A STUDY OF
THE CANE SUGAR DIFFUSION PROCESS

by

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This work was carried out in the Research and Development Department of Huletts Sugar Ltd. I was given assistance by members of this Department in carrying out the experimental part of this project, which is formally referred to in the Acknowledgements. Otherwise, unless specifically indicated to the contrary in the text, this thesis is my own original and unaided work.

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ABSTRACT

A mathematical model has been developed to represent the extraction of sugar from sugar cane in the diffusion process. As a consequence of the structure of prepared cane, extraction is postulated to occur via two first order relations in parallel .

The model was found to represent accurately the extraction behaviour observed in two different experimental configurations. Experiments on a laboratory scale were undertaken to elucidate the mechanism and basic rates of extraction. The results are consistent with the postulate that extraction takes place by a combination of washing and molecular diffusion.

The diffusion process was simulated experimentally in a fixed-bed pilot plant diffuser. Model parameter values estimated from this data provide evidence of the effect of liquid hydrodynamics on the extraction process. These results have been interpreted in terms of liquid holdup, liquid velocities and the efficiency of liquid-solid contacting.

It is shown how the model can be applied to full-scale diffusers of the moving-bed type, which will enable prediction of performance and the choice of optimum operating conditions.

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CHAPTER 1.

INTRODUCTION

Although the cane sugar diffusion process is a relatively recent innovation in the sugar industry, experiments into the extraction of sugar from cane by diffusion were carried out as early as 1886 in Louisiana (Meade, 1963). Apart from a diffusion battery system which operated in Egypt for over 50 years, the diffusion process was not applied on a large scale until fairly recently. The successful advent of the continuous sugar beet diffusion process directed attention to the possibility of using a similar system for processing sugar cane. As a result the first full-scale continuous cane diffusion system was installed some ten years ago; and since that time more than 60 diffuser installations have been commissioned throughout the cane-producing areas of the world.

The advantages of diffusion as an alternative to milling were soon realized, and catalyzed the rapid acceptance of this new extraction process. Apart from the savings accruing from lower capital, operating and maintenance costs, it has been amply demonstrated that the degree of extraction achieved via diffusion is as good as and generally better than can be obtained by milling. This is an important consideration, since the economics of sugar cane processing generally dictate that as high a level of extraction as possible should be aimed for.

Since the diffusion process represents the major development in the sugar recovery process in recent years, it is not supported by the depth of background knowledge which exists for most other unit operations in a sugar mill. As a result the development of cane sugar diffusers has followed a somewhat haphazard course.

A principal factor which hindered the application of the diffusion process to the extraction of sugar cane was the attainment of a suitable type of cane preparation. This is due mainly to the tough fibrous nature of cane in comparison with a material such as sugar beet which is easily cut into thin regular slices. It is still conceded that the type of preparation has a major effect on diffuser performance. The picture is further complicated by the variable nature of a plant product such as sugar cane.

The technology of diffusion has now reached a stage where reliable diffusion systems can be installed, based on the experience gained over a number of years. However, the state of the art is such that virtually every diffuser installation has required in situ modifications once it has been installed to improve its performance. The fact that some types of diffusers now commercially available are based on diametrically opposite concepts demonstrates that the designer is still not in a position to specify the qualities which an efficient diffuser should possess.

In order to rationalize subjective information and experience in the design and operation of diffusers, it was decided to investigate the possibility of formulating a mathematical model of the extraction process in which the parameters are in some way dependent on the characteristics of the cane being processed and the diffuser operating conditions.

The existence of such a model which provides an adequate description of extraction performance is a necessary pre-requisite for two important investigations:

1. In the design of a new diffuser installation the model could be used to find the optimum size, configuration and operating conditions, for a given cane throughput and extraction level. This would entail consideration of: the degree of cane preparation and the associated cost of cane preparation; the imbibition rate and evaporator capacity and fuel availability; the operating temperature and the extent of impurity extraction.

2. In the formulation of the optimum operating policy for an existing diffuser, it would enable prediction of extraction as a function of the control variables. This would involve the same considerations as above, but relates instead to a diffuser of fixed size; in this case an additional constraint is imposed by the necessity to maintain an efficient liquid flow system consistent with the diffuser configuration.

The primary objective of this study is to develop a mathematical model of the extraction process, which will allow diffuser extraction performance to be predicted for any given operating conditions.

In order to develop and test the model, the diffusion process was simulated on a fixed bed pilot plant diffuser. This was supplemented by information obtained from a simple laboratory extraction apparatus. Of necessity, the scope of the investigation had to be wide enough to embrace all aspects of a fundamental nature which affect performance in practice. This has resulted in a better and more basic understanding of the diffusion process.

It is patently of little benefit to look at sugar diffusion as an isolated process. Since the ultimate aim is maximum overall recovery of sucrose, this study has also included an investigation into the extent of non-sucrose species extracted in the diffusion process.

Finally, it is shown how the model can be applied to a full-scale diffuser of the moving-bed type; in this form the model can be used for design or optimization purposes.

CHAPTER 2

REVIEW OF PREVIOUS WORK

Previous investigations into sugar diffusion have been confined almost exclusively to studies of a qualitative nature. Clearly such work has limited applicability for design or control purposes, which require a far more rigorous approach. It is likely even that not all the important effects operative during the extraction process have been identified.

In the absence of a more comprehensive investigation into diffusion, the extraction system can be considered as comprising the following sub-systems:

1. Biological, dealing firstly with the nature of the cane, which is responsible for the characteristics of the bagasse to be processed, and secondly with the effect of structure on extraction from a material of vegetable origin.

2. Fluid mechanical, dealing with flow through packed beds and the attendant effects on liquid-solid contacting and mass transfer rates.

Both of these aspects have been covered per se in the literature. Together they influence the performance of sugar diffusers, but a review of published literature on diffuser performance shows that one or both have invariably been neglected.

2.1 THE STRUCTURE OF SUGAR CANE.

A pre-requisite for the understanding of diffusion is an adequate knowledge of the structure of the raw material, sugar cane. Related to this, the distribution of sucrose and impurities within the cane furnishes information on expected rates of extraction of these substances. An elementary study of the sugar cane can provide sufficient detail for the purposes of this investigation.

2.1.1 Anatomy.

Most of the information of this section has been obtained from van Dillewijn (1952). Basically, the sucrose is stored in two types of cells, the major part in large soft-walled cells called parenchyma, while a certain amount is also stored in small elongated tough tubular cells called sclerenchyma. The latter form part of the vascular bundles.

A cross section of an internode, illustrated in Fig. 2.1, shows from the outside towards the centre, the epidermis, a narrow cortex or rind, and vascular bundles embedded in a matrix of parenchyma cells. The size of these cells increases gradually towards the centre of the cane stalk. These cells are separated by small intercellular spaces, generally filled with air.

The cortex or rind consists of several layers of cells, many of which are sclerenchymatous (i.e. of the elongated thick-walled type). Many of these cells are lignified, which contributes to the hardness of the rind. The cortex varies in width and composition in different regions of the stem, as does its hardness, which depends on its fibre content.

The fibrovascular bundles are fairly widely spaced in the central part of the stalk, but towards the periphery, their number increases while their size decreases, as can be seen in Fig. 2.1. The bundles at the periphery often lie so close together as to form practically one solid ring.

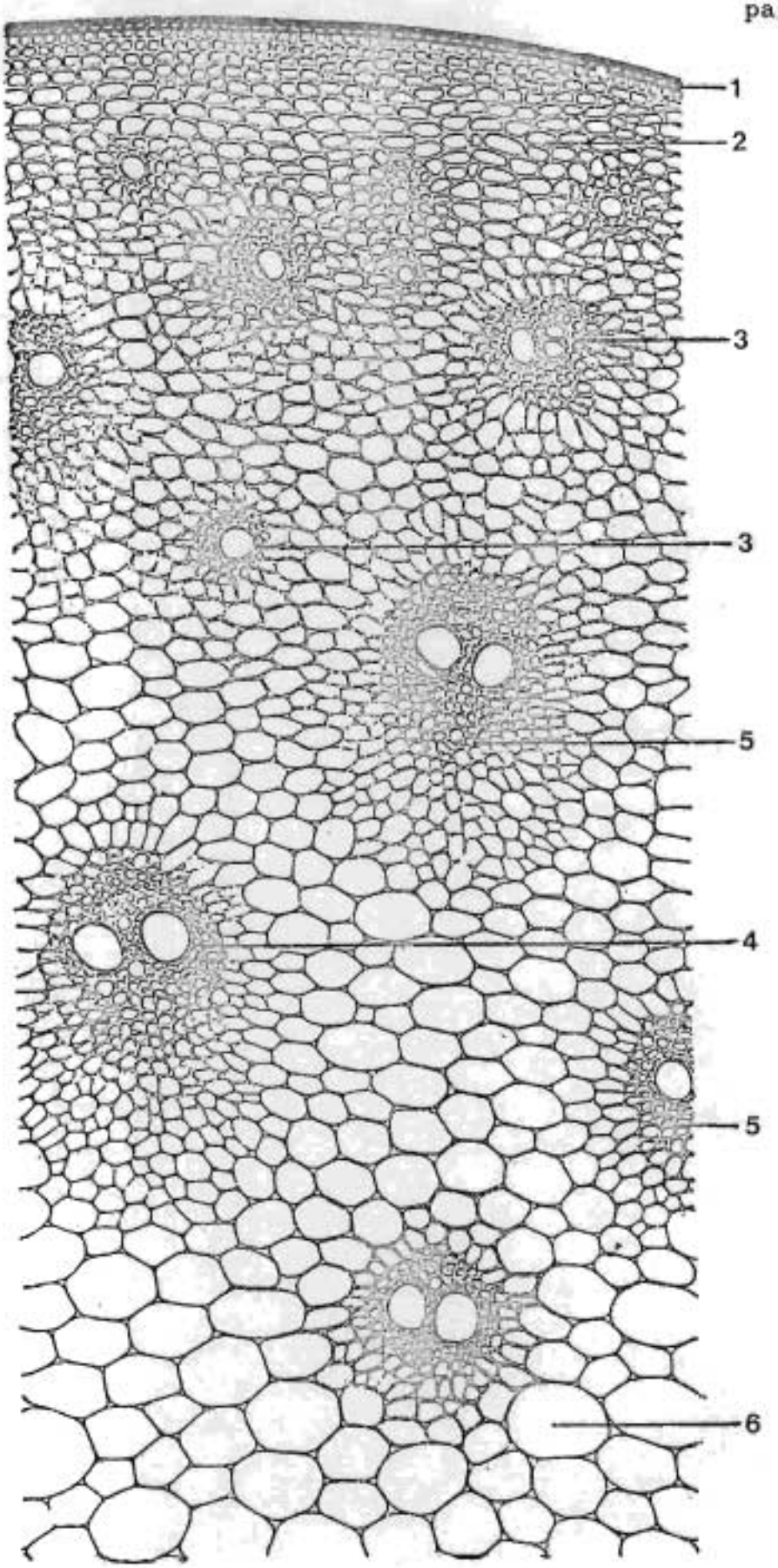


Fig 2. 1. Cross section through the outer part of an internode of a cane stalk. 1, Epidermis; 2, thick-walled cells forming the rind; 3, 4, vascular bundles of different sizes; 5, sclerenchyma; 6, parenchyma cells. From van Dillewijn (1952) after Lewton-Brain.

In the internode region, the fibrovascular bundles run more or less parallel to each other. In the node, many of them branch or bend to the leaves or buds, and moreover, since the tissue is often lignified here, the nodes are much harder than the internodes.

A transverse section of a bundle is shown in Fig. 2.2. The bundle consists of a sclerenchymatous sheath which surrounds the xylem and the phloem. The xylem consists of the protoxylem with adjacent lacuna or air tube, and two large vessels surrounded by flattened parenchyma cells. The phloem consists of sieve tubes and companion cells.

Water and nutrient from the roots flow up the large xylem vessels to the leaves. The major part of this water leaves the cane in transpiration cooling, so that the concentration of dissolved solids in the xylem is very low. The products of photosynthesis translocate from the leaves to the cells through the sieve tubes of the phloem.

Each cell is lined with a layer of protoplasm, which is so thin as to be scarcely visible under a microscope. The cell walls are composed mainly of cellulose and hemi-cellulose (pentosans). Cellulose is permeable to water and solutes, while protoplasm is permeable to water and some solutes only (Transeau et. al., 1940). Although sucrose finds its way into sugar storage cells from the xylem by diffusion, the rate of diffusion is so slow that, considering the time scale of the diffusion process, protoplasm may be considered as essentially impermeable to sucrose molecules.

2.1.2 Composition of Sugar Cane.

The composition of cane varies widely, depending on the cane variety, the region in which it was grown, climatic conditions during growth, and its degree of maturity.

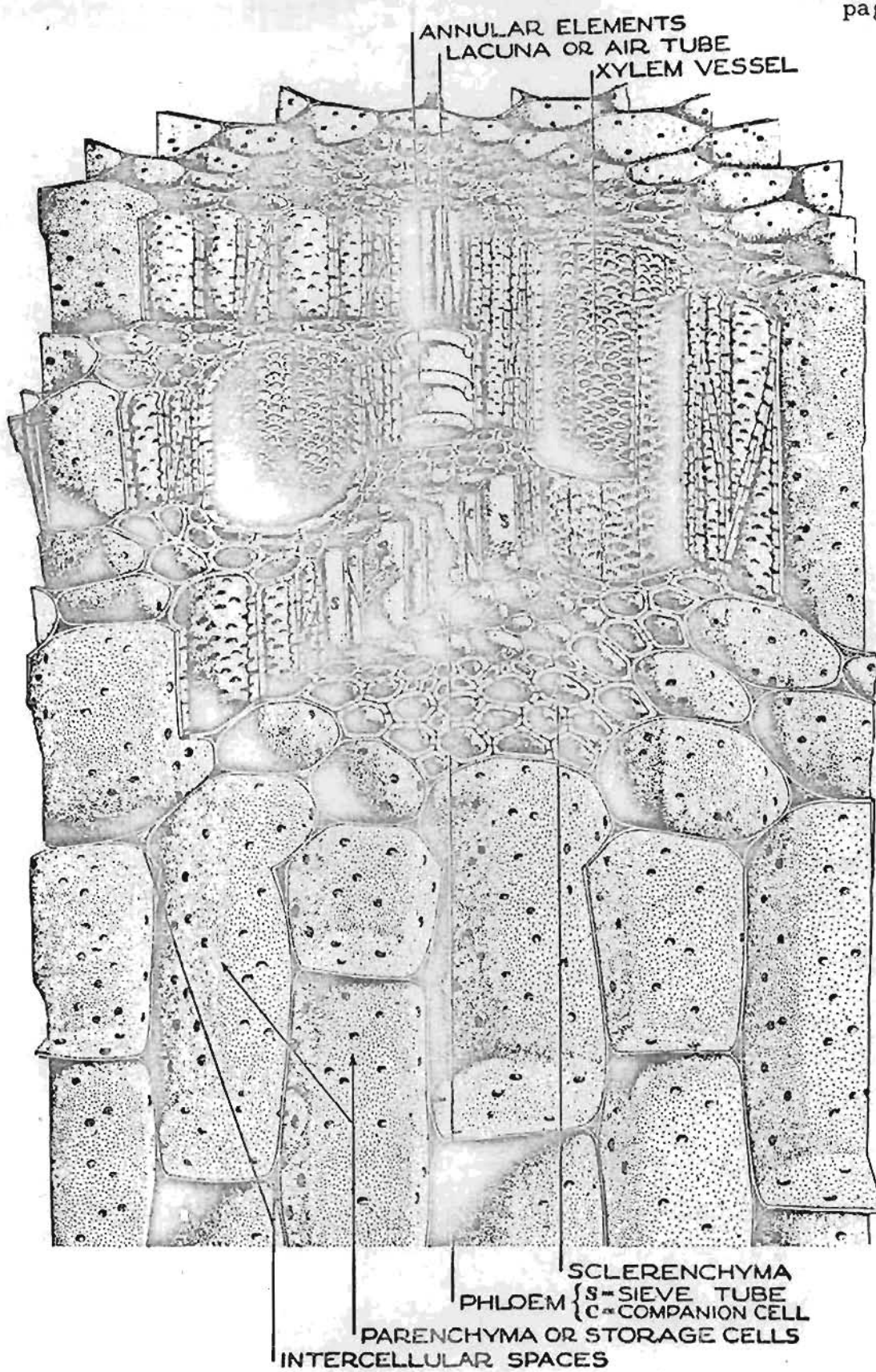


Fig. 2. 2. Three dimensional representation of a vascular bundle.
From van Dillewijn (1952) after Martin.

Average values of the sucrose and fibre content of cane processed in South Africa, calculated from figures for the last sixteen years, (Lamusse, 1971), are:

Sucrose % cane	13.41
Fibre % cane	15.39

The remainder consists largely of water.

'Fibre' is defined as the total non-soluble material in cane. It represents the fibres derived from the vascular bundles as well as the parenchyma cell wall material, termed pith. The fibre content of South African canes is generally higher than in most other parts of the world.

Other constituents of sugar cane are, in decreasing order of concentration, reducing sugars, inorganic material, nitrogenous substances, gums, fat and wax, and free and combined acids (Spencer & Meade, 1945) which account for roughly 2% of the cane.

2.1.2.1 Distribution of Sucrose.

The juice contained in the parenchyma cells of the ground tissue has a higher concentration of sucrose and a lower concentration of impurities than the juice in the fibrous schlerenchyma cells. Since the parenchyma cells occupy roughly 70% by volume of the cane, it is clear that most of the sucrose is contained in the parenchyma cells.

Similarly, more sucrose is found in the internodes than the nodes. This is illustrated by the analysis of Stubbs, reported by Deerr (1905), in Table 2.1. These figures are not representative of South African canes, and furnish a rough guide only.

Table 2.1 Analysis of Nodes and Internodes of Cane.

	Nodes	Internodes.
Brix	15.94%	17.40%
Glucose	0.13	0.94
Solids not sugar	3.21	0.96
Fibre	16.5	8.0

The rind contains relatively very little juice, of high impurity content.

2.1.2.2 The Cell Walls

These are made up of long chains of cellulose bound together by hemi-cellulose, lignin, and pectin. An idea of the relative amounts of cell wall constituents is given by figures reported by Honig (1958) shown in Table 2.2.

Table 2.2 Constituents of Cell Walls.

	% Dry solids in cane	Principal location in cane.	Principal monomer compound in polymer.
Cellulose	25-30	fibre and pith	β-D-glucopyranose in 1-4 pyranoside linkage.
Hemicellulose (pentosans)	12-18	fibre and pith	β-D-xylopyranose in 1-4 pyranoside linkage.
Lignin	8-12	fibre and pith	-
Pectin	0.005 to 0.15	Middle lamellae of cells	-
Proteins	0.1 to 0.2	cytoplasm of cells	amino acids in peptide linkage.

The pentosans in cane are of the xylan-araban type. Lignin is universally associated with the hemicellulose accompanying the cellulose in the fibre structure of plants. It has an aromatic structure, and is probably deposited in the pores. (Honig, 1958). Cellulose is insoluble, but the other cell materials are soluble or may be dispersed in the juice as colloids during processing. The gums in the juice are derived entirely from the hemicellulose of the fibre (Browne & Phillips, 1939).

The fibre content is higher in the fibro-vascular bundles than in the parenchyma. Although the parenchyma cells comprise roughly 70% of the cane by volume, its fibre content is so low that it does not amount to more than 25% of the whole quantity of dry fibre. Table 2.1 shows the fibre content of the nodes to be higher than in the internodes. Similarly, both the chemical composition and physical structure of the fibre varies according to the part of the cane stalk from which it derives.

Considerable evidence exists which indicates that natural fibre has associated with it a certain amount of water, called 'brix-free' water, which does not dilute the juice in cane. Bruijn (1963) has reviewed experimental evidence, and concludes that an amount of water, more correctly termed 'hydration water of fibre', is associated with natural cane fibre, to the extent of 20-30% of dry fibre by weight. Thus a figure of 25% will be assumed for brix-free water. Although this has been a subject for contention, the South African Sugar Industry as a whole has accepted this figure for the purposes of the new cane payment system.

2.1.2.3 The Location of Impurities.

Reducing sugars are the most abundant impurities in cane. As they are the building blocks for growth, a high reducing sugar content is found in the top portion of the cane stalk (Barnes, 1964). Likewise, the figures in Table 2.1 indicate lower reducing sugar concentrations in the nodes, which are dormant regions.

Inorganic substances are reported to constitute from approximately 0.6 - 1.0% of fresh cane stalks (Barnes, 1964). Nearly half of this is silica, while other constituents

in roughly decreasing concentration, are K, P, Ca, S, Na, Mg, Fe, and Cl, and traces of several other elements (Spencer & Meade, 1945). Most of these inorganic materials are dissolved in the sugar solution in the cells, while silica (to a large extent) and calcium are present in cell walls. This is supported by the fact that K, Na, and Cl are extracted to the same extent as sucrose in milling, while extraction of Si, Ca, Mg, and P depends on the age, variety and growth conditions of the cane (Honig, 1958). Far more of this inorganic material is associated with the vascular bundles than the parenchyma cells (Prinsen Geerligs, 1909).

Impurities which originate from the cell walls, gums, pectin, etc., were mentioned in the previous section. The existence of starch in cane has received a considerable amount of attention in South Africa because of the high starch content of South African canes, and because of the adverse effect of starch on the recovery of sucrose (Wood, 1962). It is formed by the condensation of glucose, and consists of a mixture of two polysaccharides. The major component, amylopectin, amounts to from 75 to 85% of starch, and has a highly branched structure; the remainder, amylose, is an essentially unbranched polymer. Both contain 1,4-α - D -glucopyranoside chains. Starch exists almost entirely as water - insoluble granules, and constitutes a reserve carbohydrate food. The starch can be readily transformed by plant enzymes into metabolites such as sucrose and hexose phosphates, depending on the metabolic requirements of the plant. (Manners, 1968).

Wood (1962) has demonstrated that different canes show considerable differences in starch content, which depends largely on the cane variety, and the stage of growth. He reported that most starch is located in the top 2 to 3 feet of the cane stalk, and that the nodes contain 3.5 to 4 times more starch than the internodes. Starch is present in very small quantities in each cell, but more starch is associated with the bundles than the parenchyma cells.

The structure of starch granules is affected by elevated temperatures, which facilitate its extraction. This is discussed at greater length in section 2.8.

The surface of the cane stalk is coated with a thin layer of wax, often more concentrated in the nodal regions. Traces of fatty matter, present as fatty acids and sterols, are present in the interior of the cane stalk (Barnes, 1964). It has been stated that the external waxy deposit acts as a protection against excessive evaporation of moisture from the surface of the stalk

2.1.3 Discussion

It has been shown that considerable differences in the physical structure are present in different regions of a cane stalk. Apart from the non-uniform structure, it is evident that the distribution of sucrose in cane is likewise non-uniform. The juice contained in the parenchyma cells has a higher concentration of sucrose, and a lower impurity content than that associated with the fibrovascular bundles and the nodal regions.

Furthermore, it is clear that different canes manifest significant differences in both physical structure and chemical composition. This is a well-known characteristic of materials of vegetable origin.

2. 2. CHARACTERISATION OF BAGASSE

It has been recognised that the degree of preparation of bagasse has an important influence on the performance of diffusers (e. g. , Foster & Shann, 1968 ; Silver, 1968). A consequence of the variability of cane is that the same method of preparation applied to different consignments of cane can lead to vast differences in the nature of prepared bagasse. It is necessary, therefore, to have some quantitative means of assessing the bagasse entering a diffuser.

The nature of bagasse is such that it is not amenable to normal methods of particle size analysis which have been applied to more uniform particulate systems. In the previous section, it was shown that the cane stalk is heterogeneous both with respect to structure and sucrose distribution. Size reduction of cane particles in the shredder is achieved by tearing of the soft tissue between the tough fibres, resulting in a fibrous mat of particles. The action of the 1st mill subsequently on the cane differs from that of the shredder, being one of cutting and squeezing. The 1st mill is not as efficient as regards size reduction, generally only larger particles suffering appreciable reduction in size. Subsequent to the squeezing action of the 1st mill, where a large fraction of juice is removed, the bagasse particles expand somewhat, resulting in a significant fraction of particles with some degree of internal porosity.

In spite of intensive preparation, the non-homogeneity of the cane stalk manifests itself in prepared bagasse as well. The larger particles are rough and flattened. These larger particles originate from the rind and nodal regions of the cane, i. e. , those parts which are toughest and most resistant to breakage.

The small particles can be roughly classified into pith and fibre. Particles of intermediate size are comprised of both pith and fibre, and have irregular shapes. It is evident that bagasse consists of particles of a wide range of shapes and sizes, and methods of characterising bagasse have been restricted to simple laboratory tests.

Foster and Shann (1968) reported that Behne first applied sieve analysis to measure particle size. He expressed his results in terms of a 'fineness factor', which is defined as:

$$\text{fineness factor} = \frac{1}{100} \sum \frac{w_i}{x_i} \qquad (2.1)$$

where w_i is the percentage by weight retained on the ith screen, and x_i the corresponding sieve aperture. As pointed out by Foster and Shann (1968), this may be considered as essentially a measure of surface area.

Foster and Hill (1966) reported sieving results in terms of an average particle thickness, d_{PT}, defined as:

$$d_{PT} = \frac{1}{100} \sum w_i x_i \qquad (2.2)$$

where x_i refers to a nominal average size of particle retained on the ith screen. They were able to correlate extraction in a pilot plant diffuser in terms of this quantity, lower values of d_{PT} resulting in higher extractions.

A measure of the availability of juice in prepared cane by means of a simple cold leaching test was first reported by Payne (1960). The ratio of sucrose extracted in the leaching test to the total sucrose was termed the 'leachability index', subsequently referred to as the displaceability index (DI). Although Payne refers to DI as a "direct measure of preparation", the results depend on the duration of the leaching test (equilibrium is only attained after a few hours), and this method does not account for variations in shape and size of the particles. DI is nonetheless a useful quantity, and its use has been reported by Aldrich and Rayner (1962), Foster and Shann (1968) and Markham (1969).

Similar attempts at characterisation of preparation in milling studies have also involved DI and sieve analysis. Pastega(1971) recently reported measurements of average particle thickness (from sieving) and 'pol in open cells', (similar to DI) which he related to extraction obtained in a hydraulic press. A comparison between the two methods showed a considerable dependence on cane type, which was also evident when each of the two methods was compared with extraction values. Changes in cane type or quality can affect not only the physical sizes of particles, but also their microstructure and the distribution of sucrose. DI would account for the two latter aspects, while sieve analysis would not.

Henderson (1970) reported attempts to relate the results of sieving tests on shredded cane on a routine basis to 1st mill extraction. No correlation was found, although 'pol in open cells' showed a better correlation with 1st mill extraction. Therefore he concluded that sieve analysis is too sensitive and technique - dependent to be applied on a routine basis. No indication of the reproducibility is given which might support what is otherwise a subjective conclusion. In that case, his findings could be interpreted as indicative of the fact that DI is a more suitable parameter for milling control. This is substantiated by the results of Pastega (1971). Pastega also pointed out that the long fibrous particles in shredded cane can lead to low sieving efficiencies. This is likely to be more serious with shredded cane than 1st mill bagasse.

It should be remembered that particle size from sieve analysis and DI are measures of different properties. The former yields a physical dimension, while the latter relates to the ease with which sucrose can be extracted in relation to the structure of prepared cane on the micro-scale. It is to be expected that DI would be important in both milling and diffusion; however, in diffusion the physical size also has direct significance through its effect on the hydrodynamics of liquid flow through a bagasse bed.

One further method of characterising preparation has been reported by the Australian Sugar Research Institute (Anon. 1958). The bulk density of bagasse, measured after pressing at 15 psi for five minutes, is claimed to provide a simple, consistent, and reproducible

measure of cane preparation. Bulk densities were related to the 'fineness figure' determined by sieving, and were shown to provide a reasonable correlation. However, Sohn and Moreland (1968) have shown that for log-normal size distributions, to which bagasse particles approximate (see section 5.1), packing densities are independent of particle size, but are functions of shape and size distribution only. Thus it is likely that bulk densities will be similarly affected.

The use of bulk densities has not found widespread use, but Crawford (1970) has recently advocated its use for assessment of shredder performance. However, he reports that results depend on the fibre content; this could perhaps be overcome by the use of fibre density instead.

It is clear that relatively few methods of characterising preparation have been developed, all of which are indirect methods whose results depend on the exact techniques employed. It is important that consideration be given to the properties which these indirect methods represent. From the point of view of diffusion, it appears that DI can furnish a good measure of the effect of the micro-structure of bagasse on extractability, while sieve analysis, although not as reliable, should provide a measure of the properties of bagasse which influence bagasse bed characteristics on a macro-scale.

2.3 FLOW THROUGH PACKED BEDS

The mechanics of liquid flow through a bagasse bed will affect the rate of mass transfer between bagasse and liquid. Thus in formulating a model of the extraction process, it is important that this be taken into account. Considerable attention has been given in the literature to the modelling of flow through packed beds, which attempts to account for the non-ideal flow, and has relevance for the liquid flow - bagasse bed system.

The assumption of plug flow considerably simplifies the analysis of the processes occurring in flow through porous media. In practice, this is an over-simplification. Mixing of the flowing liquid occurs, and the resulting dispersion affects performance. In mass transfer operations, this implies a reduced driving force for mass transfer. Greenkorn and Kessler (1969) have enumerated a variety of mechanicms of dispersion which occur on a macroscopic scale, and which may contribute to the overall observed dispersion. Most of these mechanisms defy accurate mathematical description, and simplifying assumptions are required to furnish a model which is not so complex as to be completely intractable.

The nature of bagasse is such that increased dispersion maybe possible due to the particle size distribution, combined with complex shapes. Further, the fibrous nature of the particles leads to an interconnecting network, which together with significant particle porosity will promote higher values of static holdup. Kyan et.al. (1970) have reported the existence of considerable regions of stagnant liquid in fibrous beds, an effect without parallel in a granular bed.

Flow models of interest can be arbitrarily classified as :-

i. Microstructure models, which consider flow in a capillary tube or other simple structure, which it is assumed, approximates fairly well to part of the porous structure. The results then need to be combined and averaged in such a way that comparison with experimental behaviour is possible.

ii. The axially dispersed plug flow model, or more simply, the dispersion model, which assumes that mixing can be described by a diffusional process superimposed on plug flow. Deviation from plug flow is accounted for by a quasi-Fickian flux term, $-D\,\delta c/\delta x$, where D is the axial dispersion coefficient.

iii. Mixing cell models, which represent the flow through a packed bed by a number of equal sized mixing tanks. Flow is characterised by the number of mixing cells, j, in series; the extreme cases are plug flow ($j \to \infty$) and complete mixing ($j=1$).

iv. Statistical models, which attempt to account for the microscopic nature of the flow process. In contrast to the continuum models, they take account of the discontinuous nature of the flow paths, by considering elements within the bed whose properties are represented by statistical distribution functions.

In a sense, the flow mechanisms should be considered in conjunction with the mass transfer process involved. Only in this way can the extent of their contribution to overall performance be assessed.

2.3.1 Micro-structure Models.

The geometry of the micro-structure in a packed bed is complex; flow occurs in the pores and void spaces in and between the solid materials. One method of attack is to consider flow in a capillary tube or other

simple structure, which it is assumed approximates fairly well to part of the porous structure. These models provide some insight into the mechanism of flow, and form a base from which other models are derived.

Micro-structure models have recently been reviewed by Nunge and Gill (1969). Most effort has been directed to flow in circular channels; the equation describing the concentration, c, of a tracer injected into laminar flow in a tube is:

$$\frac{\delta c}{\delta t} + u(r) \frac{\delta c}{\delta x} = D_m \left[\frac{\delta^2 c}{\delta x^2} + \frac{1}{r} \frac{\delta}{\delta r} \left(r \frac{\delta c}{\delta r} \right) \right] \qquad (2.3)$$

where D_m is the molecular diffusion coefficient, u(r) the parabolic velocity profile, and r the co-ordinate in the radial direction. Taylor (1953) showed that this process could be represented by a one-dimensional equation:

$$\frac{\delta C}{\delta t} + U \frac{\delta C}{\delta x} = D \frac{\delta^2 C}{\delta x^2} \qquad (2.4)$$

Radial gradients are ignored, and U and C refer to average values across the tube radius. D now represents an effective dispersion coefficient, which has been related to U, the tube radius, and D_m (Taylor 1953, Aris, 1956), on the assumption that the dispersion processes are linear, and ultimately yield a Gaussian response.

The work of Gill and co-workers has been summarised by Gill and Nunge (1969). This includes an analytical solution to equation (2.4) for both step and slug inputs; the effect of different boundary conditions and the corresponding conditions under which the results hold (Gill and Ananthakrishnan, 1966); and the effect of developing velocity fields. Work on turbulent flow is discussed; in this case material is dispersed by the additional mechanisms of eddy diffusion, and D is therefor smaller than in laminar flow.

The advantage of these models is that other effects can be incorporated relatively easily. Buoyancy effects have been analysed by Reejhsinghani, et.al., (1966), while capacitance effects have been incorporated by Turner (1958). Aris (1959) extended Turner's analysis, and showed that stagnant regions can increase overall dispersion by up to eleven times. Dayan and Levenspiel (1968) extended the Turner model to include the effect of adsorption, which also contributes to the dispersion.

Since flow through porous media is too complex to be described by any single model, micro-structure models consider a few effects, and neglect, or minimise, others. The difficulty lies in combining micro-structure units in such a way that they represent flow in a packed bed. However, these models enable an estimate of the importance of the various mechanisms of dispersion, and show how they contribute to the overall observed dispersion. Turner (1959) and Dayan and Levenspiel (1968) see the usefulness of their models in comparing different systems, in order to obtain estimates of effects such as adsorption.

2.3.2 The Dispersion Model.

The dispersion model has been widely employed to represent mixing in packed beds. It assumes that mixing can be considered as a diffusional process superimposed on plug flow. Dispersion occurs in both the longtitudinal and radial directions, but in practice radial dispersion is less important and is often neglected. Then the dispersion model is described by equation (2.4), where D is again defined as an effective axial dispersion coefficient. The generalisation of the Taylor equation (2.4) for flow in a tube to flow in packed beds is an oversimplification of a complex flow system, but has proved an effective method of characterising fluid mixing.

The results obtained from integrating equation (2.4) depend on the boundary conditions used. These are discussed by Gunn (1968), who states that a lack of knowledge of the mechanics of dispersion makes the choice of the correct boundary conditions difficult.

Evaluation of dispersion coefficients has been made by comparing the response to sinusoidal or pulse inputs with the predictions from the dispersion model. Chung & Wen (1968) and Gunn (1968) have reviewed a large amount of experimental work. Dispersion results are generally correlated using the Peclet number, Pe ($=d_p$ U/D) in terms of the Reynolds number, Re. The Schmidt number is unimportant when liquids are considered, except at very low flow rates (Gunn, 1968). A large amount of scatter is evident in the experimental data; according to Gunn and Pryce (1969), a significant part of the scatter can be explained on experimental observations that re-packing a bed and repeating the determination of Pe shows a standard deviation of 0.15 Pe. Thus the relative orientation and arrangement of particles, even at the same porosity, affects dispersion.

For liquid phase flow, values of Pe are fairly close to 1, while dispersion in the radial direction is of the order of one tenth that in the axial direction.

Values of Pe for gas flow are close to 2, which is the value predicted by the mixing cell model. Deans & Lapidus (1960) and Gottslich (1963) have shown that the existence of stagnant regions of liquid within the packing can explain the discrepancy between values of Pe in liquid and gas flows.

In a review by Turner (1964), the effect of dispersion on chemical conversion is discussed. Generally dispersion has a negligible effect, except for large deviations from plug flow, or for fast reactions in very short beds. Similarly, its effect on drying processes is important only in short beds. Experimental results in beds of ion exchange resin beads prompted Turner to state "it is unusual for longitudinal dispersion to be an important factor when kinetic or mass transfer rates are involved, but when the latter are rapid, and the bed is shallow, the longitudinal dispersion must be taken into account". This is supported by Carberry (1958) who analysed the effect of axial dispersion on first order rate processes. He found that the effect of axial mixing was negligible in beds of practical aspect ratio (bed height / particle diameter).

2.3.3 Mixing Cell Models.

A number of equal sized mixing tanks is sometimes used to represent the flow through a packed bed. This visualises a packed bed a consisting of flow paths between particles connecting the interstices, where each of the interstices is treated as a perfectly mixed region. This model describes satisfactorily the relatively small deviations from plug flow which occur in packed beds of sufficient length (Levenspiel, 1962).

Flow in this system is characterised by the number of cells in series, j. Kramers and Alberda (1953) showed that j is related to the dispersion coefficient of the dispersion model by:

$$\frac{1}{j} = \frac{2D}{UZ} \qquad (2.5)$$

where Z is the bed height. They found that this relation holds only for $j > \sim 10$. If each mixing cell is taken as equivalent to the dimension of one particle, then

$$j = \frac{Z}{d_p} \qquad (2.6)$$

Equations (2.5) and (2.6) can be combined to give:

$$Pe = 2 \qquad (2.7)$$

From equation (2.7) it can be seen that the mixing cell model predicts dispersion coefficients proportional to velocity, which is in accordance with experimental data, except at very high flow rates (Nunge & Gill, 1969). However, both Gunn (1968) and Nunge & Gill (1969) consider the implied physical process to be unlikely. Although Shulman & Mellish (1967) observed turbulent mixing at the junctions between packing elements, Nunge & Gill report other observations that fluid streams retain their identity at the junctions.

For j great enough, mixing cell models predict results very close to those of the dispersion model. In this case, the mixing cell model has the added advantage of computational simplicity compared to the dispersion model (Bischoff, 1966).

2.3.4 Statistical Models.

Models of the dispersed plug flow type ignore the microscopic nature of the flow process, and assume that dispersion occurs in one continuous medium. In fact, dispersion occurs in a medium which exhibits abrupt changes. Greenkorn & Kessler (1969) point out that precise mathematical description of the micro-structure in a porous medium is impossible, and summarise various attempts to represent the properties of the medium by stochastic models.

Typical of this approach is the work of Saffman (1959). He proposed a model consisting of randomly mixed straight capillaries of given length and diameter. Several capillaries start and finish at one junction, and the orientation of the capillaries to the direction of flow is random. Laminar flow and a pressure gradient linear with distance is assumed. A random walk was taken with this model, assuming successive steps to be statistically independent. Axial and radial dispersions were then calculated, and fair agreement with experimental data was obtained.

The amount of work reported on statistical models is meagre. Although the results are capable of representing experimental data, they rely heavily on experiment for the evaluation of the necessary parameters; they have not been developed to a stage where they may be used to predict dispersion in porous media.

2.3.5 Capacitance Effect.

Experimental evidence of Shulman et al (1955) and others points to considerable liquid stagnancy, which is not accounted for by the dispersion model. Similarly, it has been found that in some cases the dispersion model is incapable of representing impulse response measurements in packed beds, due to excessive 'tailing' of the residence time distribution curve. This is a manifestation of the capacitance effect due to the presence of stagnant liquid within the bed. This effect is more pronounced with liquid than gas flow (Bischoff,1966).

due to the lower molecular diffusivities of liquids. As a result, a number of workers have modified flow models to incorporate the capacitance effect, in micro-structure models (Turner, 1958; Aris, 1959), plug flow models (Hoogendoorn & Lips, 1965; Hochman & Effron, 1969), mixing cell models (van Swaaij et al, 1969; Levich et al, 1967; Deans and Lapidus, 1960), and the dispersion model (van Swaaij et al, 1969; Gottelich, 1963; Bennett and Goodridge, 1970).

Hochman & Effron (1969) proposed a model similar to that of Hoogendoorn & Lips (1965), termed the 'crossflow model'. This model assumes that a fraction, Φ, of the liquid flows in plug flow through the bed, while the remainder, $1 - \Phi$, comprises stagnant liquid pockets; exchange of material between the 2 fractions is represented by an exchange coefficient, k. The model is represented by the following equations:

$$\Phi \frac{\delta C}{\delta t} + U \frac{\delta C}{\delta z} + k (C - C') = O \qquad (2.8)$$

$$(1 - \Phi) \frac{\delta C'}{\delta t} + k (C' - C) = O \qquad (2.9)$$

Both Hochman & Effron (1969) and Hoogendoorn & Lips (1965) found that values of Φ were close to $\frac{h_D}{h_T}$, the ratio of dynamic to total liquid holdup (and hence $1 - \Phi$ = static holdup/total holdup). They found also that this model fitted residence time distribution curves better than the dispersion model.

Van Swaaij et al (1969) and Bennett & Goodridge (1970) included the dispersion term $D \frac{\delta^2 C}{\delta z^2}$ on the righthand side of equation (2.8) to account for longitudinal dispersion in the plug flow phase.

As a first approximation, van Swaaij et. al. assumed $\Phi = h_D / h_T$, and used experimentally determined values of Pe obtained using non-wettable packings, where stagnant regions are absent. They obtained good agreement between experimental and predicted impulse response curves.

In a subsequent paper, Villermaux and van Swaaij (1969) obtained values of k and Φ from a curve fitting procedure. They showed that Φ increases from 0.6 to 1.0 as the flow rate increases. Thus static hold-up, h_S ($= (1-\Phi)\, h_T$) decreases from the value of h_S = adherent holdup at zero throughput to $h_S = 0$ at high flow rates, depending on packing size. Good agreement of the model with experimental data was demonstrated, which was not possible with the ordinary dispersion model. They showed in addition that the phenomenon of tailing is independent of axial dispersion and is due entirely to the effect of stagnant liquid regions.

Bennett & Goodridge (1970) confirmed these results, and also demonstrated that the static holdup is less than the usual definition of h_S = adherent holdup. (This is discussed further in section 2.4).

Gottslich (1963) applied a similar analysis, but assumed instead that exchange with stagnant regions occurs only by molecular diffusion according to Fick's Law. This requires some assumption regarding the geometry of the stagnant zones, in order to formulate the rate of transfer with the stagnant liquid. Gottslich assumed the stagnant liquid to exist in a film surrounding the particles. He compared the calculated film thickness from this model with the diffusion film thickness calculated from mass transfer data (equivalent to D_m / mass transfer coefficient). He showed that if the distribution of film thicknesses is taken into account, these two kinds of film are the same. Again, it is illustrated that axial dispersion has a negligible effect on liquid phase mass transfer experiments.

Rosen (1952) and others have investigated the case where diffusion from the interior of porous particles occurs. Glaser & Litt (1963) showed that in this case, a significant contribution is made by diffusion from inter- as well as intra- particle fluid.

This review has covered only part of the evidence which confirms that dispersion models per se are inadequate for describing the flow through packed beds when stagnant pockets of liquid exist within the bed. This is particularly true at low flow rates. The correspondence demonstrated between the amount of stagnant liquid and the static holdup indicates that the major part of the static holdup is non-free flowing. Further, the results of Gottslich (1963) and others imply that the capacitance effect has a much greater effect on mass transfer in packed beds than axial dispersion alone. This will be substantiated further in section 2. 5 where exchange with stagnant zones is shown to play an important part in washing processes in packed beds.

2. 3. 6 Additional Effects.

Nunge & Gill (1969) have reviewed instabilities which may be induced by differences in viscosities and densities between displacing and resident liquids. This can result in decreased or increased mixing in the liquid phase (the latter is caused by gravity or viscous 'fingering'). Buoyancy effects have been covered by Reejhsinghani et al (1966).

Liquid flow in diffusers involves the downward displacement of liquid. In this case, if the displacing phase is less dense, or at a higher temperature, the difference in density is termed 'favourable', resulting in a reduction in overall dispersion. This situation generally obtains in moving bed diffusers, and was realised in the pilot plant experimentsof this investigation.

The wall effect becomes important if the ratio of tower to packing diameter, d_T/d_p is too low. It has been reported that d_T/d_p should be >10, (Bischoff 1966), or >12 (Gunn 1968), for variations in fluid velocity to be negligible. However Schiesser and Lapidus (1961), observed a higher than expected wall flow even at a value of d_T/d_p = 16. Consideration of the extent of increased porosity near a wall leads to the conclusion that this ratio need only be greater than 8. Stanek and Kolar (1968) confirmed the findings of Schiesser and Lapidus that a higher than expected wall flow occurs for d_T/d_p >8, which cannot be explained only on the basis of increased porosity at the wall.

2.3.7 Discussion

The development of a model of the diffusion process must of necessity result in a form which is mathematically tractable if it is envisaged that such a model find practical utility. With this in mind, it is clear that the use of statistical models of the type described above appears unattractive. It is apt at this stage to quote Turner (1958) who stated that 'the nearer the chosen model approaches actuality, the more difficult the analysis and the more laborious the calculations'.

Thus the development of a model is guided by two conflicting considerations - accuracy and practicability. In this respect, it is desirable to include only those effects which play a significant role and neglect or minimise others. Thus evidence has been presented which shows that unless very shallow beds are employed or extremely fast mass transfer occurs, axial dispersion has a negligible effect on mass transfer performance (Carberry, 1958; Gottslich, 1963; Turner, 1964). However it would appear that with liquid phase flow, the capacitance effect due to the existence of stagnant liquid can have an important effect. This has added significance in this case, since it has been pointed out that such static liquid may be higher in beds of bagasse than in beds of the more conventional packing materials.

2.4 LIQUID HOLDUP.

Liquid holdup may be considered as a basic dependent variable of packed bed operation. Its importance in mass transfer processes is shown by the number of attempts made to expain and correlate mass transfer performance in terms of liquid holdup (eg. Furnas & Bellinger, 1938; Davidson 1959, Shulman et al 1963). Further, liquid holdup measurements furnish information on the fluid mechanical properties of packed bed systems; Sater & Levenspiel (1966) have reported that axial dispersion is a function of the same factors that determine holdup, and holdup can be used to infer information on the degree of liquid-solid contact (Hochman & Effron, 1969).

The concept of the total liquid holdup as consisting of 2 components, the dynamic and static holdups, has been widely accepted. This may be represented as:

$$h_T = h_D + h_S \qquad (2.10)$$

The total holdup represents the total amount of liquid in a packed bed at a given operating condition. The dynamic holdup is a measure of the amount of flowing liquid, and the static holdup, the difference between h_T and h_D, therefore represents liquid retained in the bed which is not part of the flowing liquid stream.

2.4.1 Significance of Dynamic and Static Holdups.

The total liquid holdup is a clearly-defined quantity which needs no further elaboration. However, there appears to be some difference of opinion as to what the dynamic and static liquid holdups represent. Shulman et al (1955) pointed to evidence of considerable liquid stagnancy in a packed bed. Their observations on the flow of dye injected into a packed bed showed semi-stagnant pockets of liquid; the splashing and random motion of liquid over the packing surface deposited or removed dye from the pockets by a slow random dilution process. Van Swaaij et al (1969) identified static holdup with stagnant zones, which is valid only if the definition of h_S provides a realistic measure of stagnant liquid regions. The particular definitions of h_D and h_S will affect the physical significance of these measured quantities, as well as their numerical values.

The generally employed definitions are:

1) the dynamic holdup (also referred to as the operating holdup)is the amount of liquid which drains from the column for a specified period after the inlet flow is cut off.

2) the static holdup is the amount of liquid retained by the packing after draining for a specified time.

These definitions have a number of disadvantages; firstly, h_S (and hence h_D) is affected by the conditions in the packed bed before draining (Turner & Hewitt, 1959), and by end effects for small packings and low bed heights (Dombrowski & Brownell, 1954), while the time allowed for drainage also affects measured values. But more important is the fact that the physical significance of h_D and h_S defined in this way is not clear. With this definition of h_S, h_S is independent of flow rate (Shulman et al 1955), and dependent only on the liquid-retaining capacity of the bed.

Static liquid is retained at the points of contact between particles. Bennett & Goodridge (1970) pointed out that during percolation, the same points of contact must serve as transfer routes from one packing element to another, and so the flowing liquid activates some of the hitherto static regions. Thus during percolation, there is an effective reduction in the amount of static liquid. Both Glaser & Lichtenstein (1963) and Villermaux & Van Swaaij (1969) maintain that at high enough flow rates, static liquid in a packed bed disappears entirely. Gelbe (1968) has proposed that h_S has the value as defined above, which he terms the "adherent holdup", at zero flow rate, but that due to the influence of the flowing film, the value of h_S decreases with increasing flow rate, approaching zero at very high flow rates. Villermaux & van Swaaij (1969) fitted their dispersion model, incorporating exchange with stagnant zones, to residence time distribution measurements, and found that the static liquid showed the same behaviour as proposed by Gelbe (1968).

Thus the concept of a static holdup which decreases with flow rate appears a more realistic picture of actual physical flow conditions. The value of the dynamic holdup is naturally dependent on the definition of h_S.

2.4.2 Correlation of Liquid Holdup Measurements.

A large number of experimental investigations of liquid holdup in packed columns have been reported. Methods of measurement have been outlined by J. E. Buchanan (1967) and generally involve measuring the weight of the column dry, during percolation, and after draining, or measuring the volume of liquid which drains from the column on interrupting the inlet flow.

Shulman et al (1955) and Morton et al (1964) appear to have published the only measurements of h_T of any note. They resorted to empirical correlations in terms of L and d which are specific for certain types of packing. However, most interest has been directed towards the separate components, h_D and h_S.

2.4.2.1. Dynamic Holdup.

Two attempts have been made to predict h_D from from theoretical considerations. Davidson (1959) proposed a model for flow in a random packing. He considered the packed bed as a number of flat surfaces, inclined to the horizontal with an equal probability of all angles, and an equal probability of all lengths up to a maximum given by the particle dimension. Liquid was assumed to run down the surfaces in laminar flow, and at the end of every surface to be fully mixed before starting down the next. This allows the calculation of an average film thickness, which when multiplied by the wetted area, yields the holdup.

Buchanan J. E. (1967) recognized the existence of 2 limiting flow regimes, gravity-viscosity and gravity-inertia regimes. The former was dealt with by an analysis similar to that of Davidson (1959). For the second regime, the assumption was made that energy losses due to viscous drag are negligible. The flow is interrupted at intervals by steps, where the liquid loses a fraction of its kinetic energy before proceeding down the next slope. The results for the 2 flow regimes were then combined in order to represent flow over the full Reynolds Number range.

Even though Buchanan's equation for h_D requires the evaluation of 2 empirical constants, both these proposed equations do not represent experimental measurements as well as purely empirical correlations. Published correlations are summarized in Table 2.3.

Table 2.3 Published Correlations of Dynamic Holdup Measurements

Reference	Correlation	Range of Applicability	Remarks
Furnas and Bellinger (1938).	$h_D = A L^n$	$7.0 < L < 155$	A & n depend on packing size and shape. $0.54 \leq n \leq 0.74$
Josser and Elgin (1943)	$h_D = h_{DW}\, \mu^{0.1} / \rho^{0.78}\, a^n$	$67 < L < 400$	$h_{DW} = h_D$ of water at 20°C $n = f(L)$
Otake and Okada (1953).	$h_D = A_1\, Re^{0.676}\, Ga^{-0.44}\, (ad)^n$	$10 < Re < 2000$	A_1, A_2, & n depend on type of packing.
	$h_D = A_2\, Re^{0.51}\, Ga^{-0.44}\, (ad)^n$	$10^{-2} < Re < 10$	d = nominal packing diameter.
Shulman et al (1955b)	$h_D = A L^{0.57} \mu^{0.13} \rho^{-0.84} (\sigma/73)^{n=.262 \log L}$	$16.7 < L < 167$ 1" packings only	A & n depend on packing shape.
Davidson (1959).	$h_D = 1.217\, Re^{1/3}\, Ga^{-1/3}\, (ad)$	laminar flow	$Re = \frac{2\pi l_r}{a\mu}$ $Ga = \frac{g d^3 \rho^2}{\mu^2}$ d = size of packing element.
Varrier and Rao (1961)	$h_D = 7.12\, Re^{0.67}\, Ga^{-0.44}$	$5 < Re < 510$	$d = (1/N)^{\frac{1}{3}}$ N = no. of packing elements / unit volume.
Mohunta and Laddha (1965)	$h_D = 16.1 \left(\frac{\mu U^3 N}{\rho g^2}\right)^{0.25} (Nd)^{-0.5}$	$7.1 < L < 387$	N = no. of packing elements / unit volume d = equivalent spherical diameter.
Buchanan, J.E. (1967)	$h_D = 2.2\,(Fr/Re)^{0.33} + 1.8\, Fr^{0.5}$	$5 \times 10^{-2} < Re < 10^3$ Raschig Rings only	d = ring diameter.
Gelbe (1968)	$h_D = 1.59\,(d_1/d_2)^{-5/9}\, E\ddot{o}^{-1/7}\, Ga^{-0.3}\, Re^n$	$10^{-2} < Re < 10^2$ Raschig Rings only	$Re = \frac{U\rho}{\mu a_T}$ $Ga = \frac{\rho^2 g d_h}{\mu^2 a_T^2}$ $E\ddot{o} = \frac{\rho g d_h^2}{\sigma}$ $d_h = 4\varepsilon / a_T$ d_1 = hydraulic radius of inner area of ring. d_2 = nominal packing diameter.
Mohunta and Laddha (1968)	$h_D = 4.67 \left(\frac{\mu U^3 a_T^3}{\rho g^2}\right)^{0.25}$	$7.1 < L < 383$ Raschig rings only.	

Shulman et al (1955) found that h_D is independent of packing material and shape, which has been confirmed by Standish (1968). This supports Davidson's theory that all random packings of a given size are equivalent to a series of sloping surfaces, and are indistinguishable from one another. However, reference to Table 2.3 shows that virtually all correlations include empirical constants which are dependent on the type of packing used, or else are specific to one type of packing. Thus these correlations lack the generality required for application to different packed bed systems.

The correlation of Otake & Okada (1953) has found the widest application for conventional packing elements for which the constants have been evaluated. Nonetheless, although Otake and Okade show data scatter of up to 20% about this correlation, data reported by Buchanan (1967) indicate greater scatter, up to 32%, while Mohunta & Laddha (1968) report an average deviation of data from this correlation of 27.7%.

Gelbe (1968) claims to have reduced scatter considerably in correlating h_D by taking into account the fact that h_S varies with flow rate. At low flow rates, the exponent on Re is 1/3, which is predicted by Davidson's (1959) theory, while at higher values of Re where viscous drag is negligible, the exponent has a value of 5/11.

2.4.2.2. Static Holdup.

Shulman et al (1955) have presented the most comprehensive measurements of h_S. They found that h_S is strongly dependent not only on the size of the packing elements, but also the shape and type of material of the packing. An empirical means of correlating their data was employed:

$$h_S = A\, d^{-n} \qquad (2.11)$$

where both A and n depend on the material and shape of the packing. Shulman et al (1955b) also investigated the effect of variable liquid properties on h_S, which they expressed as

$$h_S = A\, \mu^{m}\, \rho^{-0.37}\, \sigma^{n} \qquad (2.12)$$

where the value of A depends on the packing, m is very small (~ 0.03), and $0.2 < n < 1.0$, depending on the packing.

Standish (1968 a) reported that his measurements did not agree with predictions from Shulman's equations, and that in general, measurements of h_S on the same packings by different workers show wide discrepancies. Some of the reasons for these discrepancies were cited earlier.

Turner and Hewitt (1959) and Gelbe (1968) showed that the liquid retained in a packed bed should be dependent on the Eötvos number, which represents the ratio of gravity to surface tension forces. Van Swaaij et al (1969) showed that a reasonable correlation exists between h_S and Eö, and confirmed Gelbe's proposal that h_S is independent of Eö for Eö < 10; Gelbe (1968) states that, at low values of Eö, "the absortivity imparted by the capillary forces to the packed bed is greated than the bed's real holding capacity."

The amount of liquid retained in a bed can also be predicted from the work on residual saturation of Dombrowski & Brownell (1954), and can be used to estimate the magnitude of the end effect in beds of lower bed height.

The above discussion refers to h_S defined as the adherent hold-up. Gelbe (1968) has proposed a correlation to predict h_S assuming that it is flow rate dependent. His correlation results from an analysis of forces in conjunction with measurements of adherent holdup. He proposes 2 different correlations, applicable to the 2 regions, $E\ddot{o} < 10$, and $E\ddot{o} > 10$. The correlation however, is complicated, and still requires the use of shape factors. Qualitatively, the correlation predicts values of h_S decreasing from the value of adherent holdup at zero flow rate to zero at a high enough flow rate, and that values of h_S are lower for smaller packings.

2.5 MASS TRANSFER IN PACKED BEDS.

The published investigations into mass transfer which have relevance to this study, can be separated into 2 categories; firstly, investigations into miscible displacement processes, typical of which is the washing of filter cakes, and secondly, investigations into the conventional solid-fluid mass transfer and gas absorption systems. The former work is concerned with the behaviour of the liquid phase, and its effect on displacement efficiency, while the latter aspect is directed more towards evaluating overall rates of mass transfer. Both aspects are of interest, and are discussed in turn.

2.5.1 Liquid-liquid Displacements.

The work described in section 2.3 concerned the behaviour of liquid flowing through porous media. This section concerns studies which complement those of section 2.3, in that they consider the effect of the liquid behaviour on washing processes in packed beds.

The earliest attempt at a mathematical representation of the mass transfer between a filtrate and a percolating wash liquid in a filter cake was reported by Rhodes (1934). He proposed that removal of filtrate occurs in 2 stages; firstly a mechanical displacement, followed by a period of 'diffusion washing'. For the second stage, he assumed the effluent concentration to be proportional to the concentration in the cake at that time, which leads to a simple exponential decay for the exit concentration. This fits his experimental data well. Under the conditions of his tests, it was found that the 'equilibrium constant' is in fact constant, independent of flow rate and cake thickness.

Apart from this purely empirical approach, other investigators have utilized the dispersion model (section 2.3.2), exchange with stagnant liquid regions (section 2.3.5), or a combination of the two, to describe extraction behaviour.

Kuo (1960) assumed that the solute in a filter cake remains in a uniform stagnant film on the surfaces of the channels through which the wash liquor flows. He proposed a model based on plug flow of the wash liquor, with continuous mass exchange between wash liquor and stagnant film. The model is similar to that proposed by Hochman and Effron (1969), represented by equations (2.8) and (2.9). The model predictions were compared with some of Rhodes' data, with satisfactory results, and is a more elegant treatment. However, insufficient results are given to show how the model parameters are affected by the conditions of washing.

A similar model was proposed by Han and Bixler (1967), but assumes instead that the stagnant liquid is retained in blind side-channels between particles. Mass transport between these channels and the main flow channels is specified to occur strictly by molecular diffusion. This leads to the following differential equations:

$$\frac{\delta C}{\delta t} + U \frac{\delta C}{\delta z} = a N \qquad (2.10)$$

$$\text{where} \quad N = D_m \left.\frac{\delta C'}{\delta x}\right|_{x=0} \qquad (2.11)$$

x is the co-ordinate in the direction of the channel, and a here represents the area of the side channels in the walls of the straight channels. N is evaluated from the equation representing diffusion in the side channel:

$$\frac{\delta C'}{\delta t} = D_m \frac{\delta^2 C'}{\delta x^2} \qquad (2.12)$$

This model, however, requires a strict definition of blind side channel geometry, as well as values for a and the length of the side channels. The latter were estimated from residual saturation measurements .

Han (1967) extended this washing theory to include the effects of porosity of the solid particles as well as side channels. Again molecular diffusion is assumed as the mass transfer mechanism; in this case, N in equation (2.10) also includes diffusion from spherical particles, with an effective diffusivity lower than that in the side channels, because of the tortuosity of the paths within the particles.

An approximate solution by numerical inversion of Laplace transforms was obtained, but no experimental data were obtained. The model predicts a longer washing time for a low effective pore diffusion coefficient, quicker washing with smaller particles (a smaller diffusion path length), and quicker washing for highly porous particles (larger surface area of pores).

The dispersion model was used by Dobie (1962) to represent experimental data on small scale tests on the washing of filter cakes. He considered the cake as a bundle of capilliaries, all of which are accessible to wash liquor. Axial dispersion occurs in flow through these capillaries, represented by equation (2.4). He showed that for the case of the axial dispersion coefficient tending to infinity, the exponential decay of effluent concentration predicted by Rhodes (1934) obtains. However, it appears that the dispersion model provides a far less satisfactory representation of washing performance.

Sherman (1964) studied the washing of diacetyl from beds packed with glass spheres, non-porous dacron fibres, and porous viscose fibres. The axial dispersion model was applied to data for the first 2 packings, and the values of the dispersion coefficient, D, were obtained from the best fits of model to data. It was found that for a particular bed, D/U is constant, but that different fibre beds, with the same fibre diameter, led to widely differing values. These variations are attributed to differences in packing or formation of fibre beds, and it appears that fibre diameter does not define a bed of fibrous material. Thus a comparison between beds packed with spheres and with non-porous fibres shows that the same mechanisms of fluid dispersion apply, but that values of D/U behave unpredictably in fibre beds.

Tests on viscose fibres were undertaken to investigate the effects of porous fibre structures. It was assumed that diffusion of diacetyl within the fibres is rapid, and that equilibrium between fibre and solution exists. For this case the term

$$\frac{\epsilon_f (1-\epsilon)}{\epsilon} \quad \frac{\delta C}{\delta t}$$

was added to the left hand side of the dispersion model, equation (2.4) where ϵ_f represents the fibre porosity. The solution to the equation was shown to be capable of describing experimental results at low flow rates only. At higher flow rates, the assumption of equilibrium breaks down, as the diffusion within the fibres is not fast enough for the rapid concentration changes in the surrounding solution. In this case, tailing of the effluent concentration is evident, even over the short washing times involved.

An investigation into the displacement of calcium chloride by a sodium chloride solution from cores of sand particles is reported by Coats and Smith (1964). They employed the axial dispersion model, with allowance for stagnant zones, to represent measured effluent concentrations. They assumed a first order transfer process between stagnant and flowing liquid. The differential equations are similar to those of Kuo (1960) and Hochman and Effron (1969) but include an axial mixing term:

$$D \frac{\delta^2 C}{\delta z^2} - U \frac{\delta C}{\delta z} = \phi \frac{\delta C}{\delta t} + k (C-C') \qquad (2.13)$$

$$(1-\phi) \frac{\delta C'}{\delta t} = k (C'-C) \qquad (2.14)$$

Values of ϕ were found to be of the order of 0.9, and k was reported to be roughly proportional to the fluid velocity.

Coats and Smith showed that if the stagnant zones consist of resevoirs of volume V_s, joined to the flow region by a neck of length l and cross-section A, then

$$k = \frac{(1-\phi)\ D_m\ A}{V_s\ l} \qquad (2.15)$$

They showed, too, that if the mass transfer process is assumed to take place from a film on the pore walls, as assumed by Kuo (1960), then the thickness and diffusivity indicate that diffusion from the film would be virtually instantaneous.

In summary, the work described above demonstrates that the dispersion model alone is incapable of representing washing processes, unless some allowance is made for exchange with stagnant liquid zones. The model proposed by Han and Bixler (1967), assuming exchange with blind side-channels, appears to be generally successful in describing washing of beds of solids, on the basis of comparison with a wide range of experimental data. This reinforces the conclusions of section 2.3.7 that the capacitance effect due to stagnant liquid has a greater effect than axial dispersion. However, Coats and Smith (1964) point out that while the fact that a capacitance model matches data better than the dispersion model does not 'prove' the existence of stagnant liquid volume, it indicates that the capacitance concept is capable of explaining observed experimental behaviour. A more detailed investigation into the model parameter behaviour is necessary to verify whether such behaviour is consistent with the implications of the capacitance model. The impulse-response measurements of Villermaux and van Swaaij (1969) described in section 2.3.5 do however provide strong evidence in favour of the validity of the capacitance concept.

2.5.2 Mass transfer rates.

In order to correlate mass tranfer rates between a fluid and a solid in a packed bed, use is often made of the dimensionless parameter, j_D. It has been found that for mass transfer in packed beds,

$$j_D = A\ Re^{-n} \qquad (2.16)$$

Values of n between 0.2 and 0.7 have been reported (Ergun, 1952). The j_D factor may be written as:

$$j_D = \frac{Sh}{Re\ Sc^{1/3}} \qquad (2.17)$$

so that equation (2.16) may be re-written as

$$Sh = A\ Re^{1-n}\ Sc^{1/3} \qquad (2.18)$$

which is a more convenient form for the purposes of this discussion. Sh, Re and Sc are dimensionless numbers defined in the Nomenclature.

The j_D factor was introduced by Chilton and Colburn (Ergun, 1952) who extended the Reynolds analogy between heat and momentum transfer to include mass transfer. This is based on a continuous fluid phase; however in a bagasse bed, liquid flows in film flow over the particles below flooding conditions, while even in a flooded bed, it will be shown that the existence of a significant amount of air trapped within the bed precludes the applicability of the j_D factor.

Another packed column operation, gas-liquid absorption, can be turned to as a source of a large volume of work on mass transfer. The liquid-phase hydrodynamics parallel more closely those expected in an irrigated bagasse bed. In both cases, the mass transfer rate is expected to be a combination of a rate term and a mass transfer area. However, gas absorption deals with mass transfer at a gas-liquid interface, while sugar diffusion involves mass transfer between a solid and a liquid. The use of the j_D factor involves evaluating the total packing surface area as the mass transfer area, but if stagnant liquid regions are present in the packing, the effective mass transfer area may be reduced. In this respect, the effective interfacial area for gas absorption makes allowance for ineffective stagnant regions, and so this area and the effective transfer area in sugar diffusers may manifest similar behaviour.

The conventional picture of mass transfer through a stagnant liquid layer at a mass transfer interface has been largely discarded, and replaced by the penetration theory of Higbie (1935). This was developed for the case of absorption in a packed tower by Danckwerts (1951). Davidson (1959) has suggested that Higbie's theory is more plausible than the stagnant film theory for a number of reasons. In particular, this theory predicts that the mass transfer coefficient is proportional to $D_m^{0.5}$, which is closer to experimental findings than the linear dependence on D_m implied by the stagnant film theory. In essence, Higbie's theory pictures masses of fluid moving to the transfer surface, exchanging mass at the surface, and then mixing with the bulk of the fluid, so that a continuous renewal of the surface fluid occurs.

Both Davidson (1959) and Norman & Sammak (1963) showed that this theory leads to a dimensionless relationship, which can be expressed as:

$$Sh = A\, Re^{1/3}\, Sc^{1/2}\, Ga^{1/6} \qquad (2.19)$$

where A is a constant.

In general, it is found experimentally that the exponent on Re is higher. Semmelbauer (1967) reviewed available correlations for liquid-phase mass transfer, and arrived at the following relation:

$$Sh = A\, Re^{0.59}\, Sc^{0.5}\, Ga^{0.17} \qquad (2.20)$$

where the value of A depends on the type of packing. The values of the exponents are "preferred values", based on consideration of available published work.

Although the initial applications suggested for the penetration theory were limited to mass transfer at a gas-liquid interface, it has also been suggested that the mechanism represents transfer in a fluid adjacent to a solid-liquid interface. Thus Johnson & Huang (1956) measured rates of solution of organic solids in an agitated vessel, and found that an equation similar to equation (2.18) could be applied to the results, with the exponent on Sc varying between 0.42 and 0.53. Hanratty (1956) likewise applied the penetration theory to mass transfer between a solid wall and a liquid in tube flow, and found it could be used to represent mass transfer rates.

This is unexpected, since a condition for the applicability of the theory as given by Danckwerts (1951) is that the depth of penetration of liquid is less than the depth at which the velocity is appreciably different from that at the surface. This condition is a good assumption at a gas-liquid interface, where the velocity gradient is small. However, the velocity gradient has its maximum value at a solid surface, and it is unlikely that this condition is complied with.

Kramers & Kreyger (1956) studied mass transfer between a solid surface and a falling liquid film. From their analysis, it is expected that the mass transfer coefficient should be proportional to $D_m^{2/3}$, which is also suggested by the j_D correlation in modified form, equation (2.18).

The discussion thus far has neglected the mass transfer area, which is generally combined with the rate coefficient to yield an overall mass transfer coefficient. Shulman et al (1963) showed that differences in measured mass transfer coefficients obtained for vaporization and gas absorption in packed columns could be explained in terms of different effective mass transfer areas in the 2 cases. They showed that the ratio of the areas approximates to the ratio h_T/h_D; the effective interfacial area in gas absorption in the absence of chemical reaction is postulated to be lower due to the presence of stagnant liquid regions.

Semmelbauer (1967) reviewed published investigations into the effective interfacial area for gas absorption, a_e , and arrived at the following relation:

$$a_e / a_T = A\ Re^{0.455}\ E\ddot{o}^{0.5} \qquad (2.21)$$

The overall liquid phase mass transfer coefficient is then the product of a_e from equation (2.21) and k from Sh in equation (2.20).

Values of a_e and the total wetted area, a_w, are significantly different, with the difference more pronounced with smaller packing elements (Davidson, 1959). This can be attributed to surface tension forces, which are able to retain more liquid between smaller packing elements, resulting in increased total wetting but decreased valued of a_e. Both areas, a_w and a_e, increase with flow rate; however the ratio a_w/a_T is greater for smaller packings, while a_e/a_T displays the opposite trend. This is due to the surface tension effect mentioned above, and is accounted for by the inclusion of $E\ddot{o}$ in equation (2.21).

A number of published measurements of values of a_w / a_T are summarized by Onda et al (1959). It appears that these values show roughly the same dependence on flow rate as values of a_e / a_T. However, in considering the total wetted area, some account should be taken of the extent of stagnant liquid regions, which contribute to the observed total wetting, but may well decrease the overall rate of mass transfer.

2.6 SOLID - LIQUID EXTRACTION STUDIES.

Since sugar diffusion has relatively recently been applied on a wide scale, little work has been reported on the mechanism of extraction of sugar from cane or bagasse. However, published work on the older types of solid-liquid extraction processes provides some insight into the mass transfer processes involved in extraction from materials, which like cane, are of vegetable origin. The recovery of oil from nuts and seeds by extraction processes was the forerunner of both beet and cane diffusion systems. A large volume of published literature exists on the solvent extraction of oilseeds and also the extraction of sugar from beets; this section is concerned only with studies of a fundamental nature aimed at elucidating the mechanism of extraction of a desired solute from a material of vegetable origin.

2.6.1 One Dimensional Non-stationary Diffusion Process.

Most studies of interest have used as a starting point the theory of diffusional processes, based on the differential equation:

$$\frac{\delta c}{\delta t} = D_m \frac{\delta^2 c}{\delta x^2} \qquad (2.22)$$

For conditions of constant diffusion coefficient D_m, uniform initial concentration c_0 in the solid, uniform thickness l in the x direction, and extraction to an extract of constant concentration, c_1, this equation can be integrated to give (Osburn & Katz 1944):

$$\frac{\bar{c} - c_1}{c_0 - c_1} = -\frac{8}{\pi^2} \sum_{0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 D_m \pi^2 t}{l^2}\right) \qquad (2.23)$$

where $\bar{c}$ represents the average concentration of solute in the solid.

In order to represent diffusion through a porous solid, the following conditions must be satisfied for equation (2.23) to be valid:

1. Diffusion occurs only in the x direction, i.e. the thickness of the solid, l, is very small in relation to the surface area.
2. The concentration in the extract is constant and uniform.
3. The initial solute concentration in the solid is uniform.
4. The medium through which diffusion occurs is homogeneous and isotropic.
5. The porous solid structure is rigid and inert.

In addition, D_m should be replaced by an "effective" diffusivity, or the diffusion coefficient modified to take into account the interference of the solid structure and the tortuosity of the diffusion paths.

The series given in equation (2.23) converges rapidly, and it has been shown that for values of the left hand side of this equation < 0.7, all terms other than the first can be neglected (Osburn & Katz, 1944).

2.6.2 Extraction of Oil from Seeds and Nuts.

Fan et al (1948) studied the extraction of oil from peanut kernels, using 2 different hydrocarbon solvents at room temperatures. After less than 30 minutes extraction time, a linear plot of log extraction against time was obtained, and assuming that only the first term in equation (2.23) is significant, D_m was evaluated from the slope. It was found that higher values of D_m were obtained with the lower viscosity solvent, and that high moisture contents led to lower values of D_m.

Their results showed higher extractions than are expected from equation (2. 23). The discrepancy was explained by two factors. Firstly, because very thin slices were used, the ruptured cells on the surface constituted roughly 20% of the total cells, and it is supposed that the oil from these cells is washed out relatively easily. Secondly, the difference between theory and experiment was found to be greater with low moisture contents; it is postulated that in these cases, moisture has evaporated and left void spaces in the kernel. When contacted with solvent, solvent flows into these spaces, dissolves some of the oil, and consequently reduces the oil concentration. When these 2 factors are taken into account, reasonable agreement between theory and experiment is obtained.

King and co-workers (1944) studied the extraction of oil from soybean flakes, of different size and thickness, using trichlorethylene as solvent. Additional data were obtained on extraction from porous plates previously soaked in soybean oil; the porous plate data was found to agree with diffusion theory, unlike the soybean flake data, which could not be represented in this way. This is not unexpected, since the flakes were neither uniform nor homogeneous, nor of the same size and shape, and contained both ruptured and unruptured cells.

This problem was taken further by Osburn & Katz (1944). They started with equation (2. 23) and considered the effect of different shapes and structures on the shape of the extraction-time curve. They found that a curve representing extraction from a material having two different but constant diffusivities (from different parts of the solid), or from a combination of two different thicknesses, could represent soybean extraction data satisfactorily. The data of King et al (1944) were analysed on the basis of two different but constant diffusivities; it was found that 70 to 90% of the oil is extracted with a diffusivity 4 to 9 times greater than that corresponding to the remaining fraction. As the flake thickness was increased, the fraction extracted with a higher diffusivity decreased, and both diffusivities tended to increase.

If the explanation is in terms of differing thickness, this implies that 10 to 30% of the material has a thickness three times the nominal diameter, which is not physically realistic. They conclude that the explanation is on the basis of two differing structures within the flakes, oil being more easily extracted from one than the other. They state that 2 different structures are not observable in the flakes, but an explanation on the basis of ruptured and unruptured cells seems plausible.

This interpretation in terms of the physical structure appears realistic, but a theory for ideal conditions of diffusion does not strictly apply in this case, because of the non-uniform sizes and structure of the material. Osburn & Katz (1944) in fact show that when the thickness of the material is not uniform, it is incorrect to use an average value for the thickness. These non-idealities must influence the values of the parameters obtained.

A similar study to that of Fan et al (1948) was carried out by Krasuk & co-workers (1967) on the extraction of oil from tung seeds. They employed thicker slices so that the effect of ruptured cells was negligible. They found extraction to be a function of $t/1^2$, but experimental data did not follow equation (2.23) satisfactorily, particularly at lower temperatures. Good agreement with experimental data was obtained by solving equation (2.22) numerically, assuming that D_m is not constant, but is given by:

$$D_m = D_o \exp\left[k \left(\frac{c}{c_o}\right)\right] \qquad (2.24)$$

where c_o refers to the initial concentration of oil in the tung seed.

Values of D_o and k were found by comparing experimental data with the theoretical solutions. Temperature was found to have a marked effect; at higher temperatures, D_o is higher, and k lower, indicating a closer approach to constant diffusivity at higher temperatures. The implication of this model is that extraction becomes slower as

extraction proceeds. Although the model does not give any insight into the microscopic processes occurring, the authors propose that the concentration dependence of the diffusivity may occur if the concentration is not representative of the number of molecules able to migrate, or if a non-linear adsorption process occurs. The latter explanation is supported by the closer approach to the diffusion theory at higher temperatures.

It was found that the nature of the solvent used had no effect on the extraction. This independence of extraction on the diffusivity in the liquid phase is taken to indicate that the difficulty with which oil diffuses across cell walls controls the extraction process. Krasuk et al show also that their model can adequately represent the experimental data of Fan et al (1948) and King et al (1944).

2.6.3 Extraction of Sugar From Sugar Beet.

A comprehensive study of the extraction of sugar from sugar beet has been made by Brūniche-Olsen (1962). A description of the structure of beet tissue is given and he points out that diffusion of sucrose from beet is impossible until the protoplasm which lines each cell is de-natured by heating to elevated temperatures. This so called "killing" process was investigated by measuring diminution in volume and electrical conductivity of beet in order to follow the process. At temperatures of 80°C and above, this process occurs rapidly, but at lower temperatures the process takes longer. The conditions of the experiments are such, however, that it is difficult to separate the effects of time required to heat the beet to the required temperature and the time required to effect the change. This is brought out by the fact that the two methods of studying the de-naturing process indicate different time requirements. The de-naturing process occurs at temperatures as low as 40°C, but at a very slow rate. It is also shown that certain substances such as carbon tetrachloride, chloroform and formalin can also "kill" the cells, but not as efficiently.

Brüniche-Olsen employed equation (2. 23), introducing an effective diffusivity, $q' D_m$, where q' is termed the coefficient of inhibition, and D_m is the molecular diffusivity in water. The value of q' was determined by following the diffusion through a section of beet tissue separating 2 sucrose solutions of different concentrations. Values of q' were found to vary between 0. 5 and 0. 7, the variations being attributed to differences in the structure of different beets used in the tests.

The extraction of sugar from disks of beet was studied, the disks having large end surface areas in relation to their thickness, in order to be able to use equation (2. 23). Extraction to solutions of constant, decreasing and increasing concentration were studied; only the first case will be considered here.

The initial period of extraction was neglected, so that only the first term of equation (2. 23) need be considered. Then:

$$\frac{\bar{c} - c_1}{c_o - c_1} = \frac{8}{\pi^2} \exp\left(- \frac{q' D_m \pi^2 t}{l^2} \right) \qquad (2.25)$$

The quantity $q' D_m \pi^2 / l^2$ was termed the "coefficient of extraction", designated as k. Introducing k into equation (2. 25) leads to:

$$\frac{\bar{c} - c_1}{c_o - c_1} = \frac{8}{\pi^2} \exp\left(- kt \right) \qquad (2.26)$$

Differentiating both sides of this equation with respect to t, remembering that c_o and c_1 are constant, leads to the following result:

$$\frac{d \bar{c}}{d t} = -k (\bar{c} - c_1) \qquad (2.27)$$

The correspondence between this "coefficient of extraction" and a mass transfer coefficient is clear.

At longer times, when equation (2. 26) holds, it can be seen that $\log \frac{\bar{c} - c_1}{c_o - c_1}$ should yield a straight line when plotted against time, of slope -k, and intercept $\log (8/\pi^2)$, which provides a test of the diffusion theory.

It was found that significant variations in concentration in different parts of the beet existed before experiments were started, in which case agreement between theory and experiment was poor. This was overcome by first soaking the beets in juice of approximately the same concentration at 75°C, which had the double effect of equalising initial concentrations and 'killing' the cells. A requirement for the applicability of equation (2. 23) and hence equation (2. 26) is that this concentration, c_o, is uniform. However, the equalizing of initial concentrations eliminates one of the factors which would influence beet extraction results in practice.

In this case, after 10 minutes a straight line on a semi-log plot was obtained, whose slope determined the value of k. However, extrapolation of the straight line back to t = o does not yield a value of the intercept indicated by equation (2. 26), and so the applicability of this diffusion theory must be in some doubt.

The values of k obtained showed variations of up to 30%, which are again attributed to natural variations found in different beets. Values of q' were found to lie between 0. 45 and 0. 6, somewhat lower than obtained by the other method, and to be independent of temperature. Values of k were found to be roughly 3 times those obtained at 23°C. The results showed that k is proportional to $1/l^2$ only for l>5 mm. An approximate analysis, somewhat similar to that of Osburn & Katz (1944), was proposed to explain the deviation from theoretical behaviour. This analysis assumed different values of q' for parenchyma cells and vascular bundles, to account for the apparent fall off in rate at higher extractions.

Yang & Brier (1958) ruled out the application of this theory for use with sugar beet due to the non-uniform structure of beet. It appears that, in Brüniche-Olsen's tests, this non-uniformity only manifests itself in thinner beet slices.

Although beet disks are sometimes used in industrial applications, cosettes of less regular shapes are generally used. Brüniche-Olsen (1962) also reported tests on this material. Slightly different extraction behaviour was evident, in that the data yielded a linear relation on a semi-log plot only at much longer times. Brüniche-Olsen showed that a linear combination of 2 exponential terms is capable of representing this behaviour. This assumes the beet cosettes to be divided into 2 fractions, each having a different value of k, which is attributed to differences in cosette thickness.

Schneider et al (1970) used the same approach, utilizing equation (2.23), but assumed 3 different fractions within the cosettes, each having a different value of k. The variations in k are attributed by these authors to the inhomogeneity of the beets. Yang & Brier (1958) pointed out that up to 18% of the beet cells are ruptured in the preparation of the cosettes, which also contributes to the non-uniformity of beet cosettes.

Recently, Rathje (1968) suggested that the extraction of sugar from beet does not occur by diffusion down a concentration gradient, but by "exchange of liquid". He maintains that water is drawn into the cells by osmosis, which effectively pushes the sucrose molecules out of the cells. Various experiments are described which support his views, showing that diffusion due to the random motion of molecules is a much slower process. However, both mechanisms must be dependent on the concentration difference as the driving force for mass transfer.

2.6.4 Discussion.

Diffusion under ideal conditions which leads to equation (2.23) cannot generally be applied to extraction from vegetable matter. The requirements of this diffusion

theory imply uniform initial concentration of the solute in a uniform, homogeneous material. Extraction from soybean flakes and tung seed show divergence from the theory, and extraction from beet obeys the theory only if the non-uniform initial sugar concentration is made to be uniform. Even so, thinner beet slices show a structure dependence which precludes the application of this theory.

However, the results reviewed here show that a 3 parameter model can adequately describe extraction from these materials. This model is obtained by assuming that extraction occurs from 2 parallel zones, each having a different effective diffusivity (the same result is obtained by assuming that extraction occurs from material of 2 different thicknesses (Osburn & Katz 1944)). Krasuk et al (1967) have shown that the assumption that D_m is an exponential function of concentration leads to essentially the same results. These models hold whether there is obviously a difference in structure in the material (eg. ruptured and un-ruptured cells) or not. Thus either non-uniform initial concentrations, or non-uniform micro-structures, or both, affect extraction results.

Further, it appears to be a characteristic of vegetable matter that the same material obtained from different plants displays significant differences in structure, which introduces scatter into measurements of extraction. This was demonstrated with tung seeds (Krasuk et al, 1967) and sugar beet (Brüniche-Olsen, 1962).

2.7 EXTRACTION IN THE SUGAR DIFFUSION PROCESS.

There are at present at least 6 different types of diffuser commercially available, the majority of which involve flow of liquid by gravity through a bed of bagasse. A large volume of literature on diffusion has been published, most of which reports plant operating results and mechanical details of different installations. Some widely divergent views have been presented, which have in some cases been generated by misconceptions regarding certain aspects of the diffusion process.

The objective of this review is to attempt to identify those factors which influence the performance of diffusers, and to elucidate the basic mechanisms operative. Thus this section concerns work of a more fundamental nature.

2.7.1 Effect of Structure of Cane and Bagasse.

Considerations of the structure of the material involved can be used as a guide to possible extraction mechanisms. The non-uniform structure of cane was discussed in section 2.1. This has a direct influence on mass transfer rates; Brüniche-Olsen (1969) has shown that the effective diffusivity of sugar in a direction parallel to the cane axis is roughly 1.5 to 2 times that in a direction perpendicular to the cane stalk. He showed also that the rate of diffusion in cane is roughly 1/3 of that in beet, so that if extraction were to be effected solely by molecular diffusion, the time requirements to achieve an acceptable extraction would be prohibitively long.

Brüniche-Olsen (1969) reported that sugar cannot be extracted from intact cells in cane, unless the cane is heated to elevated temperatures. This leads to a de-naturing of the protoplasm lining each cell, thus rendering the cell wall permeable to sucrose molecules. The temperature and time requirements to effect this change are similar to those found for beet, described in greater detail in section 2.6.3.

Because diffusion in cane is a slow process, the cane is subjected to a cane preparation step prior to processing, involving knifing and shredding or milling. This serves the double function of breaking the cane into small pieces, and rupturing a large proportion of the juice-bearing cells. A considerable amount of this juice can then be removed by mixing with water, which is the

basis of the DI test. For first mill bagasse, a DI of roughly 64 (Markham, 1969) indicates that up to 64% of the juice can be easily removed. The remaining juice is not accessible to the washing liquid.

Elzeini (1965) reported that after passing through 2 mills, roughly 10 to 16% of the parenchyma cells are intact, and since they are weak-walled cells, a larger proportion of the tougher-walled sclerenchyma cells must remain undamaged. This indicates that a significant fraction of the juice can only be extracted by diffusing through cell walls. Further, the slow approach to equilibrium indicated by DI tests (Markham 1969) indicates that a significant amount of the juice exists in broken cells, which must be located in the interior of particles, since this juice is not readily accessible to a washing liquid.

2.7.2 Experience with Full-scale Diffusers.

Two modes of operation are possible, with or without a mill before the diffuser. In a number of countries, including South Africa, cane payment is based on the analysis of juice expressed in the first mill, so that at least one mill before a diffuser is mandatory. This has the advantage that roughly 60% of the sugar is extracted prior to the diffusion stage, and so exposes less of the total sugar to inversion or other losses within the diffuser. Undoubtedly, however, the lower cost of purchasing, running and maintaining an extraction system without a first mill is attractive, even though a slightly longer diffuser is required.

Preparation has been recognised as a major factor influencing the performance of diffusers. The degree of preparation determines what fraction of the cells is ruptured, and this dependence infers that efficient extraction is dependent on efficient washing. For diffusion without a first mill, Payne (1968) has stated that a DI of at least 94 is required, in order to achieve an extraction of 97% or more. This naturally requires an intensive cane preparation operation. With such a high proportion of broken cells, a washing-displacement process is undoubtedly the major mechanism. But some extraction by diffusion must occur in order to achieve high extraction values.

Although roughly 60% of the sucrose in cane is expressed in a first mill, a DI for first mill bagasse of the order of 65 suggests that extraction by a diffusional mechanism must play a more important role. In both cases, since diffusion is a much slower process, it is probable that extraction by diffusion determines the overall extraction achieved (Buchanan 1968). Buchanan (1968) has pointed out that extraction by diffusion involves not only transfer through intact cell walls, but also molecular diffusion down a concentration gradient through broken cells within the particles.

In practice, the degree of preparation cannot be widely varied without seriously jeopardizing the juice flow system in a diffuser where liquid flow occurs by gravity. In particular, finer preparations limit the maximum liquid flow through the bagasse. Control action has usually been to limit the degree of cane preparation. In view of the importance of cane preparation, it might be more beneficial to rearrange the juice flow system where possible to accommodate a finer preparation. Tantawi (1964) reported that the percolation rate is higher through crushed than shredded cane; however Payne (1968) has stated that shredded cane produces a more open permeable bed.

Payne (1968) is of the opinion that the bed should not be disturbed at all for optimum displacement efficiency, so that a minimum of mixing between static and dynamic juice occurs. However, Brüniche -Olsen (1966) states that intimate mixing between juice and bagasse is necessary to ensure high extraction efficiencies.

Cane quality has been widely recognised as a factor influencing extraction. In particular, the response of different canes to the same methods of preparation leads to prepared bagasse of different characteristics.

Two recent papers by van der Riet & Renton (1971) and Foster & Shann (1971) provide excellent reports of operational problems encountered in full-scale diffuser operation.

Apart from preparation, the effects of other primary variables cannot be evaluated on the plant scale. The controlled conditions of pilot plant experiments are required to demonstrate these effects.

2.7.3 Pilot Plant Investigations.

One of the earliest reported investigations into diffusion is that of van der Pol & Young (1957). They attempted to evaluate a "diffusion constant", based on Fick's law, but found in fact that this quantity was not constant, but varied with time.

Payne (1960) first showed that extractions better than or comparable to those obtained by milling are possible by diffusion; a pilot plant processing 20 tons cane/hour yielded figures for extraction of about 97%. Again, preparation was found to be the principal factor influencing extraction; the effect of imbibition rate was overshadowed by the degree of preparation. He reported laboratory tests which showed that juice in broken cells is easily extracted by a simple washing process.

Brüniche-Olsen (1969) reported some investigations into extraction from crushed cane to an extract of constant concentration. He utilized the same equation (2.2.7) as for his tests with beet described in section 2.6.3. However, it is unrealistic to expect the diffusion theory, on which this equation is based, to hold for a material as inhomogeneous as crushed cane. Equation (2.27) could in this case be considered as an empirical definition of the coefficient of extraction, k . In the event of the inapplicability of the diffusion theory, k does not characterize the extraction process; it represents only the rate of extraction at longer times, and an estimation of the extraction achieved cannot be obtained.

The value of k was obtained from the slope of the concentration-time curve on a semi-log plot, although the period over which a linear relation was obtained is not reported. It is shown that periodic compression of the particles during extraction increases the value of k; this is explained by the fact that some juice is expressed from the particles and replaced by more dilute extracting liquor. A certain amount of scatter in the values of k obtained is ascribed to the natural variations found in a plant product.

Bruniche-Olsen (1969) also presented an analysis to predict the extraction values expected in a moving bed diffuser, which however is based on a number of dubious assumptions, not the least of which is the assumption that percolating juice within a stage is fully mixed, and so of constant concentration in a stage. The latter assumption is also inherent in an analysis by Buchanan (1968), who attempted to predict the number of stages required in a moving bed diffuser by a McCabe-Thiele graphical method which is normally used to predict the performance of a leaching battery.

Foster & Hill (1966) reported the results of a pilot plant investigation aimed at determining the effect of primary operating variables on extraction. They simulated the operation of a moving-bed diffuser by pumping a number of different volumes of juice, of successively decreasing concentrations, through a fixed bed of first mill bagasse. The bagasse was subsequently subjected to a laboratory de-watering pressing to reduce the bagasse moisture content to commonly encountered final bagasse values. Their results were summarized by expressing extraction from first mill bagasse, E, as a multi-linear regression equation:

$$E = 91.2 - 3.01\, d_{PT} + 0.10T + 0.185t \qquad (2.28)$$

Here d_{PT} represents an average particle thickness obtained from sieve analysis, defined by equation (2.2). T and t represent temperature in ^{o}C and time in minutes respectively. This shows that cane preparation has the greatest effect on extraction. They showed also that, after the extraction process, most sugar is contained in larger bagasse particles. Thus more efficient preparation should aim at reducing the maximum particle size, rather than reducing the average particle size. Their results also show that sieve analysis can effectively be used to provide a measure of cane preparation.

This work provides the only quantitative information on extraction. Extraction by a diffuser and de-watering mill can be roughly estimated, for similar types of cane, from equation (2.28). However their results show only the overall effect of the 3 variables studied, and provide little insight into the fundamental process occurring.

Foster & Shann (1968) reported that liquid permeability is reduced in beds of finer bagasse. They showed that the maximum flow rate displays a distinct dependence on particle size, but reported that some canes show unpredictable behaviour. Significantly higher flow rates in shallower beds were also observed.

A comprehensive investigation into diffusion on a laboratory scale was reported by Buchanan & Jullienne (1969). Two different experimental test rigs were used; a well stirred vessel in which bagasse and water were agitated together, and a 4" diameter column to take a fixed bed of bagasse. In both configurations, a closed system was used with fixed batches of bagasse and water, so that extraction was gauged by the approach to equilibrium conditions.

The column diameter was small in relation to the size of the particles, so that the wall effect mentioned in section 2.3.6 must have been significant. This is substantiated by the very high flow rates obtained with this equipment. They found that extraction in column operation was less efficient than in the fully-mixed system. This is ascribed to channelling through the packed bed, leading to "incomplete wetting". It is inconceivable that some of the particles remained unwetted under their operating conditions of very high flow rate in a flooded bed from which all air had been displaced. Rather, lower efficiencies should perhaps be ascribed to less effective wetting of particle surfaces, on the basis of work described in section 2.5.1.

Degree of preparation was again found to be the most important single variable. Higher temperatures were found to promote initial mass transfer rates, as well as overall extraction performance. The effect of temperature was reported to be more significant with coarser types of preparation.

Buchanan & Jullienne (1969) postulated that high rates of extraction obtained initially are representitive of a displacement process, but that the decreased rates of extraction in the later periods are indicative of a diffusional mechanism.

2.7.4 Discussion.

Published work on diffusion is characterized by a scarcity of work of a fundamental nature. A large proportion of published work consists of subjective reasoning on the mechanism of extraction, unsupported by experimental evidence. In particular no information of a quantitative nature exists which will permit diffuser design on a rational basis.

Extraction of sugar is dependent on the processes occurring on a micro-scale, as well as macro fluid flow characteristics. The latter aspect has been neglected entirely. Possible mechanisms of extraction on a micro-scale have been recognized, namely displacement washing of juice from particle surfaces, diffusion through intact cells, and molecular diffusion of sucrose down a concentration gradient to the particle surfaces.

It appears that the structure of prepared bagasse or cane must determine to a large extent the relative importance of these mass transfer processes. The greater the degree of cell rupture, the more important is the washing process and the smaller the contribution of true diffusion to the overall extraction. In this respect it is likely that DI can furnish a measure of cane preparation. Further, the structure of bagasse is of such a heterogeneous nature that classical diffusion theory is unlikely to be of any utility in describing the diffusional component of the extraction process.

The work of Foster & Hill (1966) and Buchanan & Jullienne (1969) has identified the major process variables, and shows qualitatively their effect on the diffusion process. This provides a useful starting point for designing an experimental programme.

2.8 EXTRACTION OF IMPURITIES

Although a number of diffuser installations have shown that higher extractions of sucrose are possible in a diffusion rather than a milling process, the ultimate performance criterion is the overall recovery of sugar, and not the degree of extraction. Impurities lead to a loss of sugar in molasses, and excessive amounts of certain impurities cause considerable difficulty in the subsequent processing of the juice. It is obviously necessary, therefore, to consider the effects of impurities in defining the optimum operating conditions for diffusion.

Considerable difference of opinion exists on whether higher extractions obtained via diffusion are offset by excessive extraction of impurities. On one hand, Payne (1968), reported that the introduction of diffusion in a sugar mill in Hawaii increased extraction, with no effect on boiling house recovery; similarly, Weng and Brüniche-Olsen (1968) reported that high extractions obtained with diffusion were not accompanied by a drop in juice purity or recovery. In contrast, it was reported (Anon., 1969) that milling returns from South African sugar factories showed that diffusion factories produce more final molasses relative to the amount of non-sucrose in mixed juice than conventional milling factories; the average non-sucrose ratios were 0.95 and 0.84 respectively. This is substantiated by values of boiling house recovery for diffusion factories, which lie significantly below the average value (Perk, 1970).

Only a few investigations into the quantities of various impurities extracted in diffusion have been reported, and have generally been restricted to the more abundant species of non-sucrose. This section also considers which impurities are easily removed in the clarification process; those impurities which are not removed to any significant extent in the recovery process naturally assume added importance.

2.8.1 Reported Investigations.

2.8.1.1 Inorganic Impurities.

Graham et al (1968) measured the amount of inorganic materials (as sulphated ash) extracted from a full scale diffuser under various conditions of temperature and pH. Their results show no significant dependence on either temperature or pH; work by Buchanan and Julienne (1969) substantiates their findings.

No measurements of individual inorganic species have been made, although the observations of Honig (1958) on the inorganic constituents of mixed juice in milling factories are of interest; K, Na, and most of the Ca are present as ions in mixed juice; phosphate is present as mono- or di-phosphate, while sulphate and chloride are present in inorganic form; only 10 to 35% of the silica in cane is extracted, and the order of decreasing concentration in mixed juice is roughly K, Na, S, Cl, Si, P, Mg, Ca, with smaller amounts of Fe and Al.

2.8.1.2 Reducing Sugars.

Temperature and pH were similarly found to have no effect on extraction of reducing sugars from a fixed bed of bagasse in a small diameter column (Buchanan and Julienne, 1969). Graham et al (1968) investigated the amount of inversion of sucrose to be expected in diffuser juices. They found that even at high temperature (85°C) and low pH (5.2), chemical inversion is negligible. However, their tests showed that at lower temperature (65°C), inversion due to enzyme activity was considerable, particularly at low pH. By increasing the pH from 5.2 to 6.0, inversion decreased nearly tenfold, from approximately 1.2% per hour to approximately 0.15% per hour. In practice, the extent of enzymatic inversion is variable, depending on the enzyme content of the cane. Bruniche-Olsen (1969) for instance reports that enzymatic inversion increases when cane is stored after harvesting.

Graham et al (1968)also showed that the ratio of reducing sugar/sucrose concentrations in diffuser juice decreases from the feed end to the discharge end of the diffuser. Thus it would appear that reducing sugars are

extracted at a faster rate than sucrose. On the basis of this, they propose that molecular diffusion plays an important part in the extraction process, attributing the faster extraction of reducing sugars to their smaller molecular size. This, however, assumes that the initial ratio of reducing sugars/sucrose is homogeneous throughout the bagasse particles; on the basis of the discussion in section 2.1.2.3, this does not appear to be the case. In fact, if extraction by washing is presumed to be significant in the first few stages of a diffuser, their results could also be interpreted as indicating easier washing extraction of reducing sugars than sucrose. However, neither of these theories explains the observation that the reducing sugar ratio is higher in juice from the last stage (discharge end) than the penultimate stage.

2.8.1.3 Starch

Temperature is likely to affect the extraction of starch, since at elevated temperatures, the structure of starch granules breaks down, and starch in colloidal form can be dispersed in the juice. This process is referred to as gelatinisation, and although it may be initiated at temperatures as low at 60°C, the rate of gelatinisation only becomes appreciable at somewhat higher temperatures (Collison, 1968).

Both Graham et. al. (1968) and Buchanan and Jullienne (1969) found starch extraction to be independent of pH, but significantly affected by temperature. The results of Graham et. al. show that the amount of starch extracted when the diffuser is operated between 75°C and 85°C is roughly five times that extracted at temperatures of 60°C to 65°C. The amount of starch extracted at the higher temperature is approximately the same as that extracted in milling. Thus a diffuser operated at lower temperature shows a distinct advantage in this respect; Jennings (1968) has in fact reported that the introduction of diffusion in a South African mill dropped the starch content in sugar crystal markedly from 600 to 400 ppm.

2.8.1.4 Gums, pectin and lignin.

Extraction of gums, pectin, and lignin was studied by Bjerager and Brüniche-Olsen (1968). They agitated disintegrated cane in water, and measured extraction of these substances as a function of time, temperature, and pH. It was shown that increased extraction is obtained with longer extraction times and higher temperatures. They also reported that higher pH promotes extraction of these substances. Although the effect of cane quality variation was eliminated by conducting tests on the same batch of cane, the increases in extraction with pH are not consistent, and in some cases, decreases were observed. Further, excessive extraction appeared to occur only under conditions of unrealistically high pH (>8.0).

Buchanan and Julienne (1969) showed that an increase in temperature from 68°C to 78°C results in an increase of more than 50% in the quantity of gums extracted. They found that a slight increase in the quantity of gums extracted is obtained with finely prepared bagasse at the lower temperature only. No effect of pH could be discerned. They also reported that the amount of pectin extracted increased with time, temperature, and pH.; however, the amounts of pectin constitute only a very small fraction of the total polysaccharide extracted.

2.8.1.5 Wax and Fats.

Bjerager & Brüniche - Olsen (1968) performed similar experiments on the extraction of fats and waxes. Extraction of fats was shown to decrease with increasing pH, while investigations into the effect of pH on the extraction of wax proved inconclusive.

Graham et. al. (1968) reported that extraction of wax is significantly lower in diffusion than in milling.

2.8.1.6 Miscellaneous.

Extraction of proteins was shown by Graham et. al. (1968) to be roughly the same in milling and diffusion, and not significantly affected by pH or temperature.

Buchanan & Jullienne (1969) reported that pH and temperature appear to have little effect on formation of colour in diffuser juice. This does not agree with observations of higher than normal colour in mixed juice in some diffuser factories. Further, it is well established that at high pH, reducing sugars decompose, and in the presence of amino acids give rise to complex compounds of dark colour (Jenkins, 1966). Payne (1968) has suggested that the major cause of colour development is the presence of excessive amounts of leaves with the cane

The diffusion process has the advantage that diffuser juice contains less suspended matter and shows less turbidity due to the filtering action of bagasse bed. This is substantiated by the considerably reduced amount of filter cake produced by diffuser factories. In fact, Payne (1968) has claimed that liming in a diffuser can produce a juice which does not require clarification.

2.8.2 Effects of impurity extraction on recovery of sucrose.

As a consequence of the structure of cane and the distribution of sucrose and non-sucrose species within the cane, it is commonly observed that the purity of juice extracted in a milling train decreases from the 1st mill, and that extraction of the last juice is associated with the extraction of a high proportion of impurities. Based on average figures for South Africa for the 1968/69 season, it can be calculated that an increase in the average sucrose extraction of 1% would result in increasing the overall recovery by only 0.65%, while the amount of final molasses would increase by 1.04% (Anon, 1969). Clearly there must be an optimum extraction above which increased recovery costs outweigh the advantage of additional sugar produced.

However, recovery of sucrose depends on the nature and not just the amount of non-sucrose associated with it. Thus it is desirable to be able to express quantitatively the effects of individual impurities on the recovery process. Such quantitative information would find utility in an optimisation strategy for the complete diffusion system. Quantitative relationships of this kind do not exist for the following reasons :-

i. The types and concentrations of impurities vary widely, and are not easily measured.

ii. The effects of individual impurities are not generally known.

iii. Although some such effects have been recognised, they cannot be expressed in quantitative terms.

iv. The effects of impurities on the production of sugar depend on the type, efficiency, and operating conditions of the recovery process.

An idea of the effect of impurities on the recovery process is given by the following relation, (Honig, 1958):

recoverable sucrose = pol-0.4 x non-sucrose content. (2.30).

This evaluates all non-sucrose as identical in molasses forming effect. However, Honig (1958) points out that the melassigenic effect of inorganic species is roughly four times that of reducing sugars. Further, those non-sucrose species which are largely removed in the clarification process will play a less important part.

Inorganic materials which are not removed have an appreciable effect on sucrose losses in molasses. According to Honig (1958), Na, K, and Cl are not removed at all. Thus van der Poel et. al. (1970) have observed a good correlation between the K^+ and Na^+ content of juices and loss of sucrose in molasses in a beet factory. The concentration of Ca increases during clarification, depending on clarification

conditions. Phosphate precipitates with Ca during clarification. Thus the extraction of inorganic phosphate is not undesirable; in some instances, phosphate is added to mixed juice to improve clarification. Al and Fe are largely removed, as is silica; however, the remaining silica has an adverse effect on the recovery process, (Honig 1958).

Alexander reported in 1954 that the starch content of South African sugar products must rank among the highest in the world, and thus the existence of starch in mixed juice and its effects has received a considerable amount of attention. Boyes (1960) reported that excessive amounts of starch in mixed juice influences the recovery process in a number of ways : it increases the viscosity of massecuite, retards the rate of crystallisation, co-crystallises with sucrose and slows down the filtration of liquor in the subsequent refining process. Gums have a similar effect; both starch and gums are hydrophilic, and so markedly increase the viscosity of solutions (Jenkins, 1966). They may, therefore, be regarded as principal harmful impurities.

Lignin and pectin similarly affect crystallisation and sugar quality (Bjerager & Brüniche-Olsen 1968) However, Spencer & Meade (1945) reportthe observation of Farnell that pectin is removed during clarification.

Wax andfatty matter is largely removed in clarification, although some may be retained in colloidal suspension in final molasses, and traces may be found in raw sugar (Barnes, 1964). Similarly, proteins coagulate and are largely removed in clarification (Honig, 1958).

2.8.3 Discussion.

Investigations into the extraction of impurities during diffusion do not point to higher impurity extractions in diffusion than in milling. Graham et. al. (1968) have shown that destruction of sucrose due to inversion is unlikely to be significant in diffusers. They also showed

that substantially less wax is extracted in diffusion, and that at low temperatures, less starch is extracted. This is supported by the observation of Jennings (1968) that the introduction of diffusion in a South African sugar factory resulted in a significant decrease in the starch content of raw sugar.

This conclusion, taken together with the claims of a number of authors that boiling house recovery and juice purity are not affected by the introduction of diffusion, are difficult to reconcile with the excessive production of molasses in South African diffuser factories.

Attempts to explain this anomaly are complicated by the fact that the juices in a sugar factory constitute a complex multicomponent mixture, whose nature and composition varies widely depending on the cane processed. In this respect, South African canes are notorious for their fibre and trash contents; it is well known that the leaves and tops constituting trash contain large quantities of undesirable impurities (Anon, 1952), which must have a detrimental effect.

Evidence exists which suggests that high retention times of juice in diffuser sysems are responsible for higher sucrose losses in molasses.
Inability to explain this observation underlines a fundamental lack of understanding of this complex system. Perhaps attention should be directed towards the physical nature of the impurities rather than the amounts of these impurities extracted.

The lack of quantitative information on the effect of the various non-sucrose species on the recovery of sucrose militates against the choice of optimum diffuser operating conditions on a rational basis. Thus only qualitative observations can be utilised as a guide in the specification of process operating variables.

CHAPTER 3

MATHEMATICAL MODELLING OF THE DIFFUSION PROCESS.

3.1 BASIS FOR MODEL FORMULATION.

It is required that a mathematical model of the diffusion process be formulated to represent extraction in the two experimental configurations employed in this study, namely extraction in the fully-mixed laboratory-scale experiments, and in a fixed bed pilot plant diffuser. The latter is practically of more utility, since it represents more closely the operation of a full-scale moving-bed diffuser.

Clearly, both models should have a common physical basis. However, packed bed operation requires that the liquid flow characteristics through the bed be accounted for, while extraction in a fully-mixed system requires only the specification of the basic mechanism of extraction, free of fluid flow considerations.

3.1.1 The Mechanism of Extraction.

Work on extraction of vegetable materials described in section 2.6 has shown that classical diffusion theory cannot be applied to these materials, because of their non-homogeneous nature. It has been shown that a 3 parameter model is capable of representing extraction from these materials, which is necessary as a consequence of non-uniform structure, or concentration distribution, or both.

A major characteristic of cane and bagasse has been shown to be its heterogeneous nature. In this material, structure differences are obvious within particles. This has led to the observation in diffusion investigations that at least 2 major transport processes are operative, namely displacement or washing of the free surface juice, and a diffusional type of transfer from the interior of particles. Clearly, the rates at which sucrose in different parts of the particles is extracted must embrace a wide spectrum of values, so that some distribution of transfer rates exists. In the absence of any indication of the type of distribution involved, the assumption that sucrose is extracted at 2 different rates would appear realistic, and balances the 2 conflicting considerations in model formulation of accuracy and practicability. Intuitively, these rates would be expected to represent the rate of washing or displacement of surface juice, and the rate of extraction of structurally inhibited sucrose by a diffusional mechanism.

3.1.2 The Effect of Liquid Flow Conditions.

In packed bed operation, the effect of liquid flow behaviour must be superimposed upon the basic rates of extraction. Sections 2.3 and 2.5 have shown that the only flow models which have any practical utility are the dispersion model and the mixing cells model, which take account of axial dispersion, and the capacitance models. For the case of liquid-phase mass transfer, it was shown that the capacitance effect of stagnant liquid regions overshadows the effect of axial dispersion. The nature of bagasse is such that stagnant liquid zones can be expected to be of considerable significance in a bagasse bed. Firstly, the hydrophilic nature of bagasse is such that liquid will be readily attracted to points of contact between particles; the wide range of particle shapes and sizes ensures an interconnecting network of void spaces of a wide distribution of sizes between particles; liquid may be attracted into the voids within bagasse particles; and finally, evidence of greater amounts of static liquid in fibrous beds has been presented (Kyan et al, 1970).

It was shown in section 2. 3 that the effect of axial dispersion on liquid-solid mass transfer processes in packed beds is generally negligible in practical packed bed systems. Although the assumption of plug flow in a packed bed is a theoretical condition not generally realised in practice, if the deviations from plug flow are not likely to affect mass transfer results significantly, the obvious advantages of neglecting it for computational simplicity and practical utility need not be emphasized. In particular, if the inclusion of axial dispersion precludes the attainment of a solution in closed form, a strong case for its omission can be made.

Han and Bixler (1967) assumed that mass transfer between stagnant liquid in channels between particles and the flowing liquid occurs by molecular diffusion, although they concede that some other mechanism contributes to the renewal of liquid at the entrances to the channels. Others, such as Hochman & Effron (1969) and van Swaaij et al (1969) have described movement into and out of stagnant zones in terms of a mass transfer coefficient without specifying what transport processes are operative. Because of the structure of bagasse, pockets of liquid trapped between particles, together with liquid which may be drawn into voids in the particles, would be inaccessible to a flowing liquid. Exchange of mass with this liquid can therefore only occur by a diffusional mechanism. On the other hand, some static liquid retained by gravity or capillary action must be more readily available to exchange by renewal with the percolating liquid.

Once liquid flow has been established in a bagasse bed it is impossible to distinguish between inter-and intra-particle liquid, in which case it is convenient to treat the static liquid as part of the juice holdup in bagasse. Then static liquid zones in internal regions of bagasse particles can be identified with the naturally tightly-held or bound juice, since both need to be extracted by a diffusional process. Static liquid external to the particles can be identified with juice from broken cells on particle surfaces which is more readily available for washing or displacement by the percolating liquid.

It is to be expected that the location and extent of stagnant liquid zones will affect observed mass transfer rates.

3.2 PROPOSED 3 PARAMETER DIFFUSION MODEL.

3.2.1 Extraction in a Fully-Mixed Batch System.

Extraction of sucrose from bagasse is postulated to occur as follows: part of the juice in broken cells on the surface of bagasse particles is readily extracted by a displacement-washing process, at a rate k_1, while the remainder of the juice in unbroken cells and in broken cells in the interior of particles is extracted by a slower diffusional mechanism, at a rate k_2. It is expected that k_1 will be considerably greater than k_2. The fraction of juice which is readily extracted is denoted by α, where $0 < \alpha < 1$. The remaining tightly-held or bound juice fraction is represented by $1-\alpha$.

The model is shown schematically in Fig. 3.1.

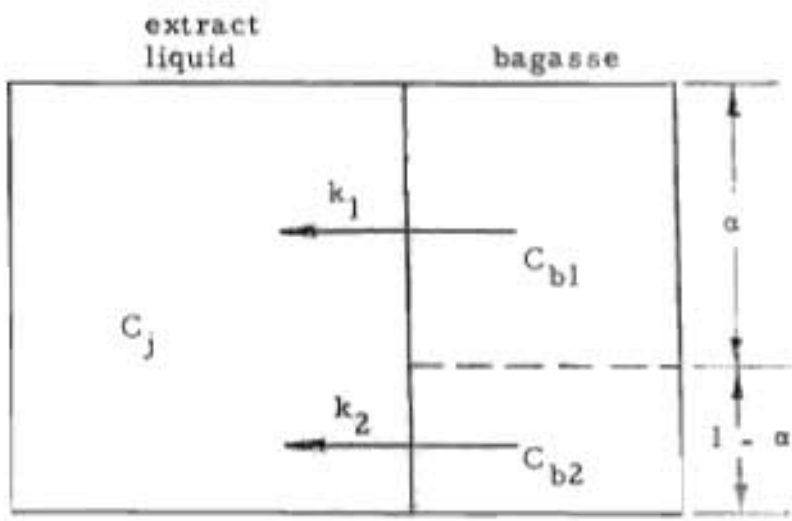


Fig. 3.1. Schematic Representation of Extraction Model.

In the laboratory extraction tests, extraction occurs to an extract liquid of constant weight, which is assumed to be fully-mixed, and therefore everywhere at the same concentration at any given time. It is assumed that mass transfer is best described in terms of mass transfer coefficients, and that the rate of extraction

is proportional to the difference in bulk concentrations of juice in bagasse and extract.

Material balances for the sucrose in bagasse over the differential time element dt lead to the following differential equations for the readily available juice and more tightly-held juice in bagasse respectively:

$$- \alpha h \frac{d C_{b1}}{d t} = k_1 (C_{b1} - C_j) \qquad (3.1)$$

$$- (1 - \alpha) h \frac{d C_{b2}}{d t} = k_2 (C_{b2} - C_j) \qquad (3.2)$$

Similarly, for the extract liquid,

$$W \frac{d C_j}{d t} = k_1 (C_{b1} - C_j) + k_2 (C_{b2} - C_j) \qquad (3.3)$$

where C_j = concentration of sucrose in extract.

C_{b1} = concentration of sucrose in readily available juice in bagasse.

C_{b2} = concentration of sucrose in tightly-held juice in bagasse.

h = juice in bagasse (g).

W = weight of extract liquid (g).

k_1 = mass transfer coefficient (g /min).

k_2 = mass transfer coefficient (g/min).

α = fraction of juice in bagasse extracted at rate k_1.

Sucrose concentrations are ideally expressed as mass fractions. In practice brix values are used. This treatment assumes that values of W and h are constant, and that the composition of the solid is uniform initially. The initial concentration of extract liquid is C_{jo}. The following boundary conditions apply :

$$\text{at } t = O, \qquad C_j = C_{jo} \qquad (3.4)$$

$$\text{at } t = O, \qquad C_{b1} = C_{b2} = C_{bo} \qquad (3.5)$$

Equations (3.1) to (3.3) can be simplified by the introduction of the dimensionless quantities:

$$\left.\begin{aligned} \theta &= \frac{k_1 t}{h} \\ \beta &= \frac{k_2}{k_1} \\ Q &= \frac{W}{h} \end{aligned}\right\} \qquad (3.6)$$

which leads to the following set of differential equations:

$$-\alpha \frac{d C_{b1}}{d \theta} = C_{b1} - C_j \qquad (3.7)$$

$$-(1-\alpha) \frac{d C_{b2}}{d \theta} = \beta (C_{b2} - C_j) \qquad (3.8)$$

$$Q \frac{d C_j}{d \theta} = C_{b1} + \beta C_{b2} - (1+\beta) C_j \qquad (3.9)$$

3.2.2 Extraction in a Packed Bed.

The process considered here is an unsteady-state mass transfer process in a packed bed. A liquid of constant concentration is applied to the top of a fixed bed of bagasse and flows down through the bed by gravity; the concentration of sucrose in the bagasse decreases with time as sucrose is progressively extracted into the flowing juice stream.

Plug flow of liquid through the bagasse bed is assumed. Again, extraction is postulated to occur by two first order relations in parallel. Stagnant liquid is considered as part of the juice holdup in bagasse. Once again, α represents the fraction of this juice which is readily extracted.

Consider an element within the packed bed, of height dz and unit cross section. Liquid of concentration C_j flows vertically downwards into this section, and is enriched as a result of mass transfer from the juice in bagasse, before flowing out of the section.

Thus the concentrations of flowing juice and juice in bagasse vary with distance from the top of the bed and time. Radial concentration gradients are considered to be negligible.

A mass balance on the sucrose in the flowing liquid phase in this element is:

$$\text{rate of input} = L\,C_j + K_1\,(C_{b1} - C_j)\,dz + K_2\,(C_{b2} - C_j)\,dz$$

$$\text{rate of output} = L\left(C_j + \frac{\delta C_j}{\delta z}\,dz\right)$$

$$\text{rate of accumulation} = \rho h_D \frac{\delta C_j}{\delta t}\,dz$$

The liquid phase material balance is then:

$$-L\frac{\delta C_j}{\delta z} + K_1\,(C_{b1} - C_j) + K_2\,(C_{b2} - C_j) = \rho h_D \frac{\delta C_j}{\delta t} \qquad (3.10)$$

Similarly, material balances for the sucrose in bagasse yields:

$$-\alpha H \frac{\delta C_{b1}}{\delta t} = K_1\,(C_{b1} - C_j) \qquad (3.11)$$

$$-(1-\alpha)H\frac{\delta C_{b2}}{\delta t} = K_2\,(C_{b2} - C_j) \qquad (3.12)$$

where C_j = concentration of sucrose in percolating liquid.

C_{b1} = concentration of sucrose in juice in bagasse (readily available phase).

C_{b2} = concentration of sucrose in juice in bagasse (tightly-held phase).

H = juice holdup in bagasse (lb/ft^3)

h_D = percolating juice holdup (ft^3/ft^3).

K_1 = mass transfer coefficient (lb/min ft^3).

K_2 = mass transfer coefficient (lb/min ft^3).

L = liquid mass flow rate (lb/min ft^2)

z = distance from top of bagasse bed (ft).

α = fraction of juice in bagasse extracted at rate K_1.

The following dimensionless variables are defined:

$$N = \frac{K_1 z}{L} \qquad (3.13)$$

$$\theta = \frac{K_1}{H} \left(t - \frac{\rho h_D z}{L} \right) \qquad (3.14)$$

The variable N can be considered as the bed height measured in terms of the number of transfer units. It may be re-written as:

$$N = \frac{K_1 V}{F} \qquad (3.15)$$

where F is the flow rate in lb/min and V the bed volume in ft^3 corresponding to the value of z. This is a more convenient form for computation purposes.

The quantity $\rho h_D z / L$ in equation (3.14) is the time, τ, required to displace the liquid held up in a column of height z. Consequently θ represents the time during which a bed element a distance z from the bed surface has been contacted by flowing liquid. Thus the relation (3.14) may be re-written as:

$$\theta = \frac{K_1}{H} \left(t - \tau \right) \qquad (3.16)$$

Introducing these dimensionless variables, and setting $\beta = K_2 / K_1$, equations (3.10) to (3.12) become:

$$\frac{\delta C_j}{\delta N} + C_j (1 + \beta) - C_{b1} - \beta C_{b2} = 0 \qquad (3.17)$$

$$\alpha \frac{\delta C_{b1}}{\delta \theta} + C_{b1} - C_j = 0 \qquad (3.18)$$

$$(1 - \alpha) \frac{\delta C_{b2}}{\delta \theta} + \beta C_{b2} - \beta C_j = 0 \qquad (3.19)$$

It is assumed that the composition of the initial solid material is uniform, and that the composition of the entering fluid is constant. For this particular case, the entering liquid concentration is zero (pure water). Thus the boundary conditions are:

$$\text{at } N = 0 \qquad C_j = 0 \text{ , all } \theta \qquad (3.20)$$

$$\text{at } \theta = 0 \qquad C_{b1} = C_{b2} = C_{bo} \text{ , all N} \qquad (3.21)$$

3.3 MATHEMATICAL SOLUTIONS.

3.3.1 Laboratory Mixing Tests.

It is required to obtain a solution for C_j from the set of ordinary differential equations (3.7) to (3.9), subject to the boundary conditions, equations (3.4) and (3.5). The solution of these equations is recorded in detail in appendix E. The final result with which comparison of experimental data may be made is:

$$C_j = C_{bo}\,\frac{1}{Q+1} + \frac{C_{bo}}{Q(a-b)}\left(\left(\frac{1}{\alpha\gamma\, a} - (1+\beta)\right)e^{-a\theta} - \left(\frac{1}{\alpha\ \gamma\, b} - (1+\beta)\right)e^{-b\theta}\right)$$

$$+ C_{jo}\,\frac{Q}{Q+1} + \frac{C_{jo}}{a-b}\left(\left(a-\left(\frac{1}{\alpha}+\frac{1}{\gamma}\right)+\frac{1}{\alpha\,\gamma\, a}\right)e^{-a\theta} - \left(b-\left(\frac{1}{\alpha}+\frac{1}{\gamma}\right)\right.\right.$$

$$\left.\left.+\frac{1}{\alpha\,\gamma\, b}\right)e^{-b\theta}\right) \qquad (3.22)$$

where $\gamma = (1-\alpha)/\beta$ (3.23)

$$a = \tfrac{1}{2}\left\{\frac{1}{\gamma}+\frac{1}{\alpha}+\frac{1+\beta}{Q} - \left[\left(\frac{1}{\gamma}+\frac{1}{\alpha}+\frac{1+\beta}{Q}\right)^2 - 4\,\frac{Q+1}{\alpha\,\gamma\,Q}\right]^{\frac{1}{2}}\right\} \qquad (3.24)$$

$$b = \tfrac{1}{2}\left\{\frac{1}{\gamma}+\frac{1}{\alpha}+\frac{1+\beta}{Q} + \left[\left(\frac{1}{\gamma}+\frac{1}{\alpha}+\frac{1+\beta}{Q}\right)^2 - 4\,\frac{Q+1}{\alpha\,\gamma\,Q}\right]^{\frac{1}{2}}\right\} \qquad (3.25)$$

a and b are the roots of a quadratic equation. If the discriminant is negative, complex roots are obtained, and the form of equation (3.22) is modified slightly. In practice, however, the possible range of the quantities comprising the discriminant ensures that the roots are always real and unequal.

3.3.2 Plug Flow Percolation.

Equations (3.17) to (3.21) were solved by first applying Laplace transforms, and then treating the transformed equations as ordinary differential equations. This yields the solution in the Laplace transform domain. Subsequent inversion leads to analytical equations for C_j, C_{b1} and C_{b2}. Details are given in Appendix E; only the final solutions are given below:

$$\frac{C_j}{C_{bo}} = 1 - e^{-(1+\beta)N}\left[1 + \int_o^\theta e^{-\frac{s}{\alpha}} \sqrt{\frac{N}{\alpha s}}\, I_1\left(2\sqrt{\frac{N s}{\alpha}}\right) ds \right.$$

$$+ \int_o^\theta e^{-\frac{s}{\gamma}} \sqrt{\frac{\beta N}{\gamma s}}\, I_1\left(2\sqrt{\frac{\beta N s}{\gamma}}\right) ds$$

$$+ \int_o^\theta \int_o^s N e^{-\left(\frac{\tau}{\alpha} + \frac{s-\tau}{\gamma}\right)} \sqrt{\frac{\beta}{\alpha \gamma \tau (s-\tau)}}\, I_1\left(2\sqrt{\frac{N\tau}{\alpha}}\right)$$

$$\left. I_1\left(2\sqrt{\frac{\beta N(s-\tau)}{\gamma}}\right) d\tau\, ds \right] \qquad (3.26)$$

$$\frac{C_{b1}}{C_{bo}} = 1 - \frac{1}{\alpha} e^{-(1+\beta)N}\left[\int_o^\theta e^{-\frac{s}{\alpha}} I_o\left(2\sqrt{\frac{N s}{\alpha}}\right) ds \right.$$

$$+ \int_o^\theta \int_o^s e^{-\left(\frac{\tau}{\alpha} + \frac{s-\tau}{\gamma}\right)} \sqrt{\frac{\beta N}{\gamma(s-\tau)}}\, I_o\left(2\sqrt{\frac{N\tau}{\alpha}}\right)$$

$$\left. I_1\left(2\sqrt{\frac{\beta N(s-\tau)}{\gamma}}\right) d\tau\, ds \right] \qquad (3.27)$$

$$\frac{C_{b2}}{C_{bo}} = 1 - \frac{1}{\gamma} e^{-(1+\beta)N}\left[\int_o^\theta e^{-\frac{s}{\gamma}} I_o\left(2\sqrt{\frac{\beta N s}{\gamma}}\right) ds \right.$$

$$+ \int_o^\theta \int_o^s e^{-\left(\frac{\tau}{\gamma} - \frac{s-\tau}{\alpha}\right)} \sqrt{\frac{N}{\alpha(s-\tau)}}\, I_o\left(2\sqrt{\frac{\beta N \tau}{\gamma}}\right)$$

$$\left. I_1\left(2\sqrt{\frac{N(s-\tau)}{\alpha}}\right) d\tau\, ds \right] \qquad (3.28)$$

CHAPTER 4.

EXPERIMENTAL DETAILS.

4.1 EXPERIMENTAL OBJECTIVES.

The vast majority of full-scale diffusers involve flow by gravity of liquid through a bagasse bed. However, plant operating data is of limited utility for the purposes of testing the model; in order to do this, the controlled conditions of pilot plant experiments are necessary. The experimental system was designed to simulate the extraction process in a fixed bed pilot plant diffuser in such a way that the primary operating variables, preparation, temperature and flow rate, could be varied over a much wider range of values than are possible on the operating plant. Thus the model parameters could be estimated over the complete range of control variable settings to permit ultimately the selection of the optimum operating conditions.

A secondary experimental objective was to apply the extraction model to a simple laboratory test rig. It was envisaged that such experiments would supply information on the basic mechanisms of extraction, and provide a frame of reference to which pilot plant results could be related. Further, such a laboratory test facility could be used as a means of characterizing bagasse in terms of the plant model.

In attempting to define the optimum diffuser operating conditions, the simultaneous extraction of impurities must be considered. Since these impurities lead to a loss of sucrose in the recovery process, they affect the ultimate economic criterion of an optimization study, which is the amount of sugar recovered and not merely the degree of sucrose extraction achieved.

Measurements were made of the principal impurities extracted; these measurements of necessity covered only a fraction of the total number of individual components, since many of them are complex and present only in trace quantities.

It should be mentioned that no experiments on shredded cane were carried out. Because of the cane payment system in South Africa, at least one mill is necessary before a diffuser, and so first mill bagasse was used in the experimental work.

4.2 THE PILOT PLANT DIFFUSER.

4.2.1 Description of Equipment.

A schematic lay-out of the pilot plant is shown in Fig. 4.1. The diffusion vessel consisted of a mild steel column, 24" in internal diameter, and roughly 6.5 ft. high. Juice or water was pumped from the surge tank, a temperature-controlled stirred vessel, through an orifice meter and flow control valve into a liquid distributor, suspended just above the bagasse bed. The juice leaving the diffuser could be collected in a series of calibrated drums, or sent to process, or returned to the surge tank.

The diffuser diameter was chosen to be large enough in relation to the bagasse particle sizes to obviate any wall effect, and to ensure that packing densities comparable to those in full-scale operation would be achieved. The bagasse bed was supported by an expanded metal screen located near the bottom of the diffuser vessel between 2 flanges. Just below this screen was another finer screen, to retain smaller particles. A sight-glass in the side of the vessel allowed the bed height to be measured, and permitted observation of the juice flow during percolation. The diffusion vessel is reproduced in the photograph Fig. 4.2, and the drawing Fig. 4.3.

A Philips load beam (Type PR 6101 P/02, 200 kg nominal load) was fitted to the vessel, to allow measurement of the weight of the transient and steady-state liquid holdup in the column. During operation, the diffuser rested on the load beam and on 2 knife edges on the opposite side of the column, parallel to and in the same horizontal plane as the knife edge on the load beam. Since the load beam knife edge and the midpoint of the line joining the other 2 knife edges were equidistant from the centre of gravity of the column, the load beam was subjected to a bending load equal to half the weight of the vessel and its contents. A counter-weight and pulley system was used to counterbalance the bulk of the weight of the vessel itself.

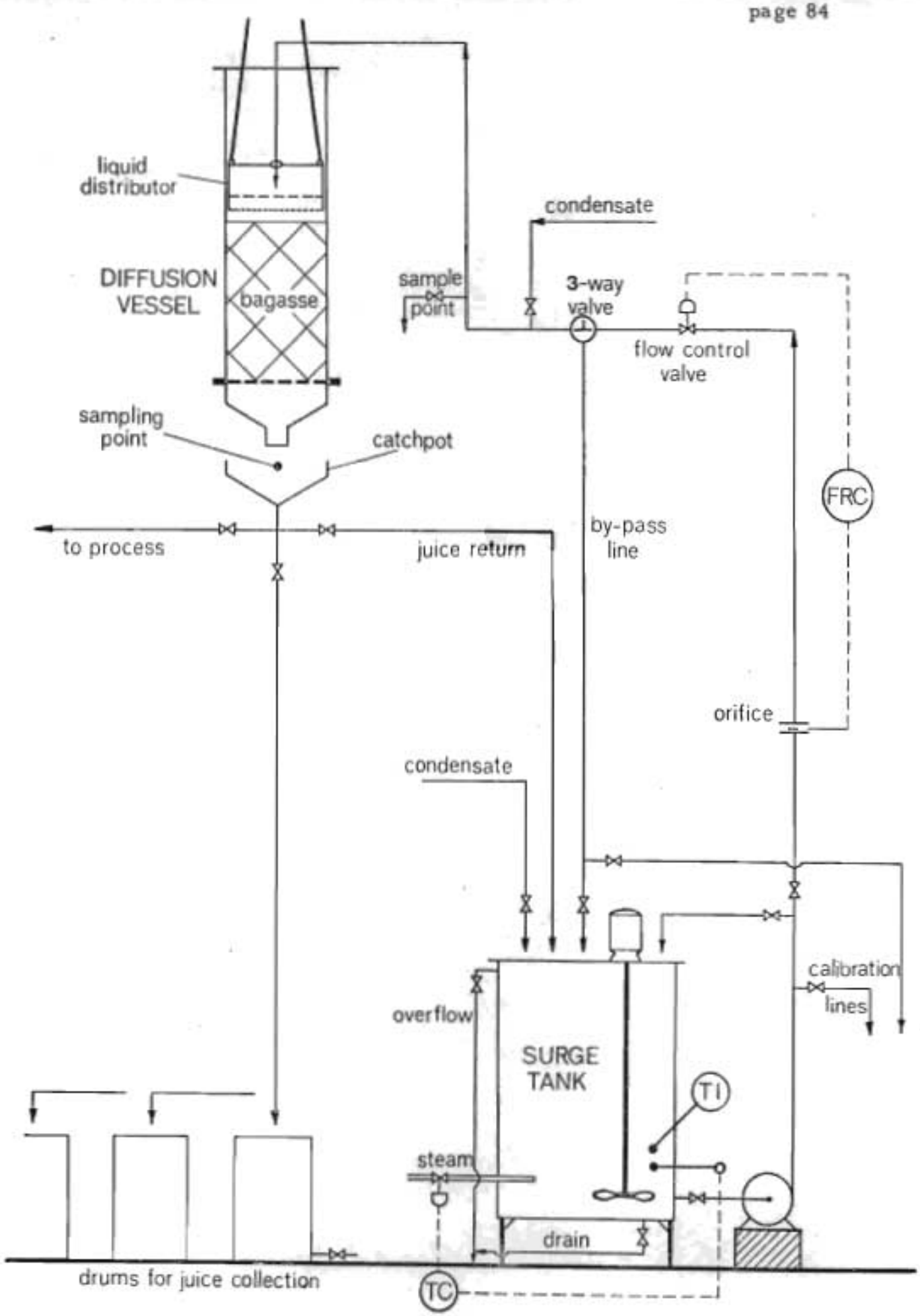


Fig. 4. 1. Schematic diagram of the pilot plant

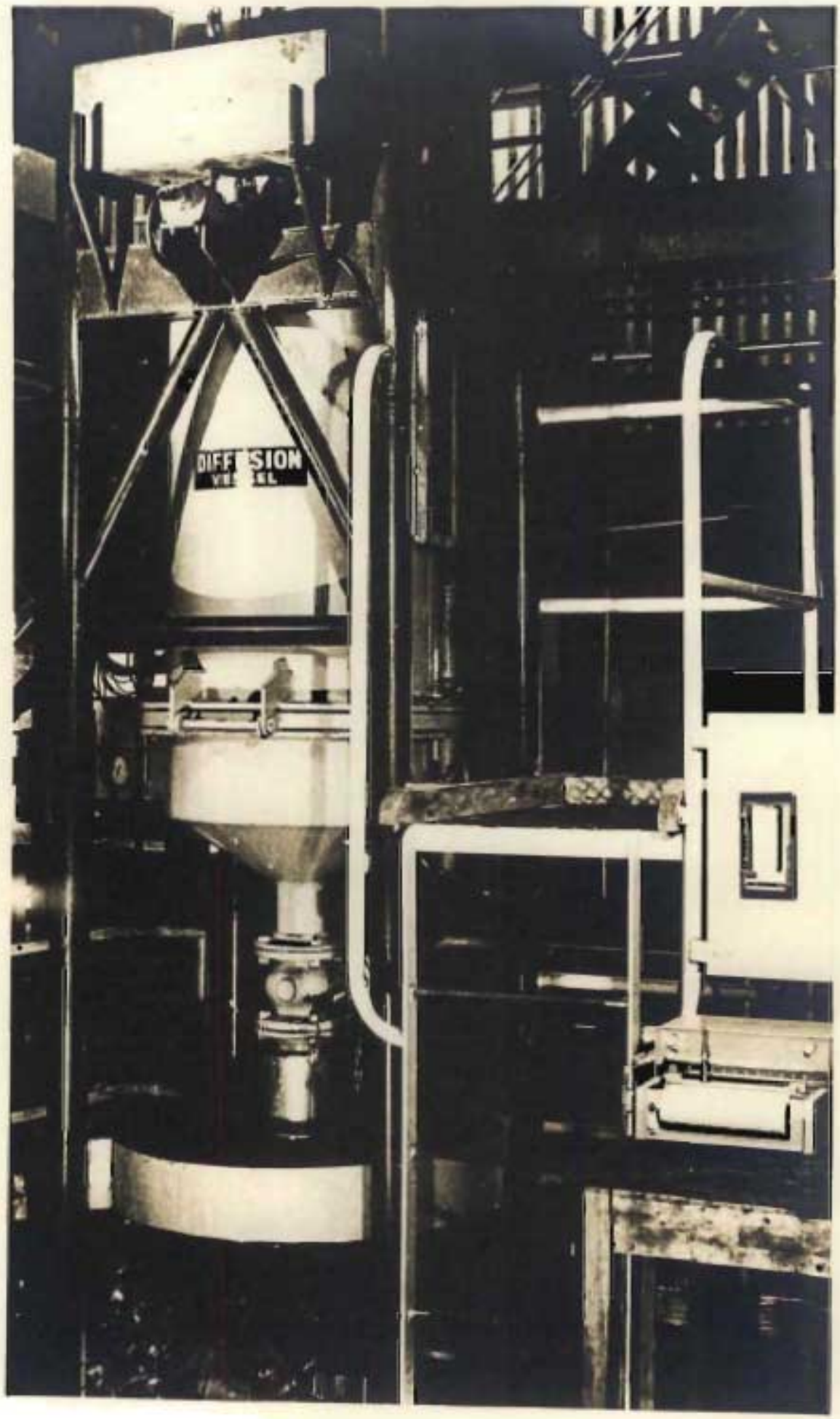


Fig 4. 2. Photograph of the diffusion vessel

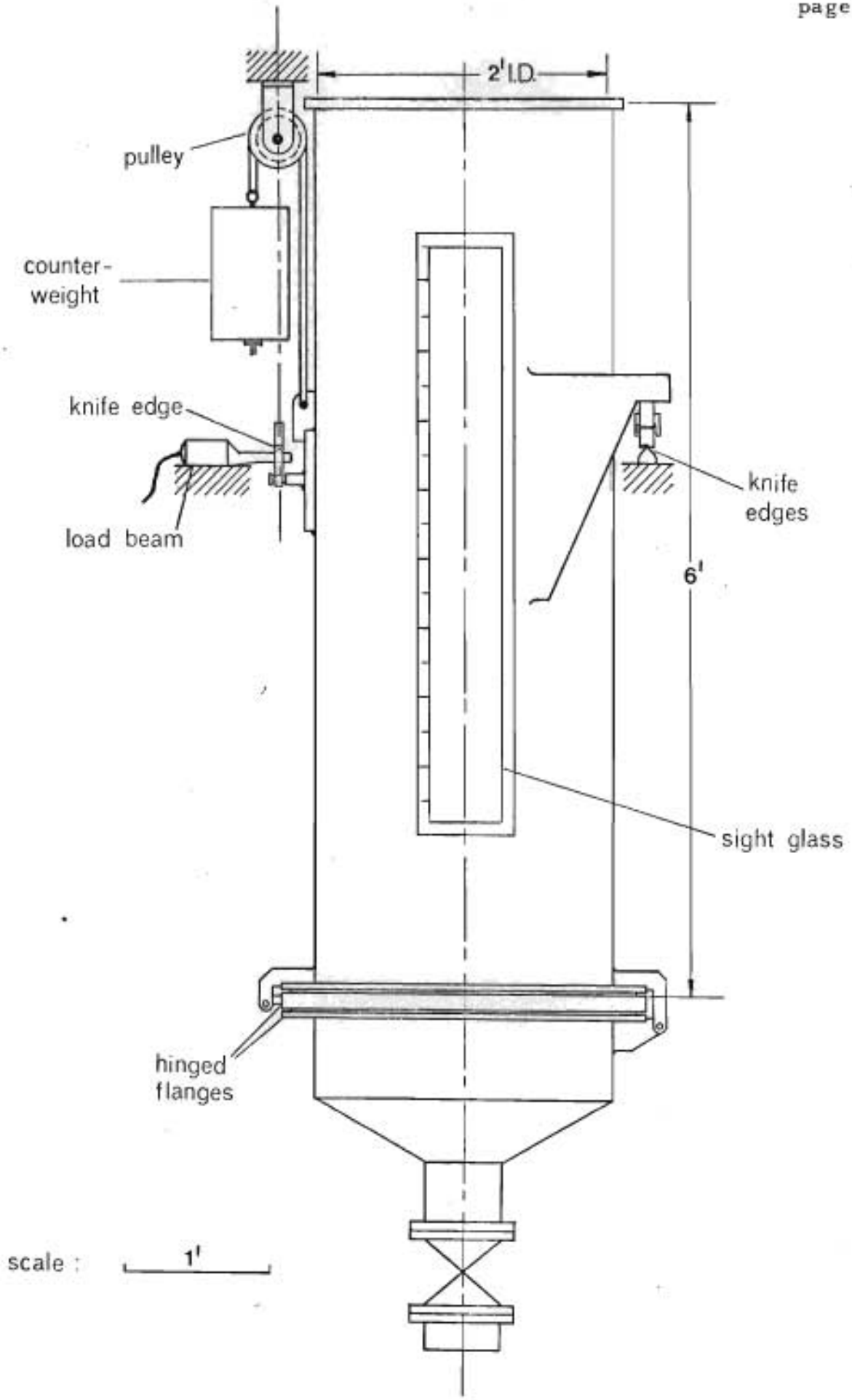


Fig. 4.3. Details of the diffusion vessel

By means of the tare adjustment on the Philips Type PR 7737/01 power supply, the contents of the vessel only could be recorded, permitting greater measurement sensitivity. With this system, the weight could be measured to within 1lb.

Because of vibration in the factory where the pilot plant was situated, the load beam output on a pen recorder had superimposed on it high frequency low amplitude fluctuations, which produced an unsuitable recorder trace. This was overcome by connecting a good quality 2 μF capacitor across the output terminals, which removed the fluctuations without significantly affecting the response time. When not in operation, the vessel was lifted off the knife edges by 4 jacking screws, and the counter-weight was lowered onto steel supports.

The bottom flange of the vessel and the screen were hinged to the vessel. The vessel was emptied by undoing 4 bolts and swinging the bottom part of the vessel down, allowing the bagasse to fall into the catchpot.

A fine screen was fitted over the outlet of the vessel to trap any fine pith particles which were washed out.

The liquid distributor was suspended independently of the diffusion vessel, so that it did not contribute to the weight recorded by the load beam. It consisted of a cylindrical frame formed from steel sheet, with a fine screen fitted over its lower diameter. Into the frame, a steel 'basket' could be inserted, whose bottom face was drilled with small holes on a 2" square pitch. Three such baskets were used, each with different sized holes. Prior to a run, a basket was selected which would maintain a liquid level of 0.5 to 1" in the basket at the selected flow rate, thus ensuring uniform flow over the column cross section.

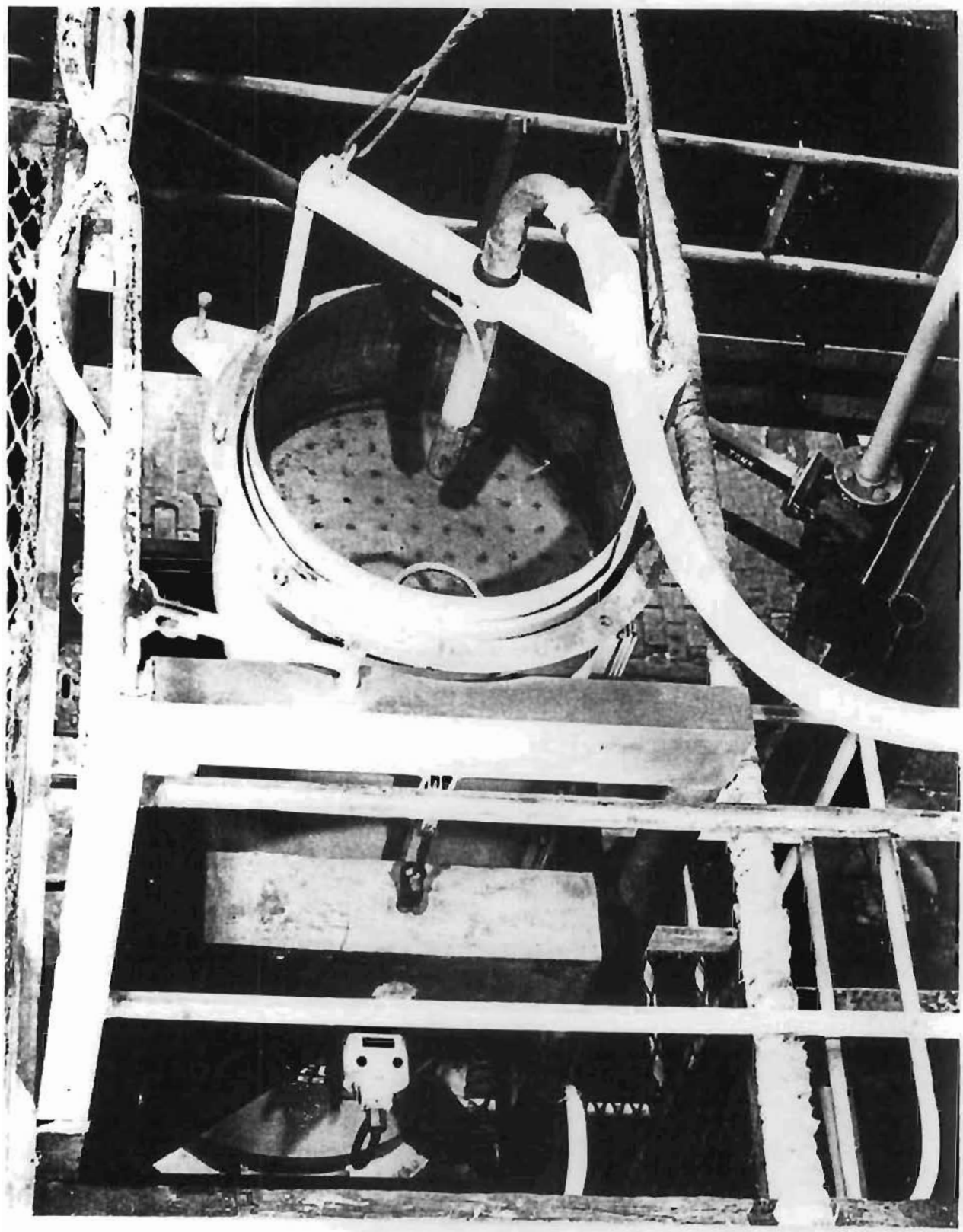


Fig 4.4. Photograph of top of diffusion vessel with the liquid distributor in position

A photograph of the top of the diffusion vessel with the distributor in position is shown in Fig. 4.4. A flexible hose was connected to the inlet pipe, so that the distributor could be raised or lowered during operation, and pulled clear of the vessel during the bagasse loading operation. The liquid inlet pipe was clamped to the frame of the distributor.

One of 3 different orifice plates could be used in the orifice meter depending on the flow rate, to ensure a pressure differential in an acceptable range over the flow rate range of 5 to 60 gal/min. A Foxboro D. P. cell transmitted the signal to a Foxboro 43 A proportional + reset controller.

The surge tank had a capacity of 300 gallons. A photograph of the surge tank in Fig. 4.5 shows in addition the pump, stirrer motor, thermometer pocket and the sightglass for liquid level indication. Temperature control was effected by a Satchwell TL 164 thermostat which controlled an electrically actuated ball valve in the steam injection line. Temperature control within 1 ^{o}C of the desired temperature was possible with this system.

4.2.2 Operation.

The operation of the pilot plant is best illustrated by following in detail the course of an experimental run.

After the bagasse had been prepared and sampled, the amount required for a run (generally 125 lb) was weighed out on a platform scale and loaded into the diffusion vessel via a metal chute inserted into the top of the vessel. After loading, the top of the bagasse bed was levelled by hand, but the bed was not compacted at all. The dry bed height was read on a scale on the side of the perspex window. The liquid distributor was then lowered into the vessel, and secured in a position just above the top of the bagasse bed.

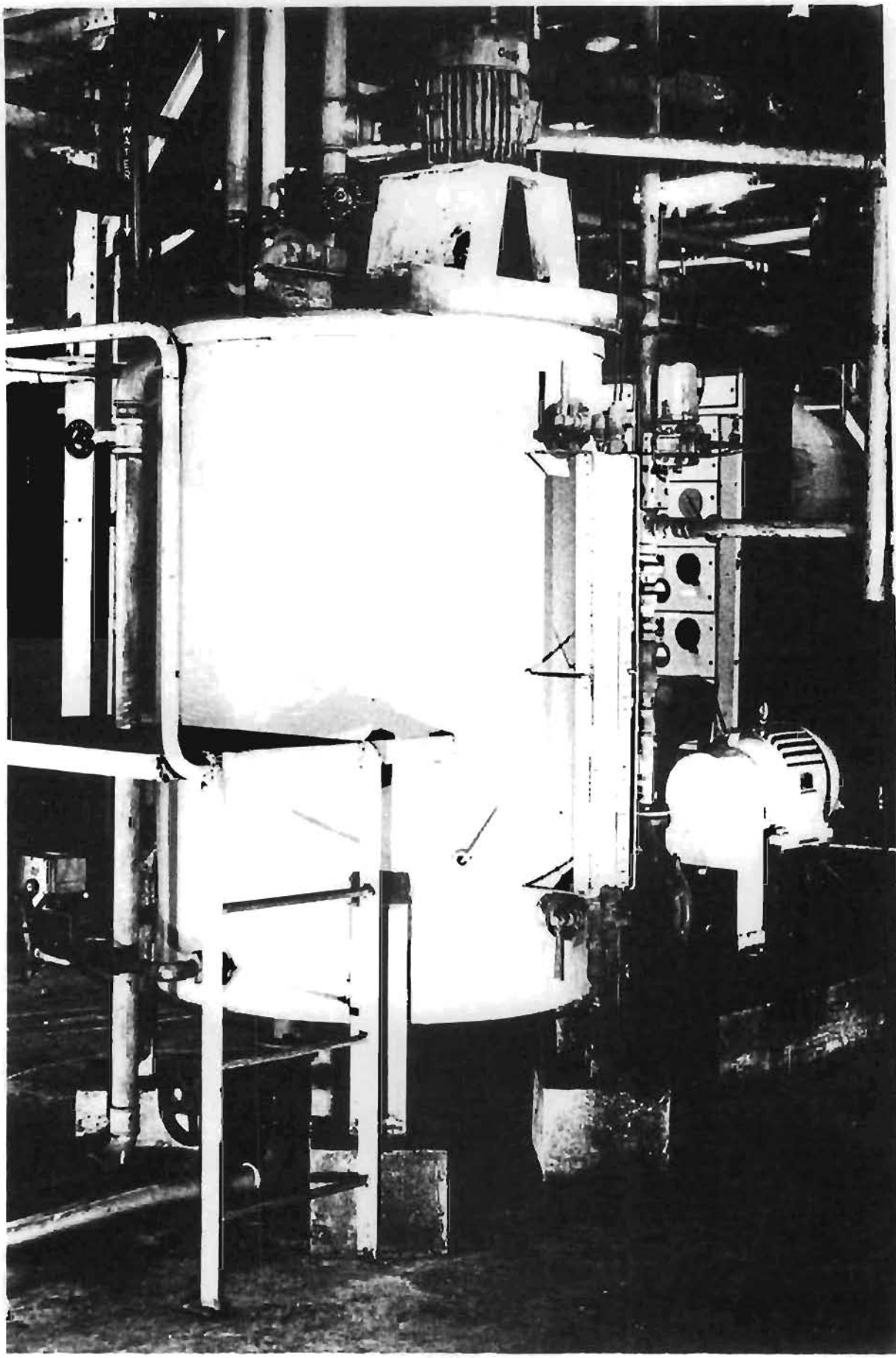


Fig 4. 5. Photograph of the surge tank and associated equipment

The load beam was commissioned by lowering the vessel onto the knife edges by screwing down the jacking screws. The tare on the power supply was adjusted to zero the recorder pen, and the calibration checked by means of the facility provided for this purpose in the power supply.

Two modes of operation of the pilot plant were employed. For most of the runs, water was pumped from the surge tank through the bed, and the effluent collected in a series of drums. Such 'once through' operation was unsuitable for the measurement of impurities extracted. In this case, the surge tank was initially filled to a given level, the liquid pumped through the bed and back to the surge tank, and continuously re-circulated.

In both cases, the water in the tank was allowed to reach the temperature set on the the thermostat before percolation was started. With the 3-way valve turned to permit flow through the by-pass line shown in Fig. 4.1, the pump was turned on and the orifice meter commissioned by bleeding air from the ports of the D.P. cell. The required flow rate was set on the flow controller; to achieve optimum control conditions, it was found necessary to adjust the proportional band of the controller depending on the control setting.

An experimental run was initiated by quickly turning the 3-way valve to close the by-pass and permit flow into the diffusion vessel. When the first liquid appeared at the diffuser outlet, a stopwatch was started, and catch samples of the effluent juice taken at regular pre-determined intervals. The temperature of the effluent liquid was also measured at regular intervals with a mercury thermometer; the inlet temperature of the first juice was considerably lower than the outlet temperature, but generally within $\pm$ 5 to 10 minutes, depending on the temperature and flow rate, the outlet temperature was substantially equal to the inlet temperature.

The bagasse bed consolidated appreciably once percolation started, but after a few minutes had essentially reached its final height. At this stage the liquid distributor was lowered to a position just above the top of the bed, and the height of liquid in the distributor was measured.

At the end of the run, the final bed height was read, before unloading the column. Then the diffusion vessel and piping were throroughly washed and flushed out for the next run.

4.2.3 Calibration of Equipment.

4.2.3.1 Effluent Drums.

Each drum was calibrated individually. A drum was placed on a scale and filled to various levels to determine a weight-level calibration curve. A dip-stick was used to measure liquid level in the drums, and the temperature measured simultaneously. Correction factors were determined for different temperatures.

In all cases, before and after filling each drum, it was covered by a lid to prevent evaporation and the ingress of foreign particulate matter. It is estimated that the weight per drum could be determined to within 3 lb., or less than 1% of its true value.

4.2.3.2 Flow Meter.

The surge tank capacity-level relationship had previously been accurately established by pumping liquid from the tank into drums, and weighing the amount of liquid pumped out. The flow meter was calibrated by pumping liquid out of the surge tank at the required set value of the flow controller over a given time interval, and measuring the reduction in volume of liquid in the surge tank during that time interval. A linear calibration curve with each orifice plate was obtained, which generally showed close agreement with expected flow rates calculated from the orifice diameters.

Whenever the orifice plate was changed, one or more flow rate checks were carried out to verify the validity of the calibration curve.

4.2.3.3 Load Beam.

Calibration of the load beam could be easily carried out by closing the outlet valve on the diffusion vessel, and introducing weighed quantities of water. The recorder pen deflection could then be accurately related to the load. A linear relation between load and pen deflection was confirmed.

As a further check, liquid was pumped into the vessel with the outlet valve closed at a number of different flow rates. In all cases, the slopes of the load-time recorder traces were found to represent accurately the known inlet flow rates.

4.2.3.4 Inlet Liquid Temperatures.

Because of heat losses in the piping between the surge tank and the diffuser, the inlet temperature was found to be slightly lower than the surge tank temperature. Simultaneous measurement of these 2 temperatures showed a drop of 2°-3° C at 90°C, while at 60°C, this drop was less than 1°C. These temperature drops were found to be essentially independent of flow rate. All surge tank temperatures were adjusted accordingly to obtain values of the liquid inlet temperature.

4.2.4 Measurement of Liquid Holdup.

Liquid holdup values are reported as a weight / unit weight of dry insoluble solids (referred to as 'fibre'), rather than as a volume fraction, since it is the amount of fibre which largely determines the amount of liquid retained in the bed. Kocatas & Cornell (1954) reported holdup measurements in beds of soybean flakes in a similar way. Holdup values can be related to values expressed as a volume fraction through liquid density and fibre packing density. Thus for the total liquid holdup, H_T,

$$H_T = \frac{h_T \rho}{q} \qquad (4.1)$$

Dynamic and static holdups, H_D and H_S are similarly related to h_D and h_S.

4.2.4.1 Total Liquid Holdup.

The first few minutes of operation represented transient flow conditions, before the liquid holdup had been established within the bed. This is illustrated by the load beam trace shown in Fig. 4.6. After the column had been loaded with bagasse, the output from the load beam was adjusted to zero, so the total weight of the contents of the diffuser is represented by the recorded weight plus the initial weight of bagasse (in this case 125 lb). Point A in Fig. 4.6 represents the point at which the first liquid is applied to the bagasse bed. The weight rises continuously at a rate determined by the inlet flow rate, until at point B the first liquid appears at the diffuser outlet. The times at which these events were observed could be conveniently marked by applying a quick slight pressure by hand to the diffuser vessel. Over the region B to C, liquid builds up in the bed and the outlet flow rate gradually increases until at point C, the inlet and outlet flow rates are equal. From C to D, the trace generally remained roughly constant (under non-flooding conditions). All holdup measurements were made over this period of steady-state flow conditions. The region D to E represents drainage of liquid from the bed.

Numerical values of the weights recorded were found to be unreliable. This was unexpected, since calibration with water showed that very accurate results could be obtained With flow through a packed bed of bagasse however, any maldistribution of liquid over the column cross-section seriously affects recorded values. With the physical geometry employed, it can be easily shown that a 1.5" shift of the centre of gravity towards or away from the load beam results in an error in the measured weight of $\pm$ 10%.

Discrepancies in the values of H_T and/or the slope of the line representing the inlet flow rate were observed. From the trace shown in Fig. 4.6, the calculated value of H_T is 9.00 lb/lb fibre compared with the estimated actual value of 8.84 lb/lb fibre. However, the slope of the trace over the region AB represents fairly well the actual inlet flow rate. These findings are indicative of various degrees of liquid maldistribution; from the magnitude of the discrepancies such maldistrubution is slight, but sufficient to invalidate accurate measurements.

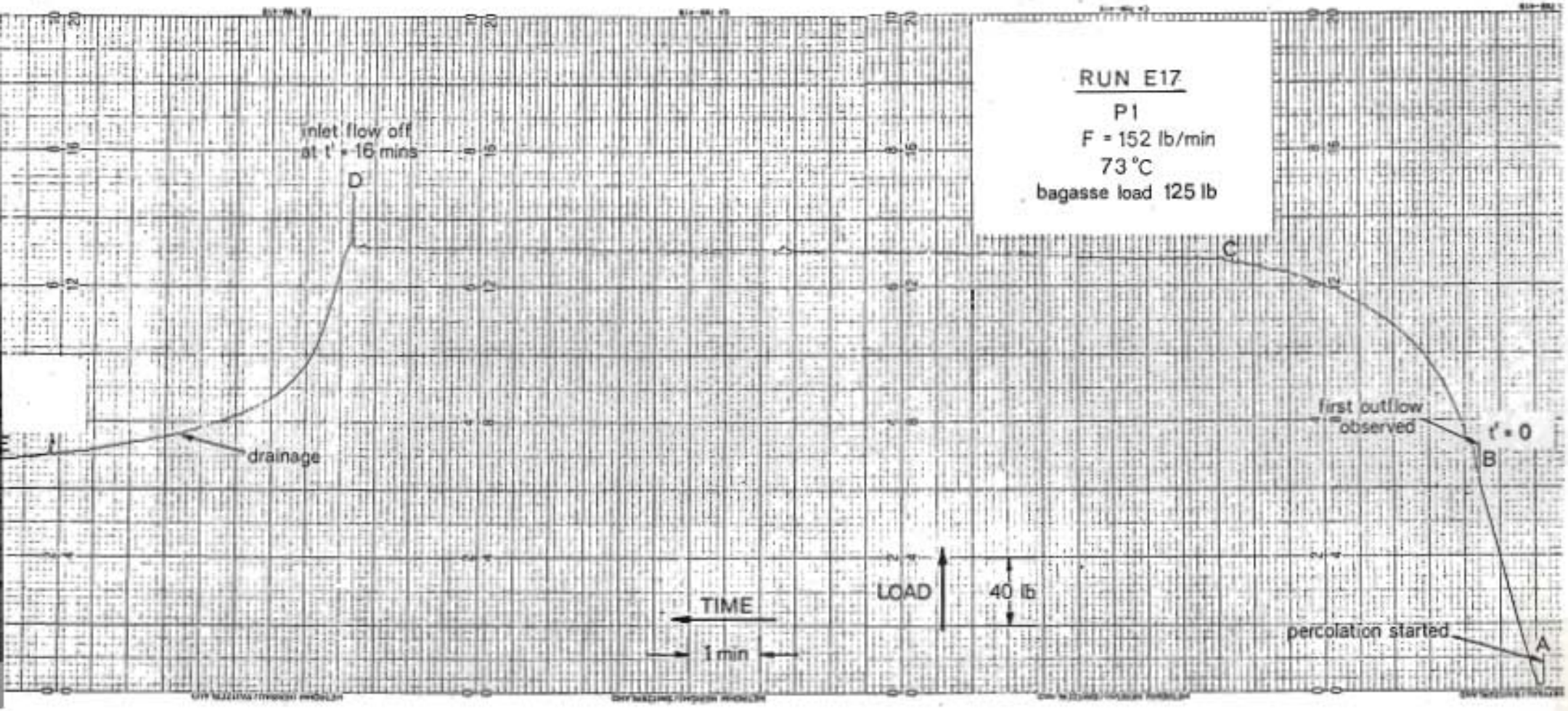


Fig. 4.6. Typical load beam trace

The load beam therefore had utility for qualitative purposes only. It could be used to infer the time required to attain steady-state flow conditions, to indicate the presence of flooding, and was sensitive enough to respond to changes in flow rate. It was also used as a check on suspect valves of H_T.

Instead the difference between cumulative inflow and outflow values was used to determine H_T. This is illustrated in Fig. 4.7; the cumulative outflow curve was obtained from the weight of liquid retained in the drums over known time intervals. The distance XY represents the weight of liquid retained in the bed, plus the liquid in the distributor. The latter was determined from the height of liquid in the distributor, and although relatively small, was subtracted from this weight. Finally, the initial weight of liquid in the bagasse was added to yield the total liquid holdup.

It is clear that if the slopes of the 2 lines in Fig. 4.7 are equal (i.e. input and output flow rates equal), then the weight represented by the distance XY is also given by the product of inlet flow rate and the time t_H. In practice this is easier to calculate. The slope of the cumulative outlow curve could be used as a check on the inlet flow rate in the absence of flooding. It is estimated that measurements of H_T are accurate to within $\pm$ 2%.

Under flooding conditions, the slope of the outlet curve was less than that of the inlet line, indicating a progressive build-up of liquid in the bed. In this case, H_T can be calculated in the same way using the distance XY, but it is then a function of time. For the purposes of the model computations, an estimate of H_S is required under flooding conditions. In this case the value of H_T at $t = t_H$ was used in calculating H_S.

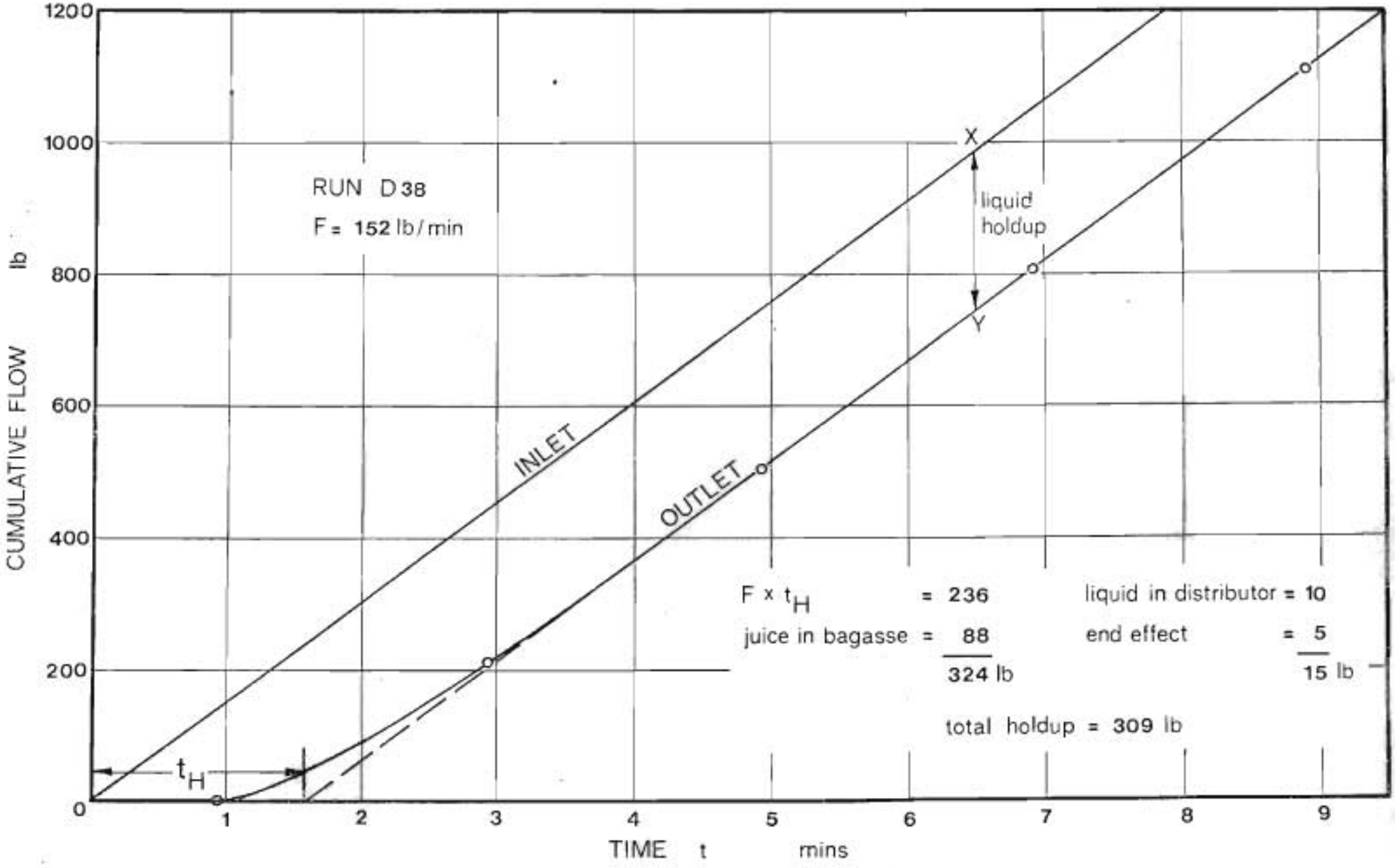


Fig. 4.7. Illustration of the method of determining the total liquid holdup in the diffuser vessel

4.2.4.2 Dynamic Holdup.

The determination of the dynamic holdup, H_D, involved the measurement of the mean residence time, τ (see section 5.3.1), which was determined by a tracer test. Roughly, 10 ml of an aqueous solution of a green vegetable dye was introduced by hand into the liquid distributor at a predetermined time to represent a pulse input. The dye employed was manufactured by Bush, Boake and Allen (S.A.) Pty Ltd. (trade name 'Permicol'). The volume of dye introduced was small enough not to affect the flow rate. The response curve was obtained from the measurement of light absorption of catch samples of the outlet liquid, measured on a Unicam S.P. 1300 Colorimeter. At least 3 such determinations were made in the course of a run; Fig. 4.8 shows typical response curves. The value of τ was determined as the time interval between the introduction of the dye and the peak in the response curve. Generally, the repeat determinations of τ in a run agreed to within 5%, and better agreement was obtained with higher values of τ. In a few cases, wide differences in the values of τ were obtained, in which case, the value of τ was rejected and a value of H_D was not obtained.

4.2.4.3 End Effects.

It was necessary to establish whether an 'end effect' should be taken into account ie. whether there is significant liquid holdup in the column in the absence of a bagasse bed. This was done in 2 ways. Firstly, the difference in the weights before and during liquid flow through the empty column was determined with the load beam. This showed an increase in weight of 5 lb $\pm$ 1 lb, independent of flow rate. Further, after the flow was stopped, the weight returned to its original value, indicating that all liquid drains freely from the vessel. Secondly, tracer tests were carried out in the empty column with the liquid distributor lowered to a few inches above the screen. These tests indicated a 'dynamic holdup' of the empty column of 3 to 7 lb. This value was more affected by the liquid height in the distributor than by flow rate.

As a result of these tests, 5 lb was subtracted from measured values of total and dynamic holdup for all flow rates. The likely error of $\pm$ 2 lb is within the accuracy of the determination of these quantities.

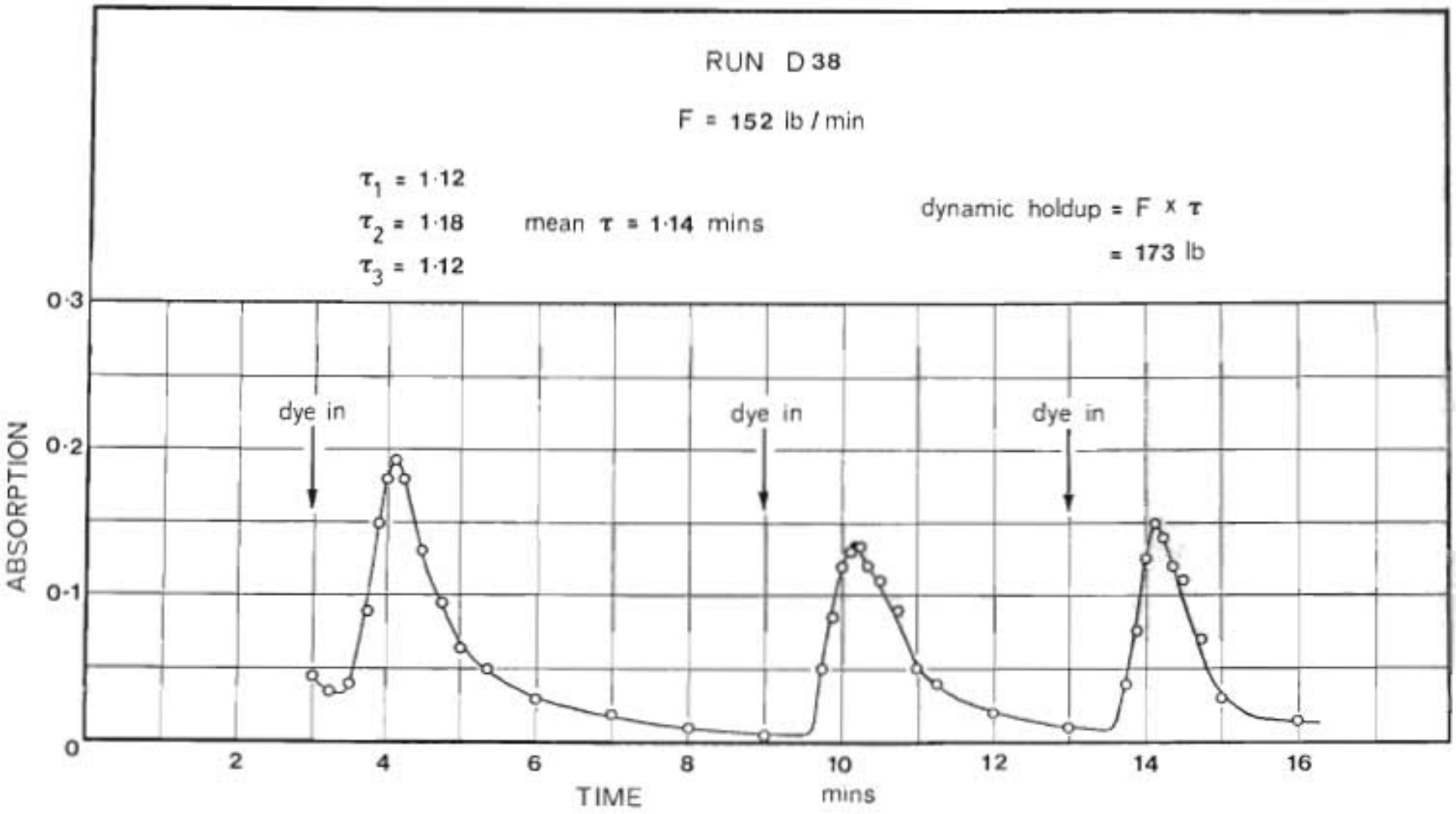


Fig. 4. 8. Typical response curves for the evaluation of the dynamic liquid holdup.

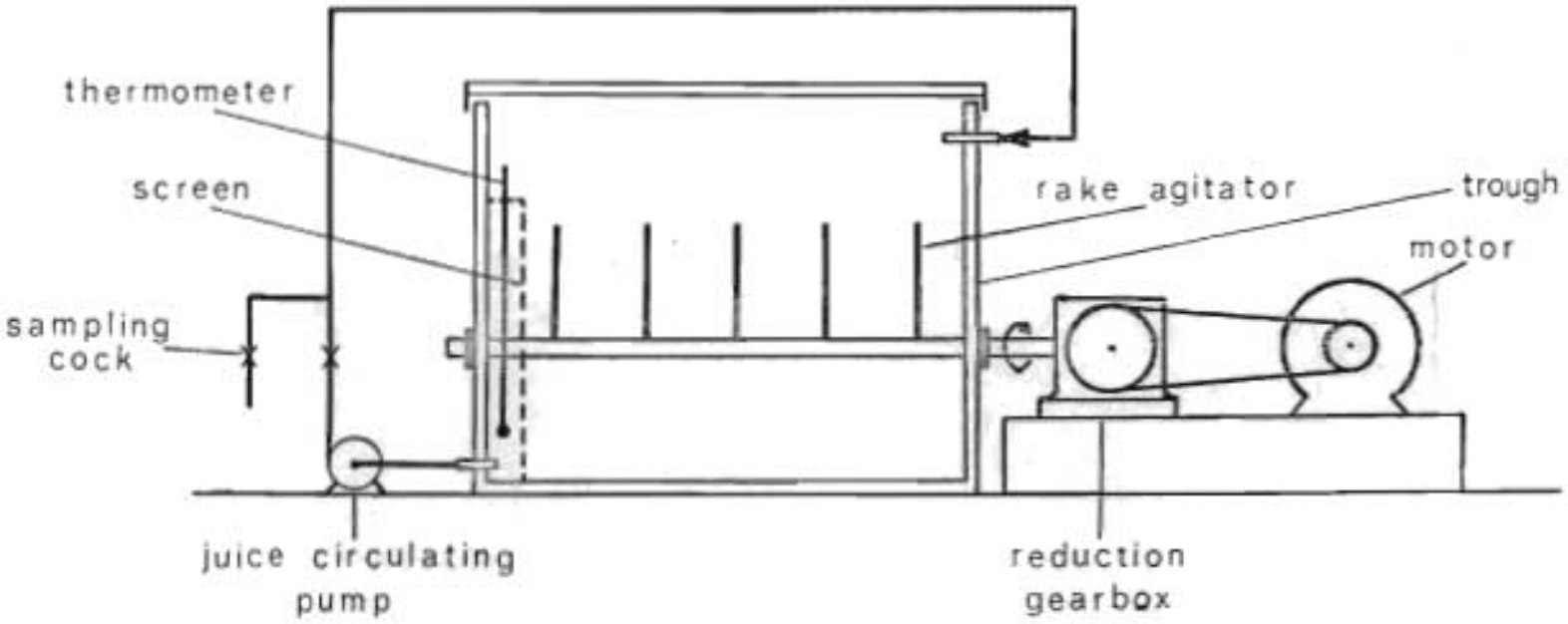


Fig 4.9. Schematic diagram of the laboratory-scale extraction equipment

4.3 LABORATORY EXTRACTION TESTS.

4.3.1 Equipment.

The equipment used in these tests was obtained on loan from the South African Sugar Milling Research Institute, and is illustrated schematically in Fig. 4.9. It consisted of a U-shaped trough 12" in diameter and 12" long, with transparent perspex ends. The trough was fitted with a rotating rake-type paddle, driven by a motor through a reduction gearbox at a speed of 19.7 r.p.m.; its function was to ensure good mixing between bagasse and juice in the trough. Juice was pumped via a small centrifugal pump from the bottom of one side of the trough round to the other side. A sampling cock in this recirculation line allowed representitive sampling of juice. The trough was fitted with a screen at the juice withdrawal end to prevent bagasse particles from being drawn into the pump.

A water jacket was attached to the trough (not shown in Fig. 4.9). Water was pumped from a thermostatically controlled tank through the jacket to maintain the contents of the trough at a desired temperature set-point.

4.3.2 Operation.

A measured quantity of water (9 kg) was introduced into the trough, and allowed to circulate until the heating system brought the water to the desired temperature. Before introducing the bagasse, a sample of the water was taken; because of the difficulty experienced in cleaning the equipment thoroughly after each run, this sample give a brix reading between 0.0 and 0.07.

Two kg of bagasse was introduced into the trough over a period of ± 5 seconds, to avoid jamming the rotating paddle. A stop-watch was started when roughly half the bagasse had been introduced, and juice samples were taken at pre-determined times for the measurement of brix. The amount of juice removed during sampling was ± 300 ml for most of the runs, but ± 500 ml for the earlier runs. Since the lid on the trough was not completely air-tight, some evaporation ocurred. This was determined as ± 200 ml over the whole run from the initial introduction of the water. Thus the average reduction in the liquid during a run was 250 ml and 350 ml for the 2 cases; although this quantity is small in relation to the initial amount, it was taken into account in the computations.

On introduction of the bagasse, the temperature indicated by the thermometer in the liquid dropped. Within 5 to 10 minutes, the juice temperature was within a few degrees of the required temperature.

4.4 BAGASSE PREPARATION, ANALYSIS AND CHARACTERIZATION.

4.4.1 Preparation.

All bagasse used in this study was collected from the milling train of the Huletts Mount Edgecombe mill, after the first mill but before the application of imbibition. Details of the preliminary cane preparation equipment and the first mill of this tandem are:

1st knife set:	direct drive 375 HP motor, 500 r.p.m.
2nd knife set:	direct drive 420 HP motor, 435 r.p.m.
Shredder:	Gruendler, 120 x 38 lb hammers, driven by 600 HP motor at 960 r.p.m.
1st Mill:	84" x 41", with feed roller, variable speed steam turbine drive.

The average crushing rate during the period of the experiments was 167 metric tons cane/hour, corresponding to 25.7 metric tons fibre/hour. No changes were made to the configuration or drives of this equipment during the course of the experimental work, except that the mill settings were changed slightly each season according to the estimated average crushing rate.

Enough bagasse for the number of runs anticipated in one day was collected early on the same day. Since the bagasse was obtained with the use of a grab, it was collected over a period of $\pm$ 1 hour. This was advantageous in that the bagasse was collected from a number of different cane consignments, and was therefore more representative of the average cane crushed.

When required, the bagasse could be more finely prepared by feeding it through a Hippo Hammer Mill (a fixed hammer shredder). The model used (Hippo Mill size 1) was 8" wide, with 4 sets of 3 hammers (or beaters) 6" long,

driven by a motor at roughly 4300 r.p.m, and rated at 6-15 HP. Four levels of bagasse preparation were employed:

(1) P1 - straight first mill bagasse.

(2) P2 - first mill bagasse, fed through the Hippo Mill.

(3) P3 - first mill bagasse, fed through the Hippo Mill, with a coarse screen over the outlet (1 1/4" D holes).

(4) P4 - first mill bagasse, fed through the Hippo Mill, with a fine screen over the outlet (1/4" D holes).

Attempts were made to reproduce each level of preparation consistently, by feeding the bagasse continuously and at a constant rate through the Hippo Mill.

4.4.2 Bagasse Analysis.

When the bagasse had been collected and further prepared if required, it was thoroughly mixed and sampled as representitively as possible before the first run of the day. After the diffusion vessel had been loaded for the first run, the remaining bagasse was well covered to prevent evaporation or contamination by foreign matter.

The bagasse sample was divided into 2 portions, one of which was used for the DI determination and sieve analysis, while the other was analysed for moisture and fibre, and brix was determined by difference. All samples were kept in air-tight containers. The latter sample was initially disintegrated in a Jeffco cutter-grinder prior to analysis (except for preparation P4 which was already fine enough). Moisture was determined by drying in a Dietert Moisture Teller for 40 minutes at 110°C, while fibre was determined according to the method of Prince (1969). This method of bagasse analysis had been previously established as being accurate to within $\pm$ 0.2% (Prince, 1969a)

4.4.3 DI and Sieve Analysis.

The DI was determined by the method outlined by Markham (1969), except that brix and not pol measurements were used. The test was done in duplicate and an average of the 2 values taken. Both results invariably agreed within 1% of each other.

The sample for sieve analysis was first thoroughly dried in a moisture teller and then sieved in 2 stages. Firstly, the bagasse was sieved by hand in a sieving box for 2 minutes, with screen apertures of 10.6, 5.0 and 2.8 mm; then the residue was transferred to a JEL mechanical shaker, and sieved in a series of Tyler screens with apertures of 1.65, 1.00, 0.589 and 0.351 mm for 10 minutes.

Values of x_i used in the evaluation of d_{PT} (equation 5.1) and S (equation 5.2) are shown below:

Fraction.	Values of x_i (mm) for Equation (5.1)	Equation (5.2)
+2	10.0	10.6
-2/+4	6.2	8.1
-4/+8	3.5	4.0
-8/+10	2.1	2.3
-10/+16	1.3	1.35
-16/+28	0.8	0.81
-28/+42	0.45	0.48
-42	0.2	0.22

The first set of values was obtained from a direct particle count (see section 5.1.4), while the second set were calculated according to the following equation given by Herdan (1960):

$$x_i = \left(1/4 \left(x_1^2 + x_2^2\right) \left(x_1 + x_2\right) \right)^{1/3} \qquad (4.2)$$

where x_1 and x_2 refer to the screen aperture sizes of the ith screen and the screen above the ith screen.

4.4.4. Evaluation of Particle Size and Shape.

A sample of dried bagasse was sieved in the normal way, as described in the previous section, and the fraction retained on each screen was studied individually. This had the dual purpose of separating the particles into fractions of approximately the same size, and also the results of the investigation could be related to sieving results.

Particles from the 3 coarse screens of the first sieving stage were large enough for direct measurement of length, breadth and thickness of each particles. This was achieved by placing the particle on graph paper ruled in mm. The images of particles retained on the finer screens were projected directly onto photographic paper, with suitable magnification. Measurement of the thickness of these particles was not attempted. Measurement of the other 2 dimensions was made with the use of transparent graph paper placed over the image of each particle. These measurements were then converted to actual sizes by dividing by the magnification factor.

Length, breadth and thickness were measured according to the definitions of Heywood (1937) (see section 5.1.4). In addition, because of the nature of bagasse, some arbitrary rules were followed. When measuring long fibres which were bent or twisted, these were first straightened so that the breadth is a true measure of the width of the particle. Otherwise the length is smaller and the breadth much larger when the fibre is bent, according to Heywood's definitions. Similarly, single fibres protruding from larger particles were aligned in a direction parallel to the other fibres in the particle.

4.5 ANALYSIS OF JUICE SAMPLES.

The methods of analysis used are summarised below:

Measurement of:	Measured by:
brix	Bausch & Lomb precision refractometer.
pol	Horne's dry lead method.
pH	Glass electrode pH meter.
reducing sugars	Reduction of ferricyanide (Comrie 1970).
sulphated ash	Method described in 'Laboratory Manual for S. A. Sugar Factories' (S. A. Sugar Technologists Assoc. 1962).
conductivity	Dionic conductivity meter, with temperature compensation (conductivity at 20°C reported)
K	Flame photometry.
Na	Flame photometry.
Ca	Atomic absorption spectrophotometry.
Mg	Atomic absorption spectrophotometry.
chloride	Automatic potentiometric titration (Comrie, 1969).
starch	Starch-iodine colorimetric method, based on method of Alexander (1954), but dissolved starch precipitated with alcohol, and precipitate washed with alcohol only.

Because of the low concentration of reducing sugars in the juice samples, the commonly used Lane and Eynon method for the determination of reducing sugars could not be employed. Instead a method based on the reduction of ferricyanide was used (Comrie, 1970) because of its sensitivity, accuracy and ease of analysis. As a check on the results obtained, the Luff-Schoorl determination was carried out simultaneously on a number of juice samples, although the latter method is known to

be unreliable due to atmospheric oxidation of cuprous ions. The results of this comparison are given below:

Sample No.	Percentage reducing sugars in sample by: Ferricyanide Method	Luff-Schoorl Method
1	0.100	0.091
2	0.058	0.052
3	0.062	0.053
4	0.063	0.055
5	0.067	0.058

Agreement is fair. Of the 2 sets of results, those obtained by ferricyanide reduction are considered to be more accurate.

CHAPTER 5.

RESULTS

The results of this study consist basically of two parts, firstly the laboratory mixing experiments and secondly the pilot plant diffuser investigations. The characterization of bagasse is discussed initially, as this is relevant to both laboratory and pilot plant results.

Experiments on the laboratory scale involve a simple physical system, and form a convenient entity per se. These results find utility as a basis for comparison with the pilot plant results.

Experiments on the pilot plant scale are complicated by the hydrodynamics of flow through a bagasse bed. Aspects of the flow conditions, namely liquid holdup and the occurence of flooding , are discussed prior to the application of the mathematical model to the results, and are utilized to explain observations on the rate of extraction. Further, parameter estimation from the pilot plant data proved to be more difficult, and the methods employed are described in relation to the pilot plant results, although the same estimation procedures were applied to the laboratory scale data.

The prime object of this investigation is the development of a mathematical model which will enable accurate prediction of extraction on the plant scale, and allow the choice of optimum operating conditions. Thus the extractions achieved in the pilot plant diffuser have little importance in this investigation. Sucrose extractions achieved are therefore briefly presented, with reference to the effect of the operating variables on extraction.

Finally, the results of investigations into the extraction of certain impurities are described.

5.1 BAGASSE CHARACTERISATION

The effects of the quality of bagasse on diffuser performance may be broadly classified as :-

i. Macro-effects, related to particle size, shape and size distribution, which affect packing density, liquid holdup and the maximum obtainable percolation rate, and

ii. micro-effects, in which the effect of the structure of the bagasse manifests itself in the rate of extraction.

Both macro- and micro- effects are significant in the extraction process. Thus methods of assessing preparation should ideally account for both the macro-effects relating to the performance in a bagasse bed, and the effect on extraction on the micro-scale. However, the diverse nature of bagasse referred to in section 2.2 has thus far prevented the attainment of this ideal facility.

In this investigation, sieve analysis and DI have been used as measures of preparation. Other methods of characterisation were considered; the use of air classification (elutriation) in a vertical column in particular was extensively investigated. This method was found to be inefficient and less reproducible than the methods used. Consistent feeding of bagasse into the air stream proved to be a major difficulty; Herdan (1960) has confirmed that this is the major experimental problem associated with this method.

Thus characterisation of bagasse was restricted to the two methods mentioned above. For the purposes of this project, in order to give a more comprehensive description of the type of bagasse used in these experiments, a direct particle evaluation was undertaken, in which particle sizes and aspect ratios of individual particles were measured, and the results related to sieving tests.

5.1.1 Visual Observations On Prepared Bagasse

Some brief observations on the nature of the bagasse employed are given in order to supplement quantitative information which is generally inadequate for a complete description of bagasse.

Photographs of bagasse subjected to the four types of preparation are given in Figures 5.1 to 5.4. Preparations P1 and P2 are similar in nature; in both cases, nearly all the larger particles consist mainly of rind material, with some softer tissue attached in some cases. These particles show signs of having been compressed in the 1st mill. The larger particles of P1 preparation appeared to be roughly 8 cm long, although longer particles have been observed. P2 preparation showed clearly that the maximum particle length was decreased to about 6 cm. Pith material appeared to be present in the form of small balls and flakes.

Preparations P3 and P4 showed a high degree of fibre separation, i.e. most of the sugar containing cells had been stripped from the fibres. This was particularly noticeable with preparation P4. Pieces of rind could be discerned in P3 bagasse, and although there still appeared to be a number of long fibres, maximum fibre length was reduced to less than 5 cm.

Prepared bagasse was also studied under a microscope. The material was first boiled in KOH solution to remove the contents of the cells, which rendered the bagasse soft and spongy. The bagasse particles could then be compressed under a coverslip without damage to unbroken cells. Nile Blue was used as a stain to outline the cell walls prior to observation.

It was hoped that an estimate of the numbers of broken and unbroken cells could be obtained from microscopic examination. This proved to be a virtually impossible task, and it was felt that no quantitative information could be obtained in this way. However, some observations are reported.

Fig 5.1. Photograph of bagasse, preparation type P1

Fig 5. 2. Photograph of bagasse, preparation type P2

Fig 5. 3. Photograph of bagasse, preparation type P3

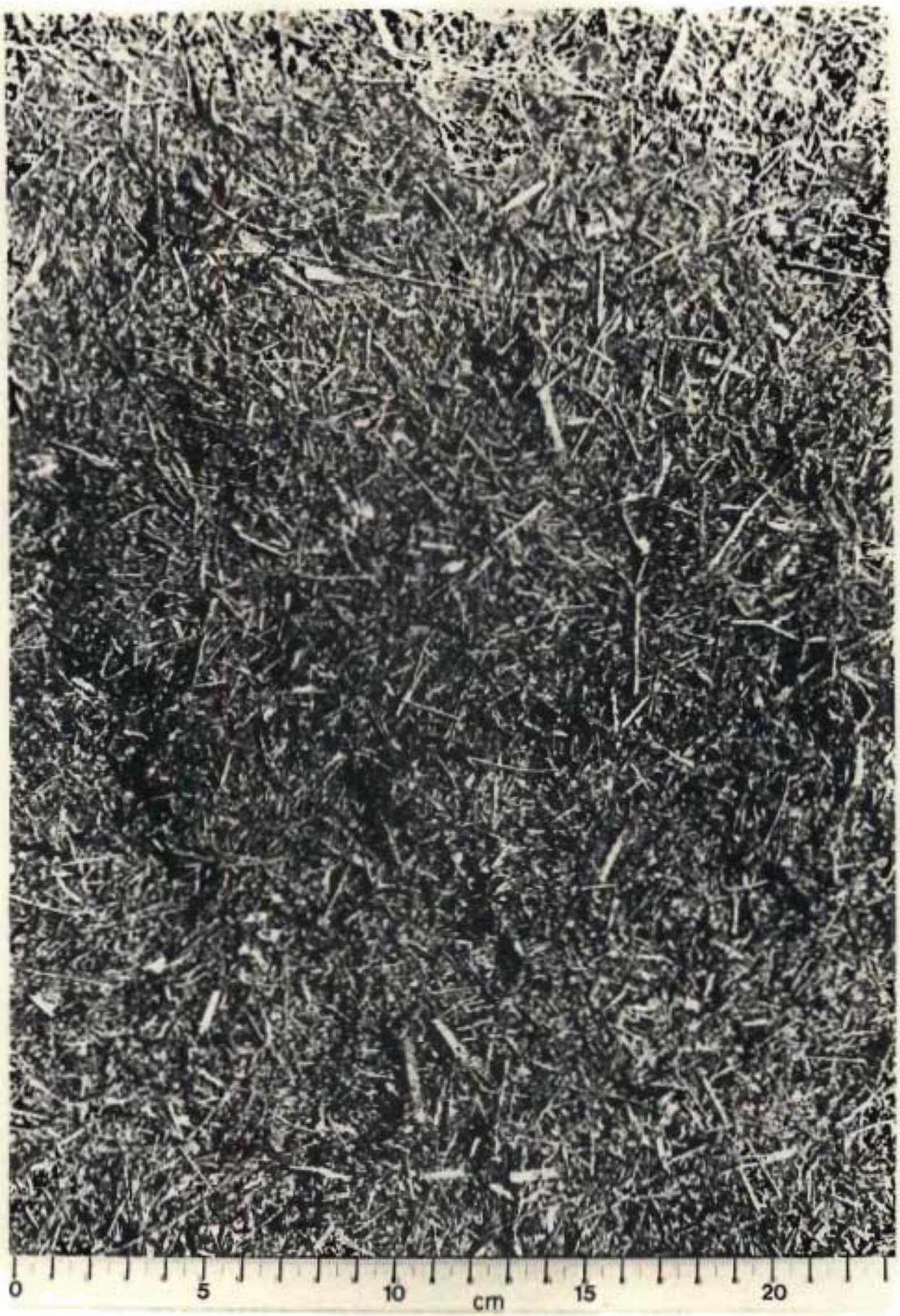


Fig 5.4. Photograph of bagasse, preparation type P4

Small particles of pith (~ 1 mm) showed that all cells had been ruptured. Complex paths between broken cells could be traced. Some unbroken cells were apparent in the interior of slightly larger pith particles (- 3 mm), possibly as a result of the cushioning effect of the outer cells. Parenchyma cells attached to single fibres were generally all ruptured. However, in some particles which had a number of closely spaced fibres traversing them, a relatively high proportion of intact cells could be discerned, presumably shielded during preparation by the surrounding fibres. This effect was less noticeable with the finer preparations employed.

As regards the extent of cell rupture, therefore, it would appear that a good preparation should aim at as high a separation of individual fibres as possible.

5.1.2 Sieve Analysis

The results of any sieve analysis are very dependent on the exact procedure followed. Errors which can be introduced in sieving are well covered by Herdan (1960). Two important sources of discrepancies over which the experimenter has control are the effect of sieve loading and the sieving time. These were investigated in the sieving of bagasse by Huletts Research & Development staff prior to the commencement of this project, and their recommendations were followed, as outlined in section 4.4.3. The maximum loading of the small screens consistent with adequate resolution was found to be ~ 30 g dried bagasse. This generally dictated the total sample size to be sieved; loading of the coarse screens was not critical in the range of sample sizes used. It was found that longer sieving times led to higher efficiencies, which is to be expected. However, it was felt that the advantage to be gained from higher efficiencies obtainable with longer sieving times was not warranted at the expense of longer time requirements.

It is obvious at the outset that sieve analysis can only yield relative results, which can be used for comparison purposes, providing the same sieving conditions are consistently adhered to. In sieving bagasse, it was found that low sieving efficiencies were obtained. The rough irregular nature and high elongation (length / breadth) ratios of the particles were a contributory factor. Furthermore tangling of fibrous particles with each other and with the screen aggravated the situation.

Sieving results in terms of cumulative weight % retained on each sieve plotted against sieve aperture size using log-normal co-ordinates are shown in Fig. 5.5 Typical results for each of the four methods of preparation, applied to the same cane, are shown, together with corresponding values of DI. The results do not plot as straight lines, but show changes in slope. These changes in slope correspond with the change from coarse to fine screens which suggests that they reflect differences in the two sieving procedures.

This was investigated further as follows: any particle retained on one of the coarse sieves which could have passed through the sieve was pushed through by hand, while sieving of the finer particles was continued for a further thirty minutes. The original distribution and the modified distribution obtained by this procedure are shown in Fig. 5.6, marked as AA and BB respectively. The modified distribution is a close approximation to a log-normal distribution. Fig. 5.6 shows the results applied to a sample of P2 preparation; similar results obtained with the other preparations as well.

5.1.2.1 Representation of Particle Size from Sieving.

The results of sieving tests were represented in three ways:

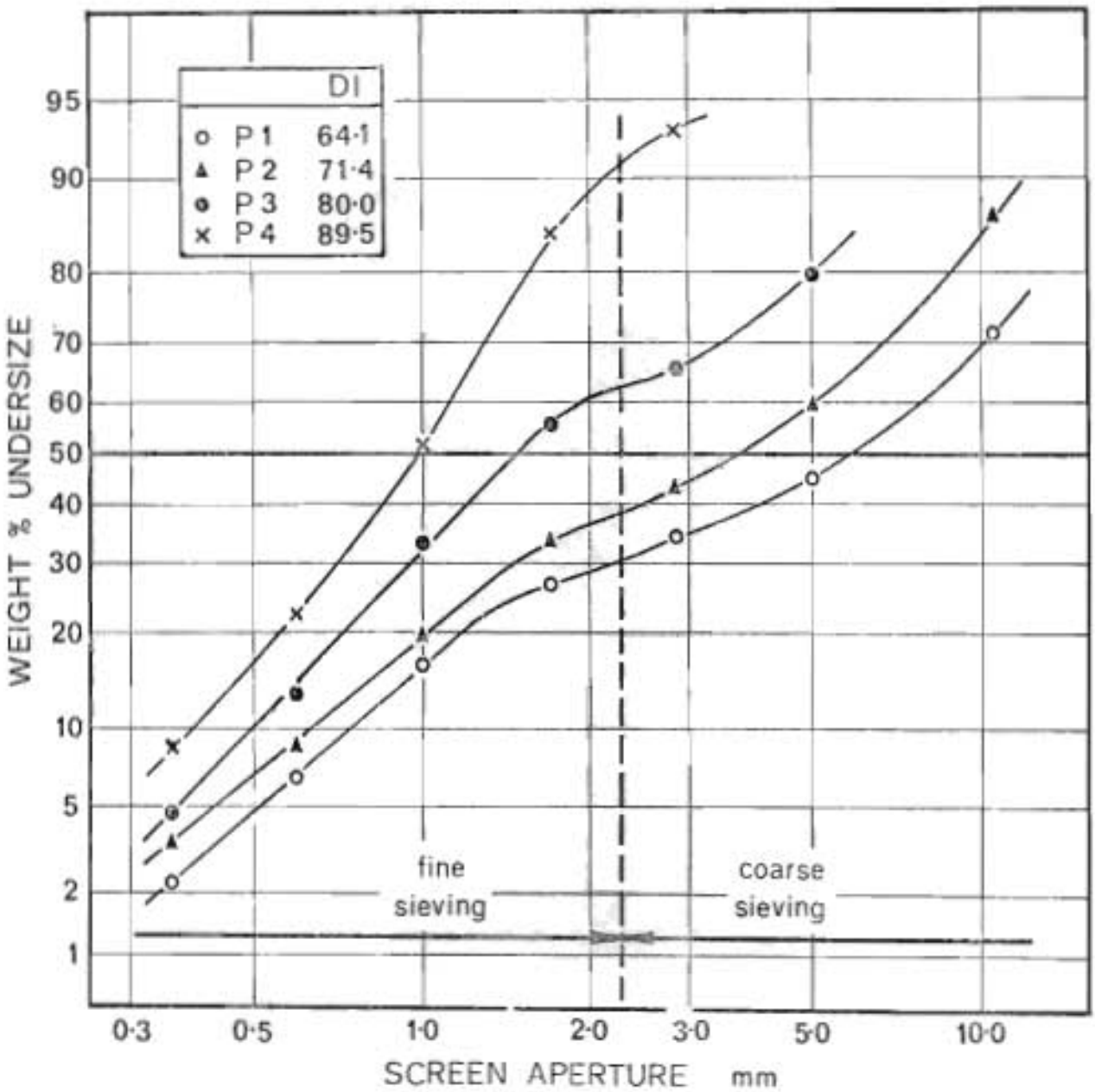


Fig. 5. 5. Typical results of sieve analyses for the 4 different types of bagasse preparation

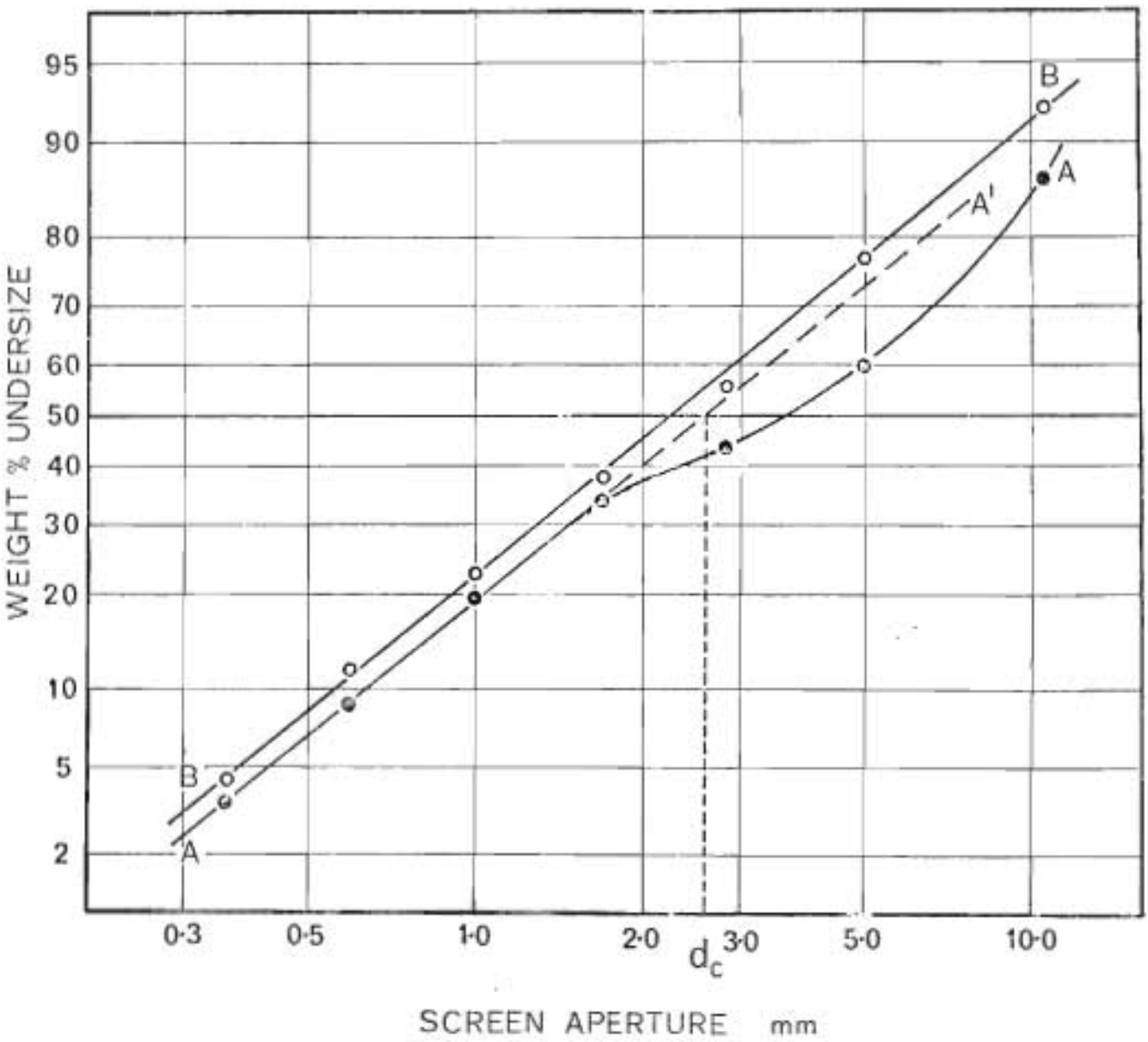


Fig 5. 6. Bagasse particle size distribution. Preparation P2

i. The average particle thickness, d_{pT}, as used by Foster & Hill (1966), was calculated i.e.

$$d_{pT} = \frac{1}{100} \sum w_i x_i \qquad (2.2)$$

The values of x_i used in this equation were chosen according to the results of direct particle measurements (see section 5.1.4) and are given in section 4.4.3. It should be noted that values of d_{pT} are weighted in favour of the larger particles.

ii. A characteristic particle diameter d_c, was obtained from a log-normal plot of the cumulative size distribution. Referring to Fig. 5.6, the straight line portion of the curve AA was extrapolated to A' ; the x co-ordinate of the intersection of the line AA' and the 50% horizontal was taken as the characteristic particle diameter, d_c , which should be a more realistic measure of mean particle size.

Plotting the distribution curves from sieve analysis in this way yields more information than calculation of d_{pT} only. Fig. 5.5 has demonstrated the relative efficiencies of the two sieving stages, and in addition, the slope of the line AA' in Fig. 5.6 yields additional information in the form of the standard deviation, σ_g , which is a potentially useful quantity. Figure 5.5 shows that the spread, and hence σ_g decreases with finer preparations.

iii. A measure of particle surface area is desirable for use in correlating mass transfer coefficients which are dependent on the mass transfer area. Herdan (1960) has reported that the specific surface (area per unit weight), S, may be obtained from sieving tests according to:

$$S = \frac{\alpha_s}{\alpha_v \rho} \quad \frac{1}{100} \quad \sum \frac{w_i}{x_i} \qquad (5.1)$$

where ρ is the solid density in g/cm^3, x_i refers to the mean particle size of the corresponding size interval calculated as the weight or volume average, and α_s and α_v are surface and volume shape factors respectively. For spherical particles, the ratio $\alpha_s / \alpha_v = 6$ and is higher for particles of any other shape. Shape factors could not be estimated for bagasse particles according to normal methods (Heywood, 1947), because of their unusual and irregular shapes. However, if it can be assumed that the ratio of shape factors remains constant, equation (5.1) could find utility in providing relative values of the specific surface. In order to obtain order of magnitude estimates of the specific surface, it was assumed that $\alpha_s / \alpha_v = 6 \rho$. Then equation (5.1) becomes:

$$S = 6 / 100 \sum \frac{w_i}{x_i} \times 1000$$

$$= 60 \sum \frac{w_i}{x_i} \qquad (5.2)$$

The additional factor of 1000 is necessary to express values of S in mm^2/g. Values of x_i were calculated from sieve aperture sizes according to the equation of Mellor as given by Herdan (1960), and are tabulated in section 4.4.3.

The specific surface is similar to the 'fineness index' introduced by Behne given in equation (2.1). Values of S are weighted in favour of small particle sizes.

If the size distribution is approximately log-normal, Herdan (1960) has shown that the specific surface, S is given by :

$$\log S = \log \frac{\alpha_s}{\alpha_v \rho} - \log x_g' + 1.151 \log^2 \sigma_g \qquad (5.3)$$

where x_g' is the geometric mean of the weight distribution. Values of S calculated from this equation, using d_c as an approximation to x_g', and the same value of α_s / α_v assumed above, are compared with values of S calculated from equation (5.2)

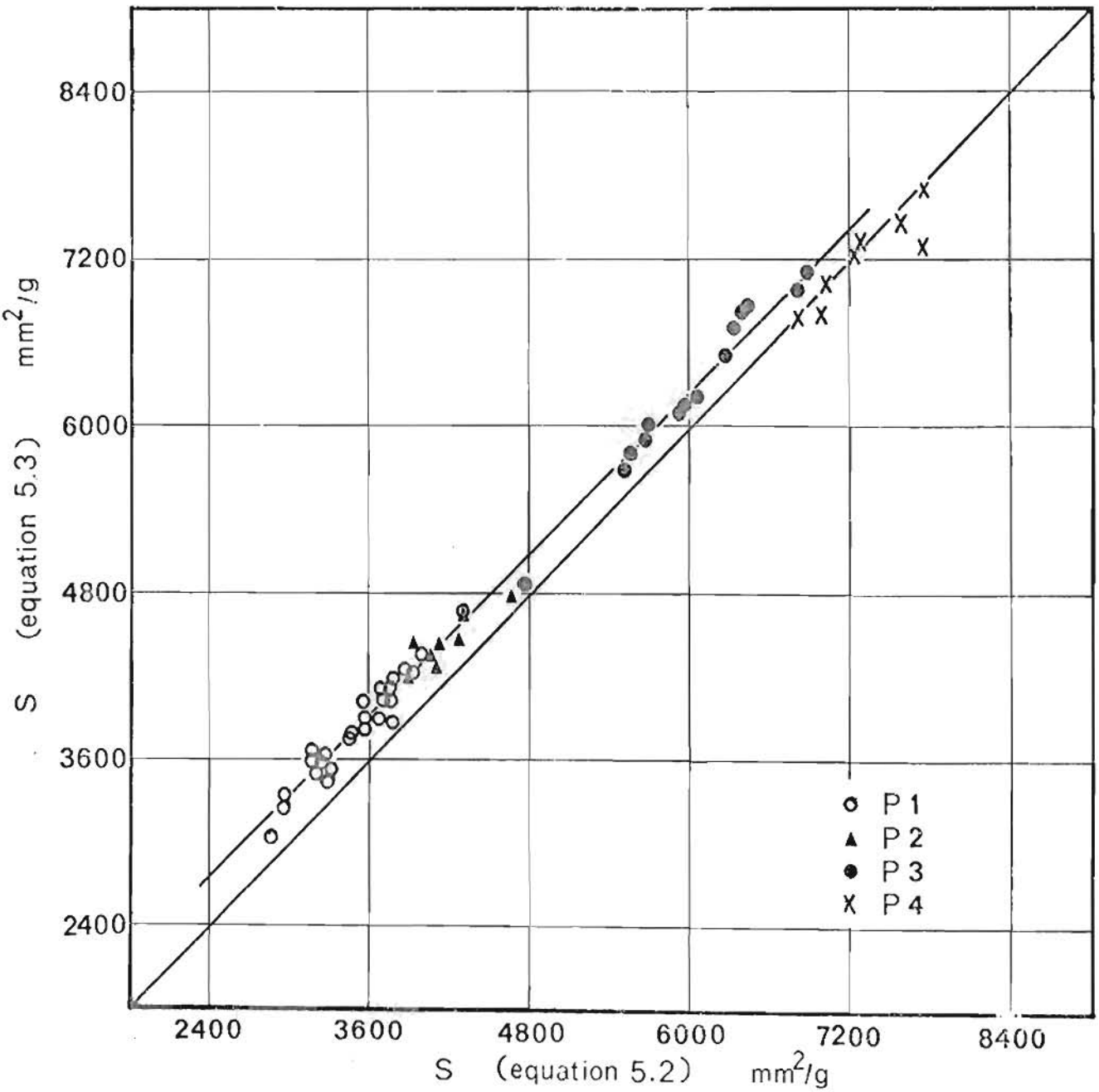


Fig. 5.7. Comparison of different methods of calculating specific surface area. All runs, D-H series.

in Figure 5.7. Values of S from equation (5.3) are consistently higher by = 300 mm^2/g for the coarser preparations. This discrepancy disappears with fine preparations where the amount retained on the coarse screens is too small to affect values of S significantly. The agreement between the two methods of calculation lends weight to the hypothesis that bagasse particles follow a log-normal distribution.

5.1.2.2 Reproducibility

The reproducibility of sieve tests has been discussed by Herdan (1960) and Heywood (1947) who show that results are very sensitive to slight changes in technique and equipment. Table 5.1 shows results of repeat determinations made on subsamples of the same bagasse.

Table 5.1 Reproducibility of Sieving Tests

	Sample A				Sample B			
	d_c (mm)	σ_g	d_{PT} (mm)	S (mm^2/g)	d_c (mm)	σ_g	d_{PT} (mm)	S (mm^2/g)
P1	2.94	3.0	5.14	3170	2.98	2.8	5.27	3020
P2	2.07	2.6	3.71	4320	2.60	2.9	4.08	3830
P3	1.41	2.3	2.19	5740	1.48	2.3	2.45	5450
P4	0.97	2.1	1.20	8090	0.94	1.9	1.16	7980

Discrepancies reflect variable amounts of particle interaction and errors due to sampling. The large difference between the values for P2 in Table 5.1 show that in some cases results can be erratic. Apart from the fact that tangling does not always occur to the same extent, the presence of one or more abnormally large chunks of cane in the sample can affect the results adversely. To overcome this latter problem a much greater sample size would be required, which would necessitate sieving in parallel and suitably combining the results of two or more determinations.

This applies in particular to the calculation of d_{pT} since its value is very dependent on the amount retained on the large screens. On the other hand, the calculation of S is less affected by the amount retained on the coarse screens, and so appears to be a more attractive method of representing sieving results.

5.1.3 Displaceability Index.

Displaceability Index (DI) is an approximation to the fraction of the juice in bagasse which is present in broken cells (expressed as a percentage). Since the test is carried out at room temperature, juice in unruptured cells is not extracted, since unruptured cell walls are permeable to sucrose molecules only at elevated temperatures (see section 2.5). Equilibrium between extract and juice is only obtained after $1\frac{1}{2}$ hours (Markham, 1969), but after 30 minutes the approach to equilibrium is slow. This indicates that all juice in broken cells is not easily extracted, and that some of the sucrose has to find its way by diffusion through broken cells to the extract. As long as a standardised time is chosen for the duration of the DI test, it should provide a reliable measure of the availability of juice. As such, DI should account for the quality of bagasse on the micro-scale, and so furnish a guide to expected extraction performance.

DI tests were carried out in duplicate, and both results were invariably within 1% of each other. The determination of DI is more accurate and reliable, and less dependent on experimental technique than sieve analysis.

5.1.3.1 Relation between DI and Particle Size

Markham (1969) showed that, with the types of bagasse preparation used in this project, DI is a function of particle size, obtained from sieve analysis, of the following form :

$$DI = m \log d_p + C \qquad (5.5)$$

where the value of the slope, m is negative, and d_p is a mean particle size. This suggests that DI could also be used as a measure of particle size.

The relation between DI and particle size was therefore investigated further. A good linear correlation between DI and d_c could be demonstrated but a significantly improved correlation was obtained in the following form:

$$DI = A\ d_c^{\,n} \qquad (5.6)$$

The form of correlation of equation (5.5) also improved on a simple linear correlation. Fig 5.8 shows this data for the 1970/71 season (series D-H). The straight line in this figure represents the best least squares line, with values of A and n equal to 91.6 and -0.308 respectively. A high correlation coefficient of -0.932, with 57 degrees of freedom (Volk, 1958) indicates a very significant correlation $p << 0.01$.

However, the standard error in DI predicted from equation (5.6) is 5.1%. Further, if each preparation is investigated individually, and a similar correlation attempted, values of the correlation coefficient drop considerably, so that for each type of preparation individually, no significant correlation between DI and particle size can be obtained. Thus a significant correlation is only obtained over a range of particle size much wider than would ever be realised in practice.

It is to be expected that higher values of DI would result from more finely prepared bagasse, since a higher degree of preparation implies a greater degree of cell rupture. However, the value

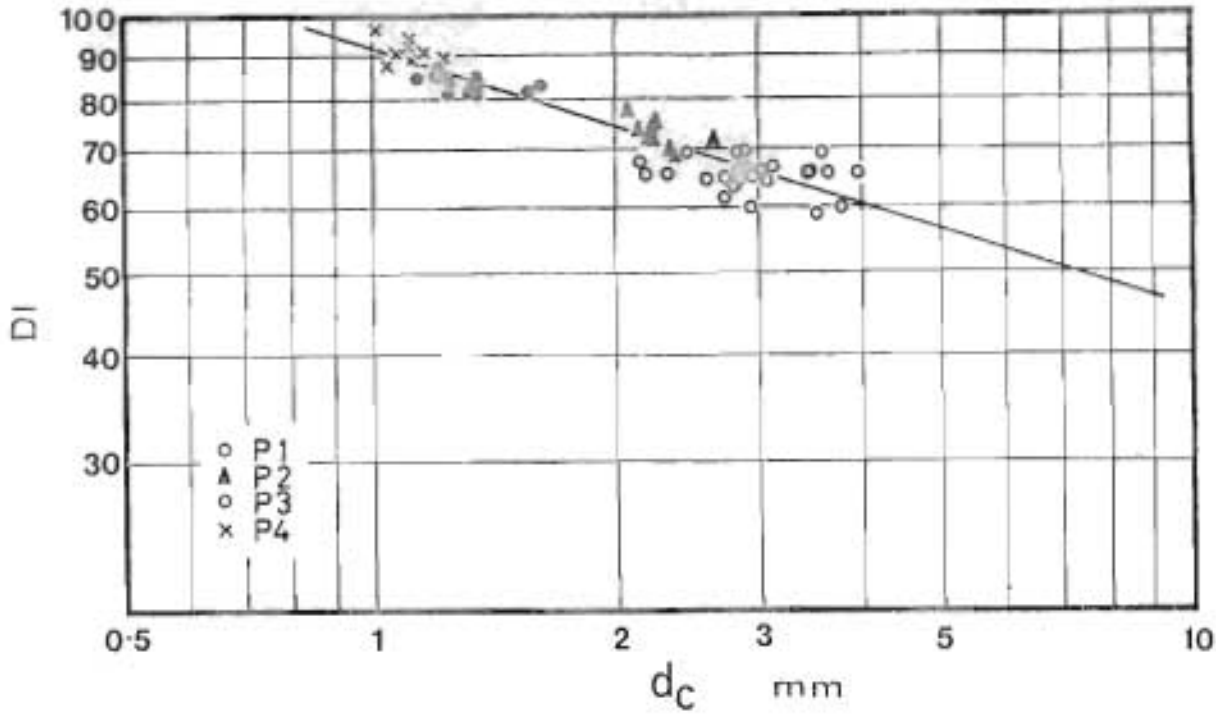


Fig. 5.8. Relation between DI and characteristic particle diameter, d_c. D-H series

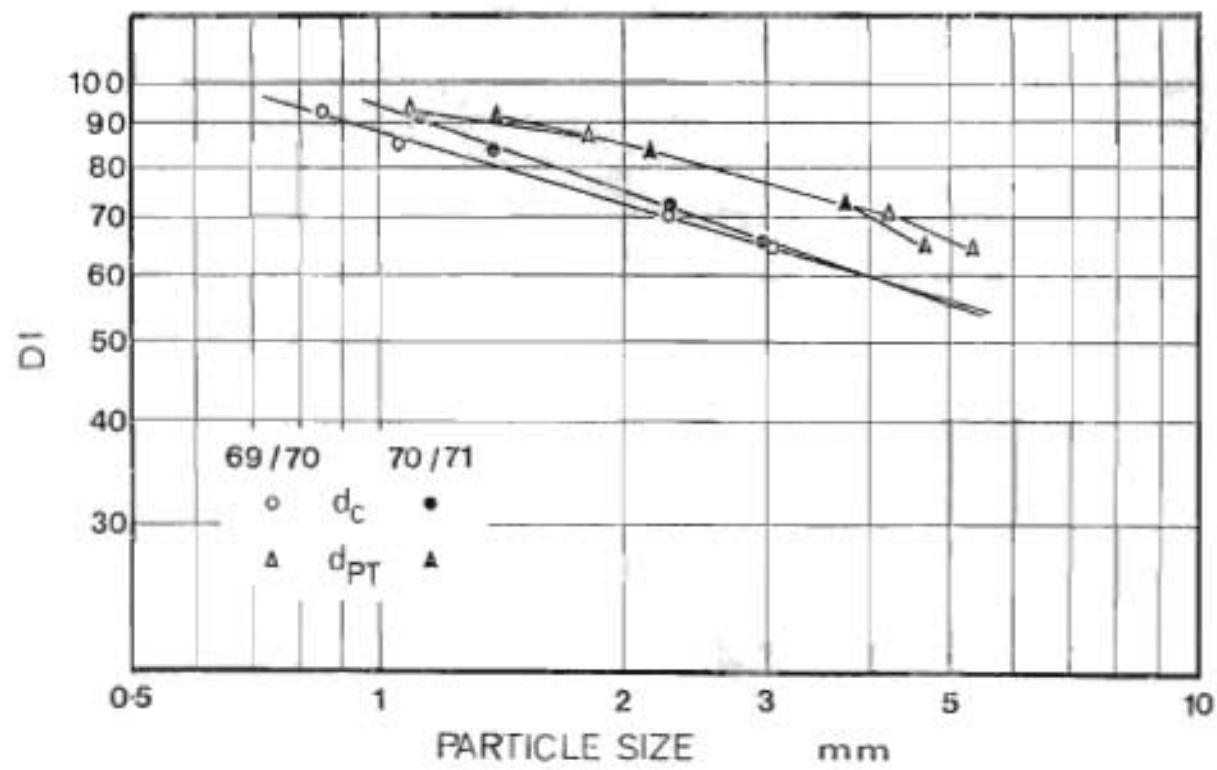


Fig. 5.9. Relation between DI, d_c and d_{PT} for 2 different seasons

of DI is largely influenced by the efficiency of the first mill in separating juice from fibre. Theoretically, two different mills producing bagasse of the same shape and size could yield vastly different values of DI depending on the amount of juice removed in the mill. In this case the mill yielding a higher extraction would result in bagasse of a lower DI. As such it is unrealistic to expect DI to furnish a measure of particle size.

An equally good correlation between DI and specific surface, S, similar to equation (5.6) was obtained, with values of A and n equal to 2.104 and 0.422 respectively. These relations of the form of equation (5.6) have utility only in comparing other types of preparation with those employed in this investigation.

5.1.3.2 Observed differences in preparation between seasons.

Data obtained in the 1969/70 season (C series) could also be correlated according to equation (5.6), but with significantly different values of A and n. This is surprising since reference to section 4.4.1 shows that no significant changes to the cane preparation equipment at Mt. Edgecombe were made between this and the 1970/71 season. There was, however, a slight change in cane quality, as the cane for the latter season experienced drought conditions in the fields, and this cane had a noticeably increased trash content.

Average values of DI, d_c, d_g, d_{pT} and S are given in Table 5.2 for the two seasons; N refers to the number of data points for each type of preparation.

Table 5.2

	N	DI	d_c (mm)	σ_g	d_{PT} (mm)	S (mm^2/g)
1969/70						
P1	12	64.4	3.04	3.0	5.36	3170
P2	4	70.4	2.28	2.8	4.23	3900
P3	10	86.3	1.05	2.0	1.81	7140
P4	3	92.5	0.85	2.0	1.10	9060
1970/71						
P1	27	65.0	2.98	3.1	4.69	3480
P2	10	72.2	2.28	2.9	3.72	4200
P3	14	83.2	1.38	2.3	2.17	6000
P4	8	91.3	1.12	2.2	1.40	7320

The average values of d_c and d_{PT} are shown in Fig. 5.9. The data suggest that some change in the quality of the bagasse has occured, particularly with finer preparations, since no change was made in the methods of preparation.

Fig. 5.9 indicates in addition that DI and d_c obey the functional form of equation (5.6) better than DI and d_{PT}.

5.1.4 Evaluation of particle size and shape

In order to furnish a more complete description of the bagasse employed, direct measurement of individual sizes and aspect ratios of particles of 1st mill bagasse (P1) was undertaken. Such direct particle measurements are ideally applied to particles of approximately uniform shape, of not

too great a size range (Herdan, 1960). Under these conditions, it is usual to measure the projected diameter or Martin's or Ferret's statistical diameters (Heywood, 1937). Heywood (1947) has shown how shape factors of roughly uniform particles can be estimated, to enable calculation of particle surface or volume from the projected diameter.

Bagasse particles are not well suited to this method of particle size determination. The shapes and sizes are highly irregular, with a wide size distribution. In particular, the use of the projected diameter was considered to be worthless, since a long thin fibre could give the same values as a small pith particle.

It was decided instead that measurement of breadth, length, and thickness of the particles would yield more meaningful information. These quantities were measured according to the definitions of Heywood (1937). The breadth is the basic dimension, taken as the minimum distance between two parallel lines tangential to the projected outline of the particle when in its most stable position. The length is the distance between tangents to the projected image perpendicular to the breadth, and the thickness is the distance between two planes tangential to the particle surface in the same plane as the projected image. With these definitions, the breadth is the basic dimension which determines whether the particle will pass though a given screen or not.

The methods employed are described in section 4.4.4.

Fig. 5.10 shows a typical projection of bagasse particles, magnified 10 times. This illustrates the varied and irregular particle shapes. Individual cells are clearly discernible in thin pith particles.

Only preparation P1 was evaluated in this way; it was hoped to apply this procedure to the other types of preparation as well, but it proved to be far too time consuming. In carrying out these measurements, it was confirmed that virtually all the large particles

Fig. 5.10. Typical photographic projection of bagasse particles. Preparation P2, bagasse retained on 28 mesh screen. Magnification 10X.

(all particles on the top screen without exception) originated from the rind or the nodes, i.e. the tougher parts of the cane stalk. In addition, it was observed that a large amount of dry leafy material was retained on the coarse sieves.

In Table 5.3 average values of the breadth, elongation ratio R_{LB}, and the flakiness ratio R_{BT} are given together with the aperture size of the screen on which the particles were retained. The ratios R_{LB} and R_{BT} are defined as :-

$$R_{LB} = \text{length/breadth} \qquad (5.7)$$

$$R_{BT} = \text{breadth/thickness} \qquad (5.8)$$

Particles in the residue (-42 mesh) were not measured, due to the very wide range of particle size in the residue and because the residue comprised a very small proportion of the total sample.

Table 5.3

Fraction	Wt. % Retained	Screen Aperture (mm)	Breadth (mm)	R_{LB}	R_{BT}
+2	26.47	10.6	9.87	10.0	2.1
-2/+4	26.73	5.0	6.19	10.0	2.3
-4/+8	12.64	2.8	3.49	11.2	2.3
-8/+10	8.14	1.65	2.10	4.2	-
-10/+16	9.58	1.00	1.28	4.4	-
-16/+28	9.30	.589	0.78	3.8	-
-28/+42	4.34	.351	0.45	2.9	-
-42	2.79	-	-	-	-

The cumulative weight distribution for this sample is the P1 curve shown in Fig.5.5. It should be remembered that the average values given in Table 5.3 are

number and not weight averages. Although fibres would have little effect on the weight average, their effect on the number average is significant. Values of R_{BT} are the least accurate in Table 5.3, due to the difficulty sometimes experienced in measuring the thickness of very thin particles.

Values of R_{LB} are much greater than those of most materials subjected to sieve analysis. A striking feature of Table 5.3 is the sharp jump in values of R_{LB} on passing from the coarse to fine screens. This indicates that a very substantial portion of the fibre content is retained on the coarse screens

The distribution of values of R_{LB} on the screens is illustrated in Table 5.4.

The fraction of material with a given value of R_{LB} drops initially as R_{LB} decreases, and then shows a slight rise in the range of R_{LB} from 22 to 40. On the three coarse screens, a similar though smaller rise occurs again near $R_{LB} = 50$.

It was found that those particles retained, on any particular screen, with a high value of R_{LB} invariably had a low value of breadth relative to the average on that screen. It is these particles which decrease the sieving efficiency by tangling with other particles and with the screen.

5.1.5. Discussion

It has been shown that bagasse is an unusually complex material, consisting of particles of varying shape, structure, and size. Its outstanding feature is its fibrous nature, which is illustrated by the high values of the elongation ratio of bagasse particles.

It is unfortunate that no means exists whereby bagasse can be completely and conveniently characterised. Due to variations in cane quality, the same method of cane preparation applied to different cane can lead to wide differences in prepared bagasse.

Table 5.4

Percentage of material of each fraction in specified range of R_{LB}

R_{LB}	Fraction						
	+2	-2/+4	-4/+8	-8/+10	-10/+16	-16/+28	-28/+42
1-4	20.4	21.3	25.2	24.5	73.0	84.5	88.4
4-7	24.5	25.1	25.2	13.3	13.9	5.4	7.4
7-10	22.4	19.7	19.0	5.0	2.6	3.6	1.4
10-16	14.1	10.8	16.0	4.0	4.4	1.8	1.4
16-22	4.0	4.5	3.3	1.0	2.6	1.8	-
22-28	12.2	3.5	5.0	-	2.6	0.6	0.5
28-34	2.0	2.0	1.3	1.0	-	0.6	-
34-40	-	2.3	1.0	-	-	1.2	1.0
40-46	-	-	0.3	-	-	-	-
46-52	-	-	0.6	1.0	0.9	-	-
52-58	-	1.6	1.0	-	-	-	-
58-64	-	0.8	-	-	-	-	-
+64	-	0.8	1.9	-	-	-	-

DI is a reliable measurement which should find utility in describing the extractability of bagasse, but it has been shown that it cannot, of itself, account for variations in size and shape which affect the hydrodynamic performance of packed beds.

For this purpose sieve analysis was employed. It has been shown, however, that in some instances unreliable results can be obtained. Furthermore, the results of sieve analysis are very dependent on technique, and great care is necessary in the method and execution of sieving tests to obtain reliable comparative results. Nevertheless, it will be seen later that the sieving results obtained can be usefully employed in the correlation of mass transfer data.

Other methods of characterisation of bagasse were investigated, but no satisfactory alternatives were found. In order to obtain a method of characterisation of bagasse to supplant or even supplement DI and sieve analysis, it is likely that a far more extensive investigation beyond the scope of this project would be required. The eventual attainment of a suitable technique should in the long run amply reward the effort involved.

5.2 LABORATORY-SCALE EXTRACTION TESTS

An investigation into the extraction of sugar from bagasse agitated in water was carried out, with the following objects in mind :-

i. It was envisaged that useful information on the mechanism of extraction could be obtained. Mass transfer in packed beds requires that two effects be delineated, namely, the basic rates of extraction, and the effect of liquid flow conditions in the packed bed on these rates. (Semmelbauer, 1967). On the other hand, this simple system should be independent of such system - induced effects, which would thus permit the evaluation of basic rate data from the results.

ii. In view of the uncertainty associated with the characterisation of bagasse, it was envisaged that experiments of this kind would constitute a laboratory testing facility, which could be used to characterise bagasse prior to processing in an operating plant, in terms of the plant model. In order to do this, clearly the models applied to each situation should have a common physical basis. These experiments provide a test of the validity of the physical concepts employed in the plant model in a simple laboratory test rig.

iii. The model parameters obtained from the laboratory scale tests should have utility in furnishing a frame of reference, to which pilot plant parameter values can be related.

These experiments covered a range of temperatures and degrees of preparation of bagasse similar to that employed in the pilot plant investigations. Experimental details are given in section 4.3.

5.2.1 Degree of Extraction

Typical curves of the brix of the extract juice as a function of time are shown in Fig. 5.11. The effect of temperature is well illustrated. The three runs illustrated in this figure were carried out on subsamples of the same bagasse, and thus are directly comparable. Significant differences are shown between results obtained under the same conditions, but with different bagasse. This is presumed to be due to a large extent to variations in cane quality, and is discussed at greater length in section 5.2.4.

As expected, extraction expressed as the percentage of extraction which would be obtained at equilibrium increases with temperature and degree of cane preparation. The effect of temperature and preparation on extraction after fifty minutes mixing time is shown in Fig. 5.12. This shows preparation to have the greater effect on extraction; equilibrium was in fact obtained with P4 preparation at 75 ^{o}C and 85 ^{o}C.

Temperature has a significant influence on extraction, particularly when more coarsely prepared bagasse is used. However, there appears to be little advantage to be gained from the use of temperatures greater than 75 ^{o}C when finely prepared bagasse is processed.

5.2.2 Application of the Model

The three parameter model applied to the laboratory extraction process is developed in section 3.3.2. The analysis assumes that both W, the weight of extract, and h, the juice holdup in bagasse are constant. W was evaluated as the quantity of water added initially less a small correction for losses due to sampling (see section 4.3.2). The value of h was taken as the amount of juice initially present in the bagasse as determined by analysis, less brix free water.

The sum of squared errors between the model (eqn. 3.22) and the experimental data was minimised using the techniques outlined

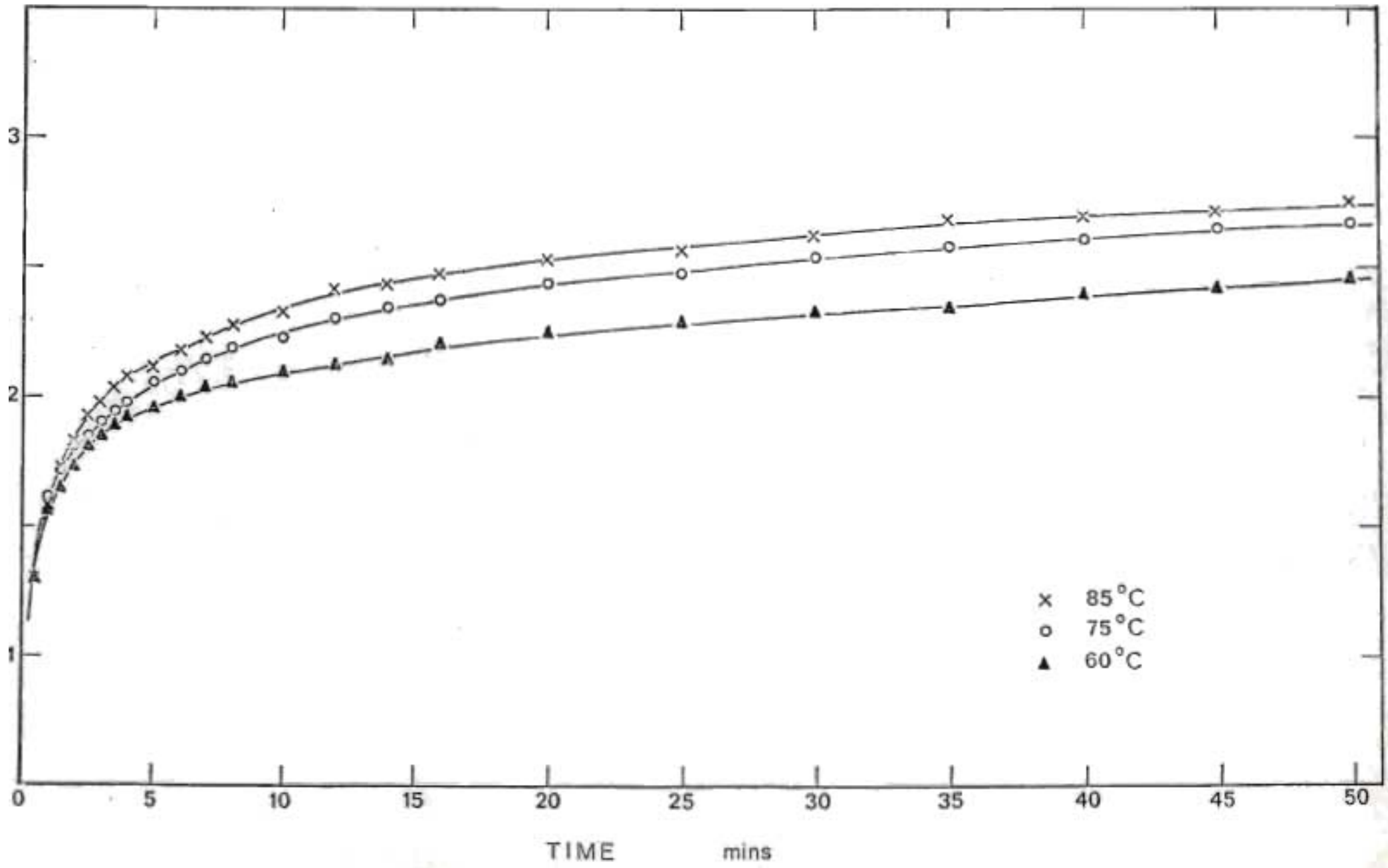


Fig. 5.11. Typical experimental data obtained in laboratory extraction tests.

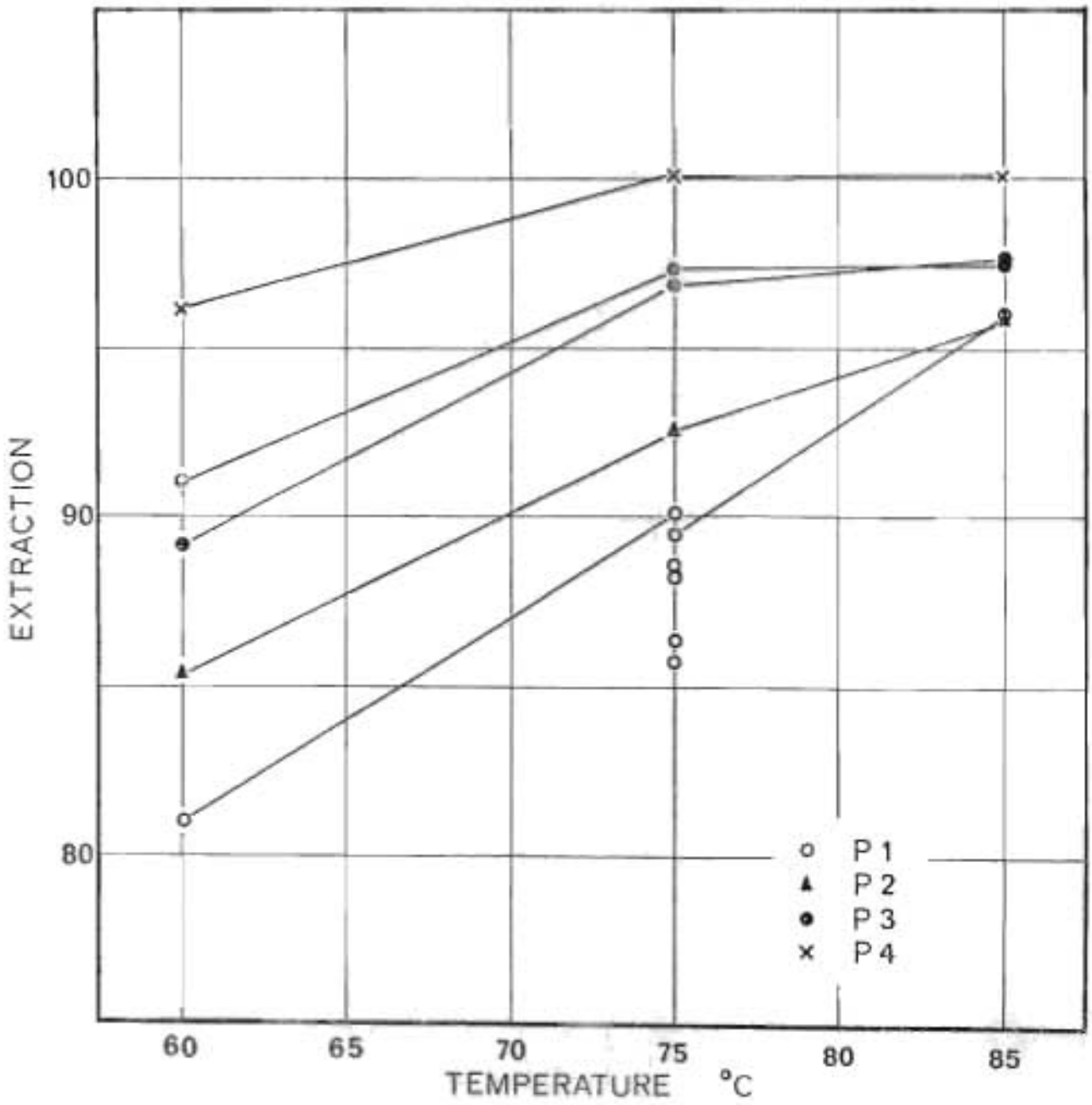


Fig. 5.12. The dependence of extraction on temperature and degree of bagasse preparation. Extraction expressed as the percentage of equilibrium attained after 50 minutes extraction time.

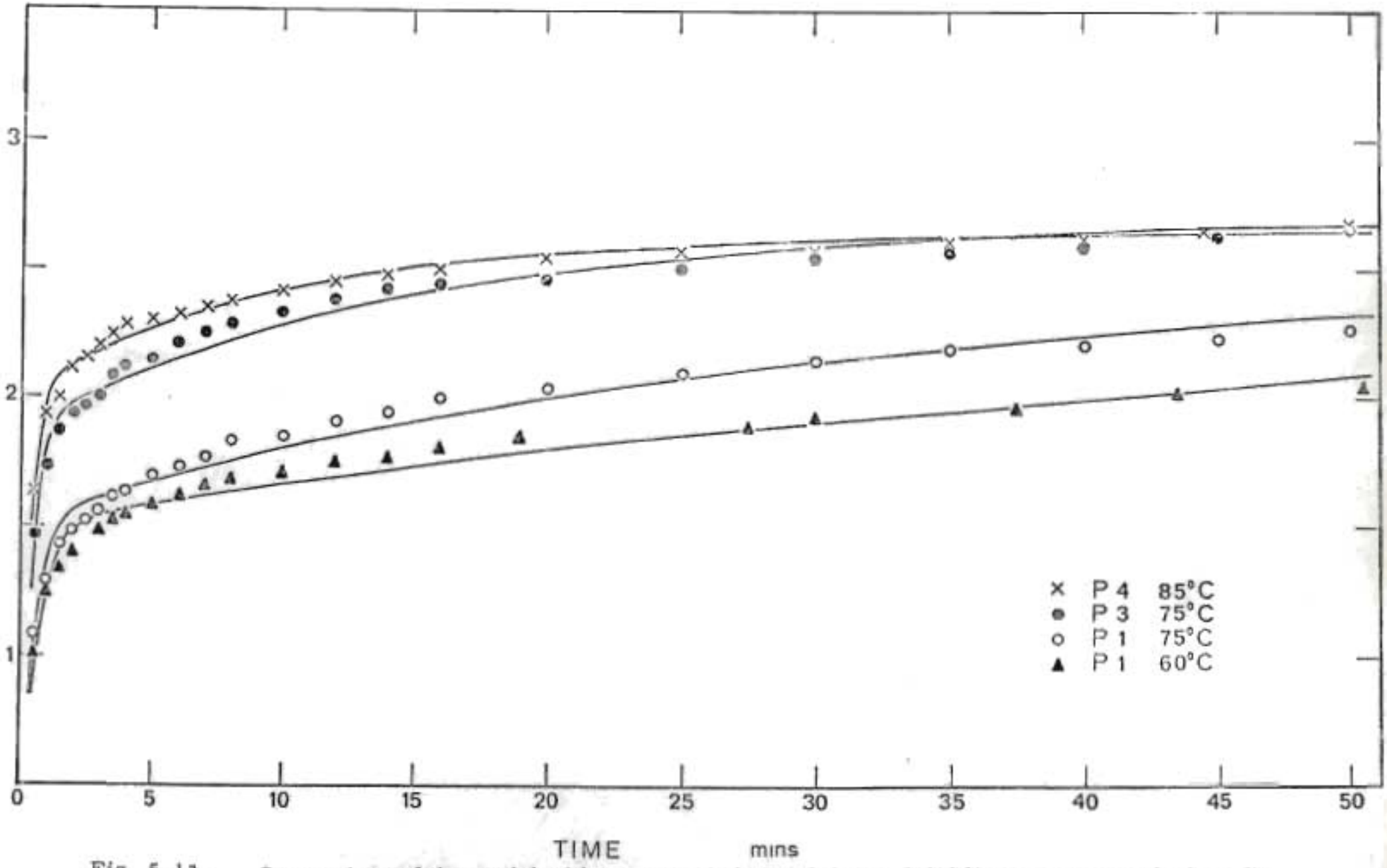


Fig. 5.13. Comparison of the model with some experimental data. Solid lines represent the best fit of the model to the data.

by Powell (1964). This parameter estimation procedure is the same as that applied to the pilot plant data; in order to preserve continuity, details of the procedures employed are referred to in section 5.5.2. where they are discussed with reference to parameter estimation from the pilot plant data.

Fit of the model to the data is satisfactory ; the fit to some of the data is shown in Fig. 5.13. It appears that the model generally predicts a faster initial rate of extraction than the data indicate. At the beginning of each run, concentration changes are rapid, and it is possible that during this period, juice sampling is not representative. Sum of squared errors values vary generally between 0.001 and 0.005 per data point, indicating a standard deviation of data points from the model of 0.03 to 0.07. This compares well with a resolution of 0.04 on the precision refractometer used in the measurement of brix.

5.2.3 Model Parameter Values.

Values of the model parameters obtained from least squares regression are recorded in Table 5.5, together with other relevant data. Since a much smaller amount of bagasse was required for these tests (2 kg per run) than for the pilot plant runs, the bagasse could be collected from only one consignment passing through the milling train. Thus cane variety is also listed in this table. During a few of the runs, small pieces of bagasse were drawn into the circulating pump, thereby choking it, and preventing sampling of the juice. Parameter estimation from these runs with an insufficient number of experimental points was not attempted, which accounts for the missing parameter values in Table 5.5. In these cases, extraction could still be determined by taking a sample of the juice at the end of the run after fifty minutes mixing time.

The dependence of the mass transfer coefficients K_1' and K_2' (referring to the rates of extraction of easily available and more tightly held juice respectively) on temperature and preparation is illustrated

Table 5.5 Results of Laboratory Extraction Tests.

Run No.	Cane Variety	Temp. (°C)	Preparation.	Fibre % Bagasse	DI	S (mm^2/g)	h (g)	W (g)	Extraction %	k_1 (g/min)	k_2 (g/min)	α	Sum of squared errors/point	K_1' (lb/min. lb fibre)	K_2' (lb/min. lb fibre)
'2	mixed	75	P1	31.8		3460	1206	8650	88.7	1510	14.3	.570	.0036	2.38	.0225
'3	376	75	P1	26.1	64.2	3310	1347	8650	86.4	1093	16.9	.515	.0069	2.09	.0323
'4		75	P1						85.8	1062	14.8	.534	.0061	2.04	.0284
'5	50/211	75	P1	32.9	62.9	3700	1177	8650	88.3	1170	15.3	.554	.0025	1.78	.0232
'8	376	75	P2	29.0	71.1	4060	1275	8650	92.7	1507	19.0	.602	.0036	2.60	.0328
'9		60	P2						85.4	1414	9.7	.606	.0045	2.44	.0168
'10		85	P2						95.8	1450	28.5	.597	.0035	2.50	.0492
'11	310	75	P3	28.6	84.0		1285	8650	97.4	2007	27.0	.659	.0024	3.51	.0472
'12		60	P3						91.1						
'13		85	P3						97.4	1886	28.9	.660	.0026	3.30	.0504
'14	50/211	75	P1	32.9	67.4	3580	1178	8750	90.1	1252	15.9	.569	.0031	1.87	.0241
'15		60	P1						81.0	1105	8.5	.560	.0030	1.68	.0129
'16	53/216	75	P1	29.9	68.8	3940	1254	8750	89.5	1088	17.6	.547	.0033	1.82	.0295
'17		85	P1						96.0	1136	24.1	.577	.0017	1.90	.0404
'18	50/211	75	P4	30.9	90.2	7750	1227	8750	100.	2184	25.2	.752	.0003	3.53	.0407
'19		60	P4						96.2						
'20		85	P4						101.	2370	30.9	.730	.0011	3.83	.0499
'21	55/805	75	P3	28.2	82.3	6040	1294	8750	96.9	1660	20.1	.709	.0024	2.94	.0356
'22		60	P3						89.2	1688	9.2	.676	.0031	2.99	.0163
'23		85	P3						99.6	2248	28.5	.678	.0035	3.98	.0504

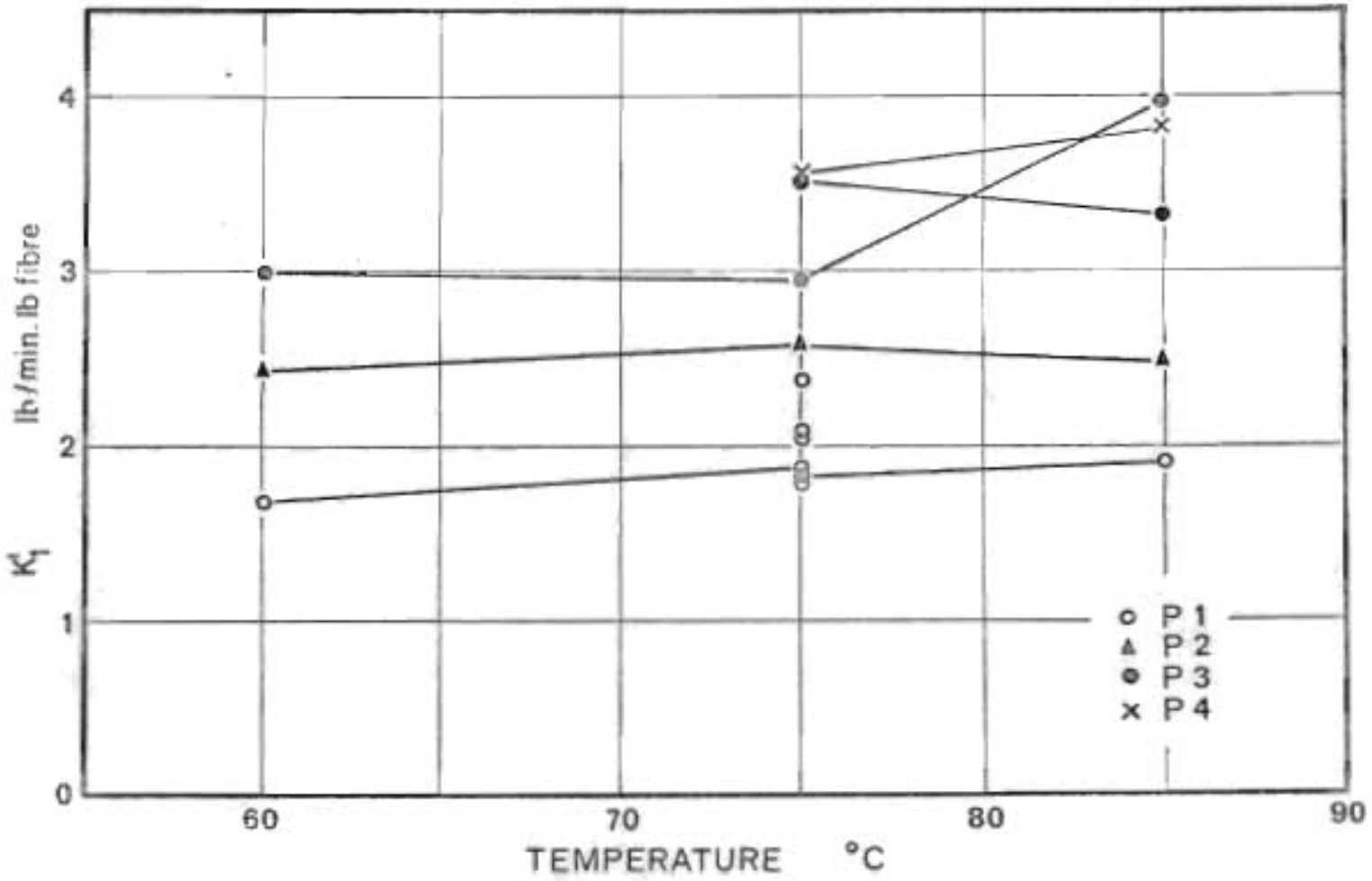


Fig. 5.14. Model parameter K_1' as a function of temperature.

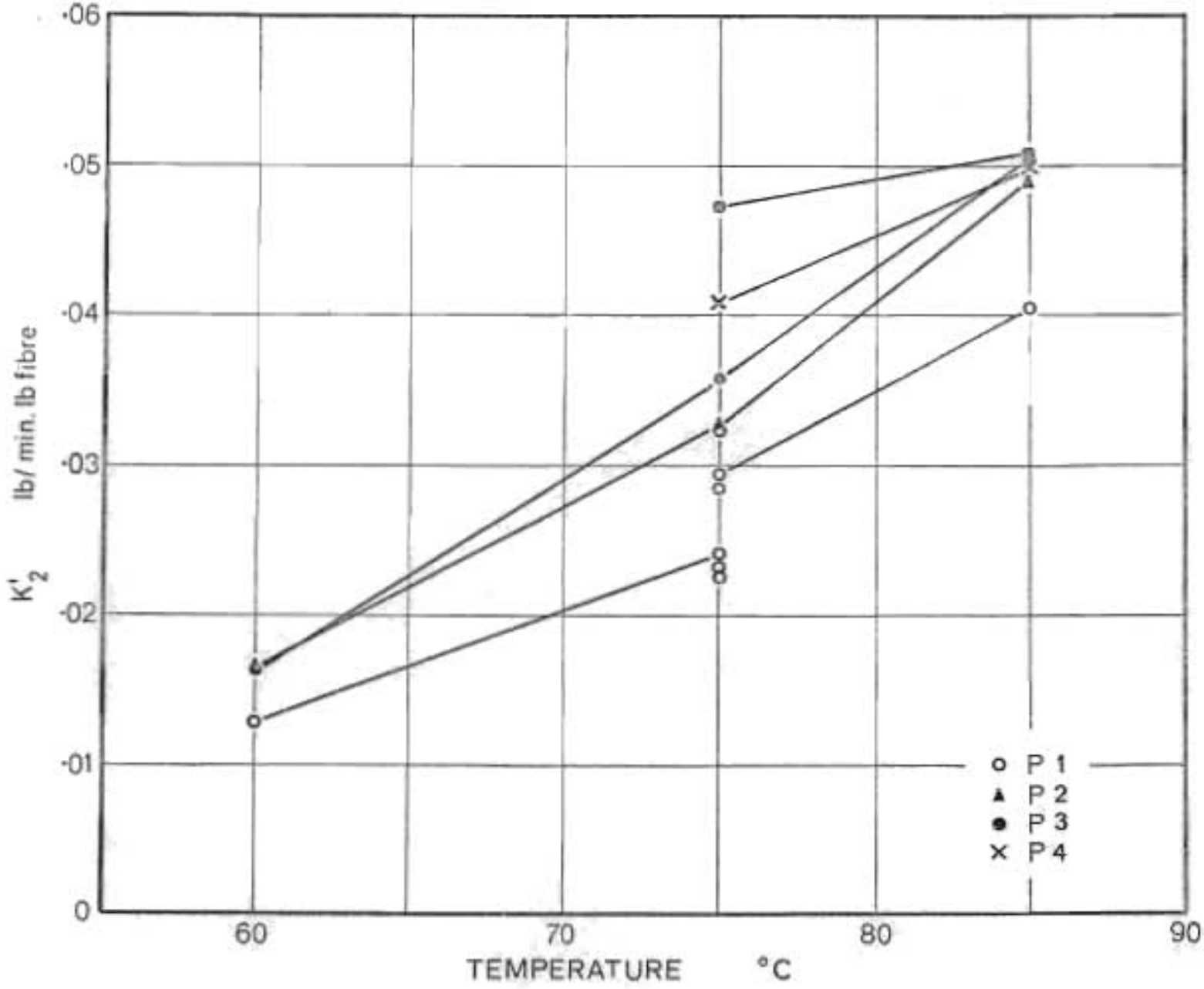


Fig. 5.15. Model parameter K_2' as a function of temperature

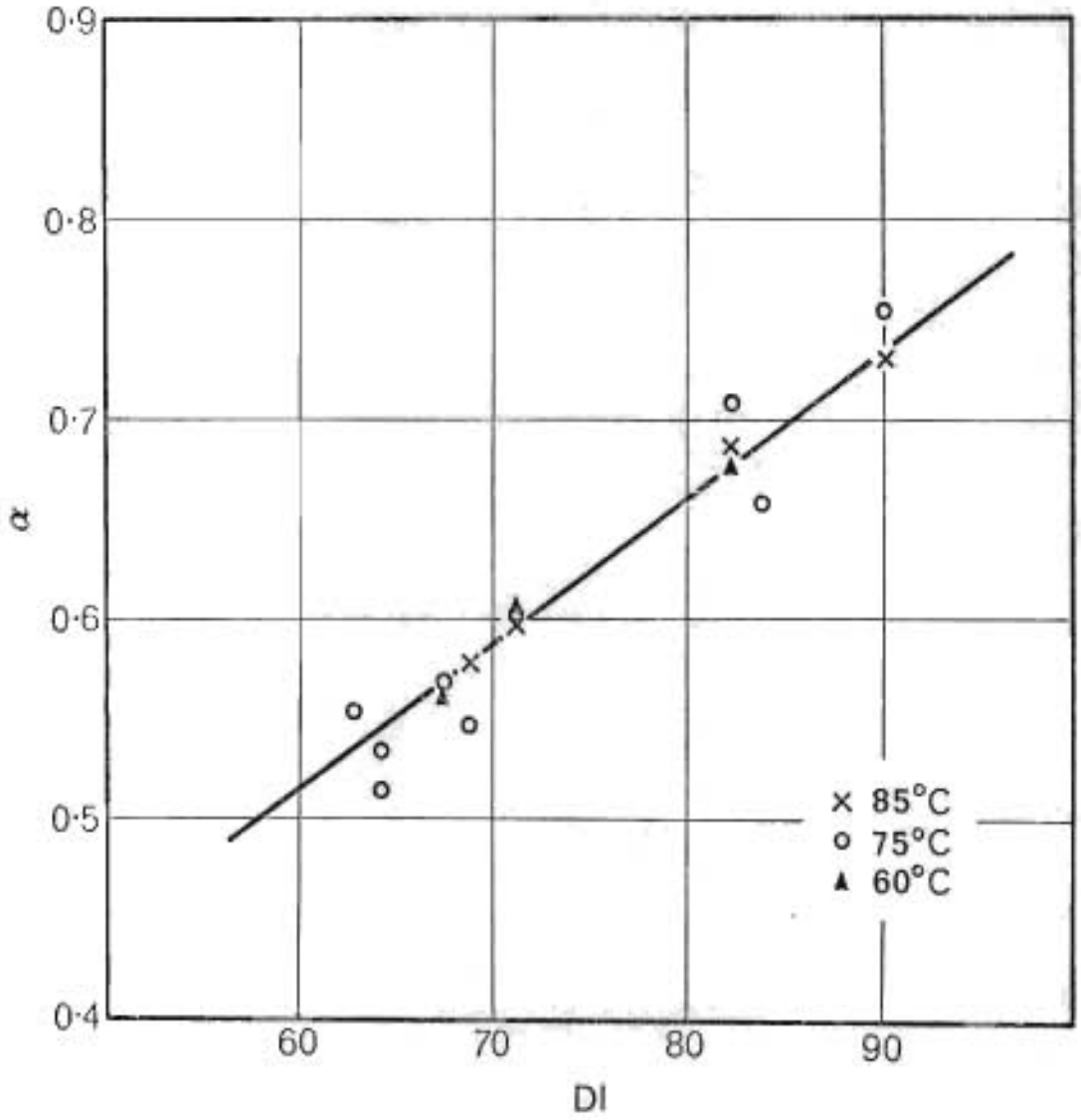


Fig. 5.16. Dependence of α on DI

in Figs. 5.14. and 5.15. The mass transfer coefficients have been normalised with respect to the weight of fibre. It can be seen that K_1' is of the order of eighty times larger than K_2' at $75^{\circ}C$, and that higher extraction rates are associated with more finely prepared bagasse. Values of K_2' vary significantly with temperature, while values of K_1' are practically independent of temperature. Solid lines in these figures connect values obtained from runs using subsamples of the same bagasse.

Values of α shown in Fig. 5.16 appear to be independent of temperature, but a marked dependence on displaceability index (DI) is demonstrated.

The value of the juice holdup in bagasse, h, was taken as the amount of juice initially present in bagasse as determined by analysis, after subtracting brix free water (25% on dry fibre). It may be argued that bagasse absorbs more water, in which case h would increase with a corresponding decrease in W. The effect of larger values of h on the model parameters was therefore investigated. It was found that :-

i. A worse fit of the model to the data was obtained, particularly over the initial period

ii. Values of k_1 and α were affected very little

iii. Values of k_2 were sensitive to the value of h, resulting in lower k_2 values; nevertheless k_2 displayed the same temperature dependence, irrespective of the value assigned to h.

It will be shown later that only values of k_1 and α can be usefully compared with pilot plant data, and that absolute values of k_2 have little significance for comparison purposes. Consequently, there would seem no point in making arbitary corrections to h here.

5.2.4. Variations due to Cane Type & Quality

It is apparent from Table 5.5 and Figs 5.12 and 5.14 to 5.16 that runs carried out under the same conditions using bagasse prepared in the same way result in values of

extraction and model parameters which differ significantly. This can be attributed either to deficiencies in experimental technique or to variations in the quality of bagasse.

Two runs, viz. F3 and F4, were carriedout using subsamples of the same P1 bagasse at a temperature of 75°C. These can be compared with four other runs carried out under the same conditions, but using different bagasse. Insufficient duplicate determinations were made to justify a statistical analysis, but the results show that the differences displayed between runs F3 and F4 are considerably less than the total variations under these conditions. This is particularly true of values of K_1' and extraction, which show a variation of the order of 10% of the total variation.

Thus it would seem that the major part of these variations can be reasonably ascribed to variations in cane type and quality. Similar observations have been made by Brüniche-Olsen (1962, 1969) in connection with extraction of sucrose from sugar beet, and from bagasse.

5.2.5 Interpretation of Results.

The implications of the model are that the mass transfer coefficient k_1 refers to washing of readily available sucrose from broken cells and the particle surfaces, k_2 refers to a slower extraction process from unbroken cells and interior parts of bagasse particles, and that a is the fraction of juice which is extracted by the easy washing process. The results will be considered in the light of this interpretation.

Firstly, the mass transfer coefficient k_1 can be thought of as the product of a rate coefficient and an area through which mass transfer occurs. It is apparent from Fig. 5.14 that higher values of K_1' are associated with finer preparations, which have a higher particle surface area. Values of K_1' were divided by the values of S, the surface area/unit weight of fibre, obtained from sieving tests; these values are given in Table 5.6, and plotted in Fig. 5.17. The change to units of ft. and lb. is made to facilitate comparison with pilot plant results. Comparison with Fig. 5.14 shows that although the scatter is not significantly reduced by the introduction of S, the

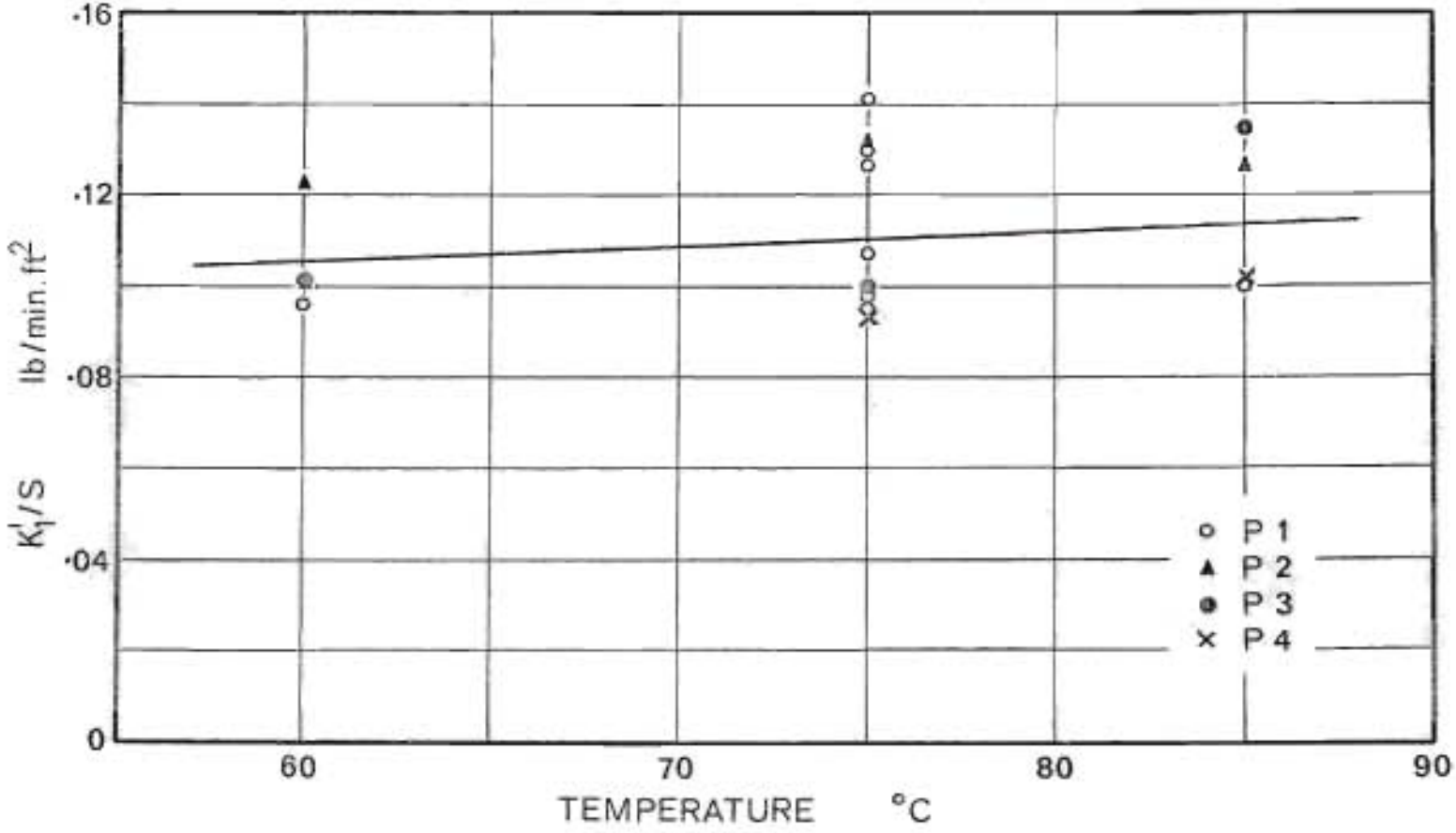


Fig. 5.17. Values of K_1'/S plotted as a function of temperature

trend with preparation is no longer evident. It is reasonable to assume complete wetting of bagasse particles in this system; thus it appears that S provides a good estimate of the mass transfer area, albeit a relative estimate (see section 5.1.2.1).

Table 5.6

Run No.	Prepara-tion.	$\frac{K'_1}{S}$ (lb/min ft^2)	$\frac{K'_2}{S(1-a)}$ (lb/min ft^2)
F2	P1	0.141	0.00345
F3	P1	0.129	0.00413
F4	P1	0.126	0.00375
F5	P1	0.099	0.00288
F8	P2	0.131	0.00416
F9	P2	0.123	0.00215
F10	P2	0.126	0.00614
F14	P1	0.107	0.00320
F15	P1	0.096	0.00168
F16	P1	0.095	0.00339
F17	P1	0.099	0.00498
F18	P4	0.093	0.00434
F20	P4	0.101	0.00488
F21	P3	0.100	0.00416
F22	P3	0.101	0.00171
F23	P3	0.135	0.00529

A washing-displacement type of process will depend on the flow patterns around the particles and the extent of eddy current mixing occurring. The mixing action in the laboratory tests was considered too vigorous to support a liquid phase resistance which might be dependent on molecular diffusion in the liquid. Thus this extraction process should be insensitive to temperature variations, and dependent only on the degree of eddy current mixing (lower viscosity at higher temperatures may conceivably promote the process slightly). As the speed of rotation of the paddle was constant for these tests, the degree of mixing should be very nearly constant. Values of K'_1 shown in Fig. 5.14 are sensibly independent of temperature, and although Fig. 5.17 suggests a slight increase in K'_1 with temperature, such a relation was found to be statistically non-significant. These facts provide evidence in favour of a displacement - washing process

Extraction from interior parts of bagasse particles occurs through cell walls and along small complex-shaped capillary passages within the particles. Mass transfer under these conditions must occur according to a diffusional type process. The mass transfer coefficient referring to this process is a combination of a number of factors; higher mass transfer rates will be obtained when the mass transfer area increases, the diffusion path length decreases and the molecular diffusion coefficient is higher. Thus if one particle is considered, the mass coefficient may be written as:

$$k \propto \frac{A\ D_m}{f(l)} \qquad (5.9)$$

where A is the area, D_m the molecular diffusion coefficient and $f(l)$ is some function of the minimum dimension of the particle, l. For a collection of particles, the same relation can be assumed to hold if some average values of A and l are used.

Reference to Fig. 5.15 shows that K'_2 is larger for finer preparations, where A is increased and l decreases; and also that higher temperatures favour higher mass transfer rates. As values of D_m for the system sucrose-water increase with temperature, these facts are consistent with equation (5.9).

In considering 'structure sensitive' diffusion of juice through bagasse, D_m in equation (5.9) should be replaced by an effective diffusivity (Treybal, 1955). This is necessary for 2 reasons; firstly, the path taken by diffusing sucrose molecules through capillary passages within the particles is tortuous as a consequence of the structure of bagasse, and secondly an additional resistance is imposed on some of the sucrose molecules by the necessity to diffuse through unbroken cell walls. Brüniche-Olsen (1962) considered the effective diffusivity to be the product of D_m and a factor q', which he termed the coefficient of inhibition:

$$D_{eff} = q'\ D_m \qquad (5.10)$$

Assuming now that K_2 follows equation (5.9) and substituting D_{eff} for D_m in this equation, leads to:

$$K'_2 \propto \frac{A\ q'\ D_m}{f(l)} \qquad (5.11)$$

Now consider two different temperatures, T_1 and T_2. The ratio of mass transfer coefficients at these two temperatures is :

$$\frac{K_2'(T_1)}{K_2'(T_2)} = \frac{A_1 q_1' D_{m1} f(l_2)}{A_2 q_2' D_{m2} f(l_1)} \qquad (5.12)$$

Where subscripts 1 and 2 on the right hand side refer to temperatures T_1 and T_2. If tests are done on the same bagasse sample at two different temperatures, A_1 and A_2 are equal, as are l_1 and l_2. Further, Brüniche Olsen (1962) has shown in experiments on sugar beet slices that q' is independent of temperature, and this can be assumed to hold for bagasse as well. Thus $q_1' = q_2'$ and the relation (5.12) becomes :

$$\frac{K_2'(T_1)}{K_2'(T_2)} = \frac{D_{m1}}{D_{m2}} \qquad (5.13)$$

In Table 5.7, values of $K_2'(T_1) / K_2'(T_2)$ are given for those runs where subsamples of the same bagasse were used at two different temperatures. These are compared with values of D_{m1} / D_{m2} calculated from the following relations for the temperature dependence of D_m in sucrose, as given by Brüniche-Olsen (1962) :

$$D_{m1} / D_{m2} = (1.025)^{(T_1 - T_2)} \qquad (5.14)$$

and as given by International Critical Tables (1929) based on the work of Oholm :

$$D_{mT} = D_{m20} \{ 1 + .029 (T-20) + 0.005(T-20)^2 \} cm^2/sec. \qquad (5.15)$$

where T is the temperature in ^{o}C.

Table 5.7 Variation of K_2' with Temperature.

Preparation.	T_1 (°C)	T_2 (°C)	$K'_2(T_1) / K'_2(T_2)$	D_{m1} / D_{m2} Eqn. 5.14	D_{m1} / D_{m2} Eqn. 5.15
P1	75	60	1.87		
P2	75	60	1.96	1.45	1.75
P3	75	60	2.19		
		mean	2.00		
P1	85	75	1.37		
P2	85	75	1.50	1.28	1.36
P3	85	75	1.07		
P3	85	75	1.42		
P4	85	75	1.22		
		mean	1.32		

In spite of scatter in the results, it can be seen that over the range 75°C to 85°C, $K'_2(T_1) / K'_2(T_2)$ approximates closely to D_{m1} / D_{m2}. A Student's t test (Volk, 1958) was employed to show that $K'_2(T_1) / K'_2(T_2)$ is very significantly different from 1.0 but that there is no justification for rejecting the hypothesis that $K'_2(T_1) / K'_2(T_2) = D_{m1} / D_{m2}$. This constitutes strong proof for the postulate of a diffusional mechanism.

Over the range 60° to 75°C, $K'_2(T_1) / K'_2(T_2)$ is consistently higher than values of D_{m1} / D_{m2}. The work of Brüniche-Olsen (1962) shows that at 60°C a significant proportion of the cell walls must still be essentially impermeable to sucrose molecules, because a much longer time is required to render the cells permeable. Then the assumption that $q'_1 = q'_2$, leading to equation (5.13), is no longer valid, since the value of q' at 60°C (ie. q'_2) must be significantly lower. Then the value of $K'_2(75°) / K'_2(60°)$ more correctly represents the quantity $q'_1 D_{m1} / q'_2 D_{m2}$. Since q'_2 is lower, higher values of $K'_2(75°) / K'_2(60°)$ are to be expected.

The quantity $A / f(l)$ in equation (5.11) is not related to the specific surface or particle size of bagasse particles in a simple way. For instance, very small particles, which have a high specific surface, may not contain any 'tightly held' juice, to which this extraction process refers. Therefore an empirical means of correlation was resorted to. It was found that values of $K'_2 / S(1-\alpha)$, given in Table 5.6 showed

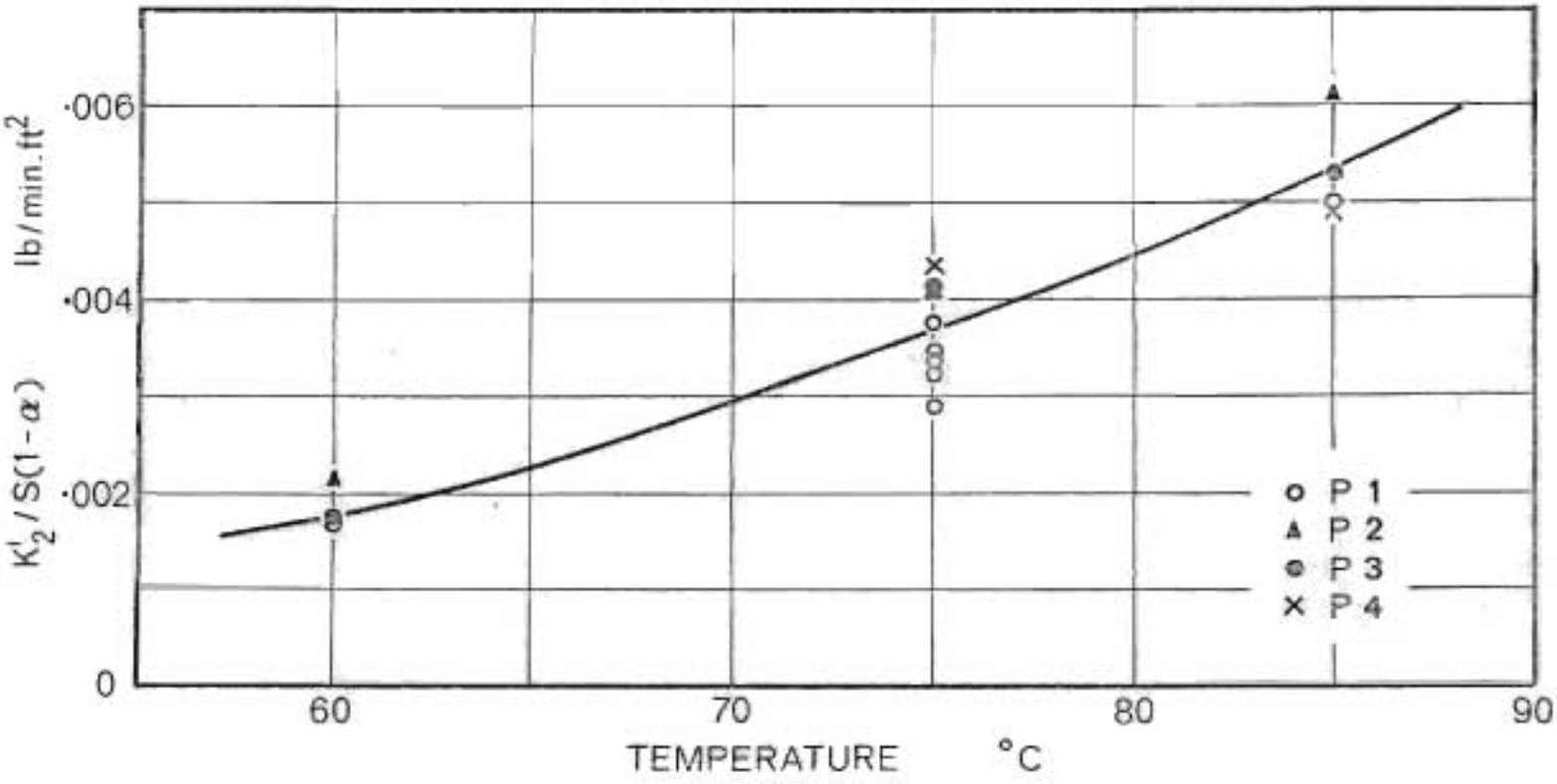


Fig. 5.18. Correlation of K_2' values in terms of $K_2'/S(1-\alpha)$

A correlation of the following form :

$$\frac{K_2}{S\ (1-\alpha)} = 6.83 \times 10^{-9}\ T^{3.055} \qquad (5.16)$$

was obtained by linear regression, with a correlation coefficient of 0.95. This relation is shown as the solid line in Fig. 5.18. Although statistically this is a very significant correlation, on the basis of the preceding discussion, a discontinuity should be expected at some temperature between 60° and 75°C where diffusion from unbroken cells begins to make a significant contribution to extraction. Thus equation (5.16) does not represent the correct temperature dependence, but is included in order to emphasize the significant effect of temperature on K_2'.

Values of α obtained do not show a significant dependence on temperature, as illustrated in Fig. 5.16. DI is used as a measure of the availability of juice, and it is logical therefore that a relationship between α and DI exists. It is expected that α should be less than DI (if expressed as a fraction, not a percentage), since the DI test employed is of 30 minutes duration during which time diffusion of juice from broken cells in the interior of bagasse particles may still occur.

Simple linear regression yielded the best least squares straight line correlation as :

$$\alpha_M = 0.723\ \frac{DI}{100} + 0.0795 \qquad (5.17)$$

where the subscript M refers to values of α obtained in these laboratory mixing tests. A correlation coefficient of 0.961 with 18 data points implies a very significant correlation, $P << 0.01$. (Volk, 1958).

It is significant that the correlation between α and DI holds in spite of variations in temperature and cane type and quality.

These results are substantial support for the proposed extraction model, indicating that the model parameters do in fact have the physical significance ascribed to them.

5.3 PILOT PLANT LIQUID HOLDUP RESULTS.

5.3.1 Definition of Holdup Quantities.

It has become common practice to express the total liquid holdup, H_T, as a combination of 2 quantities, the dynamic and static holdups. Thus:

$$H_T = H_D + H_S \qquad (5.18)$$

However, it is apparent from section 2.4 that some confusion exists over what the 2 components of the total holdup represent. The static holdup is generally taken to be equivalent to the 'adhering holdup', ie. the amount of liquid which remains in a packed bed when the bed is drained. With this definition, H_S depends on the liquid retaining capacity of the bed, and is independent of liquid flow rate.

As shown in section 2.4 it is unlikely that the 'static holdup' so defined does in fact represent a holdup of liquid which is static within the bed, and the concept of a static holdup which decreases at higher flow rates was suggested to be more realistic. Since it is expected that static liquid regions will influence mass transfer (sections 2.3 and 2.5), it is obviously preferable to employ this concept, which provides some measure of the extent of these static regions.

In a packed bed in which there is no stagnant liquid, the response to a pulse input is a Gaussian distribution curve, whose variance is a measure of the axial dispersion, provided the bed is not too shallow, or the dispersion too large (Levenspiel, 1962a). Then the product of mean residence time and liquid flow rate yields the total holdup, and the static holdup is zero. If, however, stagnant regions are present in the bed, tailing of the response curve occurs (Hoogendoorn and Lips, 1965). Levich et al (1967) showed that the response to a pulse input if stagnant zones are present, can be represented by the sum of a normal distribution and an exponentially decaying one.

Then the position of the maximum in the impulse response corresponds to a residence time τ given by (Levich et al, 1967):

$$\tau = \phi Z / U \qquad (5.19)$$

where U is the true mean liquid velocity,
where ϕ is the fraction of flowing liquid, and $(1 - \phi)$ is the fraction of liquid which is static. But Z/U is the mean residence time, $\bar{\tau}$; thus

$$\tau = \phi \bar{\tau} \qquad (5.20)$$

Since the product of $\bar{\tau}$ and liquid throughput yields the total liquid holdup, the product of τ and liquid throughput yields the flowing fraction of the total holdup, i. e. the dynamic holdup. This provides a convenient method of measuring the dynamic holdup. The time τ can be considered as a dynamic phase residence time; the closer τ is to $\bar{\tau}$, the lower is the degree of tailing, and the smaller the static holdup.

Thus H_D is defined as the product of τ and the liquid throughput, and H_S is calculated as the difference between H_T and H_D, from equation (5. 18). With these definitions of holdup, it is envisaged that the dynamic and static holdups should have the physical significance which this nomenclature implies.

The juice initially present in the bagasse is included in the value of H_S (and H_T); once irrigation of the bagasse occurs, it is impossible to distinguish between inter- and intra-particle liquid, and both are included in the values of H_S. Thus it is expected that H_S can never have zero values.

This section presents liquid holdup results for the pilot plant diffuser, and proposes correlations for the prediction of liquid holdup. The methods used to measure liquid holdup are given in section 4.2.4 and experimentally determined holdup values and other related experimental parameters are given in Appendix A.

5.3.2 Dynamic Holdup.

Values of H_D are shown in Fig. 5.19 as a function of liquid flow rate. This data was obtained with a percolating liquid temperature of 73° C, and a constant bagasse load of 125 lb. Higher values are evident at higher flow rates, and with finer bagasse preparation. There is a certain amount of overlap between data using different types of preparation. This is not unexpected, since it was pointed out in section 5.1 that the same method of preparation applied to different cane can lead to wide differences in prepared bagasse.

In order to establish the flow rate dependence, the best fit of the data, for each type of preparation, to an equation of the form

$$H_D = A L^n \qquad (5.21)$$

was obtained by simple linear regression (using log transformations to linearize the equation). Values of the constants and correlation coefficients are given in Table 5.8.

TABLE 5.8. Regression Coefficients for Equation (5.21).

Preparation.	A	n	Correlation Coefficient r	Significance Level p
P1	0.990	0.410	0.777	< .001
P2	0.769	0.514	0.933	< .001
P3	1.075	0.492	0.836	< .001
P4	1.428	0.495	0.893	< .01

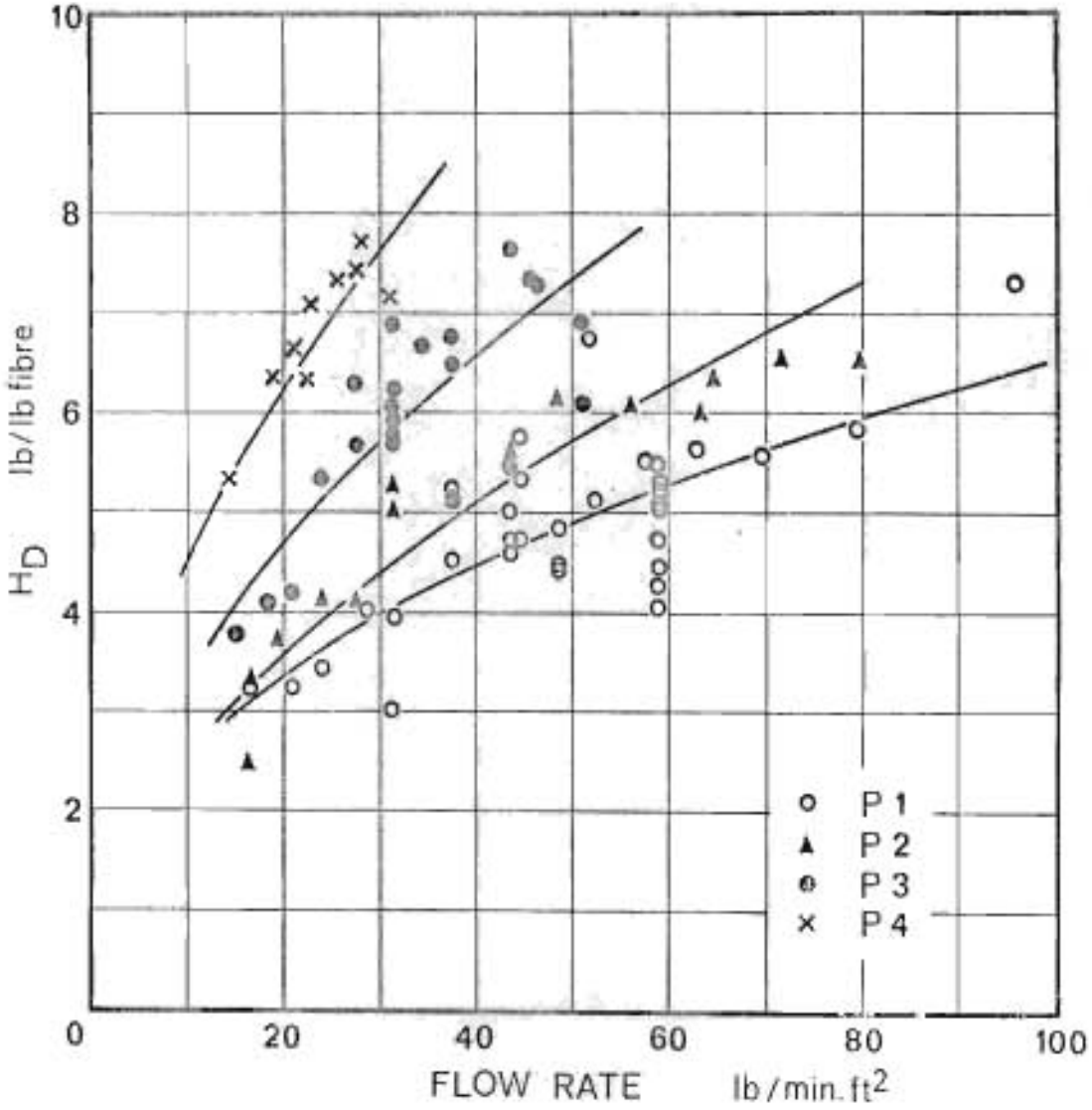


Fig 5.19. Dynamic liquid holdup as a function of liquid flow rate. Data shown represents operation at 73°C with an initial bagasse load of 125 lb.

The significance levels were obtained from tables (Volk, 1958), using the number of degrees of freedom and the values of r. For all preparations, the effect of flow rate is very significant. The equation for each type of preparation is also shown in Fig. 5.19.

5.3.2.1 Flooding.

The occurrence of flooding is treated more fully in section 5.4; it is necessary here to point out that the occurrence of flooding limits the maximum obtainable flow rate. In a full-scale diffuser, the condition of over-flooding, where flooding occurs to an extent where liquid builds up above the top of the bed, is an undesireable operating condition, which has a detrimental effect on performance. Any lower flow rate, which leads to partial or complete flooding, without the build-up of liquid above the surface of the bed, is an acceptable operating condition. Thus, in the pilot plant experiments, any run where over-flooding occurred was rejected because it represents unrealistic operation. Moreover, a pool of liquid on top of the bed would affect the tracer tests.

Data from runs where flooding occurred, but without the formation of a liquid level above the bagasse bed, are included in Fig. 5.19. This data showed no distinguishable differences from data obtained under non-flooding conditions.

5.3.2.2 Effect of Bed Height.

Most runs on the pilot plant were carried out using a bagasse load of 125 lb. in the diffusion vessel, resulting in a bed height of 2.5 - 3.0 ft. A series of 30 runs was carried out with bagasse loads varying between 75 lb. and 175 lb. with a spread in bed heights of 1.6 to 3.8 ft.

Since holdup is affected by flow rate and bagasse preparation, a series of 10 runs was carried out at the same flow rate and temperature, and using the same type of preparation, but with different bagasse loads. The data is shown in Table 5.9.

Table 5. 9. Effect of Bed Height on Liquid Holdup.

Test Conditions: Flow rate 37.6 lb/min ft^2
Temperature 73 °C
Preparation P1.

Run No.	Z (ft)	q (lb fibre/ft^3)	H_D (lb/lb fibre)	H_T (lb/lb fibre)	H_S (lb/lb fibre)
E21	1.98	4.40	5.22	8.98	3.74
E22	2.85	4.57	5.69	8.78	3.10
E23	1.71	4.08	5.07	8.67	3.61
E24	3.48	4.67	5.56	8.66	3.10
E25	2.13	4.40	4.87	8.40	3.54
E26	2.63	4.46	5.16	8.31	3.15
E27	1.75	4.16	4.94	8.78	3.84
E28	3.60	4.71	5.22	8.28	3.06
E29	2.56	4.44	5.22	9.22	3.99
E30	3.00	4.55	5.02	8.24	3.22

Each pair of values represents 2 runs carried out using subsamples of the same bagasse. Within each pair, it appears that generally a higher value of H_D is associated with a greater bed height. However, linear regression analysis yields a correlation coefficient too low to claim any association between H_D and Z.

Closer scrutiny of this data reveals that higher bed heights lead to higher values of the fibre density, q, presumably due to compaction under a greater load. Thus a dependence on bed height may be interpreted as an effect of density of packing, as higher packing densities result in a greater number of particle contact points per unit volume.

5.3.2.3

Effect of Temperature.

Values of H_D at temperatures other than 73°C, if superimposed on Fig. 5.19, suggest that H_D is independent of temperature. Because of the scatter in this data, this cannot be utilized to confirm the temperature independence of H_D. As in the previous section, a series of runs were carried out with constant conditions, but different temperatures.

This data is shown in Table 5.10.

ABLE 5.10 EFFECT OF TEMPERATURE ON LIQUID HOLDUP

Test Conditions: Flow rate 44.6 lb/min ft^2

Preparation P1

Bagasse Load 125 lb.

un No.	Temperature (^oC)	q (lb fibre/ft^3)	H_D (lb/lb fibre)	H_T (lb/lb fibre)	H_S (lb/lb fibre)
2	74	4.53	5.36	9.23	3.87
3	88	4.64	4.81	flooding	
4	89	4.54	5.25	flooding	
5	67	4.47	5.36	9.55	4.19
6	67	4.63	4.71	8.99	4.29
7	81	4.78	4.71	8.49	3.76
8	81	4.57	4.58	8.84	4.26
9	60	4.46	4.36	9.60	5.23
4	74	4.42	4.77	9.61	4.83
5	60	4.32	5.40	10.49	5.08
6	89	4.62	4.90	8.56	3.65
7	60	4.51	4.98	9.45	4.48

This data confirms the absence of any dependence of H_D on temperature.

It is possible to obtain an estimate of the expected change in H_D with temperature from the equations which have been proposed for correlating dynamic holdup. Estimates of the ratios of H_D at temperatures of 60, 73 and 90 °C are given in Table 5.11 from published correlations which were discussed in section 2.4.2.1

Table 5.11. Dependence of H_D on temperature from published correlations.

Reference.	$\frac{H_D(60)}{H_D(73)}$	$\frac{H_D(60)}{H_D(90)}$	$\frac{H_D(73)}{H_D(90)}$
Otake & Okada (1953	1.035	1.066	1.030
Gelbe (1968)	1.030	1.057	1.027
Jesser & Elgin (1943)	1.025	1.047	1.022
Mohunta & Laddha (1968)	1.050	1.098	1.046
Shulman et al (1955b)	1.044	1.088	1.042
Davidson (1959)	1.064	1.126	1.059
MEAN	1.040	1.080	1.038

This table shows that higher values of H_D are expected at lower temperatures. Although a change of only 8% is expected over the range from 60° to 90°C, such a change is not shown by the data in Table 5.10. However, Table 5.10 shows that higher values of the fibre density are obtained at higher temperatures, which on the basis of the results in section 5.3.2.2, would imply slightly higher values of H_D. Thus, at higher temperatures, it appears that the 2 effects of temperature and packing density effectively compensate each other with the result that values of H_D are apparently independent of temperature.

5.3.2.4 Correlation of Dynamic Holdup Data.

Dimensional analysis leads to the following relation for H_D (Mohunta & Laddha, 1965):

$$H_D = f\ (Fr / Re\ ,\ Re) \qquad (5.22)$$

Alternatively, with the same independent variables, it can be shown that:

$$H_D = f\ (\ Re\ ,\ Ga\ ) \qquad (5.23)$$

The form of equation (5.23) has generally found wider acceptance, and can also be derived from theoretical considerations (Davidson, 1959). Consequently, all H_D data was subjected to multilinear regression analysis, assuming a relation of the form of equation (5.23). The multilinear regression procedure used in this and following sections is described in appendix F.

Values of Re and Ga used in this study are designated as Re_1 and Ga_1 respectively, and are defined as:

$$Re_1 = \frac{L}{\mu\ a_T} \qquad (5.24)$$

$$Ga_1 = \frac{g\ \rho^2}{a_T^3\ \mu^2} \qquad (5.25)$$

The value of a_T, the packing surface area/unit volume of packed bed, was calculated as the product of specific surface, S, and fibre density, q, and expressed in ft^2 / ft^3. Since S is a relative measure of specific surface (see section 5.1.2.1), values of a_T, Re_1 and Ga_1 are not directly comparable with values of these quantities for packed beds of regular-shaped packings.

Regression analysis yielded the following equation:

$$H_D = 185.1\ Re_l^{0.415}\ Ga_l^{-0.338} \qquad (5.26)$$

In order to assess whether H_D predicted from this equation is independent of Z and q, these 2 variables were included in the regression set. This showed that q as well as Re_l and Ga_l are significant variables, resulting in the equation:

$$H_D = 990\ Re_l^{0.465}\ Ga_l^{-0.361}\ q^{-1.015} \qquad (5.27)$$

where q is expressed in lb. fibre/ft^3. This procedure does however establish that H_D is independent of bed height.

Equation (5.25) suggests that the dynamic holdup to be used in such a correlation may be better expressed as a volume fraction, since the exponent on q is so close to unity. Thus, using the following definition:

$$h_D = \frac{H_D\ q}{\rho} \qquad (5.28)$$

the following correlation is obtained:

$$h_D = 19.8\ Re_l^{0.478}\ Ga_l^{-0.379} \qquad (5.29)$$

which is independent of both q and Z.

A standard error for each correlation was calculated as :

$$\text{standard error} = \left[\frac{\Sigma\left(\frac{Y - y}{y}\right)^2}{N}\right]^{\frac{1}{2}} \qquad (5.30)$$

where Y and y are predicted and observed values, and N represents the degrees of freedom. The standard errors associated with the 3 correlations are 14.8%, 14.1% and 14.4% respectively.

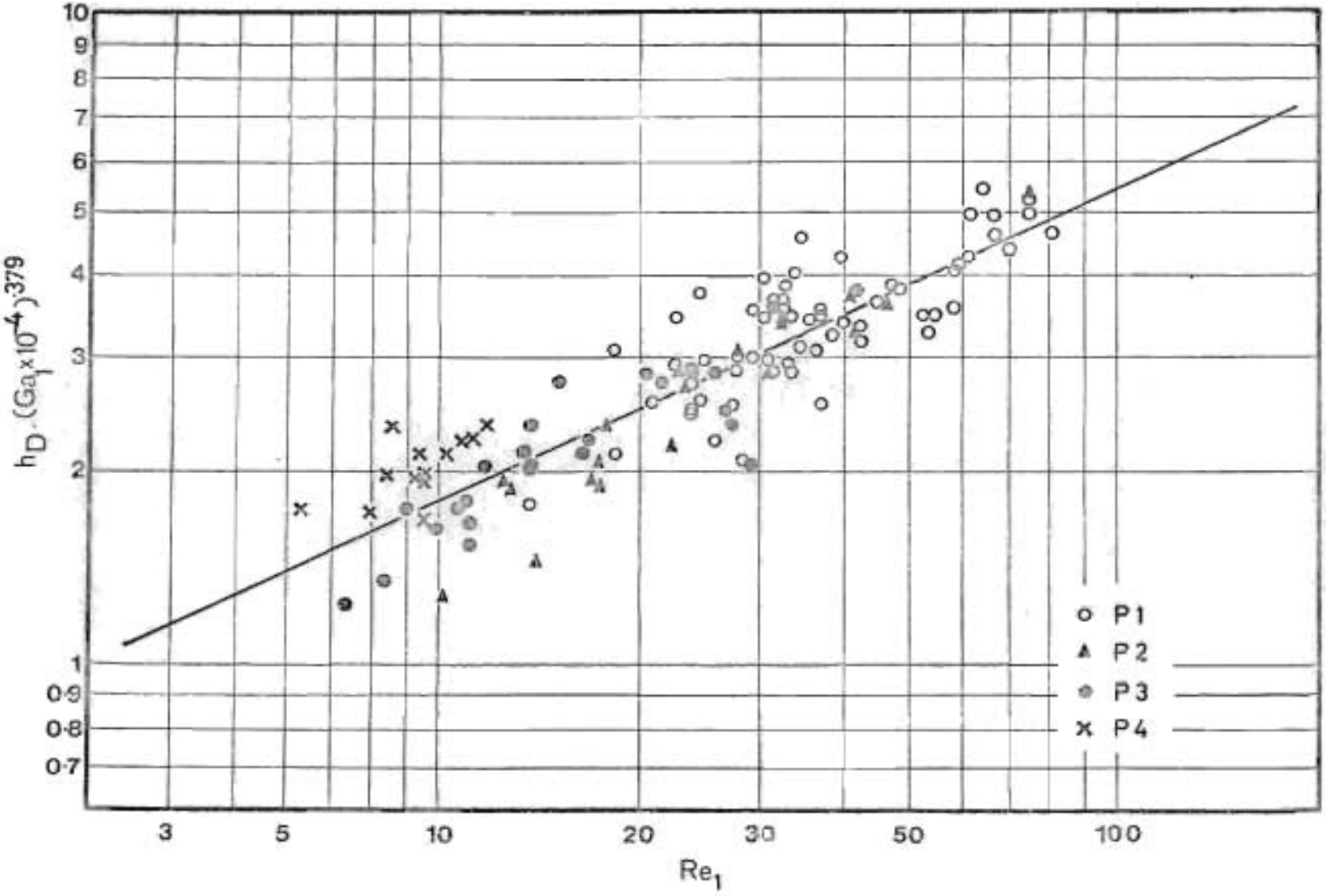


Fig. 5.20. Comparison between dimensionless correlation for dynamic liquid holdup, equation (5.29), and experimental data.

Clearly there is little to choose between these correlations; equation (5.29) is compared with experimental data in Fig. 5.20.

Mohunta and Laddha (1968) investigated various published correlations for H_D, and found the average deviation from the correlations to vary between 14.9% and 35.2%. The present correlations compare favourably.

The exponent on Re lies well within the range reported in the literature, i.e. from 0.33 to 0.75 (see Table 2.3). In particular the close agreement with the value of 0.455 suggested by Gelbe (1968) should be noted, since he alone allowed for the fact that the static-holdup varies with flow rate. Of the correlations proposed in the same form as equation (5.23), values of the exponent on Ga vary between -0.3 and -0.44 (Table 2.3). Again agreement with published values is remarkably good.

In order to investigate more closely the dependence of H_D on the individual experimental parameters, the data was subjected to multiple regression analysis, in the following form:

$$H_D = f(L, S, d_c, T, q, Z) \qquad (5.31)$$

Correlations in terms of significant variables only (5% significance level) were obtained for the data for each type of preparation separately; for all data excluding the varying bed height data; for data at 73°C only; and for all data. This yielded the following results:

1. No significant differences between the correlations for each individual type of preparation were evident.

2. In all cases, S was selected as a more significant variable than d_c, which indicates that S is a more meaningful measure of particle size. (see section 5.1.2.1).

3. Temperature was found to be a non-significant variable in all cases.

4. The correlation for all data at 73°C, including the varying bed height data, is

$$H_D = 0.00686\ L^{0.467}\ S^{0.590} \qquad (5.32)$$

with a standard error of 11.9%. The data is compared with the equation in Fig. 5.21.

5. The best correlation for all data is:

$$H_D = 0.00314\ L^{0.478}\ S^{0.691}\ Z^{-0.120} \qquad (5.33)$$

The standard error in this case is 14.4%. The dependence on bed height is surprising, in view of the facts that (1) Z was found to be non-significant at the 5%, and even the 95% level for the 73°C data, and (2) the dependence on Z is in the opposite sense to that expected from the results of section 5.3.2.2. This is assumed to be due to the observation in section 5.3.2.3 that the same values of H_D are obtained at higher temperatures, but at a greater bed density, i.e. lower bed height. Clearly such an inverse dependence on Z is at variance with the varying bed height data; the form of equation (5.33) is therefore doubtful, particularly as the standard error is considerably higher than for equation (5.32). The use of dimensionless variables is obviously to be preferred, and equation (5.29) is to be recommended ded for prediction purposes.

6. The correlations of equations (5.32) and (5.33) include data where partial flooding occurred (but not over-flooding). The exclusion of this data has a negligible effect on these correlations, with changes in the regression coefficients of less than 1.5%.

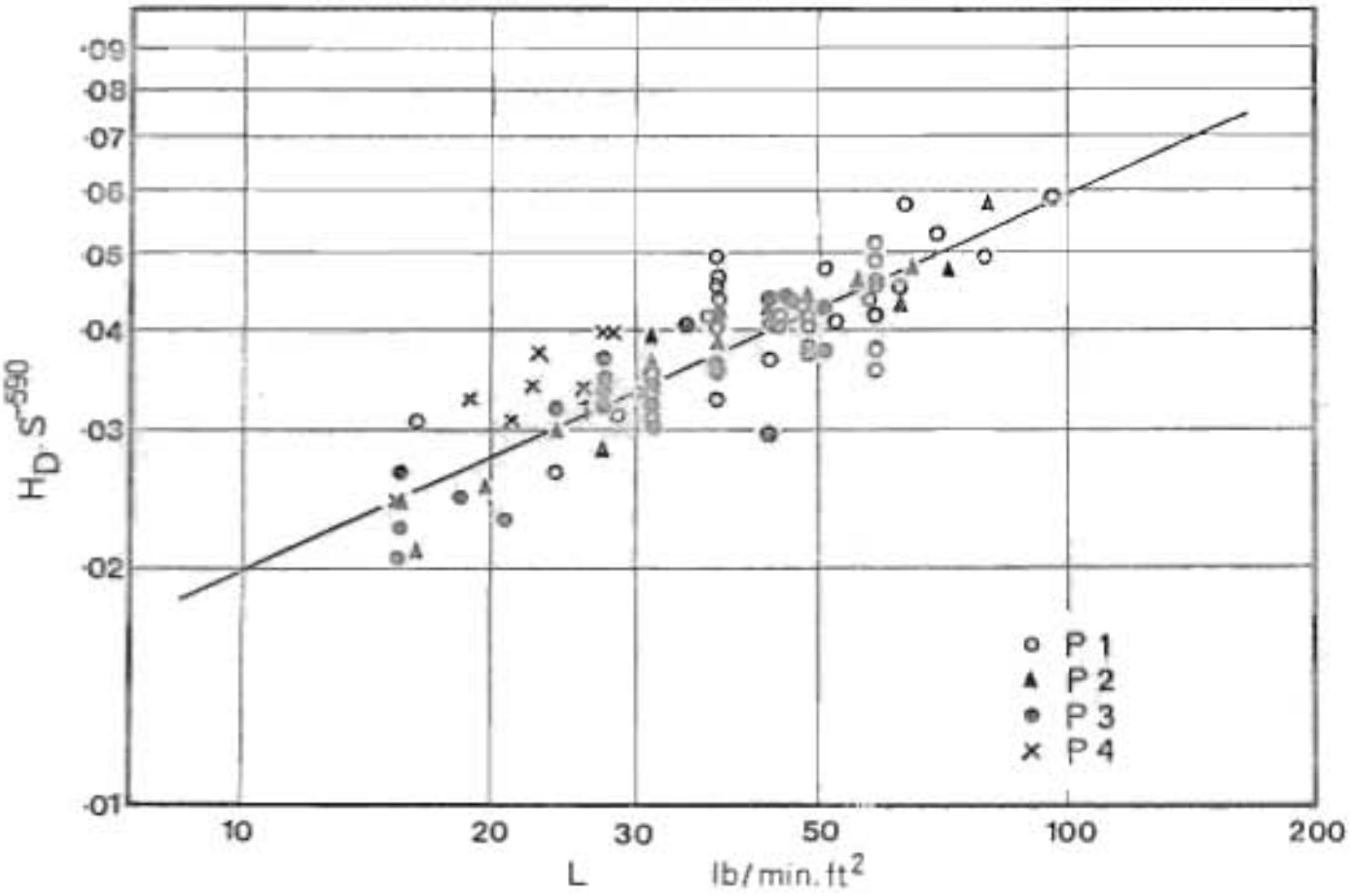


Fig. 5.21. Empirical correlation for dynamic liquid holdup. Comparison with data from runs carried out at 73°C.

5.3.3 Total Holdup.

Higher values of H_T were obtained with finer preparations and higher flow rates, as illustrated in Fig. 5.22. In addition, higher bed heights and temperatures generally resulted in lower values of H_T.

Total holdup is not a primary variable, but was determined in order to obtain values of H_S. Nonetheless, H_T is a directly measured quantity which may find utility for design purposes; as such a correlation in terms of the operating variables is desireable.

The occurrence of flooding (excluding excessive flooding runs, which were rejected) precluded the measurement of H_T. Flooding was generally observed to be progressive, with the liquid level in the bed rising continuously throughout a run (but sometimes stabilizing before the end of the run). This was associated with a continuously increasing value of H_T, and so the measurement of a steady state value of H_T was precluded. Since H_S is the difference between H_T and H_D, this implies in addition that values of H_S are also not determined under these conditions.

5.3.3.1 Correlation of Total Holdup Data.

Reported measurements of H_T in the literature are scarce; the only reported investigation (Shulman et al 1955) gives the relation:

$$H_T = L^n \qquad (5.34)$$

where n is a function of packing size. Thus an empirical means of correlation was resorted to, resulting in the equation:

$$H_T = 2.588\ L^{0.186}\ S^{0.217}\ q^{-0.485}\ T^{-0.128} \qquad (5.35)$$

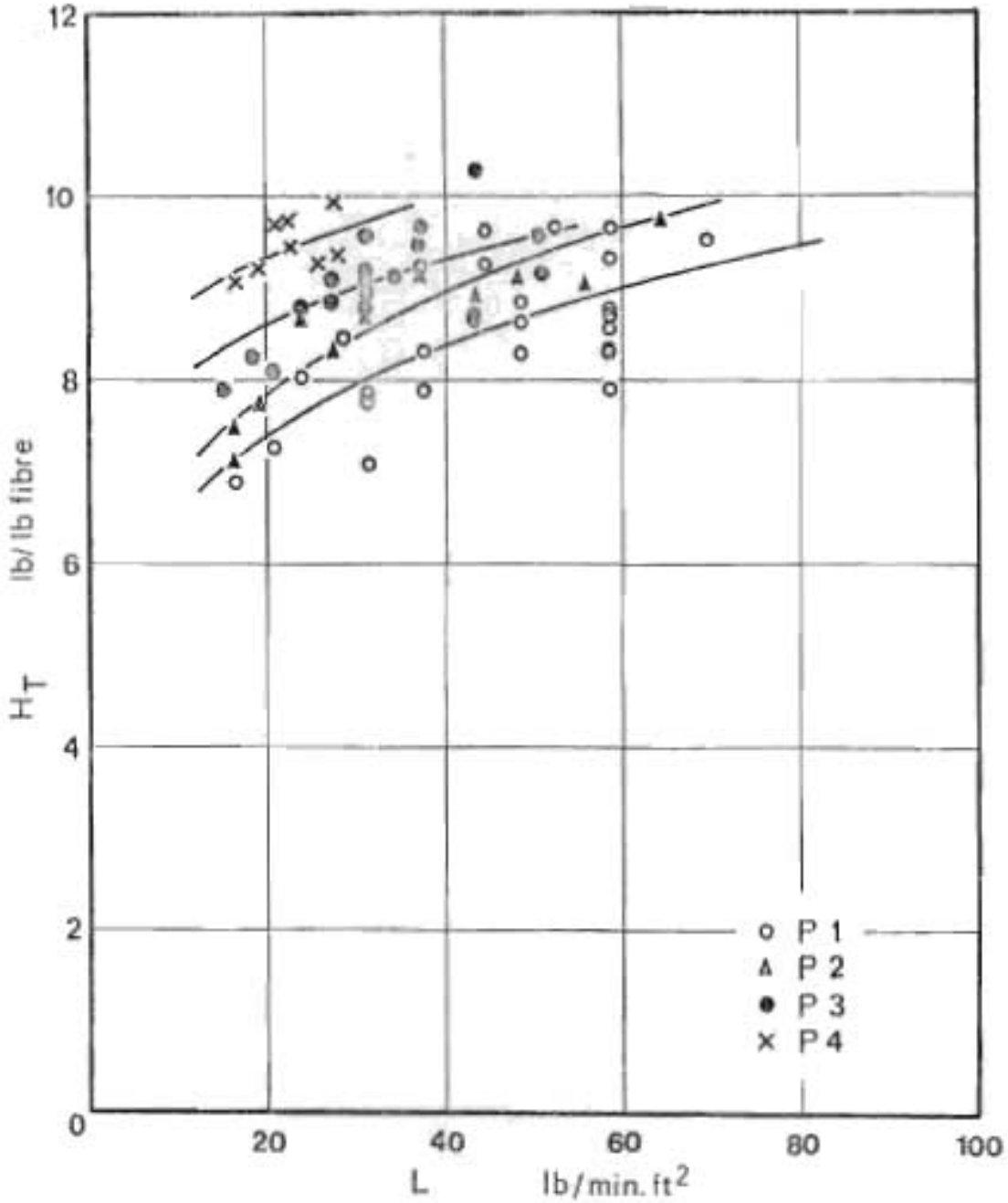


Fig. 5.22. Total liquid holdup as a function of liquid flow rate. Data shown represent operation at 73°C with an initial bagasse load of 125 lb.

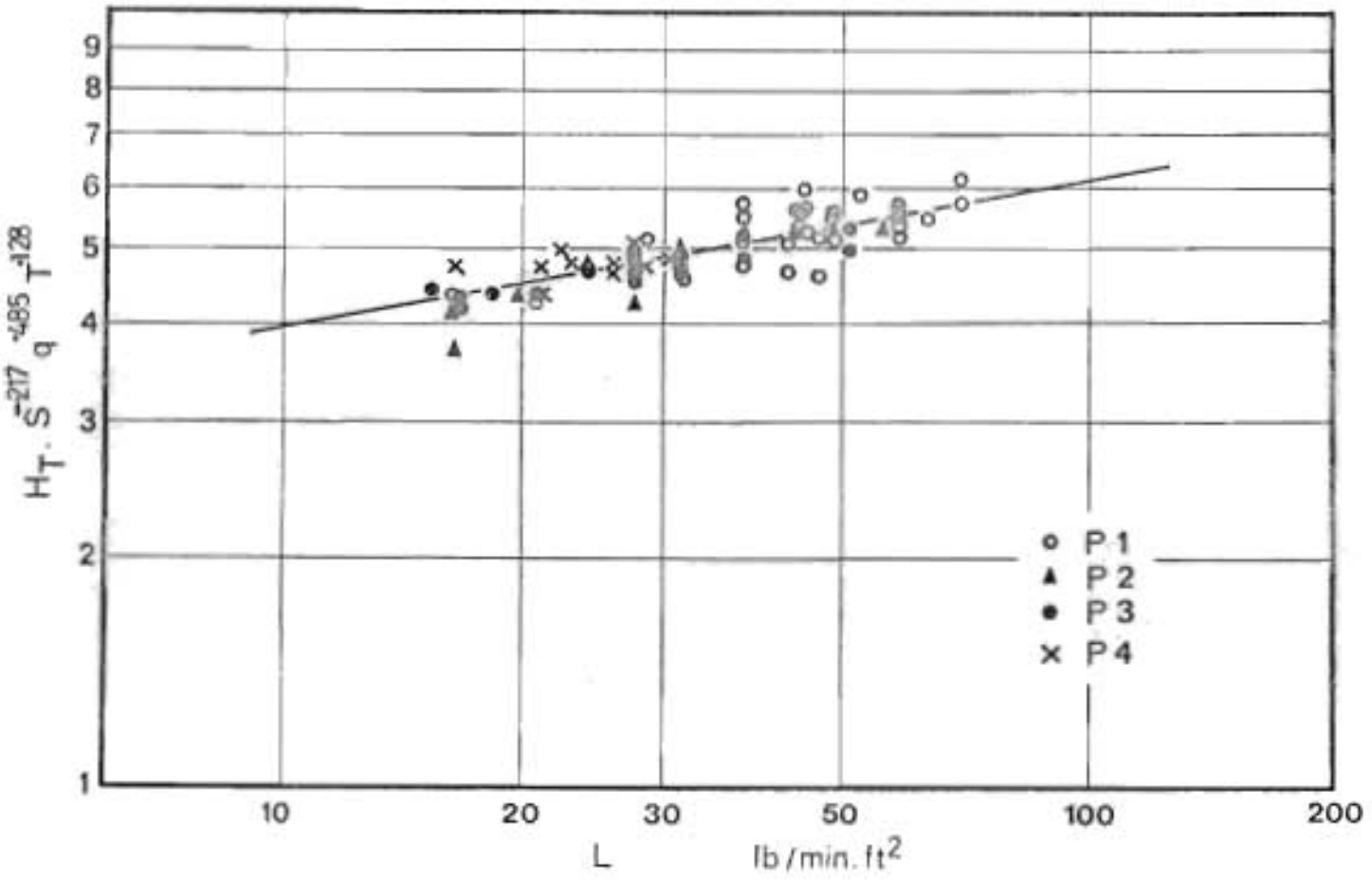


Fig. 5.23. Comparison between empirical correlation for total liquid holdup and experimental data.

This correlation is compared with experimental data in Fig. 5.23; the correlation is good, and is associated with a standard error of 5.6%. Once again, bed height was found to be a non-significant variable, with fibre density accounting for the observed apparent bed height dependence. Also S was again included as a more significant variable than d_c.

5.3.4 Static Holdup.

Measured values of static holdup are shown in Fig. 5.24 for the data obtained at 73°C, with a 125 lb. bagasse load. Lower values of H_S are obtained at higher flow rates, and with finer preparations. The effect of flow rate was again investigated by finding the best equation of the form:

$$H_S = A L^n \qquad (5.36)$$

for each type of preparation. Values of A, n and significance level are given in Table 5.12.

Table 5.12. Regression Coefficients for Equation (5.36)

Preparation	A	n	r Correlation Coefficient	p Significance Level
P1	4.19	-0.019	-0.059	> 0.1
P2	10.1	-0.285	-0.818	< .01
P3	13.5	-0.425	-0.719	< .001
P4	31.7	-0.782	-0.778	< .05

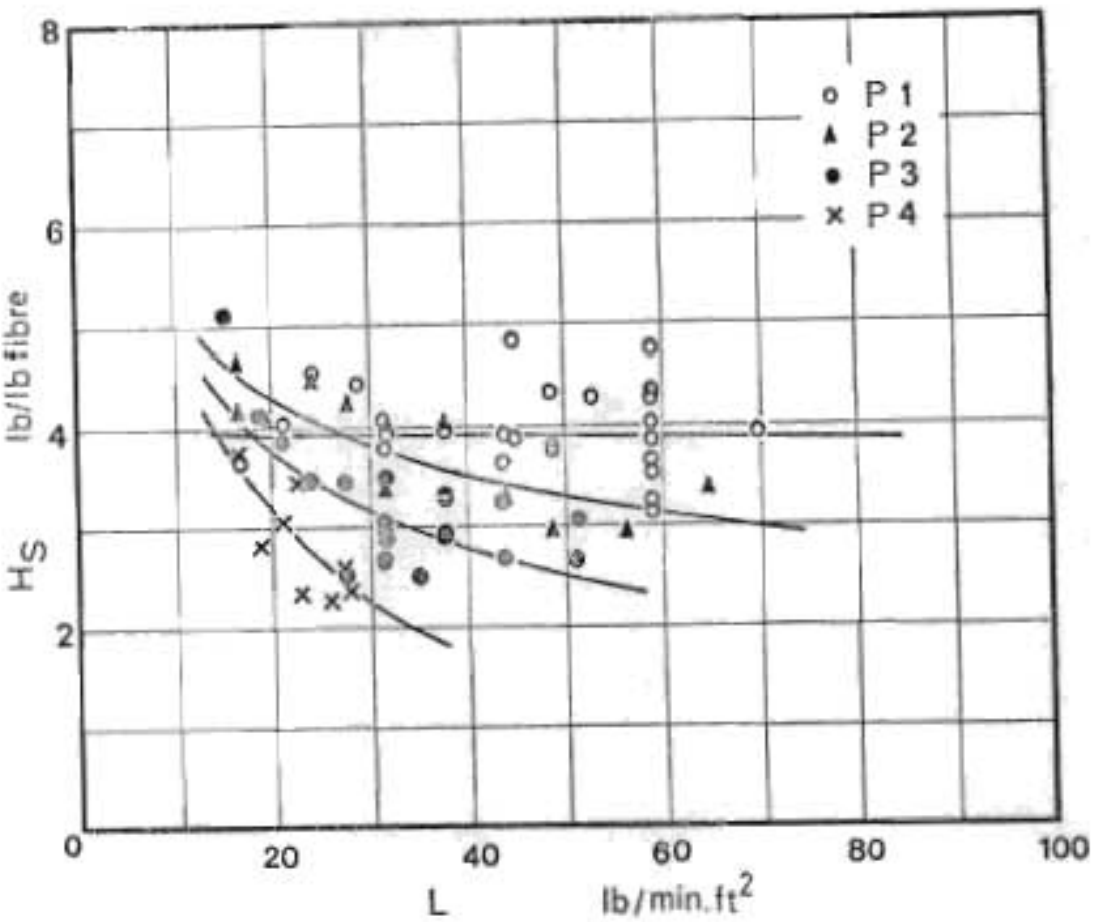


Fig. 5.24. Static liquid holdup as a function of liquid flow rate. Data shown represent operation at 73°C with an initial bagasse load of 125 lb.

All data except the P1 data show a significant dependence on flow rate. Scatter in these values is greater than shown by values of H_D and H_T. This is presumably due to the fact that H_S is a derived quantity, the difference between H_T and H_D. Further, Shulman et al (1955) reported that while H_D is dependent on packing size only, H_S varies with packing material and packing shape as well.

The data in Table 5.9 indicate that H_S is lower with greater bed heights (or higher fibre densities). This is illustrated in Fig. 5.25. The bold line through the data represents the least squares line.

$$H_S = -1.212\ q + 8.83 \qquad (5.37)$$

A correlation coefficient of -0.69 indicates that this relationship is significant at the 5% level (Volk, 1958).

The dependence on packing density can be interpreted as follows: at higher packing densities, the interstices between adjacent particles are smaller, so that each flow path supports a lower volumetric flow rate than in a less dense bed. Thus at the same flow rate, the number of flow paths must increase. Then more of the contact points between particles, where static liquid is retained, must serve as transfer routes for flowing liquid, so that the static holdup is decreased.

Table 5.10 shows that H_S is significantly lower at higher temperatures; this data is shown in Fig. 5.26, which also shows the least squares straight line:

$$H_S = -0.0389\,T + 7.18 \qquad (5.38)$$

This relation is significant at close to the 1% level.

From equation (5.38), the ratio of H_S at 60°C to the value of 90°C can be calculated as 1.30. From equations proposed by Shulman et al (1955 b), for the effect of fluid properties on H_S, the same ratio is predicted to be less than 1.10. However, the data of Table 5.10 show that higher fibre densities occur at higher temperatures, so this data demonstrates a combined effect of temperature and packing density.

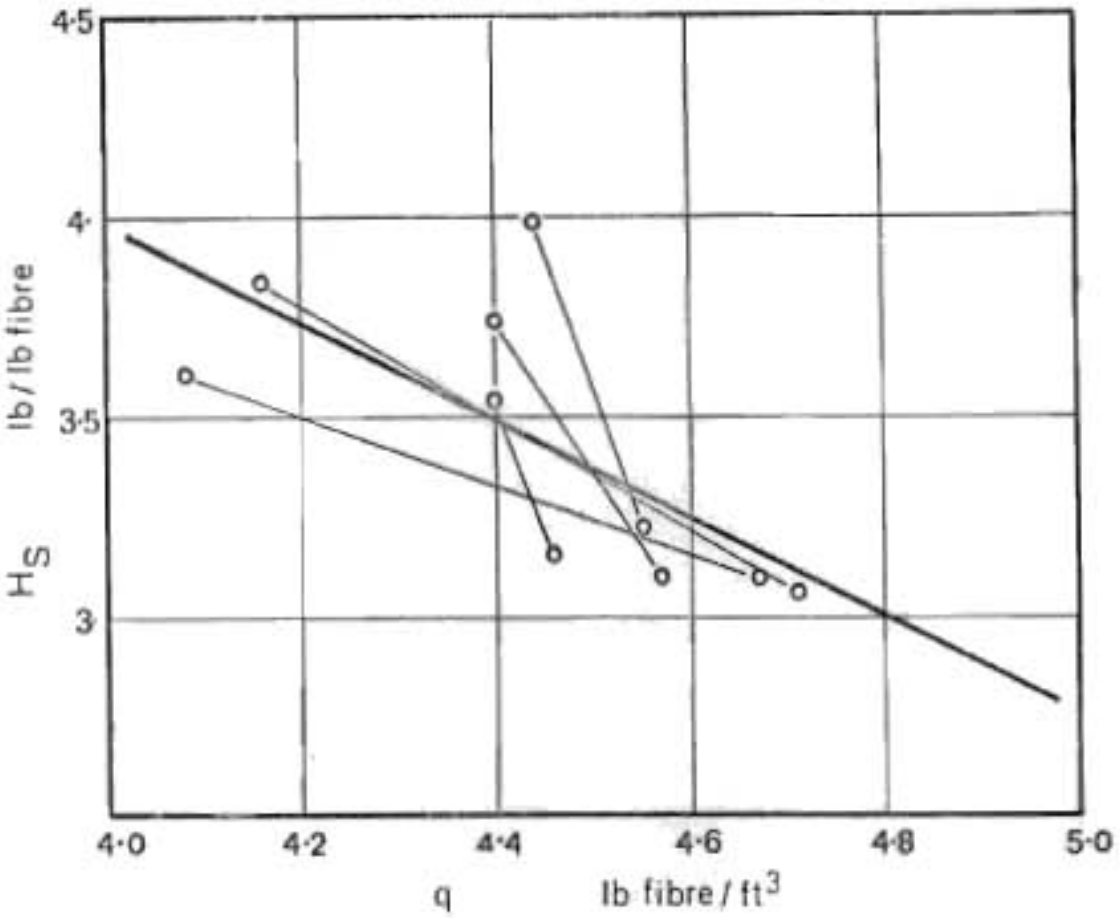


Fig. 5.25. Dependence of static liquid holdup on fibre packing density. Data shown represent operation with $L = 37.6 lb/min.ft^2$, at 73°C, with preparation type P1. Solid lines connect data obtained using subsamples of the same bagasse.

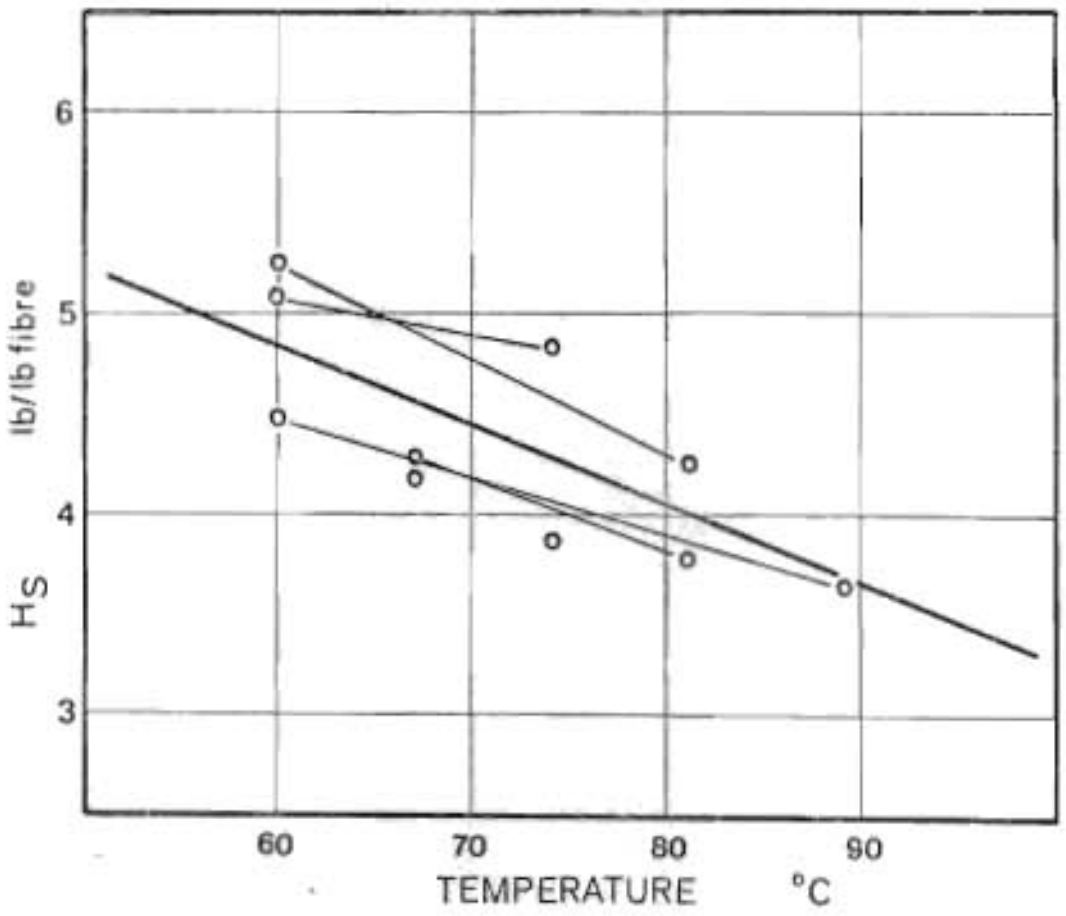


Fig. 5. 26. Dependence of static liquid holdup on temperature. Data represent operation at L = 44.6 lb/min. ft^2, with bagasse preparation P1. Solid lines connect data points obtained with subsamples of the same bagasse.

5.3.4.1 Correlation of Static Holdup Data.

Few attempts have been made to correlate static holdup measurements. These have been outlined in section 2.4.2, but lack the generality required for them to have any utility in this case.

Because they found H_S to be a function of packing shape, Shulman et al (1955) resorted to purely empirical means of correlation. A similar procedure was followed here, to obtain a correlation in terms of easily measured experimental parameters.

Multiple regression analysis applied to all the H_S data yielded the following correlation:

$$H_S = 2390\ L^{-0.204}\ S^{-0.432}\ T^{-0.506} \qquad (5.39)$$

This is compared with experimental measurements in Fig 5.27; as expected, the standard error is greater at 16.0%. The independence of H_S on q indicated by the regression analysis is unexpected. However, when the data obtained at 73°C only were subjected to regression analysis, the following equation obtained:

$$H_S = 426\ L^{-0.213}\ S^{-0.361}\ q^{-0.693} \qquad (5.40)$$

It appears that since T and q values are correlated to some extent (see previous section), the effect of temperature masks the dependence on fibre density. The standard error associated with equation (5.40) is lower at 14.7%. The absence of Z in equations (5.39) and (5.40) indicates that H_S is independent of bed height.

5.3.5 Reproducibility of Holdup Measurements.

A series of runs was carried out under the same conditions of flow rate, preparation and temperature, to investigate the magnitude of variations which can be expected under apparently identical conditions. The measured liquid holdup quantities are shown in Table 5.13.

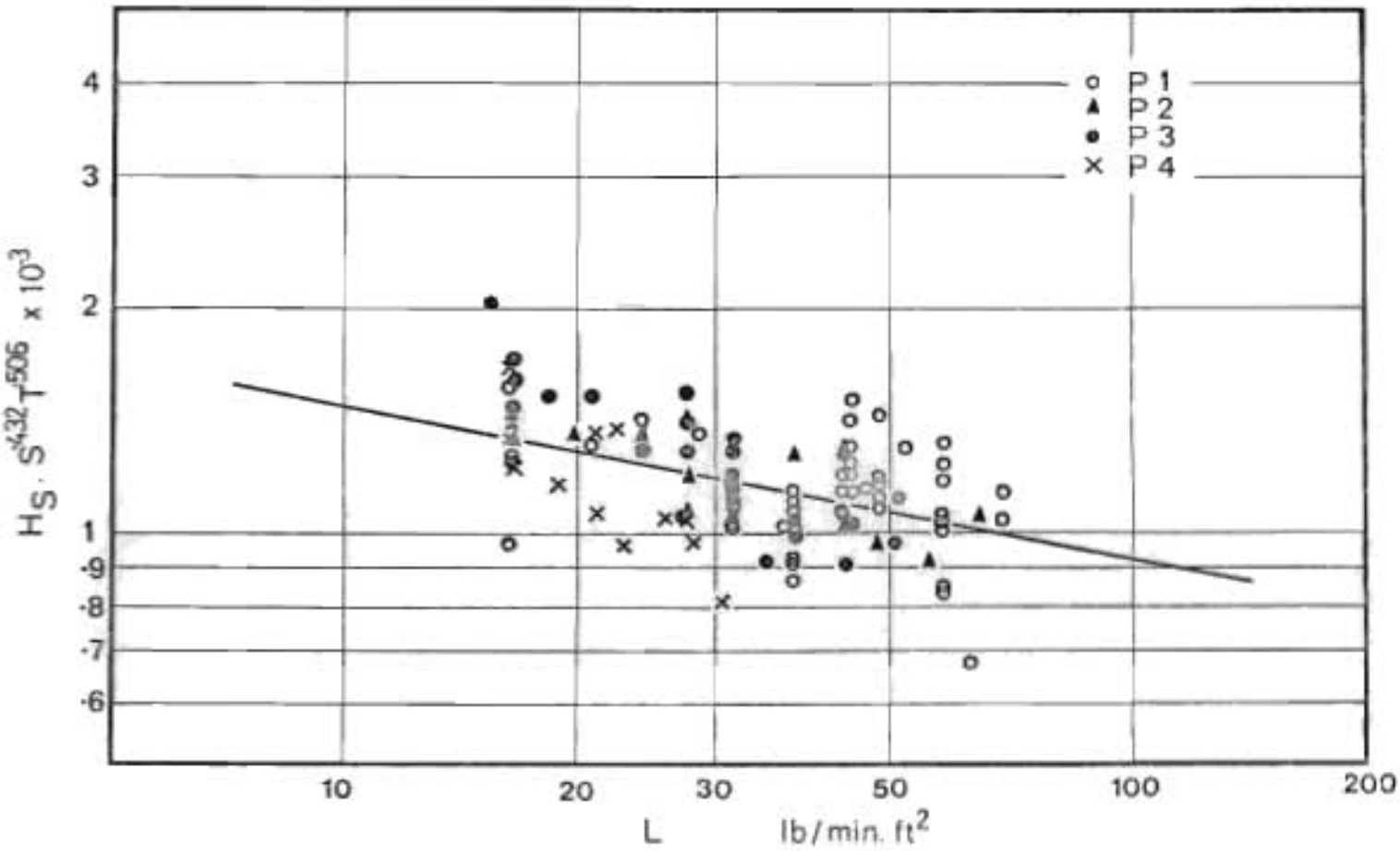


Fig. 5.27. Comparison between empirical correlation for static liquid holdup, equation (5.39), and experimental data.

TABLE 5.13. Reproducibility of Holdup Measurements.

Test Conditions: Flow rate 58.6 lb/min ft^2
Temperature 73°C
Preparation P1
Bagasse load 125 lb.

Run No	H_D (lb/lb fibre)	H_T (lb/lb fibre)	H_S (lb/lb fibre)
C 9	5.31	9.65	4.35
C10	5.24	8.76	3.52
C11	5.07	8.30	3.23
C12	4.74	8.75	4.01
C13	4.46	8.32	3.87
C14	5.49	8.55	3.07
C16	5.03	9.32	4.29
C17	4.27	7.90	3.63
C18	4.01	8.70	4.77
Mean	4.85	8.69	3.86
Std. deviation	0.51	0.53	0.55
s.d./ mean	10.5%	6.1%	14.3%

The horizontal lines in the table separate pairs of runs carried out using subsamples of the same bagasse. In order to test whether the variation within pairs of runs is any different from the total variation, an analysis of variance was carried out, and is summarized in Table 5.14.

TABLE 5.14 Analysis of Variance for Liquid Holdup Measurements.

	Between Pairs	Within Pairs	Total
H_D			
Sum of squares	1.439	0.621	2.060
Degree of freedom	4	4	8
Variance	0.360	0.155	-
H_S			
Sum of Squares	1.416	0.844	2.260
Degree of freedom	4	4	8
Variance	0.354	0.211	-
H_T			
Sum of squares	0.847	1.618	2.465
Deg. of freedom	4	4	8
Variance	0.212	0.405	-

The variance ratios for H_D, H_S and H_T are 2.32, 1.68 and 0.52 respectively. All 3 values are less than the value of $F_{4,4,.05}$ of 6.39 (Volk, 1958). Thus the hypothesis that the variation between pairs is the same as the variation within pairs cannot be rejected.

The same conclusion was obtained for a series of 7 runs using P3 preparation, even if the variance estimates for the 2 sets of data are pooled.

This result is unexpected; since particle size and shape affect holdup values, it may be expected that repeat determinations on beds of bagasse obtained from the same subsample should show less variation than determinations on different bagasse samples. The magnitude of the variations is far greater than could be expected from the accuracy of the measurement techniques employed (see section 4.2.4). Nevertheless, in the case of H_D, it was shown that the standard error of the correlation proposed is lower than that of most published correlations.

This suggests some inherent variability associated with packed bed operation. Other workers (Jesser & Elgin, 1943; Gunn & Pryce, 1969) have confirmed that the experimental variance of measurements in packed columns is an order of magnitude greater on re-packing the bed than the experimental variance found on repetition without re-packing. Thus it appears that the relative arrangement and orientation of packing elements affects the flow characteristics.

5.4 THE OCCURRENCE OF FLOODING.

The occurrence of flooding limits the maximum operating flow rate; this constitutes a constraint in the selection of optimum diffuser operating conditions and as such warrants more than cursory attention.

It was pointed out in section 5.3.2.1 that flooding is detrimental to performance if flooding occurs to an extent where the liquid level rises above the level of bagasse. Any state of flooding at a lower flow rate, where the liquid level lies below that of the top of the bed, is an acceptable operating condition. Thus it is necessary to attempt to establish the conditions under which excess flooding occurs, so that in practice, the flow rate can be controlled at some lower level. Although the existence of a liquid level anywhere within the bagasse implies some form of localised flooding, in keeping with the objective, the flooding flow rate, L_f, is defined as that flow rate which maintains the liquid level at the top of the bagasse bed. The objective can then be re-stated as establishing the dependence of L_f on process operating conditions.

5.4.1 General Observations.

It was at once apparent from operation of the pilot plant that flooding occurred at lower liquid throughputs with more finely prepared bagasse. This sensitivity to the degree of preparation is well known in the operation of full-scale diffusers. These facts are consistent with the observations of Mayo et al (1935) that flooding occurs earlier with smaller packings.

Even with the same type of preparation, the flooding flow rate appeared to be extremely variable. Clearly particle size alone is insufficient to determine when flooding will occur. In addition, experimental observations showed that flooding occurred at lower flow rate at higher temperatures, and in higher beds of bagasse.

The extent of flooding could be observed through the perspex window in the diffusion vessel. Flooding was observed to start at the bottom of the bed, followed subsequently by the movement of the liquid level up through the bed. The liquid level was seldom observed to be static in the bed,

generally rising continuously, rapidly at first, and then more slowly. A corresponding steady increase in the total weight of the column was indicated by the load beam weight recording. Thus it is clear that the flooding flow rate, as defined above, is a function of time, and that the flooding flow rate at a particular time will therefore be greater than the flooding rate at some later time. Generally, however, when excess flooding did not occur, the liquid level had for all practical purposes stabilised by the end of a run.

When flooding occurred, pockets of air were often observed within the bagasse bed. In order to estimate the extent of air occlusion during flooding, the volume of liquid and fibre within the bed was calculated, and compared with the total packed volume. Typical results are shown in Table 5.15. The calculation assumes values of density of liquid and fibre as 61.0 and 94.9 lb/ft^3 respectively. (See Appendix G). This table shows that a significant amount of air is trapped in a flooded bed, and that continuous liquid phase operation is not realized.

5.4.2 Effect of Bed Height.

Observations of lower values of the flooding flow rate with higher packed heights were recorded above. It was shown in section 5.3.2.2 that higher bed heights are associated with higher fibre densities, i.e. more compaction occurs due to the greater weight of a higher bed. This suggests that the bed height effect may be explained in terms of density of packing. The fact that flooding was observed to begin at the bottom of the bed, which is expected to be the region of greatest local packing density, is consistent with this proposal.

When liquid was re-circulated through the bagasse, flooding was sometimes observed to occur on the top of the bed. This is due to the fact that fine pith particles were washed out of the bed, deposited on top of the bed, and plugged the bed surface. Such an effect has also been observed on the factory scale; unscreened press water returned to the diffuser leads to flooding problems due to plugging of the top of the bed with fines.

Such problems can be overcome by screening, clarification or other means, and are the concern of the factory staff. However, this does suggest that flooding may occur from the bottom of the bed due to the collection of a layer of fines at the bottom of the bed. Thus, samples of

Table 5.15

Estimates of the amount of air trapped in a flooded bed.

Run No.	Preparation	Inlet Flow Rate (lb/ min)	Total liquid holdup at flooding (lb)	Weight of fibre (lb)	Volume of liquid and fibre (ft^3)	Packed volume (ft^3)	Fraction air in packed volume
C33	P1	303	434	36.4	7.49	7.83	0.046
D39	P1	250	417	37.4	7.24	8.44	0.142
C34	P2	352	372	33.9	6.46	8.12	0.204
C53	P2	303	491	40.1	8.47	9.56	0.114
D36	P3	227	416	38.2	7.22	8.38	0.138
C38	P4	98	411	38.9	7.15	8.38	0.147

bagasse were taken from the top and the bottom of the bagasse after two flooding runs. These were dried and sieved in the normal way; the values of specific surface so obtained are given in Table 5.16, together with the value obtained from the subsample obtained for analysis purposes.

Table 5.16

Comparison of sieving results of bagasse samples.

Run No.	Values of S (mm^2/g) for sample from :-		
	Top of bed	Bottom of bed	Normal analysis.
J1	3200	3600	4100
J24	3400	3100	3600

It is suggested that the small differences between samples reflect only difficulties in obtaining representative samples, and that the collection of fines at the bottom of the bed does not occur to any significant extent. This lends support to the proposal that flooding is initiated at a region of maximum density at the bottom of the bed.

The effect of bed height was investigated in greater detail by determining L_f in two sets of runs, where each set consisted of four runs at different bed heights, using subsamples of the same bagasse. The value of L_f was measured approximately eighteen minutes after the beginning of a run, thus allowing some degree of stabilization. The results are shown in Table 5.17.

Table 5.17

Effect of bed height on L_f (at 73°C)

Run	Bagasse load (lb)	Bed height (ft)	q (lb fibre/ft^3)	S (mm^2/g)	L_f (lb/min ft^2)
K1	125	2.65	4.62	4770	53.5
K2	175	3.54	4.81	4770	38.5
K3	150	3.02	4.85	4770	41.4
K4	100	2.19	4.47	4770	57.9
K5	150	3.0	4.81	4160	68.4
K6	100	2.17	4.47	4160	85.9
K7	175	3.50	4.81	4160	64.3
K8	125	2.63	4.58	4160	75.1

The data are shown plotted against bed height in Fig. 5.28 and against fibre density in Fig. 5.29. The displacement between the two sets of data illustrates the variable flooding rates which can be obtained in practice with the same type of preparation. The lower levels of L_f obtained in the first set are associated with a higher value of S.

It appears that the dependence of L_f on bed height may equally be ascribed to a variation in fibre density.

5.4.3 Effect of Temperature

The effect of temperature was investigated by determining L_f at different temperatures on the same bed of bagasse. Earlier work had shown that higher temperatures result in greater fibre densities, presumably due to the softening of the fibres, leading to a greater degree of compaction. Therefore L_f was determined at a low temperature first, and

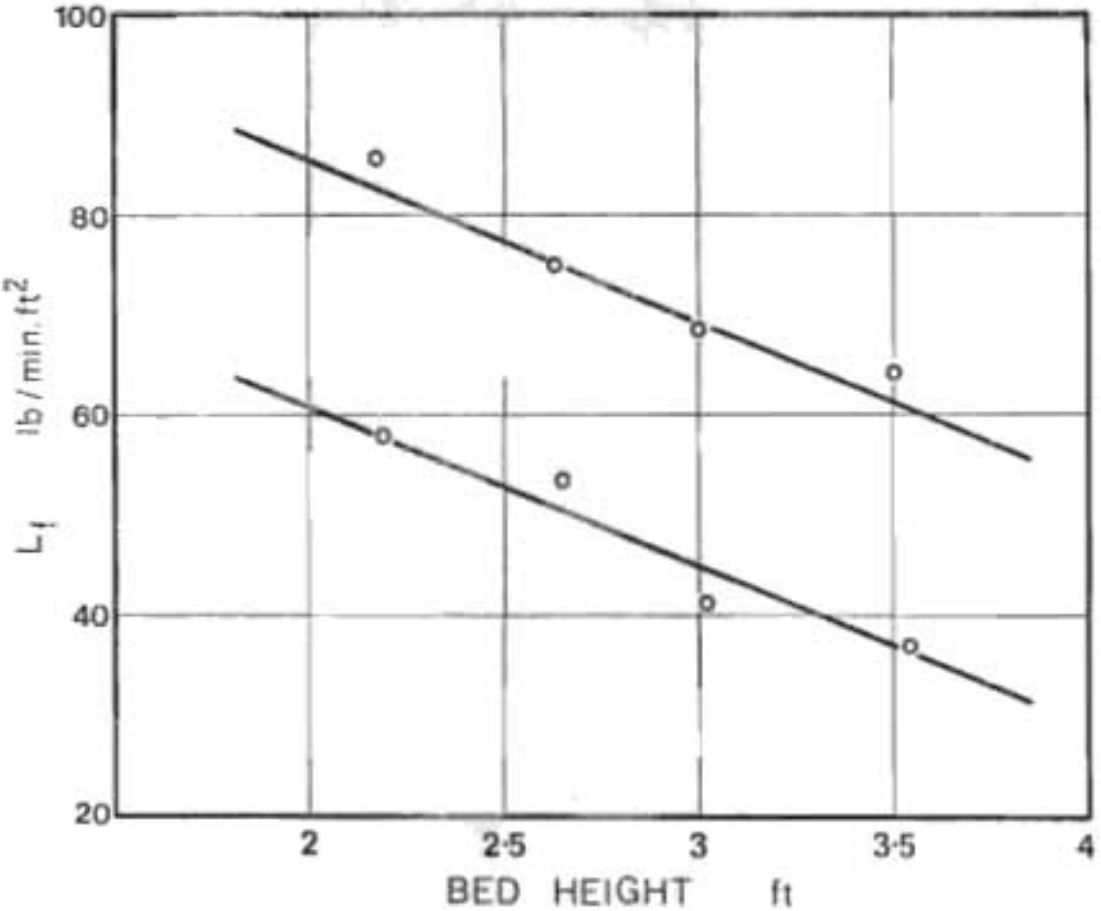


Fig. 5. 28. Effect of bed height on flooding flow rate. Graphical representation of the data in Table 5. 17. Bagasse preparation P1.

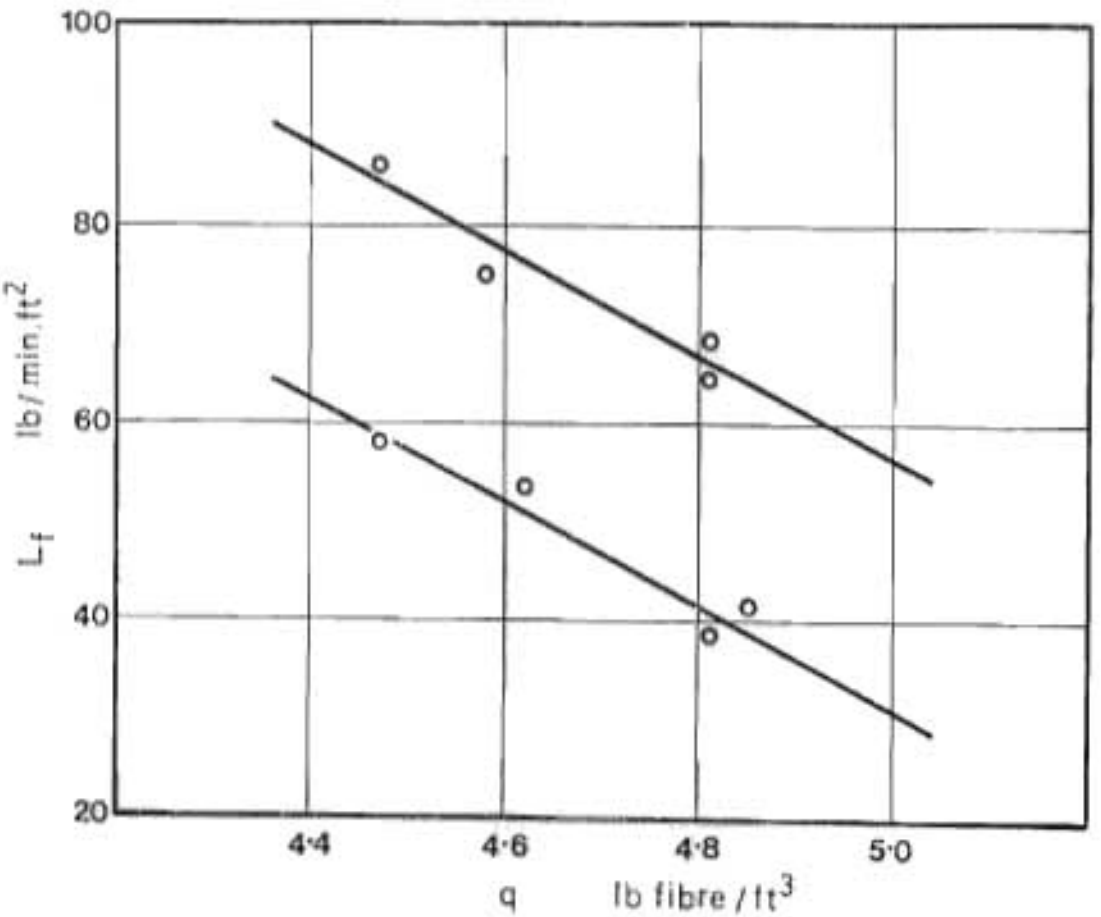


Fig. 5. 29. Flooding flow rate as a function of fibre packing

then at progressively higher temperatures.

Two determinations were carried out on two different beds (but utilising bagasse from the same sample). The results are given in Table 5.18, and are plotted in Fig. 5.30.

Table 5.18

The effect of temperature on L_f

Run No.	Temp. (^{o}C)	Bed Height (ft)	q (lb. fibre/ft^3)	L_f (lb/min. ft^2)
K9	70	2.52	4.85	90.4
	74	2.52	4.85	92.9
	81	2.52	4.85	99.6
	87	2.50	4.89	96.1
K10	67	2.60	4.70	79.3
	74	2.58	4.74	78.9
	81	2.54	4.81	77.0
	88	2.50	4.89	72.6

The results show an apparent inconsistency, in that run K9 shows an increase with temperature initially before dropping off, while K10 displays a continuous decrease with temperature. This discrepancy can be explained by considering the values of fibre density, q, given in Table 5.18. The first three points of run K9 have the same value of q, and L_f increases with temperature. This may be attributed to less flow resistance due to a lower liquid viscosity. At 87^{o}C, L_f drops in accordance with an increase in fibre density. Run K10 displays a continuous increase in fibre density. Thus it appears that packing density has a far more significant effect on L_f than viscosity (or temperature).

It should be emphasised that the data of Table 5.18 is of an approximate nature; obtaining this data entailed draining the bagasse bed after determining L_f at each temperature, since

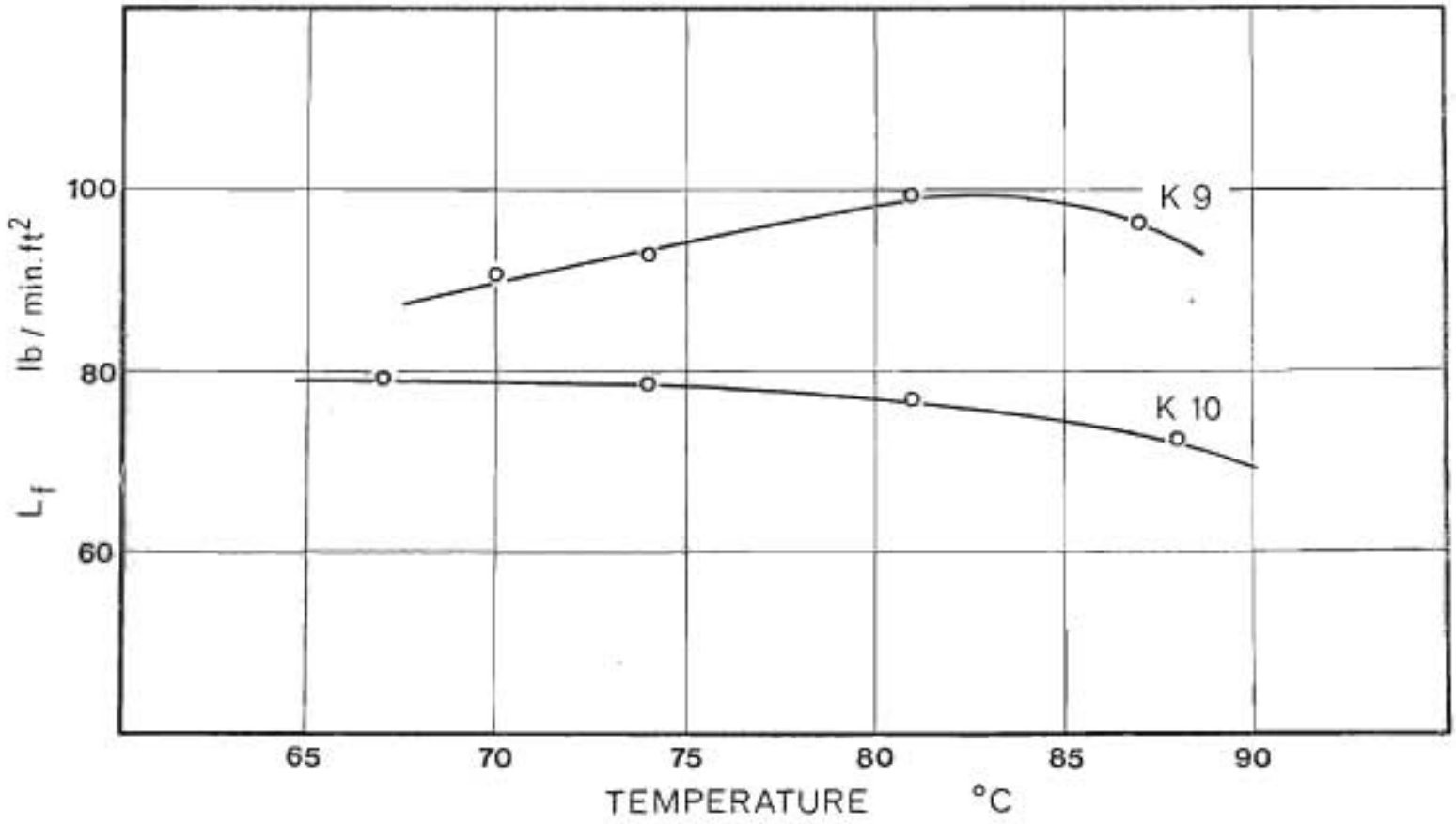


Fig 5. 30. Effect of temperature on the flooding flow rate. Graphical representation of the data in Table 5. 18. Bagasse preparation P1.

a period of some minutes was required between each determination to raise the water temperature in the tank to the required level ; then percolation had to be re-established and stabilised before the next determination could be made. Nonetheless, the difference in the levels of L_f in each run is inexplicable.

A general explanation of the occurrence of flooding can now be made. It appears that flooding occurs when the inter-particle space is not sufficiently large to permit the required volume of liquid to flow through. An increase in the fibre density results in a reduction in the inter-particle spaces, and so flooding occurs at a lower flow rate. Lower viscosities at higher temperatures allow more liquid to flow through the interstices, and so flooding occurs at a higher flow rate, provided the fibre density remains constant. Generally, however, higher temperatures lead to a softening of the fibres which results in an increase in the packing density. This effect overrides the effect of reduced liquid viscosity, and so there is an apparent decrease in L_f at higher temperatures.

5.4.4 Prediction of Flooding.

Although the literature abounds with work on two-phase flooding in packed columns, the case of single-phase flooding has been largely neglected. Only one reference to the latter case (Lavin, 1964) could be found.

Even if work on single-phase flooding had clearly established the conditions under which flooding occurs, it might be expected that significant discrepancies between theory and practice would exist in this case, for the following reasons:

1. Bagasse consists of particles of a wide range of complex shapes and sizes,

2. Beds of fibrous particles do not follow the normal equations for the pressure drop in packed beds, due to increased static liquid within the bed (Kyan et. al., 1970), and

3. It was shown earlier that a flooded bagasse bed contains significant amounts of trapped air within the bed.

The work of Lavin (1964) can be used as a guide in attempting to establish a locus of flooding. He found that flooding flow rates could be correlated by the relation -

$$L_f / \rho \left(\frac{a_T}{\varepsilon^3} \; SF \; \mu^n \right)^{\frac{1}{2}} = C' \qquad (5.41)$$

where SF represents a shape factor, ε the porosity or void fraction and C' is a constant. The exponent, n, on μ was found to depend on the value of μ, varying between 0.1 for water, to over 1 for liquids of high μ. This equation can be re-arranged to a more convenient form :-

$$L_f = C' \rho \left(\frac{\varepsilon^3}{a_T \; SF \; \mu^n} \right)^{\frac{1}{2}} \qquad (5.42)$$

This equation shows that L_f is strongly dependent on the porosity, or void volume within the bed. Since in a bagasse bed, liquid is present in the voids between bagasse particles as well as within the particles, the value of ε to be used in this equation is not clearly defined. In this case, the fibre density, q, furnishes a measure of the 'openness' of the bed, and can be used instead of ε ; it is inversely related to ε . As regards the shape factor, the only assumption that can be made is that it is constant for all the bagasse used in this study, and can therefore be incorporated in the constant. Equation (5.42) can then be rewritten as :

$$L_f = C'' \rho \left(\frac{1}{a_T \; q^m \; \mu^n} \right)^{\frac{1}{2}} \qquad (5.43)$$

If laminar flow exists, it can be expected from Darcy's law that liquid velocity, and hence flow rate, is proportional to $1/\mu$, in which case n in equation (5.43)

takes the value 2. This is considerably larger than the range of values reported by Lavin (1964); if turbulent or partly turbulent flow exists, it is to be expected that n should be lower, as viscous forces play a less important role.

In order to establish the exponent on q, values of liquid flow rate for all runs at 73°C were plotted against the corresponding values of $a_T q^m$, where m assumed only integer values. Fig. 5.31 shows L plotted against $a_T q^3$; a value of m = 3 yields the best separation between flooding and non-flooding points. The solid points represent runs where complete and/or over-flooding occurred. These values are listed in Tables 5.19 and 5.17. The semi-solid points represent runs where partial flooding occurred, evidenced by a liquid level within the bed. The solid line in Fig. 5.31 represents the equation

$$L_f = 2.17 \times 10^4 \left(\frac{1}{a_T q^3} \right)^{0.664} \qquad (5.44)$$

The present data shows that, with few exceptions, equation (5.44) represents a locus between non-flooding or partial flooding, and excess or over-flooding. Thus values of L should be kept below the value calculated from equation (5.44).

The exponent on q was chosen by visual inspection of plotted data. In addition the use of a_T^2 instead of a_T in equation (5.44) was tried with less satisfactory results. Thus it is clear that no claim to the functional correctness of equation (5.44) can be made. However, on the basis of the present data, it gives the best representation of the flooding locus, and can be used only to estimate when over-flooding is likely to occur.

Unfortunately, insufficient values were obtained for flooding at temperatures other than 73°C, so the dependance on μ cannot be clarified. It would appear, however, to be small. The best assumption which can be made is that L_f shows the same dependence on μ as that published by Lavin (1964). In any event, the effect of increased values of q at higher temperatures completely overrides the effect of changed liquid properties.

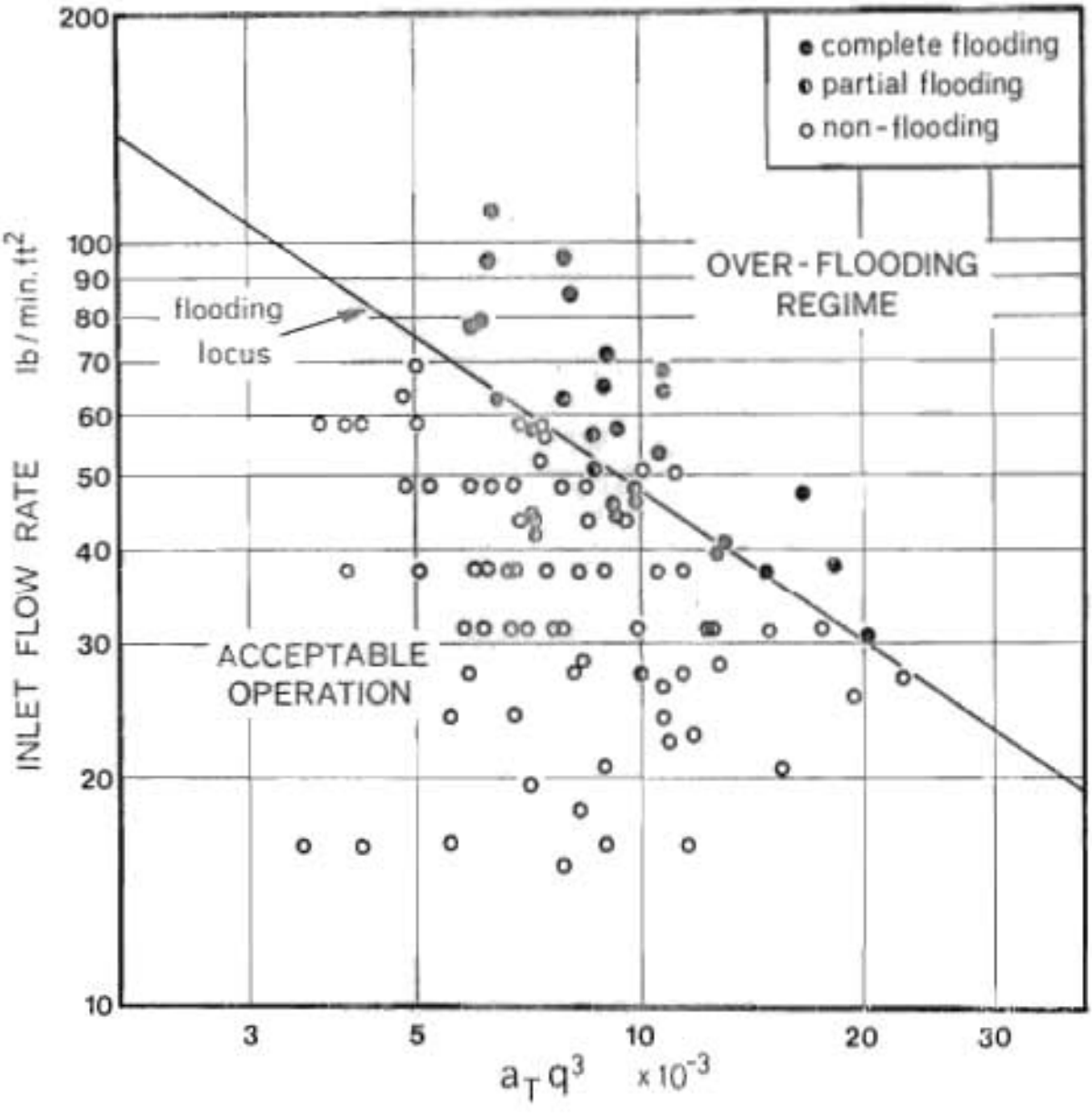


Fig. 5.31. Comparison of proposed flooding locus, equation (5.44), with experimentally observed behaviour. Flooding locus represents the boundary between regions where excess flooding occurs (above locus) and where acceptable operation attained without over-flooding (below locus).

Table 5. 19

Flooding data, 73°C.

Run No.	Preparation	L (lb/min ft^2)	S (mm^2 / g)	q (lb fibre/ft^3)	a_T (ft^2 / ft^3)	$a_T q^3$
C33	P1	95.8	3530	4.64	79.9	7980
D39	P1	79.3	3280	4.43	70.8	6160
E12	P1	62.4	2950	4.60	66.2	6440
C34	P2	110.8	4340	4.17	88.4	6410
C44	P2	79.9	3020	4.49	66.2	5990
C53	P2	94.2	4152	4.20	85.1	6310
D36	P2	71.6	4310	4.56	95.9	9090
C36	P3	47.1	7730	4.58	172.9	16600
E8	P3	37.2	6860	4.59	153.8	14900
C28	P4	38.2	9080	4.51	200.0	18400
C38	P4	30.9	8950	4.65	203.1	20400

Equation (5.44) indicates that L_f is affected to a greater extent by fibre density than particle surface area per unit volume. This indicates that the mean particle size can be reduced, with corresponding benefits in extraction, without seriously decreasing the maximum percolation rate, provided the type of bagasse preparation ensures an open bed of low fibre density.

5.4.5 Fibre Packing Density.

Discussion of liquid holdup and flooding results has shown the important effect of the fibre density of a bagasse bed on the liquid hydrodynamics. In view of this, a statistical regression analysis was performed as outlined in Appendix F, in order to establish the factors which affect the fibre density.

When P1 data only were considered for the analysis, it was found that the fibre density q is dependent on Z, σ_g, S and temperature (5% significance level). A regression equation was obtained, with a standard error of 3.5%, which indicated higher values of q with higher values of the 4 independent variables mentioned above. This confirms the conclusions drawn earlier that the value of q is larger when greater bed heights are employed (due to the greater compacting load) and at higher temperatures (due to softening of the fibres). In addition, higher values of q are favoured by finer bagasse, and a greater standard deviation of the particle size distribution.

When the data for all types of bagasse preparation obtained at 73°C only were analysed in the same way, σ_g appeared as the only significant independent variable. This indicates that σ_g is the most important single variable influencing the fibre density, and substantiates the conclusions of Sohn & Moreland (1968) that the value of σ_g defines the value of packing density of a bed of particles which follow

a log-normal size distribution.

However, when all the data obtained were subjected to the same analysis, none of these independent variables proved to be significant at the 5% level. This can only be assumed to be due to the observation that the data obtained at temperatures other than 73°C generally displayed an abnormally large degree of scatter.

5.5 APPLICATION OF THE MODEL TO PILOT PLANT DATA.

Once a mathematical model of a process has been postulated, it is necessary to establish whether the model is capable of satisfactorily representing the experimentally determined behaviour of the process. The fulfilment of this requirement is in itself an insufficient criterion for the adequacy of the model. In the formulation of the model, some physical significance of the parameters is implied. The 'best fit' in some sense of the model to experiment yields corresponding values of the parameters, which will be dependent on the experimental process conditions. The validity of the formulation may be assessed quantitatively by the relationship between the magnitudes of the parameters and values anticipated on the basis of a physical interpretation of other related experimental findings.

In Chapter 3, the model was formulated for the pilot plant situation, which resulted in a relation between the concentration of sucrose in the percolating liquid as a function of time, position in the bagasse bed, and the three model parameters (equation 3.26). This enables comparison between C_j predicted from equation (3.26) and measurements of the sugar concentration in the juice from the outlet of the diffusion vessel. Typical experimental data is shown in Figs. 5.32 and 5.33.

This section is concerned with details of the computation procedures, the degree of fit of the model to experimental data, and parameter estimation procedures. The values and behaviour of the model parameters follows in section 5.6.

5.5.1 Calculation Procedures

The input to the function evaluation routine consisted of the liquid flow rate, brix % bagasse, the juice holdup in bagasse, H, and values of the model parameters. The value of H was calculated from the value of the static holdup, less brix-free water, calculated as 25% of the fibre content. (see section 2.1.2.2.)

Ideally, measured and predicted values of C_j should be expressed as mass fractions. In practice, brix values were used, where brix represents the concentration by weight of soluble solids, expressed as a percentage.

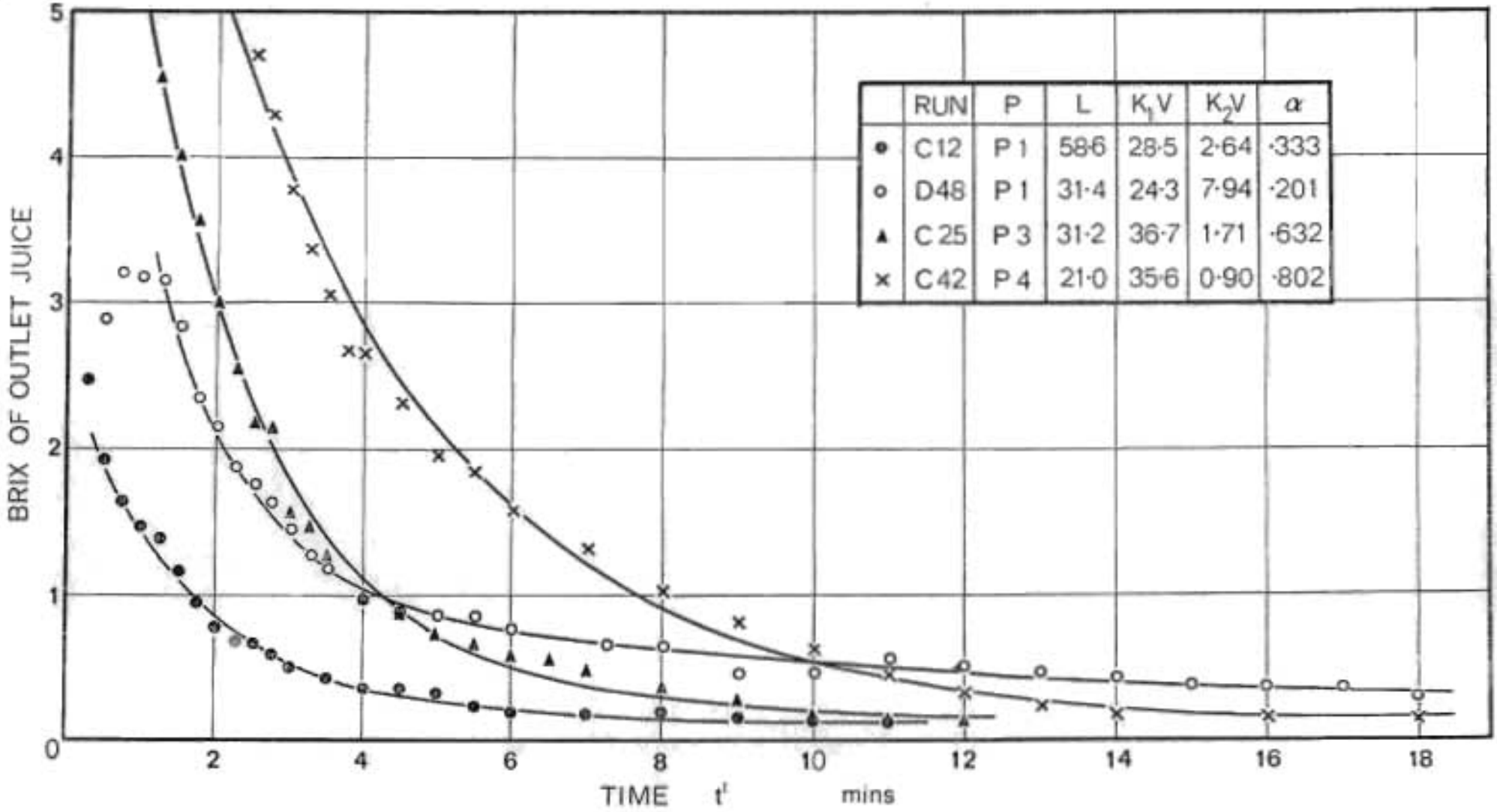


Fig. 5. 32. Comparison between typical pilot plant experimental data and model predictions. Solid lines represent the best fit of the model to the data.

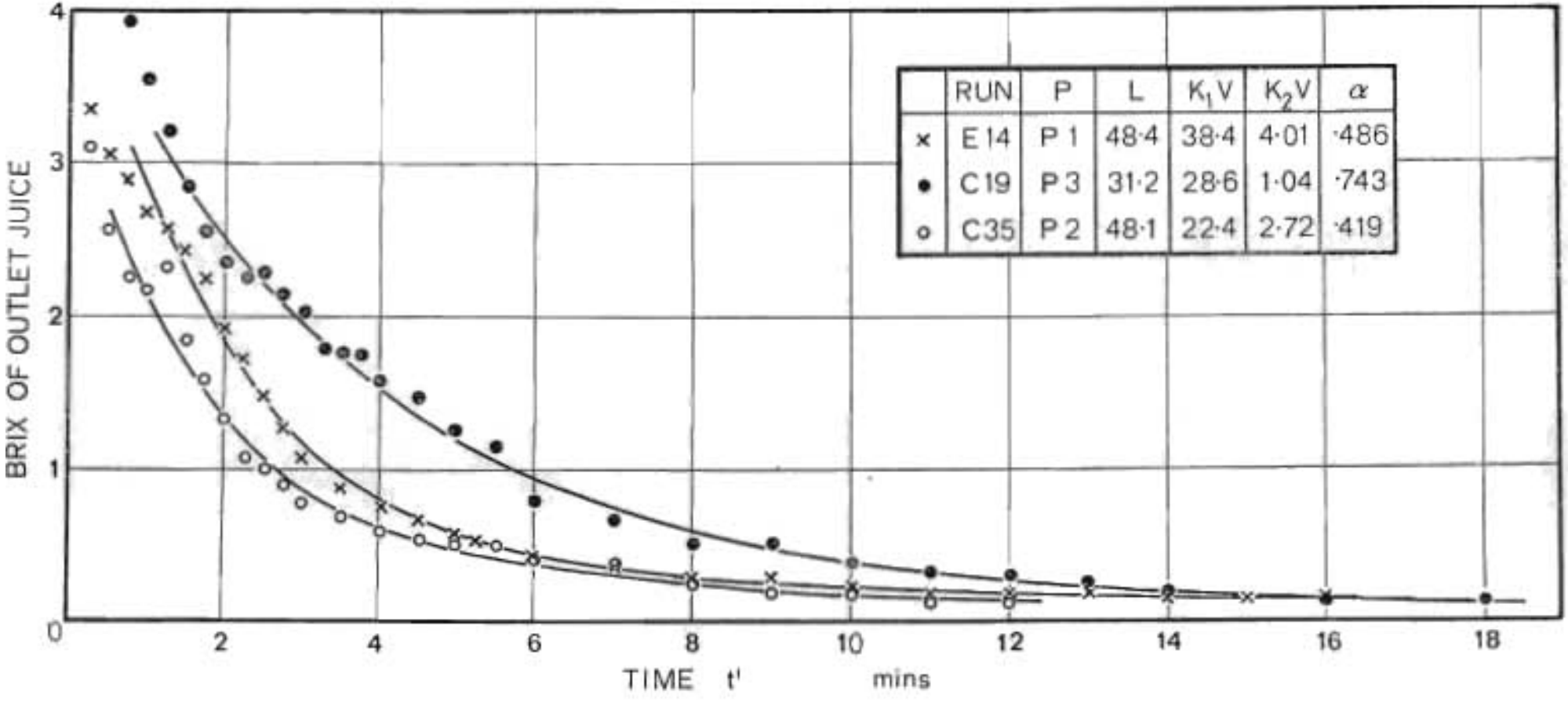


Fig. 5.33. Comparison between typical pilot plant experimental data and model predictions. Solid lines represent the best fit of the model to the data.

Computation of the function given in equation (3. 26) was complicated by the necessity to evaluate the integrals numerically. Numerical integration was carried out using the library program, GQUAD, of the Department of Chemical Engineering, University of Natal. The program performs the integration over a specified time interval using a five-point Gaussian quadrature. The integration is repeated with a Gaussian four-point formula; if these do not agree within a specified error, the time interval is halved and the procedure repeated until the error criterion is satisfied.

In the evaluation of the double integral of equation (3 . 26), it was found unnecessary to check the integration procedure in this way. This is understandable when it is considered that the value of this term was generally <5% of the two single integrals, and often <1%.

Bessel functions were evaluated using polynominal approximations given by Abramowitz and Stegun (1965).

5. 5. 1. 1 Checks on
Calculation Procedures

A number of computations were carried out which served as a check on the accuracy of the calculation procedures employed. Firstly, it was established that interchanging the values of K_1 and K_2, and replacing the value of α by $(1 - \alpha)$, led to identical values of the function C_j. This is not obvious from the form of equation (3 . 26), but is consistent with model formulation. Further, this indicates that the parameter space must contain two minima; since the basis of the model implies that $K_1 >> K_2$, this at no stage led to confusion as to which minimum represented the required parameter values.

Secondly, the concentration of sugar in bagasse, C_b , is given by :

$$C_b (N, \Theta) = \alpha C_{b1} (N, \Theta) + (1 - \alpha) C_{b2} (N, \Theta) \qquad (5.45)$$

Using this, equations (3.17) to (3.19) can be combined to yield

$$\frac{\delta C_b}{\delta \theta} = -\frac{\delta C_j}{\delta N}$$

Therefore

$$C_b(N, \theta) = C_{bo} - \int_o^{\theta} \frac{C_j(N, \theta)}{\delta_N} \, d\theta \qquad (5.46)$$

In order to obtain the average value of C_b in the bagasse bed, both sides of the equation (5.46) need to be averaged with respect to N. Thus -

$$\bar{C}_b = \frac{1}{N}\int_o^N C_b \, dN = C_{bo} - \frac{1}{N}\int_o^{\theta} C_j \, d\theta \qquad (5.47)$$

This relation furnishes two alternative methods of calculating $\bar{C}_b$, firstly by calculating C_b from equations (3.27), (3.28), and (5.45) and integrating numerically with respect to N, and secondly by calculating C_j from equation (3.26) and integrating numerically with respect to θ. Values of $\bar{C}_b$ computed in the two different ways were found to agree within 0,05%.

These findings confirm the accuracy of the calculation procedures employed.

5.5.2 Parameter Estimation

Values of the model parameters were obtained from the 'best fit' of the model to experimental data. The minimisation procedure outlined by Powell (1964) was utilised for this purpose. This is a 'direct search' method, which unlike the 'gradient methods', does not require calculation of derivatives of the model with respect to the parameters. This method was initially chosen for its simplicity, and because the calculation of derivatives would be extremely complex. Powell claims that when this minimisation scheme is applied to a quadratic form, it causes conjugate directions to be chosen for the variables, so that the ultimate rate of

convergence is rapid when the method is used to minimise a general function. In this case, Powell's method was found to be eminently suitable.

The form of the function to be fitted is complex, so that the time involved in function computation was vastly in excess of that required to organise the search. Box (1966) has shown that the number of function evaluations required by Powell's method is close to the number required when two different 'gradient methods' were applied to the same problems, if each derivative computation is considered equivalent to a function computation. Time requirements on an ICL 1901-A computer for each function computation and for complete convergence were of the order of 30 seconds and 30 minutes respectively. These times varied considerably, depending on the number of data points per run and the initial estimates of the parameters.

Only minor modifications were made to the search procedure as outlined by Powell (1964). Firstly, with poor initial estimates of the parameter, it occasionally happened that the second derivative of the function (assuming a quadratic form) was negative, in which case the procedure was terminated and re-started with new initial estimates of the parameters.

Secondly, a simplified convergence procedure was employed. Powell has outlined a comprehensive convergence criterion which was reported to always yield the required accuracy, although it causes many unnecessary function evaluations. In this case, when an iteration caused each variable to be changed by less than the required accuracy, provided a new direction was defined by the iteration, convergence was assumed. The adequacy of this criterion was confirmed by initiating the search procedure from a number of different points in the parameter space. Final parameter values well within the required tolerances confirmed that the ultimate convergence rate is better than linear.

5. 5. 2. 1 Best fit criterion.

Initially, the best fit of the model to the pilot plant data was obtained by minimising the sum of squared errors, Φ_o, where

$$\Phi_o = \Sigma (y_i - C_{ji})^2 \qquad (5.48)$$

where y_i and C_{ji} refer to observed and predicted values of the outlet juice concentrations respectively. It was found, however, that Φ_o was insensitive to the value of K_2V. The value of K_2V has the greatest effect on values of C_i at longer times; thus a normalised sum of squares function, Φ_2, was tried:

$$\Phi_2 = \sum (y_i - C_{ji})^2 / y_i^2 \qquad (5.49)$$

This, however, gave too much weight to points with low values of y_i, which adversely affected the fit to the initial data points. Thus a compromise function, defined as

$$\Phi_1 = \sum (y_i - C_{ji})^2 / y_i \qquad (5.50)$$

was employed, which did not jeopardise the fit to the initial data points, but significantly improved the sensitivity to the value of K_2V. This is illustrated by smaller confidence limits, and is discussed in section 5.5.4.

In most cases, a good fit was obtained, and visual inspection showed little difference in the quality of fit whether Φ_o or Φ_1 was employed. As expected, the use of Φ_1 gave a better fit to the data points at longer times. It was found, however, that the 'best values' of the parameters were affected by the choice of Φ_1 or Φ_o as shown in Table 5.20.

From Table 5.20 it is evident that values of K_2V are sensitive to the criterion of best fit. This will be elaborated on when confidence limits are discussed. (section 5.5.4).

5.5.2.2. Correction for transient flow conditions.

Parameter estimation was complicated somewhat by the tansient flow conditions in the initial stages of each pilot plant experiment. The model assumes plug flow through the bagasse bad, which implies that at $\theta = 0$, i.e. $t = \tau$ (the mean residence time), the percolating liquid

Table 5.20

Effect of Criterion of Best Fit on Parameter Values.

Run No.	Preparation	Minimising ϕ_1					Minimising ϕ_o				
		K_1V	K_2V	α	ϕ_o	ϕ_1	K_1V	K_2V	α	ϕ_o	ϕ_1
C12	P1	28.5	2.64	0.33	0.042	0.068	28.6	2.82	0.33	0.042	0.070
E14	P1	38.4	4.01	0.49	0.155	0.087	38.2	3.39	0.52	0.147	0.100
D34	P2	29.6	1.58	0.60	0.159	0.075	29.6	1.07	0.64	0.148	0.098
C25	P3	36.7	1.71	0.63	0.210	0.258	37.3	2.74	0.58	0.155	0.325
E3	P3	24.8	2.73	0.51	0.099	0.081	24.7	3.55	0.46	0.086	0.104

appears at the bottom of the bed, the outlet flow rate immediately assuming a value equal to the inlet flow rate. Practically, this situation is not realized. The outlet flow rate builds up gradually to its final value as shown in Fig. 5.34. These transient flow conditions are associated with a sinking and consolidation of the bagasse bed, and a build-up of liquid holdup within the bed. Thus the starting time is not physically well defined.

Clearly, two approaches are possible. Firstly, a more sophisticated model of the transient conditions could be developed, or secondly, the simplified plug flow model could be used as an approximation to the real situation. The former approach is unattractive since an accurate representation of the transient conditions would result in a mathematically intractable function, which would yield no additional useful information.

Initially, the effect of the starting time on the parameter values was investigated. The starting time is expressed as a correction, t_c, to be subtracted from recorded times, t', having as an arbitrary zero, the time at which the first liquid appears at the bottom of the diffuser. (see Fig. 5.34.). Table 5.21 shows that the choice of starting time has a significant effect on parameter values, so that care should be taken in assigning a value to t_c.

Three approaches were investigated. Firstly, it was hoped that the sensitivity to starting time would be overcome by utilising the slope of the experimental points near $t' = 0$. If equation (3.26) is rearranged and derivatives with respect to θ are taken, it can be shown that, for $\theta = O$:

$$\frac{\delta}{\delta t}\left[\ln\left(1 - \frac{C_j}{C_{bo}}\right)\right] = \frac{(K_1 V)^2}{HF}\left(\frac{1}{\alpha} + \frac{\beta^2}{1-\alpha}\right) \qquad (5.51)$$

Thus $\ln (1 - C_j/C_{bo})$ was plotted against time, and it was found that in a number of cases, a straight line could be drawn through the first four or so experimental points. The slope of this line yields an experimentally determined value of the left hand side of equation (5.51), which was designated SLOPE. Substituting

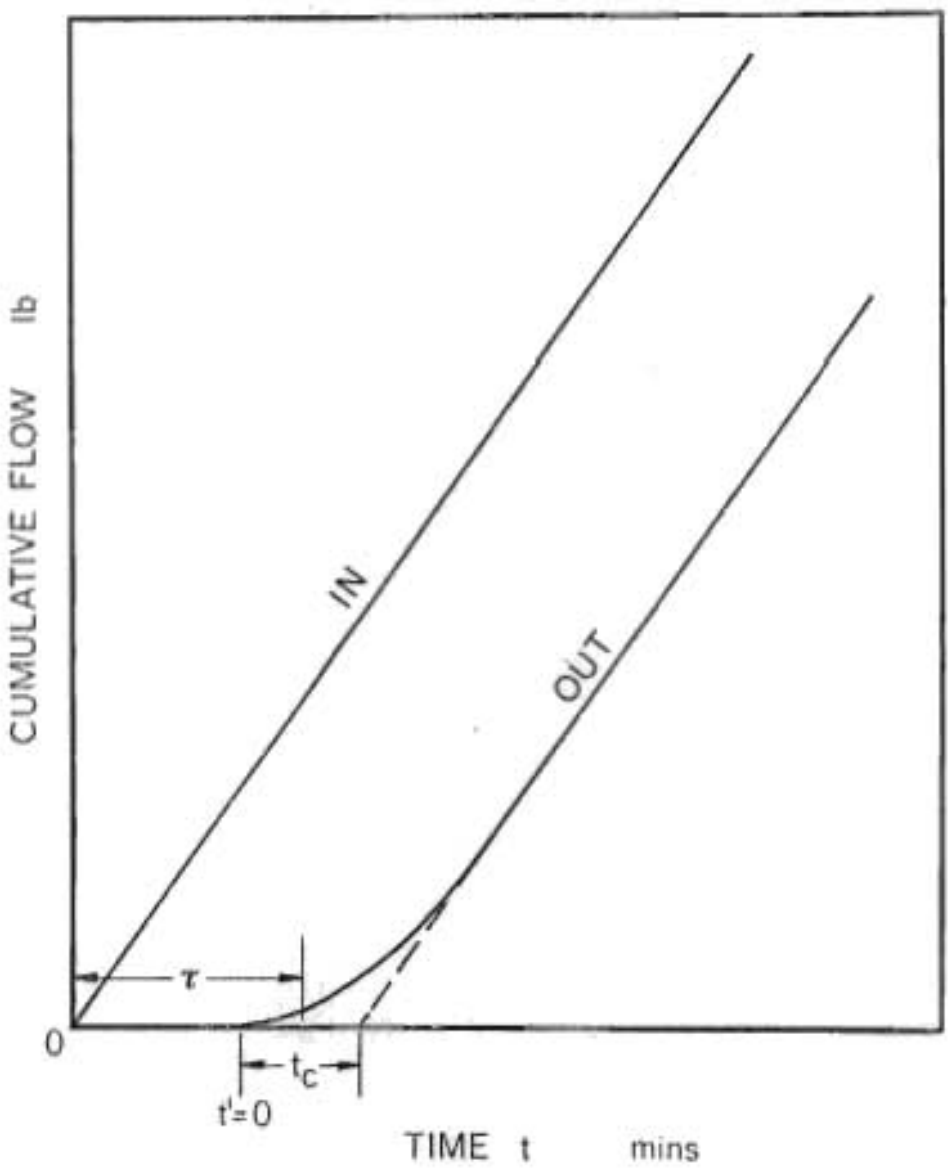


Fig. 5.34. Schematic representation of transient flow condition in the pilot plant diffuser. Value of t_c defined by equation (5.54).

Table 5.21. Effect of starting time correction on model parameter values

Run No.	Preparation	L (lb/min. ft^2)	t_c	K_1V	K_2V	α	ϕ_1	t_c	K_1V	K_2V	α	ϕ_1
C12	P1	58.6	0.47	28.5	2.64	0.33	0.068	0.17	36.7	2.96	0.40	0.080
D44	P1	43.6	0.75	38.2	2.14	0.55	0.091	0.19	49.6	3.36	0.66	0.094
D34	P2	43.6	0.68	29.6	1.58	0.60	0.075	0.40	32.5	1.73	0.66	0.098
C25	P3	31.2	0.83	36.7	1.71	0.63	0.258	0.64	40.9	2.44	0.68	0.239
D 54	P4	22.2	1.32	29.0	2.33	0.79	0.165	0.60	31.4	4.54	0.79	1.145

$$\text{SLOPE} = \frac{\delta}{\delta t}\left[\ln\left(1-\frac{C_i}{C_{bo}}\right)\right] \qquad (5.52)$$

in equation (5.51) and rearranging leads to :-

$$K_1V = \sqrt{\alpha HF(\text{SLOPE}) - \frac{\alpha}{1-\alpha}(K_2V)^2} \qquad (5.53)$$

Thus for any given values of SLOPE , H and F (from experiment), K_1V is completely determined by values of K_2V and α . The starting time is most likely to affect values of K_1V which have an important effect at the beginning of a run when the washing-displacement process is operative. It was anticipated therefore that the use of equation (5.53) would to a large extent free the parameter values from this starting time dependency. However, although this sensitivity was reduced it was still significant.

A modification to this method was made, whereby the starting time was introduced as an additional variable. Thus K_2V, α and t_c were varied to obtain the best fit of the model to the data, with K_1V determined by equation (5.53). This procedure worked satisfactorily, but was eventually abandoned, since a reliable value of SLOPE could only be obtained for less than half the experimental runs. Even small discontinuities in experimental data at small values of t' (e.g., see curve A, Fig. 5.35) precluded a reliable estimate of the slope in these cases.

The second approach employed was to introduce t_c as an additional parameter, thus requiring the evaluation of four parameters. A similar procedure was followed by Draper et.al. (1969), who introduced an extra parameter in the form of a time lag, in the estimation of reaction rate constants. The time lag was introduced to represent unknown time lag between the actual starting time of a reaction and a chosen arbitrary origin.

This 'extra parameter method' was regarded as unsatisfactory for a number of reasons. Firstly, t_c has no physical significance, and the introduction of

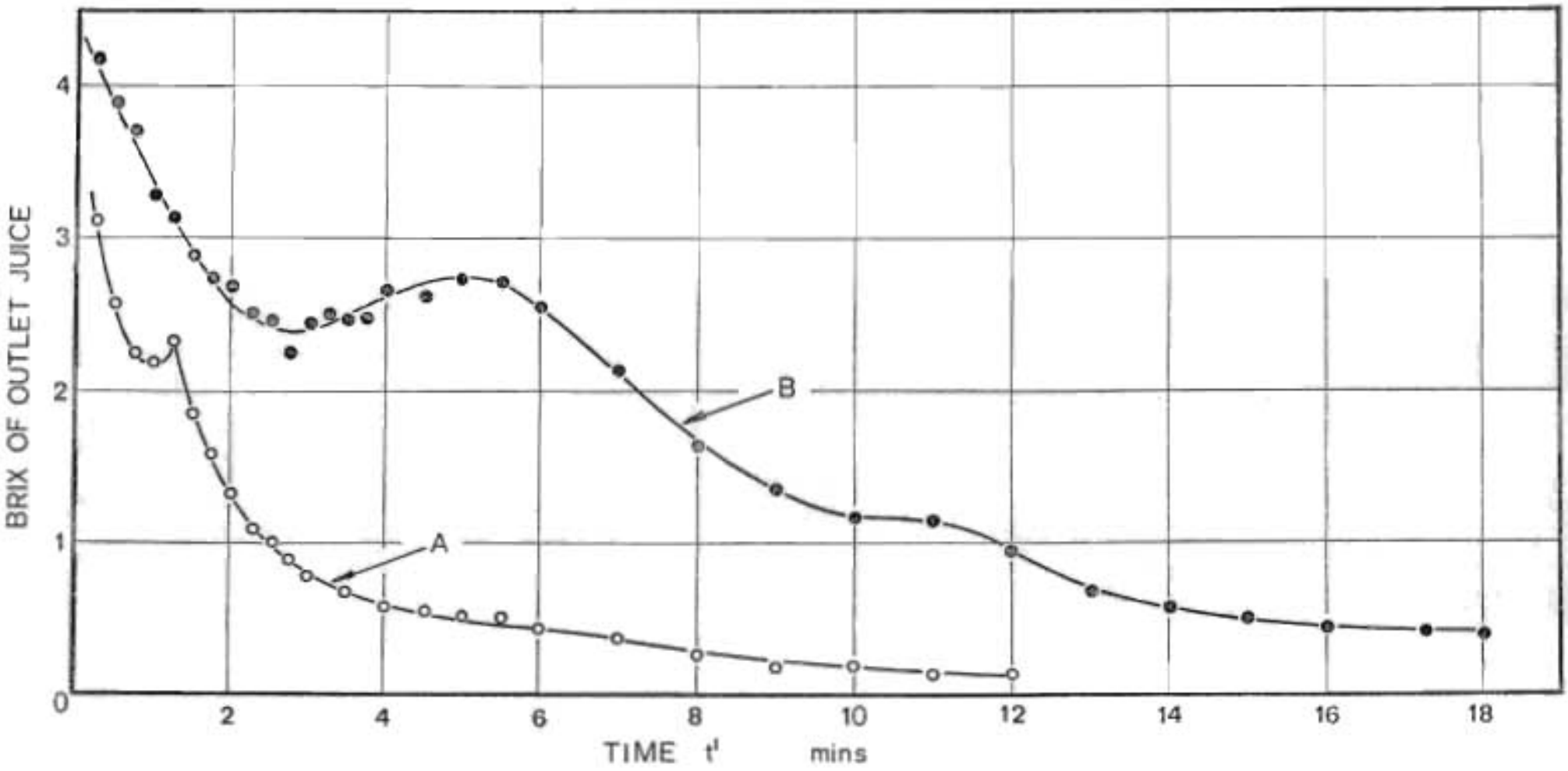


Fig. 5.35 Illustration of erratic behaviour of some experimental data.
(Note: solid lines drawn in by eye do not represent fitted curves).
Curve A : run C35, P2, $L = 48.1$ lb/min. ft^2. Curve B : run D15, P3,
$L = 18.2$ lb/min. ft^2.

an additional parameter must necessarily improve the fit, but may lead to overparameterisation with a consequent loss in physical significance of the model parameters. Secondly, values of t_c obtained sometimes displayed erratic behaviour, differing widely from values obtained in the preceeding approach using the initial slope. Thirdly, the function minimised, ϕ_1/n (where n=number of data points) involved discontinuities as t_c changed sufficiently to include or discard an additional data point. This seriously jeopardised the rate of convergence, and was not evident in the 'initial slope' procedure.

The approach ultimately used involved the choice of starting time defined by the physical flow conditions. In this case, t_c is defined by the relation :

$$F(t'-t_c) = \int_0^{t'} F_o dt \,, \quad t' > t_c \qquad (5.54).$$

where F_o is the outlet flow rate.

According to this definition, t_c is easily obtained by extrapolating the cumulative outflow curve to zero, where the intercept with the t-axis yields the value of t_c. This is illustrated schematically in Fig. 5.34 . The difference between the corrected starting time ($t'=t_c$) and the plug flow starting time ($t=\tau$) represents the time required for the build up of static liquid holdup (see section 4.2.4.1)

This method was also applied by Kozicki et. al. (1970) to constant-rate filtration studies. A time correction factor defined in the same way was introduced to account for the varying filtration rate in the initial period during cake stabilisation. They found that the use of such a time correction factor significantly improved the reproducibility of cake parameter values.

Apart from the fact that this approach attaches a physical significance to t_c , the values of t_c obtained in this way agreed fairly well with those obtained from the initial slope procedure.

5.5.3 Fit of the model to Pilot Plant Data.

Observed and predicted values of C_j are shown in Figs. 5.32 and 5.33 ; the solid lines represent model predictions. The figures show only a limited number of runs selected at random, but are sufficient to demonstrate the very satisfactory agreement between the model and experimental data. Resolution on the precision refractometer used for brix measurement was 0.04, although readings could be estimated to within 0.02.

In some cases, the experimental points showed an erratic trend (for instance, curve B, Fig. 5.35). It is obvious that the form of the model is incapable of describing such data. The results of this particular run represent the most extreme case. Nevertheless, similar though less severely abnormal behaviour occured, particularly with finer preparations, at low flow rates, and generally when higher temperatures were employed. This is presumed to be due to the consolidation of the bagasse bed, which would lead to a random establishment of new flow paths and blocking off of other flow paths. This is expected to be more serious at low flow rates where wetting efficiency is low and the total number of flow paths is small (Davidson, 1959).

On occasions, high values were obtained for $\phi_{1\ min.}$ (the minimum value of ϕ_1), which were found to be due exclusively to erratic data. Such data, and the corresponding parameter values were rejected. As a general rule, where $\phi_{1\ min}$ was >0.6, the results were rejected, and for $\phi_{1\ min} < 0.45$, all results were utilized. Runs with intermediate values of $\phi_{1\ min}$ were accepted if the model predictions and the data showed the same average trends (by inspection). Otherwise, the results were rejected, as it was felt that the parameters were not representative of stabilised operation, which might lead to unnecessary scatter in parameter correlations.

The number of runs rejected is shown in Table 5.22. Minor discontinuities (e.g. curve A, Fig. 5.35) did not jeopardise the fit of the model to the data.

Table 5.22

Number of runs rejected due to erratic behaviour of experimental data.

Preparation.	Total No. of runs processed.	No. of runs rejected at 59°C	73°C	87°C
P1	74	0	0	0
P2	21	1	0	1
P3	33	0	8	2
P4	16	1	4	2

5.5.4 Confidence limits on parameter values.

An estimate of the accuracy with which the parameters are determined is necessary if it is envisaged that these results be applied to full scale plant operation.

In the non-linear regression case, the normal methods of estimating confidence limits do not apply (Draper & Smith, 1966). In this case, approximate confidence regions can be defined as the region enclosed by ϕ_o, where

$$\phi_o = \phi_{o\,min} \left[1 + \frac{m}{n-m} F(m, n-m, 1-p) \right] \quad (5.55).$$

where n = number of observations, m= number of parameters. This provides approximate 100 (1-p) % confidence regions. The contour so determined is a proper correct confidence contour, and it is only the probability level which is approximate (Draper & Smith, 1966). Thus we may write

$$\phi_o / \phi_{o\,min.} = 1 + \frac{m}{n-m} F(m, n-m, 1-p) \quad (5.56)$$

Approximate 95% confidence regions were determined for a number of runs, assuming that equation (5.56) holds also for $\phi_1 / \phi_{1\,min.}$ The confidence regions in this case are 3-dimensional, and sectional drawings through $\phi_{1\,min}$ are

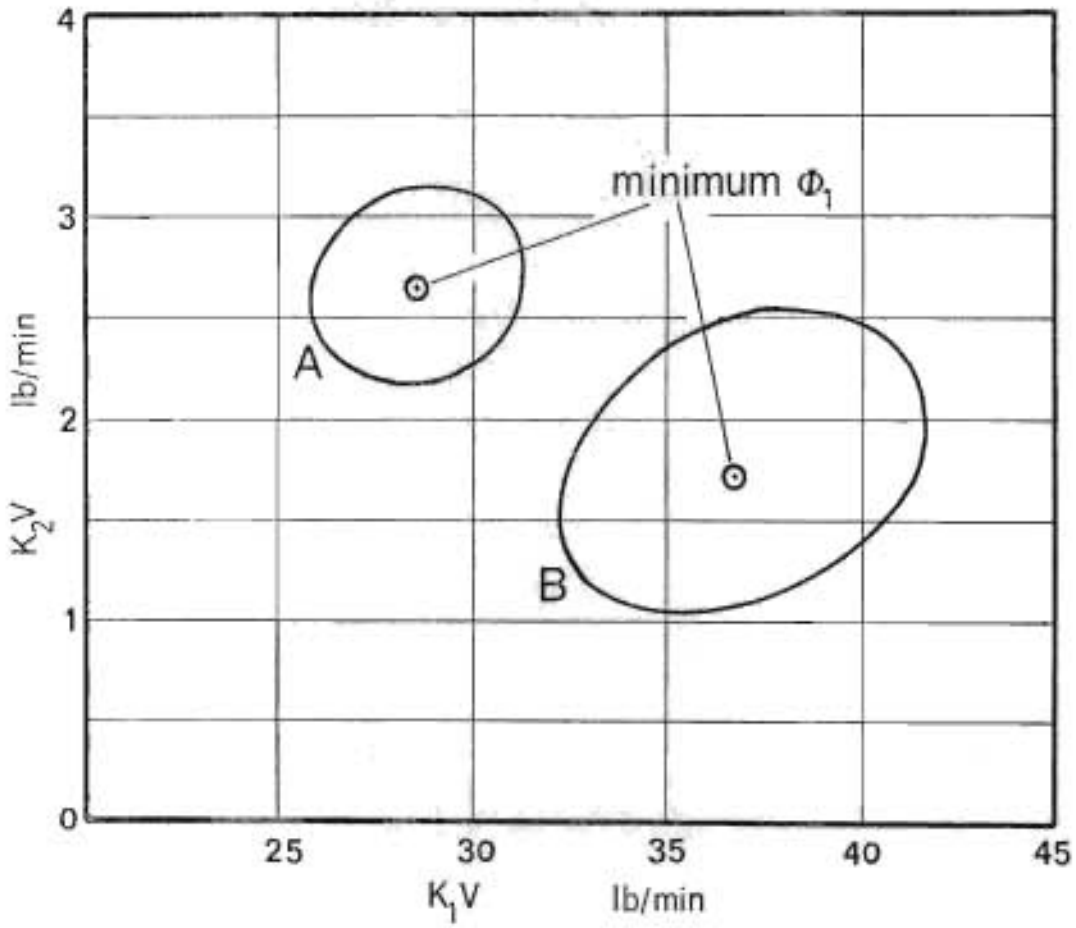


Fig. 5.36 95% confidence regions, K_1V - K_2V, A: run C12, P1, α = 0.333, Φ/Φ min. = 1.49. B: run C25, P3, α = 0.632, Φ/Φ min. = 1.47

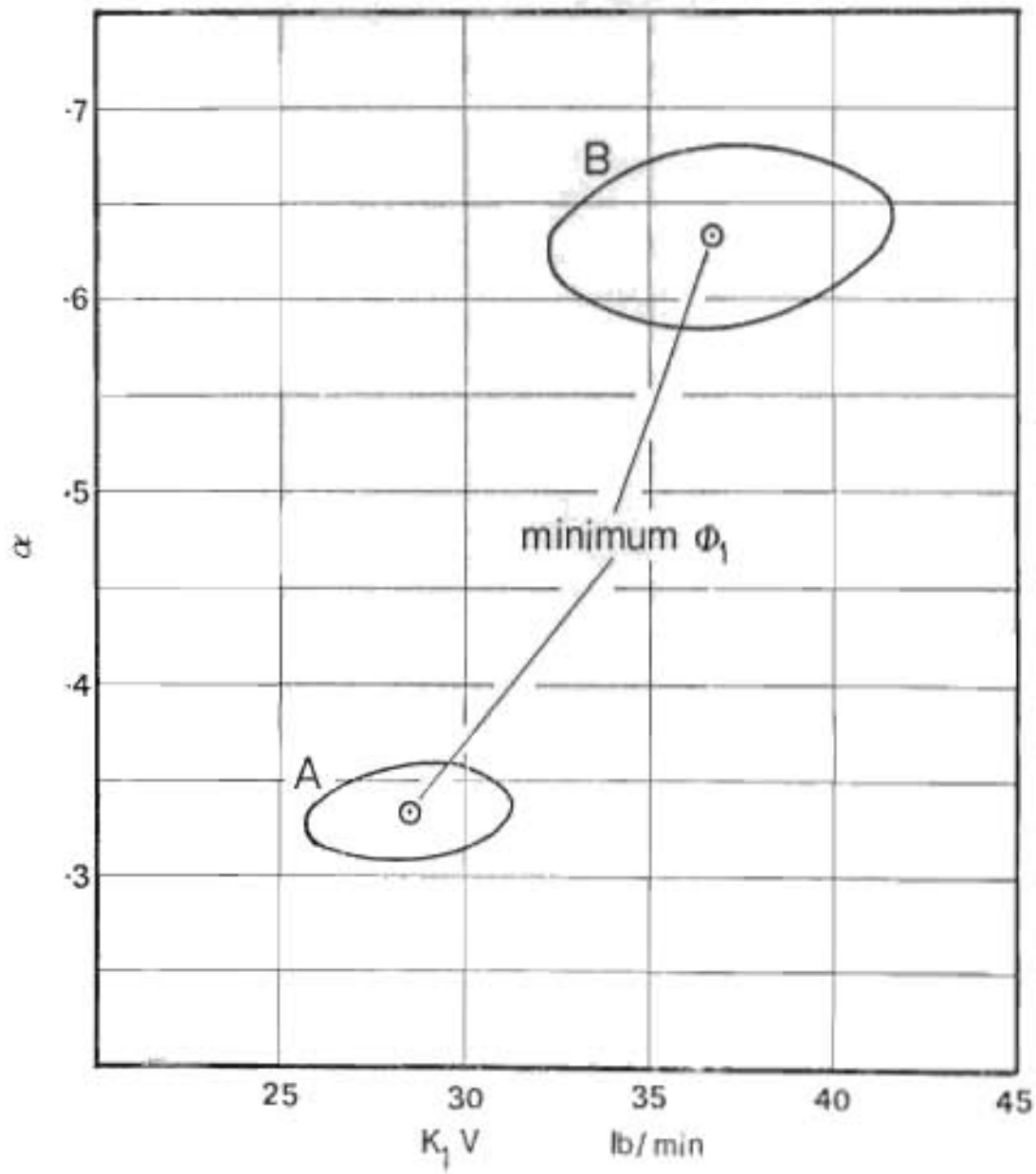


Fig. 5.37 95% confidence regions, K_1V - α .
A: run C12, P1, $K_2V = 2.64$.
B: run C25, P3, $K_2V = 1.71$.

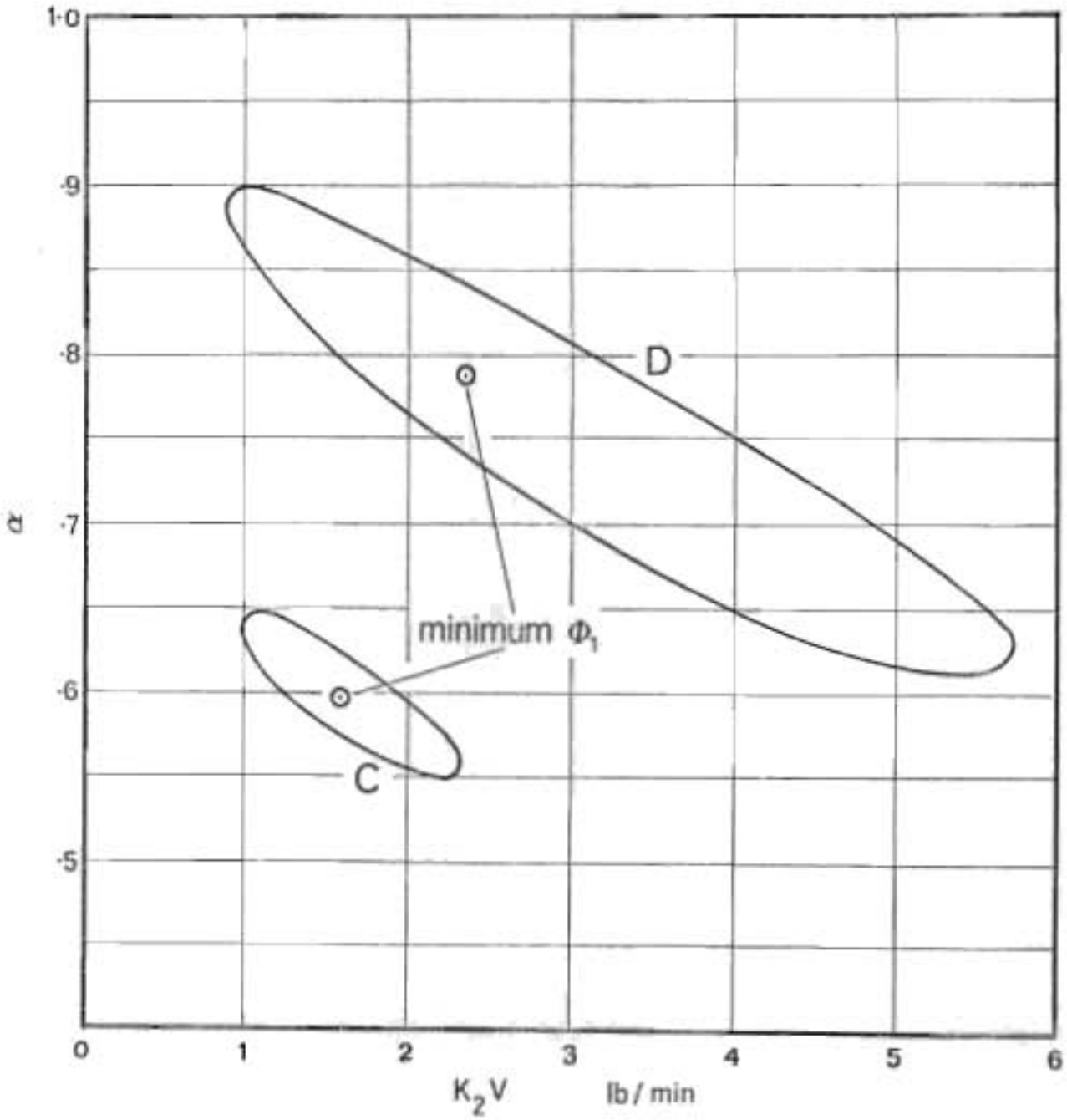


Fig. 5.30. 95% confidence regions, K_2V - α .
C: run D34, P2, K_1V = 29.6. ϕ/ϕ min. = 1.40. D: run D54, P4, K_1V = 29.0. ϕ/ϕ min. = 1.36.

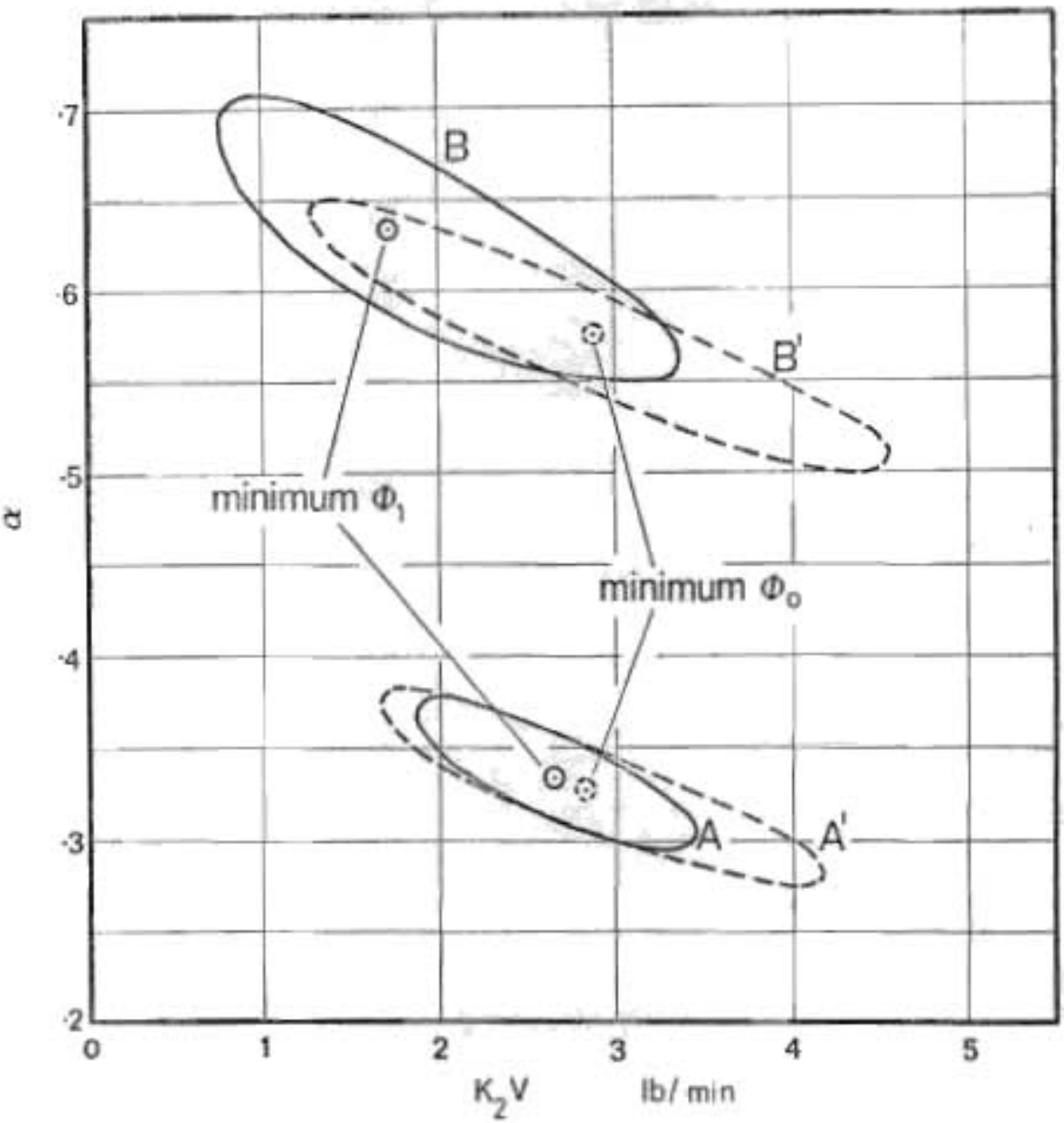


Fig. 5.39 Comparison of confidence regions obtained minimising Φ_0 and Φ_1. A: run C12, P1, minimising Φ_1, $K_1V = 28.5$. A':run C12, P1, minimising Φ_0, $K_1V = 28.6$. B: run C25, P3, minimising Φ_1, $K_1V = 36.7$. B': run C25, P3, minimising Φ_0, $K_1V = 37.3$

when the ellipses (or approximate ellipses) are obliquely orientated with the axes, a dependence between the two parameters is indicated (the parameters are correlated). The size of the contours indicates the relative precisions with which the parameters are determined.

Figs. 5.36 and 5.37 indicate that little or no correlation between K_1V and K_2V, or K_1V and α exists. However, it can be seen from Figs. 5.38 and 5.39 that K_2V and α are correlated, and that this degree of association is more pronounced with P3 and P4 in particular. It appears that with approximately 95% confidence, K_1V and α are generally determined to within approximately 10%, although this figure is greater with P4 preparation. K_2V is determined to within approximately 30% for P1 preparation, but it appears that this figure is much greater with fine preparations, particularly P4 again.

Fig. 5.39 shows the contour of ϕ_o as well as ϕ_1, calculated from equation (5.56). This shows clearly that the confidence region is much smaller for the function ϕ_1, so that ϕ_1 is far more sensitive than ϕ_o to the value of K_2V, which, as stated in section 5.5.2, is the reason for the use of ϕ_1. It can be seen that the use of ϕ_o results in the minimum moving down the valley in the α - K_2V plane; in any event, the minima predicted using ϕ_o will generally be well within the approximate 95% confidence limits determined using ϕ_1.

The ill-conditioned contours shown in Figs 5.38 and 5.39 did not significantly affect the rate of convergence, although in general, more iterations were required when processing P4 data. It was noted that the search procedure generally defined a search direction along the valley in the K_2V - α plane.

5.6 MODEL PARAMETER VALUES.

The laboratory-scale extraction tests, described in section 5.2, established the mechanisms whereby sucrose is extracted from bagasse. The results show that extraction can be considered to take place via two parallel mechanisms, firstly by a washing-displacement process, and secondly by molecular diffusion. These tests were carried out under conditions of good liquid-solid contact; however, in a packed bed environment, it is logical to expect lower contact efficiencies, which would be dependent on the flow conditions within the bed, and particularly on the degree and type of wetting of bagasse particles. This can be expected to influence the rate of extraction, manifesting itself in the values of the model parameters.

The mass transfer system in a diffuser differs slightly from those packed bed systems which have received attention in the literature. Under non-flooding conditions, film-flow, or trickle flow exists, which is similar to the liquid-phase behaviour in gas absorption operations. However, the emphasis in gas absorption studies is directed towards the behaviour of the liquid-gas interface, while this extraction process relates to liquid-solid mass transfer, rather than liquid-gas transfer. Under so-called flooding conditions, it was established in section 5.4.1 that a considerable amount of trapped air is occluded within the bed. Thus this system cannot be treated as a continuous fluid flow system; otherwise it might be expected that previous work on continuous liquid or gas flow through packed beds may have relevance in this case. Rather, flooding in a bagasse bed should be considered as an extension of the film-flow regime. In making comparisons with the results of other packed bed operations, these differences should be borne in mind.

Theoretical studies on mass transfer have generally been made on the assumption of a particular flow condition (usually laminar flow). Such work has utility in indicating the functional relationships between the mass transfer rate and the flow conditions and fluid properties. However, a more realistic picture of the flow pattern may

be envisaged as a network of regions of laminar flow, turbulent flow, and stagnant fluid zones. In interpreting the behaviour of the model parameters, two mass transfer coefficients have to be considered. The degree of turbulence may be expected to influence the value of K_1, while stagnant liquid zones are likely to affect K_2, which represents extraction by a diffusional mechanism.

All model parameter values are listed in Appendix C. The pilot plant experiments covered a wide range of operating variables; this is shown in Table 5.23.

Table 5.23. Range of Experimental Variables.

Flow rate	16.2 - 95.8 lb/min ft^2
Temperature	56 - 90^{o}C
DI	58.5 - 93.8
Specific surface	2610 - 9080 mm^2/g
Bed height	1.62 - 3.80 ft
Fibre density	3.8 - 5.0 lb/ft^3

In the following paragraphs, the values and behaviour of the model parameters are discussed.

5.6.1 α

It was shown in section 5.2 that, in the laboratory extraction tests, α is a linear function of DI, and represents the fraction of juice which can be easily extracted. The pilot plant results likewise show higher values of α for finer preparations, but Fig. 5.40 shows α is a function of liquid flow rate as well. The scatter is expected, since it was pointed out in section 5.1 that the same method of preparation applied to different cane can yield widely differing bagasse.

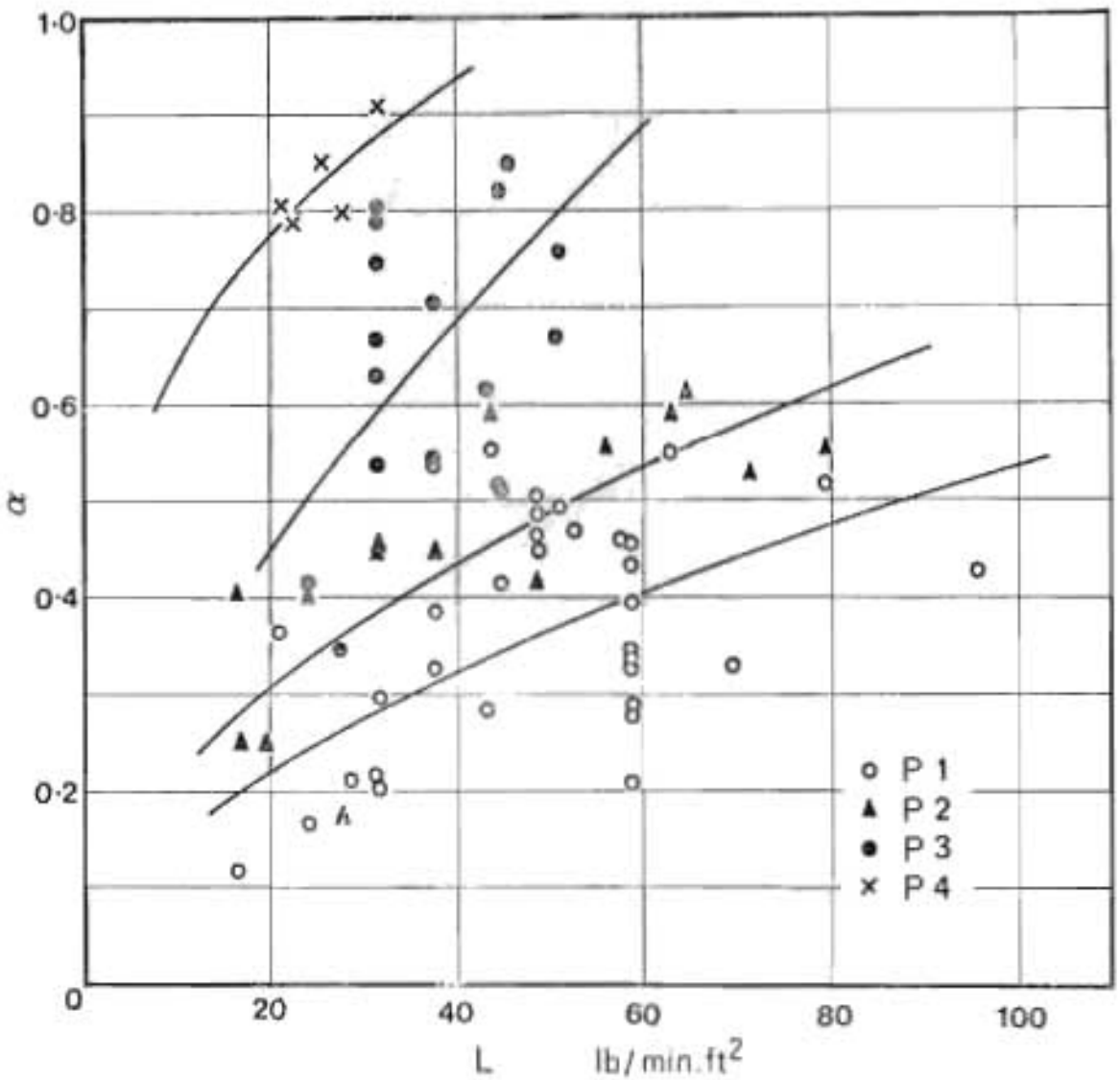


Fig. 5.40 Values of α as a function of liquid flow rate. Data shown represent operation at 73°C, with an initial bagasse load of 125 lb.

Also shown in the figure are the best correlations, for each preparation, of the form

$$\alpha = A L^n \qquad (5.57)$$

The levels of significance and values of A and n are shown in Table 5.24. This includes only data obtained at 73°C, with an initial bagasse load of 125 lb. in the diffusion vessel.

Table 5.24 Regression Coefficients for Equation (5.57).

Preparation	A	n	Correlation Coefficient	Significance level p
P1	0.0412	0.557	0.556	<.01
P2	0.0701	0.496	0.711	<.01
P3	0.0700	0.619	0.559	<.05
P4	0.358	0.259	0.723	>.1

This table illustrates that the flow rate dependence is significant for preparations P1, P2 and P3, while this dependence is not established for preparation P4, due primarily to the small number of data points, caused by difficulties in parameter estimation for P4 data. Scatter in the values of α is particularly pronounced for finer preparations. The lower degree of confidence in these values for P3 and P4 in particular (see section 5.5.4) presumably is reponsible to some extent.

Generally, the values of α obtained in the laboratory-scale tests α_M, are approached only at high flow rates in the pilot plant. A comparison between values of α_M obtained from the laboratory mixing tests and the values predicted from equations (5.57) for each preparation at limiting flow rates is shown in Table 5.25.

Table 5.25. A comparison between values of a obtained in laboratory tests and pilot plant runs at high flow rates.

Preparation	DI		Value of a	
	Mean	Std. Dev.	Lab. (a_M)	Pilot Plant
P1	64.1	2.4	0.54	0.52
P2	71.9	2.8	0.60	0.60
P3	84.6	2.7	0.70	0.79
P4	91.6	2.6	0.75	0.86

The lower values of a obtained at lower flow rates are indicative of less efficient liquid-solid contact at low flow rates. Only at high flow rates is a comparable degree of wetting obtained. With preparations P1 and P2, a_M values are approached only at the highest flow rates. With preparations P3 and P4, the pilot plant values are higher at high flow rates; however, as pointed out above, this data is the least reliable. These results confirm the anticipated dependence on liquid flow conditions.

The ratio, a / a_M, can be regarded as a measure of the efficiency of wetting. Since a_M is directly related to DI (equation 5.17), a / DI should likewise be a measure of the efficiency of wetting, but relates a to the directly measurable quantity, DI, rather than the derived quantity, a_M. At a given flow rate, values of a/DI are higher for finer preparations; this is consistent with the observations of Mayo et al (1935) that smaller packings are more effectively wetted.

5.6.1.1 Correlation of data.

Values of a obtained from runs at different bed heights or at temperatures other than 73°C diverge from the results shown in Fig. 5.40. In order to correlate this data, it is necessary to consider the significance of a in greater depth.

The parameter a relates to the quantity of sucrose which can be readily extracted. Since this generally constitutes sucrose in surface juice which is readily accessible, a should relate to an effective wetted surface area.

In other mass transfer operations in packed beds, two different mass transfer areas have been investigated, the total wetted area, and the effective interfacial area in gas absorption (see section 2.5.2). However, the area of interest in this case is likely to be different from the total wetted area, since liquid retained in small capillary passages between particles is effective in wetting particle surfaces, but is not exposed directly to the flowing liquid. Thus static liquid holdup may well decrease the mass transfer area which is effectively contacted by the dynamic liquid phase. On the other hand, the effective interfacial area in gas absorption refers to a liquid-gas interface rather than a liquid-solid interface, but does exclude static liquid which is ineffective in the transfer process (Shulman et al, 1963).

It appears that the dependency of both the total wetted area a_w and the effective interfacial area a_e on liquid flow rate is similar. Semmelbauer (1967) has shown that a_e is proportional to $L^{0.455}$, while Onda et al (1955) summarized the results of a number of experimental investigations which show that reported values of the exponent on L in the case of a_w vary over a wide range, with a mean of roughly 0.55. However, values of a_w/a_T increase with smaller packings, while values a_e/a_T show the opposite trend (Davidson, 1959).

In order to correlate values of a/DI, investigations into a_w/a_T and a_e/a_T furnish a guide as to the significant variables to be used for correlation purposes in this case. The following relationship is indicated:

$$\frac{a}{DI} = f(L, \mu, \rho, g, a_T) \qquad (5.58)$$

Surface tension is generally included as a variable in correlating effective interfacial areas (Semmelbauer, 1967). This is likely

to be more significant when a liquid-gas interface is considered than a liquid-solid interface. In any event, the surface tension of sugar cane juices is extremely variable, depending on the nature and amount of non-sucrose components present (VanHook & Biggins, 1952), and so surface tension cannot in any event be estimated with any degree of confidence.

Dimensional analysis applied to equation (5.58) yields:

$$\frac{a}{DI} = f(Re, Ga) \qquad (5.59)$$

Multilinear regression analysis (described in Appendix F) of all the a/DI values yielded the following relation:

$$\frac{a}{DI} = 0.318\ Re_l^{0.576}\ Ga_l^{-0.397} \qquad (5.60)$$

with a standard deviation of 0.169. The value of the exponent on Re_l is consistent with those reported for a_w/a_T. Moreover, the dependence of a/DI on packing size is consistent with that of a_w/a_T on packing size, and so a/DI probably approximates more closely to a_w/a_T than to a_e/a_T. Fig. 5.41 shows a comparison of the correlation with experimental data.

It should be noted that the correlation for dynamic liquid holdup shown in equation (5.29) is of the same form as equation (5.60). In fact, a strong numerical correspondence between the exponents in these 2 equations exists. From this it may be inferred that a/DI and dynamic holdup are measures of the same physical quantity. In considering the rates of gas absorption and vaporisation in packed columns, Shulman et al (1963) found that the effective transfer area for each type of process is proportional to the holdup of liquid active for that operation. This suggests the use of a phenomenological correlation for a in terms of dynamic liquid holdup.

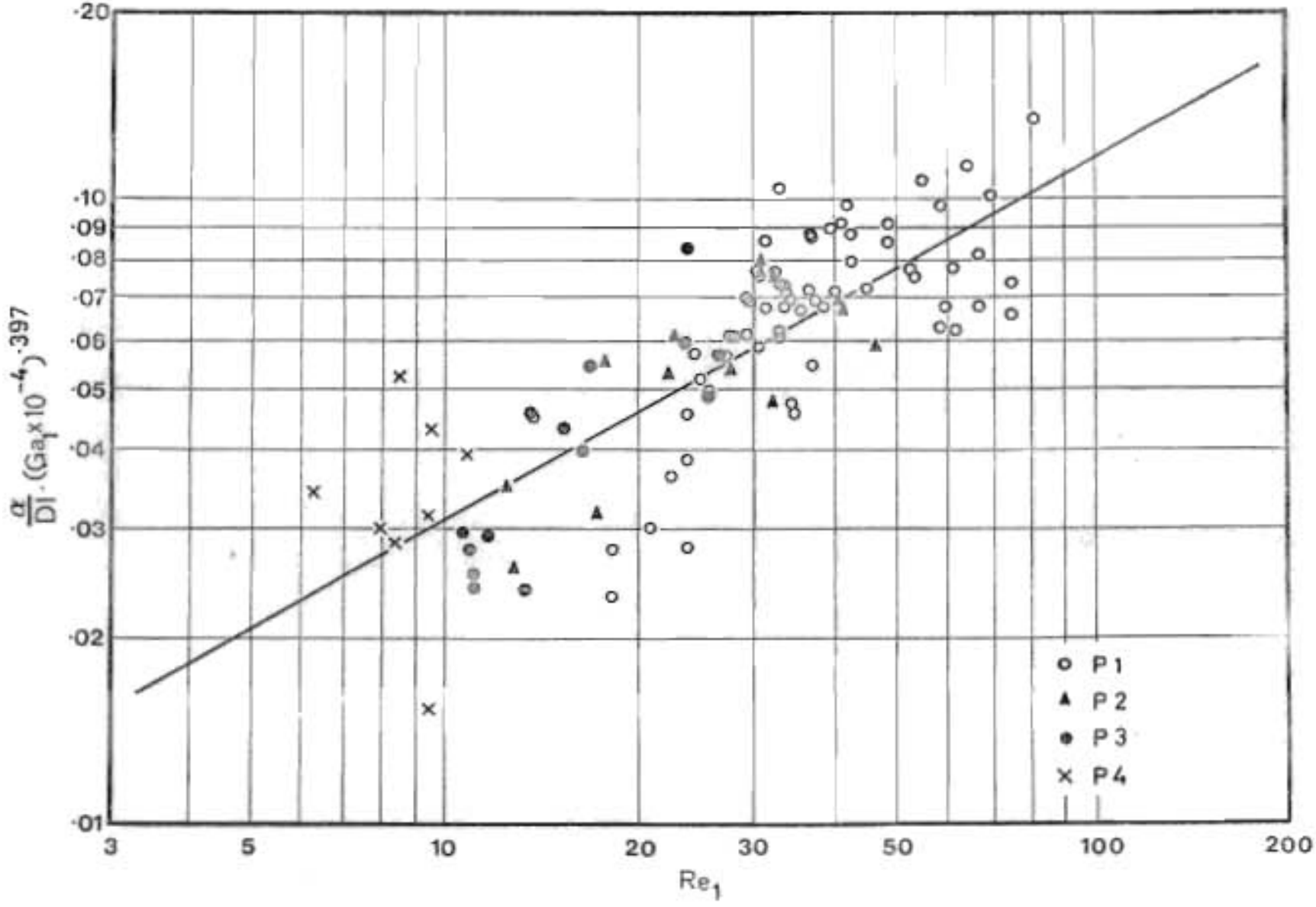


Fig. 5.41 Comparison of dimensionless correlation for α/DI, equation (5.60), with experimental data.

5.6.1.2 Correlation in terms of dynamic holdup.

Values of a and a/DI were plotted against H_D, and are shown in Figs. 5.42 and 5.43. Only data obtained at 73°C is shown; the solid lines represent the following correlations:

$$a = 0.130\, H_D - 0.226 \tag{5.61}$$

$$\frac{a}{DI} = 0.01559 \log H_D - 0.00467 \tag{5.62}$$

Correlation coefficients of 0.75 and 0.69 respectively imply a significance level in both cases of $p < 0.001$ (Volk, 1958), with standard deviations of 0.121 and 0.140 respectively. A semi-log relationship is shown in equation (5.62) only because it appears to give a slightly better correlation than a linear or log-log relationship.

It is evident that data for all types of preparation are well correlated in terms of H_D. In Fig. 5.42 the effect of preparation is included implicitly in H_D.

Since H_D represents the liquid actually flowing through the bagasse bed, it appears that a depends on the degree of active or dynamic wetting, as distinct from static wetting.

This method of correlation effectively accounts for the varying bed height data, which are included in these 2 figures. Values of a were generally found to be higher for higher bed heights. This corresponds with generally higher values of H_D, and so this behaviour is consistent with the above results.

5.6.1.3 Effect of temperature.

The correlation in terms of Re_l and Ga_l was obtained for all the data, including runs at different temperatures. However the data in Figs 5.42 and 5.43 represent runs carried out at 73°C only. Data obtained at other temperatures show a significant deviation from the correlations in terms of H_D. Fig. 5.44 shows a series of data obtained at the same flow rate but different temperatures, with P1 preparation. Values of a are higher at higher temperatures, although H_D is apparently independent of

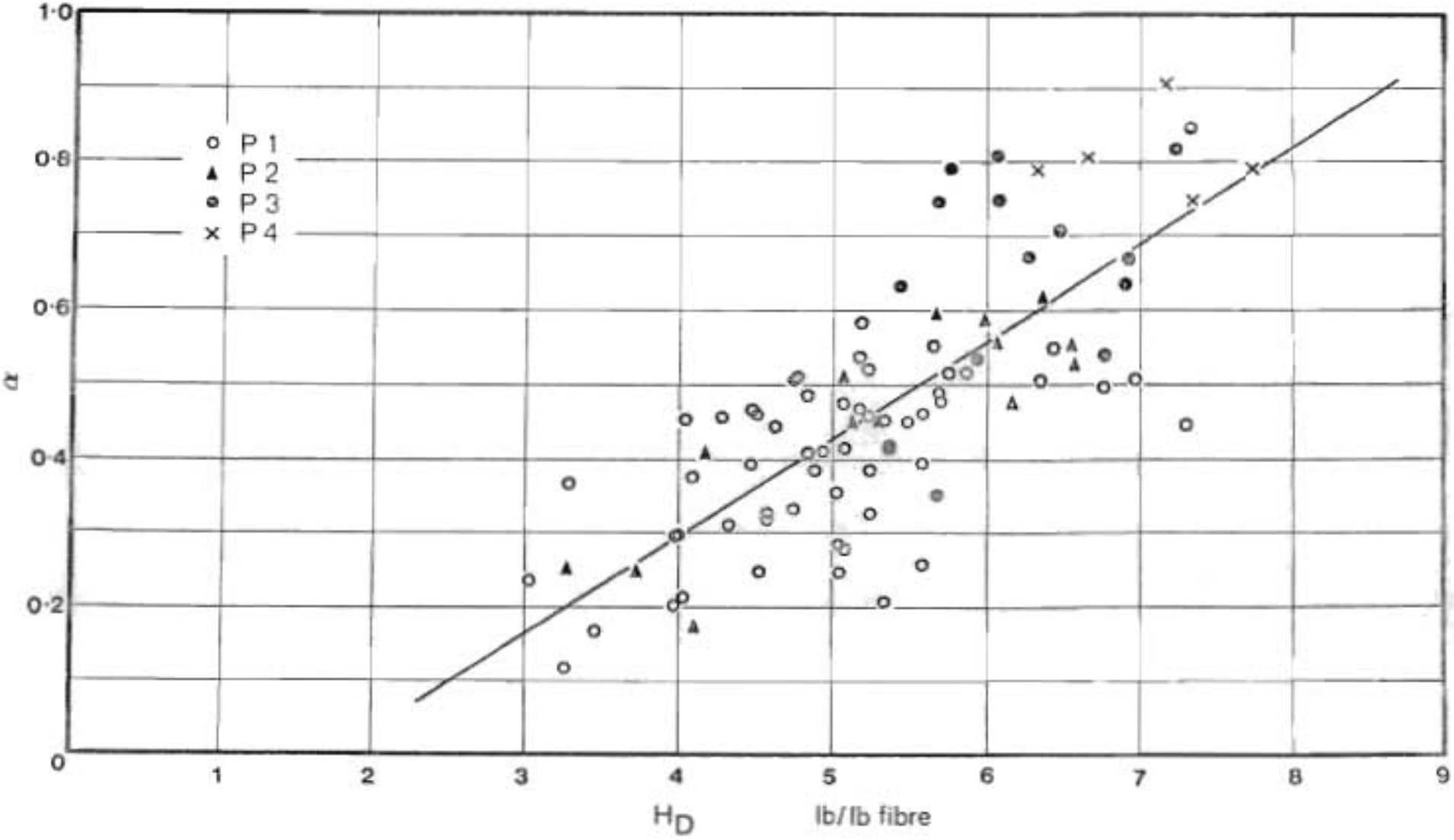


Fig. 5.42 Correlation of values of α in terms of dynamic liquid holdup. Data points represent all data obtained at 73°C.

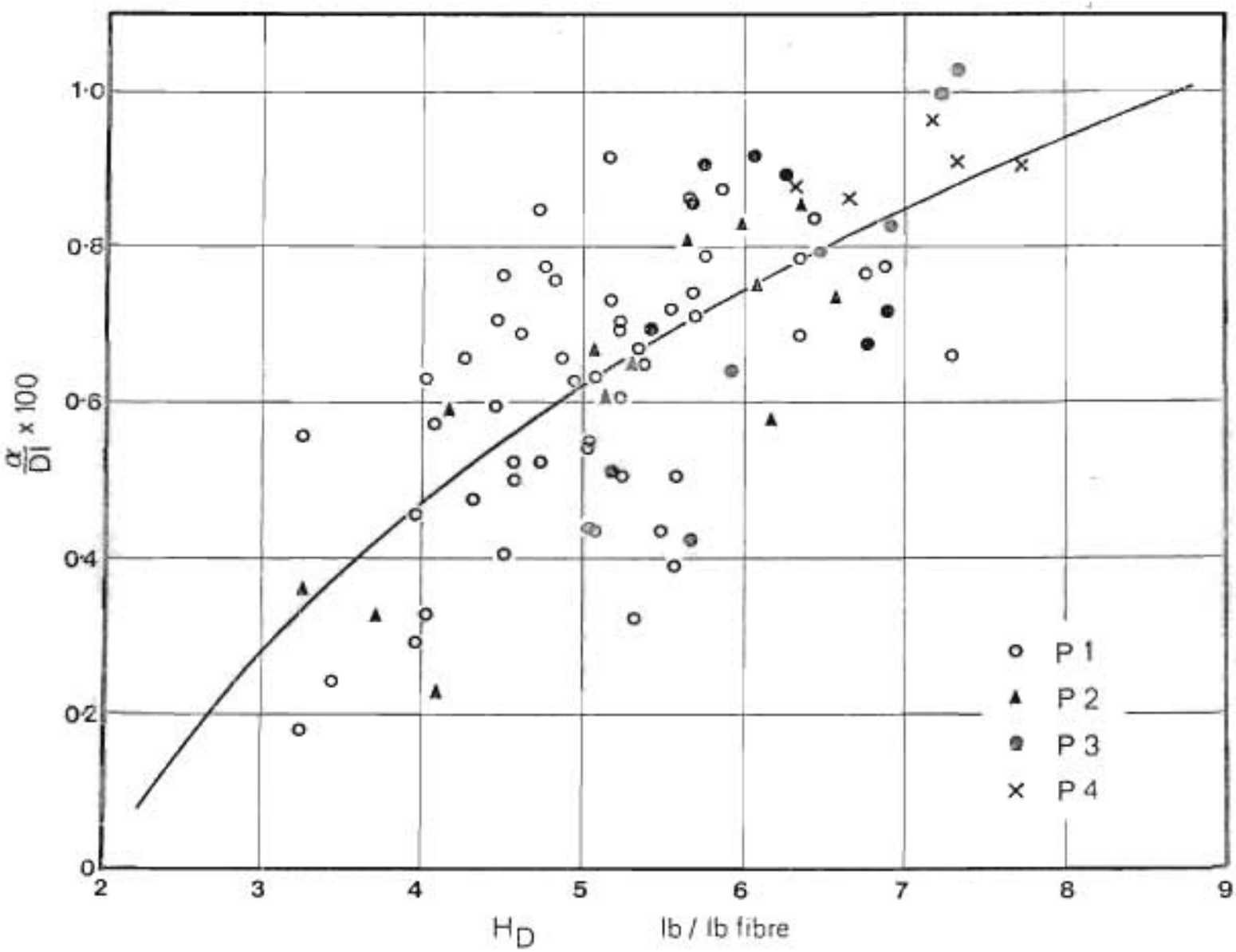


Fig. 5.43 Correlation of values of α / DI in terms of dynamic liquid holdup. Data points represent all data obtained at 73°C.

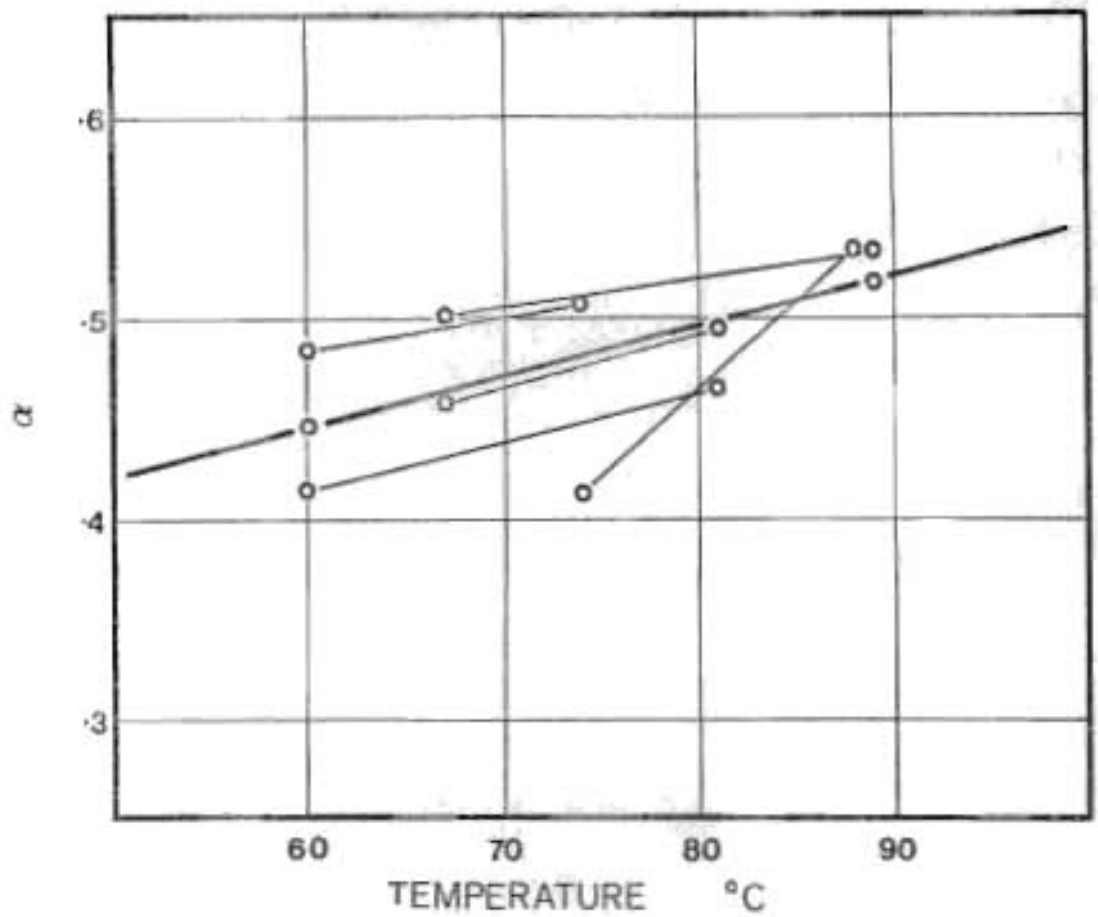


Fig. 5.44 Effect of temperature on values of α. Data points represent operation at L = 44.6 lb/min. ft^2, bagasse preparation P1. Solid lines connect data points obtained using subsamples of the same bagasse.

temperature. It is likely therefore that at different temperatures, the differerences in fluid properties induce changes in the liquid flow.

Liquid holdup can be considered as the product of a wetted area, and a mean film thickness x_f (Davidson, 1959). Since it is the wetted area that determines the value of a, it is of interest to consider how x_f and the wetted area vary with temperature.

Semmelbauer (1967) introduced an 'effective film thickness', δ which is defined as:

$$\delta = \left(\frac{\mu^2}{g \rho^2} \right)^{1/3} \qquad (5.63)$$

This quantity has the dimensions of length, and has been used as a length parameter by van Krevelen & Hoftijzer (1948) and Onda et al (1959) in correlating mass transfer data. The quantity x_f / δ is known as the Nusselt film thickness (Catchpole & Fulford, 1966), and is equivalent to $Re^{1/3} x$ (gravity force / viscous force). Thus it is postulated that δ determines the variation in film thickness, with temperature. At higher temperatures, the ratio of gravity / viscous forces is greater (lower δ), leading to a thinner film and greater spreading of the liquid. Thus the relationship between H_D and x_f changes, so that for the same value of H_D, x_f is lower and the wetted area is greater at higher temperatures. Davidson (1959) also noted that the degree of wetting is influenced by the liquid viscosity, being lower the higher the liquid viscosity.

An attempt was made to allow for the change in liquid spreading characteristics at various temperatures by incorporating δ as a correction factor for H_D. Values of a at a given temperature, T, were plotted against $H_D \cdot x$ $\delta 73 / \delta T$, and are shown in Fig. 5.45. The solid lines connect data obtained using subsamples of the same bagasse, at the same flow rates but different temperatures. (P1 data are shown only, for the purposes of clarity).

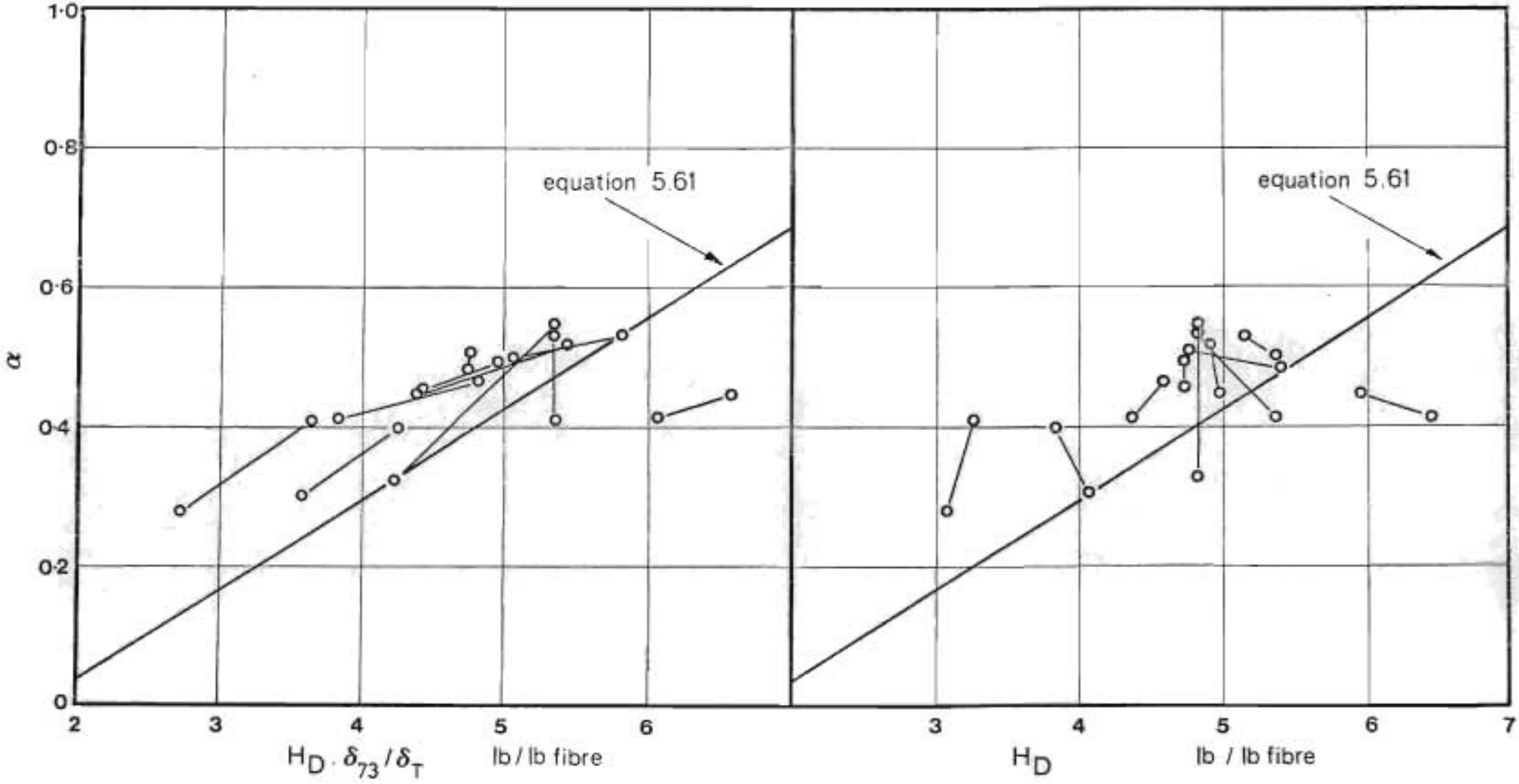


Fig. 5.45 Values of α obtained at different temperatures as a function of H_D and $H_D \cdot \delta_{73}/\delta_T$. The trends shown by the pairs of sample points should be compared in each case with the 73°C correlation, equation (5.61).

Also shown are the data plotted against H_D only. Clearly the trends shown by the data with the δ correction to H_D correspond more closely to the trend of the 73°C correlation, equation (5.61)

The data for all temperatures was subjected to regression analysis. The best correlations for α and α/DI are:

$$\alpha = 0.101 (H_D \cdot \delta_{73} / \delta_T) - 0.051 \qquad (5.64)$$

$$\frac{\alpha}{DI} = 0.0125 \log (H_D \cdot \delta_{73} / \delta_T) - 0.00216 \qquad (5.65)$$

The multi-linear regression procedure showed these correlations to be independent of bed height, fibre density, and temperature. This confirms the validity of the δ correction for temperature. Standard deviations are similar to those found for the 73°C correlations, namely 0.128 and 0.155 respectively.

These standard deviations are lower than the value obtained from the correlation in terms of Re_1 and Ga_1, equation (5.60). In fact, it was found that equation (5.60) does not account as satisfactorily for the effect of temperature.

5.6.2 K_1.

Values of K_1 obtained from the parameter estimation procedure were found to scatter widely and cover a wide range of values. Nonetheless, it was clear that in general higher values of K_1 were obtained at higher liquid flow rates and with finer preparations.

As discussed in section 5.2.5, K_1 is the product of a rate coefficient and an area through which mass transfer occurs. This can be represented as

$$K_1 = ka \qquad (5.66)$$

For the laboratory scale extraction tests, it is reasonable to assume that a is given by the total surface area, a_T. But in packed bed operation, the value of a is dependent on the liquid flow and this assumption does not strictly apply. It is however the only approximation that can be used. Consequently, values of K_l/a_T (which actually represent values of k. a/a_T) are shown in Fig. 5.46. This results in a considerably improved correlation, and effectively accounts for the effect of preparation.

This figure can be compared directly with the equivalent values obtained in the laboratory tests, shown in Fig. 5.17, since $K_l/a_T \equiv K'_l/S$. It is apparent that only at very high flow rates, of the order of 100 lb/min ft^2 , do the values of K_l/a_T approach the values obtained in the laboratory tests (roughly 0.1 lb/min ft^2). Of particular interest is the fact that, with finer preparations, the occurrence of flooding limits the maximum flow rate, and restricts the efficiency of mass transfer to values considerably lower than can be obtained in a well-mixed system.

5.6.2.1 Effect of bed height.

The data in Fig. 5.46 represent data obtained with roughly similar bed heights (with a constant bagasse load of 125 lb). Values of K_l/a_T were found to vary significantly with bed height, as shown in Fig. 5.47.

Attempts to correlate this data in terms of liquid holdup proved unsuccessful. It was shown in section 5.6.1 that the dynamic holdup can be identified with mass transfer area; thus it would appear that it is the rate of transfer which is affected at different bed heights.

It was pointed out in section 5.3.2.2. that higher bed heights lead to higher values of fibre packing density, i.e. a more compact bed. This dependence on Z is presumed to be due to the degree of compaction of the bed. Because the bed is more compact with greater bed heights, the average interstitial spaces are smaller with the result that each flow path supports a lower flow rate; attention was drawn to this point in section 5.3.4 in connection with liquid holdup behaviour. Thus the average liquid velocity in each flow path is lower.

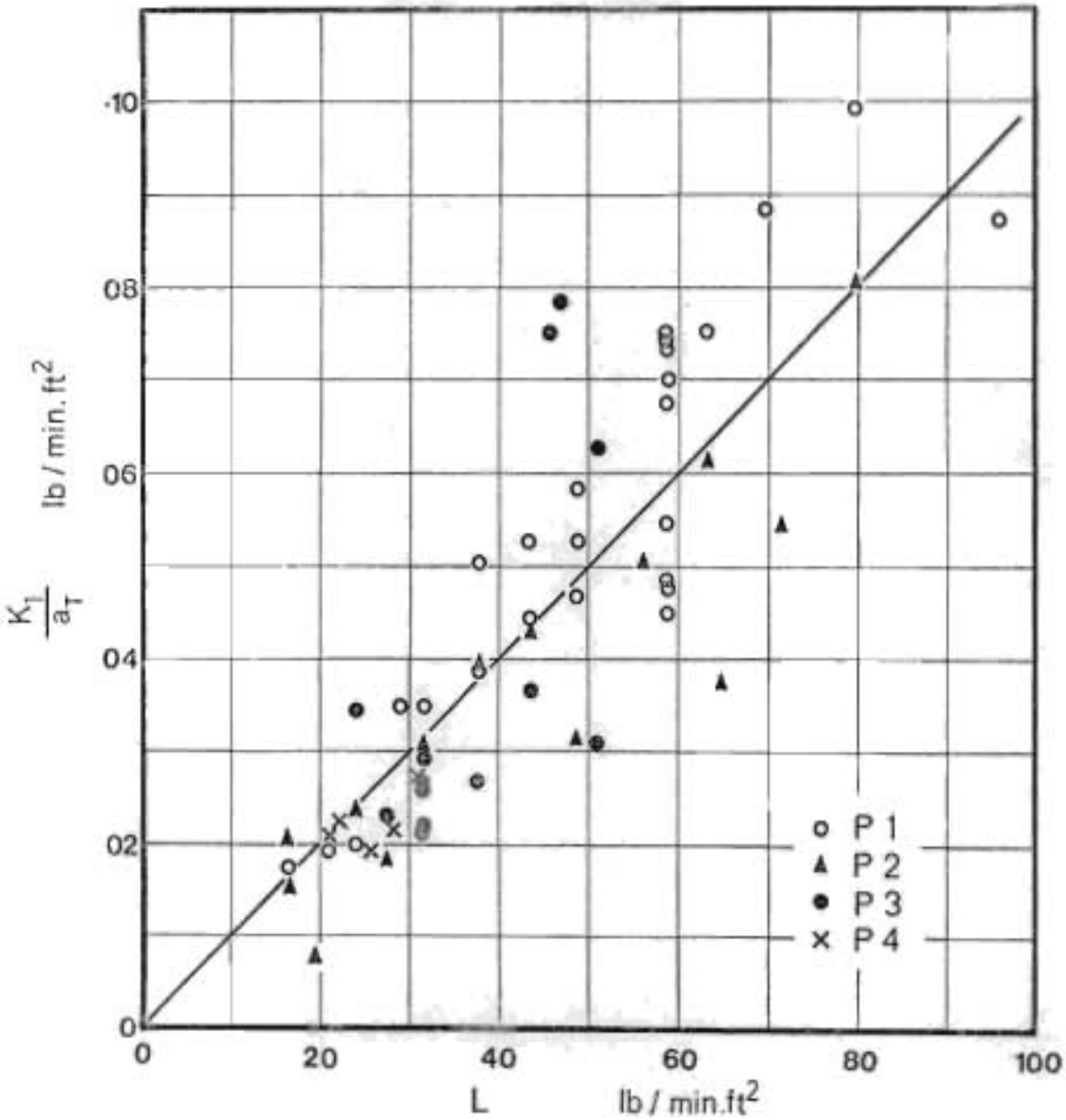


Fig. 5.46 Values of K_1/a_T as a function of liquid flow rate. Data points represent operation at 73°C with an initial bagasse load of 125 lb.

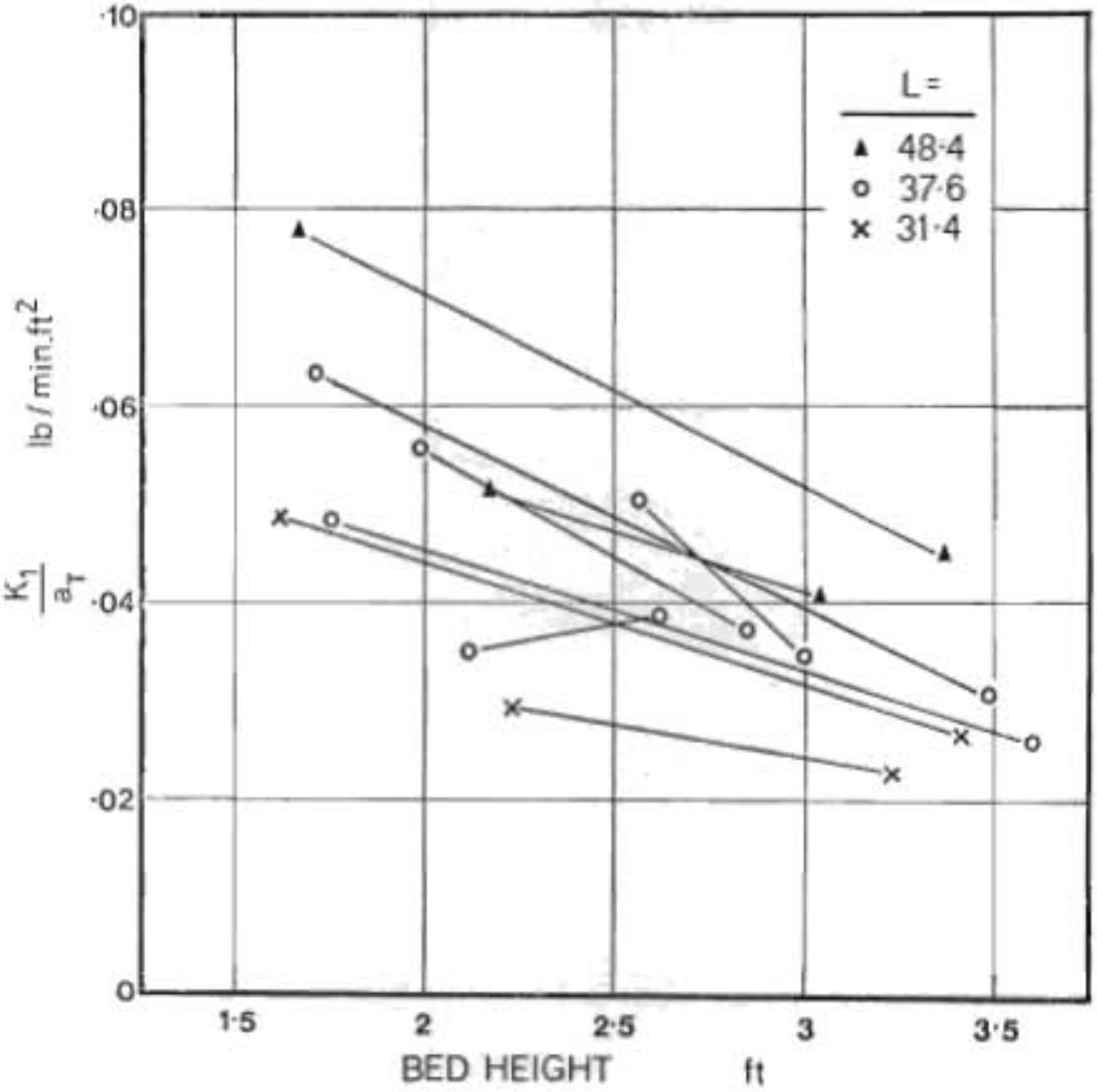


Fig. 5.47. The dependence of K_1/a_T on bed height. Bagasse preparation P1. Solid lines connect data points obtained using subsamples of the same bagasse.

Since liquid velocity is known to affect the rate of transfer from solid surfaces, the values of K_l are lower as a result.

In order to try to confirm this hypothesis, an average dynamic phase liquid velocity $\bar{U}$ was calculated as the quotient of bed height/mean residence time, τ. Values of K_l/a_T are shown plotted against $\bar{U}$ in Fig. 5.48 for preparation P1. Again solid lines connect data points representing runs using subsamples of the same bagasse but at different bed heights.

It was found that lower values of $\bar{U}$ obtain with higher bed heights. This figure demonstrates the dependence of mass transfer rate on the mean velocity of liquid flowing through the bed. Higher velocities can be expected to promote eddies which result in more frequent renewal of surface liquid, thereby promoting the displacement of surface juice.

5.6.2.2 Generalized correlation.

In section 2.5.2 it was shown that mass transfer data is generally correlated in terms of the Sherwood number, Sh, as a function of Re, Sc, and in the case of gas absorption, Ga as well. The data in this form was subjected to multi-linear regression analysis, with Sh_l as dependent variable, while Re_l, Sc, Ga_l, Z and q were considered as independent variables in the regression set. It was found that only Re_l, Sc, and Z are significant variables, leading to the following correlation:

$$Sh_l = 0.00249\ Re_l^{1.150}\ Sc^{0.870}\ Z^{-0.681} \qquad (5.67)$$

where Z is expressed in ft. The standard error is 25.2%; all data are compared with equation (5.67) in Fig. 5.49.

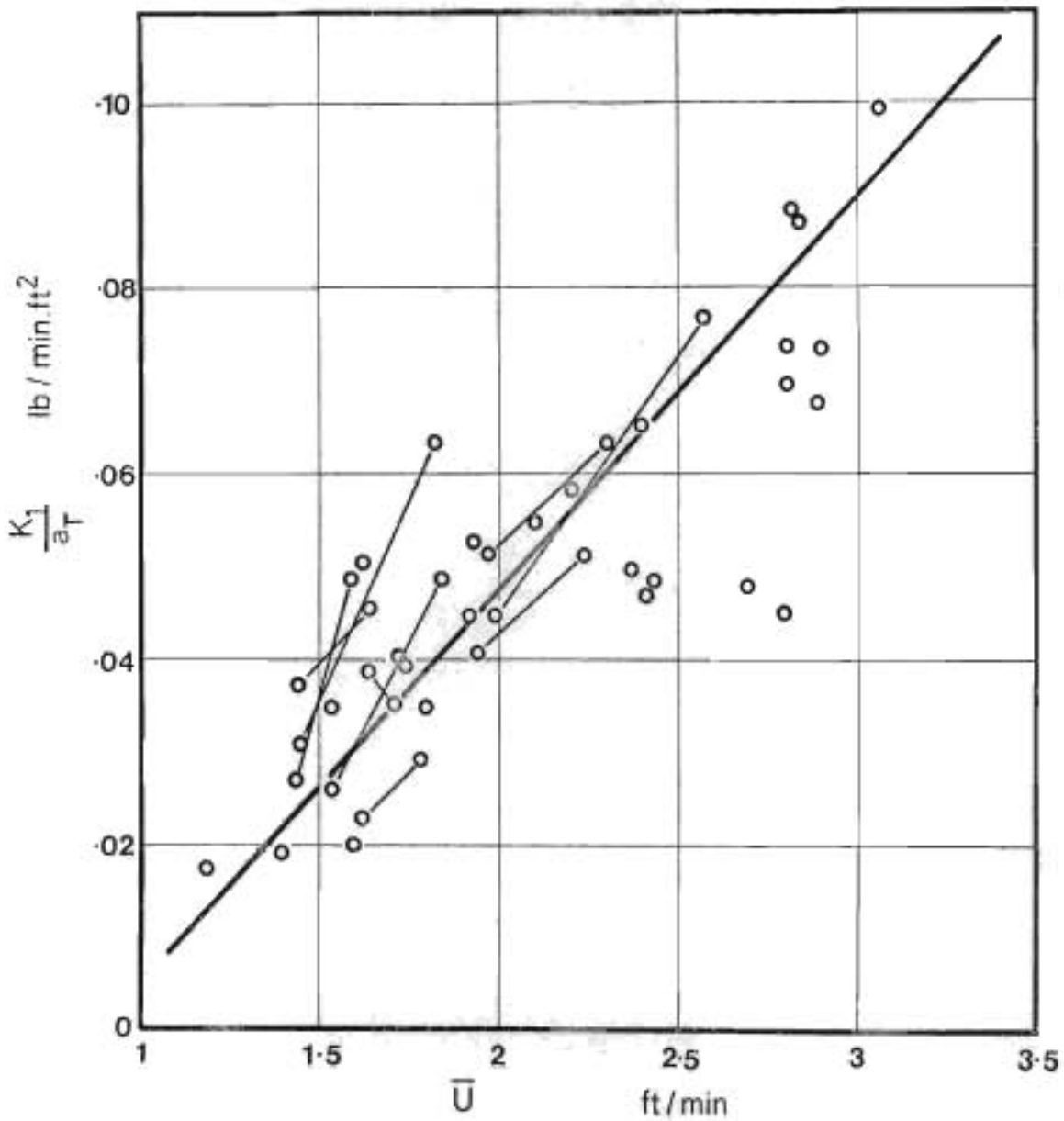


Fig. 5.48. Values of K_1/a_T as a function of dynamic liquid phase velocity $\bar{U}$; data points represent all runs at 73°C with bagasse preparation P1.

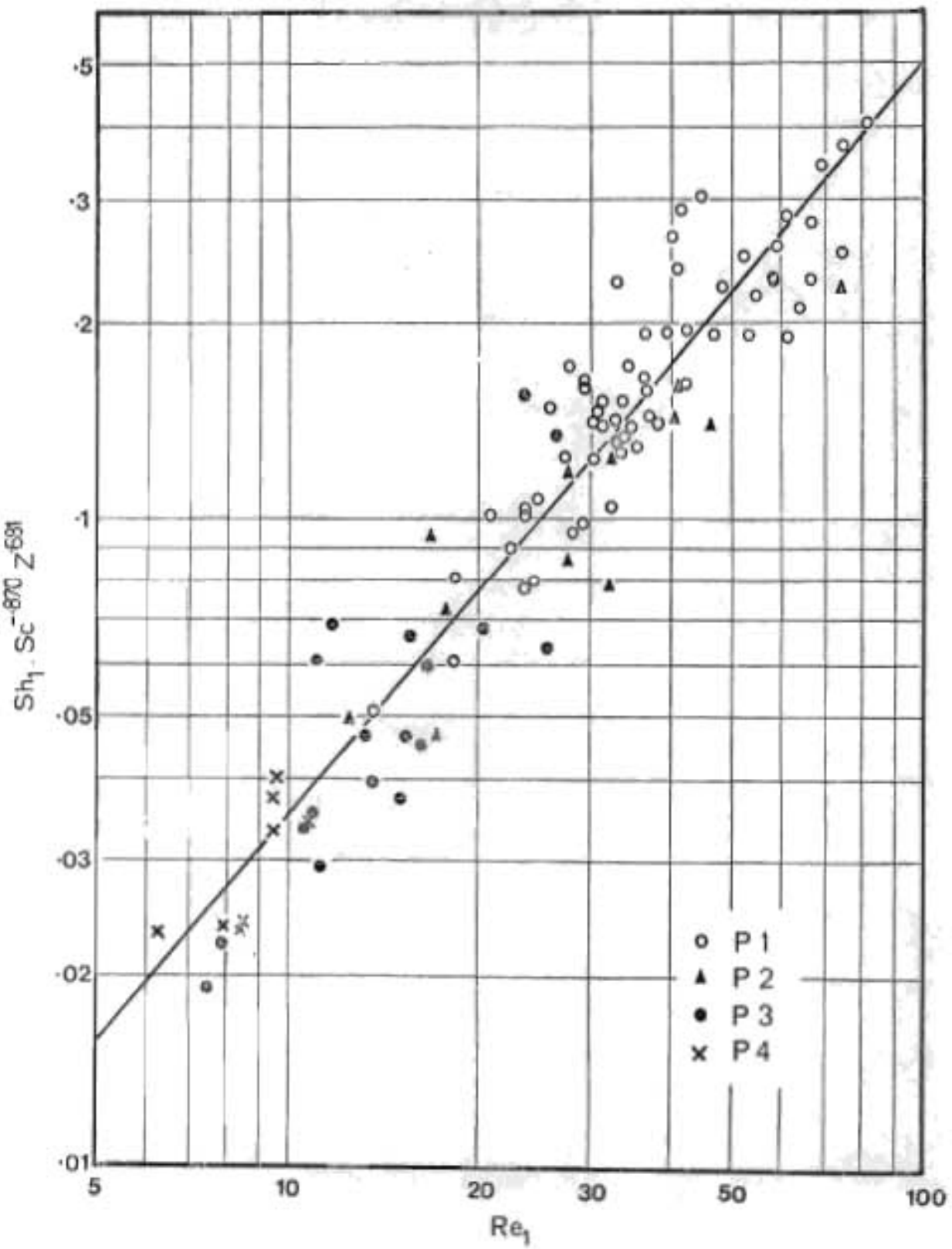


Fig. 5.49 Comparison of the dimensionless correlation of Sherwood Number, equation (5.67), with experimental data.

The inverse relationship between Sh_l and Z follows from the experimental observations. Although bed height is not generally included as a variable in mass transfer correlations, Cornell et al (1960) have reported a number of investigations where packed height is a significant variable. However, this is more likely to be due to liquid maldistribution in high packed columns than to a change in packing density.

The value of the mass transfer coefficient used in Sh_l was calculated using a_T and not the true mass transfer area a, as pointed out earlier. Thus it may be shown that Sh_l is related to the normally employed value of Sh by

$$Sh = Sh_l \frac{a}{a_T} \qquad (5.68)$$

Thus the exponent on Re_l in equation (5.67) includes a contribution due to the variation of a/a_T, with flow rate. Since it was established that a/a_T is proportional to L to the power of $\pm$ 0.5, it can be seen from equation (5.67) that the true coefficient is proportional to L to the power of $\pm$ 0.65. This agrees well with the exponent of 0.6 found for liquid-phase mass transfer coefficients in gas absorption (Semmelbauer, 1967), and lies well within the wide range of exponents on Re, based on the j_D factor, reported in the literature. (Ergun, 1952).

The dependence of Sh_l on temperature is indicated by the inclusion of Sc in the generalized correlation. The correlation correctly describes experimental observations that higher values of K_l are obtained at higher temperatures. The dependence of the mass transfer coefficient on the molecular diffusion coefficient, D_m, has generally been utilized to infer the nature of the transfer process. In section 2.5, it was shown that a square root dependence is generally accepted. Since D_m is contained in both Sh and Sc, equation (5.67) implies that k is proportional to $D_m^{0.13}$. The value of the exponent is considerably lower than the value of 0.5 which

can be expected from the surface renewal theory, as put forward by Danckwerts(1951). In addition part of the overall temperature dependence is accounted for by the variation of a/a_T with temperature. The laboratory extraction tests showed K_1 to be independent of temperature, where extraction by displacement is assumed to occur. Since K_1 refers only to the transfer of surface juice, it would appear that in a bagasse bed a large proportion of the transfer occurs by a true piston-like displacement, and that only a part of the transfer process occurs by a surface renewal mechanism implied by the penetration theory.

5.6.3 K_2.

Values of K_2V are shown in Fig. 5.60. It is clear that no well defined trend with flow rate exists, and particularly at low flow rates, scatter is considerable. All efforts to correlate K_2 in terms of flow rate or Re proved fruitless.

In general, slightly lower values are obtained with finer preparations. Thus K_2/a_T values would be even more widely spread.

In common with K_1, K_2 includes a contribution from the area through which mass transfer occurs. It has been shown that α, the fraction of juice removed by the displacement-washing process, depends on the actively wetted area, represented by the dynamic liquid holdup. In this case, it can be expected that the static holdup, H_S, influences the area through which extraction by a diffusional mechanism occurs. This is substantiated by the fact that K_2/a_T correlates with H_S. An improved correlation is obtained by using the group, $K_2/a_T(1-\alpha)$, which is suggested by the results of the laboratory extraction tests, where the same group was used for correlation purposes. This correlation is represented in Fig. 5.51, which shows data obtained at 73°C, under non-flooding conditions.

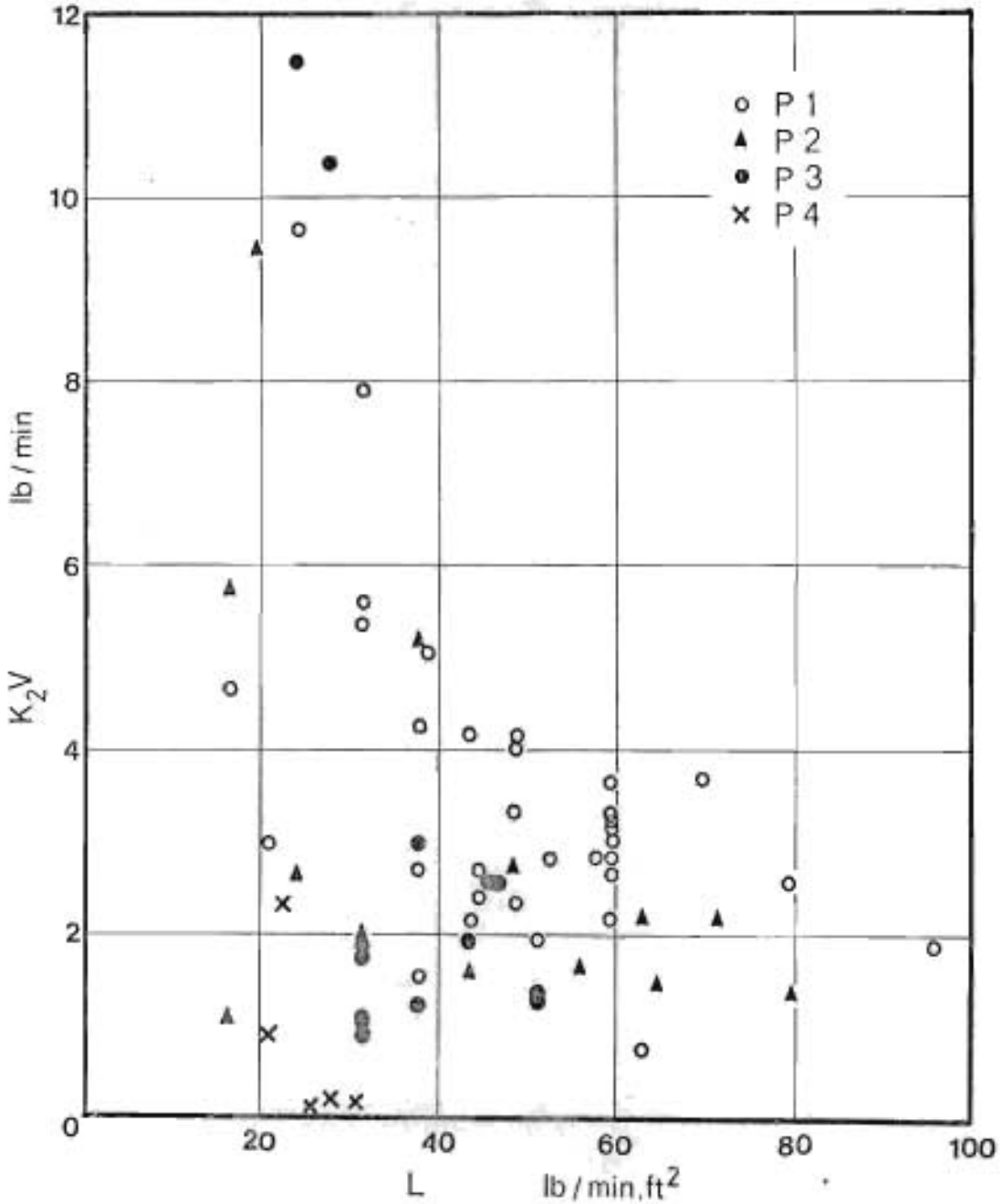


Fig. 5.50. Values of K_2V obtained from parameter estimation procedure.

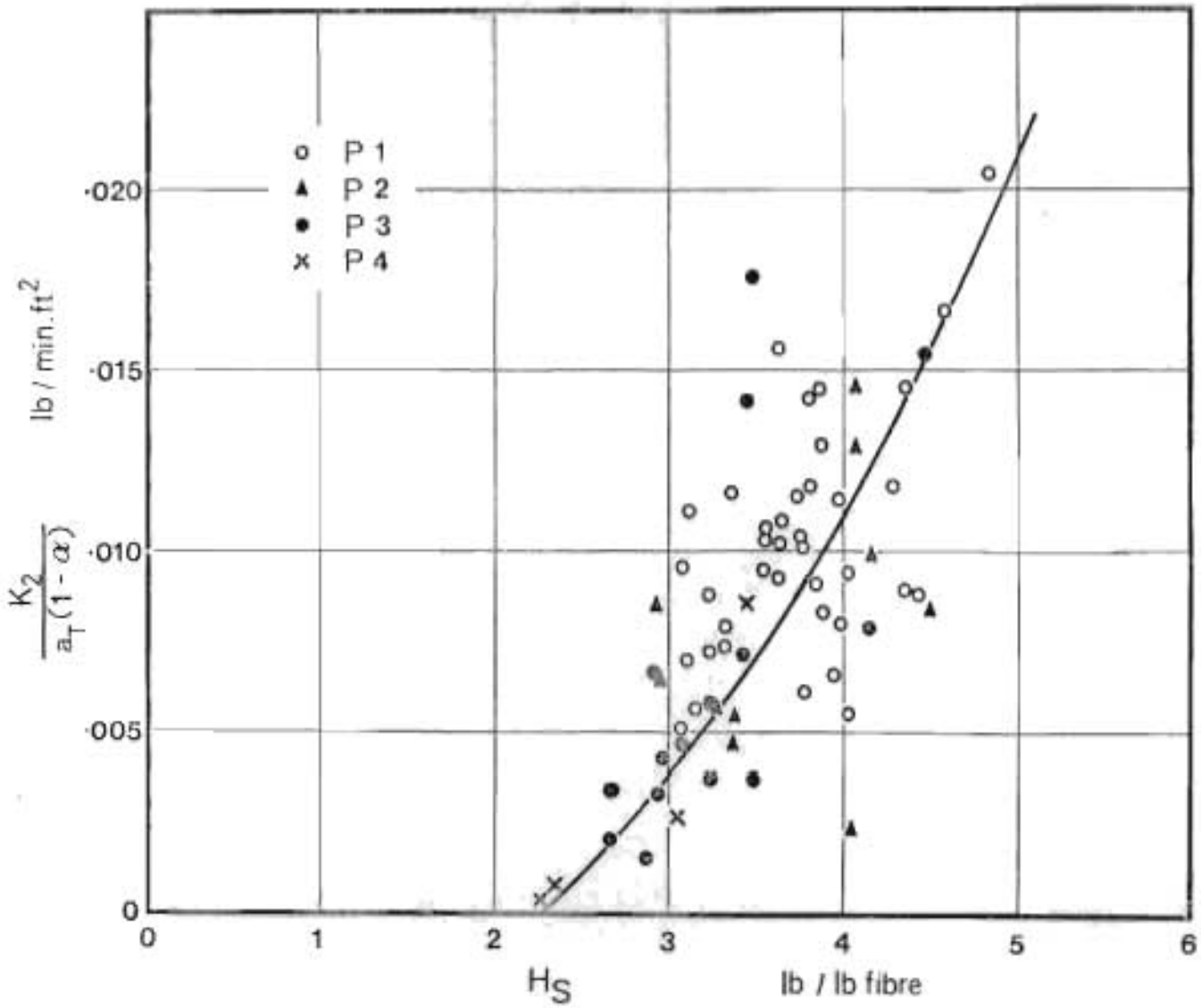


Fig. 5.51. Correlation of values of K_2 in terms of $K_2/a_T(1 - \alpha)$ as a function of static liquid holdup.

Simple linear regression yielded the equation:

$$\frac{K_2}{a_T(1-\alpha)} = 0.00580\ H_S - 0.01170 \qquad (5.69)$$

A correlation coefficient of 0.606 with 70 data points indicates that the relationship between the 2 quantities is significant at a level of less than 0.1% (Volk, 1950).

Clearly, however, the dependency on H_S is not linear. The solid line in Fig. 5.51 represents the quadratic equation :

$$\frac{K_2}{a_T(1-\alpha)} = -0.00246 - 0.00178\ H_S + .00129\ H_S^2 \qquad (5.70)$$

This figure shows that under conditions of high H_S, values of $K_2/a_T(1-\alpha)$ are higher, indicating the increased role played by diffusion in the extraction process under these conditions.

The varying bed height data are well-represented by this correlation, and are included in Fig. 5.51. Generally, lower values of K_2 are obtained with greater bed heights, which corresponds with the lower values of H_S under these conditions.

Unfortunately, data obtained under flooding conditions cannot be correlated in this way, since the value of H_S is not clearly defined under flooding conditions (see section 5.3.3). The flooding data show no clear trends with any of the operating variables. In this case, the best estimate which can be used for predictive purposes is an arithmetic mean. Since only a few results under flooding conditions with preparations P3 and P4 were obtained, a mean value has been calculated from P1 and P2 values only. Moreover, in practice the type of preparation normally employed more closely resembles preparations P1 and P2. Thus, from 13 data points, the mean value is

$$\frac{K_2}{a_T(1-\alpha)} = 0.0061 \qquad (5.71)$$

with a standard deviation of 0.0018.

From the laboratory-scale extraction results, it was shown that K_2 is proportional to the molecular diffusion coefficient (section 5.2.5). Although the extraction of 'tightly held' juice (to which K_2 refers) is clearly affected by liquid hydrodynamics, it is still likely that the same temperature dependence holds in the packed bed case.

Unfortunately, most of the temperature data show a large amount of scatter around the correlation of Fig. 5.51, which tends to mask the temperature effect. A series of data obtained from runs at the same flow rate, but different temperatures, is shown in Fig. 5.52. Solid lines join runs carried out with subsamples of the same bagasse. The bottom half of the figure shows the uncorrected data, while the top section shows the data corrected to 73°C, by multiplying values of $K_2/a_T(1-\epsilon)$ by D_{m73}/D_{mT}. Although in both cases, the data is well to the right of equation (5.70), the trend shown by the pairs of data points corrected to 73°C in this way is obviously closer to that of equation (5.70).

It is unrealistic to claim the validity of this temperature correction on the basis of a small number of data points. However, taken together with the results of the laboratory extraction tests, this suggests the use of a correction factor for the effect of temperature on D_m, in order to predict values of K_2 from Fig. 5.51 at temperatures other than 73°C.

5.6.4 Discussion.

5.6.4.1 Reliability of Parameter Correlations.

All the parameter correlations of the preceding sections are associated with a large degree of scatter. It is necessary to consider how such scatter could arise, and whether such scatter is greater than could be expected 'a priori'.

Scatter is likely to arise as a result of three factors:

i. Variability of cane quality.
ii. Errors in parameter estimation.
iii. Inherent variability of packed bed operation.

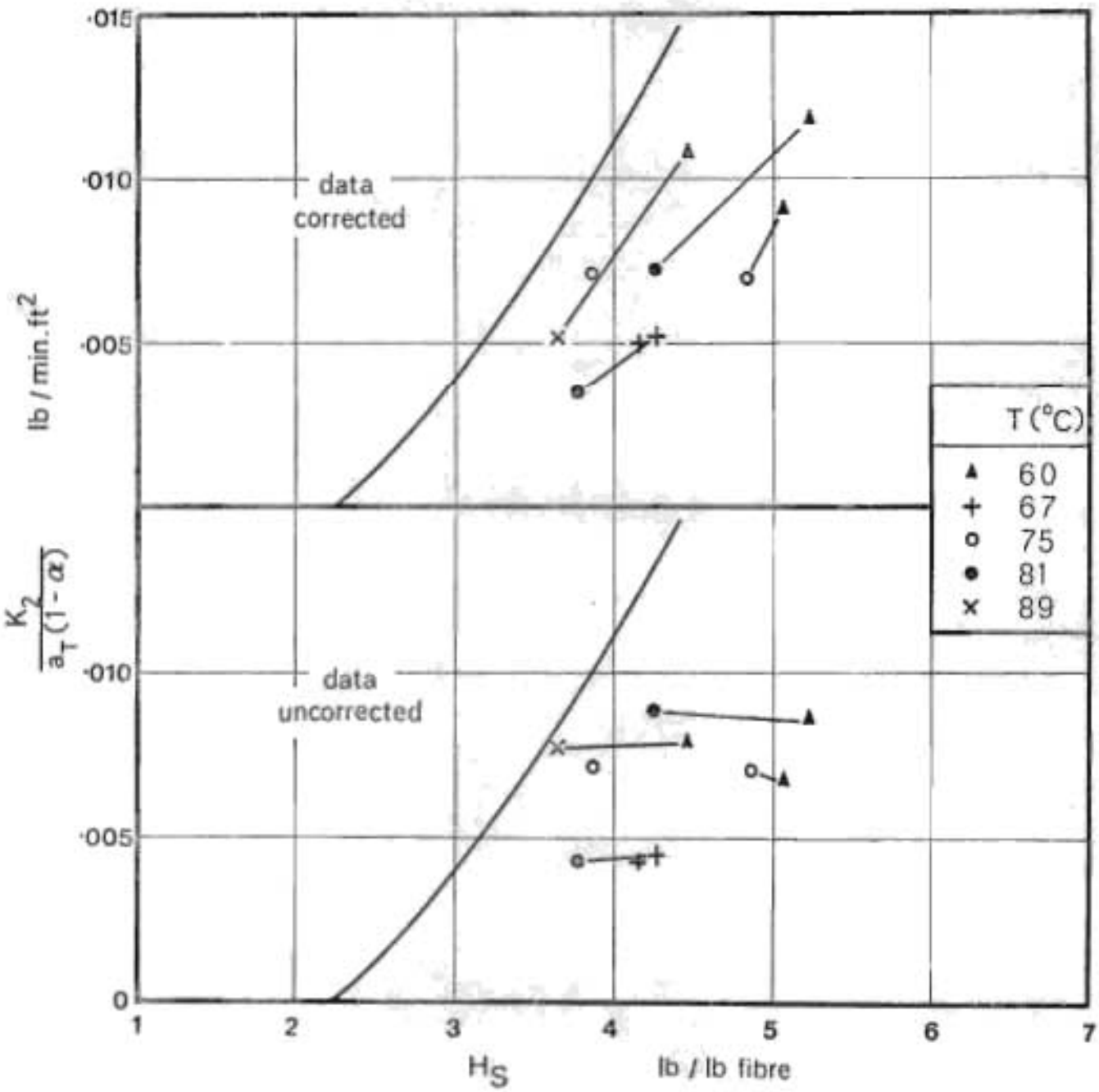


Fig. 5.52 The effect of correction of values of K_2 to account for variations in D_m with temperature. Bold line represents the 73° C correlation, equation (5.70). Data shown represent operation at L = 44.6 lb/min. ft^2; bagasse preparation P1.

The laboratory extraction tests include only the first 2 components. From Table 5.6, it can be calculated that for the laboratory data, standard errors represent ± 15% of the mean. It was pointed out in connection with these tests that most of the variation appeared to be due to variations in cane quality. This underlines the fact that the nature of bagasse is not sufficiently completely characterized by DI and sieve analysis.

Parameter estimation proved more difficult for the pilot plant data; it was shown in section 5.5.4 that with 95% confidence, K_1, is determined to within ± 10%, and K_2 only to within ± 30% or more.

The third component was discussed in section 5.3.5 in connection with the reproducibility of liquid holdup measurements. This is further illustrated by the findings of Gunn and Pryce (1969) that the principal component of scatter in measurements of Pe (or axial dispersion) in a packed column was generated on re-packing the column. Standard errors of the order of 15% were found for the correlations for H_D and H_S (see section 5.4)

It is not possible to calculate an expected variance from the individual components mentioned above, since they are not linearly related to the parameter values. Nonetheless, in the light of these remarks, observed standard errors for the model parameter correlations of 20 - 30% do not appear unreasonable. The greater degree of scatter in the correlation of K_2 is presumably due to the fact that K_2 values are less well determined by the parameter estimation procedure.

5.6.4.2 Interpretation.

It is apparent from the preceding sections that extraction is affected by the liquid hydrodynamics in a packed bed of bagasse. Lower values of α and K_1 at low flow rates are indicative of lower liquid-solid contact efficiencies. It is improbable that this is due to incomplete wetting of bagasse particles; because of the hydrophilic nature of bagasse, wetting is easily accomplished in spite of air trapped in the bed. Moreover, on emptying the pilot

diffuser, and from observation through the sightglass in the diffuser vessel, no signs of any non-wetted pockets of bagasse were evident. Rather, the inefficient liquid-solid contacting can be explained in terms of the existence of stagnant pockets of liquid within the bed, particularly because of the affinity of bagasse for water, and since such static liquid is expected to be greater in fibrous beds (Kyan et al, 1970).

The parameter a, representing the fraction of juice which is readily extracted, has been shown to depend on the dynamic liquid holdup, and not the total liquid holdup. Thus, extraction by displacement-washing depends on the actively wetted area, and not the total wetted area. The existence of static holdup indicates that some particle surfaces are not available for active wetting. At higher flow rates, dynamic holdup increases, while static holdup decreases and the rate of extraction is correspondingly improved.

Zones of static liquid within the bagasse bed can be considered to have a 'blocking' effect; thus some juice which should be easily washed from the surfaces of particles, has to find its way by diffusion through the static liquid to reach the flowing liquid. The higher values of K_2 found for the cases where the static holdup is higher confirm the increased role of diffusion under these conditions.

The results of laboratory-scale extraction tests serve as a useful frame of reference. Because of the good mixing between bagasse and liquid in these tests, it is to be expected that stagnant liquid pockets are absent. Only at high flow rates do values of a and K_1 from the pilot plant data approach those obtained in the laboratory-scale mixing tests. This indicates that extraction is more efficient in a well-mixed system than in packed-bed operation.

Higher temperatures are beneficial in two respects. Firstly, extraction by diffusion is faster, since the molecular diffusion coefficient is higher. Secondly, the degree of active wetting is higher, as evidenced by higher values of a, due to the lower liquid viscosity at higher temperatures. Thus, in addition, a smaller fraction of the sucrose has to be extracted by a diffusional mechanism. However, the effect of temperature on extraction performance is not as pronounced as the effect of liquid flow rate, or the effect of degree of preparation. This is brought out more fully in the next section.

Correlations of model parameters have not distinguished between flooding and non-flooding operation. It was shown in section 5.4.1 that, even under so-called flooding conditions, a significant amount of porosity is present, and that continuous liquid-phase operation is not realized. The occurrence of flooding does not appear to affect the rate of extraction. This confirms the view that flooding involves a gradual build-up of liquid, and that it should be regarded as merely an extension of the non-flooding regime.

5.7 EXTRACTION OF SUCROSE.

The pilot plant experiments were undertaken to provide the necessary data for the development and testing of a mathematical model of the extraction process, and for the evaluation of the model parameters over a wide range of process operating conditions. Once this has been achieved, the model can be used to predict the extraction expected for any geometry of diffuser that involves the flow of liquid through a bed of bagasse. In Chapter 6, it will be shown for instance how the model can be applied to a full-scale moving bed diffuser.

In the pilot plant tests, the extraction achieved was also determined directly. These measured extraction values have little practical utility, since the operation of the pilot plant diffuser bears little resemblence to the counter-current extraction process of a full-scale plant diffuser. Nonetheless, these measured extractions indicate directly the influence of the various control variables on extraction.

Extraction in the pilot plant diffuser can also be computed via the mathematical model if reliable estimates of the model parameters are available. In order to show how the model parameter values affect the computed extraction values, the sensitivity of these values to the model parameters is also reported here.

Factory control results generally report the combined extraction obtained in the diffusion and de-watering stages, which, however, is influenced by the efficiency of the de-watering mill(s). Extraction obtained in the factory diffuser alone cannot be determined with any reliability, since the bagasse discharged from the diffuser is associated with a large amount of adhering juice. Apart from the difficulty of sampling such wet bagasse reliably, analysis of the discharge material includes any adhering juice, the major part of which constitutes mechanically-held surface juice, which is easily removed in a de-watering mill, and should be regarded as juice already extracted.

However, extraction in the pilot diffuser could be easily determined. The brix extracted was calculated utilizing the measurements of weight and brix of the outlet juice retained in drums (see section 4.2). The extraction after t minutes, E_t, was calculated as the percentage of brix in the original bagasse extracted during that time.

It should be remembered that the experimental work of this study employed first mill bagasse as a raw material. Although individual values of first mill extraction could not be obtained for each sample of bagasse used, factory measurements show that during the periods of the experimental work, first mill pol extraction figures obtained at Mount Edgecombe mill averaged 67.6%, with a standard deviation of 2.4%.

5.7.1. Effect of process variables on measured extraction values.

Brix extraction values obtained at 73°C after 16 minutes percolation time, E_{16}, are shown as a function of flow rate in Figs. 5.53 and 5.54. This shows clearly the higher extraction values obtainable with finer bagasse preparations, and also that higher flow rates generally promote a higher extraction.

It appears that the extraction-flow rate curve levels off at a flow rate of ± 50 lb/min. ft^2. With the finer types of preparation employed, such flow rates are not attainable due to the earlier onset of flooding.

The scatter evident in these figures is not unexpected, since extraction is affected by cane quality (Brüniche-Olsen, 1966) and fibre content (Buchanan, 1967). However, first mill extraction is likely to have a considerably greater effect. Under conditions of efficient first mill performance, the juice remaining in first mill bagasse may be more difficult to extract. Ideally, first mill extraction figures should be combined with extraction obtained in the diffuser; unfortunately such first mill extraction figures were not available.

Although Figs 5.53 and 5.54 demonstrate the effect of degree of preparation on extraction, more striking is the relation between extraction and DI, shown in Fig. 5.55. Data for very low flow rates (<20 lb/min.ft^2) have been omitted; otherwise a marked dependence of E_{16} on DI is evident, despite the fact that the data points represent different levels of flow rate.

In addition, Fig 5.55 demonstrates the utility of DI as a guide to expected extraction performance.

Higher temperatures promote higher values of extraction, as shown in Fig. 5.56 for preparation P2, P3 and P4. Solid lines connect data points obtained from runs using subsamples of the same bagasse, but at different temperatures. In studying the model parameter behaviour with temperature in section 5.6, it was shown that higher temperatures promote the rates of extraction, as well as increased values of α due to lower liquid viscosities.

Lower bed heights were also found to result in slightly improved extraction performance. Since full-scale diffusers operate with bed heights generally greater than were used in the pilot plant tests, it would appear beneficial to operate diffusers with reduced bed heights.

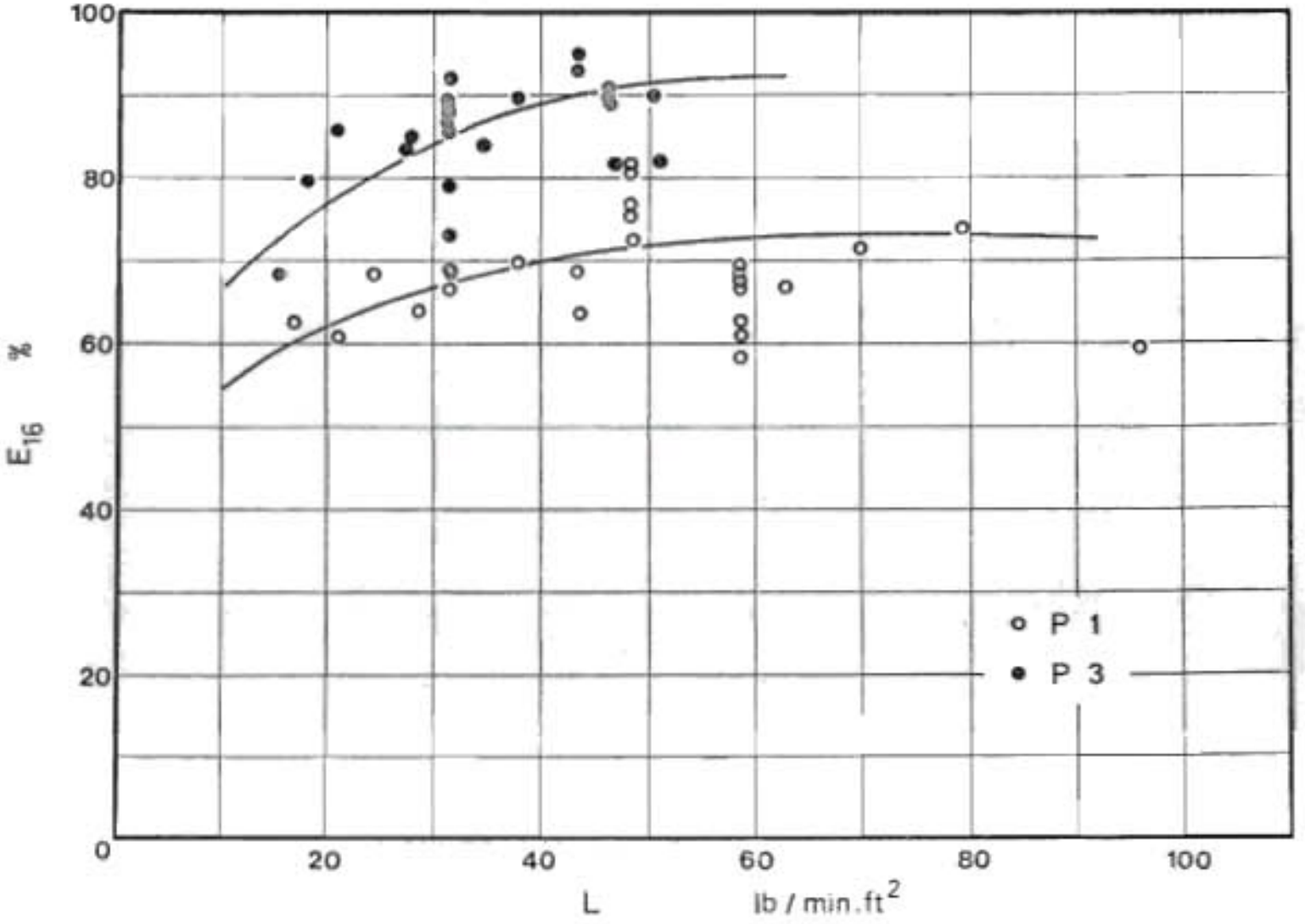


Fig. 5.53 Measured values of extraction after 16 mins percolation time. preparations P1 and P3. Liquid temperature 73° C.

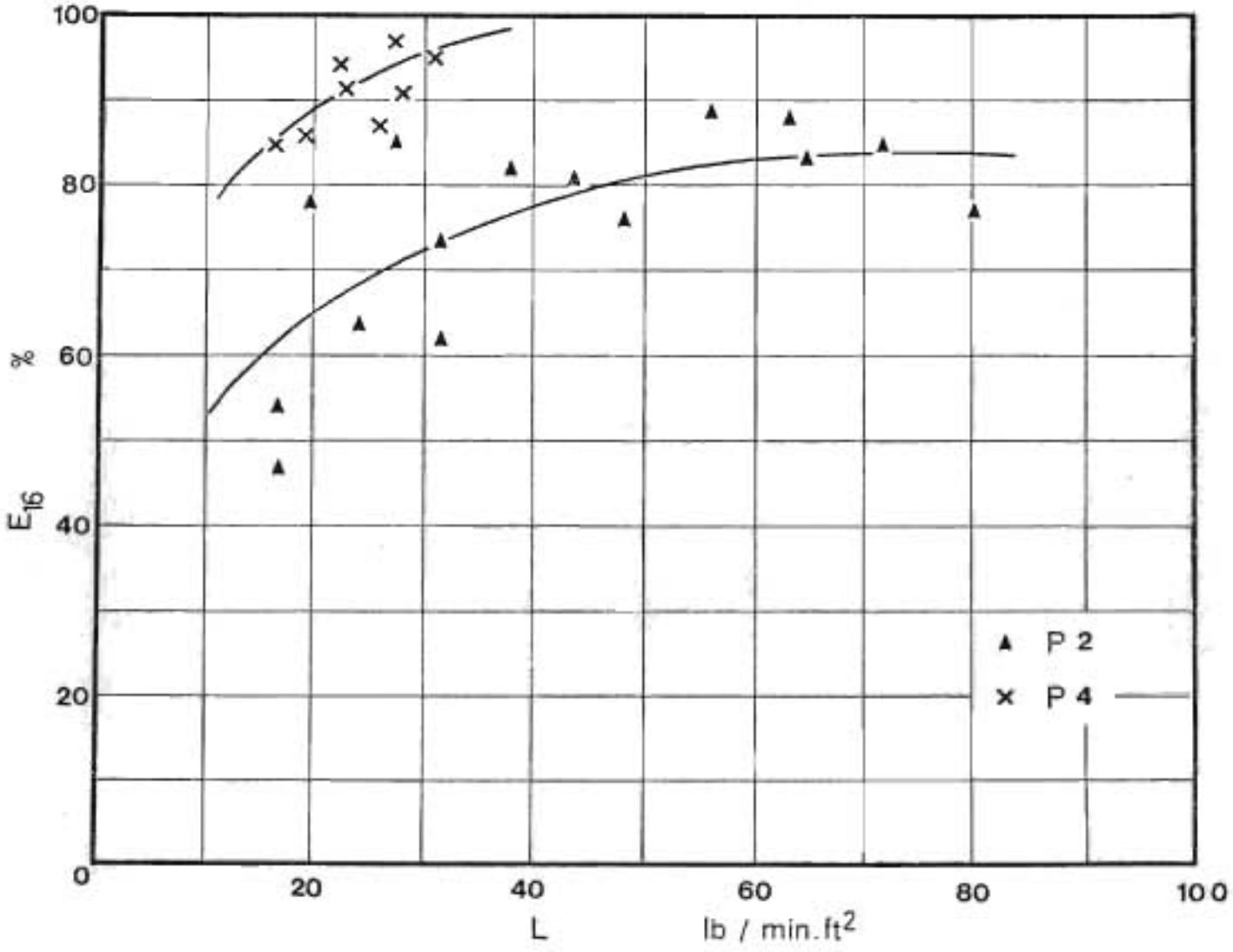


Fig. 5.54 Measured values of extraction after 16 minutes percolation time, preparations P2 and P4. Liquid temperature 73° C.

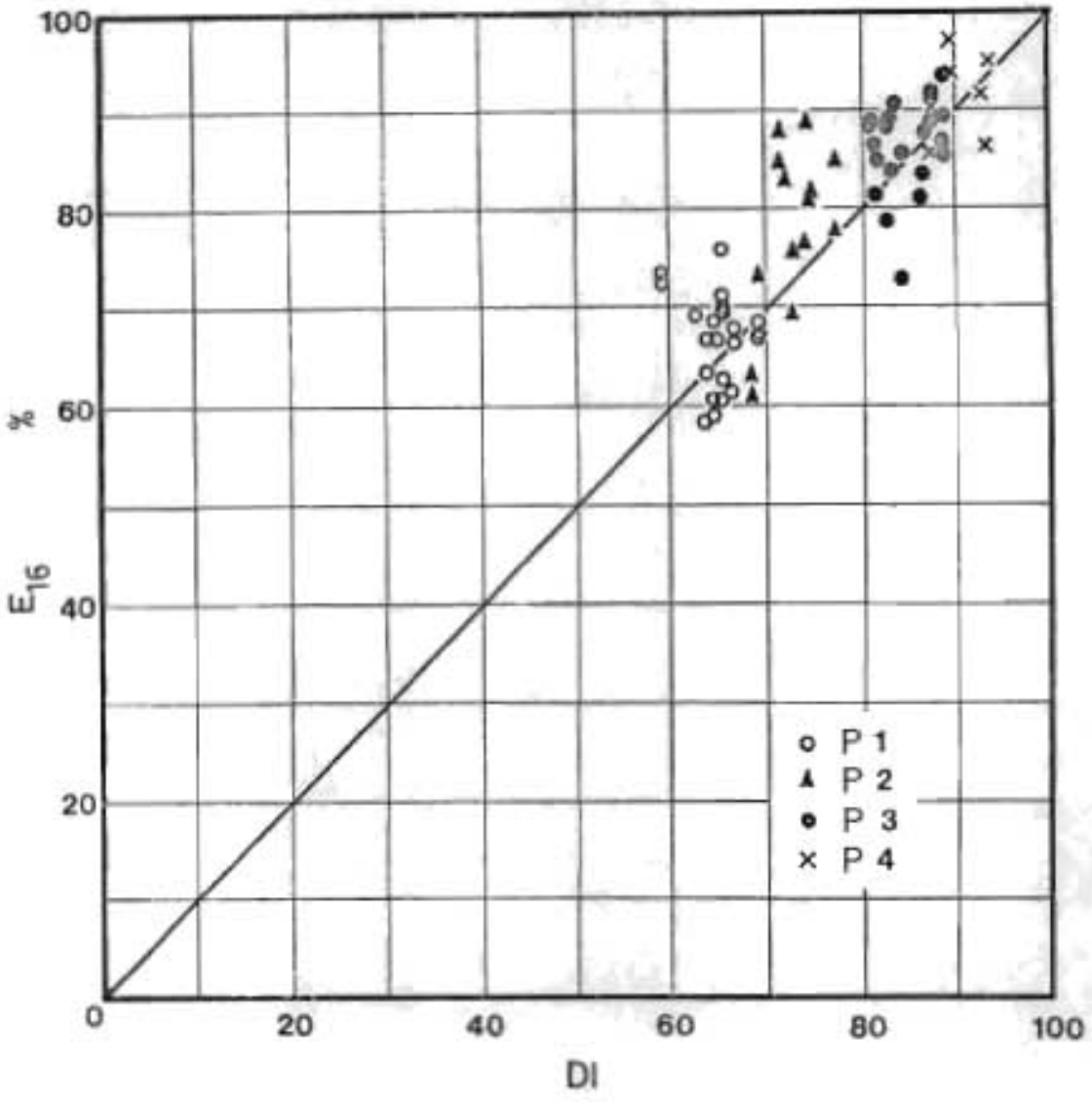


Fig. 5.55. The dependence of extraction on the DI of bagasse. Data points represent all runs where L > 20 lb/min. ft^2, at 73°C.

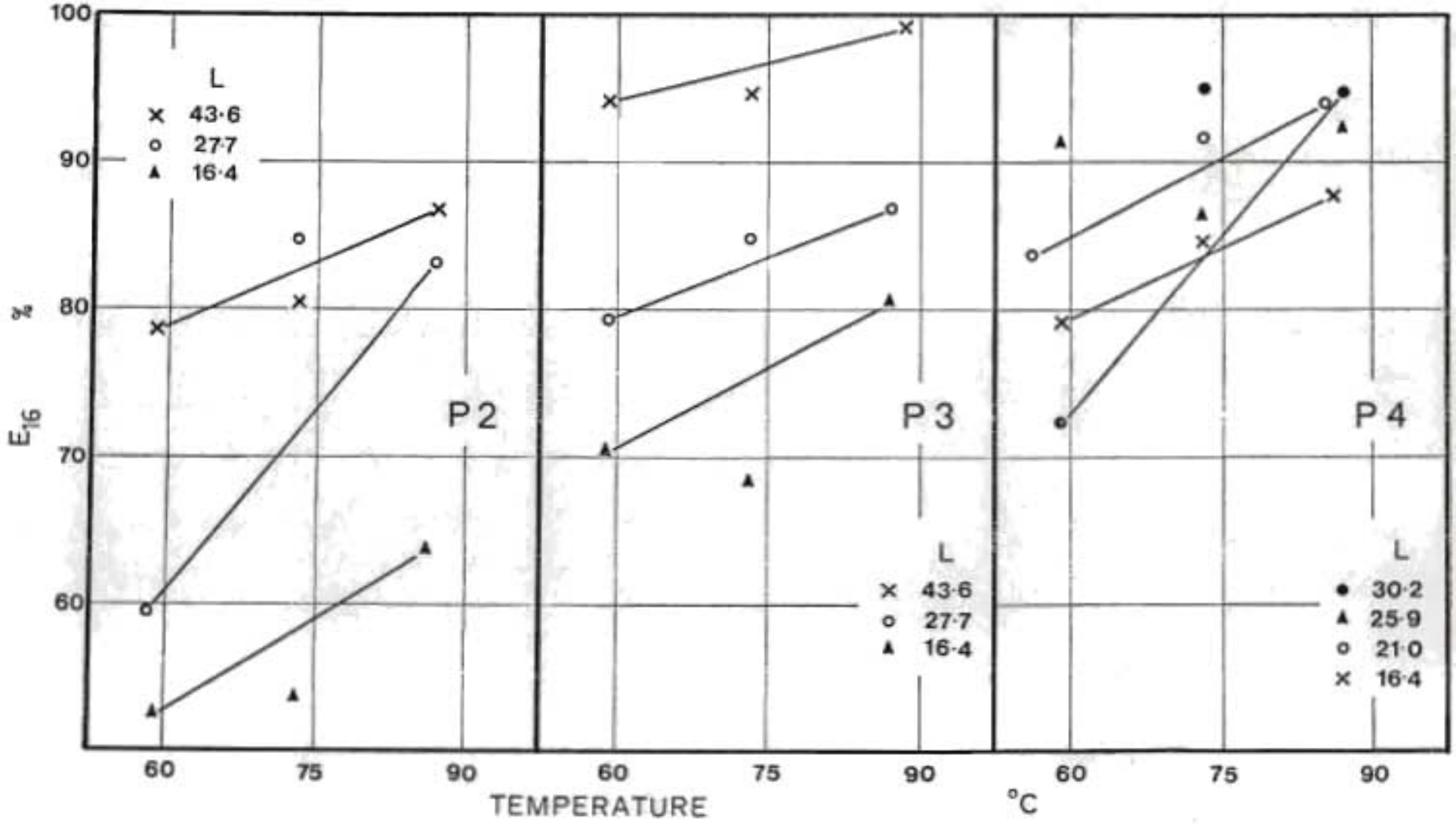


Fig. 5.56 Effect of temperature on extraction.

5. 7. 2. Diffuser Extraction Related to Overall Expected Extraction.

In order to relate the brix extraction obtained in the diffusion stage to the overall sucrose extraction obtained by a first mill, diffuser, and de-watering mill(s), it is necessary to know:

(a) first mill extraction,

(b) the relation between brix and pol extraction,

(c) an estimate of the performance of the de-watering mill(s).

Buchanan (1967) and Brüniche-Olsen (1966) have shown that the overall extraction is very dependent on first mill extraction. As mentioned earlier, an average first mill extraction of 67. 6% was obtained in the Mount Edgecombe milling train.

The purity of the juice extracted in the 1st mill is much greater than that of the last expressed juice (see section 2. 8. 2). Thus, sucrose is extracted at a faster rate than brix, because of the larger amounts of soluble non-sucrose species associated with the vascular bundles. Use can be made of the purities of the juice extracted and the juice remaining in the bagasse to calculate the sucrose extraction from brix extraction figures. Thus, using typical values, for South African conditions, of 85 and 65 for mixed and last expressed juice purities respectively, it can easily be shown that sucrose extractions are greater by 1 to 2% than the equivalent brix extractions, for brix extractions of the order of 95%.

The performance of de-watering mills lies outside the scope of this project. However, it is worthwhile to consider briefly the expected effect of the de-watering mill on diffuser discharge bagasse. The diffuser model assumes that sucrose is present in 2 "phases", one fraction readily extracted, and the other more tightly held in the bagasse. The easily extractable phase,because it can be readily removed by a washing process, must be located on or near the particle surfaces. It is to be expected therefore that it will also be more easily removed in any mechanical expression process. Thus the concentration and amount of the tightly held juice fraction probably determines to a large extent the sucrose left in the bagasse after the de-watering mill.

A simplistic model of a de-watering mill can be formulated, assuming that all readily available juice is removed prefentially in the de-watering mill, before any tightly-held juice is expressed. The introduction of a reabsorption factor, as defined by Murry & Holt (1967), and specification of the moisture content of final bagasse, would then be sufficient to define the brix remaining in the bagasse. The reabsorption factor could be adjusted so that agreement between the model and actual mill performance is obtained.

The lack of a suitable method of measuring the extraction performance of full-scale plant diffusers was mentioned at the beginning of section 5.7. This concept suggests that a method of measuring diffuser efficiency on the plant scale should be directed towards the measurement of the extraction of "tightly-held" sucrose. Adhering juice and the readily available juice fraction could be removed by a simple laboratory washing test, so that any sucrose not removed is defined by the experimental washing process as tightly-held sucrose. This method would also circumvent difficulties associated with sampling wet diffuser bagasse, since it is only the tightly-held sucrose/fibre ratio of input and output bagasse which is of interest.

5.7.3. Dependence on Model Parameters.

The sensitivity of extraction to the values of the model parameters was evaluated by computing values of extraction expected in the pilot plant as described in section 5.5.1.1. As a base case the values of process variables and parameters given in Table 5.26 were used.

Table 5.26

Specification of Base Case Variables.

Flow rate	47.7 lb/min ft^2
Brix % bagasse	13.0
Bagasse load	125 lb.
Juice holdup in bagasse	100 lb.
Percolation time	16 mins.
K_1V	30 lb/min.
K_2V	3 lb/min.
α	0.4

These values were chosen as being representative of first mill bagasse (preparation P1). The sensitivity of E_{16} to model parameters was computed varying only 1 of the model parameters at a time. The results are shown in Fig. 5.57.

This figure indicates that K_1 has very little effect on computed values of E_{16}, while α and K_2 show a pronounced effect. Clearly, since $K_1 >> K_2$, after 16 minutes percolation time, all readily available sucrose has been removed. This is illustrated by the fact that, for $K_2V = 0$, $E_{16} = 40\%$, corresponding to a value of α of 0.4. However, in a counter-current moving bed diffuser, the bagasse residence time within a stage is only of the order of 3-4 minutes. Thus values of extraction after 4 minutes percolation time, E_4, were computed, and are shown in Fig. 5.58. This presents a very different picture, in that K_1 now has a significant effect, although even in this case, after a high enough value of K_1 is reached, its effect becomes negligible.

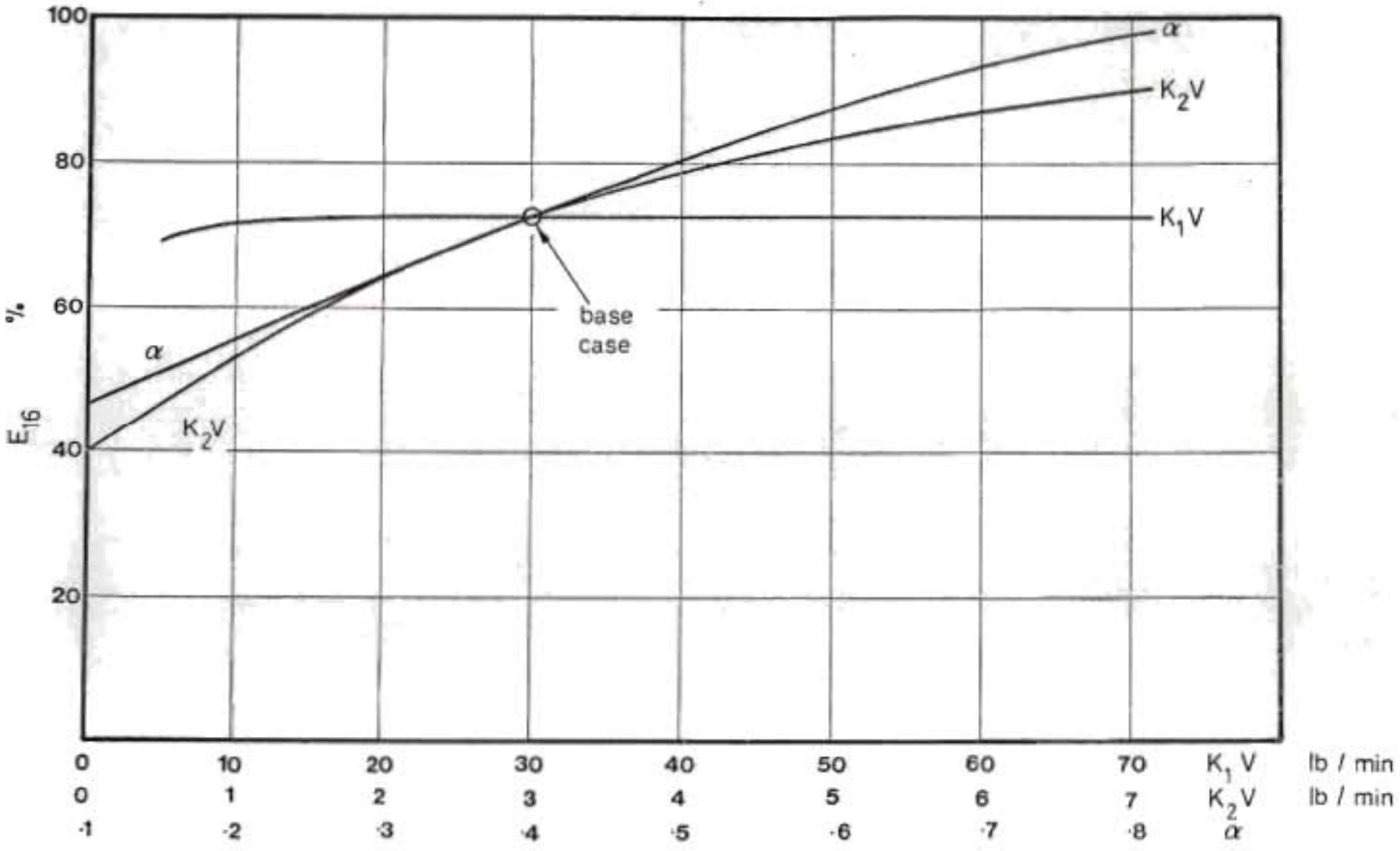


Fig. 5.57 The sensitivity of computed values of extraction after 16 mins to the values of the model parameters.

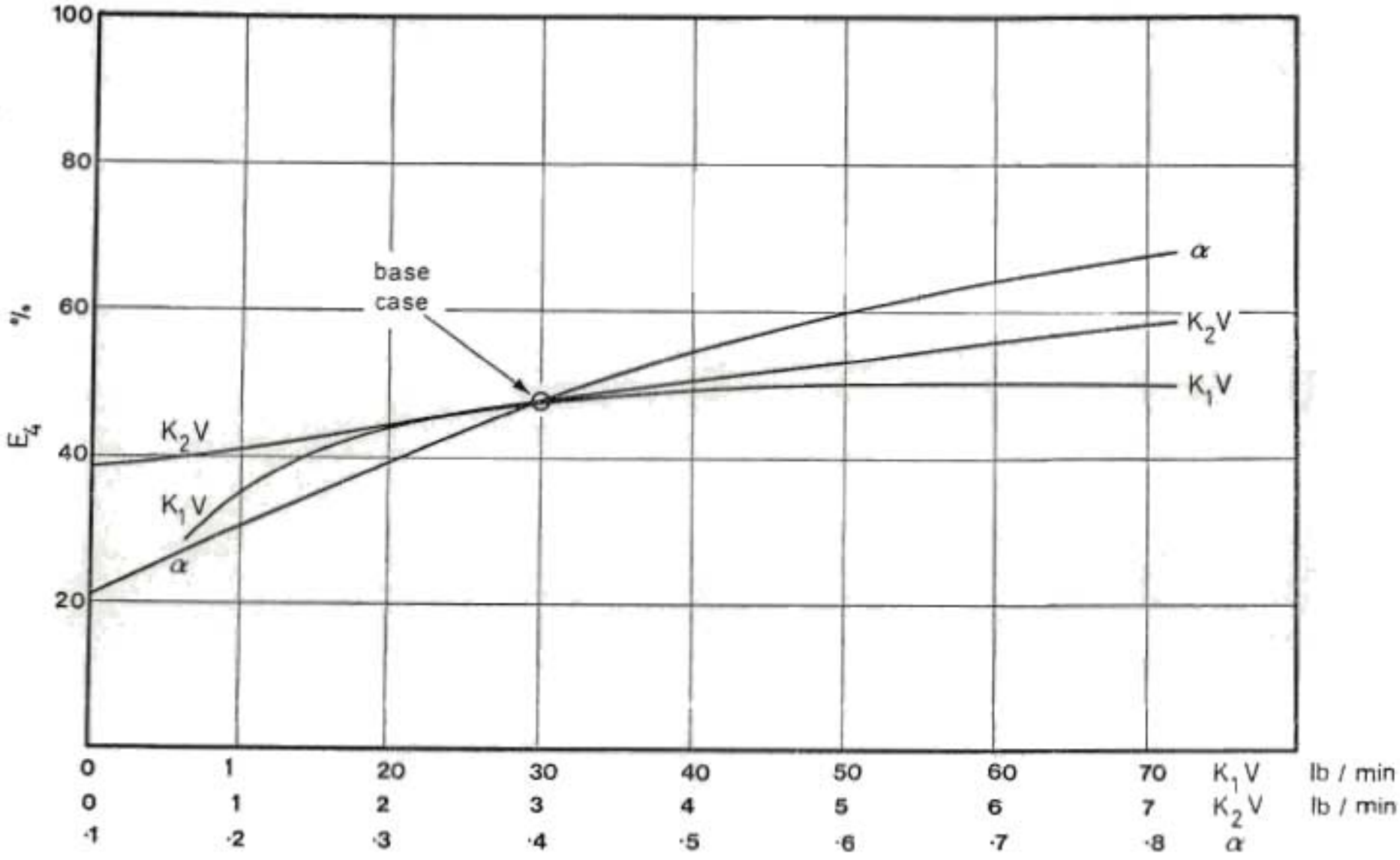


Fig. 5.58 The sensitivity of computed values of extraction after 4 mins to the values of the model parameters.

It should be remembered that for the conditions of flow rate and preparation assumed, values of the parameters far removed from the base case can represent hypothetical conditions only. Nevertheless, Fig. 5.58 demonstrates that the value of α has the most significant effect on extraction.

5.8. EXTRACTION OF IMPURITIES

The recovery of sucrose from mixed juice depends on the quantity and nature of the impurities in the juice. In view of the disagreement regarding the relative magnitudes of sucrose losses in molasses in diffusion and milling factories, there is clearly a need for a controlled investigation to rationalise subjective information on the extrac - tion of impurities in the diffusion process. Diffuser juice contains a host of different substances, and an intensive investigation embracing measurement and identification of all impurities constitutes a major research project in its own right. For the purposes of this project, attention has been confined to only a few impurities which are considered to have a major effect on recovery.

Thus attention has been focussed on the extraction of reducing sugars, inorganic materials, and starch. Reducing sugars are the most abundant impurity in mixed juice. Inorganic impurities have a pronounced melassigenic effect, since the major part of inorganic material is not removed in the recovery process and results in a high loss of sucrose in molasses (see section 2.8.2). Starch is a major organic impurity which has a profound effect on recovery; although gums are present in higher concentrations in diffuser juice (Graham et. al., 1968), the attention which starch has received in South Africa qualifies it as a principal harmful organic impurity.

Temperature, degree of preparation, and flow rate were varied in these tests to investigate the effect of these variables on impurity extraction. Flow rate was varied only over a fairly narrow range of higher flow rates, since this flow rate range leads to higher sucrose extractions, and thus represents the range of interest. It was found, however, that flow rate had no discernible effect on the extraction of the

impurities studied. pH was not included as a variable, since it appears that within the range of pH employed in practice, extraction of pectins only is significantly influenced by pH. The amount of pectin extracted is generally small, and according to Spencer & Meade (1945), it is in any case completely removed in clarification. Thus all extraction tests were carried out at a pH of ~ 6.

5.8.1 Operation of the pilot plant.

Extraction of impurities was measured in the pilot plant diffuser, rather than in the laboratory scale mixing equipment, since this would be more representative of conditions in percolation diffusers. In order to maintain the levels of impurities high enough for measurement, with adequate accuracy, it was necessary to re-circulate liquid through the diffuser rather than use the 'once-through' procedure employed for sucrose extraction tests. The latter method of operation was, however, utilised for a few runs to investigate the initial rates of extraction of reducing sugars and inorganics.

The brix profile in the re-circulation runs is naturally different from that obtaining in normal once through operation. Typical curves are shown in Fig. 5.59. The brix drops as in the once through case, but after a few minutes the brix of the juice entering the diffuser rises above zero. Thus the outlet brix values rise again, and gradually continue rising until an equilibrium value is reached.

Prior to a run, the water level in the surge tank was adjusted to the same height for each run. Thus the initial amount of water in the system was set at the same value for each run (roughly 140 gals.). This was the minimum amount which, after allowing for liquid hold-up in the diffusion vessel and the piping, would be sufficient to cover the stirrer blades and ensure thorough mixing in the tank. The final brix values of the juice samples depended on the initial brix content of the bagasse and the final amount of liquid in the system. The latter quantity was variable, depending on the amount of steam introduced for direct heating purposes.

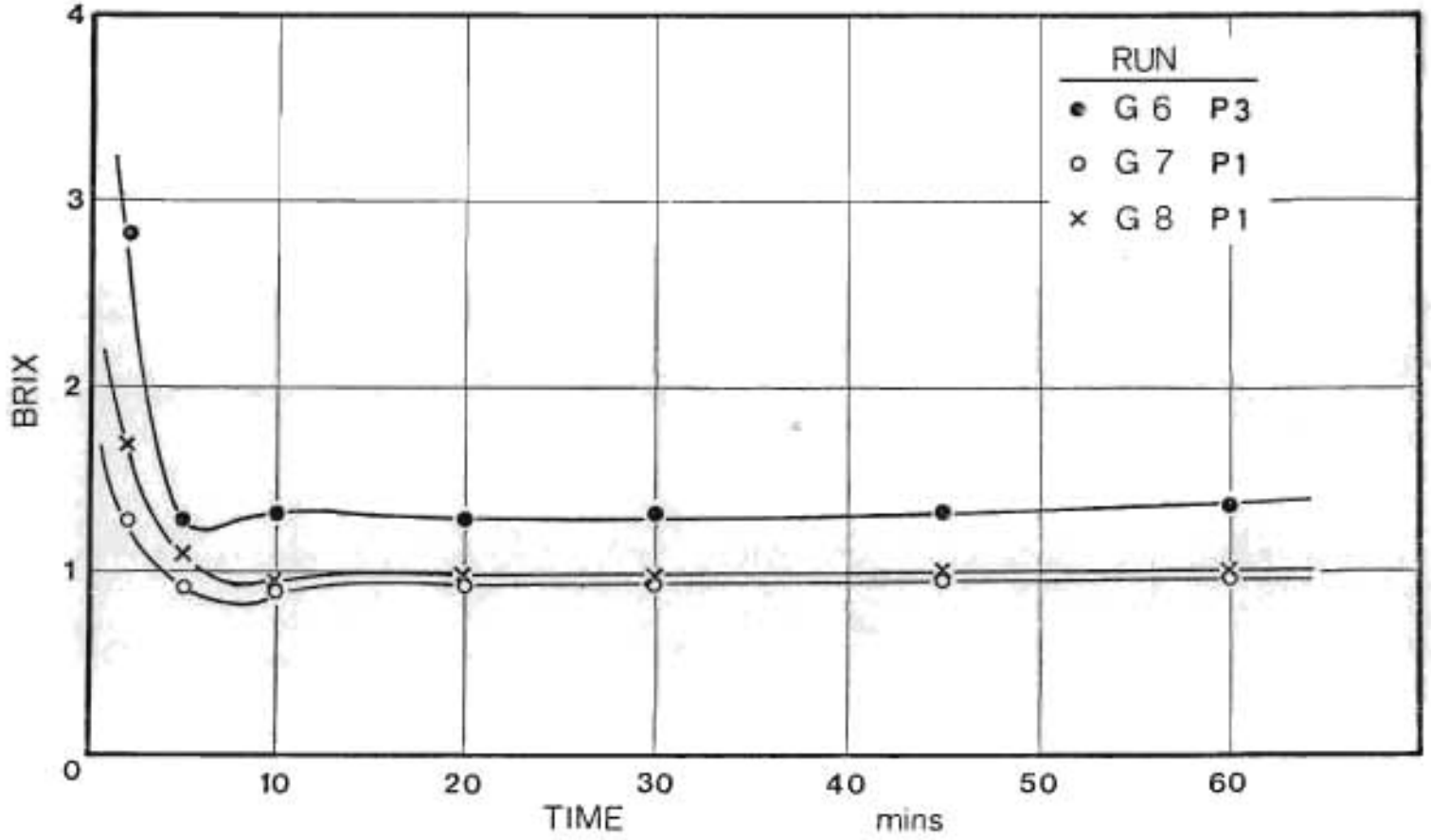


Fig. 5.59. Typical brix-time curves realized in re-circulation runs.

Because the quantity of water in the system varied slightly, impurity concentrations were expressed as percentages or ppm on brix. Results not tabulated in the text are given in Appendix D.

5.8.2. Juice Purity

The apparent purity (defined as the ratio pol/brix) of juice samples was measured for a number of runs. No trend with temperature or preparation could be discerned, in keeping with the findings of Buchanan & Jullienne (1969). In most cases, a slight initial drop in purity was observed, prior to a slight increase and subsequent gradual decrease.

5.8.3. pH

The water used in these tests was obtained from the hot condensate lines, and generally had a pH of just over 8. The shape of the pH-time curves was similar to those for purity, showing an initial decrease to approximately 5.8 before rising to a value of 6.0 or just over. After ten minutes the pH assumed a constant value within 0.1 units. Thus pH values were in the range of full-scale diffuser operating pH.

It was found that if more than one run was carried out in a day, the pH of juices from runs subsequent to the first were significantly lower. This was attributed to a breakdown of sucrose in bagasse which had been left standing for a few hours. This was confirmed by higher reducing sugar levels and is discussed further in the next section.

5.8.4. Reducing Sugars.

Typical measurements of reducing sugar concentrations are shown in Fig. 5.60. There appears to be no trend with extraction temperature; however, inspection of the final pH values shows that high values of reducing sugar/brix are associated with low pH values. The breakdown of sucrose by enzyme action yields reducing sugars, and is associated with the production of organic acids which lower the pH. The two runs with the highest values in Fig. 5.60, runs G5 and G8, manifested the effect described in the previous section, and followed runs G4 and G7 respectively. These

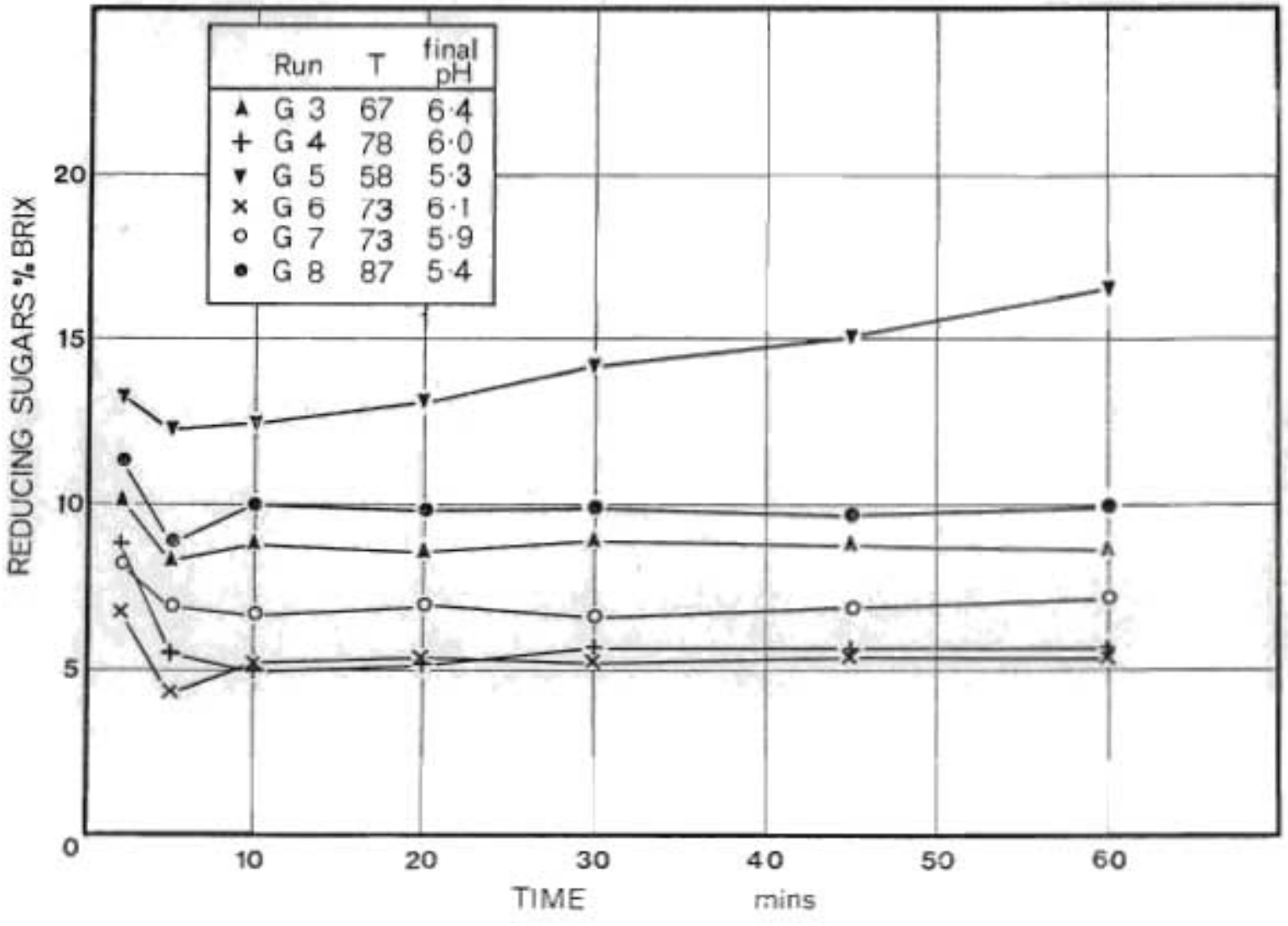


Fig. 5.60 Measured values of reducing sugars % brix as a function of time.

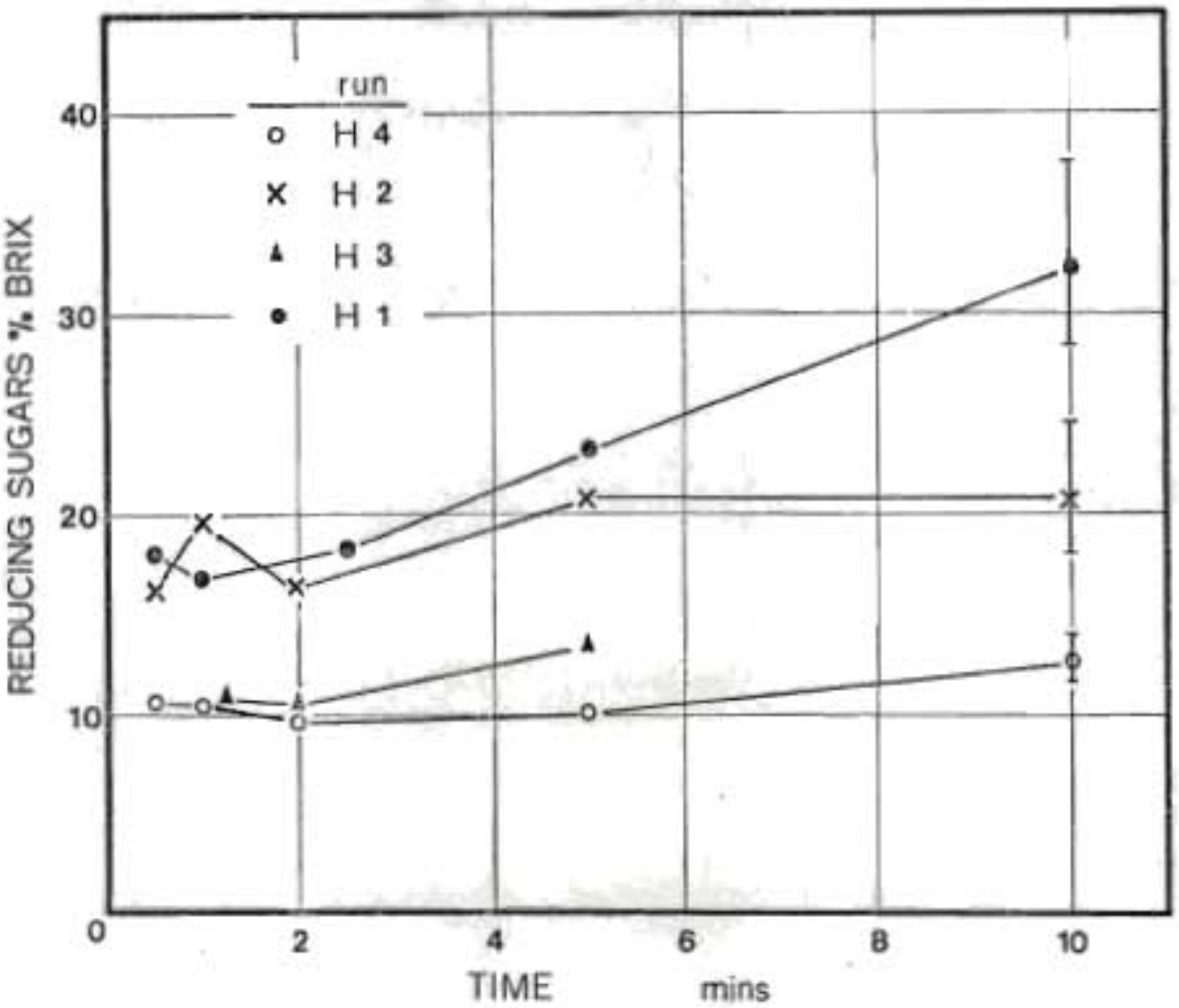


Fig. 5.61. Measured values of reducing sugars % brix in once-through percolation runs. Bagasse preparation P1.

high values are attributed therefore to inversion of sucrose in bagasse which was collected some four to five hours prior to the test. It should be remembered however that the amount of inversion involved is not sufficient to affect brix values significantly, and in no way invalidates brix measurements obtained for comparison with model predictions.

Graham et.al. (1968) have shown that inversion due to enzyme activity can be considerable at temperatures of 65°C, and many times greater than would be expected due to chemical inversion. At higher temperatures, the enzymes are inactivated. All the results shown in Fig. 5.60 appear to be approximately constant after ± ten minutes, except for the run at 59°C. The increase with time shown by this run is presumed to be due to progressive enzymatic destruction of sucrose; this is confirmed by a corresponding increase in pH. It is clear therefore that the operating temperature should be restricted to the range of temperatures $> 65^{\circ}C$ to prevent appreciable enzymatic inversion of sucrose.

In spite of these effects, no significant dependence of the amount of reducing sugars extracted on temperature or preparation is evident. Fig. 5.60 indicates that the initial rate of extraction of reducing sugars is higher than that of sucrose. In order to investigate the rate of extraction of reducing sugars, four of the normal once-through runs were undertaken, with P1 preparation at different temperatures. The results are shown in Fig. 5.61. Once again, the higher values shown by runs H2 and H4 are due to the time lag between collecting the bagasse and the execution of the run.

This shows that the reducing sugar / brix ratio is approximately constant during the initial period, but there is a tendency for the ratio to increase with time. The values at ten minutes include limits on experimental error which may be induced as a result of dividing by low brix values. A marked increase in the reducing sugar ratio is demonstrated, which is directly opposed to the results of Fig. 5.60.

A tentative explanation may be put forward as follows; according to the once-through data, sucrose and reducing sugars are extracted at the same rate initially where the washing displacement mechanism is operative, while at longer times, reducing sugars are

extracted more quickly in the diffusion controlled stages because of their higher molecular diffusivities and small molecular size. The re-circulation data manifest what may be termed a 'chromatographic column effect', where the smaller reducing sugar molecules are preferentially absorbed by the bagasse. This explains the initial drop in reducing sugar / brix ratio and the lower equilibrium value. On the other hand, in the runs where water is continuously passed through the bagasse, the reducing sugar molecules are continuously eluted from the bagasse.

This chromatographic column effect may also be used to explain the variation in the reducing sugar / pol ratio measured by Graham et. al. (1968) on a full-scale diffuser. The value of reducing sugar /pol is higher from the last stage where imbibition water is added (i.e. an elution stage) than in juice from the penultimate stage. Also, the ratio of reducing sugar / pol increases as the feed end is approached, as the equilibrium is displaced at higher reducing sugar concentrations.

This effect may also provide an explanation for the observation that the introduction of diffusion resulted in a lower extraction of reducing sugars than milling (Kulkarni & Unde, 1969). This was realised with a diffuser operating at the natural pH of cane juice, and so it is unlikely that this drop can be due to lower inversion of sucrose.

5.8.5 Inorganic Impurities

In order to obtain a quick measure of the inorganic content of diffuser juice, the specific conductance was measured (more simply, this will be referred to as conductivity). Such measurements naturally only record the presence of dissociated ions; thus no indication of silica or undissociated organic phosphate is obtained.

The conductivity of syrups, molasses and solutions of sucrose crystals has been fairly widely used as a measure of inorganic content (Browne & Zerban, 1941). More recently, its use has been reported in determining the inorganic content of cane juices (Stevenson & Daniels, 1971).

The relation between conductivity and sulphated ash of diffuser juice was investigated by determining both quantities on the same samples of juice. It was found that this relationship could be represented by a straight line, of the following form :-

$$\text{sulphated ash} = 8.6 \times 10^{-5} \times \text{conductivity } (\mu \text{ Siemen/cm}) \qquad (5.72)$$

This is illustrated in Fig. 5.62. and was found to be independent of brix, preparation, temperature, or time at which the sample was obtained, within the range of interest of these variables. This allows an estimate of ash content from the conductivity reading. However Browne and Zerban (1941) point out that such a relation is restricted to the factory or area where it is determined.

All conductivity readings were converted to equivalent ash or 'conductivity ash' using equation (5.72) and expressed as a percentage on brix. Conductivity ash % brix was found to be independent of temperature and degree of preparation. This can be seen in Table 5.27, where all the data obtained are recorded as average values.

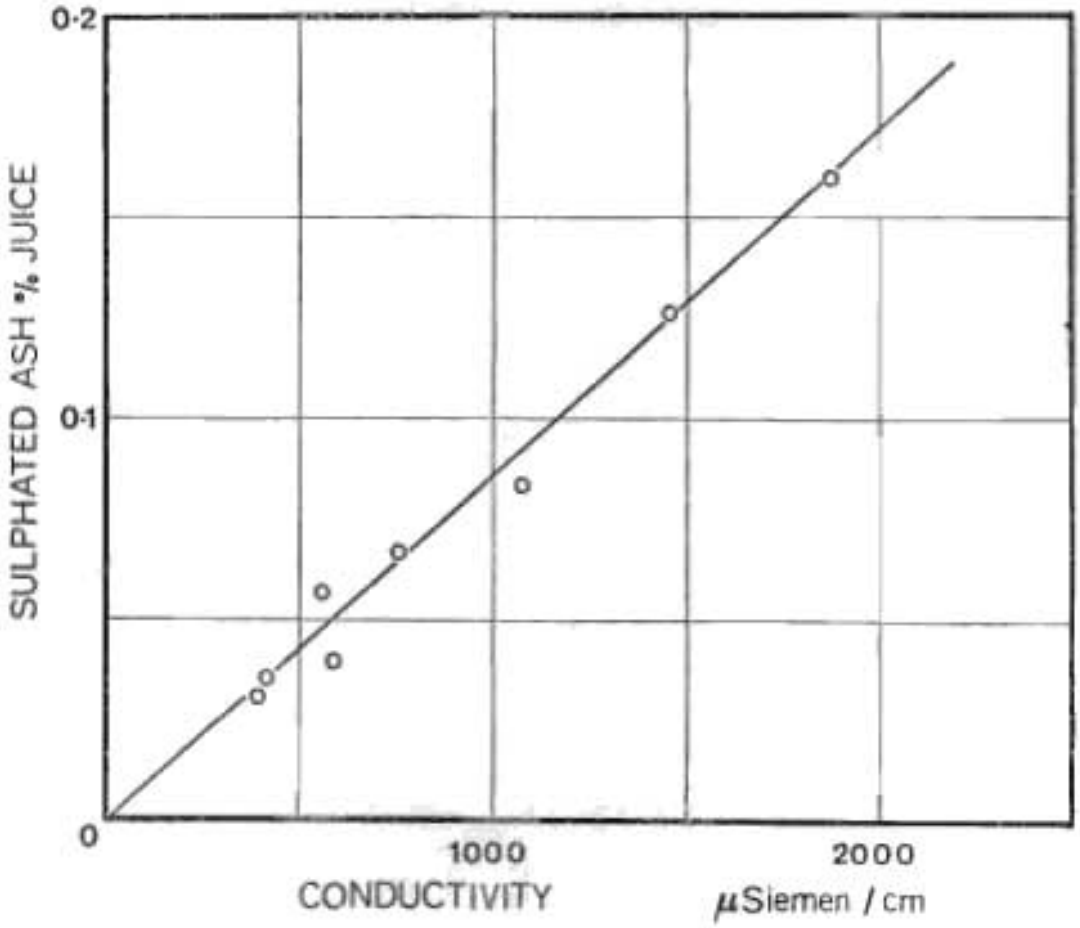


Fig. 5.62. The relation between juice conductivity and sulphated ash content.

Table 5.27 Summary of conductivity ash measurements.

Preparation	Temperature (°C)	Conductivity ash % brix values at t =					
		10 mins.		30 mins.		45 mins.	
		mean	std. dev.	mean	std. dev.	mean	std. dev.
P1	59	4.4	0.1	4.5	0.2	4.6	0.0
P1	69	4.4	0.3	4.5	0.4	4.4	0.4
P1	73	4.5	0.6	4.4	0.7	4.3	0.7
P1	78	4.5	0.4	4.4	0.4	4.4	0.4
P1	87	4.6	0.2	4.4	0.2	4.5	0.2
P2	73	4.3	1.0	4.3	1.0	4.3	1.0
P3	73	4.3	1.0	4.3	1.2	4.3	1.2
All data		4.4	0.5	4.4	0.5	4.4	0.5

The lack of dependence on preparation is more clearly illustrated in Fig. 5.63, which shows the results of 3 runs carried out on subsamples of the same bagasse, with different levels of preparation. This figure also demonstrates the general behaviour of the measured values as a function of time.

The concentrations of some of the major inorganic elements were measured by flame photometry or atomic absorption spectrophotometry. The ratios of concentrations of Na, Mg, K, and Cl to brix were nearly constant throughout the course of a run, although the latter two ions sometimes displayed a slight decrease initially. The ratio of Ca to brix generally showed a slow increase with time. According to Honig (1958), some calcium is associated with the cell walls, and the increase observed with time is indicative of a progressive leaching of this calcium.

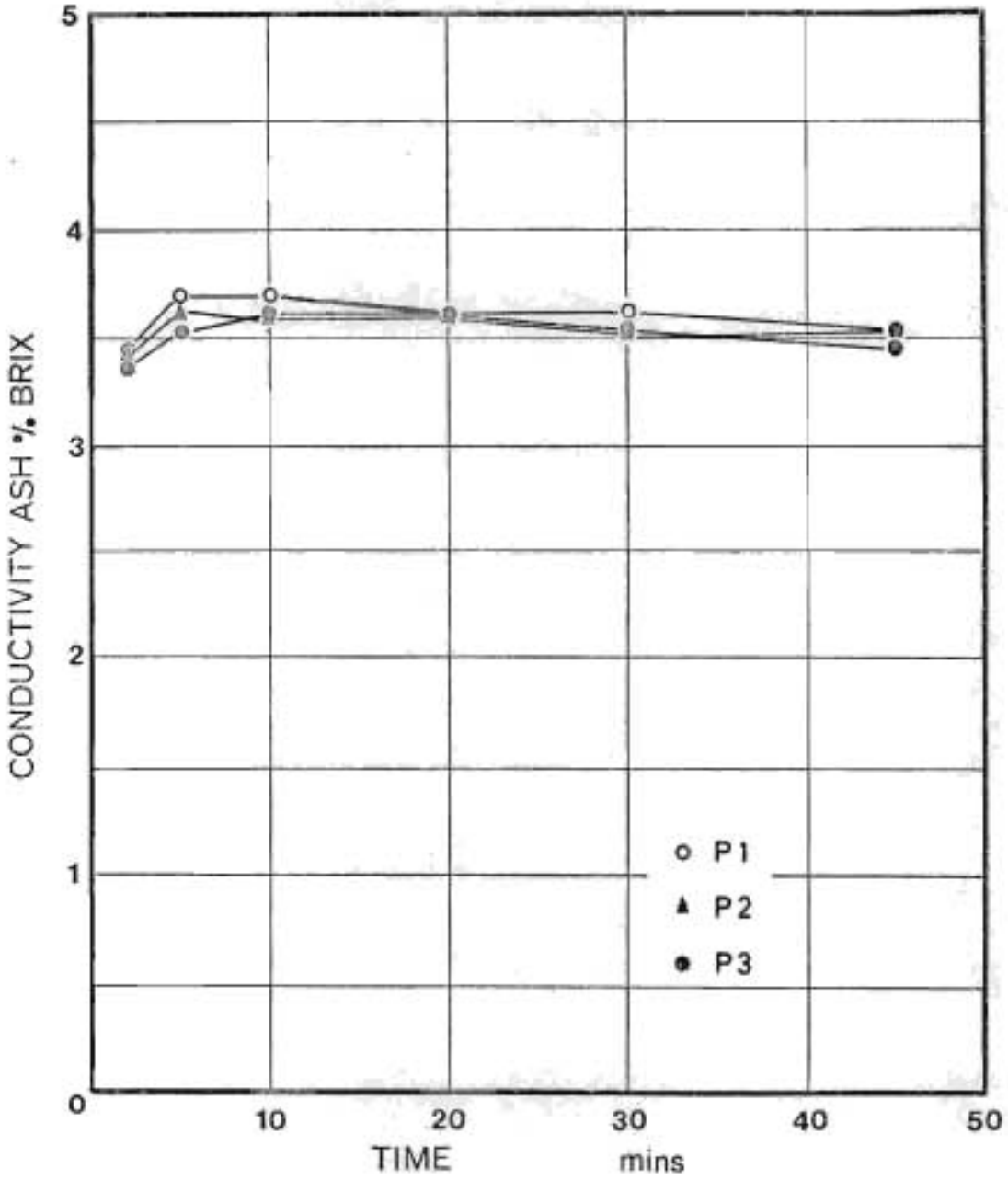


Fig. 5.63 Conductivity ash % brix as a function of time for different levels of preparation.

The concentrations of the individual inorganic species were also found to be independent of temperature. The average values of all measurements made with P1 preparation are shown in Table 5.28.

Table 5.28. Average concentrations of individual inorganic species in diffuser juice as a function of recirculation time. P1 preparation. Concentrations expressed as percentage on brix. No. of data points = 20

	10 mins.		30 mins.		45 mins.	
	mean	std.dev.	mean	std.dev.	mean	std.dev.
K	1.31	0.14	1.28	0.13	1.27	0.12
Cl	0.72	0.08	0.70	0.09	0.69	0.09
Ca	0.21	0.07	0.21	0.07	0.22	0.06
Mg	0.15	0.04	0.16	0.04	0.16	0.04
Na	0.06	0.03	0.06	0.03	0.06	0.03

With the exception of some measurements reported by Douwes-Dekker (1952), to the author's knowledge, no similar measurements in mixed juice of milling factories have been reported in South Africa. Douwes-Dekker reported measurements of calcium and magnesium in mixed juice, which show order of magnitude agreement with those in Table 5.28.

However, Honig (1958) gives an extensive table of measurements of individual inorganic constituents of mixed juice in Java. The order of decreasing concentrations in Table 5.28 should be compared with that shown by the Java data i.e. K, Na, S, Cl, Si, P, Mg, Ca, Fe. Of interest is the low concentration of Na in diffuser juice. Apart from this, the values given in Table 5.28 show order of magnitude agreement with the Java data.

The degree of bagasse preparation appeared to have little effect on most of the elements measured, although the concentrations referred to brix values generally showed a decrease with finer bagasse, due to the higher brix values. However, the Ca /brix ratio generally showed an increase with degree of preparation. This is illustrated in Figure 5.64, where the ratio of Ca / K concentrations is plotted to eliminate the effect of brix values. This increase with finer preparation is consistent with the existence of calcium in the cell wall material; the greater the damage to the cell wall material, the more accessible is calcium associated with the cell walls to the leaching juice.

Thus excessive damage to the fibre can result in undesireable side effects; as regards the extraction of inorganic materials, these effects are likely to be small.

Fig.5.63 is typical of the shape of the conductivity ash % brix -time curves. This suggests that initially sucrose is extracted at a relatively higher rate, but that after a short time, equilibrium between juice and bagasse is established. Ash % brix values measured in once-through runs are shown in Fig. 5.65. This shows that the initial increase suggested by the

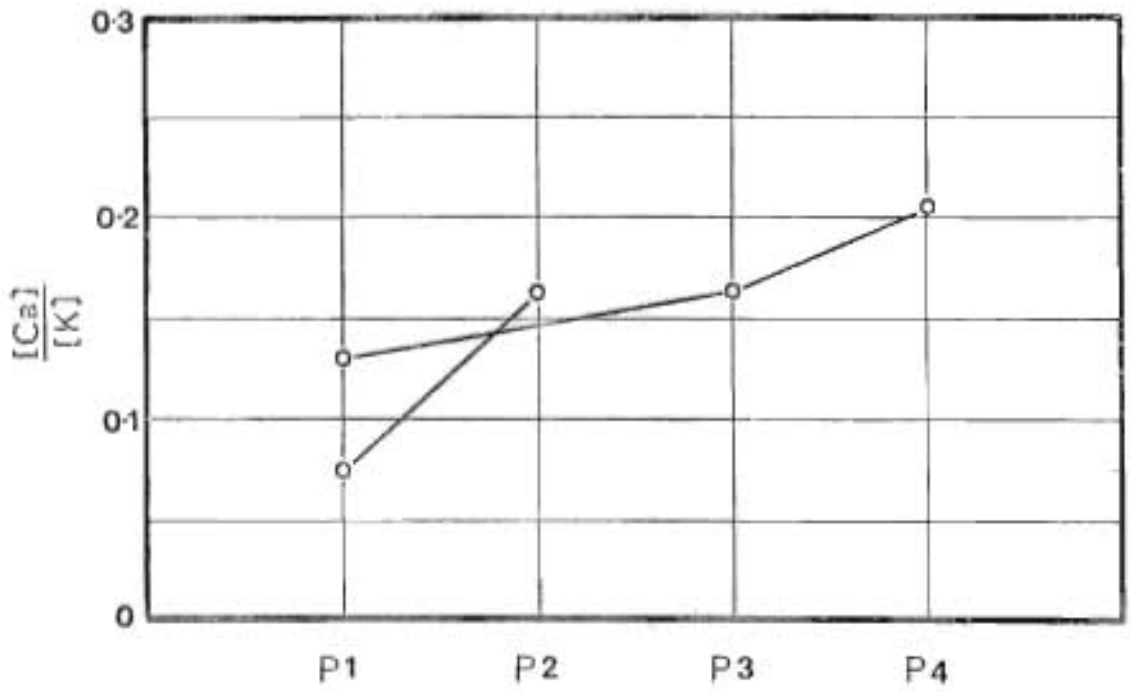


Fig. 5.64. The ratio of concentrations of calcium to potassium after 45 minutes re-circulation time, as a function of degree of bagasse preparation.

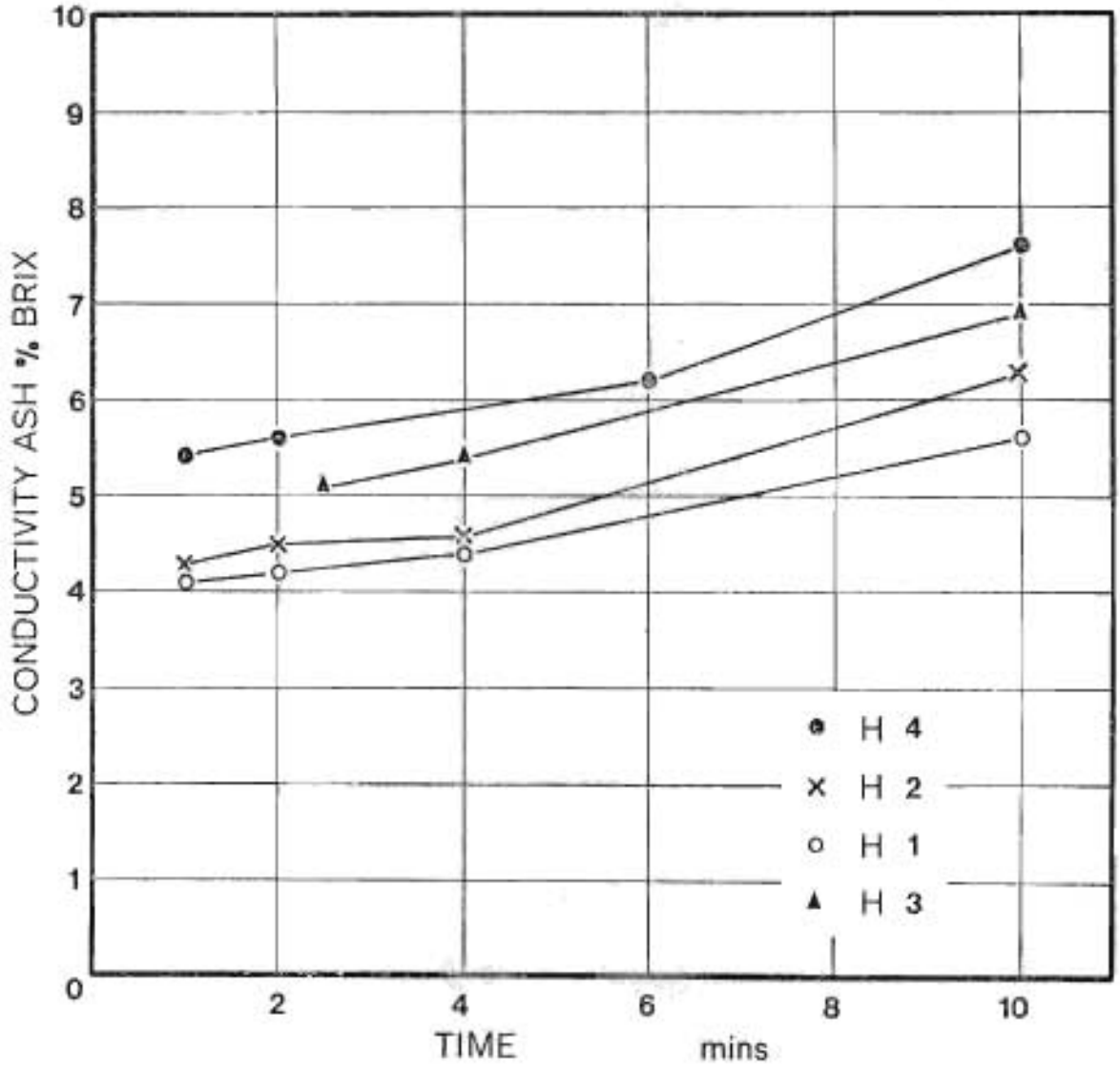


Fig. 5.65. Conductivity ash % brix as a function of time in once-through percolation runs. Bagasse preparation P 1.

re-circulation data is sustained. Thus inorganic materials are extracted at a relatively faster rate at progressively longer times, an effect which is obscured in re-circulation runs where equilibrium between juice and bagasse is attained. The higher diffusion coefficients of the small inorganic ions manifest as a higher rate of extraction of these species as extrac - tion by diffusion becomes important.

Thus in a full scale diffuser, extraction of inorganic materials may reasonably be expected to depend on the time of contact of juice and bagasse. Within each stage, however, a close approach to an equilibrium condition between inorganic material in juice and bagasse can be expected. The equilibrium value will naturally depend on the initial inorganic content of the cane, which is likely to have the greatest effect on the quantity of inorganic material in diffuser draft juice.

5.8.6 Starch

Different varieties of cane contain widely different amounts of starch (Wood, 1962), and futhermore, the starch content depends on the age, growth conditions, etc., of the cane. A wide variation in measured starch contents of re-circulating juice confirmed this, and necessitated that the effect of temperature or cane preparation on starch extraction be demonstrated using subsamples of the same bagasse. This necessitated collection of a larger sample from the milling train, which proved advantageous in that this sample collected over a longer time was more representitive of 'average' bagasse, which tended to mask inter-consignment variations.

The execution of four runs using subsamples of the same sample of well-mixed bagasse required that the bagasse for the fourth run experience a time lag of the order of six hours before processing. Since virtually all starch is present in granular form (Manners, 1968), it is unlikely that degradation of starch occurred to any significant extent. In order to eliminate any possible effect, the levels of temerature and preparation were varied in a random way for each set of runs.

The effect of temperature on the starch content of the diffuser juice after re-circulation for thirty and forty-five minutes is shown in Fig.5.66. A striking increase in the amount of starch extracted at temperatures greater than 73°C is demonstrated; the amount of starch extracted rises roughly six-fold from 73°C to 88°C. Boyes (1960) reported a number of results of analyses of starch in juice from milling. From his results, it appears that the concentration of starch in mixed juice generally lies in the range 2 000 to 2 500 ppm on brix. Comparison with Fig.5.66 shows that at lower temperatures, the level of starch extraction is very much less than obtained by milling, while at high temperatures, starch extraction is of the same order. Because of the adverse effect of starch on sugar recovery, there is a strong incentive for maintaining diffuser operating temperatures below 75°C.

In contrast to the extraction of reducing sugars and inorganic materials, extraction of starch is not a diffusion-controlled phenomenon. The sharp increase in starch extraction with temperature is due to gelatinisation of starch granules. Although gelatinisation occurs to some extent at temperatures as low as ± 60°C (Collison, 1968), enzymes in the juice can effectively remove this starch as fast as it appears. At temperatures of 70° to 80°C, the rate of enzymatic destruction falls off rapidly in low brix juices (Smith, 1970); together with a rapid increase in gelatinisation, this results in excessive extraction of starch.

Graham et. al. (1968) reported an increase in starch extracted between 60° and 80°C in a full scale diffuser, of similar magnitude to that shown in Fig. 5.66. However, this study has succeeded in pinpointing more closely the temperature above which excessive extraction of starch occurs.

The effect of preparation on starch extraction is illustrated in Fig.5.67. Solid lines join points obtained using bagasse originating from the same sample. These results were obtained with a re-circulation juice temperature of 73°C, i.e. close to the maximum temperature consistent with low starch extraction. It appears that preparations finer than 1st mill bagasse

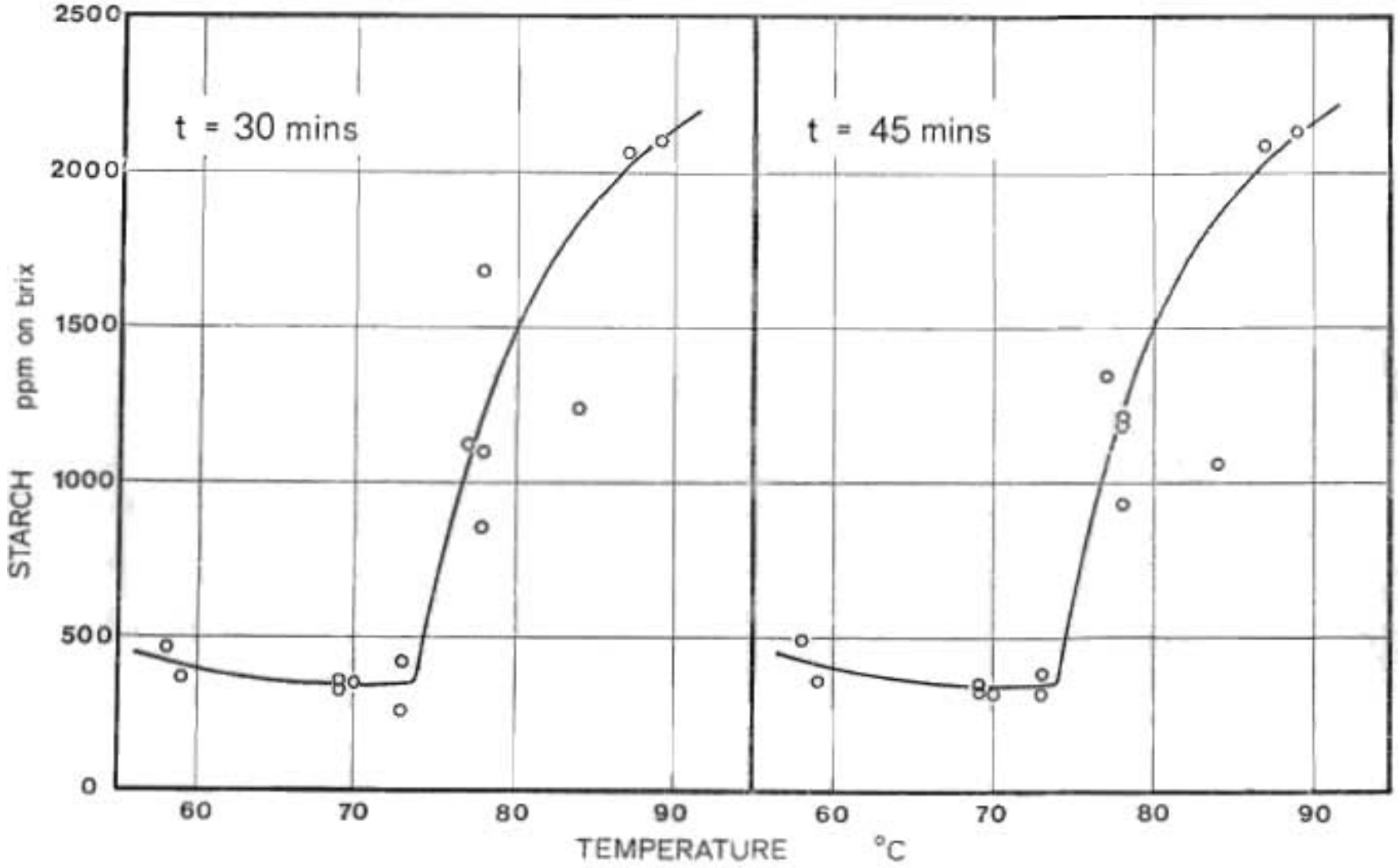


Fig. 5.66. Extraction of starch as a function of temperature. Values shown for 2 different recirculation times. Bagasse preparation P1.

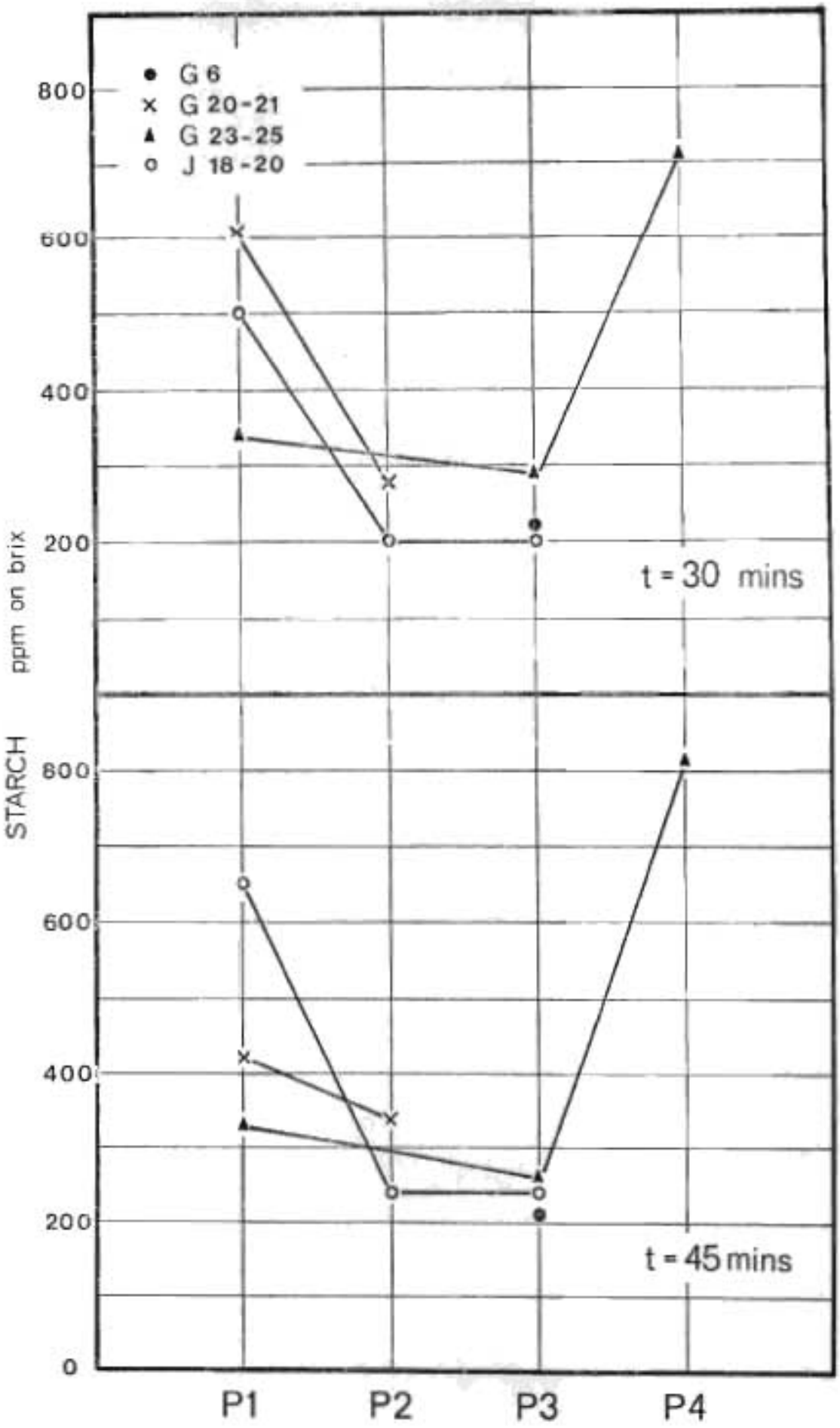


Fig. 5.67. Effect of level of preparation on the extraction of starch. Extraction temperature 73° C.

actually result in slightly lower starch extraction. However, one data point representing preparation P4 suggests that this trend is not maintained with excessively fine bagasse. This material is considered to be finer than would ever be achieved in practice; thus for all practical preparations, it would seem that, as far as starch extraction is concerned, there is no reason to limit the degree of preparation.

5.8.7 Discussion

In keeping with the conclusions reached by other authors (Buchanan & Jullienne, 1969; Graham et. al., 1968) no significant effect of temperature on juice purity or extraction of inorganic materials was found; preparation was also found to have no significant effect. Measurement of some of the major inorganic constituents has shown that potassium is the major inorganic species in diffuser juice, as also in mill juices (Honig, 1958). Other inorganic species measured occurred in the following order of decreasing concentration : Cl, Ca, Mg, Na. However an increase in the amount of calcium extracted with more finely prepared cane has demonstrated how the degree of preparation influences the availability of cell wall constituents for extraction.

Destruction of sucrose in bagasse prior to some of the test runs obscured any possible minor trends in extraction of reducing sugars with the operating variables. In any event, Graham et. al., (1968) have shown that conditions of pH and temperature within a diffuser have a much greater effect on the amount of reducing sugars in diffuser juice, as these variables affect the enzymatic breakdown of sucrose to reducing sugars. Results of this study show that operation at temperatures below 65^{o}C can result in considerable breakdown of sucrose. Thus low temperatures should not be employed unless active steps are taken to ensure that the pH remains well above 6.0

Observations on the rate of extraction of inorganic impurities and reducing sugars show that these substances are extracted at a faster rate during the diffusion-controlled stages of extraction. This is attributed to their smaller molecular size. Evidence has been put forward, however, to suggest that reducing sugars may be preferentially absorbed by bagasse.

A very marked increase in starch extraction with temperature has been demonstrated, which indicates that diffusers operated at temperatures not greater than 73°C can effect a considerable reduction in the amount of starch extracted. However, it appears that the amount of starch in diffuser juice is largely dependent on the amount of starch originally present in the cane, which can vary within wide limits.

Taken in conjunction with the survey of previous work (section 2.8), this investigation shows that no more of the recognised impurities are extracted in diffusion than in milling, and that some impurities are actually extracted to a lesser extent. However, recovery of sucrose depends not only on the amounts of the various impurities extracted; it is possible that although the same chemical impurities are extracted in milling and diffusion, the physical natures of the impurities may be different (e.g. colloidal or non-colloidal), which will influence the degree of removal of the impurities.

In general, extraction of the two major impurities, inorganics and reducing sugars, is independent of temperature and degree of preparation, and is roughly the same as in milling. In this respect, it would appear that extraction of these impurities is not limiting on the process operating variables. The degree of preparation generally has little effect on the extraction of impurities, although excessively fine preparations can result in higher levels of impurity extraction. The operating temperature can have an important influence on the extraction of organic impurities, particularly starch.

CHAPTER 6

MODELLING OF FULL-SCALE DIFFUSERS.

Pilot plant tests showed that the 3 parameter model is capable of accurately representing extraction in the diffusion process. It remains to apply the model to a full-scale diffuser of the moving-bed type, so that the model can be utilized for design or optimization purposes.

The purpose of this chapter is to show how the model can be formulated and applied to a moving-bed diffuser. A brief comparison between model predictions and actual observed behaviour of a diffuser is made; a comprehensive appraisal of the model predictions is beyond the scope of the present study. Attention is drawn to a number of additional factors which may influence diffuser performance and which may require incorporation into the model.

6.1 APPLICATION OF THE MODEL TO A MOVING-BED DIFFUSER.

6.1.1 Model Formulation for a Single Stage.

In the first instance the model is formulated for a single stage within the diffuser. Consider the nth stage shown in Fig. 6.1. Juice percolating through the bed is displaced in the x-direction due to the movement of the bagasse. In order to formulate the model it is necessary to make the following assumptions:

1. Steady-state operation, so that all quantities concerned are independent of time.
2. Liquid is uniformly applied over the top of a stage, and flows in plug flow through the bed.

In addition, the same assumptions regarding the mechanism of extraction used in the pilot plant model are made in this case.

Mass balances over an element within the bed of height dz, length dx and unit width yield the following differential equations:
For the percolating juice,

$$- L \frac{\delta C_j}{\delta z} - \frac{L}{\tan \psi} \frac{\delta C_j}{\delta x} + K_1 (C_{b1} - C_j) + K_2 (C_{b2} - C_j) = 0 \qquad (6.1)$$

and for the juice in bagasse,

$$- \alpha H v \frac{\delta C_{b1}}{\delta x} = K_1 (C_{b1} - C_j) \qquad (6.2)$$

$$- (1 - \alpha) H v \frac{\delta C_{b2}}{\delta x} = K_2 (C_{b2} - C_j) \qquad (6.3)$$

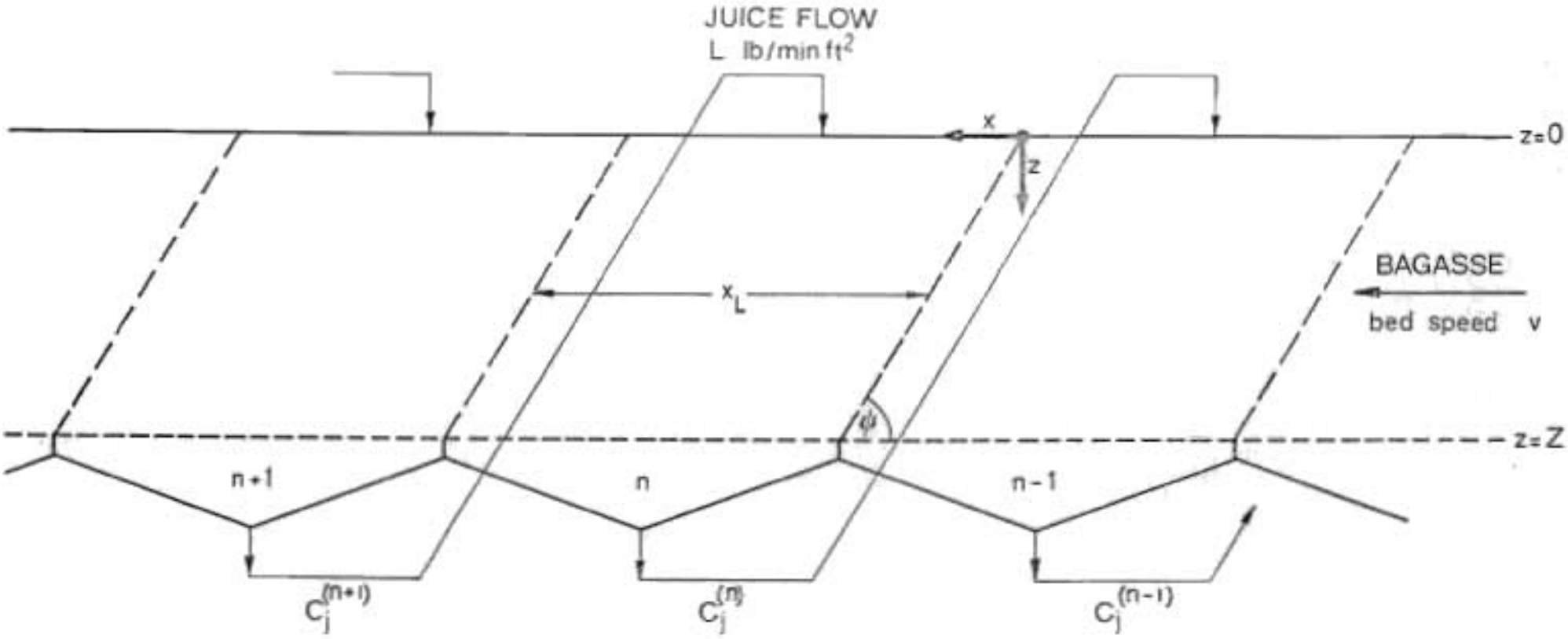


Fig. 6.1 Basis for formulation of model of a single stage in a moving-bed diffuser.

Introducing the dimensionless variables:

$$N = \frac{K_1 z}{L} \tag{6.4}$$

$$Y = \left(x - \frac{z}{\tan \varphi} \right) \frac{K_1}{H v} \tag{6.5}$$

$$\beta = \frac{K_2}{K_1} \tag{6.6}$$

into equations (6.1) to (6.3) leads to:

$$\frac{\delta C_j}{\delta N} = C_{b1} + \beta C_{b2} - C_j (1 + \beta) \tag{6.7}$$

$$- \alpha \frac{\delta C_{b1}}{\delta Y} = C_{b1} - C_j \tag{6.8}$$

$$- (1 - \alpha) \frac{\delta C_{b2}}{\delta Y} = \beta (C_{b2} - C_j) \tag{6.9}$$

This treatment assumes that the concentrations of percolating juice and juice in bagasse vary with both distance from the top of the bed and distance along the bed, within a stage. The boundary conditions are given by the concentration of juice pumped onto the nth stage from the (n+1)th tray, and the concentration of juice in bagasse leaving the (n-1)th stage. The former condition may be expressed as:

$$\text{at } N = 0,\ C_j = C_j^{(n+1)}, \quad 0 \leqslant Y \leqslant \frac{K_1 x_L}{H v} \tag{6.10}$$

The superscript is used to denote the stage number. The second boundary condition is more complicated since C_{b1} and C_{b2} depend on both Y and N. This makes the situation over-complicated. If however C_{b1} and C_{b2} are considered to depend on Y only within a stage, the boundary condition is considerably simplified. Then:

$$\text{at } Y = O \quad C_{b1} = C_{b1}^{(n-1)} \tag{6.11}$$

$$\text{at } Y = O \quad C_{b2} = C_{b2}^{(n-1)} \tag{6.12}$$

Since the variation in C_b across the length of the diffuser is much greater than the variation with bed height within a stage, this assumption is not likely to affect the results significantly. In this case, C_j in equations (6.8) and (6.9) must be replaced by an average value; these 2 equations must then be replaced by the following integro-differential equations:

$$-\alpha \frac{d\,C_{b1}}{d\,Y} = C_{b1} - \frac{1}{N} \int_o^N C_j\, dN \tag{6.13}$$

$$-(1-\alpha)\frac{d\,C_{b2}}{d\,Y} = \beta\, C_{b2} - \frac{\beta}{N} \int_o^N C_j\, dN \tag{6.14}$$

The method of solution of the set of equations (6.7), (6.13) and (6.14) together with boundary conditions (6.10) to (6.12) is given in appendix E. The final solution only is given below:

$$C_j = f_o\, C_j^{(n+1)} + f_1\, C_{b1}^{(n-1)} + f_2\, C_{b2}^{(n-1)} \tag{6.15}$$

$$C_{b1} = e_o\, C_j^{(n+1)} + e_1\, C_{b1}^{(n-1)} + e_2\, C_{b2}^{(n-1)} \tag{6.16}$$

$$C_{b2} = d_o\, C_j^{(n+1)} + d_1\, C_{b1}^{(n-1)} + d_2\, C_{b2}^{(n-1)} \tag{6.17}$$

$$\text{where } a_o = \frac{\gamma m_2 + A}{\gamma (m_1 - m_2)} \tag{6.18}$$

$$a_1 = \frac{1 - A}{\gamma (m_1 - m_2)(1 + \beta)} \tag{6.19}$$

$$a_2 = \frac{\gamma m_2 (1+\beta) + 1 + A\beta}{\gamma (m_2 - m_1)(1+\beta)} \tag{6.20}$$

$$b_o = \frac{\gamma m_1 + A}{\gamma (m_2 - m_1)} \tag{6.21}$$

$$b_1 = -a_1 \tag{6.22}$$

$$b_2 = \frac{\gamma m_1 (1+\beta) + 1 + A\beta}{\gamma (m_1 - m_2)(1+\beta)} \tag{6.23}$$

$$d_o = 1 + a_o e^{m_1 Y} + b_o e^{m_2 Y} \tag{6.24}$$

$$d_1 = a_1 e^{m_1 Y} + b_1 e^{m_2 Y} \tag{6.25}$$

$$d_2 = a_2 e^{m_1 Y} + b_2 e^{m_2 Y} \tag{6.26}$$

$$e_o = 1 + \frac{a_o b_2}{a_1} e^{m_1 Y} + \frac{b_o a_2}{b_1} e^{m_2 Y} \tag{6.27}$$

$$e_1 = b_2 e^{m_1 Y} + a_2 e^{m_2 Y} \tag{6.28}$$

$$e_2 = \frac{a_2 b_2}{a_1} e^{m_1 Y} + \frac{a_2 b_2}{b_1} e^{m_1 Y} \tag{6.29}$$

$$f_o = AN(e_o + \beta d_o) + e^{-(1+\beta)N} \tag{6.30}$$

$$f_1 = AN(e_1 + \beta d_1) \tag{6.31}$$

$$f_2 = AN(e_2 + \beta d_2) \tag{6.32}$$

$$\begin{matrix} m_1 \\ m_2 \end{matrix} \} = \frac{1}{2\,\alpha\,\gamma} \{ -(1 + \gamma A + \alpha A \beta) / (1+\beta) \pm \left[\frac{(1 + \gamma A + \alpha A \beta)^2}{(1+\beta)^2} - 4\,\alpha\,\gamma\,A \right] \} \quad (6.33)$$

$$\gamma = \frac{1 - \alpha}{\beta} \quad (6.34)$$

$$A = \frac{1}{(1+\beta)N} \left(1 - e^{-(1+\beta)N}\right) \quad (6.35)$$

Equation (6.15) relates the value of C_j to position within the bagasse bed. The quantity of interest however is the concentration of juice in the nth tray, $C_j^{(n)}$; this can be evaluated by integrating equation (6.15) over the range $O \leq Y \leq (x_L - Z/\tan\psi)\, K_1/Hv$. Note that this assumes that all juice entering stage n finds its way into the correct tray (this implies that the beginning of the nth tray is positioned at $x = Z/\tan\psi$). If this were not the case, the range of integration would be different.

Thus:

$$C_j^{(n)} = \frac{1}{Y} \int_o^Y C_j \, dY \quad (6.36$$

$$= g_o\, C_j^{(n+1)} + g_1\, C_{b1}^{(n-1)} + g_2\, C_{b2}^{(n-1)} \quad (6.37)$$

where:

$$g_o = 1 + \frac{A\,N\,a_o}{m_1 Y} \frac{(1+\beta)(\gamma m_1 + 1)}{1 - A} (e^{m_1 Y} - 1)$$

$$+ \frac{A\,N\,b_o}{m_2 Y} \frac{(1+\beta)(\gamma m_2 + 1)}{1 - A} (e^{m_2 Y} - 1) \quad (6.38)$$

$$g_1 = \frac{A\,N(1+\beta)}{Y(1-A)} \left[\frac{a_1}{m_1} (\gamma m_1 + 1)(e^{m_1 Y} - 1) + \frac{b_1}{m_2} (\gamma m_2 + 1)(e^{m_2 Y} - 1) \right]$$

$$(6.39)$$

$$g_Z = \frac{AN(1+\beta)}{Y(1-A)} \left[\frac{a_2}{m_1} (\gamma m_1 + 1)(e^{m_1 Y} - 1) + \frac{b_2}{m_2} (\gamma m_2 + 1)(e^{m_2 Y} - 1) \right] \quad (6.40)$$

6.1.2 Overall Mass Balance.

A mass balance over the stages of interest ($1 \leq n \leq J$) can be drawn up. Referring to Fig. 6.2, this can be stated as:

$$L x_L w C_j^{(J+1)} + \frac{M_b H}{q} C_b^{(o)} = L x_L w C_j^{(1)} + \frac{M_b H}{q} C_b^{(J)} \quad (6.41)$$

where C_b = concentration of juice in bagasse defined by:

$$C_b = \alpha C_{b1} + (1 - \alpha) C_{b2} \quad (6.42)$$

M_b = fibre throughput (lb fibre/min)
q = fibre packing density (lb fibre/ft^3)
w = width of diffuser (ft)
x_L = length of 1 stage (ft)

Equation (6.41) may be re-arranged to give:

$$C_b^{(J)} = C_b^{(o)} - \frac{L x_L w q}{M_b H} (C_j^{(1)} - C_j^{(J+1)}) \quad (6.43)$$

6.1.3 Calculation Procedure.

For the purposes of comparing the model predictions with actual diffuser operating data, attention will be confined to those stages of a diffuser after the maceration stage and up to and including the stage where the press-water is introduced.

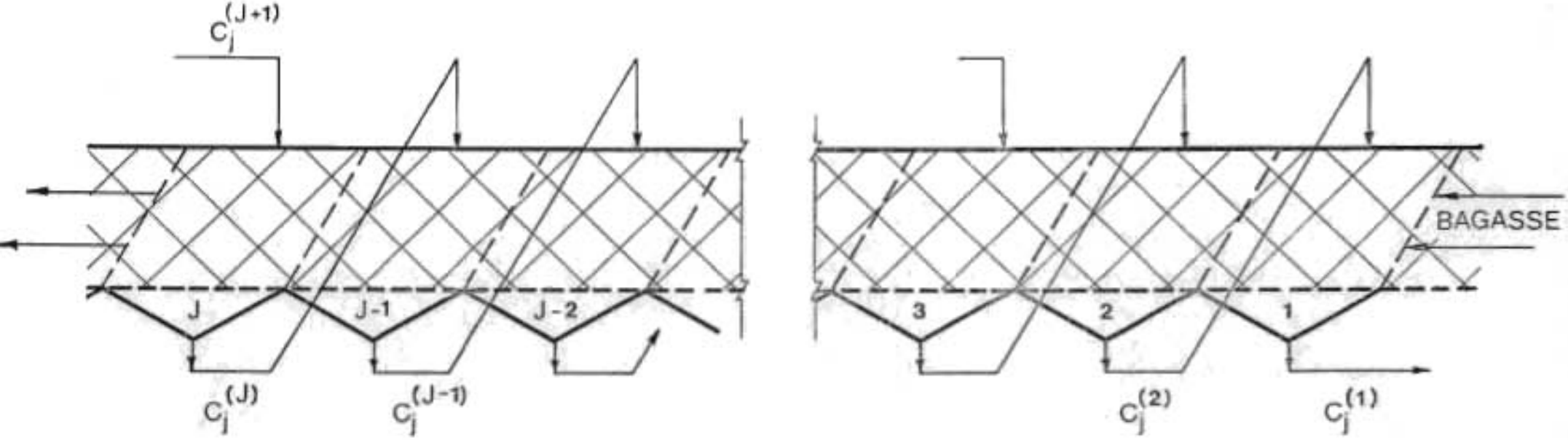


Fig. 6.2 Schematic representation of a moving bed diffuser.

These stages constitute the major part of a diffuser. Beyond the press-water stage, the flow rate through the bed is lower, while in the maceration stage the physical geometry is different. The model can be applied to both these regions as well, but for the purposes of simplicity are neglected here.

Equation (6. 37) relates the concentration of the juice leaving a stage to the values of C_j, C_{b1} and C_{b2} entering the stage. A stage-by-stage calculation procedure is necessary; the result of the calculations must however also satisfy the overall material balance, equation (6. 41)

As a basis for comparison, predicted values of C_j in each tray can be compared with observed values. Since there are 3 dependent variables (C_j, C_{b1} and C_{b2}), 3 boundary values are required. The observed values of $C_j^{(1)}$ $C_j^{(2)}$ and $C_j^{(J+1)}$ are chosen for this purpose.

The calculation procedure is as follows:

1. Assume a value of $C_b^{(o)}$.
2. Calculate $C_{b1}^{(o)}$ and $C_{b2}^{(o)}$ from the following equations, derived from equations (6. 42) and (6. 37):

$$C_{b1}^{(o)} = \frac{C_j^{(1)} - g_o C_j^{(2)} - g_2 C_b^{(o)}/(1-a)}{g_1 - g_2 a/(1-a)} \qquad (6.44)$$

$$C_{b2}^{(o)} = \frac{1}{1-a} \left(C_b^{(o)} - a C_{b1}^{(o)} \right) \qquad (6.45)$$

3. From equations (6. 16) and (6. 17), calculate $C_{b1}^{(1)}$ and $C_{b2}^{(1)}$.
4. From equation (6. 37), calculate $C_j^{(3)}$
5. Calculate $C_{b1}^{(2)}$ and $C_{b2}^{(2)}$ from equations (6. 16)and (6. 17).

6. Continue the calculations stage-by-stage until stage J is reached.

7. Check that the material balance equation (6.43) is satisfied. If not, a new value of $C_b^{(o)}$ must be assumed.

8. Repeat steps (1) to (7) until the overall mass balance is satisfied within a given tolerance. The calculated and observed values of $C_j^{(J+1)}$ should then agree within the specified tolerance.

6.2 COMPARISON WITH DIFFUSER PERFORMANCE.

The calculation routine was programmed on an ICL 1903-A computer. Input to the calculation routine consisted of values of x_L, w, M_b, H, Z, v , L, values of the model parameters, and a set of observed values of C_j. The liquid flow rate, L, was computed from imbibition and press-water flow rates. The value of H was determined from the static holdup (less brix free water), calculated from equation (5.40), while values of the model parameters were computed from the correlations given in section 5.6, equations (5.60), (5.67) and (5.71).

The operating data used in comparing the model predictions with the observed performance of a moving-bed diffuser was obtained from the Empangeni diffuser installation, details of which are given by van der Riet and Renton (1971). Average data for one week's operation, selected at random, were used; most values used represent the averages of hourly analyses made throughout a week. Average data for a week were used in order to eliminate the effect of short-term fluctuations due to the large random variations observed over short time periods.

The results of this comparison are shown in the computer print-out reproduced in Fig. 6.3, and are illustrated graphically in Fig. 6.4. All quantities used in the calculations are shown in Fig. 6.3 with the exception of x_L and w; these values are 10.76 ft and 15.75 ft respectively. In addition, a value of S representitive of the bagasse processed (4000 mm^2/g) and a DI of first mill bagasse of 68.3 were used in the correlations for the evaluation of model parameters.

It can be seen that good correspondence between observed and predicted values of C_j is obtained. It should be noted that predicted and observed values of C_j are forced to have the same values for stages 1, 2, and 11, in which case the observed value (3) overprints the predicted value (2). From Fig. 6.3 it can be calculated that an extraction of 67.5% of the brix entering the 1st stage is achieved in the 10 stages of interest. The fact that C_{b1} in the bagasse entering stage 1 is lower than C_{b2} shows that some extraction has been effected in the maceration stage.

DIFFUSER SIMULATION

DATE OF THIS PRINT 23/05/72 AT TIME 11/38/15

IMBIBITION PER CENT FIBRE 30%.

PRESS WATER PER CENT FIBRE 350.

FLOW RATE 3.65 GAL/MIN,SQ.FT

FIBRE THROUGHPUT 56.80 TONS/HR

BED HEIGHT 3.91 FT

BED VELOCITY 2.90 FT/MIN

JUICE HOLDUP IN BAGASSE 3.18 LB/LB,FIBRE

FIBRE DENSITY 4.54 LB/FT**3

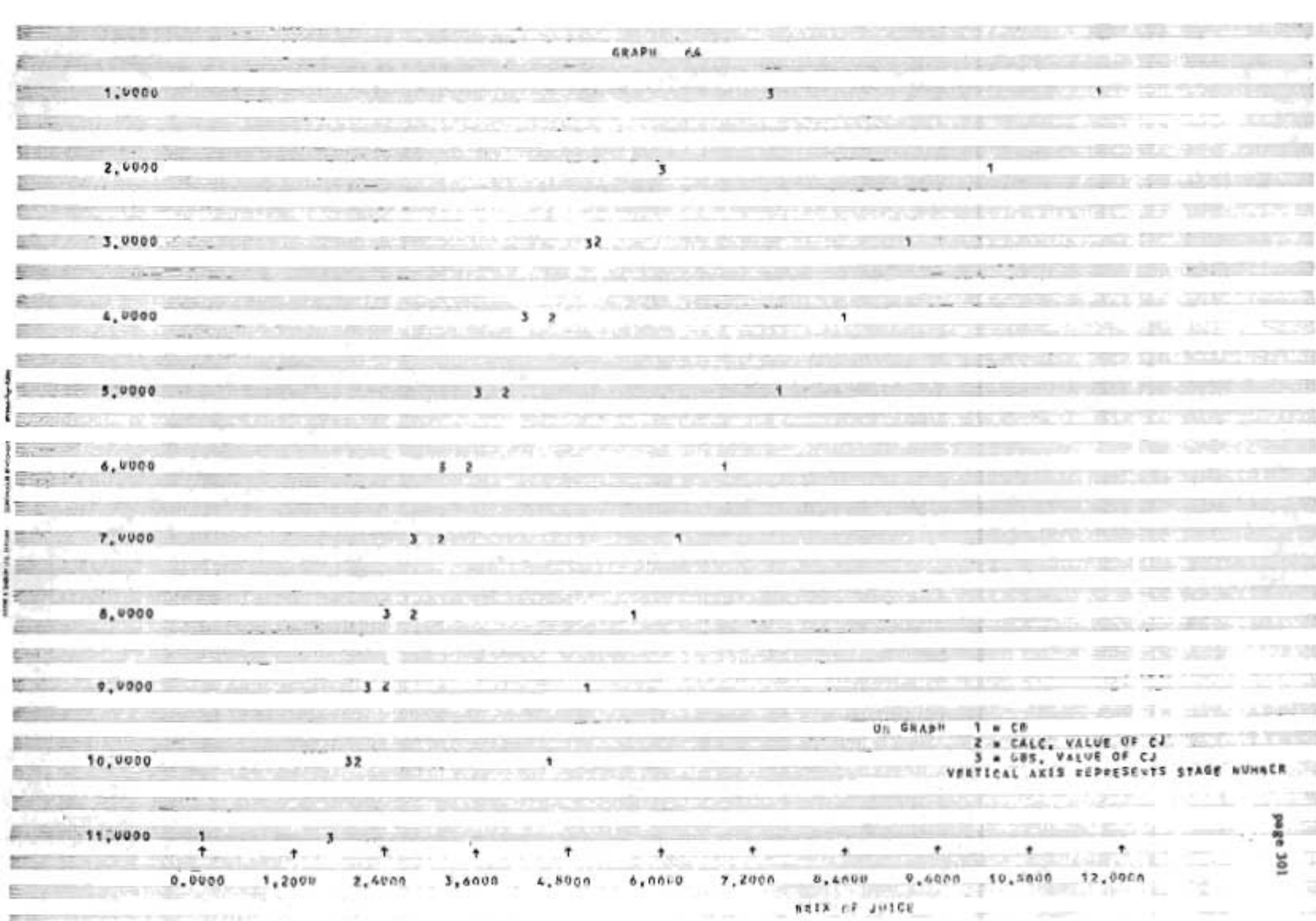
FLOW RECIRCULATION 0.000

MASS BALANCE OVER STAGES 1 TO 10

K1 = 4.13 LB/MIN,FT**3 K2 = 0.518 LB/MIN,FT**3 ALPHA = 0.413

STAGE	OBS. CJ	CALC. CJ	Cn1	Cn2	Cn
0			12.35	15.35	14.11
1	7.50	7.50	8.26	14.26	11.79
2	6.10	6.10	6.37	13.15	10.15
3	5.10	5.23	5.31	12.09	9.29
4	4.30	4.60	4.60	11.19	8.41
5	3.60	4.07	4.04	10.16	7.63
6	3.20	3.66	3.56	9.20	6.93
7	2.80	3.17	3.14	8.49	6.27
8	2.40	2.78	2.76	7.72	5.67
9	2.20	2.41	2.38	7.02	5.10
10	2.00	2.07	2.14	6.36	4.56
PW	1.76	1.76			

Figure A.3


GRAPH 64
1.0000
2.0000
3.0000
4.0000
5.0000
6.0000
7.0000
8.0000
9.0000
10.0000
11.0000
ON GRAPH 1 = CB
2 = CALC. VALUE OF CJ
3 = OBS. VALUE OF CJ
VERTICAL AXIS REPRESENTS STAGE NUMBER
0.0000
1.2000
2.4000
3.6000
4.8000
6.0000
7.2000
8.4000
9.6000
10.8000
12.0000
BRIX OF JUICE

It would be naive to suggest that this brief comparison confirms the validity or the accuracy of the model predictions. A comprehensive comparison is beyond the scope of this project. In any event, it is anticipated that data obtained from the full-scale diffuser will not be entirely relaible, due to the variable operation realised in practice. It is for this reason in fact that pilot plant tests were resorted to for developing and testing the model. These variations are largely due to the variable nature of the cane being processed.

This again emphasizes the need for a quick and efficient method of characterizing the degree of cane preparation. If the quality of the bagasse could be continuously monitored, the cane preparation process could be controlled to present a feed of uniform quality, to the diffuser. In this respect, model predictions can only be representative of an 'average' bagasse.

No account of evaporation of water in the diffuser was taken. However, this has been found to be small ($\pm$ 2% over the whole extraction system) and a correction for evaporation is probably not warranted.

Of the assumptions made in the formulation of the model, the assumption that all juice applied to a given stage finds its way into the correct tray is particularly open to criticism. Even if the trays are correctly positioned with respect to the liquid application areas, transverse dispersion would result in some liquid finding its way into the trays on either side of the correct tray. The effect is compensated to a large extent by the flow of liquid of similar concentration from adjacent stages into the tray under consideration.

A more serious error would be introduced if the trays were all consistently misplaced with respect to the juice application areas. Consider the case where a fraction of the juice applied to the nth stage finds its way into the (n+1)th tray. Let the fraction of liquid entering the nth tray be λ (where $0 < \lambda \leq 1$). Under these conditions

recirculation of juice within the nth stage occurs, whose magnitude is determined by the value of λ. As a result the brix of juice in the trays is affected, and in addition the flow rate through each stage is increased from L to L/λ. In practice, such recirculation could also result in flooding problems in the diffuser.

This situation can be handled by the model. If the degree of recirculation can be measured or estimated, the liquid flow rate and new model parameter values can be calculated. The average concentration of juice in the nth stage which enters the correct tray can be obtained by integrating equation (6.15) with respect to Y over the limits O to λY (instead of O to Y as in equation (6.36)). The average concentration of the remaining fraction of the juice from the nth stage (which enters the (n+1)th stage) is given by integrating over the limits λY to Y. Thus the average concentration of juice in each tray is calculated from 2 components; a fraction λ entering the correct tray and a fraction $(1-\lambda)$ originating from the previous stage.

A similar procedure can be employed for the case of significant 'carry-over' of juice into the following tray by the moving bagasse bed.

CHAPTER 7.

SUMMARY AND CONCLUSIONS.

This study has resulted in the development of a mathematical model of the extraction process which can be used to predict the performance of sugar diffusers. The attainment of this objective has involved a detailed study of the factors which influence extraction performance. This chapter summarizes the major conclusions of the study.

7.1

Consideration of the structure of cane and the nature of bagasse has emphasized the heterogeneous character of bagasse. Also, since cane is a plant product, its quality varies widely. As a result, the response of different canes to the same methods of preparation can result in wide differences in prepared bagasse.

The structure of bagasse has been emphasized as playing a major role in the extraction process. Attention was drawn to the need for characterizing bagasse in two respects, firstly the microstructure, which determines the extraction properties, and secondly particle size and shape which influences the liquid flow characteristics of a bed of bagasse. In this project, this has been achieved through the use of DI and sieve analysis. Because of the fibrous nature of bagasse, sieving of bagasse is not entirely satisfactory. Nonetheless, the specific surface area of bagasse, obtained from sieve analysis, provides an adequate measure of particle size, and has been successfully utilized to correlate liquid holdup data, in the prediction of flooding, and as a measure of mass transfer area in

correlating model parameters.

Although other process variables such as flow rate and temperature can be quickly and accurately measured, these methods of characterizing bagasse are fairly laborious and provide relative measures only of the degree of preparation.

7.2

In a sugar diffuser two fundamental factors influence extraction, namely the basic mechanisms and rates of extraction, and the effect of liquid hydrodynamics on these rates. In this project two experimental configurations were employed. Experiments in a laboratory-scale fully-mixed extraction system provided information on the mechanism of extraction in the absence of the effect of liquid hydrodynamics. These results were then related to experiments in a fixed-bed pilot plant diffuser, where both effects are operative.

7.3

Formulation of the mathematical model of the extraction process was based on previous work on extraction from materials of vegetable origin and on consideration of the structure of bagasse. The model assumes that sucrose is extracted via two 1st order relations in parallel. Model formulation implies that part of the juice in broken cells on the surfaces of bagasse particles is readily extracted by a displacement washing process, at a rate K_1, while the remainder of the juice in unbroken cells and in ruptured cells in the interior of particles is extracted by a slower diffusional process, at a rate K_2. A third parameter a is introduced to represent the fraction of the juice in bagasse extracted at a rate K_1.

7.4

The model was found to fit experimental data obtained in the laboratory extraction tests very satisfactorily. The results of these tests provide substantial support for the validity of the model formulation. Values of K_1 were found to be independent of temperature and

and inversely proportional to surface area, consistent with a washing extraction process; the variation of K_2 with temperature is the same as the variation of D_m with temperature, confirming a diffusion mechanism; and values of α were correlated in terms of DI only, independent of cane quality or temperature.

7.5

Liquid holdup measurements in the pilot plant diffuser were used to infer information on the liquid hydrodynamics in a bagasse bed. Measurements of dynamic and total holdup were made. In keeping with other published work, dynamic holdup was correlated in terms of Reynolds and Galileo numbers, and compares favourably with other published correlations. In the case of total and static holdups, empirical correlations were resorted to.

Evidence of some inherent variability associated with flow through packed beds was demonstrated. This is confirmed by other published work, and is assumed to be generated on re-packing the column. Thus the relative arrangement and orientation of the particles affects the flow characteristics.

7.6

The fibre packing density q (in lb fibre/ft^3) was found to be an important process variable, affecting values of holdup and model parameters. Measured values of q are greater in higher beds because of increased compaction, and at higher temperatures due to softening of bagasse fibres.

7.7

In applying the model to the pilot plant diffuser, axial liquid dispersion was neglected since its effect is likely to be negligibly small. However, the capacitance effect due to stagnant liquid regions was taken into account, since the literature shows that it plays an important part in

other similar displacement processes. The static holdup was therefore considered as part of the juice holdup in bagasse.

The same physical concepts used in the laboratory extraction case regarding the rates of extraction were employed in the pilot plant model.

7.8

The pilot plant model was also found to represent observed experimental behaviour very well. Parameter estimation was complicated by the transient liquid flow conditions observed at the beginning of each pilot plant run. Some assumption was necessary to handle this situation; thus the true starting time was defined on a rational basis according to the physical flow conditions.

Confidence intervals on the parameter estimates were evaluated. This demonstrated a degree of correlation between K_2 and a, which resulted in a lower degree of confidence in values of K_2.

7.9

Results of the pilot plant experiments demonstrate clearly the dependence of extraction on liquid hydrodynamics. The variation in liquid-solid contact efficiency with liquid flow rate may be explained in terms of a capacitance model or stagnant liquid zones model.

Although this model may not provide a unique description of the flow conditions, evidence of previous published work, together with the fact that stagnant zones are likely to be more significant in bagasse beds, provide strong evidence for the validity of this concept.

The parameter a was correlated in terms of the dynamic holdup, which suggests that the fraction of the juice which is readily available is dependent on the actively wetted area, rather than the total wetted area.

The existence of static liquid in the bed indicates that some particle surfaces are not available for active wetting. At higher flow rates, dynamic holdup increases, static holdup decreases, and consequently the rate of extraction is improved.

Zones of static liquid are considered to have an inhibiting effect; thus some juice which would otherwise be washed from particle surfaces has to find its way by diffusion through the static liquid to reach the percolating liquid phase. The higher values of K_2 obtained under conditions of higher static holdup confirm the increased role of diffusion under these conditions.

A comparison of parameter values with those obtained in the laboratory extraction tests shows that extraction is generally more efficient in a fully-mixed environment than in packed bed operation, which is ascribed to better liquid-solid contacting.

The degree of preparation of bagasse has the greatest effect on model parameter values. Higher liquid flow rates are beneficial in increasing the values of K_1 and a, and higher temperatures are advantageous in that they promote the rate of molecular diffusion and improve the effectiveness of liquid-solid wetting.

Correlations for the model parameters have been developed which allow predictions under any given operating conditions (within a broad range of possible conditions). All correlations of this study show a significant degree of scatter, which is to a large extent due to natural variations in the quality of cane. This necessitated the collection of a large volume of experimental data to establish relationships statistically.

7.10

Correlations of liquid holdup and model parameters have not distinguished between flooding and non-flooding data (excluding the conditions of excess flooding, where the liquid level rises above the top of the packed bed). It was established that in a so-called flooded bed, air occluded within the bed constitutes roughly 10-15% of the packed volume. Flooding involves

a gradual build-up of liquid within the bed and may be regarded merely as an extension of the non-flooding regime.

7.11

Flooding was found to occur at lower flow rates with finer preparations. Although higher temperatures should increase the flooding flow rate due to decreased liquid viscosity, generally the effect of more dense beds obtained at higher temperatures overrides the effect of decreased viscosity, resulting in a net decrease in the flooding flow rate.

An approximate locus of flooding was presented, which allows an estimate of when excess flooding will occur, so that in practice the flow rate may be controlled at some lower level to prevent the occurrence of this undesireable operating condition. This correlation shows that fibre density has a far more significant effect on flooding than particle size.

Flooding is initiated at the region of highest fibre density, generally at the bottom of the bagasse bed.

7.12

Extraction is promoted by finer bagasse preparation, higher flow rates and higher temperatures. Of these, preparation is the dominant factor. DI generally dictates the degree of extraction obtained, and is a useful parameter to determine the 'extractability' of bagasse.

Similarly, the most significant model parameter is a, which is related to DI. Since $K_1 >> K_2$, a small increase in the value of a results in a large increase in the overall rate of extraction.

7.13

Because diffuser juice is a complex multi-component mixture, only a limited number of the individual

non-sucrose components could be measured. Attention was confined to the more abundant non-sucrose species. These results together with a literature survey of previous work suggest that the degree of extraction of impurities is if anything, lower than can be expected in milling. This cannot be reconciled with the excessive losses of sucrose in molasses reported in South African diffusion factories.

No quantitative relationships between impurity concentrations and overall recovery exist which can aid in the choice of optimum operating conditions on a rational basis. Thus only qualitative observations can be used as guidelines. In this respect, it appears that the major control variable, degree of preparation, does not affect the extraction of impurities unless unrealistically fine preparations are employed.

7.14

The model used to represent the extraction process in the pilot plant diffuser has been formulated for the geometry of a full-scale moving-bed diffuser. This results in a relation between juice concentrations into and out of a single stage within the diffuser and the concentration of juice in bagasse, and permits a stage-wise calculation procedure.

A brief comparison between model predictions and observed diffuser operating results was made, which demonstrates good agreement between theory and practice. It was pointed out that such factory data cannot be considered as accurate. In addition, attention was drawn to a number of factors which may affect observed diffuser performance. In particular, the effect of mis-alignment of juice application areas and their corresponding catch-trays was considered, and it was shown how the model can be adapted to incorporate this factor.

7.15

The correlations for liquid holdup and model parameter values given in sections 5.4 and 5.6 may be used to predict these values under any given operating conditions. Together with the model of a full-scale moving-bed diffuser, they constitute a means of predicting performance, which may be utilized in the design of new diffuser installations, or to optimize the performance of an existing diff

NOMENCLATURE

a	Mass transfer surface area/unit volume. (ft^2/ft^3)
a_e	Effective interfacial area / unit volume. (ft^2/ft^3)
a_T	Packing or particle surface area / unit volume (ft^2/ft^3)
a_w	Wetted area per unit volume. (ft^2/ft^3)
C	Concentration.
C'	Concentration in stagnant liquid.
C_b	Concentration of juice in bagasse (brix).
$\bar{C}_b$	Average concentration of juice in bagasse (brix).
C_{bo}	Concentration of juice in bagasse at $\theta = O$ (brix)
C_{b1}	Concentration of readily available juice in bagasse (brix).
C_{b2}	Concentration of tightly-held juice in bagasse (brix).
C_j	Concentration of juice (brix).
c	Local concentration.
$\bar{c}$	Average concentration.
D	Axial dispersion coefficient (ft^2/min).
D_m	Molecular diffusion coefficient (ft^2/min).
DI	Displaceability index.
d	Size of packing element (ft).
d_c	Characteristic particle size (mm).
d_p	Particle size (ft).
d_{PT}	Average particle thickness, defined by equation (2. 2) (mm).
d_T	Tower diameter (ft)
E	Extraction (%)
E_t	Extraction after t minutes (%.)

Eö	Eötvos number = $\rho g d^2 / \sigma$.
F	Liquid flow rate (lb/min)
F_o	Outlet flow rate (lb/min)
Fr	Froude number = $\frac{U^2}{gd}$
g	Acceleration due to gravity (ft/min^2).
Ga	Galileo number = $d^3 g \rho^2 / \mu^2$
Ga_1	Galileo number = $g \rho^2 / a_T^3 \mu^2$
H	Juice holdup in bagasse (lb/ft^3)
H_D	Dynamic liquid holdup (lb/lb fibre)
H_S	Static liquid holdup (lb/lb fibre)
H_T	Total liquid holdup (lb/lb fibre)
h	Juice holdup in bagasse (g).
h_D	Dynamic liquid holdup (ft^3/ft^3)
h_S	S tatic liquid holdup (ft^3/ft^3)
h_T	Total liquid holdup (ft^3/ft^3)
j	Number of mixing cells.
j_D	Mass transfer factor, defined by equation (2.17).
K_1	Mass transfer coefficient ($lb/min\ ft^3$)
K_1'	Mass transfer coefficient (lb/min lb fibre)
K_2	Mass transfer coefficient ($lb/min\ ft^3$)
K_2'	Mass transfer coefficient (lb/min lb fibre).
k	Mass transfer coefficient (ft/min or $lb/min\ ft^2$)
k_1	Mass transfer coefficient (g/min).
k_2	Mass transfer coefficient (g/min).
L	Liquid mass flow rate ($lb/min\ ft^2$).
L_f	Flooding liquid flow rate ($lb/min\ ft^2$)
l	Particle or slice thickness (ft)
M_b	Fibre throughput (lb/min)
m	Number of parameters.
N	Dimensionless bed height = $K_1 Z/L$

p	Probability
Pe	Peclet number = $d\,U/D$
Q	W / h
q	Fibre density in bagasse bed (lb/ft^3)
q'	Coefficient of inhibition or tortuosity factor.
Re	Reynolds number = dL / μ
Re_l	Reynolds number = $L/\mu\, a_T$
R_{BT}	Particle flakiness ratio, equation (5.8)
R_{LB}	Particle elongation ratio, equation (5.7)
r	Radial co-ordinate (ft).
S	Specific surface from sieve analysis, equation (5.2) (mm^2 / g)
S_c	Schmidt number = $\mu/ \rho D_m$.
Sh	Sherwood number = $k\,d / D_m$
Sh_l	Sherwood number = $(K_l/a_T\ \rho)\ /a_T\, D_m$.
T	Temperature (^oC)
t	Time (mins).
t'	Experimental time (min).
t_c	Time correction factor (min).
U	Average liquid velocity (ft/min).
$\bar{U}$	Mean dynamic phase liquid velocity = $Z/\ \tau$ (ft/min)
u	Point velocity (ft/min).
V	Volume of packed bed (ft^3)
v	Speed of bagasse bed (ft/min).
W	Weight of extract liquid (g)
w	Width of diffuser (ft)
w_i	Percent by weight of fraction retained on ith screen (%).
x	Axial co-ordinate (ft).
x_f	Mean film thickness (ft).
x'_g	Geometric mean of weight distribution (mm)
x_L	Length of a diffuser stage (ft)
x_i	Mean dimension of particles retained on ith screen (mm).

Y	Dimensionless length parameter, defined by equation (6.5).
Z	Bed height (ft).
z	Axial co-ordinate, measured from top of packed bed (ft).
α	Fraction of juice in bagasse readily available for extraction.
α_M	Values of α obtained in laboratory mixing tests.
α_S	Particle surface shape factor.
α_V	Particle volume shape factor.
β	K_2/K_1 or k_2/k_1 .
γ	$(1 - \alpha) / \beta$
δ	Effective film thickness = $(\mu^2 / g \rho^2)^{1/3}$ (ft)
ϵ	Fractional void volume.
θ	Dimensionless time = $K_1/H(t - \tau)$. (or $k_1 t/h$ in section 5.2)
ϕ	Fraction flowing liquid.
ϕ_b	Sum of squared errors. Equation (5.48).
ϕ_1	Sum of squared errors function, equation (5.50)
ϕ_2	Sum of squared errors function , equation (5.49)
λ	Fraction of juice in plug flow entering correct tray.
μ	Liquid viscosity (lb/ft min).
ρ	Liquid density (lb/ft^3)
σ	Surface tension (lb/min^2)
σ_g	Geometric standard deviation of particle size distribution.
τ	Mean residence time of dynamic liquid phase (min).
$\bar{\tau}$	Mean residence time (min).

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APPENDIX A.

DETAILS OF PILOT PLANT RUNS

Details of the primary measurements for the pilot plant runs are given here, as well as sieve analysis results and liquid holdup measurements. For simplicity, units are not given in the following tables; these may be found in the Nomenclature.

Table A. 1. Details of pilot plant runs. Bagasse preparation P1.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{pT}	d_c	σ_g	a_T	H_D	H_S	H_T
C9	58.6	73	125	2.46	3.93	64.6	3200	5.47	2.65	2.68	61.4	5.31	4.35	9.65
C10	58.6	73	125	2.42	4.00	64.6	3200	5.47	2.65	2.68	62.5	5.24	3.52	8.76
C11	58.6	73	125	2.46	4.29	63.8	2610	6.39	4.02	3.24	54.7	5.07	3.23	8.30
C12	58.6	73	125	2.46	4.29	63.8	2610	6.39	4.02	3.24	54.7	4.74	4.01	8.75
C13	58.6	73	125	-	-	66.4	2770	6.14	3.18	3.28	-	4.46	3.87	8.32
C14	58.6	73	125	2.46	4.39	66.4	2770	6.14	3.18	3.28	59.2	5.49	3.07	8.55
C16	58.6	73	125	2.50	4.16	62.7	2940	5.51	2.38	2.43	59.6	5.03	4.29	9.32
C17	58.6	73	125	2.54	4.72	69.2	3010	5.60	3.12	2.97	69.5	4.27	3.63	7.90
C18	58.6	73	125	2.58	4.65	69.2	3010	5.60	3.12	2.97	68.4	4.01	4.77	8.78
C22	31.2	73	125	2.50	4.78	-	-	-	-	-	-	3.01	4.07	7.09
C30	16.2	73	125	2.79	4.24	65.4	2680	5.98	3.75	3.07	55.5	3.25	3.63	6.88
C31	69.7	73	125	2.67	4.43	65.4	2680	5.98	3.75	3.07	58.1	5.58	3.96	9.53
C32	43.3	73	125	2.58	4.49	64.1	3530	4.80	2.84	3.09	77.3	5.02	3.62	8.65
C33	95.8	73	125	2.50	4.64	64.1	3530	4.80	2.84	3.09	79.9	7.30	-	-
C45	16.2	61	125	-	-	-	3100	5.43	2.51	2.57	-	2.22	4.94	7.14
C46	16.2	88	125	-	-	-	3100	5.43	2.51	2.57	-	2.22	5.10	7.30
C47	69.4	90	125	2.58	4.40	60.9	2910	5.90	3.12	2.94	62.6	4.81	-	-
C48	69.7	58	125	2.67	4.27	60.9	2910	5.90	3.12	2.94	60.6	4.81	4.67	9.48
C49	95.8	63	125	2.69	4.60	59.1	-	-	-	-	-	6.46	-	-
C50	95.2	87	125	2.67	4.64	59.1	-	-	-	-	-	5.95	-	-
C51	43.3	87	125	2.77	4.49	62.7	2650	5.80	3.71	3.09	58.0	3.27	3.71	6.98
C52	43.3	59	125	2.71	4.59	62.7	2650	5.80	3.71	3.09	59.3	3.09	4.81	7.91
D38	48.4	73	125	2.67	4.46	59.0	3280	4.85	3.77	3.45	71.3	4.50	3.77	8.27
D39	79.3	73	125	2.69	4.43	59.0	3280	4.85	3.77	3.45	70.8	5.86	-	-
D40	62.7	73	125	2.65	4.63	63.8	3580	4.47	2.82	2.94	80.9	5.65	-	-
D41	43.6	73	125	2.71	4.52	63.8	3580	4.47	2.82	2.94	79.0	4.60	-	-
D44	43.6	73	125	2.62	4.78	65.2	3760	4.32	2.86	3.01	87.7	4.74	3.94	8.68
D45	28.5	73	125	2.71	4.63	65.2	3760	4.32	2.86	3.01	85.0	4.02	4.42	8.43
D46	20.8	73	125	2.85	4.55	65.2	4290	3.83	2.19	2.81	95.3	3.26	4.02	7.28
D47	37.6	73	125	2.75	4.72	65.2	4290	3.83	2.19	2.81	98.9	4.57	3.31	7.87
D48	31.4	73	125	2.69	4.40	69.0	3860	4.61	2.44	2.84	82.9	3.97	3.80	7.76
D49	24.0	73	125	2.71	4.36	69.0	3860	4.61	2.44	2.84	82.2	3.45	4.58	8.03

Table A. 1. (continued) Details of pilot plant runs. Bagasse preparation P1.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{PT}	d_c	σ_g	a_T	H_D	H_S	H_T
E1	31.4	73	75	1.62	4.38	61.3	3680	4.76	2.71	3.04	78.7	4.56	3.36	7.94
E2	31.4	73	175	3.42	4.86	61.3	3680	4.76	2.71	3.04	87.3	4.51	3.55	8.06
E9	48.4	73	75	1.67	4.07	64.0	3560	4.66	2.71	2.98	70.9	4.62	4.84	9.44
E10	48.4	73	175	3.37	4.69	64.0	3560	4.66	2.71	2.98	81.7	5.18	3.78	8.96
E11	63.3	73	75	1.62	4.28	65.3	2950	5.73	3.96	3.44	61.5	6.43	2.44	8.86
E12	62.4	73	175	3.52	4.60	65.3	2950	5.73	3.96	3.44	66.2	6.89	-	-
E13	48.4	73	125	2.37	4.83	64.0	3750	4.59	2.60	2.92	88.5	6.35	-	-
E14	48.4	73	125	2.52	4.55	64.0	3750	4.59	2.60	2.92	83.3	4.83	3.80	8.61
E15	48.4	73	100	2.17	4.05	67.5	3990	5.45	2.15	2.56	78.8	5.34	3.71	9.06
E16	47.7	73	150	3.04	4.32	67.5	3990	5.45	2.15	2.56	84.2	5.70	-	-
E17	48.4	73	125	2.65	4.56	65.7	2870	5.34	3.65	3.02	63.9	4.48	4.35	8.84
E18	31.4	73	125	2.62	4.60	65.7	2870	5.34	3.65	3.02	64.4	3.98	3.85	7.84
E19	31.4	73	100	2.23	4.34	65.5	3780	4.46	2.31	2.66	80.1	4.08	3.88	7.97
E20	31.4	73	150	3.23	4.49	65.5	3780	4.46	2.31	2.66	82.9	4.31	3.31	7.63
E21	37.6	73	100	1.98	4.40	65.8	3460	4.95	3.02	3.08	74.3	5.22	3.74	8.98
E22	37.6	73	150	2.85	4.57	65.8	3460	4.95	3.02	3.08	77.3	5.69	3.10	8.78
E23	37.6	73	75	1.71	4.08	65.7	2980	5.77	3.48	3.16	59.4	5.07	3.61	8.67
E24	37.6	73	175	3.48	4.67	65.7	2980	5.77	3.48	3.16	68.0	5.56	3.10	8.66
E25	36.6	73	100	2.12	4.40	58.5	3260	5.54	3.51	3.41	70.2	4.87	3.54	8.40
E26	37.6	73	125	2.62	4.46	58.5	3260	5.54	3.51	3.41	71.0	5.16	3.15	8.31
E27	37.6	73	75	1.75	4.16	65.4	3470	5.07	3.00	3.09	70.5	4.94	3.84	8.78
E28	37.6	73	175	3.60	4.71	65.4	3470	5.07	3.00	3.09	79.9	5.22	3.06	8.28
E29	37.6	73	125	2.56	4.44	65.4	3190	5.09	3.42	3.23	69.2	5.22	3.99	9.22
E30	37.6	73	150	3.00	4.55	65.4	3190	5.09	3.42	3.23	71.0	5.02	3.22	8.24
3	45.8	87	125	-	-	-	4010	4.68	2.40	2.94	-	3.84	-	-
4	46.2	59	125	2.71	4.26	-	4010	4.68	2.40	2.94	83.3	4.06	4.09	8.18
2	44.6	74	125	2.69	4.53	63.3	3440	4.90	2.90	3.05	76.2	5.36	3.87	9.23
3	44.6	88	125	2.62	4.64	63.3	3440	4.90	2.90	3.05	78.0	4.81	-	-
4	43.9	89	125	2.65	4.54	61.7	3950	4.33	2.43	2.91	87.6	5.25	-	-
5	44.6	67	125	2.69	4.47	61.7	3950	4.33	2.43	2.91	86.2	5.36	4.19	9.55
5	44.6	67	125	2.65	4.63	62.8	4100	4.41	2.44	3.04	92.8	4.71	4.29	8.99
7	44.6	81	125	2.56	4.78	62.8	4100	4.41	2.44	3.04	95.8	4.71	3.79	8.49

Table A. 1. (continued) Details of pilot plant runs. Bagasse preparation P1.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{PT}	d_c	σ_g	a_T	H_D	H_S	H_T
J8	44.6	81	125	2.58	4.57	63.4	4030	4.44	2.56	3.16	89.8	4.58	4.26	8.84
J9	44.6	60	125	2.65	4.46	63.4	4030	4.44	2.56	3.16	87.7	4.36	5.23	9.60
J14	44.6	74	125	2.62	4.42	65.4	3890	4.31	2.51	2.99	84.0	4.77	4.83	9.61
J15	44.6	60	125	2.69	4.32	65.4	3890	4.31	2.51	2.99	82.0	5.40	5.08	10.49
J16	44.6	89	125	2.58	4.62	64.4	3520	4.62	2.74	2.98	79.4	4.90	3.65	8.56
J17	44.6	60	125	2.65	4.51	64.4	3520	4.62	2.74	2.98	77.5	4.98	4.48	9.45
J21	50.9	74	125	2.75	4.47	65.1	4450	3.74	2.20	2.90	97.1	6.76	-	-
J22	44.2	74	125	2.71	4.54	65.1	4450	3.74	2.20	2.90	98.6	5.75	-	-
J23	52.2	74	125	2.52	4.52	63.9	3670	4.61	3.05	3.37	81.0	5.16	4.28	9.69
J24	57.6	74	125	2.56	4.45	63.9	3670	4.61	3.05	3.37	79.7	5.53	-	-

Table A.2 Details of pilot plant runs. Bagasse preparation P2.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{PT}	d_c	σ_g	a_T	H_D	H_S	H_T
C6	64.6	73	125	-	-	72.1	4000	4.06	1.97	2.46	-	6.37	3.39	9.76
C35	48.1	73	125	2.46	4.39	72.9	4340	3.67	1.80	2.43	92.9	6.17	2.95	9.12
C43	16.2	73	125	3.12	3.90	67.5	3140	5.26	2.76	2.82	59.8	2.43	4.67	7.10
C44	79.9	73	125	2.67	4.49	-	3020	5.63	2.67	2.72	66.2	6.54	-	-
D18	16.4	73	125	3.00	4.08	69.4	4100	3.64	2.33	2.74	81.7	3.27	4.18	7.45
D19	31.4	73	125	-	-	69.4	4100	3.64	2.33	2.74	-	5.30	3.38	8.68
D20	31.4	73	125	2.75	4.09	68.4	4260	3.71	2.35	2.90	85.1	5.06	4.04	9.11
D21	24.0	73	125	2.79	4.03	68.4	4260	3.71	2.35	2.90	83.9	4.16	4.50	8.65
D34	43.6	73	125	2.62	4.36	74.2	3940	3.68	2.26	2.76	83.8	5.64	3.28	8.92
D35	56.0	73	125	2.58	4.43	74.2	3940	3.68	2.26	2.76	85.2	6.09	2.92	9.01
D36	71.6	73	125	2.67	4.56	71.7	4310	4.03	2.66	3.33	95.9	6.59	-	-
D37	63.0	73	125	2.69	4.52	71.7	4310	4.03	2.66	3.33	95.1	5.99	-	-
D42	43.6	87	125	2.77	4.42	73.3	4780	3.53	2.14	3.34	103.1	5.14	-	-
D43	43.6	59	125	2.85	4.29	73.3	4780	3.53	2.14	3.34	100.1	5.17	4.31	9.48
D50	37.6	73	125	2.79	4.30	74.7	4000	3.93	2.24	2.67	84.0	5.12	4.06	9.18
D51	27.4	73	125	2.79	4.34	77.5	4660	3.70	2.08	2.74	98.8	4.10	4.23	8.33
D52	19.7	73	125	2.90	4.18	77.5	4660	3.70	2.08	2.74	95.2	3.71	4.07	7.77
D55	16.4	86	125	2.98	4.29	70.8	4100	3.84	2.23	2.72	85.8	2.22	4.06	6.28
D56	16.4	59	125	3.13	4.09	70.8	4100	3.84	2.23	2.72	81.8	2.54	4.98	7.52
D57	27.7	87	125	2.83	4.53	71.7	3920	4.52	2.26	2.63	86.7	3.96	3.47	7.43
D58	27.7	58	125	3.02	4.25	71.7	3920	4.52	2.26	2.63	81.3	3.60	3.86	7.49

Table A. 3. Details of pilot plant runs. Bagasse preparation P3.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{PT}	d_c	σ_g	a_T	H_D	H_S	H_T
C19	31.2	73	125	3.00	4.10	86.8	5830	2.50	1.33	1.36	116.8	5.68	3.49	9.15
C20	31.2	73	125	2.46	4.88	84.1	7690	1.54	0.92	2.02	183.2	6.26	2.65	8.92
C21	31.2	73	125	2.58	4.65	84.1	7690	1.54	0.98	2.02	174.3	5.92	2.86	8.78
C23	31.2	73	125	2.75	4.44	82.8	6650	1.97	1.16	2.15	144.0	6.06	3.02	9.07
C25	31.2	73	125	2.50	4.37	88.8	8270	1.48	0.91	2.04	176.6	6.90	2.64	9.55
C26	31.2	73	125	2.83	4.44	87.6	6590	1.92	1.11	2.10	142.7	6.05	2.99	9.04
C27	31.2	73	125	-	-	87.6	6590	1.92	1.11	2.10	-	5.75	3.42	9.19
C37	27.1	73	125	2.46	4.97	86.2	7730	1.75	0.98	2.17	187.3	6.32	2.52	8.84
C39	37.6	73	125	2.75	4.31	88.8	6790	1.86	1.05	2.03	142.8	6.48	2.93	9.41
C40	15.3	73	125	3.00	3.94	88.8	6790	1.86	1.05	2.03	130.4	3.77	5.13	8.90
C54	43.3	73	125	-	-	88.7	6780	1.61	1.04	1.89	-	5.41	3.24	8.66
C55	20.7	73	125	2.79	4.66	88.7	6780	1.61	1.04	1.89	154.1	4.19	3.87	8.08
D11	37.6	73	125	2.75	4.28	80.6	5500	2.11	1.35	2.01	115.0	6.77	2.90	9.68
D12	27.4	73	125	2.79	4.39	81.7	5960	2.12	1.32	2.18	127.7	5.66	3.43	9.07
D13	24.0	73	125	2.79	4.39	81.7	5960	2.12	1.32	2.18	127.7	5.38	3.48	8.89
D14	34.4	73	125	-	-	83.2	5670	2.10	1.33	2.08	-	6.63	2.49	9.12
D15	18.2	73	125	3.00	4.16	83.2	5670	2.10	1.33	2.08	115.0	4.04	4.17	8.21
D22	27.1	87	125	2.75	4.28	80.4	6260	1.99	1.36	2.41	130.6	6.83	-	-
D23	27.4	59	125	2.88	4.10	84.4	6260	1.99	1.36	2.41	125.4	4.91	4.10	9.01
D24	16.4	87	125	-	-	84.3	6810	1.94	1.21	2.28	-	5.52	3.38	8.90
D25	16.4	59	125	-	-	84.3	6810	1.94	1.21	2.28	-	4.70	4.51	9.21
D26	50.6	73	125	2.75	4.52	81.5	5540	2.37	1.57	2.49	122.4	6.91	2.66	9.57
D27	50.9	73	125	2.83	4.39	81.5	5540	2.37	1.57	2.49	118.8	6.06	3.07	9.13
D28	46.2	73	125	2.71	4.31	82.4	5810	2.39	1.61	2.66	122.1	7.22	-	-
D29	45.8	73	125	2.75	4.24	82.4	5810	2.39	1.61	2.66	120.3	7.33	-	-
D31	43.6	73	125	2.67	4.09	83.7	6330	2.07	1.25	2.27	126.2	7.62	2.66	10.29
D33	43.6	59	125	-	-	86.8	6430	2.14	1.22	2.26	-	7.25	2.60	9.86
E3	27.4	73	75	2.04	3.80	80.7	5920	2.14	1.35	2.21	109.8	5.84	4.14	9.96
E4	27.4	73	150	3.58	4.32	80.7	5920	2.14	1.35	2.21	124.7	6.18	-	-
E5	16.4	73	100	2.50	4.14	84.7	6370	2.34	1.23	2.27	128.7	3.94	4.46	8.40
E6	16.4	73	150	3.54	4.38	84.7	6370	2.34	1.23	2.27	136.3	4.66	3.24	7.89
E7	37.6	73	100	2.37	4.30	84.3	6860	1.88	1.14	2.17	143.9	6.61	2.49	9.09
E8	37.2	73	150	3.33	4.59	84.3	6860	1.88	1.14	2.17	153.8	6.82	-	-

Table A. 4. Details of pilot plant runs. Bagasse preparation P4.

Run No.	L	T	Load (lb)	Z	q	DI	S	d_{PT}	d_c	σ_g	a_T	H_D	H_S	H_T
C29	16.2	73	125	-	-	90.6	9080	1.06	0.85	1.94	-	5.31	3.76	9.07
C38	30.9	73	125	2.67	4.65	93.8	8950	1.04	0.87	2.08	203.1	7.17	-	-
C41	25.8	73	125	2.67	4.58	93.1	9050	1.05	0.84	2.04	202.3	7.33	2.26	9.28
C42	21.0	73	125	-	-	93.1	9050	1.05	0.84	2.04	-	6.65	3.05	9.70
D2	21.0	56	125	3.08	3.86	89.9	6790	1.35	1.18	2.13	127.9	6.90	3.05	9.95
D3	21.0	85	125	2.75	4.40	96.6	7770	1.22	1.02	2.08	166.7	6.27	2.34	8.40
D4	25.9	59	125	2.71	4.05	93.7	7030	1.34	1.12	2.09	139.0	7.00	2.87	9.86
D5	16.4	59	125	2.92	4.01	90.5	6830	1.45	1.21	2.22	133.6	6.22	3.73	9.95
D6	16.4	86	125	2.75	4.25	90.5	6830	1.45	1.21	2.22	141.7	6.43	2.86	9.29
D8	30.2	59	125	2.92	4.25	89.5	7240	1.40	1.13	2.19	150.0	8.12	2.22	9.87
D9	25.1	87	125	2.75	4.25	92.8	7270	1.35	1.11	2.18	150.6	6.73	-	-
D10	21.9	73	125	2.96	4.29	92.8	7270	1.35	1.11	2.18	152.1	7.06	2.36	9.43
D16	18.9	73	125	-	-	87.8	7580	1.24	1.04	2.04	-	6.39	2.81	9.20
D17	28.0	73	125	2.75	4.33	87.8	7580	1.24	1.04	2.04	160.4	7.72	2.34	9.39
D53	27.4	73	125	2.79	4.29	89.5	6700	1.44	1.24	2.28	140.2	7.41	2.57	9.97
D54	22.2	73	125	2.81	4.23	89.5	6700	1.44	1.24	2.28	144.6	6.31	3.45	9.77

APPENDIX B

EXPERIMENTAL DATA FROM PILOT PLANT RUNS AND LABORATORY EXTRACTION TESTS.

Measured juice concentrations are presented here, together with the values calculated via the mathematical models for the pilot plant runs and the laboratory extraction tests. In addition, the bagasse analysis and values of C_{bo} and t_c for each individual run are given. In the tables, obs. C_j represents the measured brix values, and calc. C_j the values calculated from the model, at experimental times t' in minutes. Further details of the pilot plant runs are given in Appendix A, and the laboratory extraction tests in Table 5. 5.

Because of the large number of runs carried out, only a few runs are tabulated here, i. e. those plotted in Fig. 5.32 (pilot plant) and Fig. 5. 13 (laboratory tests). It was felt that the inclusion of the data for all runs would serve only to inflate the size of an already unwieldy volume. Copies of the full set of data have been lodged with the University of Natal and Huletts Sugar Ltd. These may be obtained by interested parties on request from:

1. The Secretary, Department of Chemical Engineering,
   University of Natal,
   Durban, South Africa.

2. The Information Officer,
   Research and Development Department,
   Huletts Sugar Limited,
   P. O. Mt. Edgecombe,
   Natal, South Africa.

RUN NO. C12			RUN NO. D48			RUN NO. C25			RUN NO. C42		
brix % bagasse = 12.6 fibre % bagasse = 26.5 C_{bo} = 12.57 t_c = 0.47 mins.			brix % bagasse = 13.6 fibre % bagasse = 29.7 C_{bo} = 12.89 t_c = 1.02 mins.			brix % bagasse = 11.5 fibre % bagasse = 27.5 C_{bo} = 17.52 t_c = 0.83 mins			brix % bagasse = 12.4 fibre % bagasse = 30.7 C_{bo} = 14.51 t_c = 1.45 mins.		
t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j
0.25	2.46		0.25	3.06		0.25	4.86		0.25	5.26	
0.50	1.93	1.92	0.50	2.88		0.50	5.74		0.75	5.78	
0.75	1.62	1.67	0.75	3.20		0.75	5.63		1.00	5.45	
1.00	1.47	1.45	1.00	3.18		1.00	5.28	5.17	1.25	5.52	
1.25	1.39	1.26	1.25	3.14	3.14	1.25	4.52	4.50	1.50	5.57	
1.50	1.16	1.10	1.50	2.72	2.73	1.50	4.00	3.93	1.75	5.79	5.62
1.75	0.93	0.96	1.75	2.35	2.39	1.75	3.55	3.43	2.00	5.45	5.21
2.00	0.77	0.83	2.00	2.15	2.11	2.00	2.99	2.99	2.25	5.08	4.83
2.25	0.69	0.75	2.25	1.87	1.88	2.25	2.54	2.62	2.50	4.70	4.48
2.50	0.66	0.66	2.50	1.75	1.69	2.50	2.17	2.30	2.75	4.30	4.15
2.75	0.58	0.59	2.75	1.56	1.53	2.75	2.14	2.02	3.00	3.78	3.85
3.00	0.50	0.53	3.00	1.45	1.40	3.00	1.56	1.77	3.25	3.37	3.57
3.50	0.42	0.43	3.25	1.29	1.29	3.25	1.47	1.56	3.50	3.06	3.31
4.00	0.35	0.35	3.50	1.18	1.19	3.50	1.27	1.38	3.75	2.69	3.07
4.50	0.35	0.30	4.00	0.97	1.05	4.00	0.99	1.09	4.00	2.65	2.84
5.00	0.31	0.26	4.50	0.89	0.94	4.50	0.85	0.87	4.50	2.31	2.45
5.50	0.23	0.23	5.00	0.87	0.86	5.00	0.73	0.70	5.00	1.96	2.11
6.00	0.19	0.21	5.50	0.87	0.80	5.50	0.66	0.58	5.50	1.85	1.81
7.00	0.19	0.18	6.00	0.77	0.75	6.00	0.58	0.48	6.00	1.58	1.57
8.00	0.19	0.16	7.25	0.68	0.66	6.50	0.54	0.41	7.00	1.31	1.17
9.00	0.15	0.15	8.00	0.64	0.62	7.00	0.48	0.36	8.00	1.04	0.88
10.00	0.11	0.14	9.00	0.48	0.58	8.00	0.35	0.28	9.00	0.81	0.67
11.00	0.11	0.13	10.00	0.48	0.53	9.00	0.27	0.24	10.00	0.62	0.52
			11.00	0.56	0.50	10.00	0.17	0.21	11.00	0.46	0.41
			12.00	0.50	0.46	11.00	0.13	0.19	12.00	0.31	0.33

RUN NO. C12 brix % bagasse = 12.6 fibre % bagasse = 26.5 C_{bo} = 12.57 t_c = 0.47 mins.			RUN NO. D48 brix % bagasse = 13.6 fibre % bagasse = 29.7 C_{bo} = 12.89 t_c = 1.02 mins.			RUN NO. C25 brix % bagasse = 11.5 fibre % bagasse = 27.5 C_{bo} = 17.52 t_c = 0.83 mins.			RUN NO. C42 brix % bagasse = 12.4 fibre % bagasse = 30.7 C_{bo} = 14.51 t_c = 1.45 mins.		
t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j
			13.00	0.48	0.43	12.00	0.11	0.17	13.00	0.23	0.2[illegible]
			14.00	0.42	0.40				14.00	0.19	0.22
			15.00	0.38	0.37				16.00	0.15	0.16
			16.00	0.37	0.34				18.00	0.13	0.13
			17.00	0.35	0.32						
			18.00	0.29	0.30						
			19.00	0.29	0.28						
			20.00	0.27	0.26						

RUN NO. F11 brix % bagasse = 13.4 fibre % bagasse = 28.6 C_{bo} = 20.84			RUN NO. F14 brix % bagasse = 12.5 fibre % bagasse = 32.9 C_{bo} = 21.21			RUN NO. F15 brix % bagasse = 12.5 fibre % bagasse = 32.9 C_{bo} = 21.21			RUN NO. F20 brix % bagasse = 13.0 fibre % bagasse = 30.9 C_{bo} = 21.13		
t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j	t'	obs. C_j	calc. C_j
0.50	1.47	1.41	0.50	1.08	0.98	0.50	1.00	0.90	0.50	1.64	1.60
1.00	1.73	1.80	1.00	1.29	1.35	1.00	1.24	1.27	1.00	1.94	1.98
1.50	1.87	1.93	1.50	1.43	1.50	1.50	1.33	1.43	1.50	2.00	2.09
2.00	1.93	1.98	2.00	1.48	1.56	2.00	1.39	1.49	2.00	2.12	2.14
2.50	1.96	2.01	2.50	1.52	1.59	3.00	1.48	1.54	2.50	2.15	2.16
3.00	2.00	2.04	3.00	1.56	1.61	3.50	1.52	1.56	3.00	2.21	2.19
3.50	2.08	2.06	3.50	1.62	1.63	4.00	1.54	1.57	3.50	2.25	2.21
4.00	2.12	2.08	4.00	1.64	1.65	5.00	1.58	1.58	4.00	2.29	2.23
5.00	2.15	2.12	5.00	1.70	1.67	6.00	1.62	1.60	5.00	2.31	2.27
6.00	2.21	2.16	6.00	1.73	1.70	7.00	1.66	1.62	6.00	2.33	2.31
7.00	2.25	2.19	7.00	1.77	1.73	8.00	1.68	1.63	7.00	2.36	2.34
8.00	2.29	2.23	8.00	1.83	1.75	10.00	1.71	1.66	8.00	2.38	2.37
10.00	2.33	2.29	10.00	1.85	1.80	12.00	1.75	1.69	10.00	2.42	2.42
12.00	2.38	2.34	12.00	1.91	1.85	14.00	1.77	1.72	12.00	2.46	2.46
14.00	2.42	2.39	14.00	1.95	1.89	16.00	1.81	1.75	14.00	2.48	2.50
16.00	2.44	2.43	16.00	2.00	1.93	19.00	1.85	1.79	16.00	2.50	2.52
20.00	2.46	2.49	20.00	2.04	2.01	27.50	1.89	1.89	20.00	2.54	2.57
25.00	2.50	2.56	25.00	2.10	2.09	30.00	1.93	1.91	25.00	2.57	2.60
30.00	2.54	2.60	30.00	2.15	2.15	37.50	1.96	1.99	30.00	2.59	2.62
35.00	2.57	2.63	35.00	2.19	2.21	43.50	2.02	2.04	35.00	2.61	2.64
40.00	2.59	2.66	40.00	2.21	2.26	50.50	2.04	2.10	40.00	2.63	2.64
45.00	2.63	2.68	45.00	2.23	2.30	60.00	2.10	2.16	44.00	2.65	2.65
50.00	2.65	2.69	50.00	2.27	2.33				50.75	2.69	2.65
60.00	2.69	2.70	60.00	2.29	2.39						

APPENDIX C.

PILOT PLANT MODEL PARAMETER ESTIMATION DATA

Values of the pilot plant model parameters and the quantities derived from them are presented in the following tables. In addition, the values of the best fit criterion ϕ_1 and the number of data points fitted are given.

Again the reader is referred to the Nomenclature for the units of the quantities tabulated.

Table C. 1. Parameter estimation. Bagasse preparation P1.

Run No.	K_1V	K_2V	α	HV	Φ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{a_T(1-\alpha)}$	$\frac{\alpha}{DI}$
C9	33.0	3.33	0.208	124	0.223	20	1.086	17.1	59.2	7.15	0.110	.00887	0.322
C10	21.3	3.00	0.326	99	0.180	23	0.701	10.9	58.2	6.79	0.0988	.00938	0.505
C11	20.2	2.19	0.275	99	0.100	19	0.609	13.2	66.5	10.1	0.0661	.00715	0.431
C12	28.5	2.64	0.333	125	0.068	22	0.860	18.7	66.5	10.1	0.0796	.00937	0.522
C13	34.3	3.56	0.396	123	0.079	23	1.013	-	-	-	0.105	.01288	0.596
C14	22.1	3.13	0.289	96	0.311	21	0.652	12.3	61.4	7.97	0.0924	.00962	0.435
C16	34.6	3.64	0.344	132	0.189	22	1.060	18.7	61.0	7.80	0.112	.01184	0.549
C17	40.8	3.09	0.455	128	0.097	20	1.082	16.0	52.4	4.94	0.0819	.01022	0.658
C18	30.3	2.82	0.433	171	0.197	20	0.803	12.1	53.2	5.18	0.0748	.00896	0.626
C22	16.0	5.59	0.217	144	0.067	23	0.426	-	-	-	0.149	-	-
C30	8.6	4.63	0.118	126	0.147	24	0.231	4.82	18.2	9.70	0.125	.01079	0.180
C31	43.0	3.73	0.329	138	0.144	19	1.157	23.0	74.6	8.46	0.100	.01143	0.603
C32	27.9	4.19	0.281	123	0.143	26	0.766	8.70	34.8	3.58	0.115	.00929	0.438
C33	54.8	1.89	0.423	90	0.085	15	1.504	16.5	74.5	3.25	0.0519	.00522	0.660
C45	9.2	3.35	0.134	174	0.037	25	0.247	-	-	-	0.0904	.00690	-
C46	10.4	0.31	0.545	184	0.146	22	0.281	-	-	-	0.0084	.00122	-
C47	48.7	0.49	0.546	133	0.084	17	1.362	15.4	80.9	9.17	0.0137	.00212	0.896
C48	32.9	4.89	0.323	158	0.123	20	0.920	23.2	58.4	5.04	0.137	.01422	0.530
C49	44.9	1.76	0.414	116	0.013	15	1.156	-	-	-	0.0453	-	0.701
C50	50.2	1.53	0.443	101	0.051	15	1.292	-	-	-	0.0394	-	0.750
C51	23.1	2.57	0.410	135	0.158	27	0.591	8.52	54.5	11.5	0.0658	.00863	0.654
C52	20.0	3.27	0.280	178	0.048	25	0.512	14.2	37.0	5.38	0.0837	.00900	0.447
D38	27.9	3.33	0.449	132	0.101	27	0.747	9.90	42.1	4.56	0.0891	.01011	0.761
D39	59.5	2.58	0.516	135	0.095	19	1.593	21.3	69.6	4.67	0.0691	.00892	0.874
D40	50.6	0.74	0.550	119	0.325	17	1.314	14.1	48.2	3.13	0.0193	.00246	0.862
D41	36.7	0.04	0.619	158	0.037	19	0.953	10.5	34.3	3.36	0.0011	.00017	0.970
D44	38.2	2.14	0.552	145	0.091	25	0.970	9.10	30.9	2.45	0.0543	.00660	0.847
D45	25.1	5.03	0.211	164	0.119	28	0.637	6.17	20.8	2.69	0.128	.00881	0.324
D46	16.4	2.98	0.364	154	0.287	29	0.402	3.05	13.6	1.91	0.0731	.00548	0.558

Table C. 1. (contd) Parameter estimation. Bagasse preparation P1.

Run No.	K_1V	K_2V	α	HV	ϕ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{a\overline{T}(1-\alpha)}$	$\frac{\alpha}{DI}$
D47	33.9	4.23	0.326	125	0.315	28	0.831	6.07	23.6	1.71	0.104	.00735	0.500
D48	24.3	7.94	0.201	132	0.070	29	0.654	6.34	23.6	2.91	0.214	.01421	0.291
D49	14.0	9.66	0.166	161	0.062	28	0.377	3.68	18.2	2.98	0.260	.01656	0.241
E1	19.6	3.18	0.319	66	0.087	26	0.878	9.38	24.8	3.40	0.143	.01162	0.521
E2	25.2	7.50	0.247	176	0.433	32	0.484	4.66	22.4	2.49	0.144	.01063	0.403
E9	28.9	4.21	0.444	98	0.037	27	1.355	16.6	42.4	4.65	0.197	.02040	0.693
E10	38.9	2.18	0.582	176	0.191	32	0.782	8.32	36.8	3.04	0.0438	.00602	0.909
E11	23.1	1.26	0.548	44	0.026	19	1.058	18.1	64.0	7.12	0.0577	.00888	0.839
E12	37.9	2.55	0.507	115	0.190	27	0.744	11.8	58.5	5.70	0.0501	.00706	0.776
E13	34.7	2.35	0.502	80	0.245	26	0.963	8.99	34.0	2.39	0.0652	.00715	0.784
E14	38.4	4.01	0.486	128	0.087	25	1.065	10.6	36.1	2.86	0.111	.01182	0.759
E15	27.5	3.38	0.450	95	0.065	26	0.999	9.84	38.1	3.38	0.123	.01145	0.667
E16	32.6	3.55	0.479	130	0.281	30	0.789	7.28	35.2	2.77	0.0859	.00847	0.710
E17	26.5	4.16	0.461	156	0.134	25	0.699	11.8	47.0	6.34	0.110	.01453	0.702
E18	21.3	5.36	0.299	137	0.218	31	0.561	9.41	30.3	6.19	0.141	.01439	0.455
E19	16.4	2.92	0.376	109	0.312	25	0.540	5.52	24.4	3.22	0.0961	.00834	0.574
E20	19.3	4.55	0.312	142	0.045	32	0.423	4.18	23.6	2.90	0.100	.00786	0.476
E21	25.8	2.31	0.521	95	0.035	26	0.944	11.4	31.4	4.03	0.0845	.01044	0.792
E22	25.7	2.47	0.487	117	0.489	31	0.627	7.25	30.2	3.58	0.0602	.00695	0.740
E23	20.2	2.91	0.416	74	0.095	26	0.923	16.1	39.3	7.90	0.133	.01563	0.633
E24	23.0	6.10	0.258	149	0.334	32	0.450	6.87	34.3	5.25	0.119	.01105	0.392
E25	16.4	2.97	0.384	95	0.197	26	0.558	7.54	32.4	4.79	0.101	.01029	0.657
E26	22.7	1.54	0.536	107	0.046	29	0.618	8.26	32.9	4.62	0.0419	.00567	0.917
E27	18.8	2.09	0.410	78	0.050	23	0.822	10.4	33.1	4.72	0.0914	.00914	0.627
E28	23.5	2.50	0.459	153	0.403	32	0.441	4.92	29.2	3.25	0.0469	.00511	0.702
E29	28.1	2.70	0.394	134	0.193	29	0.786	11.0	33.7	4.99	0.0755	.00799	0.603
E30	23.1	3.82	0.354	129	0.196	30	0.538	7.36	32.9	4.63	0.0890	.00884	0.541
H3	31.5	1.87	0.400	136	0.066	17	0.870	-	-	-	0.0516	.00440	-
H4	25.7	2.01	0.302	139	0.023	20	0.709	9.22	28.1	1.94	0.0555	.00406	-

Table C. 1. (contd) Parameter estimation. Bagasse preparation P1.

Run No.	K_1V	K_2V	α	HV	Φ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{a_T(1-\alpha)}$	$\frac{\alpha}{DI}$
J2	38. 2	2. 70	0. 411	138	0. 180	26	0. 999	11. 5	36. 4	3. 75	0. 0706	. 00713	0. 649
J3	56. 0	2. 31	0. 533	140	0. 196	22	1. 465	11. 8	41. 7	4. 75	0. 0604	. 00769	0. 842
J4	75. 3	2. 28	0. 532	148	0. 080	22	1. 997	12. 1	36. 6	3. 35	0. 0605	. 00669	0. 862
J5	42. 9	1. 57	0. 500	149	0. 078	22	1. 138	11. 9	29. 2	2. 15	0. 0416	. 00 431	0. 810
J6	39. 0	1. 87	0. 457	155	0. 067	22	1. 013	9. 52	27. 1	1. 73	0. 0486	. 00446	0. 728
J7	41. 4	1. 66	0. 496	136	0. 058	22	1. 075	6. 97	31. 2	2. 18	0. 0431	. 00427	0. 790
J8	42. 5	3. 45	0. 464	149	0. 233	27	1. 146	8. 08	33. 3	2. 65	0. 0930	. 00883	0. 732
J9	44. 1	3. 69	0. 411	185	0. 137	19	1. 189	14. 3	25. 8	1. 67	0. 0995	. 00859	0. 649
J14	55. 4	2. 40	0. 507	167	0. 671	28	1. 520	14. 1	33. 0	2. 79	0. 0659	. 00703	0. 775
J15	54. 8	2. 40	0. 482	176	0. 203	27	1. 504	19. 9	27. 6	2. 03	0. 659	. 00669	0. 737
J16	46. 7	2. 38	0. 518	128	0. 508	28	1. 245	9. 38	41. 0	4. 50	0. 0634	. 00767	0. 804
J17	38. 5	2. 80	0. 446	159	0. 301	27	1. 026	16. 0	29. 2	2. 41	0. 0746	. 00785	0. 692
J21	46. 5	1. 96	0. 498	93	0. 424	23	1. 204	8. 42	32. 6	1. 81	0. 0508	. 00465	0. 765
J22	57. 2	2. 40	0. 514	137	0. 591	28	1. 481	10. 2	27. 9	1. 72	0. 0622	. 00588	0. 789
J23	59. 5	2. 81	0. 467	153	0. 149	27	1. 661	16. 9	40. 1	3. 12	0. 0784	. 00822	0. 731
J24	67. 1	2. 82	0. 460	144	0. 402	23	1. 873	19. 4	44. 9	3. 28	0. 0787	. 00814	0. 720

Table C. 2. Parameter estimation. Bagasse preparation P2.

Run No.	K_1V	K_2V	α	HV	Φ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{\alpha_T(1-\alpha)}$	$\frac{\alpha}{D1}$
C6	26.2	1.45	0.614	113	.159	22	0.729	-	-	-	0.0403	.00535	0.852
C35	22.4	2.72	0.419	92	.306	22	0.661	5.08	32.1	2.06	0.0803	.00653	0.575
C43	12.0	1.05	0.405	169	0.062	23	0.314	5.16	16.9	7.72	0.0274	.00350	0.600
C44	44.5	1.36	0.552	92	0.089	17	1.184	18.3	74.9	5.69	0.0362	.00547	-
D18	11.6	5.72	0.250	151	0.145	27	0.301	2.79	12.5	3.03	0.149	.00990	0.360
D19	23.6	2.00	0.449	120	0.200	26	0.613	-	-	-	0.0519	.00471	0.647
D20	21.3	0.94	0.454	134	0.387	25	0.602	5.15	22.9	2.68	0.0266	.00234	0.664
D21	17.4	3.64	0.403	150	0.380	25	0.492	4.27	17.8	2.81	0.103	.00829	0.589
D34	29.6	1.58	0.597	109	0.075	26	0.823	7.73	32.3	2.81	0.0439	.00567	0.804
D35	34.8	2.62	0.554	96	0.095	26	0.968	8.94	40.9	2.68	0.0729	.00850	0.746
D36	43.7	2.18	0.525	83	0.078	24	1.144	8.58	46.4	1.88	0.0571	.00571	0.732
D37	49.3	2.17	0.592	114	0.272	26	1.291	9.76	41.2	1.92	0.0568	.00662	0.825
D42	50.3	1.97	0.707	123	0.682	25	1.307	5.87	30.9	2.05	0.0512	.00749	0.965
D43	38.0	2.63	0.595	156	0.099	25	0.987	8.97	22.1	1.12	0.0683	.00724	0.812
D50	29.0	5.19	0.449	144	0.269	25	0.769	7.08	27.8	2.79	0.138	.01278	0.601
D51	15.7	18.08	0.173	151	0.287	30	0.412	2.77	17.2	1.72	0.475	.02523	0.223
D52	6.7	9.42	0.249	145	0.218	32	0.176	1.23	12.9	1.92	0.248	.01448	0.321
D57	19.7	1.00	0.792	130	0.255	28	0.488	3.18	23.3	3.45	0.0247	.00620	1.114
D58	17.7	4.11	0.269	146	0.092	28	0.439	6.13	17.3	2.09	0.102	.00728	0.378

Table C. 3. Parameter estimation. Bagasse preparation P3.

Run No.	K_1V	K_2V	a	HV	ϕ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{\sqrt{T(1-a)}}$	$\frac{a}{DI}$
C19	28.6	1.04	0.743	125	0.154	27	0.739	3.37	16.6	1.04	0.0269	.00368	0.856
C20	37.0	0.94	0.667	91	0.210	25	0.981	2.16	10.6	0.269	0.0249	.00199	0.793
C21	29.8	1.00	0.538	99	0.078	23	0.790	1.83	11.1	0.313	0.0265	.00153	0.639
C25	36.7	1.71	0.632	82	0.258	23	1.068	2.27	10.9	0.301	0.0498	.00335	0.712
C26	27.8	1.05	0.803	108	0.503	27	0.704	2.32	13.6	0.570	0.0266	.00420	0.917
C27	36.9	1.88	0.791	125	0.160	26	0.934	-	-	-	0.0476	.00708	0.903
C39	32.6	1.20	0.703	100	0.235	26	0.876	2.80	16.3	0.569	0.0323	.00328	0.792
C54	49.0	1.94	0.614	123	0.203	22	1.195	-	-	-	0.0473	.00370	0.692
D11	30.6	2.99	0.540	98	0.311	26	0.827	4.05	20.3	1.09	0.0808	.00654	0.670
D12	26.2	10.35	0.344	122	0.375	26	0.681	2.77	13.3	0.795	0.269	.01409	0.421
D13	38.3	11.47	0.417	124	0.119	27	0.996	4.05	11.7	0.795	0.298	.01757	0.510
D23	38.1	10.04	0.407	143	0.185	27	1.029	5.69	11.1	0.570	0.271	.01495	0.482
D25	19.7	2.77	0.628	151	0.320	27	0.555	-	-	-	0.0780	.00631	0.745
D26	32.8	1.37	0.669	94	0.286	22	0.839	3.83	25.7	0.902	0.0351	.00391	0.821
D27	66.5	1.23	0.749	110	0.087	19	1.702	8.00	26.6	0.987	0.0315	.00463	0.919
D28	81.3	2.57	0.819	97	0.283	22	2.219	9.69	23.5	0.909	0.0701	.01367	0.994
D29	78.2	2.57	0.846	91	0.172	20	2.134	9.47	23.7	0.952	0.0701	.01606	1.027
D33	33.1	2.56	0.727	89	0.594	27	0.879	-	-	-	0.0680	.00794	0.838
E3	24.8	2.73	0.505	95	0.081	24	1.018	4.85	15.5	1.25	0.112	.00783	0.625
E5	12.3	6.32	0.594	137	0.415	26	0.378	1.43	7.91	0.777	0.194	.01540	0.701
E6	13.2	7.51	0.147	146	0.111	29	0.271	0.97	7.47	0.654	0.154	.00581	0.173
E7	30.6	10.82	0.308	72	0.302	26	0.954	3.00	16.2	0.556	0.338	.01457	0.366
E8	32.1	1.35	0.746	91	0.321	32	0.667	1.96	15.0	0.455	0.0281	.00330	0.885

Table C. 4. Parameter estimation. Bagasse preparation P4.

Run No.	K_1V	K_2V	α	HV	Φ_1	No. of points	K_1'	Sh_1	Re_1	Ga_1 ($\times 10^6$)	K_2'	$\frac{K_2}{a_T(1-\alpha)}$	$\frac{\alpha}{DI}$
C38	46.8	0.15	0.903	82	0.503	20	1.202	2.05	9.44	0.198	0.0038	.00089	0.963
C41	33.2	0.10	0.849	77	0.036	21	0.865	1.46	7.92	0.200	0.0025	.00038	0.912
C42	35.6	0.90	0.802	107	0.261	26	0.928	-	-	-	0.0235	.00269	0.861
D2	15.4	2.97	0.529	105	0.205	30	0.412	2.22	8.35	0.537	0.0794	.00509	0.589
D4	28.1	9.94	0.326	90	0.197	25	0.815	3.62	9.45	0.419	0.288	.01247	0.348
D5	16.5	2.88	0.670	128	0.279	26	0.449	2.14	6.22	0.472	0.0784	.00713	0.740
D6	15.0	4.80	0.837	96	0.867	28	0.409	0.96	8.43	0.791	0.131	.02406	0.925
D17	30.1	0.19	0.797	79	0.485	24	0.804	2.05	10.9	0.401	0.0051	.00068	0.908
D54	29.0	2.33	0.787	120	0.165	28	0.775	2.37	9.54	0.547	0.0623	.00856	0.880

APPENDIX D.

IMPURITY EXTRACTION DATA

Table D. 1 records results obtained operating the pilot plant with re-circulation of juice through the diffusion vessel. Measurements of juice pH and apparent purity were inconclusive, and are not given here. Measurements of inorganic impurities are given in section 5. 8. 5.

Table D. 2 gives the results of a limited number of 'once-through' percolation runs aimed at elucidating the rate of extraction of reducing sugars and inorganic species.

Table D. 1. Details of impurity extraction runs.

Run No.	Preparation.	Temp. (°C)	L (lb/min ft^2)	S (mm^2/g)	Brix at t =			Starch ppm on brix at t =			Reducing sugars % brix at t =		
					10 min.	30 min.	45 min.	10 min.	30 min.	45 min.	10 min.	30 min.	45 min.
G1	P1	73	58.6	3160	1.12	1.24	1.27	340	400	660	5.2	5.1	5.3
G2	P1	73	48.4	3250	0.97	1.04	1.06	390	600	470	-	-	-
G3	P1	67	48.4	-	0.93	0.98	1.04	740	450	420	8.8	8.9	8.8
G4	P1	78	48.4	2960	1.06	1.08	1.10	2180	2600	2610	5.0	5.7	5.6
G5	P1	59	48.4	2960	0.95	0.97	1.00	1380	1290	1000	12.5	14.6	15.1
G6	P3	73	43.6	5480	1.31	1.31	1.31	240	210	210	5.2	5.3	5.5
G7	P1	73	48.4	3740	0.89	0.93	0.95	490	340	-	6.7	6.6	6.8
G8	P1	87	48.4	3740	0.95	0.98	1.00	2660	2230	2630	10.0	9.9	9.7
G9	P1	59	48.4	4030	0.79	0.81	0.83	550	460	490			
G10	P1	78	48.4	4030	0.93	0.98	1.00	770	1120	1340			
G11	P1	69	48.4	4030	0.93	0.97	0.98	470	350	320			
G12	P1	87	48.4	4030	0.95	1.00	1.00	1880	2060	2090			
G13	P1	69	48.4	3750	0.79	0.85	0.85	400	330	370			
G14	P1	87	48.4	3750	0.91	0.95	0.97	2090	2100	2130			
G15	P1	59	48.4	3750	0.85	0.85	0.87	400	370	360			
G16	P1	78	48.4	3750	0.89	0.93	0.95	880	1680	1180			
G17	P1	73	48.4	3680	0.83	0.89	0.91	490	420	380			
G18	P1	69	48.4	3680	0.85	0.89	0.93	440	350	340			
G19	P1	78	48.4	3680	0.85	0.91	0.93	510	1100	1210			
G20	P1	73	48.4	3230	0.82	0.87	0.89	420	610	420			
G21	P2	73	48.4	4250	0.86	0.90	0.92	360	280	340			
G22	P1	73	58.6	3230	0.82	0.87	0.87	880	1110	1190			
G23	P1	73	48.4	3780	0.74	0.82	0.85	420	340	330			
G24	P3	73	37.6	6010	0.99	0.97	0.97	280	290	260			
G25	P4	73	25.6	7360	1.43	1.41	1.39	270	710	810			

Table D. 1. (contd) Details of impurity extraction runs

Run No.	Preparation	Temp. (°C)	L (lb/min. ft²)	S_2 (mm^2/g)	Brix at t =			Starch ppm on brix at t =			Reducing sugars % brix at t =		
					10 min.	30 min.	45 min.	10 min.	30 min.	45 min	10 min.	30 min.	45 min.
G26	P1	73	48.4	3480	0.89	0.96	0.97	220	260	320			
G27	P1	82	48.4	3480	0.92	0.96	0.97	1060	1240	1060			
G28	P1	78	48.4	3480	0.87	0.96	0.97	430	850	930			
G29	P1	69	48.4	3480	0.82	0.87	0.89	390	360	320			
J18	P1	73	44.6	3770	0.87	0.95	0.98	250	500	650			
J19	P3	73	34.1	5280	1.03	1.05	1.05	180	200	240			
J20	P2	73	37.6	4450	0.98	1.03	1.03	190	200	240			

Table D. 2. Results of once-through percolation runs.
Preparation P1, L = 48.4 lb/min. ft^2.

Run No.	Time (mins)	Brix	Reducing sugars % brix	Conductivity ash % brix	Conc. of K % brix	Conc. of Ca % brix	Conc. of Mg % brix	Conc. of Na % brix
H1 T = 80°C S = 3070 mm^2/g	0.5	2.59	10.6	4.1	1.35	0.22	0.14	0.05
	1	1.93	10.5	4.2	1.30	0.23	0.15	0.05
	2	1.26	9.7	4.4	1.32	0.21	0.15	0.06
	5	0.48	10.2	5.6	1.52	0.25	0.17	0.10
	10	0.19	12.6	8.3	2.10	0.16	0.26	0.10
H2 T = 67°C S = 3070 mm^2/g	0.5	2.57	16.1	4.3	1.36	0.23	0.16	0.05
	1	1.72	19.7	4.5	1.36	0.22	0.16	0.06
	2	1.35	16.3	4.6	1.33	0.21	0.18	0.06
	5	0.42	20.4	6.3	0.93	0.29	0.24	0.10
	10	0.13	20.7	8.2	2.54	0.23	0.23	0.15
H3 T = 87°C S = 4010 mm^2/g	1.25	1.65	10.8	5.1	1.66	0.21	0.17	0.08
	2	1.20	10.6	5.4	1.95	0.22	0.16	0.09
	5	0.35	13.3	6.9	2.41	0.29	0.14	0.14
H4 T = 59°C S = 4010 mm^2/g	0.5	2.15	18.0	5.4	1.77	0.23	0.19	0.07
	1	1.55	16.9	5.6	1.77	0.24	0.18	0.08
	2.5	0.76	18.3	6.2	1.97	0.25	0.20	0.09
	5	0.27	23.3	7.6	2.89	0.33	0.26	0.19
	10	0.14	32.1	9.2	3.57	0.39	0.29	0.21

APPENDIX E

SOLUTION OF MODEL EQUATIONS.

E. 1 Laboratory mixing tests.

The Laplace transformation of equations (3. 7) to (3. 9) leads to the following set of transformed equations:

$$- \alpha p \bar{C}_{b1} + \alpha C_{bo} = \bar{C}_{b1} - \bar{C}_j \qquad (E. 1)$$

$$- (1 - \alpha)\, p\, \bar{C}_{b2} + (1 - \alpha)\, C_{bo} = \beta (\bar{C}_{b2} - \bar{C}_j) \qquad (E. 2)$$

$$Q\, p\, \bar{C}_j - Q\, C_{jo} = \bar{C}_{b1} + \beta \bar{C}_{b2} - \bar{C}_j (1 + \beta) \qquad (E. 3)$$

The bar above a variable represents its Laplace transform, and p represents the Laplace transform variable.
From equation (E. 1):

$$\bar{C}_{b_1} = \frac{\alpha C_{bo} + \bar{C}_j}{\alpha p + 1} \qquad (E. 4)$$

similarly,

$$\bar{C}_{b2} = \frac{\gamma C_{bo} + \bar{C}_j}{\gamma p + 1} \qquad (E. 5)$$

where

$$\gamma = (1 - \alpha) / \beta \qquad (3. 23)$$

Substituting equations (E. 4) and (E. 5) into (E. 3) and rearranging leads to:

$$\bar{C}_j = \frac{C_{bo}\left[\alpha\gamma p(1+\beta)+1\right]}{\alpha\gamma Q p\left[p^2 + p\left(\frac{1}{\alpha}+\frac{1}{\gamma}+\frac{1+\beta}{Q}\right)+\frac{Q+1}{\alpha\gamma Q}\right]}$$

$$+ \frac{C_{jo}(\alpha p+1)(\gamma p+1)}{\alpha\gamma p\left[p^2 + p\left(\frac{1}{\alpha}+\frac{1}{\gamma}+\frac{1+\beta}{Q}\right)+\frac{Q+1}{\alpha\gamma Q}\right]} \qquad \text{(E. 6)}$$

In order to make the inverse transformation, the 2 terms in equation E. 6 are expanded in partial fractions. Thus:

$$\bar{C}_j = C_{bo}\frac{1}{Q}\left[\frac{A}{p}+\frac{B}{p+a}+\frac{C}{p+b}\right] + C_{jo}\left[\frac{D}{p}+\frac{E}{p+a}+\frac{F}{p+b}\right] \qquad \text{(E. 7)}$$

a and b are obtained as the roots of the term in square brackets in the denominators of the terms in equation (E. 6) and are given as equations (3. 24) and (3. 25). For equations (E. 6) and (E. 7) to be identical, the coefficients in equation (E. 7) must have the following values:

$$A = D = \frac{1}{\alpha\gamma a b}$$

Since,

$$ab = \frac{Q+1}{\alpha\gamma Q}$$

$$A = D = \frac{Q}{Q+1}$$

$$B = \frac{1-\alpha\gamma a(1+\beta)}{\alpha\gamma a(a-b)}$$

$$C = \frac{\alpha\gamma b(1+\beta)-1}{\alpha\gamma b(a-b)}$$

$$E = \frac{a^2 - a\left(\frac{1}{\alpha}+\frac{1}{\gamma}\right)+\frac{1}{\alpha\gamma}}{a(a-b)}$$

$$F = \frac{b\left(\frac{1}{\alpha}+\frac{1}{\gamma}\right) - b^2 - \frac{1}{\alpha\gamma}}{b(a-b)}$$

Now, from Erdélyi (1954), $\mathcal{L}^{-1}\left(\frac{1}{p-a}\right) = e^{at}$.

Therefore, inverse transformation of equation (E. 7) leads to:

$$C_j = C_{bo}\frac{1}{Q}\left[A + Be^{-a\theta} + Ce^{-b\theta}\right] + C_{jo}\left[D + Ee^{-a\theta} + Fe^{-b\theta}\right] \quad (E.8)$$

Substitution for the coefficients A to F in equation (E. 8) yields the final result, equation (3. 22).

E. 2 Plug Flow Percolation.

Laplace transformation of equations (3. 17) to (3. 19) with respect to the variable θ leads to:

$$\frac{d\bar{C}_j}{dN} + \bar{C}_j(1+\beta) - \bar{C}_{b1} - \beta\bar{C}_{b2} = 0 \quad (E.9)$$

$$\alpha p\bar{C}_{b1} - \alpha C_{b1}(0,N) + \bar{C}_{b1} - \bar{C}_j = 0 \quad (E.10)$$

$$(1-\alpha)p\bar{C}_{b2} - (1-\alpha)C_{b2}(0,N) + \beta\bar{C}_{b2} - \beta\bar{C}_j = 0 \quad (E.11)$$

Using the boundary condition, equation (3. 21), and putting $\gamma = (1-\alpha)/\beta$, equation (E. 10) and (E. 11) become:

$$\alpha p\bar{C}_{b1} - \alpha C_{bo} + \bar{C}_{b1} - \bar{C}_j = 0 \quad (E.12)$$

$$\gamma p\bar{C}_{b2} - \gamma C_{bo} + \bar{C}_{b2} - \bar{C}_j = 0 \quad (E.13)$$

From equation (E. 12):

$$C_{b1} = \frac{\bar{C}_j + \alpha C_{bo}}{\alpha p + 1} \quad (E.14)$$

and from equation (E. 13),

$$C_{b2} = \frac{\bar{C}_j + \gamma C_{bo}}{\gamma p + 1} \qquad \text{(E. 15)}$$

Introducing equations (E. 14) and (E. 15) into equation (E. 9), and re-arranging, leads to:

$$\frac{d \bar{C}_j}{d N} + \left(C_j - \frac{C_{bo}}{p}\right) \left[\frac{\alpha \gamma p^2 (1 + \beta) + p}{(\alpha p + 1)(\gamma p + 1)} \right] \qquad \text{(E. 16)}$$

Integration of equation (E. 16) gives:

$$\bar{C}_j - \frac{C_{bo}}{p} = A \exp \left[- \frac{(\alpha \gamma p^2 (1 + \beta) + p) N}{(\alpha p + 1)(\gamma p + 1)} \right] \qquad \text{(E. 17)}$$

The integration constant A is evaluated using the boundary condition, equation (3. 20):

$$\text{At} \quad N = 0 , \; C_j = 0$$

$$\text{Hence} \quad A = -C_{bo} / p$$

Therefore,

$$\bar{C}_j = \frac{C_{bo}}{p} \left\{ 1 - \exp \left[- \frac{(\alpha \gamma p^2 (1 + \beta) + p) N}{(\alpha p + 1)(\gamma p + 1)} \right] \right\} \qquad \text{(E. 18)}$$

The exponential term in equation (E. 18) can be simplified.

It can easily be shown by simple rearrangement, that

$$\frac{\alpha\gamma p^2(1+\beta)+p}{(\alpha p+1)(\gamma p+1)} = (1+\beta) - \frac{\frac{1}{\alpha}}{p+\frac{1}{\alpha}} - \frac{\frac{\beta}{\gamma}}{p+\frac{1}{\gamma}}$$

Therefore, equation (E. 18) may be re-written as

$$\bar{C}_j = \frac{C_{bo}}{p} - \frac{C_{bo}}{p} \exp\left[-(1+\beta)N + \frac{N/\alpha}{p+1/\alpha} + \frac{\beta N/\gamma}{p+1/\gamma}\right] \quad \text{(E. 19)}$$

Direct inverse transformation of equation (E. 19) is difficult. It can be manipulated into the following form:

$$\bar{C}_j = \frac{C_{bo}}{p} - \frac{C_{bo}}{p} \exp\left[-(1+\beta)N\right] \left\{\left[\exp\left(\frac{N/\alpha}{p+1/\alpha}\right)-1\right]+1\right\}$$

$$\left\{\left[\exp\left(\frac{\beta N/\gamma}{p+\frac{1}{\gamma}}\right)-1\right]+1\right\}$$

$$= \frac{C_{bo}}{p} - \frac{C_{bo}}{p} \exp\left[-(1+\beta)N\right] \left\{1+\left[\exp\left(\frac{N/\alpha}{p+1/\alpha}\right)-1\right]\right.$$

$$\left. + \left[\exp\left(\frac{\beta N/\gamma}{p+\frac{1}{\gamma}}\right)-1\right] + \left[\exp\left(\frac{N/\alpha}{p+\frac{1}{\alpha}}\right)-1\right]\left[\exp\left(\frac{\beta N/\gamma}{p+\frac{1}{\gamma}}\right)-1\right]\right\} \quad \text{(E. 20)}$$

From Erdélyi (1954), $\mathcal{L}^{-1}\{e^{a/p}-1\} = a^{\frac{1}{2}} t^{-\frac{1}{2}} I_1(2\sqrt{at})$

$$\therefore \mathcal{L}^{-1}\{e^{\frac{a}{p+b}}-1\} = a^{\frac{1}{2}} t^{-\frac{1}{2}} I_1(2\sqrt{at})\, e^{-bt} \quad \text{(E. 21)}$$

Using this relation (E. 21), the inverse transformation of equation (E. 20) can be obtained by convolution as:

$$C_j / C_{bo} = 1 - e^{-(1+\beta)N} \left[1 + \int_o^\theta e^{-\frac{s}{\alpha}} \sqrt{\frac{N}{\alpha s}}\ I_1 \left(2\sqrt{\frac{Ns}{\alpha}}\right) ds \right.$$

$$+ \int_o^\theta e^{-\frac{s}{\gamma}} \sqrt{\frac{\beta N}{\gamma s}}\ I_1 \left(2\sqrt{\frac{\beta N s}{\gamma}}\right) ds$$

$$+ \left. \int_o^\theta \int_o^s N\, e^{-\frac{\tau}{\alpha} + \frac{s-\tau}{\gamma}} \sqrt{\frac{\beta}{\alpha\gamma\tau(s-\tau)}}\ I_1 \left(2\sqrt{\frac{N\tau}{\alpha}}\right) I_1 \left(2\sqrt{\frac{\beta N(s-\tau)}{\gamma}}\right) d\tau\, ds \right] \qquad \text{(E. 26)}$$

In order to obtain solutions for C_{b1} and C_{b2}, equation (E. 19) is substituted in equations (E. 14) and (E. 15) respectively. Substituting in equation (E. 14) leads to:

$$\bar{C}_{b1} = \frac{\alpha C_{bo}}{\alpha p+1} + \frac{C_{bo}}{p(\alpha p+1)} - \frac{C_{bo}}{p(\alpha p+1)} \exp\left[-(1+\beta)N + \frac{N/\alpha}{p+1/\alpha} + \frac{\beta N/\gamma}{p+1/\gamma} \right]$$

$$= \frac{C_{bo}}{p} - \frac{C_{bo}}{\alpha p} \exp\left[-(1+\beta)N \right] \frac{\exp\left(\frac{N/\alpha}{p+1/\alpha}\right)}{p+1/\alpha} \left[1 + \left(\exp\left(\frac{\beta N/\gamma}{p+1/\gamma}\right) - 1 \right) \right]$$

(E. 22)

From Erdelyi (1954), $\mathcal{L}^{-1}\left\{ \frac{1}{p} \exp\left(\frac{a}{p}\right) \right\} = I_o\left(2\sqrt{at}\right)$

$$\therefore\ \mathcal{L}^{-1}\left\{ \frac{1}{(p+b)} \exp\left(\frac{a}{p+b}\right) \right\} = e^{-bt}\ I_o\left(2\sqrt{at}\right) \qquad \text{(E. 23)}$$

Utilizing the inverse transform relations (E. 21) and (E. 23), equation (E. 22) can be inverse transformed to yield:

$$C_{b1} = C_{bo} - \frac{1}{\alpha} C_{bo} e^{-(1+\beta) N} \left[\int_o^{\theta} e^{-\frac{s}{\alpha}} I_o \left(2 \sqrt{\frac{N s}{\alpha}}\right) ds \right.$$

$$\left. + \int_o^{\theta} \int_o^{s} e^{-\left(\frac{\tau}{\alpha} - \frac{s-\tau}{\gamma}\right)} \sqrt{\frac{\beta N}{\gamma (s-\tau)}} I_o \left(2 \sqrt{\frac{N \tau}{\alpha}}\right) I_1 \left(2 \sqrt{\frac{\beta N (s-\tau)}{\gamma}}\right) d\tau \, ds \right]$$

(3.27)

The solution for C_{b2} follows the same procedure as shown above to yield equation (3.28); by inspection, it can be seen that it has the same form as equation (3.27).

E. 3 Moving Bed Diffuser.

Since in this case C_{b1} and C_{b2} are independent of N, equation (6.7) can be integrated, using boundary condition (6.10) to give:

$$C_j = \frac{1}{1+\beta} \left(C_{b1} + \beta C_{b2} \right) \left(1 - e^{-(1+\beta)N}\right) + C_j^{(n+1)} e^{-(1+\beta)N}$$

(E. 24)

The integral in equations (6.13) and (6.14) can then be evaluated as:

$$\frac{1}{N} \int_o^N C_j \, d N = \frac{1-A}{1+\beta} \left(C_{b1} + \beta C_{b2} \right) + A C_j^{(n+1)} \qquad \text{(E. 25)}$$

where A is defined by equation (6.35). Substituting for the integral in equations (6.13) and (6.14) leads to:

$$\alpha \frac{d C_{b1}}{d \gamma} + \frac{A + \beta}{1 + \beta} C_{b1} - \frac{\beta (1-A)}{1 + \beta} C_{b2} = A C_j^{(n+1)} \qquad \text{(E. 26)}$$

$$\gamma \frac{d C_{b2}}{d \gamma} + \frac{1 + A \beta}{1 + \beta} C_{b2} - \frac{(1-A)}{1 + \beta} C_{b1} = A \beta C_j^{(n+1)} \qquad \text{(E. 27)}$$

These 2 ordinary differential equations are solved by routine methods to give:

$$C_{b2} = c_1 e^{m_1 y} + c_2 e^{m_2 y} + C_j^{(n+1)} \qquad \text{(E. 28)}$$

$$C_{b1} = \frac{c_1}{1-A} \left(\gamma m_1 (1+\beta) + 1 + A\beta \right) e^{m_1 y} + \frac{c_2}{1-A} \left(\gamma m_2 (1+\beta) \right.$$

$$\left. + 1 + A\beta \right) + C_j^{(n+1)} \qquad \text{(E. 29)}$$

where m_1 and m_2 are the roots of the auxilliary equation, given in equation (6. 33). In practice it is found that these roots are always real and unequal.

The constants c_1 and c_2 are evaluated from the boundary conditions, equations (6. 11) and (6. 12):

$$c_1 = a_o C_j^{(n+1)} + a_1 C_{b1}^{(n-1)} + a_2 C_{b2}^{(n-1)} \qquad \text{(E. 30)}$$

$$c_2 = b_o C_j^{(n+1)} + b_1 C_{b1}^{(n-1)} + b_2 C_{b2}^{(n-1)} \qquad \text{(E. 31)}$$

where the a_i and b_i are given in equations (6. 18) to (6. 23). Substituting for c_1 and c_2 in equation (E. 28) and (E. 29) leads to equations (6. 17) and (6. 16) respectively. Then simple substitution of equations (6. 16) and (6. 17) in equation (E. 24) leads to the final solution for C_j, equation (6. 15).

APPENDIX F.

MULTILINEAR REGRESSION ANALYSIS PROCEDURES.

In general, it was required to correlate a given variable in terms of other related measurable quantities. A relationship of the following form was generally sought:

$$y = A\, x_1^{a_1}\, x_2^{a_2}\, x_3^{a_3} \ldots\ldots \qquad (F.1)$$

In order to perform a linear regression analysis, it was first necessary to transform the variables to furnish a linear relation. This was achieved by taking the log of each variable. Then the objective becomes to obtain a correlation in the following form:

$$Y = b_o + b_1 X_1 + b_2 X_2 + b_3 X_3 + \ldots\ldots \qquad (F.2)$$

where $Y = \log y$ and $X_i = \log x_i$. The regression coefficients in equation (F. 2) are related to the constants in equation (F. 1) by:

$$\log A = b_o$$

$$a_i = b_i \,, \qquad i > 0$$

The I.C.L. statistical package was used to perform the multilinear (or multiple linear) regression analyses. A number of alternative analysis procedures are available in this program. In the procedure used here, a dependent variable, a subset of independent

variables to be considered by the program, and a significance level for the contribution of any independent variable in the subset is specified for each analysis. The program then follows an iterative procedure, successively including and/or removing variables from the regression set. The final regression equation includes only those variables which are significant at the specified significance level.

This procedure was found to be extremely useful. In order to obtain a regression equation for a given dependent variable, any possible independent variables could be specified for consideration by the program, which then selected only the significant variables from the subset of all possible variables. In all cases, a significance level of 5% was specified.

In a few cases, a set of observations for a run was incomplete (for example, a value of bed height may be missing for the run). The program included the facility for handling missing values in the observation matrix. For certain parts of the analysis, a complete observation matrix is required and so the program substituted for missing elements values derived from those already known (in this case by a 2nd order regression procedure). The calculation procedure however is designed in such a way that contributions to the cross product and co-variance matrices from the missing values are omitted, with the result that the significance tests and regression coefficients are not affected by the substitution for missing values.

APPENDIX G.

PHYSICAL PROPERTIES.

G1. Liquid Viscosity.

Measurements of the viscosity of low brix sucrose solutions have been reported by Brüniche-Olsen (1962) covering the range 0 to 20 brix in 5 brix intervals, over a temperature range of 23° - 70°C. Values of μ for 0 and 20 brix solutions are also reported by Perry (1963) and extend over a wider temperature range. Good agreement between the 2 sets of values at 0 and 20 brix supports the reliability of Brüniche-Olsen's measurements.

Average values of 2 and 5 brix were assumed for the pilot plant and full-scale diffuser juice concentrations respectively, and values of μ were obtained by interpolation from the published values at these brix values. Deviations from these average values are small enough so that the values of viscosity are accurate to within 5% for most of the juice brix values observed.

The values of μ used are tabulated below.

Table G1. Values of liquid viscosity (in cp.)

	55°C	60°C	65°C	70°C	75°C	80°C	85°C	90°C
2 brix	0.53	0.49	0.45	0.42	0.39	0.37	0.35	0.34
5 brix	0.59	0.54	0.50	0.47	0.44	0.41	0.39	0.38

These values are accurately represented by the following quadratic equations:

at 2 brix, $\mu = 1.273 - 0.0184\ T + 0.0000884\ T^2$ (G. 1)

at 5 brix, $\mu = 1.397 - 0.0201\ T + 0.0000976\ T^2$ (G. 2)

Values of μ were converted from cp to lb/min ft by multiplying by 0.0402.

G. 2 Liquid density.

A constant value of 61.0 lb/ft^3 was used throughout for the liquid density. Calculations from tabulated values of ρ (Spencer & Meade, 1945) show that over the range of brix and temperature encountered, the maximum error introduced is 1%.

G. 3 Molecular Diffusion Coefficient.

Brüniche-Olsen (1962) expressed the results of a comprehensive series of measurements of D_m for sucrose in water by the following equations:

$$\text{at } 23^{\circ}C,\ D_m = (5.0 - 0.05C) \times 10^{-6}\ cm^2/sec \qquad (G.3)$$

where C represents the brix of the solution, and

$$D_m = D_{mo}\ (1.025)^{T-T_o} \qquad (G.4)$$

for all brix values up to 15 brix. D_{mo} represents the value of D_m at temperature T_o.

Combining the 2 equations leads to

$$D_m = (5.0 - 0.05C)\ (1.025)^{(T-23)} \times 10^{-6}\ cm^2/sec \qquad (G.5)$$

This equation was utilized to calculate values of D_m. A multiplication factor of 0.0646 was applied to express values of D_m in ft^2/min.

G. 4 Density of Dry Fibre.

The commonly used figure of 1. 52 g/cm^3 was employed for the density (no-void) of dry fibre. This figure is also given by Jenkins (1966). Multiplying by 62. 43 to convert the units leads to a figure of 94. 9 lb/ft^3.