"THE DEVELOPMENT OF A THREE-PHASE FILTRATION CELL"

by

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As the candidate's Supervisor I agree/do not agree to the submission of this thesis.

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ABSTRACT

The chemical and process industries rely largely on filtration processes to separate solid-liquid process streams. Their industrial applications include water purification, processing streams in a refinery, and numerous others. Three-phase reactor vessels require contact between a gas, liquid and solid catalyst phase. The concept of three-phase filtration vessels stems from the need to separate the catalyst from the gas-liquid mixture in situ.

This investigation focused on the design and development of a three-phase filtration cell. Although the system is frequently applied, knowledge about their operation and development is limited. This dissertation serves to provide the knowledge required to develop competency in this field.

Prior to conducting any experimental work, a detailed literature review was conducted. Here, information was obtained that described the filtration process, the mechanism of filtration and the types of three-phase reactor vessels. This was done so as to assist in the design and construction of the test rig required for the investigation.

The test rig was designed to allow for both two-phase and three-phase filtration experiments to be conducted and compared. The test rig resembled a Nutsche filtration vessel, which was modified to include a sparger to introduce the third phase during filtration. After commissioning, experiments were then conducted by varying a single factor at a time and monitoring the system behaviour, through determination of the filtration constants (α_{av} and R_m). The factors that were varied were the solids concentration in the slurry, applied pressure to the system and the pore size of the filter medium. These initial sets of experiments allowed for the operational range of factors to be determined, although they could not provide any indication of the combined effects of these factors on the filtration behaviour. The solids concentration in the slurry was varied from 1% mass/mass to 10% mass/mass, the pressure was varied between 1.5bar abs and 2.5bar abs, and a 10µm and 20µm filter cloth was used.

The α_{av} values for both the two-phase and three-phase experiments ranged from 5.067×10^8 m.kg^{-1}- 1.2×10^9 m.kg^{-1} and the R_m values ranged from 1.051×10^{10} m^{-1} - 1.515×10^{10} m^{-1}. The values of the filtration constants for diatomaceous earth were not readily available in literature, however these calculated values were similar to those obtained in literature for other materials (such as talc and kaolin).

A statistical design of experiments was performed in order to indicate which parameters, and their combinations, affected the system behaviour. An *ANOVA* analysis indicated the level of significance of the parameters on the system. The analysis showed that the two-phase and three-phase systems were generally influenced by the same factors (either the applied pressure, solids concentration of the slurry, filter cloth pore size or a combination of these factors) and experienced the same level of significance. The effect of the applied pressure and solids concentration on the α_{av} was significant in both the two-phase and three-phase system. The applied pressure and filter cloth pore size had a combined effect on the α_{av} for the two-phase experiments, but this was not so for the three-phase experiments. The R_m for the two-phase and three-phase systems was influenced by the applied pressure and solids concentration as well as the combined effect of the applied pressure and solids concentration. Regression models were also fitted to the data. The adequacy of these models was then tested using normal probability plots and found to be a suitable description of the behaviour of the filtration systems. The correlation coefficients for the actual versus the predicted values from the regression model were also calculated. These ranged from 0.72 to 0.97, indicating a suitable fit of the model to the data.

NOMENCLATURE:

Notation: | | Units:

Notation:		Units:
A	Area	m^2
c	Mass of dry cake solids per unit volume of filtrate	kg solids per m^3 filtrate
D	Diameter	m
D_p	Diameter of the particle	m
e	Residual value	-
H_o	Null hypothesis	-
H_1	Alternative hypothesis	-
H_{slurry}	Height of the slurry in the filtration column	m
L	Length/Cake thickness	m or mm
m	Ratio of wet to dry cake masses	$kg.kg^{-1}$
m_{dc}	Mass of dry cake	kg
m_s	Mass of filtrate collected at time t_s	g
m_{solid}	Mass of solid	kg
m_{water}	Mass of water	kg
m_{wc}	Mass of wet cake	kg
MS	Mean sum of squares	-
n	Cake compressibility index	Dimensionless
r	Radius of filtration column	m
R	Radius of filter cloth	m
R^2	Coefficient of multiple determination	-
R_m	Filter medium resistance	m^{-1}
s	Mass fraction of solids in the feed slurry	$kg.kg^{-1}$
S_o	Specific surface area of particle	m^2 of particle area per m^3 volume of solid particle
SS	Sum of squares	-
t	Time	s
t_{2p}	Time at which the three-phase experiments transition to two-phase filtration	mm:ss
t_s	Start up time	s

v	Velocity	$m.s^{-1}$
v'	Superficial velocity	$m.s^{-1}$
V	Volume /Volume of filtrate collected up to time t	m^3
V_s	Start up volume of filtrate collected at time t_s	m^3

Greek letters:

α	Level of significance	-
α_{av}	Specific cake resistance	$m.kg^{-1}$
α_0	Specific cake resistance at unit compressive pressure	$m.kg^{-1}$
ΔP	Pressure drop	bar *or* Pascal
ΔP_c	Pressure drop across the filter cake	bar *or* Pascal
ΔP_m	Pressure drop across the filter medium	bar *or* Pascal
ε	Voidage/Porosity of the bed	Dimensionless
μ	Viscosity	bar.s *or* Pa.s
μ_{water}	Viscosity of water	bar.s *or* Pa.s
ρ	Density	$kg.m^{-3}$
ρ_{solid}	Density of the solid particles	$kg.m^{-3}$
ρ_{water}	Density of water	$kg.m^{-3}$
ϕ	Sphericity of particles in a packed bed	Dimensionless

Subscripts and superscripts:

A	with respect to factor *A*
B	with respect to factor *B*
C	with respect to factor *C*
AB	with respect to the interaction between factor *A* and *B*
AC	with respect to the interaction between factor *A* and *C*
BC	with respect to the interaction between factor *B* and *C*
ABC	with respect to the interaction between factor *A*, *B* and *C*
E	with respect to the error term
R	with respect to the regression
T	with respect to the total value

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CHAPTER 1:

INTRODUCTION

1.1. Scope of the investigation

The chemical and process industries rely largely on filtration processes to separate solid-liquid process streams. Their industrial applications include water purification, processing streams in a refinery, and numerous others. Filtration is generally done through the application of an applied force which forces this suspension through a permeable membrane. The solid particles are generally retained on the surface of this medium whilst the liquid filters through. Filtration processes are frequently applied due to its simplicity of execution. The particles in the suspension can be of various shapes or sizes, or could be present in a large or small concentration. The fluid could be either a gas or liquid, and the liquid suspension could be highly viscous or not. The principle underlying the filtration process remains the same for all system variations, what changes is the filtration technique used to execute the separation.

Three-phase filtration systems can be applied in three-phase reactor vessels. These vessels are frequently used in the chemical, metallurgical, pharmaceutical and process industries (Zaruba, et al., 2005). These reactor types involve bringing a gas, liquid and solid into contact for the reaction to occur. In these reactors the liquid phase is generally stationary, the gas phase is the dispersed phase and the solid particles are usually those of the catalyst. The solid catalyst may need to be separated from the liquid product through filtration. However, external filtration is not deemed feasible as the slurry mixture would now be exposed to alternate conditions and the slurry would have to be pumped to a new location, introducing additional costs (Shah, 1979). The relocation of the slurry may also lead to catalyst deactivation. This unwanted situation can be avoided by conducting internal filtration within the column.

By using three-phase filtration, the relocation of the slurry is avoided and the filtration of the slurry occurs within the reactor vessel. Their industrial use is wide-spread, be it in the Fischer-Tropsch synthesis, catalytic coal liquefaction processes, catalytic hydro-cracking of petroleum fractions and numerous others listed to a great degree by Shah (1979, pages 2-5).

This investigation focused on the design and development of a three-phase filtration cell. Although their application is frequently applied, knowledge about the principles and theory that govern their operation and development is limited. This dissertation serves to provide the knowledge required to develop competency in this field.

1.2. Project objectives

The objectives of this project can be stated as follows:

- Discuss the filtration process and the mechanism of filtration along with the range of three-phase reactor types and their modes of operation. This will be done to ensure that the necessary knowledge required is obtained prior to embarking on test rig design and experimentation.
- Construct a test rig that would allow for both two-phase and three-phase filtration experiments to be conducted and compared.
- Conduct a statistical design of experiments in order to allow for constructive data analysis which will allow for the effects of various factors on the system performance to be examined.
- To fit a model that describes the behaviour of the system, describing the important parameters which influence the filtration behaviour, and to provide recommendations for further study.

1.3. Structure of dissertation

The structure of this dissertation would be compiled to illustrate how the three-phase system was studied, developed and its performance assessed. The chapter that follows (Chapter 2) is divided into sections that detail the filtration process, the methods of filtration, the filtration system design and factors affecting the performance of the filtration process. This chapter also discusses the types of three-phase reactors, their mode of operation and the factors that influence their performance. Lastly, the methods of statistical experimental design are further discussed.

Chapter 3 deals with the design, construction and commissioning of the test rig and the finely tuned experimental procedure that was developed to achieve peak performance.

The discussion regarding the decisions made follows in Chapter 4, along with the presentation and analysis of the results obtained and the determination of the important factors for two-phase and three-phase test systems. Chapter 4 also describes the behaviour of the system by illustrating the fit of a regression model from the data obtained.

Chapter 5 provides a set of conclusions drawn and recommendations that could be considered in further studies.

Lastly, the Appendices follow, which contain the raw data, sample calculations, graphs and other important information that may be consulted.

CHAPTER 2:

LITERATURE REVIEW AND THEORETICAL BACKGROUND

2.1. Chapter overview

Literature in the field of three-phase filtration is limited, as this field is not well developed. In order to develop a system that is fully operational and meets the constraint of reproducing the behaviour within a bubble column reactor, a literature review would certainly have to be conducted. The objectives of the literature review conducted are as follows:-

-   Gain an understanding of the process of filtration, the methods, devices and factors affecting its operation.
-   Obtain information on three-phase reaction vessels, the type of reactors available and the important factors that affect their operation.
-   Acquire any information possible that would assist in the design of a test rig that would incorporate a third phase into the filtration process.
-   Employ the experience of previous research investigations to determine the method of experimentation and the factors that would be analysed during the experiments.
-   Determine a method of data analysis that would quantify the performance of the filtration test system.
-   Obtain information that would assist in experimental design that could be used to assess the filtration behaviour.

The sections that follow encompass the listed objectives with the actual test rig design and construction following in Chapter 3.

2.2. Separation processes

Most chemical processes require the use of separation methods within the production process in order to obtain the desired product, or to separate phases during a stage of the chemical process. Their industrial applications include use within mineral processing plants, waste water treatment, paper making plants, etc.

The separation of phases can be done for gas-liquid, vapour-liquid, liquid-liquid and liquid-solid mixtures; each of which have a specialized method that allows for the separation to occur. Most of these methods depend on physical-chemical differences of the molecules to be separated as well as on mass transfer of the molecules, thus allowing for separation to occur based on molecular differences.

Some separation techniques rely on mechanical-physical forces rather than on molecular or chemical forces or diffusion. These mechanical-physical forces include gravitational and centrifugal forces, physical forces, and kinetic forces arising from flow. These separation techniques include filtration, settling and sedimentation, centrifugation and expression.

2.3. Separation process stages

Any separation system design has to consider all possible stages of the separation process. The focus of this investigation is on solid-liquid separation with the introduction of a gas phase. Thus, filtration will be considered, along with the separation process stages that assist in its execution. These stages include:
- Pre-treatment
- Solids Concentration
- Solids Separation
- Post-treatment

These stages require the use of various types of equipment and techniques, which are best described by Wakeman and Tarleton (1999a, page 2) as presented in Figure 2-1:
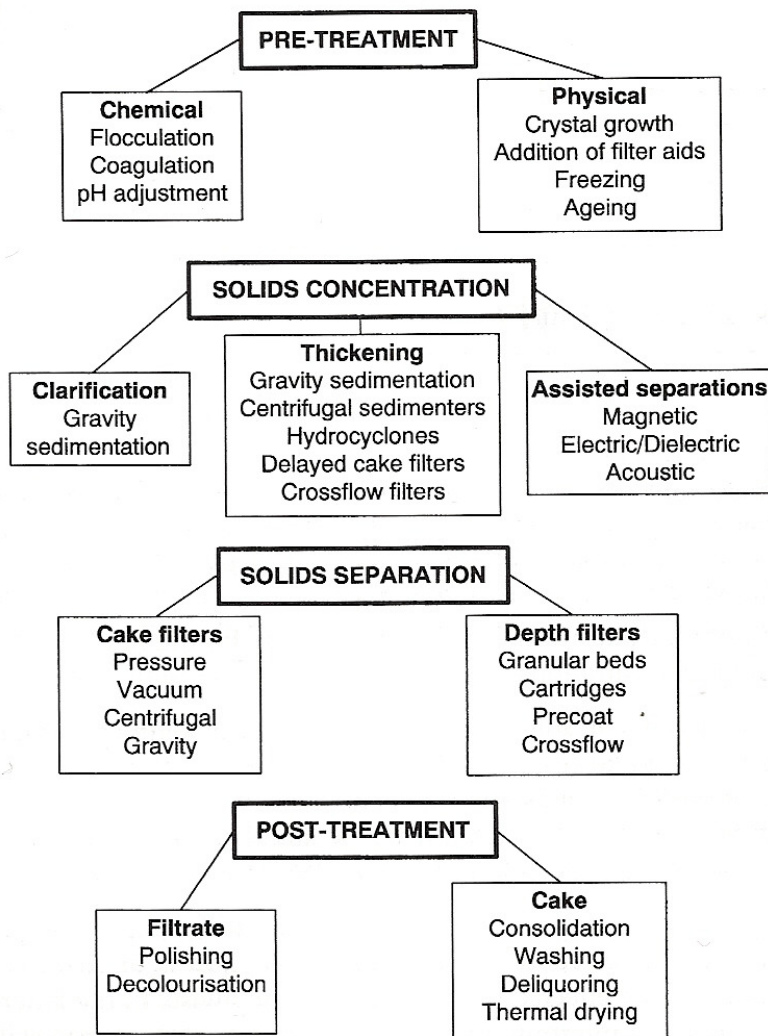
Figure 2-1: Components of the solid-liquid separation process (*Source: Wakeman & Tarleton, 1999a, page 2, Figure 1.1)*

2.3.1. Pre-treatment

These techniques are done to facilitate subsequent processing of the suspension. For example, pre-treating slurries that are difficult to filter can improve the filterability of the suspension. Pre-treatment methods usually involve changing the nature of the solids within the suspension by chemical or physical means. Pre-treatment could also include the use of filter-aids (discussed in section 2.6.5.).

2.3.2. Solids concentration

Solids concentration methods allow for the filter cake to be formed more readily by removing some of the liquid from the suspension, and thus decreasing the throughput load on the filter medium.

2.3.3. Solids separation

This describes the use of filtration methods, be it cake filters or deep bed filters etc., to execute the actual separation process. This is further discussed in section 2.4.

2.3.4. Post-treatment

Post-treatment is done to further enhance the degree of separation and improve the quality of the final product. For liquid products, the filtrate may be further purified by removing small contaminants through the process of polishing. If the cake is the desired product, this is done by washing soluble impurities from the cake voids and removing any excess liquid from the cake voids.

2.4. Filtration

This research project requires the development of a filtration cell that can be used to study three-phase filtration and thus required a good understanding of the filtration process. To gain further information regarding the developments in the field of filtration, the author attended the tenth annual World Filtration Conference in April 2008. The conference was held in Germany and lectures and presentations which concentrated on solid-liquid separation were attended. The conference also provided the opportunity to network with several other research institutions who showed keen interest to assist with the investigation and provide any advice if necessary. In addition, the conference included an exhibition which featured various filtration and separation equipment and devices. Here, samples of filter media were obtained along with ideas for modification of the proposed test rig design. The contact details of several members of industry and manufacturers were also acquired. A combination of the knowledge

gained at the conference as well as research conducted, provided the details regarding the filtration process, the stages in filtration and filtration system design that are discussed in the sections that follow.

Filtration involves the separation of two phases, solid and liquid, from a suspension. This is done through the use of a separating agent, which could be energy, matter or even both (Tien, 2006). In filtration the suspended solid particles in the liquid mixture are removed by forcing the suspension flow, physically or mechanically, through the porous medium. Thus, this allows for the solids to accumulate on the surface of the porous medium whilst allowing for the liquid to flow through the medium. The separating agents for a filtration process are viewed to be both energy and matter. The energy is usually viewed as that which drives the flow of the suspension and the matter to be that which allows for the separation, i.e. the filter media (Tien, 2006).

Since the filtration process provides a distinct separation of two phases, either of the phases, or even both may be the desired product:
-   The solid component may be recovered as the desired product with the liquid being discarded
-   The liquid component being the preferred product with the solid not needed
-   Recovery of both phases as being valuable
-   Neither phase being required, as in the case of purifying water prior to its discharge.

Filtration generally occurs through the use of an applied force to promote the separation of the different phases. These driving forces include the use of a vacuum, pressure or through an applied centrifugal force (Gösele & Alt, 2005). Vacuum filtration requires a vacuum to be created that sucks the slurry through the permeable medium in order for the separation to occur. Pressure filtration occurs via pressure being applied to the slurry mixture, forcing the slurry to flow through the permeable medium, thus, promoting separation. Centrifugal filtration occurs when a centrifugal force is applied within a centrifuge with a perforated rotor, resulting in the phase separation.

The filtration process has varied use due to the simplicity of execution. The particles in the suspension can be of various shapes or sizes or be present in a large or small concentration. The fluid could be either a gas or liquid, with the liquid suspension being highly viscous or not. The principle underlying the

filtration process remains the same for all system variations, what changes is the filtration technique used to execute the separation.

2.4.1. Types of filtration techniques

Tien (2006) discusses cake filtration in his book with his aim being to provide information for those interested in cake filtration research and developmental work. He also describes, in less detail, the other types of filtration that can occur, namely, deep bed and cross-flow filtration. The types of filtration processes are discussed below, with each being compared to cake filtration, as this would be used in this investigation. Details are also given to provide an idea of when to favour the use of one method over the other.

2.4.1.1. Cake/Surface filtration

Cake filtration or surface filtration describes the method of filtration that utilizes a porous medium, through which the solid-liquid suspension flows. The solid material (i.e. the filter cake) accumulates on the upstream side of the filter medium and the liquid (i.e. the filtrate) passes through the filter medium. After a short initial period, the deposited solids form a cake through which the liquid must pass. The process continues, increasing the height of the cake and in turn, the resistance to filtrate flow. If the cake structure is able to withstand the force applied on it during the filtration process and produce a uniform, time-dependant cake porosity, the cake is considered to be incompressible. If a porosity gradient exists through the height of the cake based on the particle arrangement, the cake is considered to be compressible. For a compressible cake, the maximum solids concentration occurs at the interface between the cake and the filter medium (Tarleton, 1999). The process continues until the space available for cake formation is filled to capacity or until the filtrate flow is reduced to an uneconomical level.

Cake filtration is the most widely employed method of filtration in the process industries and is well suited to the filtration of concentrated suspensions and the recovery of large quantities of solid (Orr, 1977).
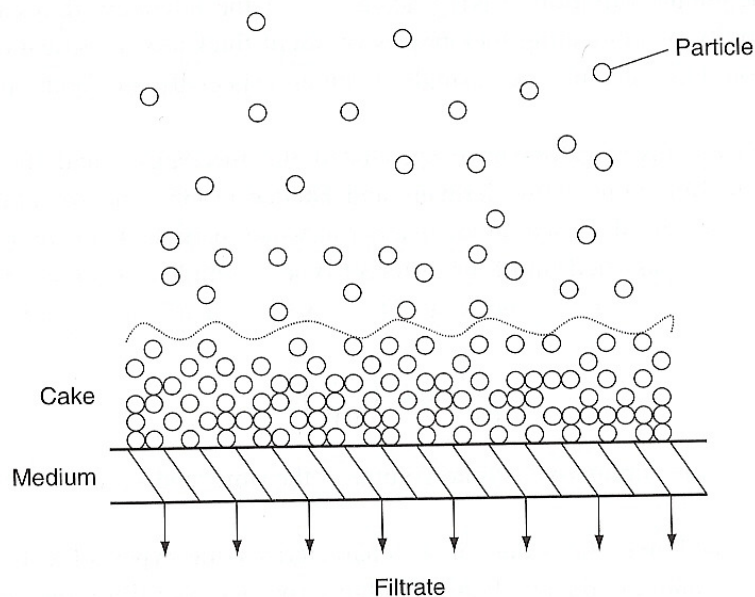
Figure 2-2: Cake filtration (*Source: Tien, 2006, page 5, Figure 1.5)*

2.4.1.2. Deep bed/Depth filtration

Deep bed filtration and cake filtration both utilize a porous medium to allow for the separation of solids to occur. The difference in the methods lies in the roles played by the filter medium. In cake filtration, the filter medium acts as a screen that allows for the solid particles to be retained by the medium, thus forming a filter cake. However with deep bed filtration, separation is executed by the solid particles being deposited throughout the entire depth of the medium. This method of filtration is commonly used for clarification of drinking water through the use of sand filters. Cleaning these filters is usually done by back-flushing, reversing the flow to remove the deposited solids.

For practical applications, cake filtration is preferred when treating suspensions of a high solid concentration whereas deep bed filtration is preferred when dealing with the separation of suspensions of a low solid concentration. In deep bed filtration, once the filter bed has been saturated with solids, the solids concentration in the filtrate begins to approach that of the feed slurry (Gösele & Alt, 2005).
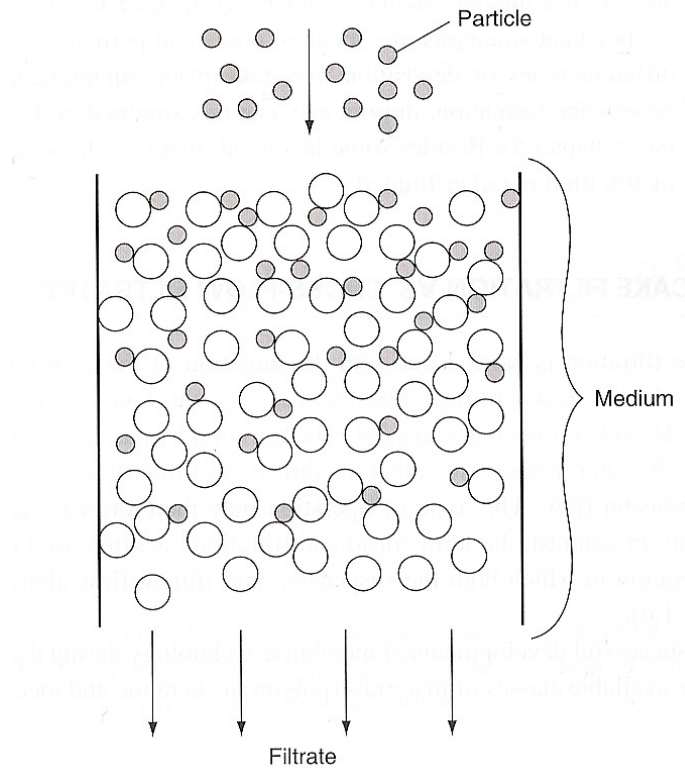
Figure 2-3: Deep bed filtration (*Source: Tien, 2006, page 5, Figure 1.5)*

2.4.1.3. Cross-flow filtration

Cake filtration involves the direction of the suspension flow to be the same as that of the filtrate flow, with the cake growth occurring in the opposite direction. However, with cross-flow filtration, the direction of flow of the suspension is normal to that of filtrate flow.

The tangential flow of the slurry to the filter medium prevents the formation of a cake, with a very small flow of fluid passing through the filter medium. The collection of the solid particles on the filter surface also results in a reduced filtrate flow across the filter.

Cake filtration is thus referred to as "dead end filtration" when compared to cross flow filtration, due to the flow of the suspension and filtrate being in the same direction. Both these processes require different operating conditions in order to be executed. Cake filtration requires high pressures to force the suspension to pass through the cake which continues to grow in height. Whereas with cross-flow filtration, the cake heights that are formed are small, and the pressure required is thus not very high.
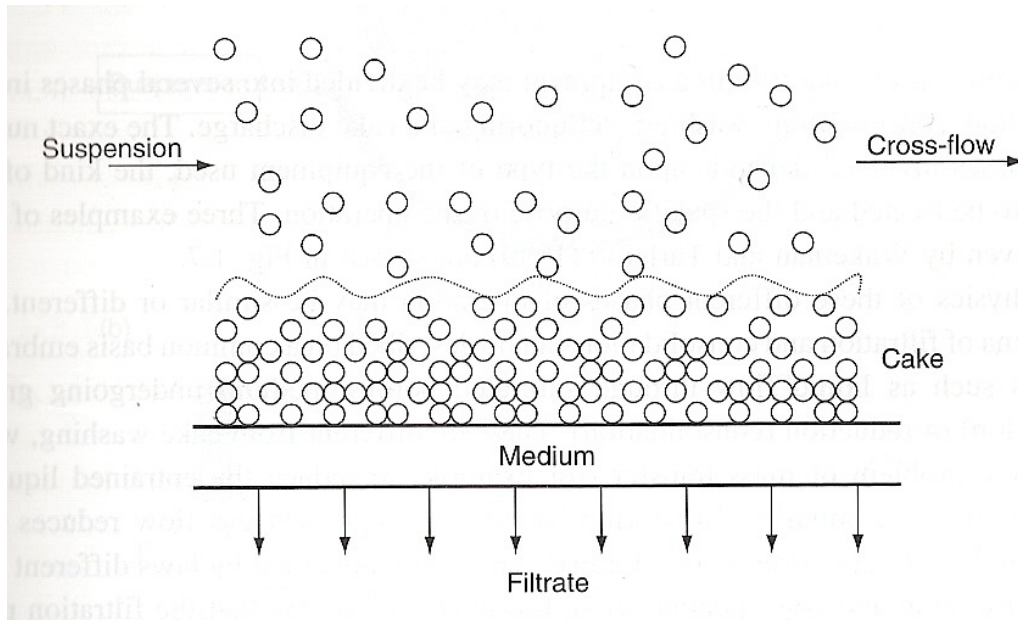
Figure 2-4: Cross-flow filtration (*Source: Tien, 2006, page 7, Figure 1.6)*

2.5. The filtration cycle

The operation of a filtration device can be divided into several operating phases which include: filtration, consolidation, washing, deliquoring and cake discharge. The number of phases for any given separation process is dependent on the type of filtration device used along with the product specifications needed at the end of the separation process.

2.5.1. Cake formation (Filtration)

The first step of pressure filtration is the formation of the cake through the forcing of a suspension through a filter medium. Burger, et al., (2001) developed a physical model that details the filtration process and the formation of the cake. The authors defined a parameter, Φ, which represented the volumetric solids concentration, which is dependent on the height, z, and time, t. The authors found that cake formation results in two processes occurring simultaneously, sedimentation of the suspension

and, flow through the porous cake and filter medium. Their ideas are best described by the following diagram:
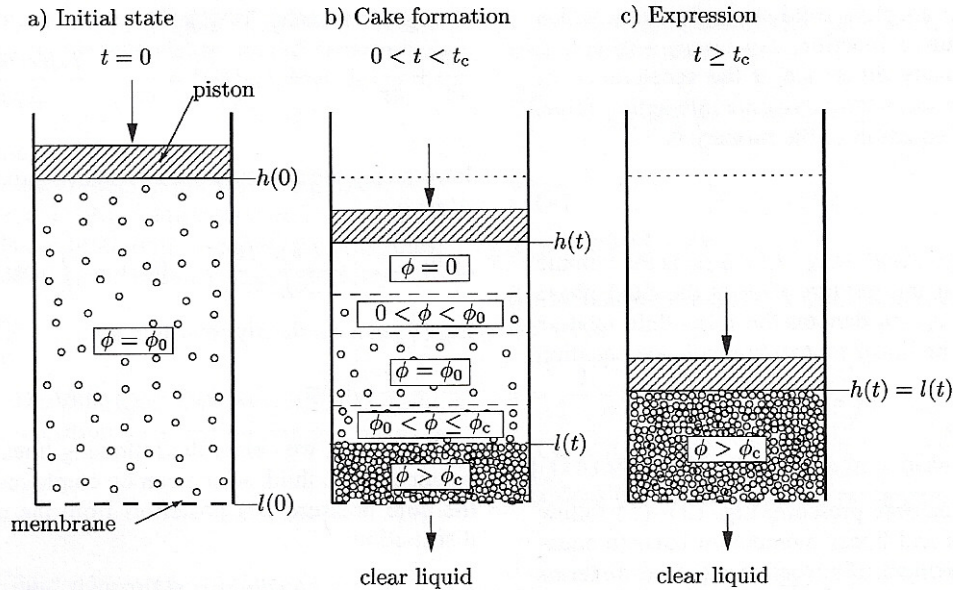
Figure 2-5: Stages in pressure filtration (*Source: Bürger, et al., 2001, page 4539, Figure 1*)

Prior to the start of filtration, the solids concentration throughout the slurry is homogenous, thus $\Phi(z, 0) = \Phi_o$ for $0 \leq z \leq h_o$.

Once the filtration begins, the authors found that there are five different zones that may be distinguished in the pressure vessel.

- At the top, a supernatant clear liquid zone may appear, $\Phi = 0$
- Below that, a transition zone where the concentration varies between zero and Φ_o
- A zone of initial concentration of the slurry, $\Phi = \Phi_o$
- A transition zone $\Phi_o < \Phi \leq \Phi_c$;where Φ_c = the critical concentration at which solid forces begin to touch each other
- A zone of consolidating porous cake with $\phi \geq \phi_c$

2.5.2. Consolidation

Consolidation occurs after the suspension of solid-liquid particles is depleted and the filter cake has completely formed. It is known to be the stage in which the bulk volume of the deposited cake is reduced. This is done through compression of the filter cake by a mechanical load.

2.5.3. Cake washing

Cake washing is categorized as a post-treatment stage and is the process of using a wash liquor to remove any solute present in the voids of the cake. Cake washing is usually undertaken to remove any impurities from the cake and replace it with a more pure liquid, or to recover the liquid product that was present in the voids of the cake.

Cake washing in batch systems continues until the desired level of purity is achieved in the cake or until it is deemed fit to stop, whereas with continuous systems, cake washing is restricted to occur within a specific time, in order to allow for all the other stages of the filtration cycle to also be completed.

Cake washing can occur by:
- passing the wash liquor through the pores of the cake once or,
- by using the wash-liquor collected downstream of the cake to be used again upstream of the cake or,
- by discharging the filter cake from the filter medium, mixing the cake with the wash liquor and creating a new solid-liquid mixture and filtering again.

2.5.4. Deliqouring

Deliquoring is also categorized as a post-treatment stage. The process of the deliquoring was described in great detail by Wakeman (1979, 1982). He described the kinetics of deliquoring and developed correlations and models to describe the process. Although his work was beyond the scope of this

research project, his research did provide the groundwork for understanding the process of cake deliquoring.

Deliquoring describes the desaturation of the filter cake by blowing a gas (e.g. air) through the cake or, by mechanical squeezing, also known as compression dewatering. As the deliquoring proceeds, the moisture content of the cake is reduced until filtrate flow ceases and only air flows through the cake voids, i.e. irreducible saturation.

The potential side effects to deliquoring are cake cracking or cake shrinkage. This is due to the incompressible liquid in the cake pores being forced out and replaced with a compressible gas, drying out the cake completely. Once a crack has formed in the cake, a channel is created. This channel provides a preferred path for flow of the gas. If the crack is formed prior to irreducible saturation being reached, then further deliquoring would be ineffective as the gas would not flow through the pores of the cake that contain liquid, but rather through the channel. The channel also makes further cake washing after the deliqouring problematic as the wash fluid would flow through the channel rather than through the pores to wash out the liquid present.

2.5.5. Cake discharge

This refers to the physical removal of the filter cake from the filter medium. This is usually done by mechanical/physical means.

2.6. Filtration system design

Within the types of filtration techniques available, lie numerous types of filtration devices which make the selection of the right filtration device for the right system crucial. The details on the range of devices and methods of operation are detailed below:

2.6.1. Constant pressure vs. constant flow rate filtration

Filtration can either be executed at levels of constant pressure applied to the system, constant flow rate of suspension being supplied or a combination of both, when one occurs first and is then followed by the other.

If the solid-liquid suspension is subjected to a source of compression at a constant pressure across the filter during the course of the filtration process, the filtration is said to occur at a constant pressure differential. This results in a decrease in the rate of filtration with cake growth, as the cake provides further resistance to flow.

If the solid-liquid suspension is fed to the filter using a positive displacement pump, the filtration is said to occur under constant flow rate conditions. This case occurs when a liquid feed is delivered at a constant flow rate and results in an increase in the pressure drop over the filter with time.

The use of centrifugal pumps to deliver the suspension results in a decrease in the feed rate as the cake resistance increases, implying conditions of variable pressure and variable rate.

Filtrations occurring by means of positive displacement or centrifugal pumps are most common for industrial use (Tarleton, 1999).

2.6.2. Batch vs. Continuous processes

Tarleton and Wakeman (2008, page 19) describe batch processes to be "a basic specification of duty". The decision regarding whether to operate a system as a batch or continuously operating device is dependent on a number of factors (see section 2.6.3.). Batch filtrations are generally preferred to continuous filtration systems. This is because the continuously operating systems are sometimes not feasible, as a result of the resistance of the cake to suspension flow becoming too high and thus, they become uneconomical to force any further separation.

Filtration processes are essentially considered to be discontinuous because they require that the process eventually be stopped to clean or replace the filter and discharge the filter cake (Porter, et al., 1971).

2.6.3. Types of filtration equipment

Filter selection can be a complex procedure due to the numerous equipment types available. In order to select the best type of device for a given system, the unit operations that supply the suspension for separation must be considered along with the latter processing of the solid or liquid products, as these factors would in turn affect the separation device. Porter, et al., (1971) compiled a list of the essential factors that must be considered when selecting a filtration device:

- Slurry characteristics and the cake formed
- Solids concentration of the feed
- Production level (required throughput)
- Physical properties of the liquid and its nature (density, viscosity, flammability, toxicity, corrosiveness)
- Process conditions
- Required results (if the desired product is the solid or liquid or both)
- Cost of operation

Usually the cake forming characteristics of the suspension are important, as this dictates if the process can be operated as continuous or batch. If high product rates are required, then continuous operation is favoured due to low labour costs, whereas with low production rates, continuous operation is not deemed feasible.

The various types of filtration devices are listed below, with a brief description about their structure and operation obtained by referencing Sinnott (2005, pages 409-414) and Geankoplis (2003, pages 903-918).

Nutsche filter (gravity and vacuum operation)
The Nutsche filter is the simplest form of filtration device available. It usually operates in batch mode and comprises of a sealed tank in which the suspension is contained, and a perforated base through which the filtrate passes and the cake is formed. Nutsches are capable of allowing dewatering, washing and drying to occur. These filters have a wide application due to their ease of operation. They can handle highly concentrated slurries with thick cakes, as well as rapidly-settling slurries and suspensions with volatile solvents. Nutsche filters are known for their low equipment costs.
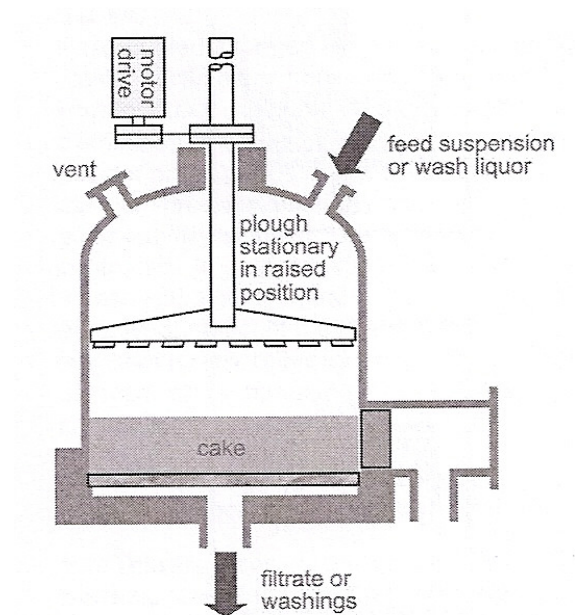
Figure 2-6: Nutsche filtration device (*Source: Tarleton & Wakeman, 2008, page 239)*

Plate and frame filter press (pressure operation)

This filter press comprises of alternating plates and frames, with the filter medium attached on the sides of the plates. The suspension is pumped into the filter device and enters the frames in the unit. The filtrate then flows between the filter cloth and the face of the plate, and exits through channels cut in the plates for filtrate drainage. The filtration continues until the frames are completely filled with cake. Usually each frame has a separate filtrate collection point, which allows for the clarity of the filtrate to be checked. A clear filtrate indicates smooth operation of the filter press, whereas cloudy filtrate indicates a problem, such as a tear in the filter medium. This press supports the use of wash liquor which is usually collected in a separate frame outlet. Although an effective device, there are disadvantages with its operation, which lie in the labour costs to disassemble the apparatus, collect the cake and then reassemble the apparatus. These filters are capable of handling viscous liquids and cakes with a high specific cake resistance but cannot be used for high throughput processes.
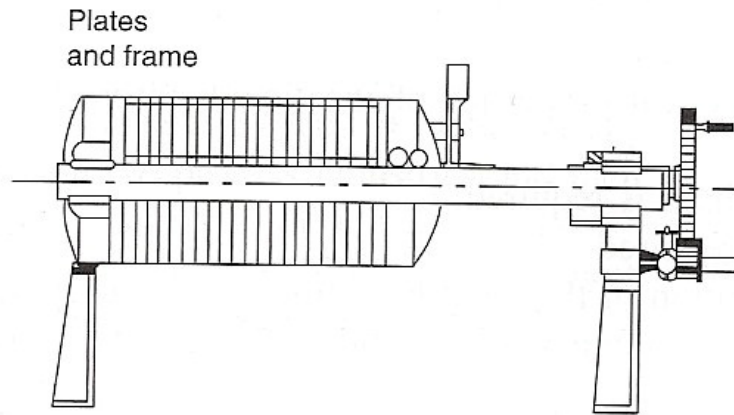
Figure 2-7: Plate and frame filter press (*Source: Sinnott, 2005, page 412, Figure 10.12)*

Leaf filters (pressure and vacuum operation)

This filtration device has a low operating cost and was developed to handle larger volumes of slurry. The device also allows for efficient washing of the cake with a small quantity of waste water. The filter press comprises of a number of leaves hung parallel to one another within a closed tank. The leaves consist of metal frames over which filter cloths are draped. The suspension enters tank and is forced through the filter cloth, through applied pressure. The cake is deposited on the outside of the leaf and is either removed mechanically or by sluicing it off using high pressured water streams. The filtrate flows out through the leaf and exits into a header. Although efficient, they do require batch operation and therefore cannot handle producing large outputs.
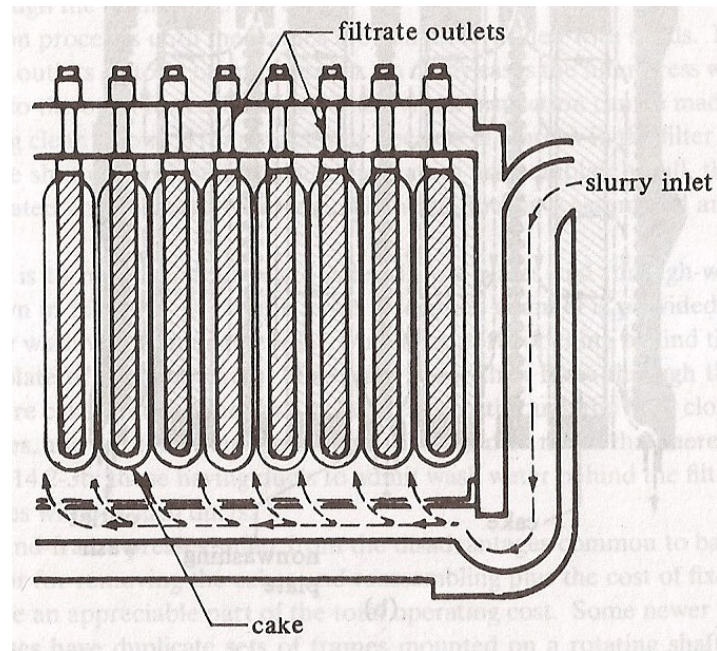
Figure 2-8: Leaf filtration device (*Source: Geankoplis, 2003, page 908, Figure 14.2-4)*

Rotary drum filter (vacuum operation)

The rotary drum filter consists of large hollow drum around which the filter medium is fitted. The drum is partially submerged in a pool of suspension. The cake is collected on the outside of the drum by creating a vacuum within the drum, drawing the filtrate to collect within the drum. For systems requiring cake washing, the drum usually has multiple compartments so that a new compartment can be used to collect the wash liquor. The filter cake is removed via mechanical removal with knives or removal with high pressure streams of water. The operation of the rotary drum filter allows it to handle large volumes of suspension and may be operated on a continuous basis.
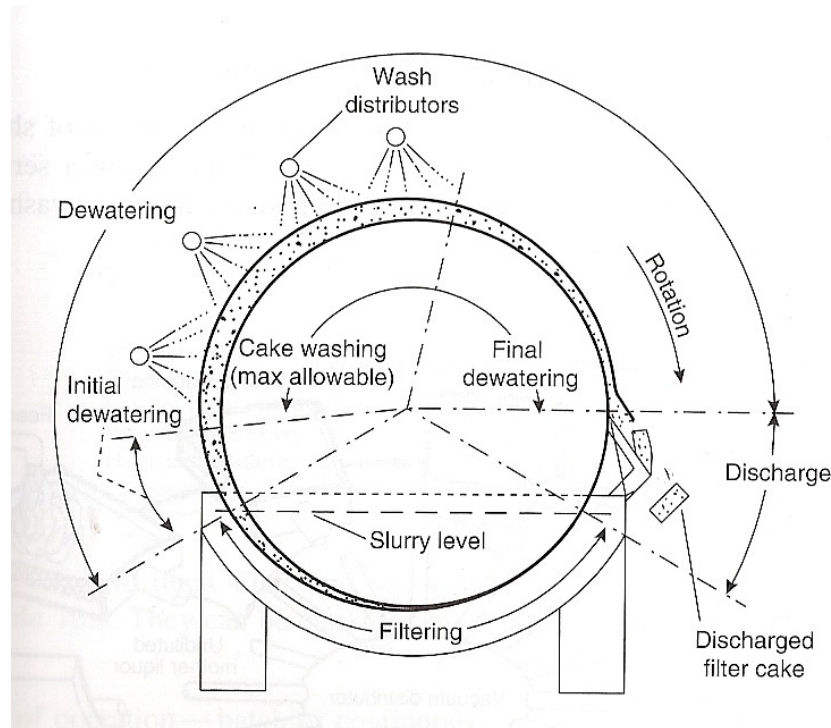
Figure 2-9: Rotary drum filter (*Source: Sinnott, 2005, page 413, Figure 10.13*)

Disc filter (pressure and vacuum operation)

These filters are similar to rotary drum filters with the only difference being the replacement of the drum with multiple thin discs mounted on the shaft. The discs then rotate through a tank submerged with slurry. The slurry is suctioned through the discs via a vacuum created, the cake collects on the outside of the discs and the filtrate flows into compartments within the disc. The filtration occurs in continuous mode and the device is capable of allowing for dewatering and washing to occur. This setup provides a relatively large filter area when compared to the floor-space occupied by the drum filter and is thus preferred when large volumes of product is required. The disadvantage in using this type of filter is the inability to use pre-coats or wash liquor.
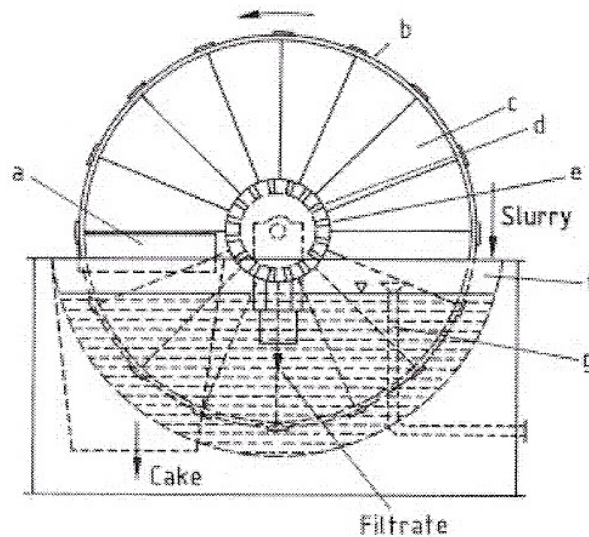
Where a = scraper, b = filter disc, c = trapezoidal sectors, d = outlet points, e = automatic valve, f = filter tank and, g = overflow.

Figure 2-10: Disc filter (*Source: Gösele & Alt, 2005, page 42, Figure 50)*

Belt filter (vacuum operation)

A belt filter comprises of a belt on which the filter medium rests. The suspension is sucked through the top of the filter and the filtrate exits through a drainage channel that runs along the centre of the belt. This filter allows for the use of wash water.
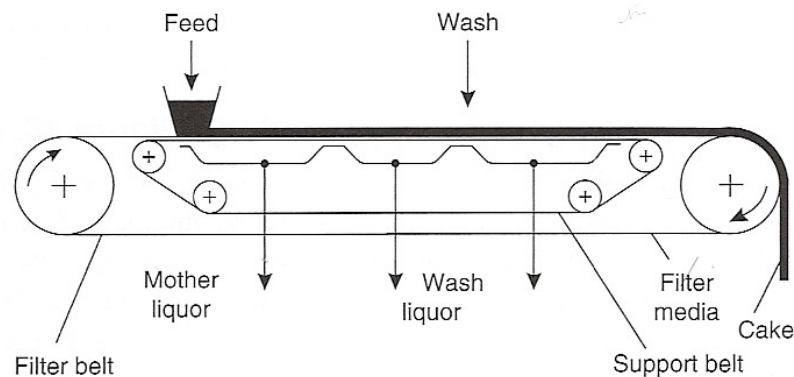
Figure 2-11: Belt filter

Horizontal pan filter (vacuum operation)

This filtration device consists of several pans mounted in a ring. The pans each have a perforated base on which the filter medium rests. The ring rotates allowing the different stages of the filtration process to occur simultaneously.
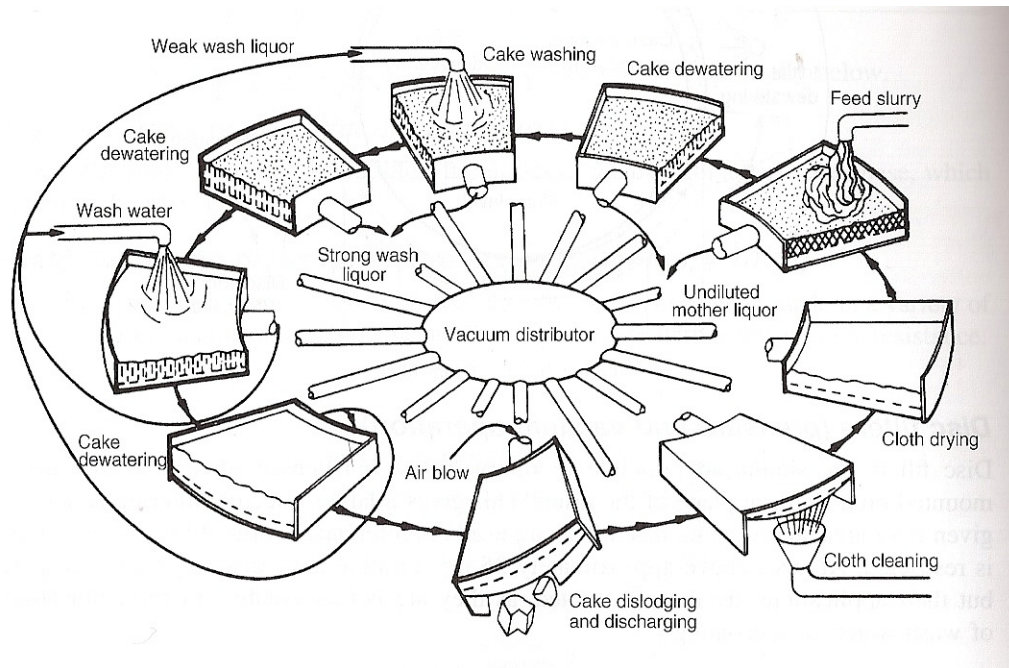
Figure 2-12: Horizontal pan filter (*Source: Sinnott, 2005, page 414, Figure 10.15)*

2.6.4. Filter media

The filter medium is described as the permeable material through which the solid-liquid suspension flows, and allows for the liquid to pass through the pores whilst retaining the solid particles. The filter medium not only has to be able to filter a given suspension, but also has to be able to pair with the filtration device requirements. Specific filtration processes require certain types of filter media in order to allow for optimum performance.

Filter media can also be classified into different types, depending on how the solid particles are separated from the liquid. These include surface types or depth media types. In surface types the solid

particles from the suspension are mostly collected on the surface of the medium with little penetration into the pores. Whereas with depth media types, the particles penetrate into the pores where they are then retained.

Filter media can be made of:
- Textiles: woven cloth or; unwoven fabric from natural materials such as cotton, silk or wool or; from unwoven synthetic fibres
- Wire cloths or meshes which comprise of ferrous or non-ferrous metals woven in complex or plain weaves
- Filter papers made from fibrous cellulosic materials, glass fibres or synthetic polymers
- Felt materials

Geankoplis (2003, pages 909-910) states that the optimum filter material should provide resistance to stretch or structural deformation and flex fatigue and should not be affected by mechanical and abrasive force. The medium also has to be able to allow for removal of the filter cake to be done with ease and should be chemically resistant to the fluids that are used.

Filter medium selection is also dependant on whether the desired product is the liquid or solid as this would dictate the degree of separation required. Mais (1971) discussed the various types of filter media available and the factors which influence the selection of a filter. Some of his findings are listed below:

The vital factors that influence the selection of a filter medium for a given separation process are:
- Size, range and shape of the solid particles
- Specific gravity of the solids
- Temperature, chemistry, viscosity and specific gravity of the suspension
- The solids concentration of the suspension
- Particle flocculation of the slurry
- The characteristics of the cake: rate of cake build-up, compressibility and resistance to fluid flow
- The permeability of the medium (when clean and used)
- The particle retention capacity of the medium
- The material from which the medium is made (polymer, metal etc.)
- The details of construction of the medium (woven etc.)
- The operating conditions: cleaning, washing and operational stability

2.6.5. Filter Aids

The addition of filter aids can also influence the performance of the filtration device. These filter aids are chosen to improve filtration performance by improving clarity or by preventing filter medium blinding. They are inert powders and can be added to the system as a pre-coat or body mix.

When added as a pre-coat on the medium, the filter aid acts as a surface and depth medium, this is done to trap fine particles which would otherwise pass through the pores of the medium. When added as a body mix in the suspension, the filter aid is used to increase the porosity of the cake and increase the flow rate. This is done by the filter aid being deposited with the solid particles on the filter medium, a porous structure is then formed which supports the solid particles but still allows the liquid from the suspension to pass through.

The pre-coat is selected to improve filtrate clarity and protect the filter medium and is thus chosen to be comprised of fine solid particles whereas the body mix is used to increase the filtration rate and is thus chosen to be a material of coarser particle size.

The filter aids available range from various grades (particle size) of material as well as different materials with specific chemical resistance and purity (Gösele & Alt, 2005). Some commonly used filter aids include diatomaceous earth, cellulose fibre, perlite, activated carbon etc. The removal of the filter aid from the filter cake can be costly, but this cost is usually outweighed by the benefits that the filter aid provides.

2.7. Factors affecting filtration

Wakeman and Tarleton (1999a, pages 8-13) discussed that three parameter types must be identified to fully describe a solid-liquid system. The authors referred to these as the primary properties, the state of the system and the macroscopic properties.

2.7.1. Primary properties

The primary properties are those which can be measured and are independent of the other components of the system. These are the solid and liquid physical properties, viz. the size and shape of particles, size distribution and the surface properties of the particles in the solution environment.

Particle sizes range from fine to coarse granular solids. The interaction of a particle with its surroundings dictates whether a particle will settle quickly or slowly. Wakeman and Tarleton (1999a, pages 9-11) mention that for small particles, the net repulsive or attractive forces greatly affect the particles behaviour. The authors also discuss that greater rates of filtration may be expected with larger particles in the feed. This is because the cake formed is more permeable, with a larger pore volume, allowing for more of the liquid to pass through the cake. The particle shape dictates the cake structure and thus, the porosity of the cake and filtration rate.

The interactions of the particles within the liquid in which it is suspended, as well as the particle surface charge (zeta potential) have great influence on the separation. At high or low values of zeta potential there is a net repulsion between the particles which are well dispersed in the suspension, but at zero zeta potential (the iso-electric point) the particles tend to aggregate.

2.7.2. State of the system

The state of the system is described by properties such as the porosity, concentration, the homogeneity and extent of dispersion of the particles, etc.

The porosity of the filter cake directly alters the filtration rate. Cakes having a higher porosity have greater pore volumes available to allow for more filtrate to flow through the cake, thus increasing the rate of filtration. Cakes with a smaller porosity have a more limited pore volume and thus reduce the ability of the filtrate to flow as rapidly as the higher porosity cakes.

The concentration of solids in the feed suspension dictates the thickness of filter cake that will be obtained. A higher solids concentration results in a cake of greater height. Cakes of greater height offer an increased resistance to filtrate flow than cakes of smaller heights, thus reducing the filtrate flow rate.

2.7.3. Macroscopic properties

The primary properties combine with the description of the state of the system to control the macroscopic properties. These are the properties that are measured, such as, permeability, specific resistance of the filter bed or cake, the bulk settling rate of particles in a suspension etc. These properties are used to describe the filtration performance of a given system. By determining these properties, the efficiency of the filtration can be evaluated and adjustments to the primary properties or state of the system can be made. The performance of the filtration device can also be assessed by determining the filtration constants (see section 2.8), measuring the solids recovery of the filter cloth, determining the solute concentration within the filter cake or by monitoring any other property that may be of importance.

2.8. Filtration theory

Several theories on filtration are available, but the theory that was used for the data analysis would be that which combines Poiseuille's law, the Carmen-Kozeny and the Blake-Kozeny equations (Geankoplis, 2003). Research by Palica (1996) provided the details regarding the processing of the data that would be obtained during the experiments in order to obtain the filtration constants. He did this by utilizing the basic filtration equations listed below.

The flow of filtrate through a packed bed of cake can be described using Poiseuille's equation for laminar flow in a straight tube is described by the following equation (Geankoplis, 2003):

$$\frac{-\Delta P}{L} = \frac{32\mu v}{D^2}$$

(2-1)

Where ΔP = pressure drop (Pa)

L = length (m)

μ = viscosity (Pa.s)

v = open tube velocity (m.s^{-1})

D = diameter (m)

The Carman-Kozeny relation for laminar flow in a packed bed of particles (Equation 2-2) and the Blake-Kozeny equation (Equation 2-3) for laminar flow have also been shown to apply to filtration (Geankoplis, 2003):

$$\frac{\Delta P}{L} = \frac{150 v' \mu}{\phi^2 D_p^2} \frac{(1-\varepsilon)^2}{\varepsilon^3}$$

(2-2)

Where L = total height of the bed (m)

v' = superficial velocity (m.s^{-1})

Φ = sphericity of particles in a packed bed

D_p = diameter of the spherical particle (m)

ε = porosity of the bed

Blake-Kozeny equation for laminar flow (Geankoplis, 2003):

$$\Delta P = \frac{150 v' \mu \Delta L}{D_p^2} \frac{(1-\varepsilon)^2}{\varepsilon^3}$$

(2-3)

The relation used to describe filtration processes was found by researchers through the combination of equations (2-2) and (2-3) as described by Geankoplis (2003, page 910):

$$\frac{-\Delta P_c}{L} = \frac{k_1 \mu v (1-\varepsilon)^2 S_0^2}{\varepsilon^3} \qquad\qquad (2\text{-}4)$$

Where ΔP_c = the pressure drop across the filter cake (Pa)

L = thickness of cake (m)

k_1 = constant = 4.17 for random particles of definite size and shape

μ = viscosity of the filtrate (Pa.s)

v = linear velocity based on filter area (m.s^{-1})

S_o = specific surface area of particle (m^2 of particle area per m^3 volume of solid particle)

In experimental filtration studies the cumulative volume of filtrate collected (V) is recorded. This is then converted to a volumetric flow rate (dV/dt). The linear velocity (v) is based on an empty cross sectional area and is defined by Geankoplis (2003, page 911) as:

$$v = \frac{dV/dt}{A} \qquad\qquad (2\text{-}5)$$

Where V = volume of filtrate collected up to time t (m^3)

A = filter area (m^2)

To describe the continuous process, a model is used that provides the relationship between the mass of solids in a suspension and the mass of the cake (Geankoplis, 2003). This is done via a material balance on the solids and is found to be:

$$LA(1-\varepsilon)\rho_{solid} = c(V + \varepsilon LA) \qquad\qquad (2\text{-}6)$$

Where L = cake thickness (m)

ρ_{solid} = density of the solid particles in the cake (kg.m^{-3})

c = mass of dry cake solids per unit volume of filtrate (kg solids/m^3 filtrate)

In equation (2-6), the mass of dry cake solids per unit volume of filtrate (c) is found from the following equation, described by Wakeman and Tarleton (1999a, page 59):

$$c = \frac{s\rho}{1 - ms} \tag{2-7}$$

Where s = mass fraction of solids in the feed slurry (kg/kg)

ρ = density of the filtrate (kg.m^3)

m = ratio of wet to dry cake masses (kg/kg)

Wakeman and Tarleton (1999a, page 69) show that the cake formation rate can be found by rearranging equation (2-6) and using the following relation to replace the voidage (ε) with the ratio of the masses of wet to dry cake (m).

$$m = 1 + \frac{\rho\varepsilon}{\rho_{solid}(1 - \varepsilon)} \tag{2-8}$$

Thus the cake height (L) can be found from the following equation (Wakeman & Tarleton, 1999a):

$$L = \frac{V}{A} \frac{s[\rho_{solid}(m - 1) + \rho]}{\rho_{solid}(1 - ms)} \tag{2-9}$$

The cake height (L) can thus be calculated at each measured value of filtrate volume collected (V), and the cake formation rate with time can be determined.

Substituting equation (2-5) in (2-4) and using equation (2-6) to eliminate the cake thickness (L), the following equation is obtained (Geankoplis, 2003):

$$\frac{dV}{Adt} = \frac{-\Delta P_c}{\alpha_{av} \dfrac{\mu c v}{A}} \tag{2-10}$$

Where $\alpha_{av} = \dfrac{k_1(1 - \varepsilon)S_o^2}{\rho_s \varepsilon^3}$ (2-11)

and α_{av} is defined as the specific cake resistance (m.kg^{-1})

By analogy, Geankoplis (2003, page 911) described the filter medium resistance to filtrate flow, R_m (m^{-1}) as:

$$\frac{dV}{Adt} = \frac{-\Delta P_m}{\mu R_m} \tag{2-12}$$

Where $-\Delta P_m$ = pressure drop across the filter medium (Pa)

Since the resistance of the cake and filter medium are in series, equations (2-10) and (2-12) are combined (Geankoplis, 2003):

$$\frac{dV}{Adt} = \frac{-\Delta P}{\mu\left(\dfrac{\alpha cv}{A} + R_m\right)} \tag{2-13}$$

Where $\Delta P = \Delta P_c + \Delta P_m$ (2-14)

and ΔP is defined as the total pressure drop (Pa)

By rearranging, a relationship between the filtrate flow rate, the applied pressure and the filter cake properties is produced (Wakeman & Tarleton, 1999a):

$$\frac{dt}{dV} = \frac{\alpha_{av} \mu c}{A^2(-\Delta P)}V + \frac{\mu R_m}{A(-\Delta P)} \tag{2-15}$$

Equation (2-15) applies to the initial start-up period of operation and can be manipulated by integrating over the entire constant pressure period to derive the following equation which describes filtration behaviour in a Nutsche filter press under constant pressure filtration conditions (Wakeman & Tarleton, 1999b):

$$t - t_s = \frac{\alpha_{av} \mu c}{A^2(-\Delta P)} \frac{1}{2}\left(V^2 - V_s^2\right) + \frac{\mu R_m}{A(-\Delta P)}\left(V - V_s\right) \tag{2-16}$$

Rearranging Equation (2-16) provides the characteristic filtration equation for constant pressure filtration:

$$\left(\frac{t-t_s}{V-V_s}\right) = \frac{\alpha_{av}\mu c}{2A^2(-\Delta P)}(V+V_s) + \frac{\mu R_m}{A(-\Delta P)}$$

(2-17)

Where t = total time of operation (s)

t_s = start up time (s)

V = total filtrate volume collected since start up (m^3)

V_s = start up volume (m^3)

α_{av}= specific resistance of the cake ($m.kg^{-1}$)

μ = viscosity of the filtrate (Pa.s if the pressure used in measured in units of Pascal's, if the

pressure is measured in bar, the units of viscosity are in bar.s)

c = mass of dry cake solids per unit volume of filtrate ($kg.m^{-3}$)

ΔP = pressure drop across the filter (Pa or bar)

R_m = filter medium resistance (m^{-1})

It is most common to describe constant pressure filtration processes using this equation of the reciprocal volumetric flow rate of filtrate. A start time and volume (t_s and V_s) is included in this equation to account for the finite time required (t_s) for the operating pressure to reach the desired value (Tien, 2006). These values are also included to eliminate the data points which may be recorded prior to the start of the actual filtration process.

For compressible filter cakes, in which the cake resistance grows with increasing pressure (Holdich, 2003):

$$\alpha_{av} = \alpha_0(1-n)(-\Delta P_c)^n$$

(2-18)

Where α_0 = specific cake resistance at unit compressive pressure (a constant) ($m.kg^{-1}$)

ΔP_c = pressure drop across the filter cake (Pa)

n = cake compressibility index. This value is zero for incompressible cakes.

For practical applications, compressible and incompressible cakes are often treated with the same equations, ignoring the use of equation (2-18), provided that the α_{av} is defined as the average specific cake resistance under the conditions of operation (Gösele & Alt, 2005).

Rewriting equation (2-17) in terms of (-ΔP):

$$(-\Delta P) = \frac{\alpha_{av}\mu c}{A^2} \frac{1(V^2 - V_s^{\,2})}{2(t-t_s)} + \frac{\mu R_m (V - V_s)}{A(t-t_s)}$$

(2-19)

In order to calculate the pressure drop across the medium (Geankoplis, 2003):

$$(-\Delta P_m) = \frac{\mu R_m (V - V_s)}{A(t-t_s)}$$

(2-20)

where ΔP_m is the pressure drop across the filter medium (Pa)

The pressure drop across the filter cake (ΔP_c) can thus be found by using the value obtained for the pressure drop across the medium from equation (2-20), and substituting this in equation (2-14) (Geankoplis, 2003).

Teoh, et al., (2006) in their work provided details regarding the various methods that may be used to calculate the specific filter cake resistance. These included:

- Using a linear plot t/V vs. V for constant pressure data (using the equations listed above)
- Using cake filtration data obtained with stepwise increase in the operating pressure
- Using data from the measurements of cake internal properties
- And a proposed method which establishes a relationship between the specific cake resistance and the cake compressive stress.

Teoh, et al., (2006) found that the last method is seemingly tedious and requires a much longer processing time than the first method which is the most well-known method. Teoh, et al., (2006) also found that both methods produce the same results and have the same efficiency. The method of execution to calculate the filtration constants will thus be via the first method, as it is the most well understood and well established.

Thus in order to compute the properties of the cake and filter medium for this experiment, the methodology applied by Palica (1996) would be used. This would require plotting a graph of $\left(\frac{t-t_s}{V-V_s}\right) vs. (V + V_s)$ - as indicated from Equation (2-17). This generally leads to the formation of a straight line graph, whose gradient and intercept can be used to evaluate the specific resistance of the cake (α_{av}) and the filter medium resistance (R_m) from Equation (2-17).

2.9. Three-phase reactors

The work of Shah (1979) provided the basis for understanding the operation and types of gas-liquid-solid reactors available. The author not only described the operation of the different types of reactors but also listed industrial applications for each type. His findings, in combination with the research of Egenes (1990), were used to discuss the sections that follow.

Three-phase reactors are vessels that are capable of allowing a gas-liquid-solid reaction to occur within the vessel. These vessels are used frequently for oxidation, hydrogenation, chlorination and biotechnological applications. Although this investigation did not delve into the development of a three-phase reactor vessel, their operation and factors which affect their operation are key to understanding the application of three-phase filtration within these units. Thus, the sections that follow discuss the various types of three-phase reactors available. They have numerous operating states and each unique process can be performed to reach its optimum, based on the correct selection of one of the three-phase reactor types listed below.

2.9.1. Types of three-phase reactors

Gas-liquid-solid reactions can be classified according to the role that the phases play in the reaction. These are divided into three types:
1. Reactions where the gas, liquid or solid are either reactants or products. E.g. thermal coal liquefaction.
2. A gas-liquid-solid reaction, with the solid acting as the catalyst. E.g. catalytic hydro-cracking of petroleum fractions.

3. Two of the phases being the reacting phases and the third phase being inert, e.g. Fischer Tropsch process. This type is further broken down into:

   o Inert solid phase: This phase is present to impart momentum and to provide better transfer coefficients and improve contact.

   o Inert liquid phase: This phase is present to act as a heat transfer medium or to act as an agent for redistributing the concentration of the various reacting species at the catalyst surface.

   o Inert gas phase: This phase is present to allow for mixing to occur.

Although there are three main types of reactions that can occur within a three-phase reactor, there are two categories of reactors that can be used. The first category is the fixed-bed reactor type, in which the solid phase is stationary and the second type allows for the solids to be in a suspended or fluidized state.

2.9.1.1. Fixed-bed reactors

In fixed-bed reactors the orientation of the gas and liquid flow dictates the manner in which the fixed bed reactor type is operated. The gas and liquid can flow cocurrently in an upwards or downwards direction; or the gas and liquid can flow counter-currently, with the usual direction of flow being gas flow upwards with liquid flow downwards. The difference in the direction of flow for each scenario dictates a specialized unit for a specific application. Fixed bed reactors are thus divided into three types:

- Trickle bed reactors
- Cocurrent fixed bed reactors
- Segmented fixed bed reactors

Trickle bed reactors are the most frequently applied form of fixed bed reactors and are used widely in the petroleum industry for processes such as hydrocracking, hydrodesulpherisation and hydrodenitrogenation. Westerterp and Wammes (2005) discussed the details of operation and flow regimes within this type of reactor, as well as their design. The authors described the operation of the reactor to be one in which the liquid flows down onto the solid catalyst particles in a thin film, where it then comes into contact with the flowing gas phase. The gas phase can flow cocurrently or counter-currently. The general industrial practice encourages cocurrent flow in a downward direction to reduce the possibility of flooding. Industrial application of this reactor type generally requires the reactor to be

operated under plug-flow conditions for effective catalyst wetting, allowing for a high conversion to be attained.

Cocurrent fixed-bed reactors generally have the gas phase as the dispersed phase and the liquid phase being the continuous phase. These reactors can operate as cocurrent upflow or cocurrent downflow reactors. Shah (1979, page 13) mentions that when compared, the upflow reactor provides better mixing; higher mass transfer coefficients; higher liquid hold-up; better liquid distribution; better heat transfer between liquid and solid; lower concentration of solid particles; less solids plugging and better aging of the catalyst. Along with these come shortcomings of higher pressure drop; poorer conversion; more homogenous reactions and more intraparticle diffusional effects and flooding which is avoided in downflow operation.

Segmented fixed-bed reactors allow for gas and liquid flow to occur cocurrently upward, downward or counter-currently. These reactors allow for more flexibility in the mixing characteristics within the reactor. This, in turn, allows for better variations in homogenous and heterogeneous reaction rates.

2.9.1.2. Gas-liquid-suspended-solid reactors

This reactor type can be further subdivided into five categories (Shah, 1979):

1. Agitated gas-liquid-suspended-solid reactors
2. Non agitated three-phase slurry reactors
3. Non agitated three-phase cocurrent upflow fluidized bed reactors
4. Non agitated three phase counter-current flow reactors (spouted bed reactors)
5. Pulsating three-phase reactors

The first three reactor types are commonly used in practice. They operate under batch conditions and are used when a small quantity of product is required.

Gas-liquid-suspended-solid reactors have high liquid hold-ups, which allow them to achieve a high heat capacity. Hot spots are also infrequent due to high levels of mixing. They are also advantageous in that they provide good temperature control. However, in order to minimize the intraparticle diffusion

effects, they require the use of fine catalyst particles which are difficult to separate from the product. They also have poor conversions due to axial mixing.

The four reactor types in this category used for industrial applications are:
- Three-phase fluidized bed reactors
- Continuously stirred autoclaves
- Loop reactors
- Bubble column reactors

Three-phase fluidized bed reactors are used for waste water treatment and hydroliquifaction of coal, along with other industrial processes. These reactors can operate in several ways (Jena, et al., 2008):
- Cocurrent flow, with the liquid as the continuous phase
- Cocurrent flow, with the gas as the continuous phase
- Counter-current flow of the gas and liquid phases, allowing inverse three-phase fluidisation
- Counter-current flow of the gas and liquid phases, allowing fluidization to occur by a turbulent contact absorber

Their general operation is such that the liquid flow is in an upwards direction with the gas flow being cocurrent or counter-current. The liquid is in the continuous state and the gas is in the dispersed state. The upwards flow of the gas and liquid phases allows for the solid particles to be suspended and experience buoyancy. These reactors allow for improved contact between gas and liquid phases and the solid phase, which usually acts as the catalyst.

Continuously stirred autoclaves have wide use in industrial applications such as crystallization, leaching and hydrogenation. These mechanically agitated reactors require a minimum amount of energy to maintain the suspended solid situation. Along with this comes turbulence, which must be distributed evenly to allow for effective use of the catalyst. Egenes (1990, pages 2-43 and 2-44) discussed their three primary modes of operation:
- Dead end operation: in this mode no components leave the reactor. The rate of consumption of the gas dictates the rate of replenishment with new gas.
- Semi-batch operation: Here the gas flows continuously.

- Continuous operation: the liquid-solid suspension and the gas flow continuously through the reactor.

Dead end operation is preferred on a laboratory scale, where the gas build-up is on a very small level. Semi-batch operation is used for slow reactions, such as hydrogenations, and when producing a product from a batch of liquid reactants. Continuous operation is preferred for fast reactions.

In loop reactors a liquid jet is used to force the liquid circulation and gas distribution. The jet, in turn, allows for circulation within the column (Deckwer, 1992).

The last reactor in the fluidized bed reactor type is the bubble column reactor. The application of three-phase filtration is generally applied in this reactor type. Thus, this reactor will be discussed in greater detail in the sections that follow.

2.9.2. Bubble column reactors

The work of Kantarci, et al., (2004) was invaluable for the understanding and information provided on bubble column reactors. The authors' research detailed the operation, design and important characterisation parameters from an abundant number of published findings.

In these reactors the dispersed phase is the gas, which is introduced into a stationary liquid phase, in which the solid phase is suspended. The turbulence induced by the rising gas bubbles allows for the solid phase to remain suspended during operation. The gas phase enters through the base of the column and is dispersed via a sparger. The gas phase travels upwards in bubble form and begins to entrain the liquid phase that is present. Thereafter the gas bubbles begin to travel in a downwards direction.

Huizenga, et al., (1998, page 1277) discussed that the solid phase commonly acts as the catalyst and may need to be separated from the liquid product through filtration. The authors' mentioned that external filtration is not deemed feasible as the slurry mixture would now be exposed to alternate conditions and the slurry would have to be pumped to a new location, introducing additional costs. The relocation of the slurry may also lead to catalyst deactivation. This unwanted situation can be avoided by conducting internal filtration within the column. However, Huizenga, et al., (1998, page 1277) found

that this method also offers disadvantages. The formation of a cake can lead to less effective utilization of the catalyst, along with hot spot formation, product decomposition and catalyst deactivation.

These reactors are commonly employed in the biochemical, metallurgical, pharmaceutical and petrochemical industries and are generally favoured for kinetically slow reactions. These reactors are commonly used for exothermic reactions due to their excellent heat transfer behaviour. They are capable of operating at high gas throughput levels and high liquid circulation levels. In addition, these columns also allow for the use of fine solid particles, minimizing intraparticle diffusion effects (Shah, 1979). However, these reactors do create back-mixing as a consequence or liquid circulation. The reactors also have a short gas residence time, which is dependant on the bubble rise velocity, although this can be improved upon if a downflow column is used.

Deckwer (1992, page 5) commented on the ability to modify the simple bubble column for specialised operation. He suggested that the addition of perforated plates could transfer the column into one that operates in a multistage cascade form. The plates would intensify mass transfer, prevent back-mixing and prevent the formation of larger bubbles, if the plates have small holes. He also suggested that the same effect could be achieved in a packed bubble column.

Their simplistic operation, lack of moving parts, cost effectiveness, low maintenance and ability to develop a large interfacial area makes these reactors continue to grow in popularity for multiphase reaction systems.

2.9.3. Factors affecting bubble column reactor operation

The factors influencing the operation of bubble column reactors are discussed in detail by Kantarci, et al., (2004) and Shah (1979). They both agree that efficient operation of three-phase reaction vessels depends on the quantification of the following parameters: momentum, mass and heat transport mechanisms and coefficients; along with chemical kinetics of the reacting system and mixing parameters. The effects of these are discussed briefly in the headings that follow.

2.9.3.1. Flow regime

The performance of a bubble column is directly dependant on the fluid dynamic characterization within the reactor. The flow regimes are strongly dependant on the flow rates of the gas and liquid and their relative orientation to each other, be it cocurrent upward or downward or counter-current. The flow regimes are also significantly affected by the distributor type. The flow regimes within a reactor are classified according to the superficial gas velocity in the column. Zhang, et al., (1997) investigated flow regime identification and the velocities at which they occur. These are divided into the:

- Homogenous (Bubbly flow) regime
- Heterogeneous (Churn-turbulent) regime
- Slug flow regime

The homogenous regime occurs under low superficial gas velocities and is usually present in semi-batch columns that have spargers with small holes (Chaumat, et al., 2007). They exhibit narrow bubble size distributions with small bubbles. There is minimal interaction between bubbles, allowing them to travel vertically and allow for a gentle mixing action. There is virtually no bubble coalescence or break-up with the bubble size being completely dictated by the sparger design and system properties.

The superficial gas velocity in the heterogeneous regime is higher than that of the homogenous regime. A large distribution of bubble sizes exists with the large bubbles concentrated along the column axis. The flow patterns are unsteady with high gas throughputs resulting in large amount of coalescence and a vigorous mixing action. This regime is common amongst large diameter columns and is the most popular regime for use in industrial bubble columns.

The slug flow regime occurs seldomly and is noted to generally occur at high gas flow rates in tall, small diameter columns. In this regime the large bubbles present become stabilized by the column wall. They then become elongated and move upwards through the column, continuing to grow as smaller bubbles join them. Slug flow should generally be avoided due to their low reaction capacity and conversion rates.

2.9.3.2. Pressure drop

The gas-liquid and liquid-solid mass transfer coefficients are correlated to the pressure drop and known to be affected by changes in it. Significant changes in pressure drop alter the partial pressure of the gas in the reactor, ultimately affecting conversion.

2.9.3.3. Gas hold-up

The gas hold-up is an important parameter for design of the bubble column reactor, it is affected by the superficial gas velocity, liquid properties, column dimensions, operating temperature and pressure, gas distributor design and solid phase properties.

Katarci, et al., (2004, pages 2269-2273) summarises the effects of all these parameters on the gas hold-up to be:

The gas hold-up increases with increasing gas velocity and operating pressure, and decreases with increasing liquid viscosity and solid concentration. The effects of the column dimensions on gas hold-up were also reported to be negligible for aspect ratios greater than 5. The gas hold-up was also found to be dependant on the orifice pitch and size for low gas velocities.

It should also be mentioned that liquid hold-up also plays an important role in the behaviour of bubble column reactors. The liquid hold-up is responsible for determining the reaction rates of homogenous and catalytic reactions, when they occur simultaneously (Katarci, et al., 2004).

2.9.3.4. Bubble characteristics

Bubble populations within a column dictate the behaviour of the hydrodynamics of the column, along with the heat and mass transfer coefficients. The bubble size distribution is also crucial in determining the reaction efficiency as it dictates the gas-liquid interfacial area available for mass transfer (Chaumat, et al., 2007). The bubble size within the reactor is strongly dependant on the coalescence behaviour of the liquid phase. Katarci, et al., (2004, pages 2273-2275) gathered that the average bubble size in a bubble column is affected by gas velocity, liquid properties, gas distribution, operating pressure and column diameter. The bubble size tends to increase with increasing superficial gas velocity, solid

concentration, liquid viscosity and surface tension. Whereas, the bubble size was seen to decrease for increasing foaming agent concentrations.

2.9.3.5. Transfer coefficients

The importance of mass transfer coefficients on reactor performance is pivotal. Katarci, et al., (2004, pages 2275-2277) noted that the volumetric mass transfer coefficient ($k_l a$) increases with increasing gas velocity, gas density and pressure but decreases with increasing solid concentration and liquid viscosity. For effective mass transfer to occur large bubbles should not be present within the column.

Along with mass transfer coefficient findings, Katarci, et al., (2004, pages 2277-2279) collected and summarized findings for the heat transfer coefficient. The heat transfer coefficient was found to increase with an increase in temperature, superficial gas velocity, and particle size, but decrease with liquid viscosity and particle density. The effect of solid concentration on the heat transfer coefficient is still unknown. In terms of the bubble behaviour, the heat transfer coefficient was found to be higher at the centre of the column, since the bubbles collect at the centre.

2.10. Statistical design of experiments

Based on the literature reviewed above, the filtration process can be influenced by numerous factors. In order to determine the effect of each of these factors, or the extent of their effect on the filtration behaviour, a statistical design of experiments should be embarked on.

An experiment is simply described as an inquiry into the effect of one or more input variables (factors) on the output variable. Although the concept of experimentation is easy enough to understand, the plan of the experimental design is what creates the complexity (Berger & Maurer, 2002).

Due to the numerous factors that would have to be considered when conducting the experiments on the designed test rig, it was essential to gain a greater understanding into the statistical design of experiments. This was done by attending an introductory course at WITS University, titled "The design and analysis of experiments", which provided the basic knowledge required. The knowledge gained at

the short course was further supplemented through use of textbooks compiled by Berger and Maurer (2002), Montgomery and Runger (1999), and Montgomery (1997).

2.10.1. The steps of experimental design

Berger & Maurer (2002, pages 1-8) discussed that any experimental design has to consider the following questions:

- Which factors should be studied?
- How should the levels of these factors vary?
- What combination of the levels should be used?

The authors provided a six step method for the experimental design of the proposed experiment. These steps included:

1. Planning the experiment
2. Designing the experiment
3. Conducting the experiment
4. Analysing the data from the experiment
5. Confirming the results of the experiment
6. Evaluating the conclusions drawn from the experiment

Step 1: Planning the experiment

The planning stage of experimentation is a key factor that contributes to the actual investigation. This ought to be tackled as follows:

- Identifying the dependant (output) variables
- Translating the output variables to measurable quantities
- Determining the factors (input/independent) variables that could affect the required output variables
- Determining the number of levels for each factor and what these values are
- Identifying possible interaction effects between different factors

These ideas were important factors in identifying the plan of execution for experimentation and are further discussed in Chapter 4.

Step 2: Designing the experiment

The actual design of the experiment can be done through various statistical techniques. A factorial design can be conducted, allowing for varying combinations of factors at different levels to occur; or one factor can be varied at time. If the design has a large number of factors at different levels, fractioning could be done, when only a fraction of the possible experiments are conducted. Blocking is another technique which also can be applied.

Once one of these design techniques has been selected, the actual experimentation may be as suggested by the statistical method. This investigation used the method of factorial design which is further discussed in section 2.10.2.

Step 3: Performing the experiment

This step involves the actual execution of the experiment which is dictated by the investigator. However, the method of experimentation is provided by the statistical technique chosen, indicating which factors should be varied at what level, and in what sequence.

Step 4: Analysing the data from the experiment

This involves the deduction as to whether a given factor has an effect on the output variable. This usually accomplished through *hypothesis testing*. This analysis assumes that a factor will not have an effect on the dependant variable unless otherwise proven. The confirmation of this is done through a method known as the *analysis of variance* (*ANOVA*) which is discussed in section 2.10.2.2.

Step 5: Confirming the results of the experiment

This is usually done through logical inspection of the given results. The results obtained can also be further verified by replicating the tests that were done and checking that the proven effects remain the same.

Step 6: Evaluating the conclusions drawn

This step is self explanatory as all investigations require the findings to be discussed and for conclusions to be drawn.

2.10.2. Factorial designs

This method of experimental design is used when several factors of importance are involved in an investigation. This method also serves useful when wanting to determine the combined effect of factors. For this investigation, a three factor, two-level design was used, the reasoning behind this deduction is further explained in Chapter 4.

Factorial design allows for the determination of the effects of k factors, each of which has two levels. These levels may be quantitative values (measureable parameters e.g. flow rate of air) or qualitative (immeasurable parameters e.g. different types of machinery used). The values assigned to these two levels are chosen with one being at the "high" level and other being at the "low" level. A complete design is this case requires two-levels to be used for k factors, and is thus termed a 2^k design. This design method provides the minimum amount of experiments required to analyse the effect of k factors.

2.10.2.1. Factorial design for three factors each at two levels (2^3 design)

The understanding and knowledge required to conduct a statistical experiment design was obtained from Montgomery and Runger (1999). The authors provided details about the selection of a design method and information on how to analyse the effects of the factors from the results that would be computed.

A 2^3 design has 8 treatment combinations. The authors geometrically describe this design as a cube, with the eight runs forming the corners of the cube. The three factors that are investigated (e.g. factors *A*, *B* and *C*) are considered to be the main effects that will be analysed. The interaction effects between these factors are divided into 2 factor interactions (here, *AB*, *AC* and *BC*) and 3 factor interactions (here, *ABC*). This method also allows for the inclusion of replicate runs, with the number of replicates being *n*.
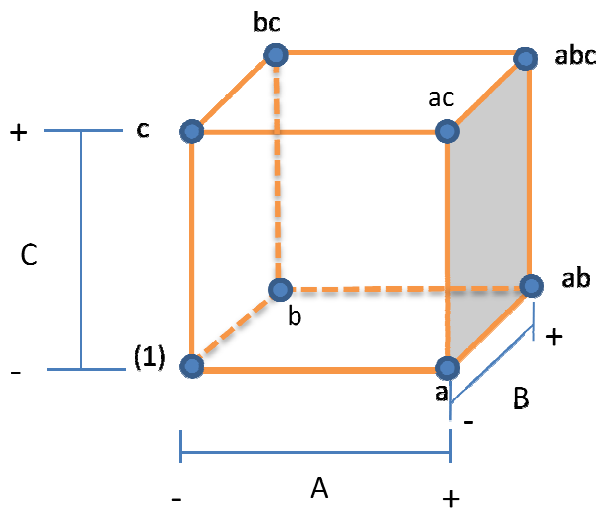
Figure 2-13: Geometric representation of a 2^3 design

In order to label the treatment combinations, Yates order notation is used (Berger & Maurer, 2002). This utilizes lowercase letters to indicate that a factor is at its high level and the absence of the letters indicates that the factor is at its low level. The treatment combinations are thus:

1 = *A*, *B* and *C* are at their low level

a = Factor *A* is at its high level and factors *B* and *C* are at their low levels

b = Factor *B* is at its high level and factors *A* and *C* are at their low levels

c = Factor *C* is at its high level and factors *A* and *B* are at their low levels

The estimation of the effects of each of the factors along with the effects of the interaction terms can be found quite easily. As illustrated by Montgomery and Runger (1999, pages 657-659).

For factor *A*, this is the difference of the average of the runs were *A* is at its high level, and the average of the runs were *A* is at its low level. This is done similarly for factors *B* and *C*.

Thus for the main effects (factors *A/B/C*),

$$A = \frac{1}{4n}[a + ab + ac + abc - (1) - b - c - bc] \tag{2-21}$$

$$B = \frac{1}{4n}[b + ab + bc + abc - (1) - a - c - ac] \tag{2-22}$$

$$C = \frac{1}{4n}[c + ac + bc + abc - (1) - a - b - ab] \tag{2-23}$$

Where *a/b/c* respectively refers to the case when each of these factors are at their high level.

ab/ac/bc refers to the experiments where the two of the three factors (*A/B/C*) are at their high levels at the same time.

abc refers to the experiment when all three factors are at their high level.

(1) refers to the scenario when all the factors are at the low level values.

n refers to the number of replicates conducted for the statistical investigation

For the interaction effects:

For *AB*, this is the difference between the average *A* effects at the two levels of *B*. This is also done similarly for *AC* and *BC*.

$$AB = \frac{1}{4n}[abc - bc + ab - b - ac + c - a + (1)] \tag{2-24}$$

$$AC = \frac{1}{4n}[(1) - a + b - ab - c + ac - bc + abc] \tag{2-25}$$

$$BC = \frac{1}{4n}[(1) + a - b - ab - c - ac + bc + abc] \tag{2-26}$$

For *ABC*, this is the average difference between the *AB* interaction for the two different levels of *C*.

$$ABC = \frac{1}{4n}[abc - bc - ac + c - ab + b + a - (1)] \tag{2-27}$$

These effect terms for the main factors (*A*, *B*, *C*) and the interaction terms (*AB*, *AC*, *BC*, *ABC*) can be used to provide an idea as to which effects and in what combination are significant on the results obtained. This is done by comparing these effect values. The values that differ significantly from the others indicate the significance of the main effect or interaction effect.

In order to indicate in what combination the factors should be varied and in what order the experiments need to be conducted, a table of signs is created as shown by Table 2-1. Here, the low levels of the factors *A*, *B* and *C* are denoted by a negative sign (-) whereas their high levels are denoted by a positive sign (+).The interaction effects for each run are found by multiplying the signs of the factors involved for that specific run. For example for the *AB* effect for the first run (Table 2-1, row 2), this is found by multiplying the level of factor *A* for the first run with the level of factor *B* for the first run.

Table 2-1: Table of signs

Run	Treatment combination	Factorial Effect						
		A	B	C	AB	AC	BC	ABC
1	1	-	-	-	+	+	+	-
2	a	+	-	-	-	-	+	+
3	b	-	+	-	-	+	-	+
4	ab	+	+	-	+	-	-	-
5	c	-	-	+	+	-	-	+
6	ac	+	-	+	-	+	-	-
7	bc	-	+	+	-	-	+	-
8	abc	+	+	+	+	+	+	+

2.10.2.2. The Analysis of variance (*ANOVA*) method

In order to determine the extent of the effect of factors or interaction factors are on the output of an experiment, an *ANOVA* table must be constructed and analysed. The layout of the *ANOVA* is obtained by referring to Montgomery and Runger (1999, page 642) and is shown below along with the equations that need to be solved in order to analyse the table.

Table 2-2: _ANOVA_ table

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p-value
A	SS_A	a-1	MS_A	M_{SA}/MS_E	From F-tables
B	SS_B	b-1	MS_B	M_{SB}/MS_E	From F-tables
C	SS_C	c-1	MS_C	M_{SC}/MS_E	From F-tables
AB	SS_{AB}	(a-1)(b-1)	MS_{AB}	MS_{AB}/MS_E	From F-tables
AC	SS_{AC}	(a-1)(b-1)	MS_{AC}	MS_{AC}/MS_E	From F-tables
BC	SS_{BC}	(b-1)(c-1)	MS_{BC}	MS_{BC}/MS_E	From F-tables
ABC	SS_{ABC}	(a-1)(b-1)(c-1)	MS_{ABC}	MS_{ABC}/MS_E	From F-tables
Error	SS_E	abc(n-1)	MS_E		
Total	SS_T	abcn-1			

The number of levels of factor _A_ is denoted by _a_, for _B_ it is given by _b_ and for _C_ it is given by _c_.

_n_ indicates the number of replicates for each run.

Montgomery and Runger (1999, pages 641-643) use the following model to describe three-factor experiments:

$$Y_{ijkl} = \mu + \tau_i + \beta_j + \gamma_k + (\tau\beta)_{ij} + (\tau\gamma)_{ik} + (\beta\gamma)_{jk} + (\tau\beta\gamma)_{ijk} + \epsilon_{ijkl} \begin{cases} i = 1, 2, \ldots, a \\ j = 1, 2, \ldots, b \\ k = 1, 2, \ldots, c \\ l = 1, 2, \ldots, n \end{cases}$$

(2-28)

The sums of the squares within the *ANOVA* table are computed using the following equations:

For the total sum of squares:

$$SS_T = \sum_{i=1}^{a}\sum_{j=1}^{b}\sum_{k=1}^{c}\sum_{l=1}^{n} y_{ijkl}^2 - \frac{y_{....}^2}{abcn}$$

(2-29)

For the sum of squares for factor *A*:

$$SS_A = \sum_{i=1}^{a} \frac{y_{i...}^2}{bcn} - \frac{y_{....}^2}{abcn}$$

(2-30)

For the sum of squares for factor *B*:

$$SS_B = \sum_{j=1}^{b} \frac{y_{.j..}^2}{acn} - \frac{y_{....}^2}{abcn}$$

(2-31)

For the sum of squares for factor *C*:

$$SS_C = \sum_{k=1}^{c} \frac{y_{..k.}^2}{abn} - \frac{y_{....}^2}{abcn}$$

(2-32)

For the sum of squares for the interaction of *AB*:

$$SS_{AB} = \sum_{i=1}^{a}\sum_{j=1}^{b} \frac{y_{ij..}^2}{cn} - \frac{y_{....}^2}{abcn} - SS_A - SS_B$$

(2-33)

For the sum of squares for the interaction of *AC*:

$$SS_{AC} = \sum_{i=1}^{a}\sum_{k=1}^{c} \frac{y_{i.k.}^2}{bn} - \frac{y_{....}^2}{abcn} - SS_A - SS_C$$

(2-34)

For the sum of squares for the interaction of *BC*:

$$SS_{BC} = \sum_{j=1}^{b} \sum_{k=1}^{c} \frac{y_{.jk.}^2}{an} - \frac{y_{....}^2}{abcn} - SS_B - SS_C \qquad (2\text{-}35)$$

For the sum of squares for the interaction of *ABC*:

$$SS_{ABC} = \sum_{i=1}^{a} \sum_{j=1}^{b} \sum_{k=1}^{c} \frac{y_{ijk.}^2}{n} - \frac{y_{....}^2}{abcn} - SS_A - SS_B - SS_C - SS_{AB} - SS_{AC} - SS_{BC} \qquad (2\text{-}36)$$

For the sum of squares for the error term, *E*:

$$SS_{ABC} = SS_{T} - SS_A - SS_B - SS_C - SS_{AB} - SS_{AC} - SS_{BC} - SS_{ABC} \qquad (2\text{-}37)$$

where $y_{i...}$ = the total number of observations taken at the *i*-th level of factor *A*

$y_{.j..}$ = the total number of observations taken at the *j*-th level of factor *B*

$y_{..k.}$ = the total number of observations taken at the *k*-th level of factor *C*

$y_{ij..}$ = the total number of observations taken at the *ij*-th cell

$y_{i.k.}$ = the total number of observations taken at the *ik*-th cell

$y_{.jk.}$ = the total number of observations taken at the *jk*-th cell

$y_{....}$ = the grand total of all the observations

The mean squares for the factors, the interactions and the error is found using:

$$MS_A = \frac{SS_A}{a-1} \qquad (2\text{-}38)$$

$$MS_B = \frac{SS_B}{b-1} \qquad (2\text{-}39)$$

$$MS_C = \frac{SS_C}{c-1} \qquad (2\text{-}40)$$

$$MS_{AB} = \frac{SS_{AB}}{(a-1)(b-1)} \qquad (2\text{-}41)$$

$$MS_{AC} = \frac{SS_{AC}}{(a-1)(c-1)} \qquad (2\text{-}42)$$

$$MS_{BC} = \frac{SS_{BC}}{(b-1)(c-1)} \qquad (2\text{-}43)$$

$$MS_{ABC} = \frac{SS_{ABC}}{(a-1)(b-1)(c-1)} \qquad (2\text{-}44)$$

$$MS_E = \frac{SS_E}{ab(n-1)}$$
(2-45)

The *F-statistic* for all the factors and their interaction effects are found by dividing the respective mean square by the mean square of the error. This *F-statistic* will be used to determine if the effect of a factor or interaction terms are significant. This is detailed below:

To test the significance we have to test the hypotheses that follow. The null hypothesis (H_o) will be accepted unless proven otherwise, in that case H_1 is accepted

1. For the effect of *A*: $H_o : \tau_1 = \tau_2 = \ldots = \tau_a = 0$ (no main effect of *A*)

     H_1: at least one $\tau_i \neq 0$

     This is tested by using the F-ratio for *A*

2. For the effect of *B*: $H_o : \beta_1 = \beta_2 = \ldots = \beta_b = 0$ (no main effect of *B*)

     H_1: at least one $\beta_j \neq 0$

     This is tested by using the F-ratio for *B*

3. For the effect of *C*: $H_o : \gamma_1 = \gamma_2 = \ldots = \gamma_b = 0$ (no main effect of *C*)

     H_1: at least one $\gamma_k \neq 0$

     This is tested by using the F-ratio for *C*

4. For the effect of *AB*: $H_o : (\tau\beta)_{11} = (\tau\beta)_{12} = \ldots = (\tau\beta)_{ab} = 0$ (no effect of *AB*)

     H_1: at least one $(\tau\beta)_{ij} \neq 0$

     This is tested by using the F-ratio for *AB*

5. For the effect of *AC*: $H_o : (\tau\gamma)_{11} = (\tau\gamma)_{12} = \ldots = (\tau\gamma)_{bc} = 0$ (no effect of *AC*)

     H_1: at least one $(\tau\gamma)_{ik} \neq 0$

     This is tested by using the F-ratio for *AC*

6. For the effect of *BC*: $H_o : (\beta\gamma)_{11} = (\beta\gamma)_{12} = \ldots = (\beta\gamma)_{bc} = 0$ (no effect of *BC*)

     H_1: at least one $(\beta\gamma)_{jk} \neq 0$

     This is tested by using the F-ratio for *BC*

7. For the effect of *ABC*: H_o : $(\tau\beta\gamma)_{111} = (\tau\beta\gamma)_{112} = ... = (\tau\beta\gamma)_{abc} = 0$ (no effect of *ABC*)

H_1: at least one $(\tau\beta\gamma)_{ijk} \neq 0$

This is tested by using the F-ratio for *ABC*

First a level of significance (α) must be chosen, this value is usually chosen to be at a 95% significance level, thus α=0.05. The hypothesis for each case can be rejected, (indicating an effect of the factor tested), if the calculated *F-statistic* is greater than those obtained from data tables at the degree of freedom of the effect, and the degree of freedom of the error.

I.e. for a case with three main factors:

Reject the null hypothesis if $F_{effect} > F_{\alpha, \; degree \; of \; freedom \; of \; the \; effect, \; abc(n-1)}$.

Alternatively the *p-value* approach can be used. The *p-value*s are found from *F-statistic* tables. The *p-value* for each effect can be compared to the level of significance (α = 0.05). If the *p-value* for a given effect is smaller than α = 0.05, then the effect is considered to be influential on the output of the investigation.

2.10.2.3. Modelling via regression

Once the effects that are of significance are identified from the *ANOVA* tables, a regression would be done in order to develop an equation that describes the effect of the significant terms on the dependant variable of the experiment.

For this investigation the system was assumed to behave linearly and in a first order manner. If this assumption was incorrect the fit of the regression model would be poor and the order of the regression equation would have to be reassessed. A multiple linear regression model with interaction terms would then be used to describe the behaviour of this system. The general form of this type of model is given by Montgomery and Runger (1999, page 485) as:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 + \epsilon \quad \text{(2-46)}$$

where y = the dependant variable

$x_1/x_2/x_3$ = the main factors (here, *A*, *B* and *C*)

$x_{12}/x_{13}/x_{23}/x_{123}$ = the interaction effects (here, *AB*, *AC*, *BC* and *ABC*)

$β_0$ = the intercept of the plane

$β_1/ β_2/ β_3/ β_{12}/ β_{13}/ β_{23}/ β_{123}$ = the regression coefficients (for *A*, *B*, *C*, *AB*, *AC*, *BC* and *ABC* respectively)

$Є$ = error term

The regression coefficients are generally determined through the use of the least squares method. However when fitting a regression model after conducting an *ANOVA* analysis,

$β_0 = y....$ = the grand average of all observations

The remaining regression coefficients for each respective effect term are calculated as follows: The regression coefficient for a certain factor is calculated as being one half of the total effect for that respective factor term, i.e. $β_1$ is half of the sum of the effects for factor 1 (i.e. factor *A*) and so on.

Assessing the model

The residuals from a model are defined to be the difference between the observed value of the dependant variable and the estimated value of the dependant variable, determined from the statistical model fitted.

Analysis of the residuals allows the investigator to determine if the selected model adequately describes the system and whether the form of the model should be changed (from first order to second, or from linear to quadratic etc.). This can be done by constructing a normal probability plot of residuals which is a plot of the residuals against the factor levels on a normal probability scale. Data points that lie far outside the distribution of the rest of the data are termed outliers. These data points are the points which do not agree with the behaviour of the rest of the data. Graphs of the residuals against the predicted values from the model are also useful.

The adequacy of a model can also be determined by the coefficient of multiple determination (R^2) which is calculated from:

$$R^2 = \frac{SS_R}{SS_T} = 1 - \frac{SS_E}{SS_T} \qquad (2\text{-}47)$$

where SS_R = regression sum of squares

This coefficient is used to determine the correlation between two sets of data. The coefficient has a range that lies between a value of 0 and 1. With values closer to 1 indicating a better fit of the model to the data.

CHAPTER 3:

EQUIPMENT AND EXPERIMENTAL PROCEDURE

3.1. Introduction

The equipment that would be required for this investigation would have to meet the following requirements:

- The equipment would have to be operated as a filtration column that would allow for two-phase and three-phase filtration experiments to be conducted and compared.
- The introduction of the third phase (i.e. the gas) would have to be similar to the manner in which the gas is distributed in a three-phase reactor unit.

The existing types of equipment at the Chemical engineering laboratory at UKZN did not meet the specific requirements needed for the proposed experimentation. Furthermore, the existing units could not be modified suitably to allow for three-phase gas introduction during filtration. Thus, a new test rig would have to be constructed in order to allow for the above mentioned design criterion to be met.

The literature review conducted provided the information and insight required to develop a three-phase filtration cell. A summary of the decisions regarding the chosen modes of operation, and construction of the test rig are listed below:

- The type of filtration used would be cake filtration so that the cake properties could be analysed in order to determine the effect of the introduction of the third phase.
- Batch operation would be superior to continuous operation in order for constructive data analysis to occur.
- In order to determine the effect of pressure on the system and perform comparisons across all the tests that would be conducted, constant pressure filtration would be performed.

The next step was to determine the type of filtration device that could be adapted to include the introduction of the third phase. Upon analyzing the various filtration devices available as described by Porter, et al., (1971), it was found that the device that provided the ability to introduce the third phase, whilst still meeting the above requirements, was a Nutsche filtration unit. In this unit, the introduction

of the third phase into the system would resemble the flow of the gas phase within a bubble column reactor with internal filtration. The solid particles in the slurry would represent the suspended catalyst in a bubble column reactor. The Nutsche filtration device was also chosen due to its simplistic setup and ease of modification. The Nutsche device was chosen as a preliminary filtration unit whose functionality and suitability would be tested during experimentation. If the Nutsche filter was found to be unsuitable for the experimentation, an alternative filtration device would be used.

The Nutsche filter configuration consists of four major components:
- The vessel
- The filter floor and cloth
- Mechanical arms for cake washing and smoothing
- The cake discharge mechanism

The mechanical arms and cake discharge mechanism is generally applied for industrial use, when working with large volumes of slurry and large filter cakes. These two components will thus be excluded from the design of the test rig.

The initial design of the equipment is described below, however, with continued experimental work and commissioning of the apparatus, modifications were made to optimize the process of filtration. The initial design is discussed, detailing any difficulties that were encountered.

3.2. Equipment Design

Several papers by Tarleton were reviewed. Tarleton (1998a) provided a detailed explanation of the usage of a computer driven experimental apparatus. He focused on the properties of the controller rather than the filtration process/filtration data. He provided details about a system that can be used to acquire variable pressure filtration. Although this was not relevant for this research project, he did provide a schematic representation of the fully automated apparatus that he had used for his investigation. It integrated electronics, computer technology, control principles and mechanical systems. In another paper, Tarleton and Wakeman (1999), the authors' focused on the simulation and software development of filtration processes. Details regarding the structure and dimensions of the equipment were provided along with a range of pressures and temperatures. Holdich (2003) also provided details

about the structure of his equipment. These were all utilized in the equipment design and the operational guidelines of the test rig.

The design of the equipment was based on that obtained from the works of Tarleton (1998a, b) as it met all the requirements regarding the method of operation and structure. The design also implemented the use of computer control and data recording which would be essential for this investigation by allowing the filtration data to be obtained in a reliable and repeatable manner.

The set-up of the equipment (Figure 3-1) comprises of a cylindrical airtight slurry feed vessel (0.0574m^3) which houses a stirrer. The feed vessel is connected via a pipeline and valve (Valve V1) to a filtration column. Valve V1 is opened when filling the filtration column with slurry and is thereafter closed when the slurry feed vessel has been emptied.

The filtration column (Figure 3-2) is a double jacketed cylinder, with the inner annulus (1m long x 0.1m wide). The inner annulus is a glass column and acts as the Nutsche filtration column and houses the slurry during an experimental run. The glass column was selected to be the inner annulus of the filtration column to allow for visual observations to be made during the experiments. This would allow for the behaviour of the suspension and filtering ability to be monitored once the third phase was introduced. The outer annulus (1m long x 0.15m wide) is made up of Perspex. The outer cylinder allows for the circulation of a cooling or heating fluid if necessary, and also acts as a safety mechanism to protect against shattering of the glass column if the pressure becomes too great.

The filter medium, on which the cake will be deposited, lies at the base of the filter column. The filter medium is supported by a series of gaskets and a porous steel filter plate which rests on a glass funnel (Figure 3-3). The filtrate liquid passes through the filter medium, through the porous steel plate and into the glass funnel from which it exits through Valve V2 (Figure 3-1) when opened. The glass funnel is attached to the filtration column by six nuts and bolts. The filtrate which exits the glass funnel is collected below in a plastic bucket which rests on an electronic mass balance (Figure 3-4), manufactured by *Radwag®*. This mass balance is used to measure the mass of the filtrate collected, in grams, to one decimal place. The mass balance is capable of reading values up to a maximum of 6kg. In order to discharge the filter cake after each run, the glass funnel has to be removed by removing the nuts using a spanner. The filter medium is then removed, along with the filter cake.
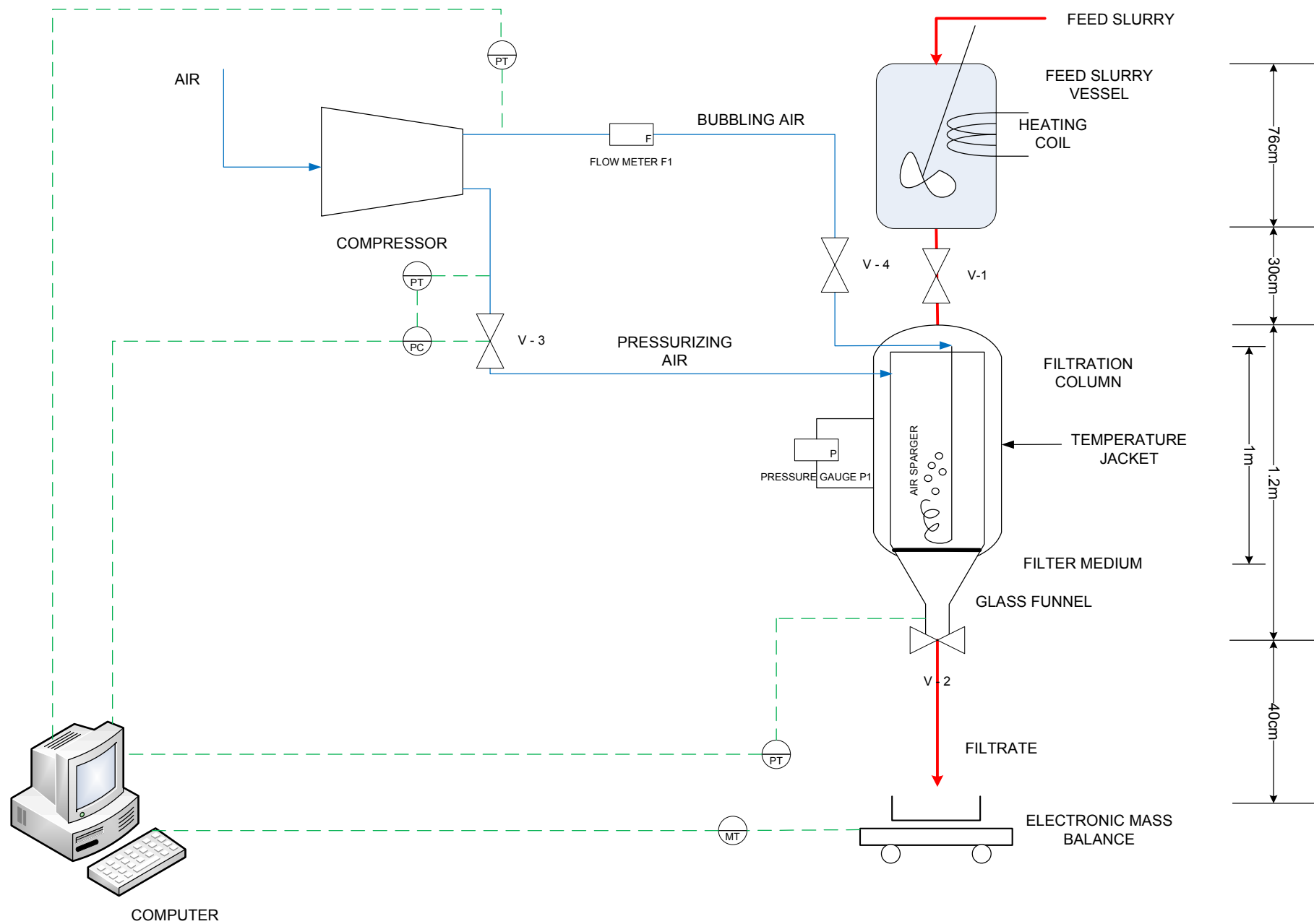
Figure 3-1: Equipment setup

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The modifications made to the equipment described by Tarleton (1998a, b) are those that would account for the introduction of the gas required for the three-phase investigation. The gas is chosen to be compressed air, as air is non-reactive. The bubbling air for the three-phase experiments could be introduced in one of the following ways:

1. Bubbled in through the top of the filtration cell where the air enters a sparger that would distribute the air through the slurry that would be present within the filtration column.

2. Bubbled through the bottom of the column through the base of the glass funnel. This would however dislodge the cake during bubbling air entry.

3. And lastly the air can be introduced by the usage of a piston which would pressurize the introduced air. This would however require modification of the equipment and would thus be used as the last alternative.

It was decided that the method of entry via the sparger would be used as this closely resembles the gas distribution within a bubble column reactor. The use of the sparger to introduce the air also prevented any disturbance to the cake formation. This would allow for the cake properties to be studied during the filtration, as this is essential when observing filtration behaviour (Tien, 2006).The air thus enters the filtration column through Valve V4 (Figure 3-1).

A compressor provides the pressurized air that would be required for the filtration. The compressor also provides the bubbling air that would be needed during three-phase investigations. The pressurizing air is fed into the filtration cell via pipework and a valve, Valve V3 (Figure 3-1). This air line is also linked via a transducer to an electronic pressure regulator which can be adjusted by the computer, via an electrical signal to ensure a constant pressure during filtration.

A pressure gauge (P1) is mounted onto the filtration cell (Figure 3-1) so that the pressure within the filtration cell can be read. Electronic sensors, produced by *Mensor®*, are also present to read this pressure along with the pressure of the bubbling air, and the pressure of the filtrate exiting the column through the use of a pressure sensor present in the glass funnel.

The computer software, programmed in *LabVIEW*, captures data at one second intervals. This data includes: the pressure of the pressurizing air, the exit filtrate pressure, the bubbling air pressure and bubbling air flow rate, as well as the mass of the filtrate being collected. (Figures 3-5 and 3-6)

The slurry that would be introduced into the system would be a mixture of water and diatomaceous earth (Figures 3-7 and 3-8). These were chosen due to the non-reactive nature of each of these components. This solid was also chosen because it is cheap, readily available and can be reused once dried. In addition, diatomaceous earth is frequently used in the filtration industry as a filter aid, which indicates that the solid has good filterability characteristics. This can be attributed to the needle-shaped particle structure of the solid and its complex structure with numerous microscopic pores (Martinovic, et al., 2006).

The solid particles were of a small size (<180µm) so a filter medium would have to be selected that would ensure that these particles were retained during the filtration. The medium was chosen by referring to the research done by Mais (1971) in which he discussed the types of filter media available. Thus, a felt filter cloth of pore size 10µm was chosen (Figure 3-9). The cloth was ordered in 3m x 3m sheets of felt and would have to be cut into the appropriately sized circles that could be used for each experiment.

The ability to safely operate the test rig was also considered. Precautionary measures needed to be taken against the possible scenario of the pressure within the filtration column reaching an unsafe value, leading to possible shattering of the glass column. Thus, a pressure release valve was linked to the column which would allow for the pressure to be released in an emergency. A Perspex shield was also mounted onto the frame of the test rig. This shield would be closed when the experiments were in operation to shield people from possible injury if the glass column shattered.
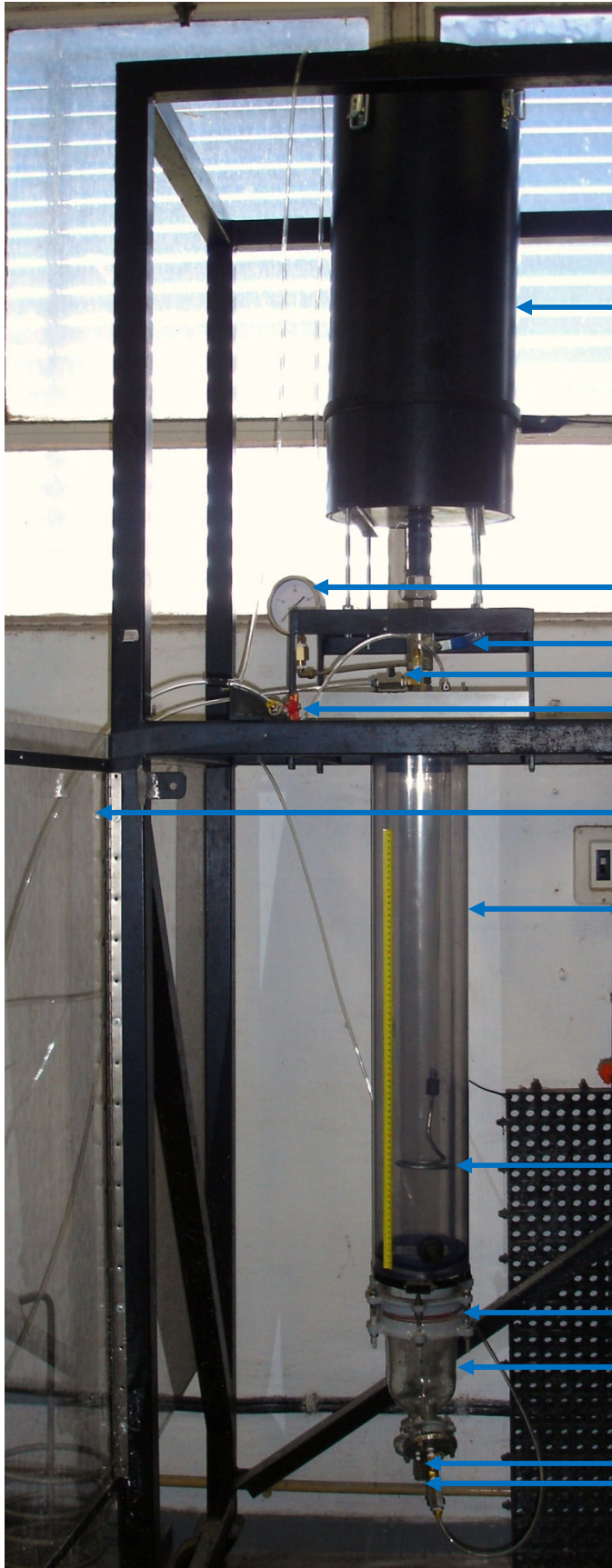
Figure 3-2: Test rig

Slurry feed vessel

Pressure gauge P1

Valve V1

Valve V3

Valve V4

Perspex shield that is closed during experimentation

Filtration column surrounded by the Perspex column

Air sparger (the third phase enters the filtration column at this point)

Filter medium

Glass funnel

Valve V2

Filtrate exit point

Filter cake

Filter medium

Glass funnel

Exiting filtrate pressure sensor

Valve V2
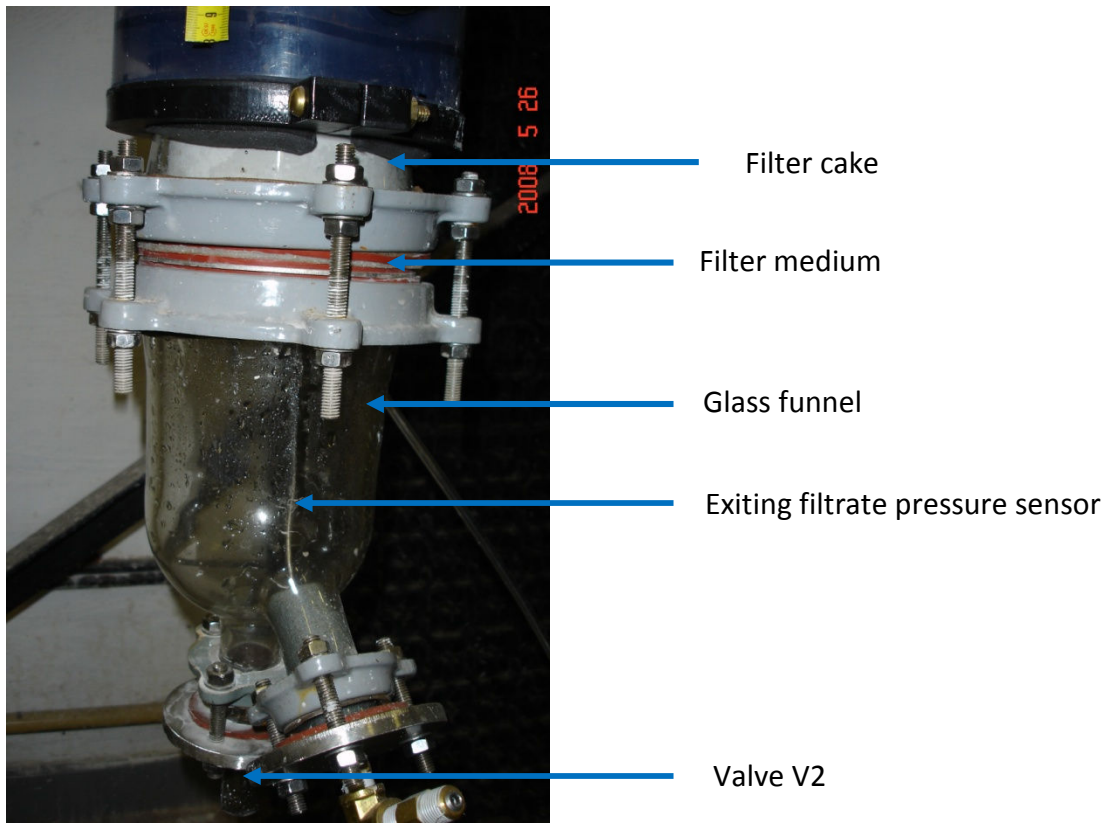
Figure 3-3: The glass funnel at the base of the filtration column

Glass funnel

Valve V2

Filtrate exit point

Electronic mass balance
(plastic bucket rests here)
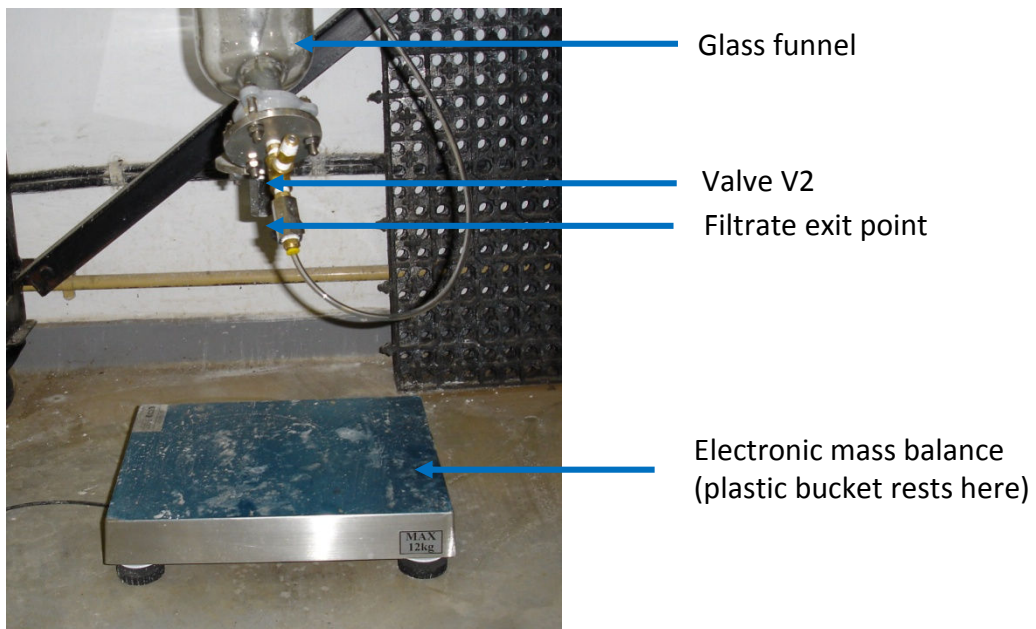
Figure 3-4: The electronic mass balance

Pressure display

Flow-meter

Figure 3-5: The computer setup
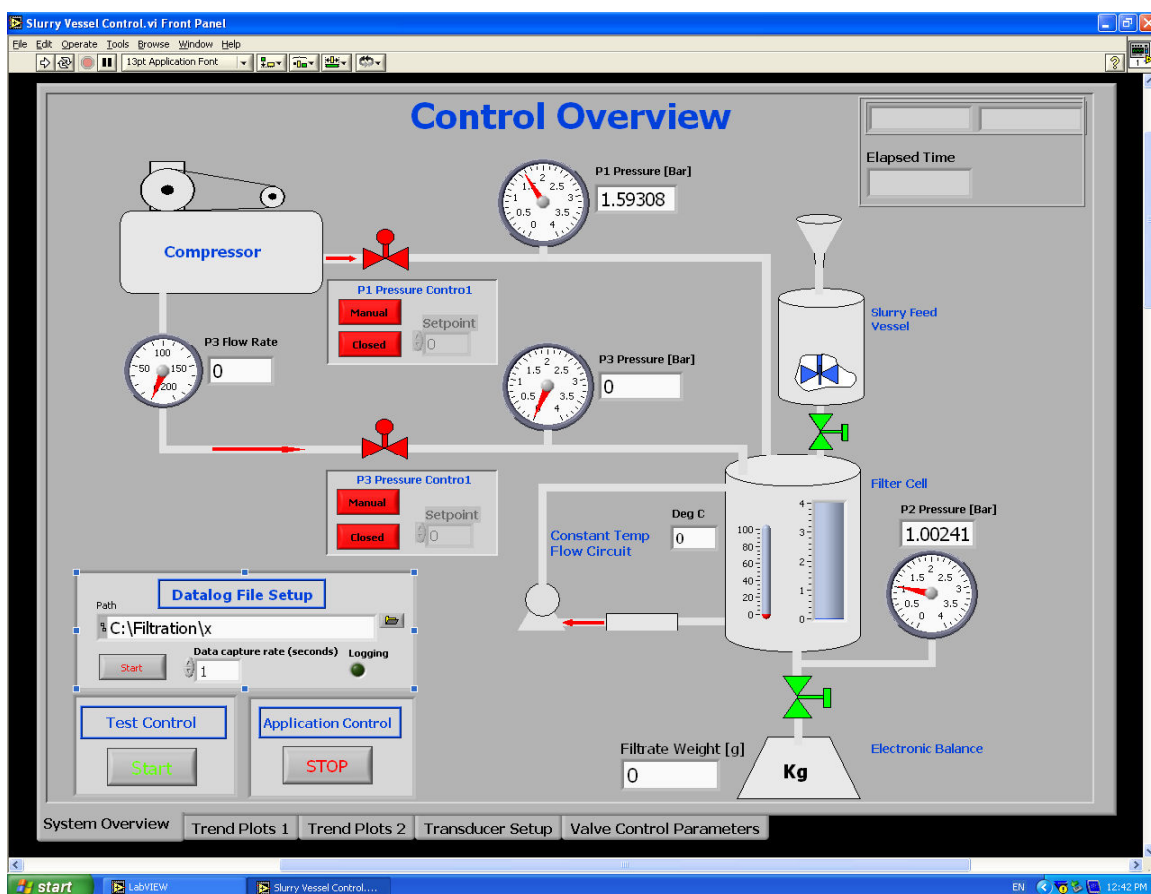
Figure 3-6: GUI of the software program

Figure 3-7: The solid material-diatomaceous earth

Figure 3-8: The slurry mixture

Figure 3-9: The felt filter cloth

3.3. Commissioning and modifications made to the initial equipment design

Any newly designed and constructed system and equipment usually has various operational deficiencies and difficulties which must be corrected for the system to be acceptable. This equipment and system needed a number of modifications to be made due to various problems encountered during commissioning and running of the system and equipment.

Listed in this section are the variations modifications that were made to the test rig during this investigation. Each modification was made in order to achieve optimum test rig operation and experimentation, and were viewed as solutions to problems encountered whilst conducting the experiments. A detailed discussion of the modifications made, can be found below.

Software problems, such as the inability to record and store data, the inability to control the valves according to the required set-point etc., were rectified by reprogramming the software and consulting with the software technician.

A leak in the system (at the point of the filter medium attachment to the glass column), was rectified by coating the outer circumference of the filter cloth with a resin that hardened. Thus, preventing the seepage of the liquid, as a better seal was provided.

Resin coating

Figure 3-10: Filter cloth with resin coating along the circumference

An air leak in the system, namely from Valve V1 (Figure 3-1), made it impossible for the column to maintain the pressure needed for filtration. This was rectified by replacing the plastic ball valve with a new steel valve, as it was believed that the particles from the slurry were creating a blockage in the old valve preventing it from being closed securely.

Over-tightening of the glass funnel during reattachment after each test run resulted in a leak from the point of contact between the filter medium and glass funnel. This resulted in the glass funnel not sitting flush against the filter medium and was corrected by realignment of the glass funnel through replacement of the flange.

A pressurised air line was used to link to the pipeline that led from the slurry feed vessel to the filtration column. This prevented the piping from becoming clogged with dried slurry. When the valve for this air line is opened, air is blasted through the pipeline into the slurry feed vessel, removing any blockages.

Cleaning air entry
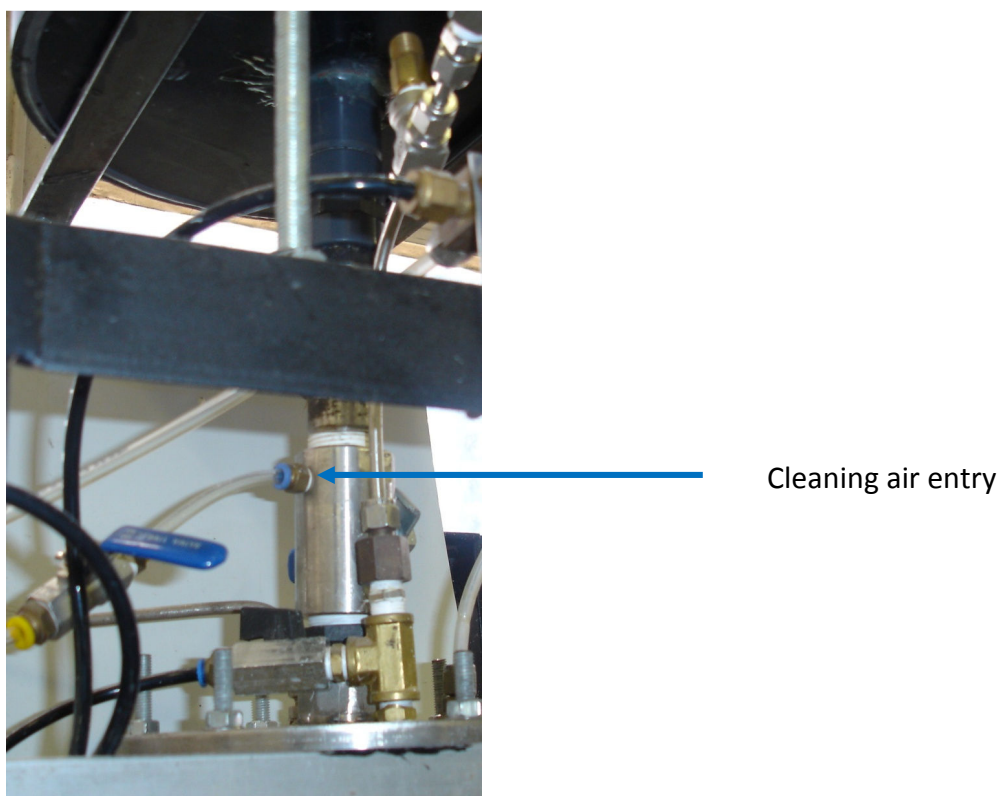
Figure 3-11: Position of the air line used to remove any blockages from the slurry feed vessel to the filtration column

Initially it was thought that the three-phase experiments would require an air line for providing the pressurized air for filtration and, a separate air line to provide the air that would be bubbled through the sparger. It was then discovered that the air that was bubbled through the sparger was capable of creating pressure within the filtration column, thus, the need for two separate air lines was unnecessary. This was rectified on the equipment design by using a single air line that exits the compressor and enters a three way valve (Figure 3-12) on the filtration column. The valve could be moved to point to the left, where the air entering the column would enter at the top of the column and push downwards onto the slurry (i.e. for two-phase experiments) or point to the right, where the air would enter the sparger and bubble upwards through the bottom of the slurry (i.e. for three-phase experiments).
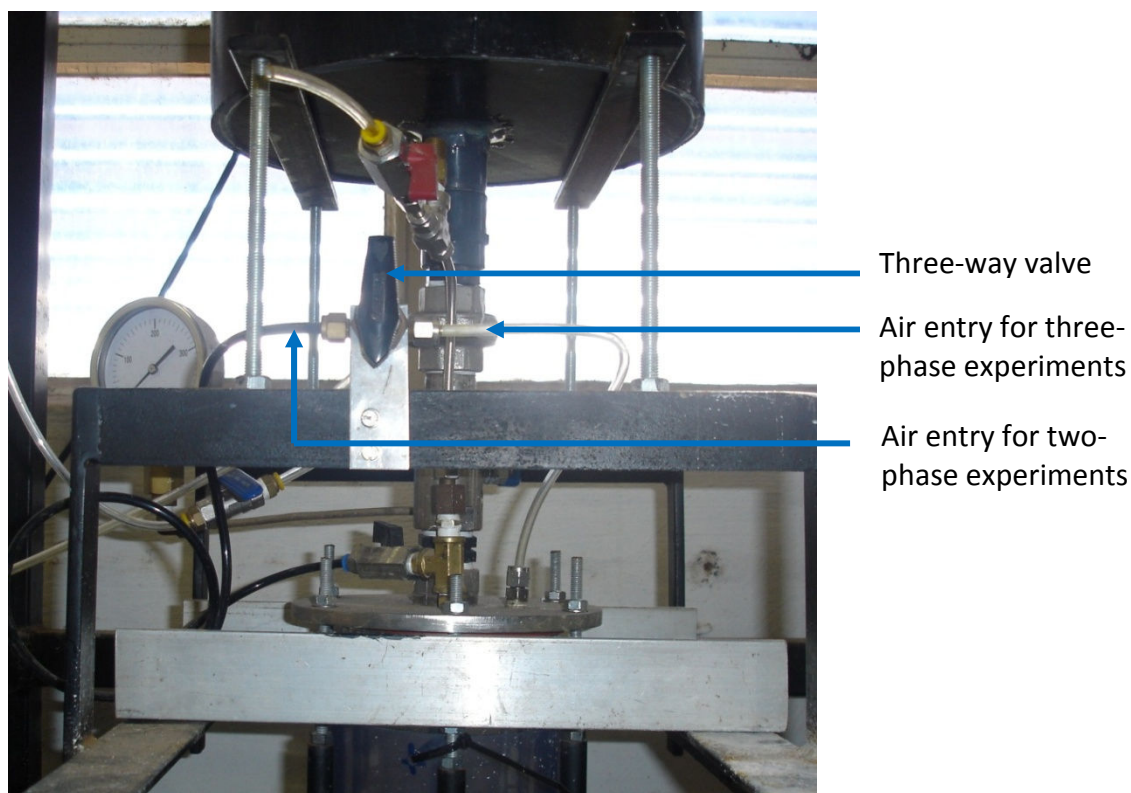
Figure 3-12: The three-way valve

A flow-meter, manufactured by *Alicat Scientific,Inc.,* was connected to the inlet air line that led to the three-way valve. The flow-meter was capable of measuring volumetric flow rates up to a maximum value of 100litres/min. The use of a single inlet air line, as opposed to the original design which incorporated two separate air lines for the two and three-phase tests, had to be

accounted for in the computer program. The pressure control for the two separate air lines was then replaced with control for a single air line that led to a three way valve. This is illustrated in Figure 3-13.
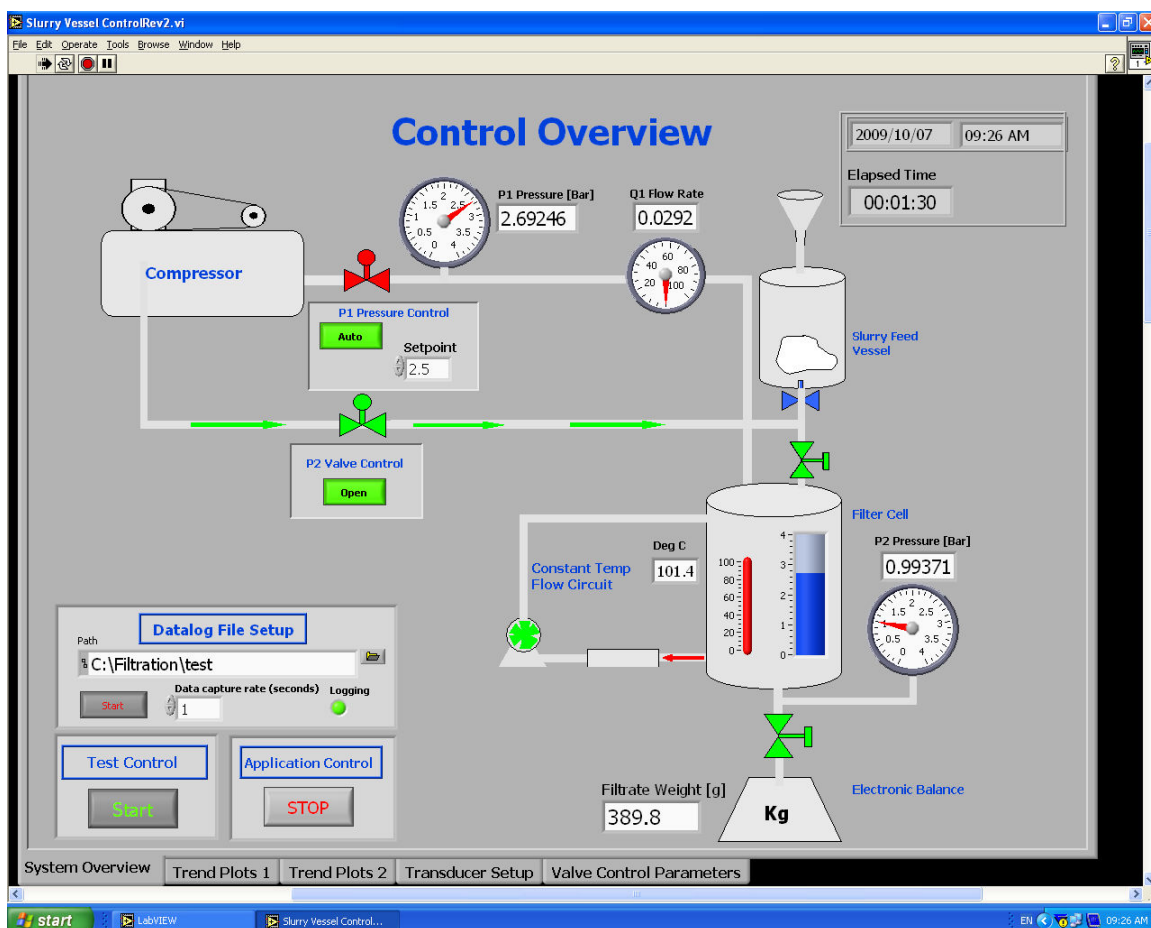
Figure 3-13: New GUI for the software program

The glass funnel (see Figure 3-14), proved to be very problematic due to numerous physical design problems, which resulted in frequent filtrate leaks at this point, during the course of an experiment. The details of the problems encountered with the glass funnel are listed below:

Ring that was free to swivel

Alternative point of entry for the third phase
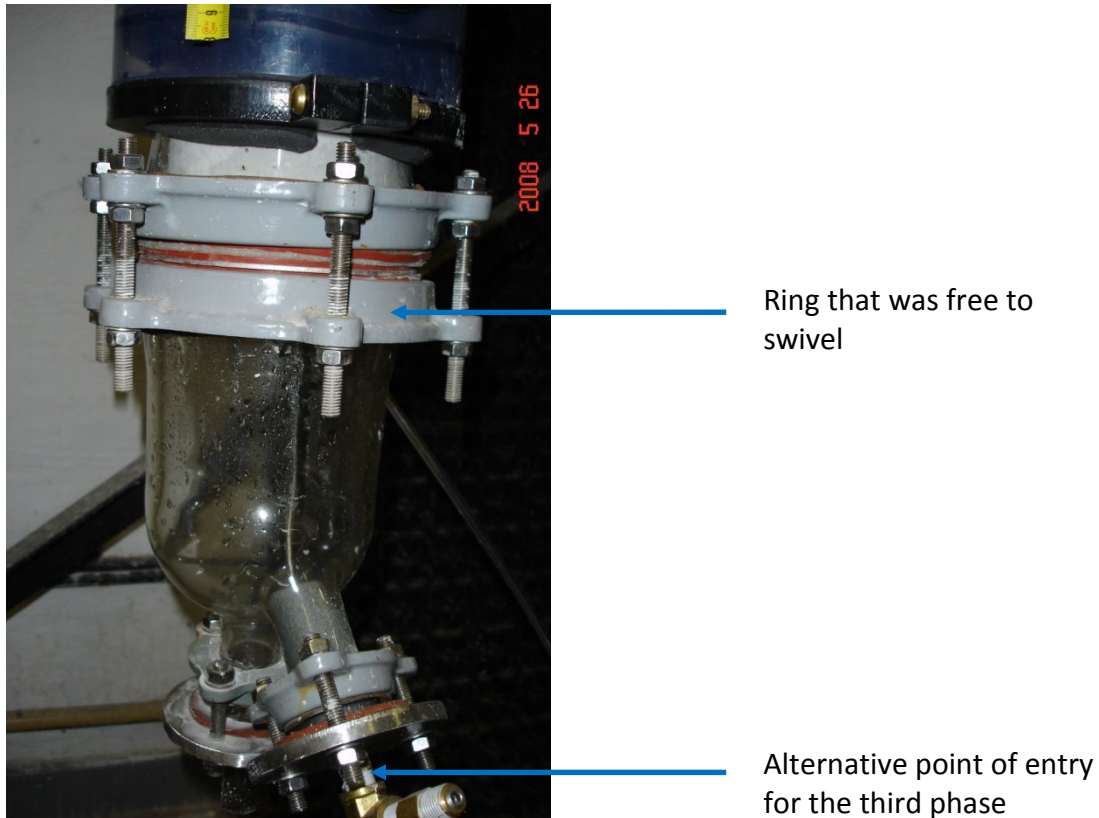
Figure 3-14: The glass funnel

The grey ring labelled on Figure 3-14 was unable to be fixed onto the glass funnel, as the funnel was tapered in shape and the ring could not fit flush against it. This made single person assembly of the unit difficult, as one hand was needed to support the glass funnel against the column, (against the force of gravity), while the other hand was used to screw the nuts into position, all whilst trying to maintain the grey ring (Figure 3-14) in a fixed position, to allow for assembly. The second design problem encountered was that the constant force of tightening and loosening the nuts for each experiment compromised the integrity of the glass funnel, which began to crack, just below the grey ring, after several experiments had been conducted.

The glass funnel was used in the setup of the system at the outset, to allow for an alternative point of entry of the bubbling air for the three-phase experiments (i.e. the air could enter through the valve indicated on Figure 3-14). This alternative would only be put into use, provided that the initial tested method of air entry, via the air sparger, was unsuccessful. Bubbling through the base of the system (through the valve shown on Figure 3-14) would not be

tackled as the first option to introduce the third phase, as this would dislodge the filter cake, making data analysis difficult. The use of the air sparger to introduce the air was tested and was found to be suitable. After much consideration, it was decided to remove the glass funnel and replace it with a solid steel base plate through which the filtrate will pass (Figures 3-15 and 3-16). A steel ring, on which a porous steel plate rests, would act as the support for the position of the filter medium. The filter medium would then be sandwiched in between this lower steel ring and another steel ring, which would be mounted against the glass column (Figures 3-15, 3-16, 3-17 and 3-18).

The only disadvantage to using the steel base plate setup as opposed to the glass funnel setup would be that the pressure of the filtrate exiting the column would no longer be able to be measured. This was not seen as a critical factor to maintain the glass funnel as part of the experimental setup, as the pressure of the filtrate exiting the column was consistently measured during various experiments and was found to be atmospheric pressure, thus eliminating the need to maintain the use of a pressure sensor at this point.

The use of the steel base plate as opposed to the glass funnel, now allowed for the filter medium to be firmly sandwiched between the two steel rings. This meant that it was no longer necessary to coat the outer circumference of the filter medium with the resin, as there was no longer any seepage of liquid occurring. The new position of the filter within the steel rings is shown below:

Solid steel base plate

Filtrate exit point
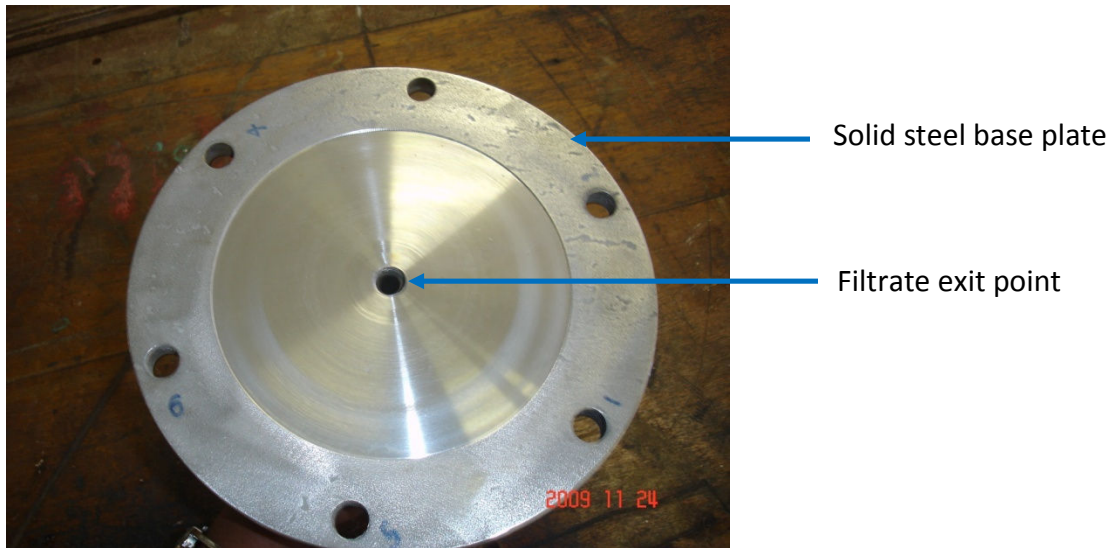
Figure 3-15: Top view of the solid steel base plate

Filtrate exit point

Valve through
which the
filtrate exits
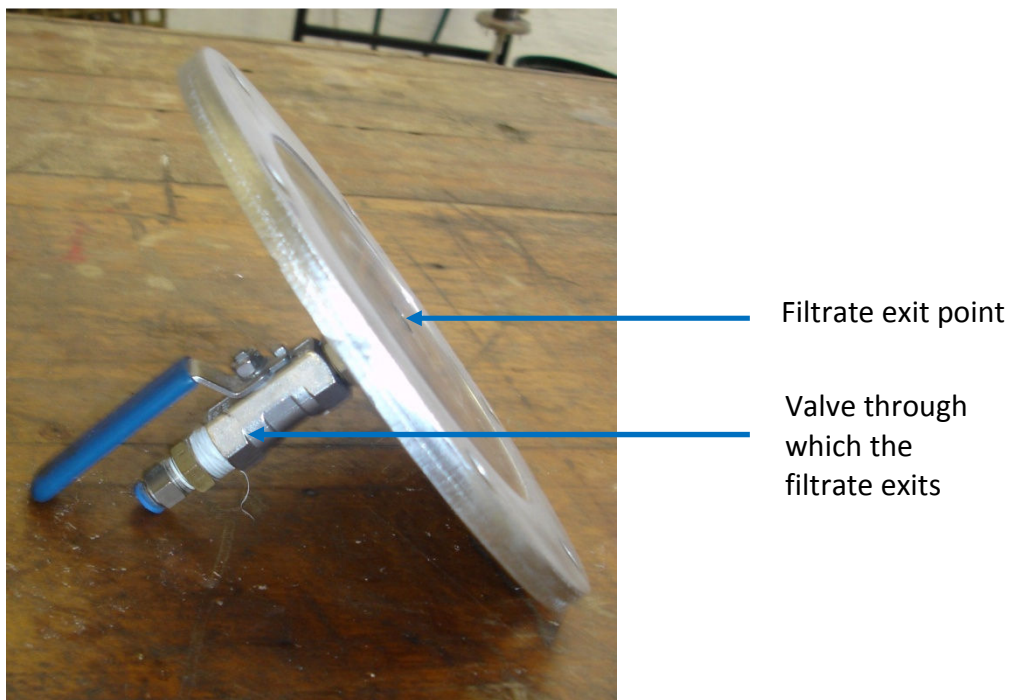
Figure 3-16: Side view of the solid steel base plate

Steel ring

Porous plate that will support the filter cloth
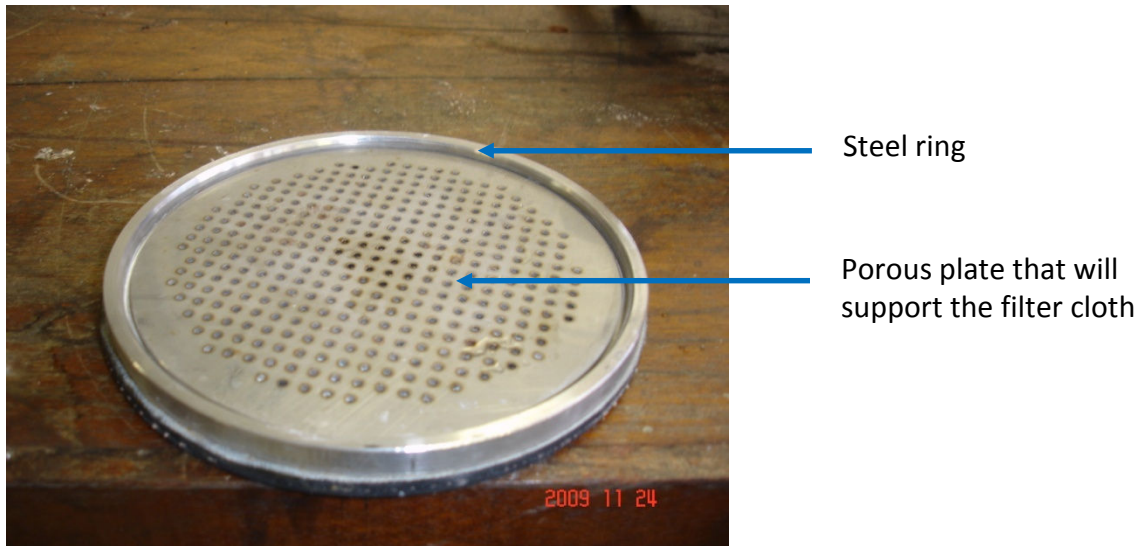
Figure 3-17: Steel ring and porous plate that will be positioned on the steel base plate

Second steel ring with a gasket

Filter cloth supported by the porous plate

First steel ring

Gasket
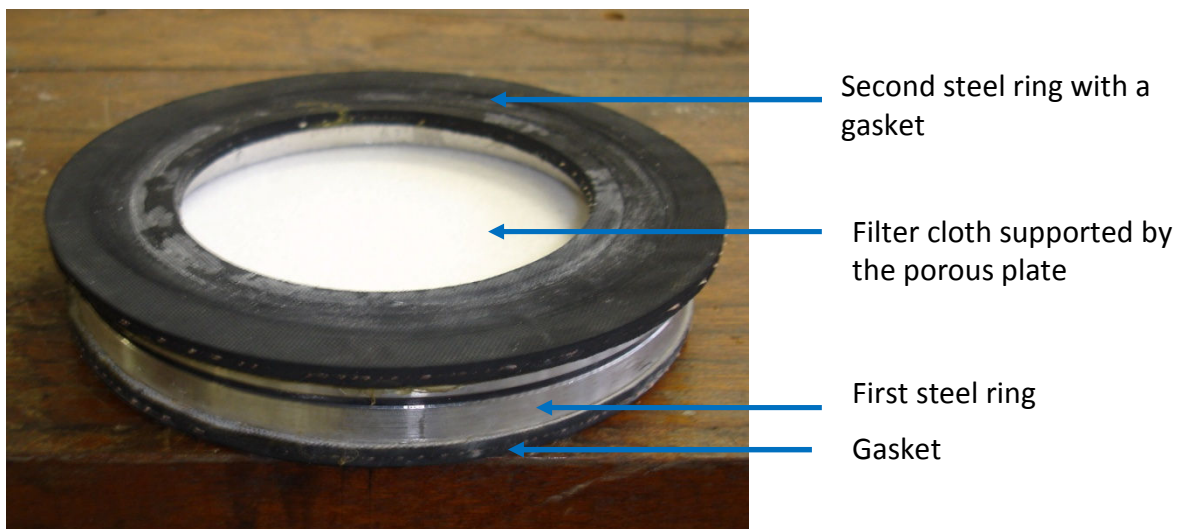
Figure 3-18: Filter medium sandwiched between the two steel rings

First steel ring with a gasket

Second steel ring with a gasket

Steel base plate

Filtrate exit valve
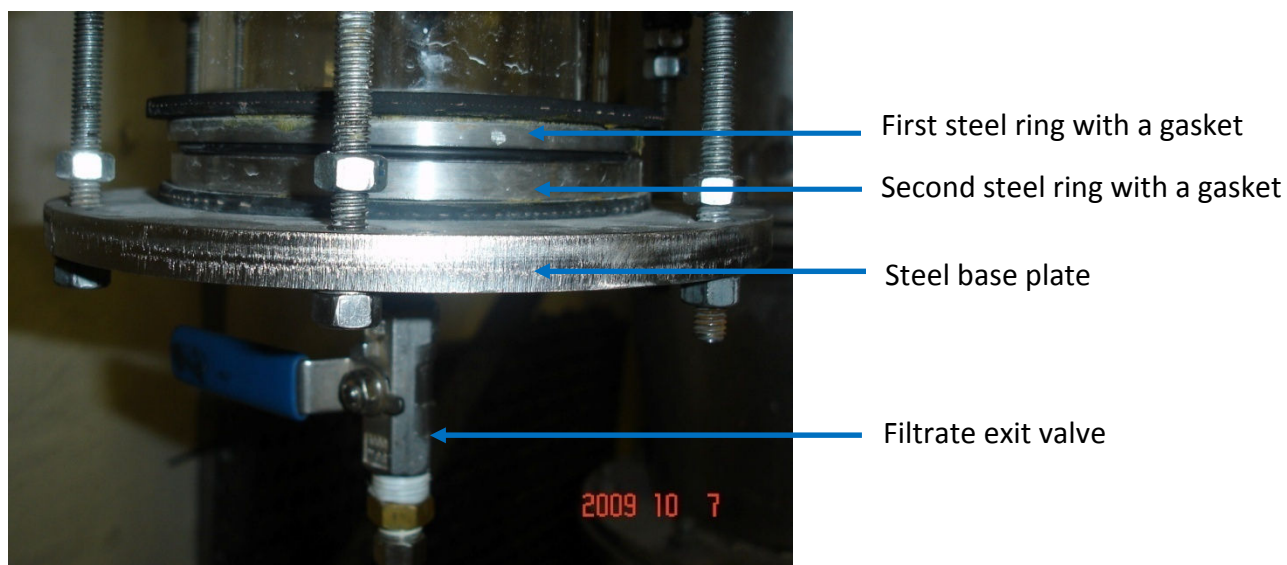
Figure 3-19: The steel plate mounted onto the filtration column

The initial design of the equipment used a glass filtration column of length 1m. The tests conducted proved that the entire 1m length of the column was not required as only one third of the column height was actually filled with slurry for any given experiment, as the time taken to fill the entire column with slurry was too long. This resulted in the solids in the slurry settling within the column, due to sedimentation. The 1m column was thus replaced with a column of length 0.5m.

Now that the length of the column was much shorter, a clear plastic tube was used to channel the filtrate from the exit point of the column (Figure 3-20) into the bucket that rested on the mass balance. This was done to prevent any loss of filtrate through splashing out of the bucket. It was then decided to place a desk above the electronic mass balance through which the tube passed, this served two purposes:

1. It acted as a "safety net" to protect the electronic mass balance, in the event that the steel plate was dropped during assembly.
2. It also acted as a convenient workspace that allowed for placement of the spanners, steel plates and nuts during assembly for each run.
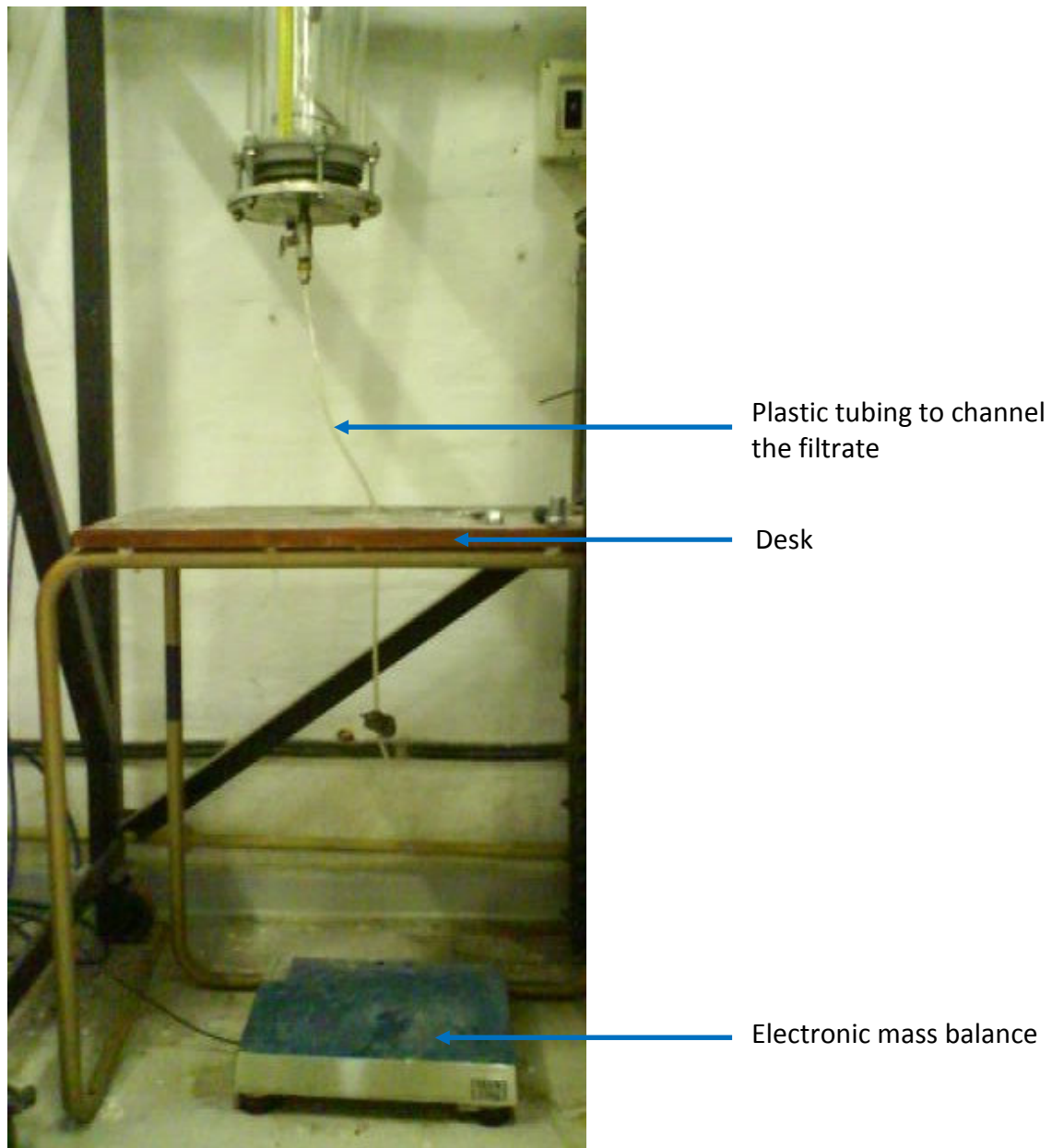
Figure 3-20: The steel plate mounted onto the filtration column

The sparger height also had to be adjusted to ensure that there was no cake build-up over it, as this made cake discharge quite difficult and air distribution through the sparger for the three-phase tests impossible. The sparger was set to be at a height of 2cm above the cake height for the experiment with the highest solids concentration of 10% (i.e. the sparger was set at a height of 8cm above the filter medium).

Repeated problems were experienced with the mass balance. The balance would frequently stop recording the filtrate mass for any given time period during the course of an experiment. This meant that the data for that specific experiment was unavailable and that the experiment would have to be repeated. This was attempted to be rectified by consulting the software company, reprogramming the software, rewiring of the balance to the pc and even replacing the pc and mass balance. It was decided that the experiments which had data recording errors would just have to be repeated, with the hope that the error would not occur during the repeat tests, however, the data recording problems occurred frequently during repeat experiments as well and the repeat experiments would usually have to be repeated, even up to five times, before the data would be recorded properly.

The equipment design was changed during the course of the experiment because part of the glass filtration column snapped off during the course of setup for an experiment. Extreme difficulty was encountered when attempting to obtain another glass column as the column was a QVF column which was imported, and allowed for the attachment of flanges (the column was flared at the top, tapered inwards at the centre of the column and then flared at the base). As a solution, the previous 1m long column was cut in half, which resulted in the new column not being flared at both ends. This altered the location of the steel bolts that were used for mounting of the steel plate, steel rings and filter medium to the glass column. The steel bolts that were previously present at the base of the column were replaced with steel rods that extended from the top of the column all the way down to the point where the steel plate would need to be attached (Figures 3-21 and 3-22).

Position of steel rods
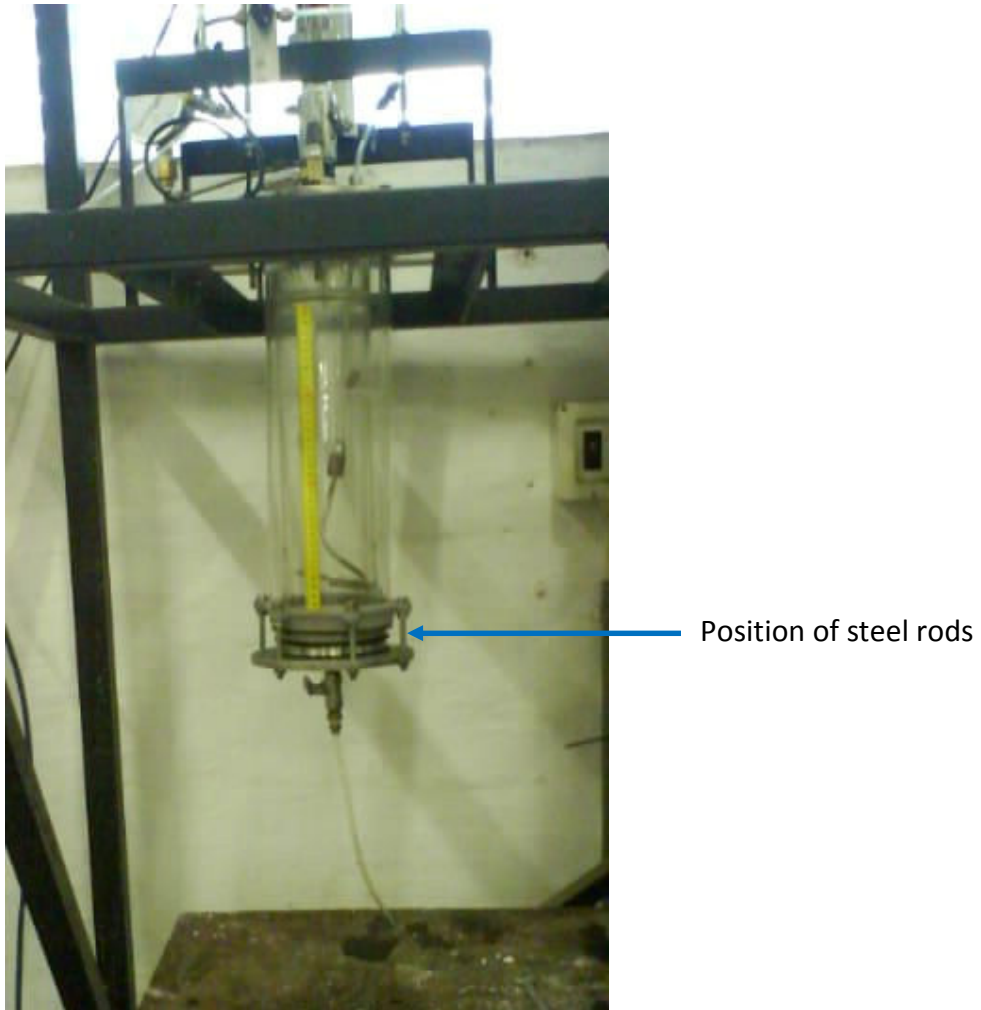
Figure 3-21: Old QVF column and position of steel rods

Position of steel rods
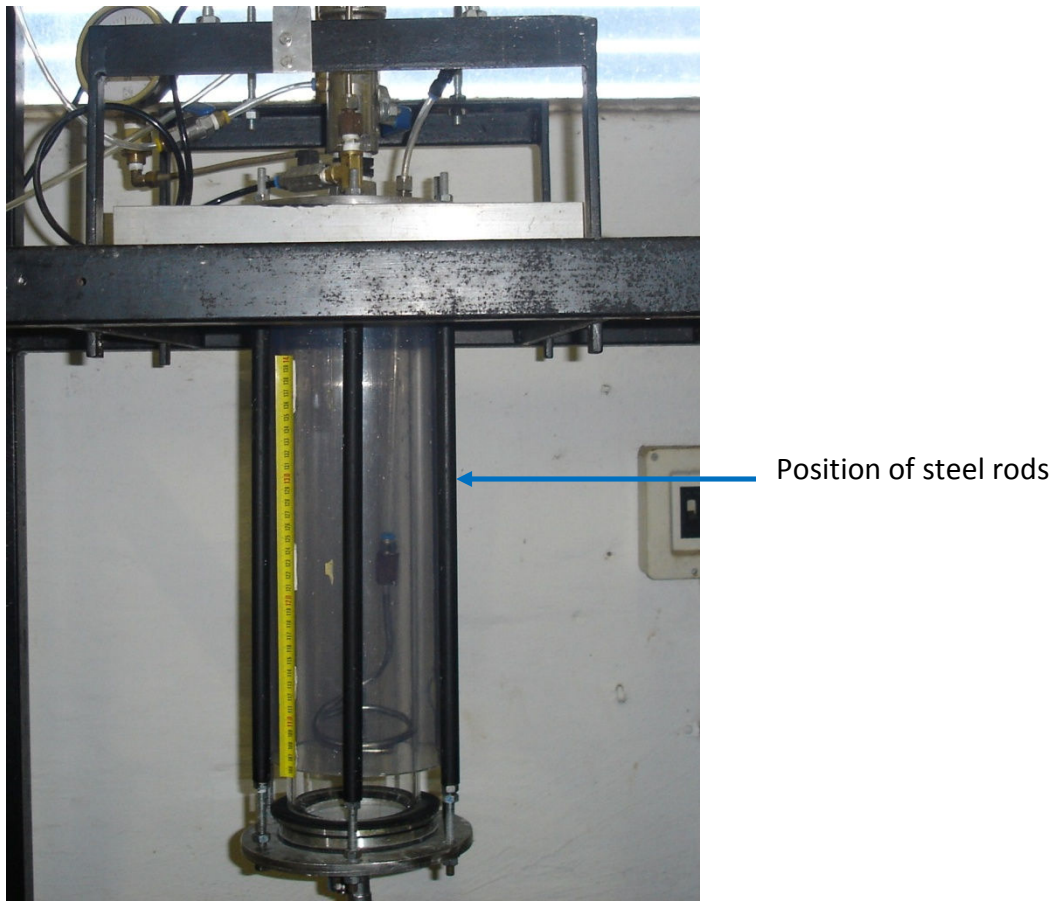
Figure 3-22: New column and new position of steel rods

The final setup of the equipment, taking all of the above modifications into account is shown below in Figures 3-23, 3-24 and 3-25.
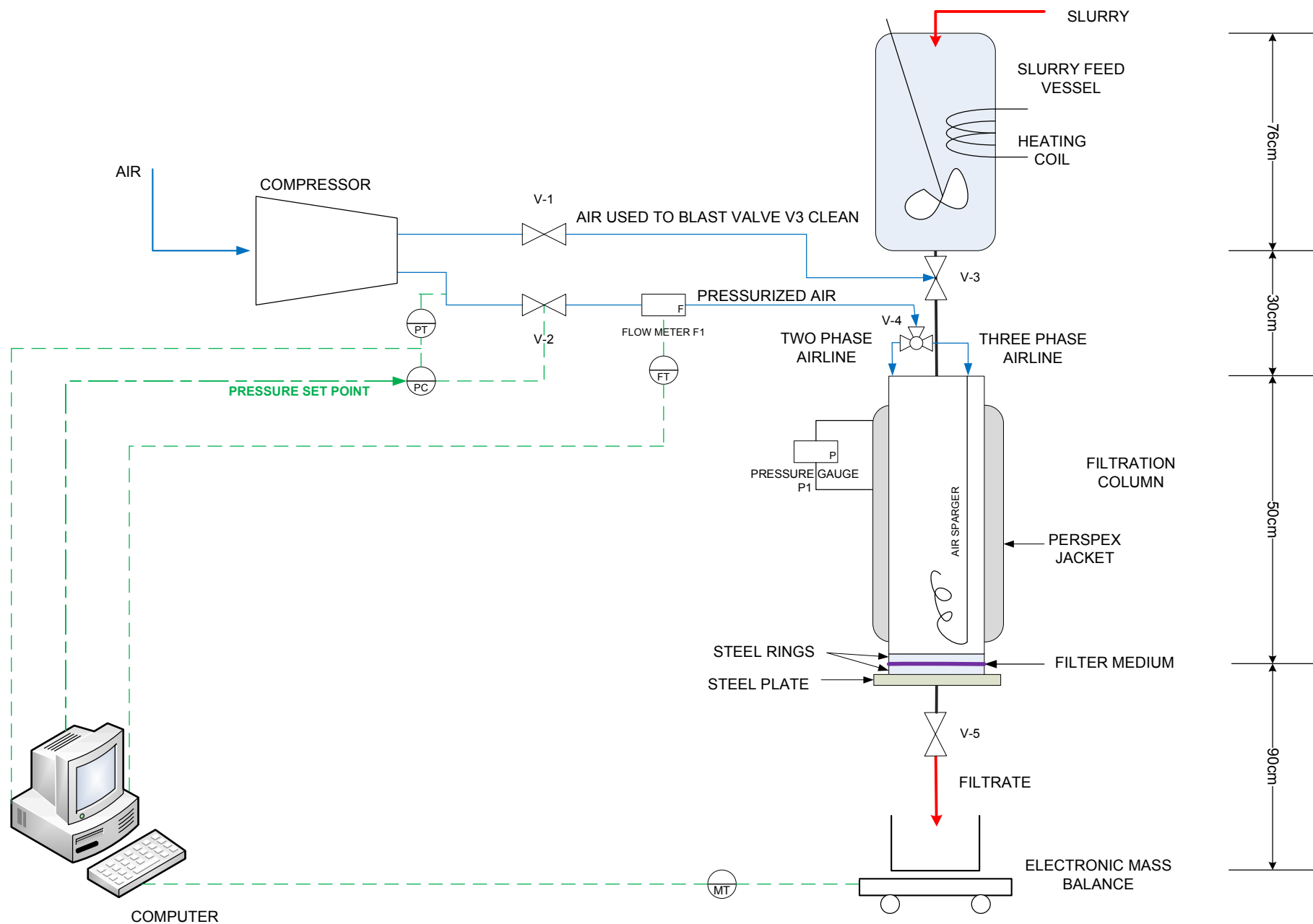
Figure 3-23: Modified equipment setup

Valve V1 (behind Valve V2)

Valve V2

One of the pressure regulators
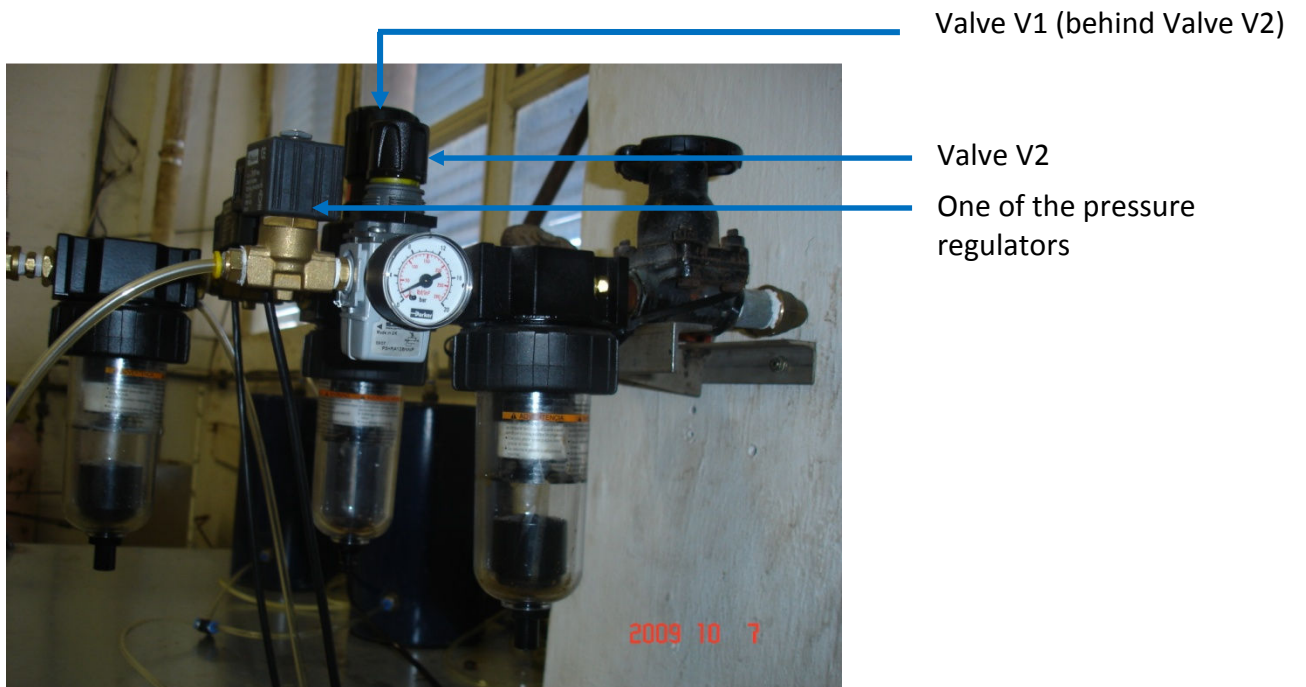
Figure 3-24: Air lines from the compressor and the pressure regulators
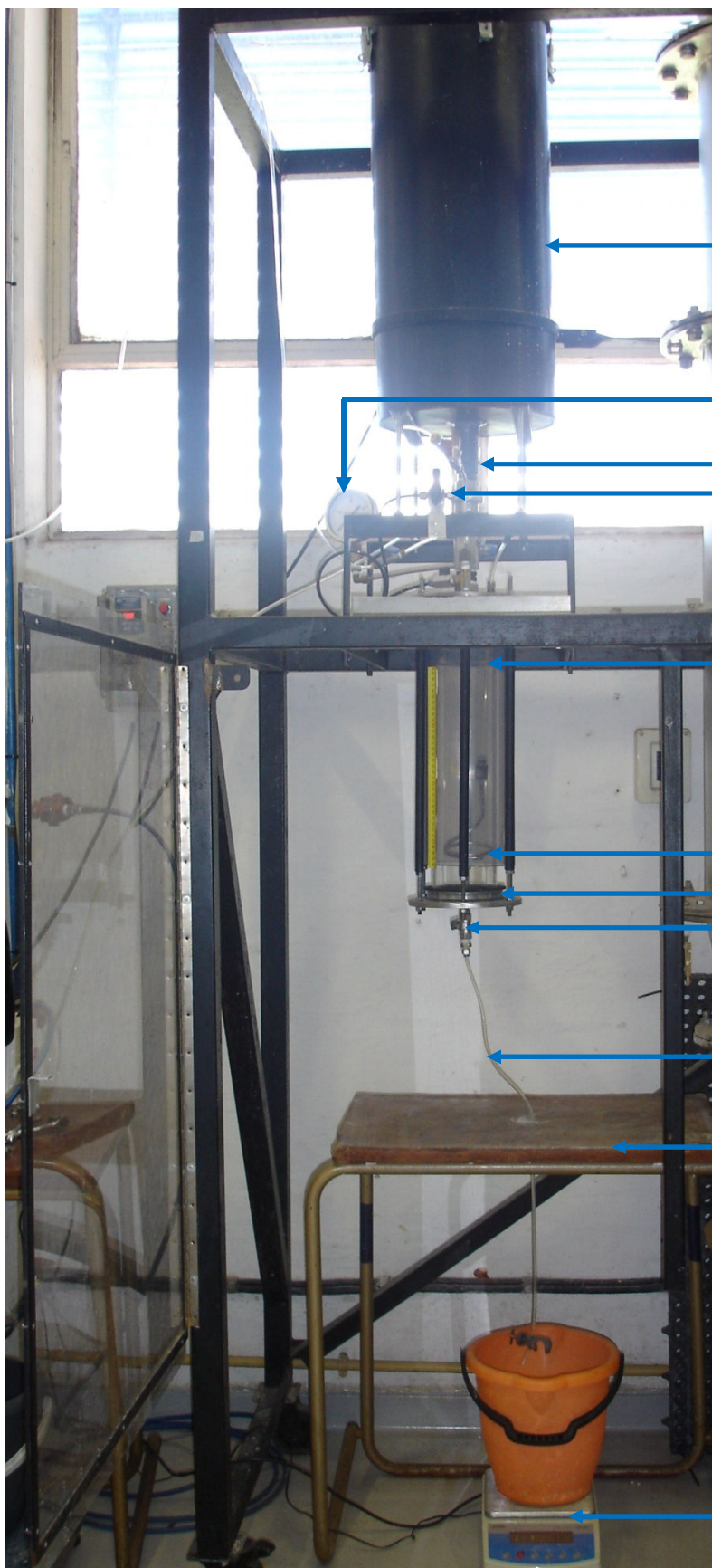
Figure 3-25:
Modified test rig

Slurry feed vessel

Pressure gauge P1

Valve V3
Three-way valve (Valve V4)

Filtration column and
Perspex cover

Air sparger
Filter medium sandwiched
between steel plates

Valve V5

Plastic tubing to channel
filtrate

Desk

Electronic mass balance

A smoothly operating test rig was finally developed, however a new set of challenges were encountered:

1. The test rig was situated on the ground floor of the chemical engineering laboratory. On the floor above this, two students were beginning experimental work on a reaction apparatus. In April 2009, the students had conducted a pressure test on the apparatus incorrectly, which resulted in a leak that flowed onto the ground floor and wet the 25kg bag of diatomaceous earth used for the experiments. A new bag of diatomaceous earth had to then be reordered, which took a month to deliver from the supplier.

2. A second leak occurred two weeks later but was noticed on the ground floor near the test rig just in time to put an end to it before any major damage could occur.

3. However, in June 2009, the students had left a cooling water valve open overnight. This resulted in a continuous gushing downpour of water throughout the night into the following morning. The three-phase test rig was completely damaged, with the electrical instrumentation beyond repair, even though it was all housed with the pc within a protective wooden box. The electrical instrumentation had to be replaced. This included a flow-meter that had to be reordered from the U.S and took 4 weeks to deliver, along with replacement of the electronic mass balance and rewiring of all the instrumentation.

4. The test rig was rebuilt once again and commissioned and finally up and running by September 2009. A galvanised steel roof was built as a preventative measure over the wooden box that housed the pc and all the instrumentation.

3.4. Experimental procedure

The experimental procedure for the two-phase system was modified until the optimal operational procedure was obtained. This is as follows: (Refer to Figures 3-13, 3-23 and 3-25)

1. Valves V1, V2, V3, V4 and V5 are closed.
2. The stirrer for the slurry feed tank is switched on.
3. Three litres of water is measured in a bucket. This quantity of water was chosen as this occupies 75% of the volume of the filtration column, allowing for the filtration to occur with the maximum allowable amount of suspension possible within the filtration column.
4. The required mass of diatomaceous earth needed to create the solids concentration on a mass/mass% basis is added to the water in order to create a slurry. The solid concentration levels that would be used to make up the slurry are discussed in Chapter 4.
5. The slurry is continuously mixed in a bucket using a glass rod to prevent any sedimentation and is then added to the slurry feed tank via the funnel at the top of the slurry feed vessel.
6. The computer program is now started. The program is started by clicking the white arrow on the top left hand corner of the screen (below the "*edit*" tab) (Refer to Figure 3-13).
7. Thereafter the file to which the program will record the data must be given a name. This is done in the "*Datalog File Setup*" box on the bottom left hand corner of the screen. The file name given in this example is "*x*" and the default path in which the files are saved in the *C:\ drive*, in the folder labelled "*filtration*". The "*Start*" button below this is then clicked to create the file in which the data will be recorded. (Refer to Figure 3-13)
8. In order to begin recording of the data, the "*Start*" button in the "*Test Control*" box on the bottom left corner of the screen (below the "*Datalog File Setup*" box) is clicked. (Refer to Figure 3-13)
9. Valve V2 now needs to be opened electronically to allow for the flow of the pressurized air that would be used within the column. The valves that are red in colour in Figure 3-

13 can be opened by clicking on the red "*closed*" button next to each valve. This would then turn green in colour and the valves would be displayed as "*open*".

10. To allow for automatic adjustment of the pressurized air flow rate via Valve V2, the desired pressure set-point for the experiment must now be entered in the program. The valves can be set for automatic control by clicking on the red "*manual*" button (Figure 3-13) which would then turn green in colour and be displayed as "*automatic*". The desired pressure set-point of the system can now be entered in units of bar, in the box below the word "*setpoint*". (The range of pressures that would be used in the system is discussed in Chapter 4).

11. Thereafter Valve V3 is opened manually to allow the slurry to empty from the slurry feed vessel and fill the filtration column. Once all the slurry has emptied from the slurry feed vessel, Valve V3 is closed and the filtration cycle is ready to begin. The height of the slurry within the filtration column (H_s) is then recorded.

12. Valve V4 is the three way valve and is turned to the left for airflow for two-phase experiments or turned to the right for airflow for three-phase experiments. Valve V4 is now opened manually in the direction required for the duration of the experiment.

13. Valve V5 is opened simultaneously with Valve V4 to allow for the filtrate to collect in the bucket that rests on the mass balance. The opening of this valve is done manually.

14. During the experiment, the computer records the following parameters at one second intervals: Time, Filtrate weight, Pressure of the pressurizing air and Flow rate of the pressurizing air.

15. If conducting a three-phase experiment, the time at which the level of the slurry drops below the level of the sparger is recorded (t_{2p}). This is explained in Chapter 4.

16. Upon completion of the filtration, the experiment is complete. The computer program must now be stopped from logging data and Valve V2 will be closed electronically. The program is stopped from recording data by clicking the "*Stop*" button in the "*Application control*" box on the bottom left hand corner of the screen (Figure 3-13). The program can now be stopped completely by clicking on the red circle button on the top left hand corner of the screen below the "*Operate*" tab. By stopping the program, all valves that were opened electronically are now returned to their default setting, which is closed.

17. Valves V4 and V5 will then be physically closed.

18. The steel plate on which the filter medium rests must now be removed; this is done by removing the six nuts that are attached to the bolts that hold the steel plate and steel rings flush against the filtration column. These nuts are removed with the use of a spanner. The steel plate and steel rings are removed, along with the filter medium and the cake is discharged.

19. The cake height is measured and the cake is removed and weighed to determine the wet cake mass. The cake is then placed to dry in an oven. This is weighed again after 24hours to determine the dry cake mass. These values are also used to determine the moisture content of the cake.

20. In order to cleanse the system of any remaining filter cake or slurry, the system is rinsed. This is done by placing an empty bucket on the desk below the unsealed glass column. Valve V3 is then opened and using a hose, the slurry feed vessel is washed with the wash liquor (clean water) which flows through the filtration column and collects in the bucket below the filtration column.

21. Valve V1 is then opened and compressed air is blasted through the pipeline into the slurry feed vessel to remove any blockages.

22. The steel plate and steel rings along with the filter medium are then mounted back onto the filtration column, ensuring that all nuts are screwed back on tightly.

23. All the valves are now closed and the system is ready for a new experiment.

CHAPTER 4:

RESULTS AND DISCUSSION

The complete experimental and computing cycle for this investigation comprised of the following:

1. Preparing the slurry to meet the desired specifications.
2. Performing the experiment and varying the necessary factors.
3. Analysing the experimental data by constructing the filtration characteristic curve (from Equation 2-17) and determining the filtration range by selecting the start time for the filtration and the cut off time for the filtration (see later).
4. Computing the filtration constants: α_{av} and R_m for the experiment.
5. Constructing the cake formation rate curve for the experiment.
6. Comparing the behaviour for the three-phase and two-phase tests.
7. Determining the factors that had significant effect on the filtration constants, through a statistical analysis.
8. Fitting a regression model that describes the behaviour of the system.

The experimental results presented are representative of more than 300 filter cycle tests inclusive of the initial commissioning experiments. Prior to the execution of the three-phase experiments, tests were carried out on two-phase systems. The purpose of this was twofold. First was to establish the effectiveness and smooth operation of the test rig. The second purpose was to obtain data, against which the three-phase tests could be compared, thereby establishing the effects of the gaseous phase on filtration.

Each of the steps listed above and the decisions made regarding their execution is discussed in the headings that follow. These are divided into several categories:

1. Selecting the parameters that would be varied and determining their range of operation
2. Decisions made regarding test rig operation
3. Results from the tests in which one factor was varied at a time
4. Results from the factorial experiments
5. The regression model

4.1. Experimental decisions

4.1.1. Selecting the parameters that would be varied and their range of operation

Once commissioning was complete, experimentation commenced. The experimental method of operation of the equipment was well understood, but the manner in which the factors would be manipulated and their operational range was difficult to determine. The filtration process is influenced by a large number of factors (as discussed in section 2.7), however, for the initial experiments it was decided to investigate three factors out of some of the most significant factors known. The chosen factors that would be investigated are: the effect of the solids concentration of the slurry, the applied pressure used for the filtration and the pore size of the filter cloth. These factors would be varied one at a time and the effect of each on the filtration would be observed independently.

The experiments discussed in the section that follows, were used to help to determine the operational range of the parameters that would be tested. Once the optimum operational range was determined, these experiments were then used to assess the filtration behaviour by determining the filtration constants (the experimental results for these experiments follow in section 4.2).

A discussion regarding the variation of each of these parameters follows, with an explanation detailing the difficulties encountered.

(a) Varying the solids concentration

This set of experiments required that the solid concentration of the slurry be varied to different values whilst all other factors (i.e. the applied pressure and filter cloth pore size) were fixed.

Prior to experimentation, exactly what solid concentrations should be used and whether these solid concentrations should be measured on a volume basis or mass basis needed to be determined. Although the volume basis was an option, a mass basis was selected. The mass basis allowed for the mass of solid to be directly linked to the mass of liquid used. The mass of the wet cake and the dry cake were two parameters that were measured for each experiment. By creating the solids concentration on a mass

basis, the mass of the dry cake could be compared to the mass of solids used to create the slurry. This was done so as to assess the efficiency of the filtration device, based on the solids recovery of the filter medium. Thus, all solid concentrations that are mentioned throughout this dissertation were measured on a mass/mass percentage basis.

Difficulty was experienced when deciding what solid concentration values should be used. Initially these concentrations were chosen to be 5, 10 and 15%. It was immediately seen from the experiments and data analysis that additional concentration levels would be required to produce quantifiable results. This was then modified to include additional concentration values that lay in-between the concentration levels that were tested. This was done to monitor how the transition between the concentration levels altered the behaviour of the filtration system. The concentration values used were 5, 7.5, 10, 12.5 and, 15%. The experiments were conducted and the two-phase experiments ran smoothly. However, visual observations were made during the three-phase experiments that caused concern. During the actual filtration, the third-phase of bubbling air ought to create a buoyancy effect within the filtration column. However, during these experiments the solids in the slurry would settle too rapidly during the filtration, not allowing for much turbulence to be created by the bubbling air. This was as a result of the solids concentrations that were used in the system being too high and it was decided to reduce the solids concentration to much smaller values. This would prevent the rapid settling of solids and allow the bubbling air to serve its intended purpose.

The use of a solids concentration as high as 15% in the test rig, also resulted in the air sparger having to be positioned quite high in the column so as to prevent cake build-up over the sparger. An experiment of 15% solids concentration would produce a cake height of approximately 10cm, the sparger would thus have to be positioned to bubble air at a height of 13cm above the filter cloth to prevent any cake build-up over the sparger.

Taking the above mentioned into consideration, the amended solids concentrations that were used were 1, 2, 3, 4, 5, 6, 7, 8, 9 and, 10%. These provided sufficient concentration levels to perform comparisons of the filtration behaviour within the concentration range. A decrease in the concentration levels also proved to reduce the rapid settling, as previously experienced. The sparger height was then lowered to 8cm, as the maximum cake height that would be reached for a slurry with solids concentration of 10%, would be approximately 6cm.

(b) Varying the applied pressure

This set of experiments required that the applied pressure for each experiment be varied whilst all other factors (i.e. the solids concentration of the suspension and the filter cloth pore size) were maintained as constant.

The glass column was used as part of the filtration column structure to allow for visual observations to be made during the operation of the test rig. The use of a glass column provided a restriction on the maximum pressure that could be applied in the system. In order to prevent shattering of the glass column, the maximum allowable operating pressure of the system was 3bar abs. Thus, the applied pressures could range from 1bar abs (atmospheric pressure) to 3bar abs. The control system implemented in the system design, although accurate, could not be precise enough to provide control to one decimal place. This was explained by Tarleton (1999), who suggested that the rate and magnitude of the pressure adjustments by a controller was dependant on the nature of the feed, the compressibility of the filter cake and the desired process conditions. The pressures that could be applied to the system where thus further constrained to differ by units of 0.5bar. Thus, the pressures that were used for experimentation were 1.5bar abs, 2.0bar abs and 2.5bar abs.

All pressures mentioned in this dissertation were all quantified on an absolute pressure basis (being 1 atmosphere above the gauge pressure).

(c) Varying the pore size of the filter medium

This set of experiments required that the filter cloth be varied by using cloths with different pore sizes and the remaining parameters (solids concentration and applied pressure) are fixed and not allowed to fluctuate.

The filter cloth that was readily available, and could be purchased in large sheets and cut to the required size, was the felt filter cloth of pore size 10μm. Despite attempts to obtain the filter cloth in alternate pore sizes, one other pore size (of 20μm) was available for commercial use. These two cloths were then used to compare the effect of the pore size on filterability.

(d) General decisions made regarding test rig operation

The set of experiments in which the solids concentration was varied (from 1% to 10%) required that the pressure applied be fixed at a specific value for all the experiments. The filter cloth pore size used would also have to be the same for each of these experiments.

The applied pressure that was fixed for this set of experiments was chosen to be 2.5bar. This was selected as this was the highest pressure that could be applied to the system [as discussed in section 4.1.1 (b)] and would thus be able to create the maximum air flow rate needed for the buoyancy required during three-phase experiments.

The filter cloth pore size that was fixed for this set of experiments was chosen to be 10μm. This cloth was chosen as it had a smaller pore size, as compared to its alternative, and thus had the potential to trap more of the smaller sized solid particles.

The set of experiments in which pressure was varied (from 1.5bar to 2.5bar) required that the solids concentration of the slurry be fixed along with the filter cloth pore size used. The concentration used for these tests was chosen to be fixed at 5% as this was the central concentration value within the concentration range. The filter cloth used was the 10μm cloth, for the same reason as explained for the experiments in which the solids concentration was varied.

The set of experiments in which the filter cloth pore size was varied, used a fixed solids concentration of 5% and fixed pressure of 2.5bar. The reasons for selecting these values are based on the justifications listed above.

Just as the factors and their ranges were determined, the experimental method and decisions that would affect data analysis also needed to be looked at. The details regarding these are listed in the discussion that follows.

While observing the settling behaviour of the solid particles within the filtration column, during the initial stages of experimentation, it was found that after emptying the slurry from the slurry feed vessel into the filtration column, the solids in the slurry would settle (due to sedimentation) within a minute of filling the filtration column. The experiments thus had to be executed rapidly, trying to make the start of

filtration occur almost immediately after the slurry feed vessel was emptied and the filtration column filled. This method of operation was also suggested by Westerterp and Wammes (2005, page 29).

For the initial experiments, the filter cloth was reused multiple times. The cloth would be removed once an experiment was complete, the cake scraped off the surface and the cloth cleaned by washing with water and then reused for the next experiment. This was done initially as there was a limited quantity of cloth available to use for the experiments. The effects of reusing the cloth were immediately noticed when analyzing the results. If a new filter medium was used for each experiment, the calculated value of the resistance of the filter medium (R_m) ought to be similar for each experiment as the same type of filter material (diatomaceous earth) would be tested each time. However in this case, since the filter medium was reused for the experiments, it was expected that the resistance of the medium would increase with continued use, due to blinding of the pores of the cloth as a result of entrapment of fine particles. However, upon analyzing the results from the experiments, it was seen that this value would fluctuate erratically for different experiments. This fluctuation could be attributed to the inability to consistently achieve the same level of filter medium cleansing after each experiment when the filter cloth was washed. This ultimately altered the resistance of the medium for different experiments. The blinding of the medium also reduced the efficiency of the cloth, restricting filtrate flow. The inability to guarantee that the cloth was rinsed after each experiment with the same level of solids removal from the pores, contributed to the proposed idea of now using one filter cloth per experiment and disposing of the cloth after use.

The diatomaceous earth filter cake that was produced at the end of each experiment was dried in an oven in order to determine the mass of the dry cake. The solid could then be reused for other experiments. This was thereafter deemed erroneous as the dried material may not have contained the very fine particles that were present in a fresh sample. These fine particles could have been lost due to blinding of the filter cloth, which was thereafter disposed off. The fines could also have been too small to be retained on the cloth and may have exited with the filtrate. Thus, the idea of re-use was altered to using a fresh sample for each investigation.

Westerterp and Wammes (2005, page 29) defined the end of filtration to occur at the point when the slurry was depleted. Thus, the filtration for the test rig was deemed to be completed once the slurry within the filtration column was depleted. However, the cake was still very moist at this point in time,

and almost in a liquid state. This made cake discharge messy and the cake height was difficult to measure. It was then decided that the filtration would continue for 1 minute after the last bit of slurry was filtered. This would remove any excess moisture from the pores of the cake. The cakes were then effortlessly discharged and the cake heights were easily obtained due to the inclusion of this cake compression stage.

The filtration of the last bit of slurry present in the filtration column forced the remaining liquid through the pores of the cake as a blast of air-liquid mixture. This was due to the applied pressure forcing the liquid through the cake pores. The pressure applied to the system would suddenly sail to lower values with the inability to increase thereafter. This was deemed to occur due to the final forcing out of the liquid within the cake pores and the beginning of the cake compacting.

The filtration column was capable of holding a maximum of 30cm of liquid suspension. The filtration time for the various experiments ranged from 1 minute to 5 minutes. Each experiment took approximately 30 minutes to complete, this included the removal of the nuts; the discharge of the filter cake; washing of the system; replacement of the filter cloth and tightening the nuts for a new experiment.

4.2. Experimental Results

4.2.1. Filtration characteristic curves and data summaries

Figures 4-1 and 4-2 illustrate the original filtration characteristic curves, and are used to explain how the start time of filtration (t_s) and end time of filtration were determined. Initially, the start time was chosen to be the time at which the first drop of filtrate was collected on the electronic mass balance. This provided the start up mass (m_s) which can be converted to the start up volume (V_s) using the density of the filtrate. However, when using this time as the t_s in the data analysis, the filtration characteristic curve, which ought to be a straight line, was graphed as follows:
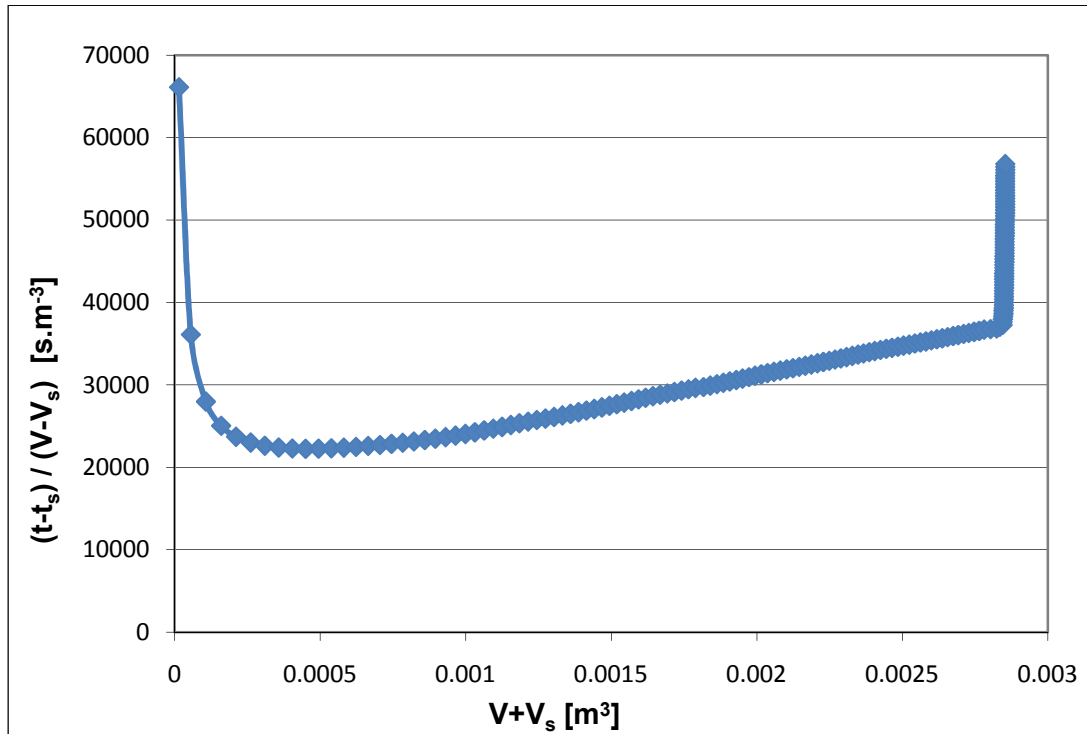
Figure 4-1: Original filtration characteristic curve (Run 61)

The steep slope at the end of the curve occurred during the 1 minute cake compression stage. The increase in the gradient was due to the fact that all the slurry within the filtration column had been filtered and the only liquid that continued to collect on the mass balance was the liquid that was progressively removed from the cake pores, through the applied compressed air. It was decided to remove data following transition to the compression stage when plotting the filtration characteristic curve, as there was no further cake growth in this region. The filtration characteristic curve would then have the following shape:
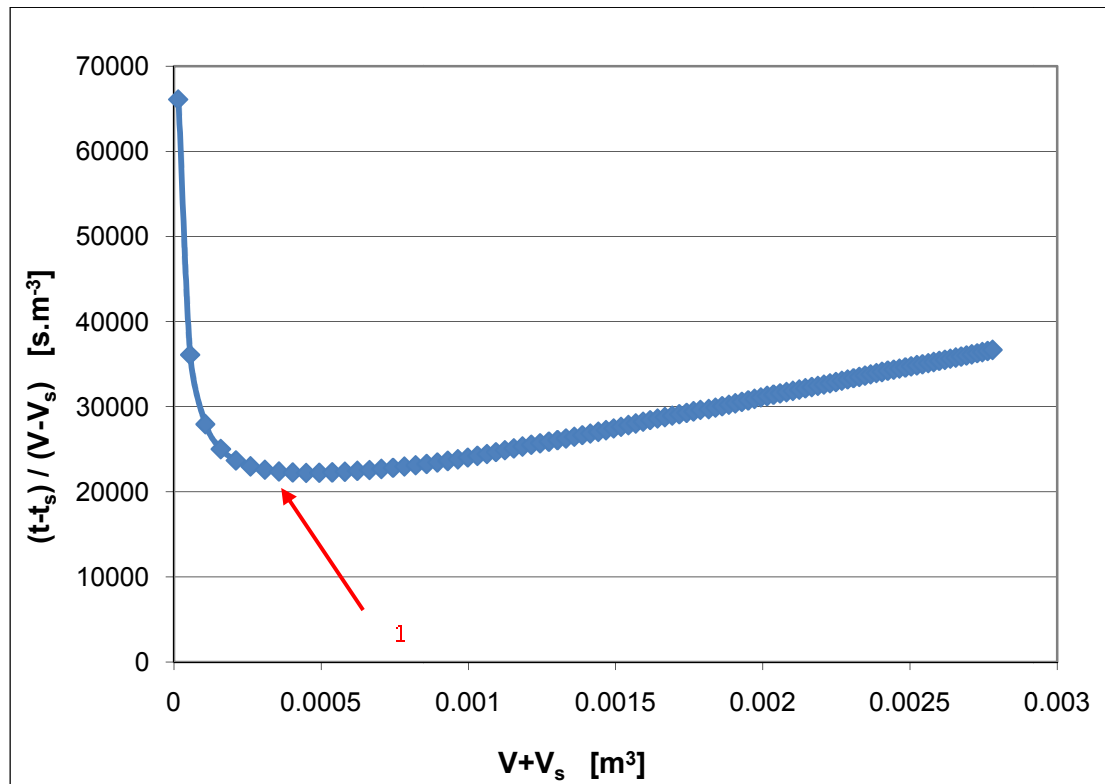
Figure 4-2: Filtration characteristic curve after removing the data from the cake compression stage (Run 61)

Although the steep slope at the end of the filtration was removed, the "dip" at the start of the filtration characteristic curve was still present (Figure 4-2). Palica (1996, page 341) mentioned that the start of cake filtration is non-stationary. He used this to justify why parts of the characteristic curves were not taken into account when determining the filtration constants. Tarleton and Wakeman (2008, page 292) also discussed this type of shape for the filtration characteristic curve. Tarleton and Wakeman mentioned that this "dip" in the data is generally as a result of the prevalence of blocking over bridging during the filtration. Tarleton and Wakeman illustrated that the filtration analysis is only conducted in the linear region of the curve and that the behaviour in the initial region (i.e. the "dip") is neglected. Thus, in order to remove the "dip" present at the start of filtration, the start up time for the filtration would then be moved from the time when the first drop of filtrate was collected (as was used in Figure 4-1 and 4-2) to the time when the $[(t-t_s)/(V-V_s)]$ values began to increase (See Appendix B for how this was done). This time occurs at the point labelled as "1" on Figure 4-2. This method of correcting the t_s was also embarked upon by Tien (2006, pages 178-179). This method was deemed suitable for the

filtration data analysis as its application was not only justified by Tarleton and Wakeman but by Tien as well. Correcting the t_s allowed the analysis to be conducted without having any of the experimental data lost or excluded. Once the start up time was moved to this new point, the characteristic curve had a linear shape and the filtration constants were then determined.

Thus, the final characteristic curve, once removing the cake compression stage and using the corrected start up time was of the form:
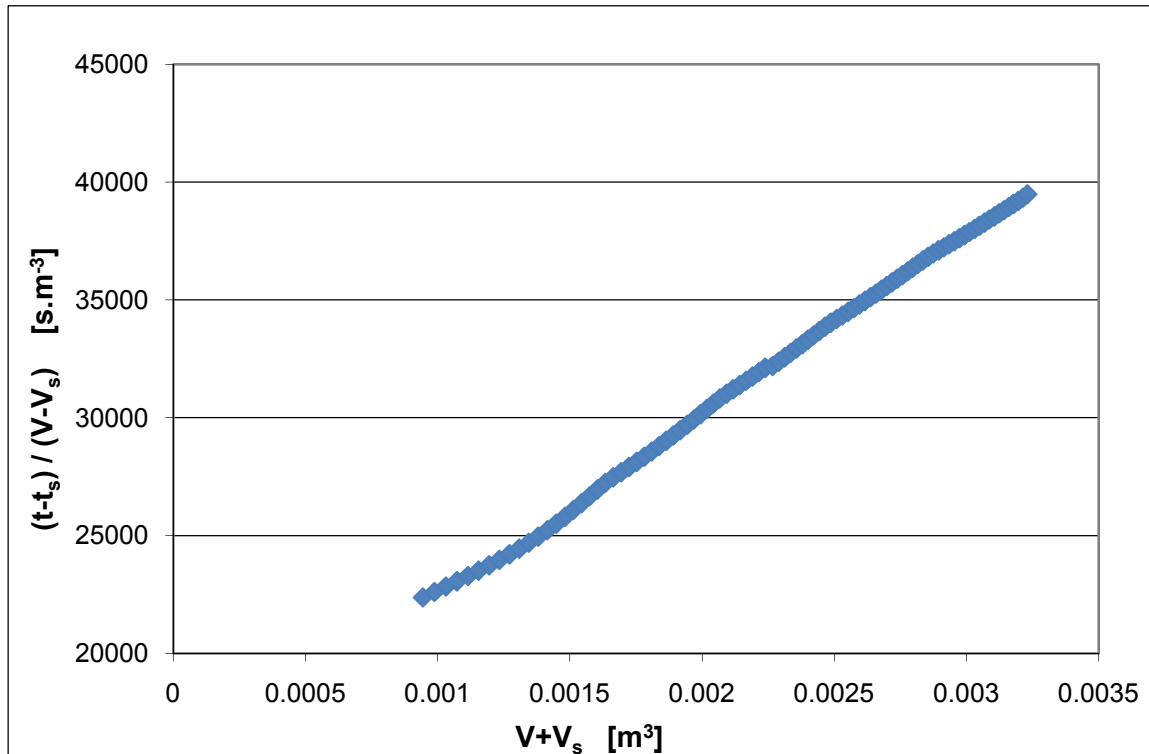
Figure 4-3: Final filtration characteristic curve

Each set of experimental runs (varying concentration, varying pressure and varying filter cloth), produced a characteristic filtration curve, $[(t-t_s)/(V-V_s)]$ vs. $(V+V_s)$, and a cake formation rate curve (the details as to how these were produced can be found in Appendix B). The characteristic filtration curves for these experiments are shown below (Figures 4-4 to 4-18), with a discussion about their behaviour to follow. The cake formation rate curves can be found in Appendix C and are discussed in section 4.2.2. Summaries of the results obtained from the data analysis are shown in Tables 4-1 to 4-12. The details regarding the calculation of the filtration constants (α_{av} and R_m) along with ΔP_c are described in section 2.8 and Appendix B.

Table 4-1: Data summary for the *two-phase* experiments in which *concentration was varied* whilst pressure and filter cloth pore size was fixed (at 2.5 bar abs and 10µm respectively)

Run	Concentration (mass/mass %)	s (kg/kg)	m (kg/kg)	c (kg/m³)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m⁻¹)	ΔP_c (bar)
1	1	0.0111	2.629	11.439	61.96	01:33	12.435	733.4	1.200E+09	1.369E+10	0.211
2	2	0.0196	2.383	20.510	58.04	01:40	11.491	1097.7	1.022E+09	1.323E+10	0.351
3	3	0.0292	2.992	31.971	66.58	01:41	9.517	496.9	9.436E+08	1.329E+10	0.351
4	4	0.0382	2.067	41.448	51.63	01:42	10.333	916.0	6.980E+08	1.385E+10	0.432
5	5	0.0477	3.000	55.506	66.67	01:40	9.443	480.2	6.384E+08	1.464E+10	0.383
6	6	0.0568	3.341	70.037	70.07	01:43	10.091	257.8	5.160E+08	1.503E+10	0.338
7	7	0.0655	3.078	81.840	67.51	01:45	9.669	447.8	5.067E+08	1.500E+10	0.415
8	8	0.0739	2.881	93.747	65.29	01:47	8.231	481.6	5.597E+08	1.439E+10	0.494
9	9	0.0828	2.801	107.526	64.30	01:50	7.576	401.6	5.900E+08	1.424E+10	0.541
10	10	0.0909	2.693	120.080	62.86	01:45	7.680	414.8	5.352E+08	1.484E+10	0.533

Table 4-2: Data summary for the *two-phase repeated* experiments in which *concentration was varied* whilst pressure and filter cloth pore size was fixed (at 2.5 bar abs and 10µm respectively)

Run	Concentration (mass/mass %)	s (kg/kg)	m (kg/kg)	c (kg/m³)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m⁻¹)	ΔP_c (bar)
11	1	0.0100	2.948	10.241	66.08	01:36	12.545	734.1	6.724E+08	1.395E+10	0.110
12	2	0.0199	2.250	20.815	55.56	01:40	11.624	577.5	1.077E+09	1.304E+10	0.305
13	3	0.0294	2.213	31.374	54.81	01:40	10.653	515.2	7.848E+08	1.287E+10	0.319
14	4	0.0385	3.145	43.724	68.20	01:42	10.819	672.8	6.199E+08	1.366E+10	0.350
15	5	0.0477	3.300	56.533	69.70	01:44	9.767	457.2	5.905E+08	1.394E+10	0.361
16	6	0.0567	3.270	69.509	69.42	01:49	8.850	535.0	6.806E+08	1.490E+10	0.458
17	7	0.0654	2.840	80.187	64.79	01:43	9.860	544.2	6.012E+08	1.479E+10	0.446
18	8	0.0741	2.727	92.699	63.33	01:50	8.131	301.5	6.552E+08	1.397E+10	0.511
19	9	0.0826	2.647	105.498	62.22	01:52	7.844	292.3	7.407E+08	1.452E+10	0.558
20	10	0.0909	2.557	118.241	60.89	01:48	8.557	429.9	8.694E+08	1.437E+10	0.682

Table 4-3: Data summary for the *three-phase* experiments in which *concentration was varied* whilst pressure and filter cloth pore size was fixed

(at 2.5 bar abs and 10μm respectively)

Run	Concentration (mass/mass %)	s (kg/kg)	m (kg/kg)	c (kg/m³)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	$m_s(g)$	α_{av} (m/kg)	R_m (m⁻¹)	ΔP_c (bar)
21	1	0.0101	2.836	10.373	64.74	01:37	12.103	523.8	8.810E+08	1.364E+10	0.121
22	2	0.0197	2.291	20.629	56.34	01:40	11.145	434.6	9.176E+08	1.336E+10	0.245
23	3	0.0291	2.106	30.962	52.53	01:45	10.971	673.1	9.888E+08	1.326E+10	0.390
24	4	0.0385	2.207	41.992	54.69	01:43	10.169	929.2	6.662E+08	1.420E+10	0.396
25	5	0.0480	3.221	56.715	68.95	01:49	9.192	605.9	7.712E+08	1.485E+10	0.441
26	6	0.0566	2.718	66.781	63.21	01:43	8.841	546.2	5.827E+08	1.563E+10	0.370
27	7	0.0657	2.839	80.534	64.78	01:45	9.324	604.4	5.097E+08	1.500E+10	0.461
28	8	0.0744	2.310	89.615	56.70	01:46	8.254	411.0	5.132E+08	1.535E+10	0.463
29	9	0.0827	2.469	103.762	59.50	01:59	7.544	325.5	8.099E+08	1.504E+10	0.591
30	10	0.0909	2.519	117.602	60.30	01:49	7.609	242.6	6.172E+08	1.595E+10	0.530

Table 4-4: Data summary for the *three-phase repeated* experiments in which *concentration was varied* whilst pressure and filter cloth pore size was fixed (at 2.5 bar abs and 10μm respectively)

Run	Concentration (mass/mass %)	s (kg/kg)	m (kg/kg)	c (kg/m³)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	$m_s(g)$	α_{av} (m/kg)	R_m (m⁻¹)	ΔP_c (bar)
31	1	0.0099	2.706	10.153	63.04	01:35	12.424	827.1	7.522E+08	1.353E+10	0.117
32	2	0.0197	2.237	20.536	55.29	01:42	11.262	645.2	1.000E+09	1.305E+10	0.298
33	3	0.0292	2.320	31.236	56.89	01:44	10.335	671.9	9.361E+08	1.360E+10	0.365
34	4	0.0385	2.406	42.287	58.44	01:45	9.574	466.7	9.512E+08	1.337E+10	0.432
35	5	0.0477	3.242	56.264	69.16	01:48	9.572	487.2	7.526E+08	1.447E+10	0.412
36	6	0.0569	3.006	68.491	66.73	01:50	9.146	477.6	8.047E+08	1.431E+10	0.502
37	7	0.0655	2.889	80.674	65.38	01:52	8.399	458.0	6.702E+08	1.519E+10	0.472
38	8	0.0742	2.624	91.941	61.90	01:52	7.809	299.7	7.617E+08	1.424E+10	0.549
39	9	0.0826	2.506	103.990	60.10	01:54	7.251	292.1	7.330E+08	1.448E+10	0.571
40	10	0.0912	2.517	118.079	60.28	01:56	7.906	399.8	7.546E+08	1.365E+10	0.677

Table 4-5: Data summary for the *two-phase* experiments in which *pressure was varied* whilst concentration and filter cloth pore size was fixed (at 5% mass/mass and 10µm respectively)

Run	Pressure (bar)	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
41	1.5	0.0483	3.353	57.555	70.18	02:22	2.931	323.0	5.308E+08	8.380E+09	0.165
42	2	0.0477	3.279	56.435	69.50	01:56	6.083	384.9	8.370E+08	1.082E+10	0.369
43	2.5	0.0477	3.000	55.506	66.67	01:40	9.443	480.2	6.384E+08	1.464E+10	0.383

Table 4-6: Data summary for the *two-phase repeated* experiments in which *pressure was varied* whilst concentration and filter cloth pore size was fixed (at 5% mass/mass and 10µm respectively)

Run	Pressure (bar)	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
44	1.5	0.0479	3.367	57.034	70.30	02:27	2.580	303.1	7.921E+08	6.927E+09	0.246
45	2	0.0476	3.280	56.315	69.51	01:56	5.697	454.0	8.045E+08	1.051E+10	0.397
46	2.5	0.0477	3.300	56.533	69.70	01:44	9.767	457.2	5.905E+08	1.394E+10	0.361

Table 4-7: Data summary for the *three-phase* experiments in which *pressure was varied* whilst concentration and filter cloth pore size was fixed (at 5% mass/mass and 10μm respectively)

Run	Pressure (bar)	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
47	1.5	0.0479	3.355	56.912	70.20	02:22	2.629	363.6	5.765E+08	8.405E+09	0.165
48	2	0.0479	3.307	56.826	69.76	01:50	5.818	301.2	7.096E+08	1.076E+10	0.325
49	2.5	0.0480	3.221	56.715	68.95	01:49	9.192	605.9	7.712E+08	1.485E+10	0.441

Table 4-8: Data summary for the *three-phase repeated* experiments in which *pressure was varied* whilst concentration and filter cloth pore size was fixed (at 5% mass/mass and 10μm respectively)

Run	Pressure (bar)	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
50	1.5	0.0476	3.359	56.533	70.23	02:36	2.569	349.5	7.735E+08	7.630E+09	0.209
51	2	0.0478	3.263	56.483	69.36	02:01	5.636	409.2	7.296E+08	1.190E+10	0.303
52	2.5	0.0477	3.242	56.264	69.16	01:48	9.572	487.2	7.526E+08	1.447E+10	0.412

Table 4-9: Data summary for the *two-phase* experiments in which *filter cloth pore size was varied* whilst concentration and pressure was fixed (at 5% mass/mass and 2.5 bar abs respectively)

Run	Cloth	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
53	10µm	0.0479	2.404	54.061	58.41	01:42	9.507	586.8	5.341E+08	1.489E+10	0.343
54	20µm	0.0480	3.254	56.740	69.27	01:41	9.921	545.0	5.526E+08	1.473E+10	0.370

Table 4-10: Data summary for the *two-phase repeated* experiments in which *filter cloth pore size was varied* whilst concentration and pressure was fixed (at 5% mass/mass and 2.5 bar abs respectively)

Run	Cloth	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
55	10µm	0.0477	3.300	56.533	69.70	01:44	9.767	457.2	5.905E+08	1.394E+10	0.361
56	20µm	0.0476	3.240	56.180	69.13	01:47	9.486	444.7	7.717E+08	1.375E+10	0.444

Table 4-11: Data summary for the *three-phase* experiments in which *filter cloth pore size was varied* whilst concentration and pressure was fixed (at 5% mass/mass and 2.5 bar abs respectively)

Run	Cloth	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
57	10µm	0.0476	2.272	53.288	55.98	01:41	9.830	467.4	5.825E+08	1.491E+10	0.343
58	20µm	0.0479	3.244	56.566	69.18	01:41	10.242	657.0	5.818E+08	1.483E+10	0.373

Table 4-12: Data summary for the *three-phase repeated* experiments in which *filter cloth pore size was varied* whilst concentration and pressure was fixed (at 5% mass/mass and 2.5 bar abs respectively)

Run	Cloth	s (kg/kg)	m (kg/kg)	c (kg/m^3)	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s(g)	α_{av} (m/kg)	R_m (m^{-1})	ΔP_c (bar)
59	10µm	0.0477	3.242	56.264	69.16	01:48	9.572	487.2	7.526E+08	1.447E+10	0.412
60	20µm	0.0477	2.987	55.467	66.53	01:47	8.804	327.6	8.759E+08	1.367E+10	0.435

During the three-phase experiments, a point occurred in the experimental run where the slurry level dropped below the level of the sparger. The air from the sparger then pressurized the slurry from above the slurry level, as with two-phase filtration. The time at which the slurry level fell to below the sparger level was recorded and is referred to as t_{2p}. The time at which the filtration moved from three-phase to two-phase filtration (t_{2p}) was used as the end point for the three-phase calculations so as to not include the behaviour of the two-phase transition.

The flow rate of the pressurized air into the system, whether introduced as two-phase pressurizing air or three-phase bubbling air was recorded and used for qualitative comparisons rather than quantitative. The flow rate provided an indication of the resistance offered by the cake, with the runs of higher concentrations having lower flow rates, and those of lower concentrations having higher flow rates. The air flows for the two-phase systems as compared to the three-phase systems were almost similar, indicating similar cake structure in both systems.

Experiments in which the solids concentration was varied and the remaining factors were kept constant:
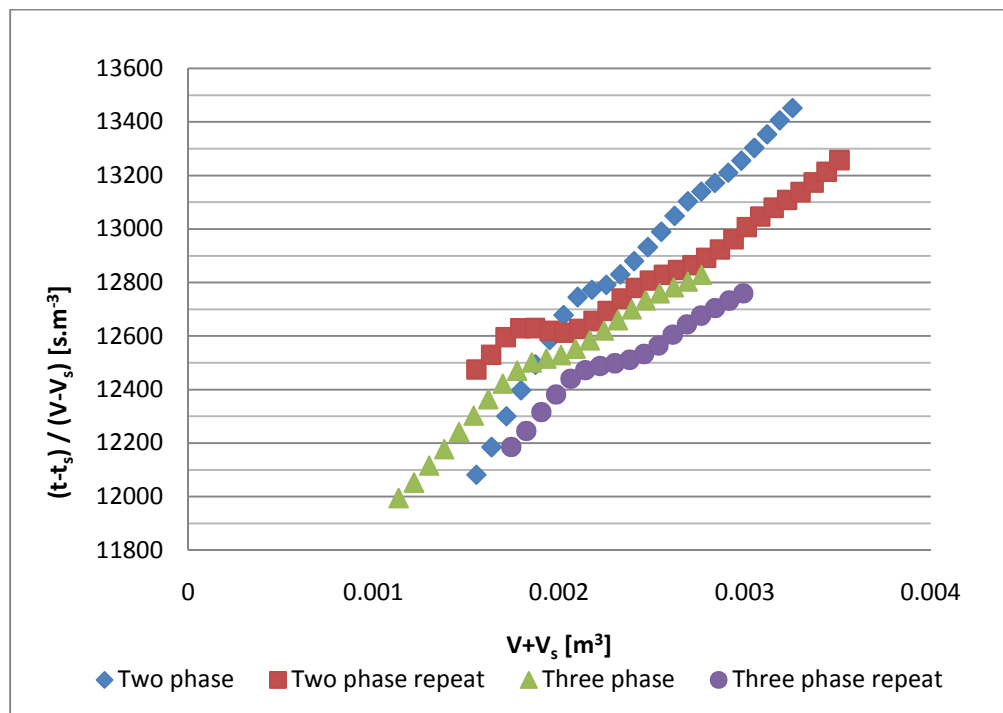
Figure 4-4: Filtration characteristic curve for a solids concentration of 1% (mass/mass) (Runs 1, 11, 21 and 31)
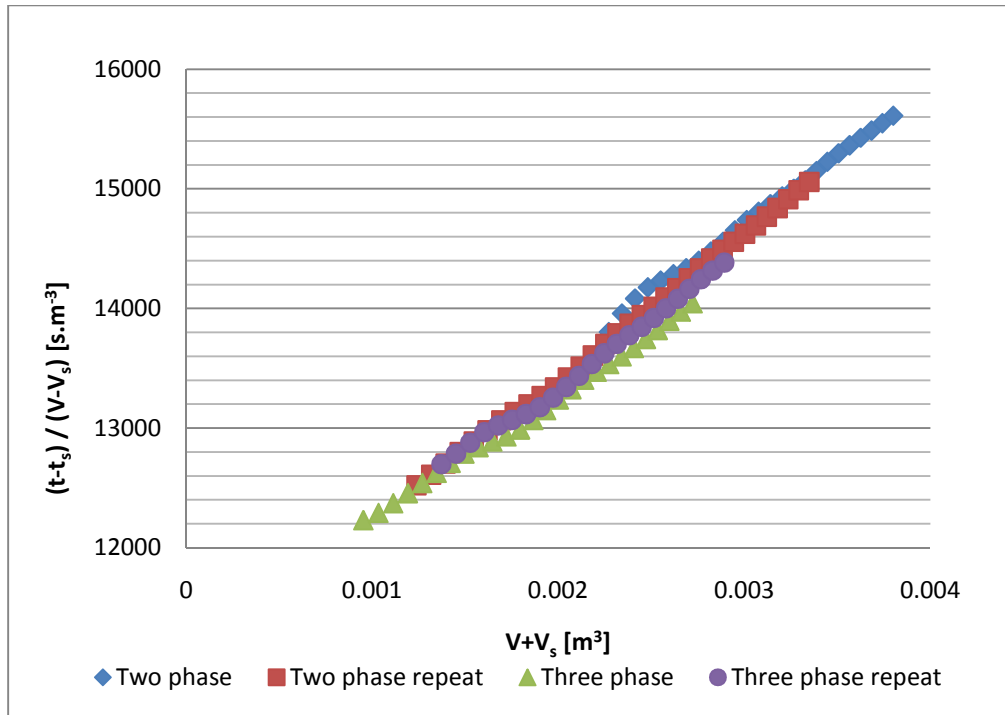
Figure 4-5: Filtration characteristic curve for a solids concentration of 2% (mass/mass) (Runs 2, 12, 22 and 32)
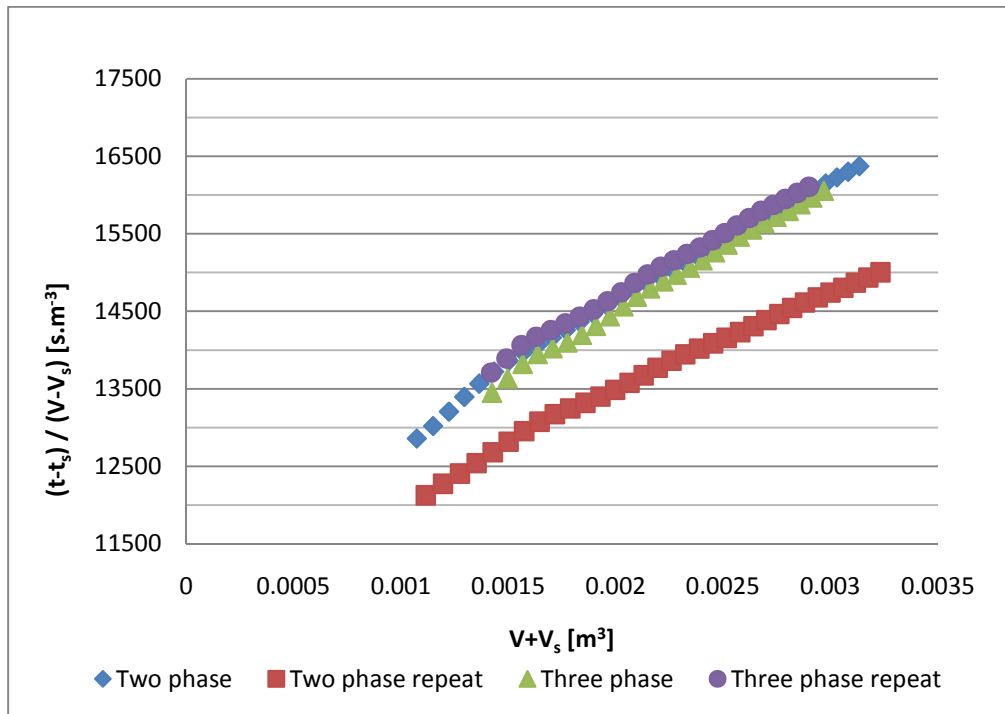
Figure 4-6: Filtration characteristic curve for a solids concentration of 3% (mass/mass) (Runs 3, 13, 23 and 33)
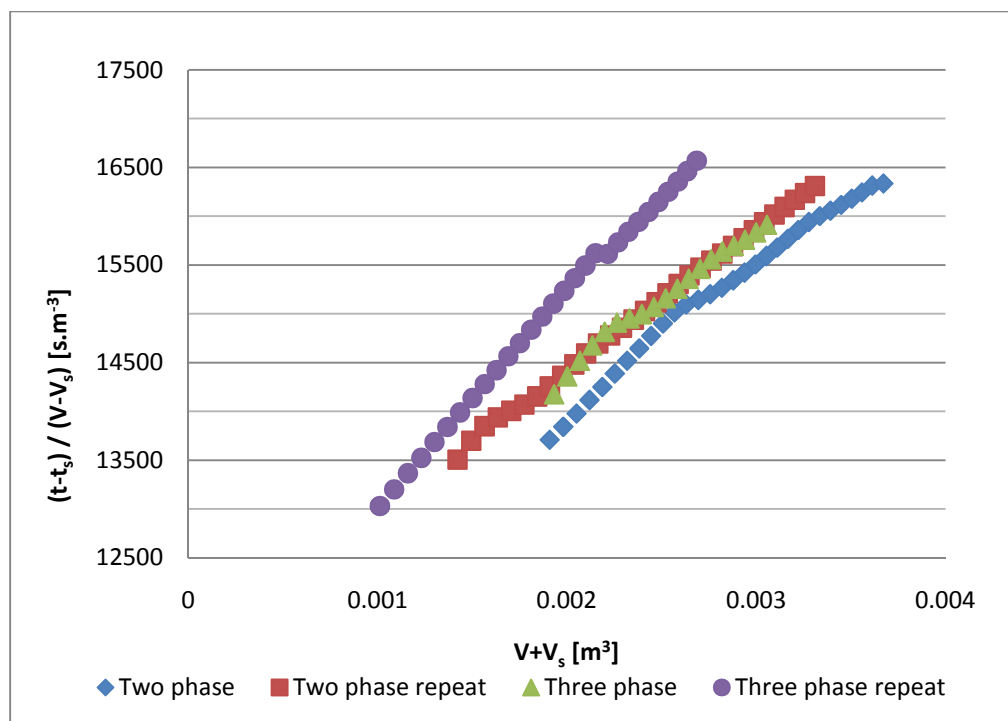
Figure 4-7: Filtration characteristic curve for a solids concentration of 4% (mass/mass) (Runs 4, 14, 24 and 34)
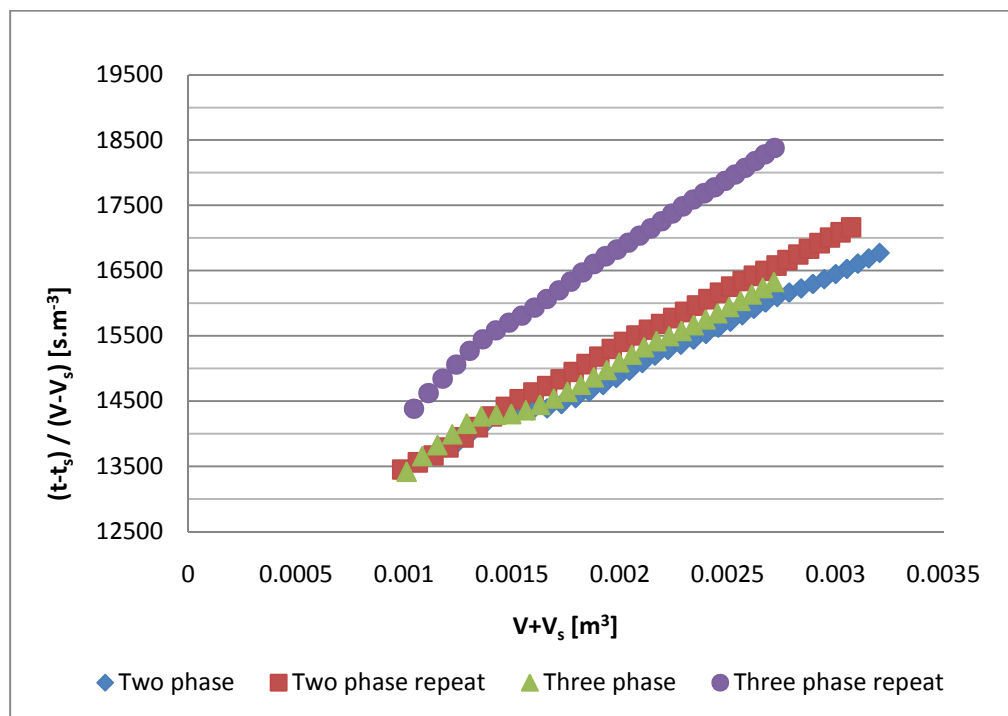
Figure 4-8: Filtration characteristic curve for a solids concentration of 5% (mass/mass) (Runs 5, 15, 25 and 35)
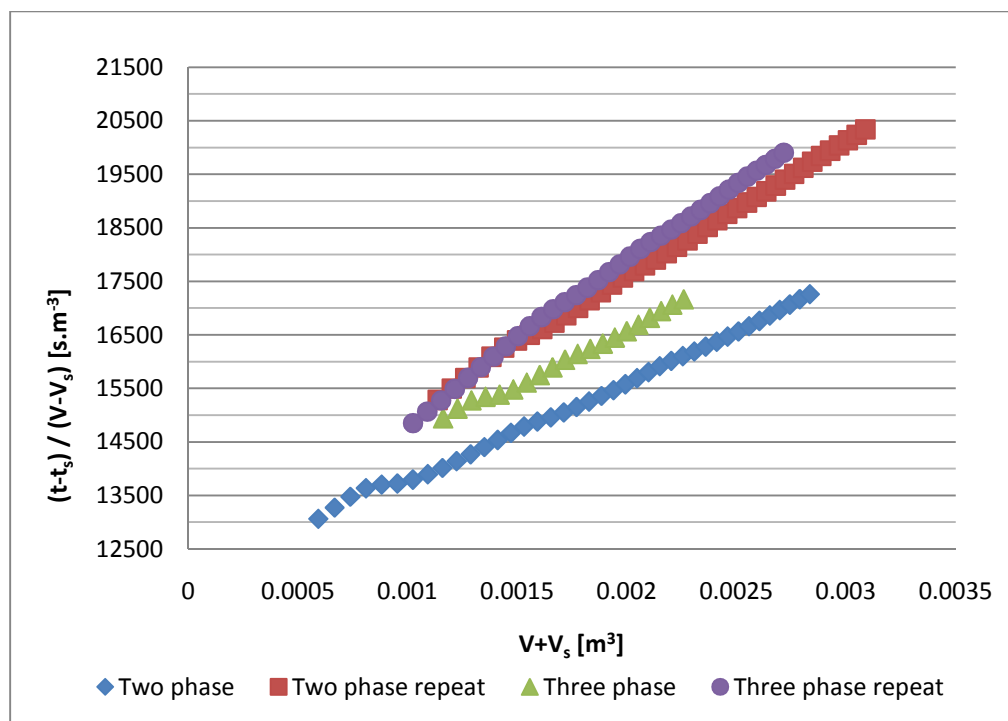
Figure 4-9: Filtration characteristic curve for a solids concentration of 6% (mass/mass) (Runs 6, 16, 26 and 36)
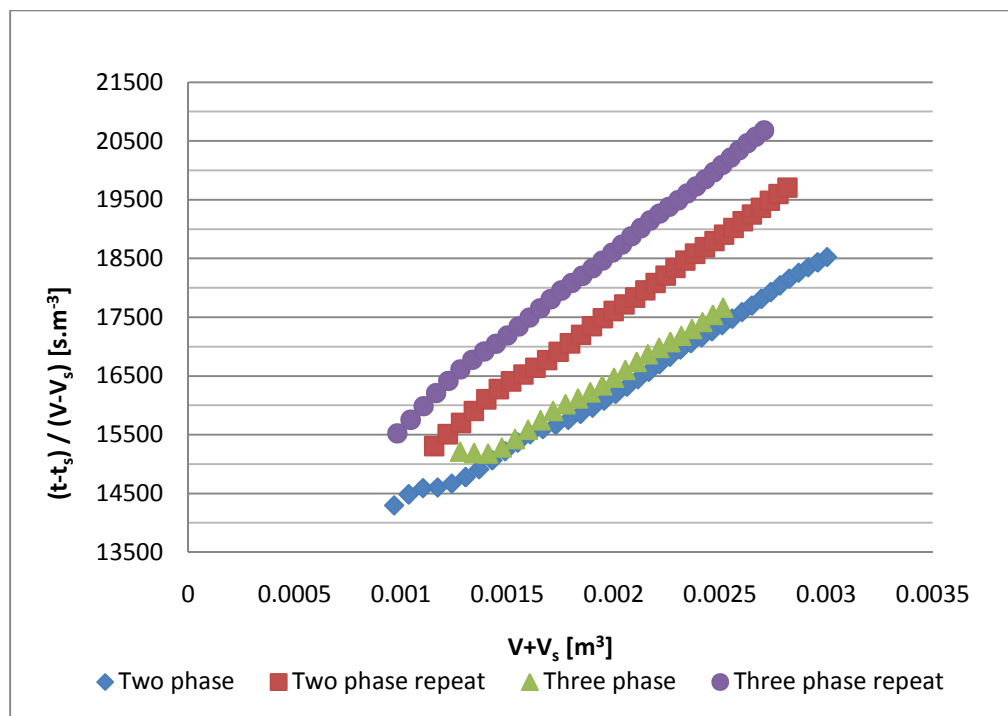
Figure 4-10: Filtration characteristic curve for a solids concentration of 7% (mass/mass) (Runs 7, 17, 27 and 37)
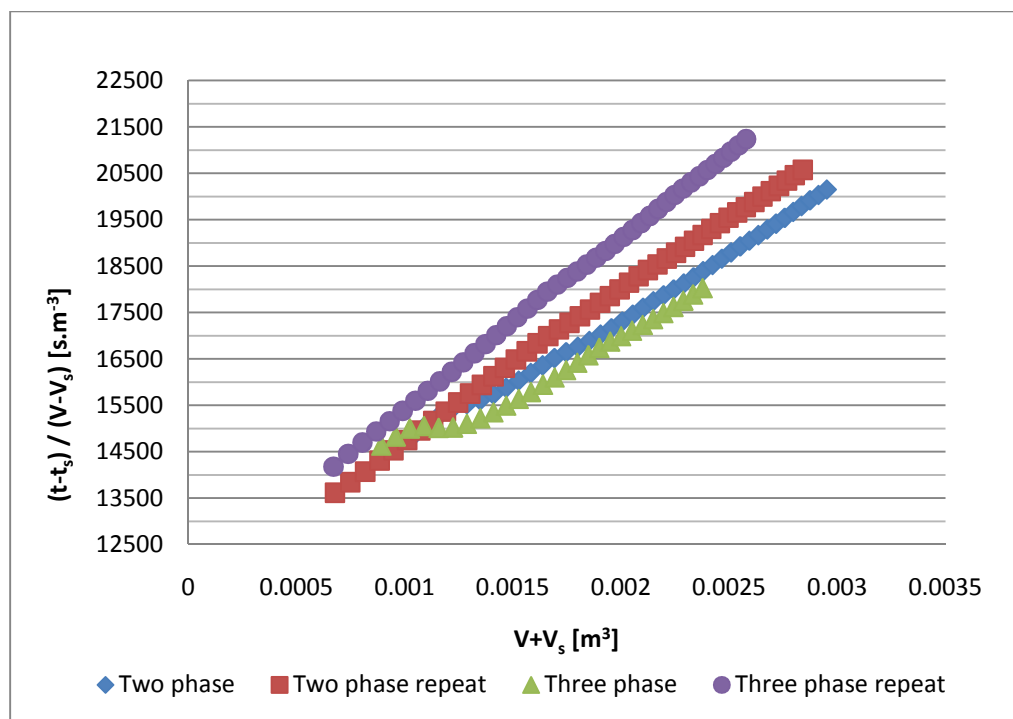
Figure 4-11: Filtration characteristic curve for a solids concentration of 8% (mass/mass) (Runs 8, 18, 28 and 38)
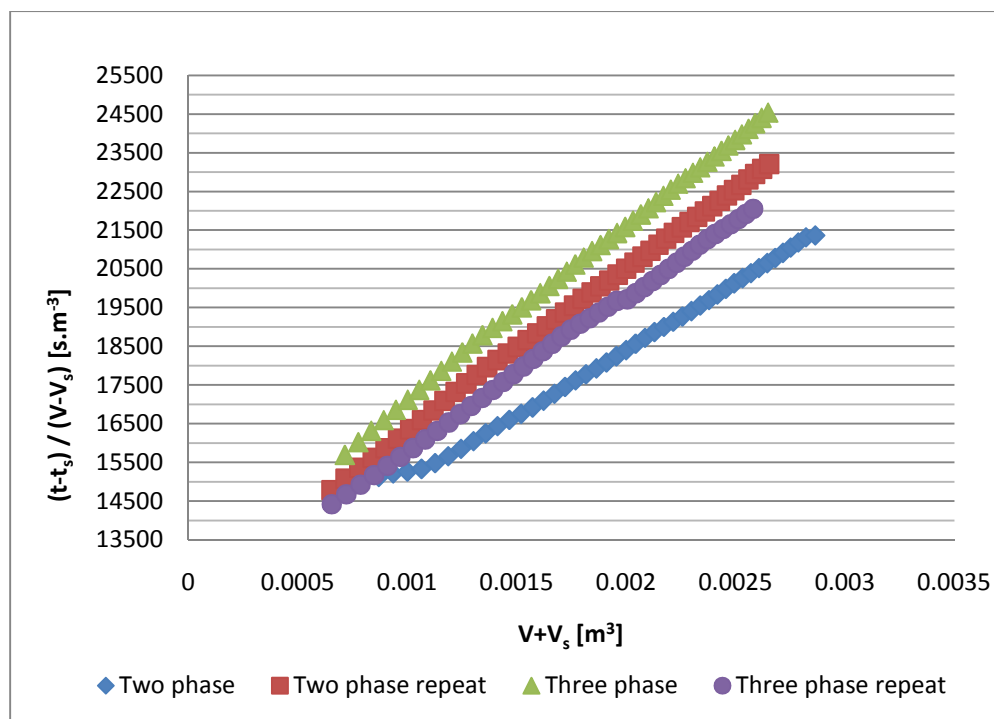
Figure 4-12: Filtration characteristic curve for a solids concentration of 9% (mass/mass) (Runs 9, 19, 29 and 39)
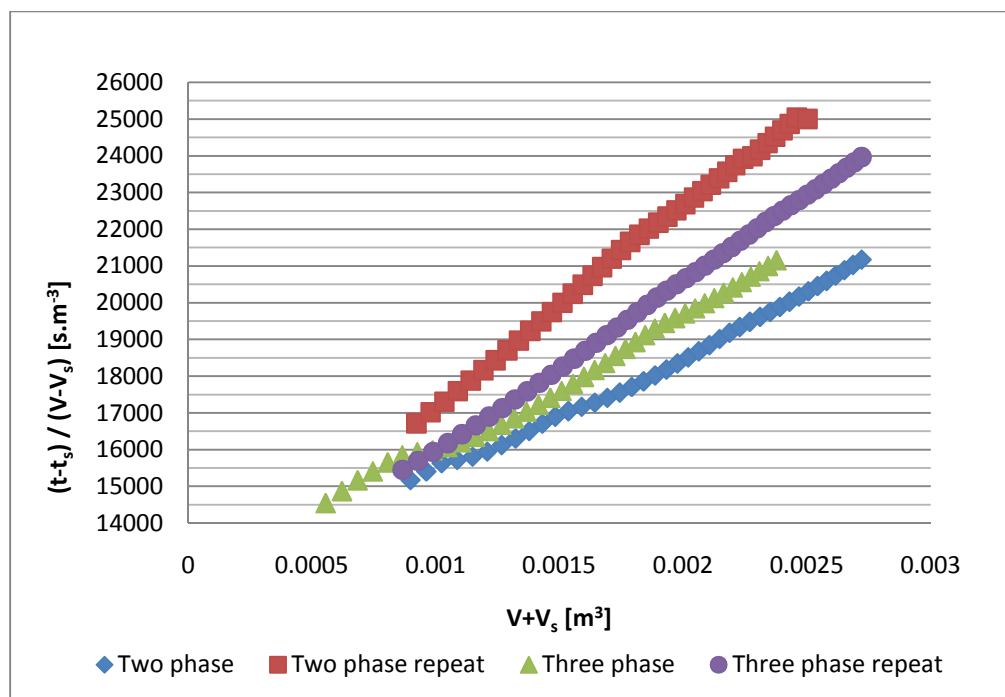
Figure 4-13: Filtration characteristic curve for a solids concentration of 10% (mass/mass) (Runs 10, 20, 30 and 40)

Experiments in which the applied pressure was varied and the remaining factors were kept constant:
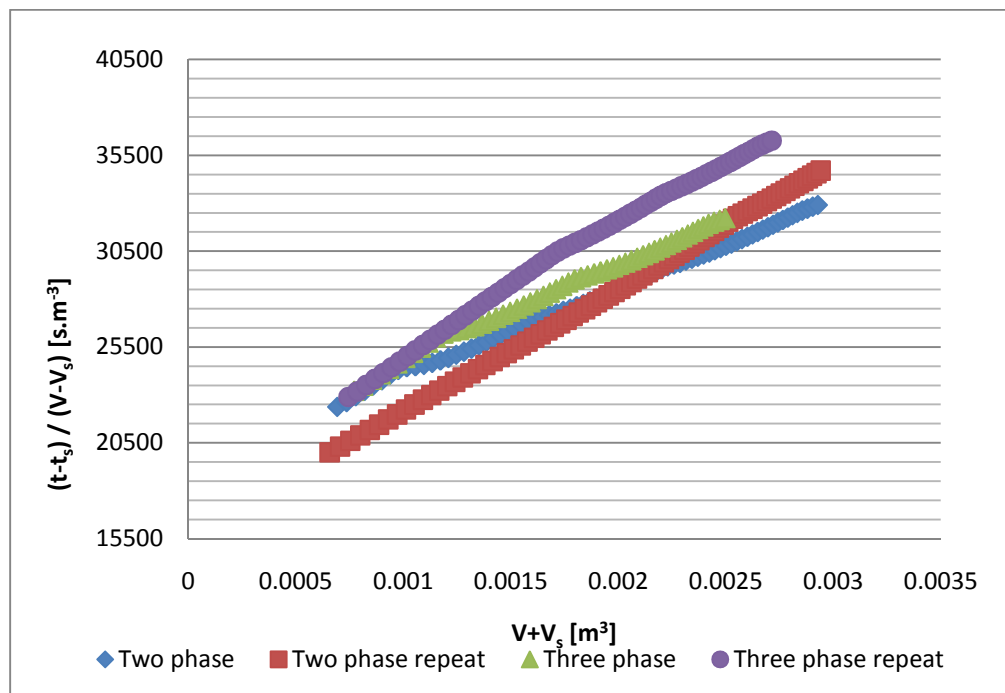
Figure 4-14: Filtration characteristic curve for an applied pressure of 1.5bar abs (Runs 41, 44, 47 and 50)
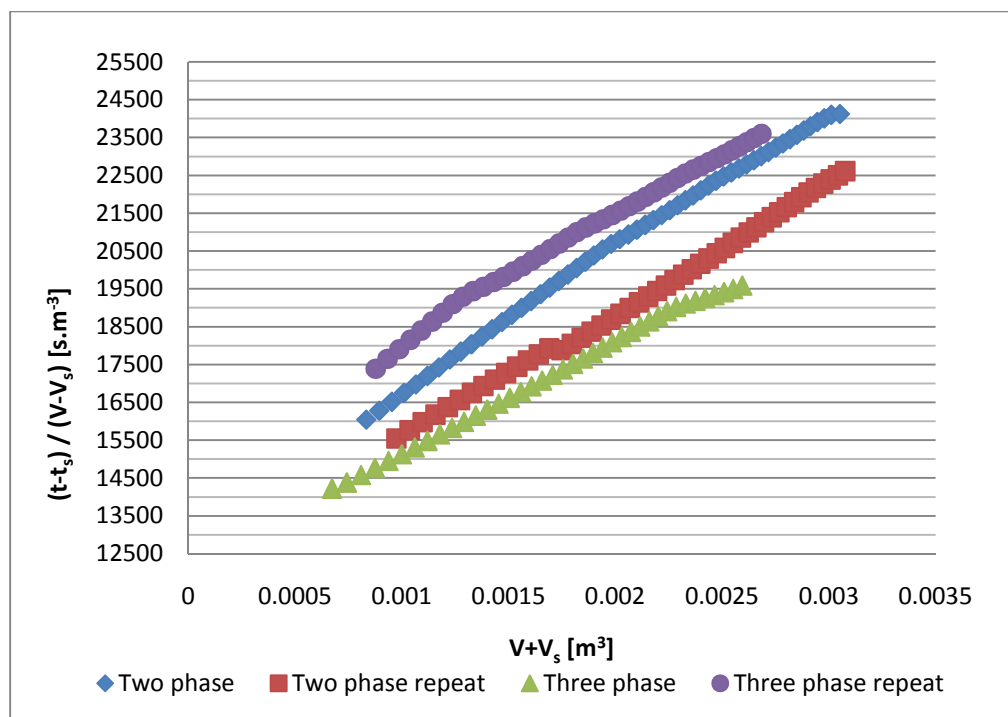
Figure 4-15: Filtration characteristic curve for an applied pressure of 2.0bar abs (Runs 42, 45, 48 and 51)
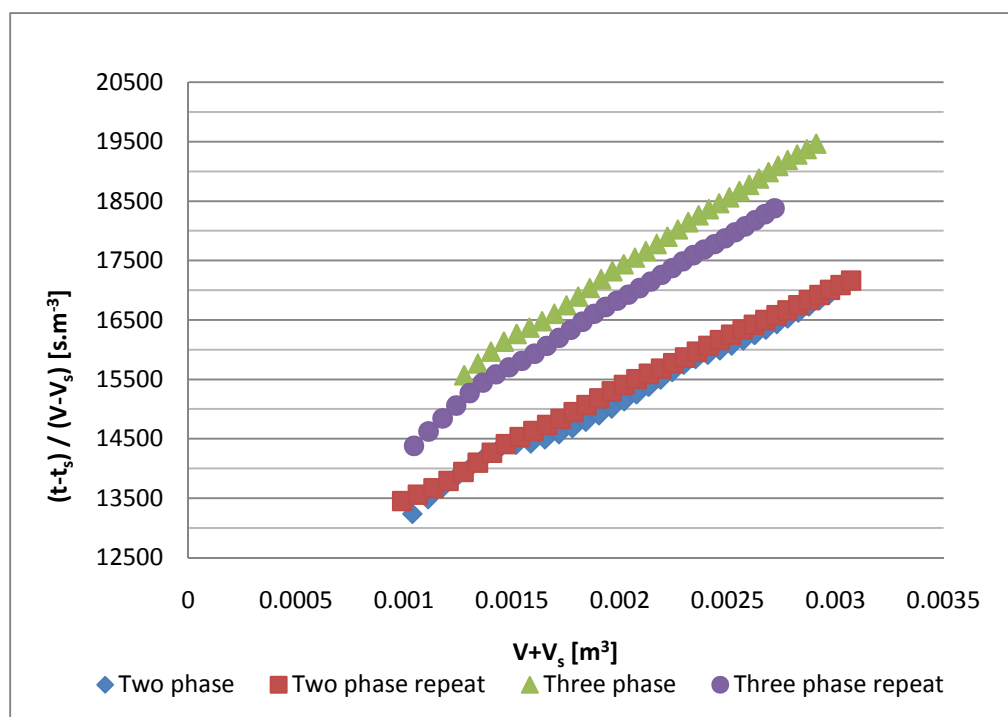
Figure 4-16: Filtration characteristic curve for an applied pressure of 2.5bar abs (Runs 43, 46, 49 and 52)

Experiments in which the filter cloth pore size was varied and the remaining factors were kept constant:
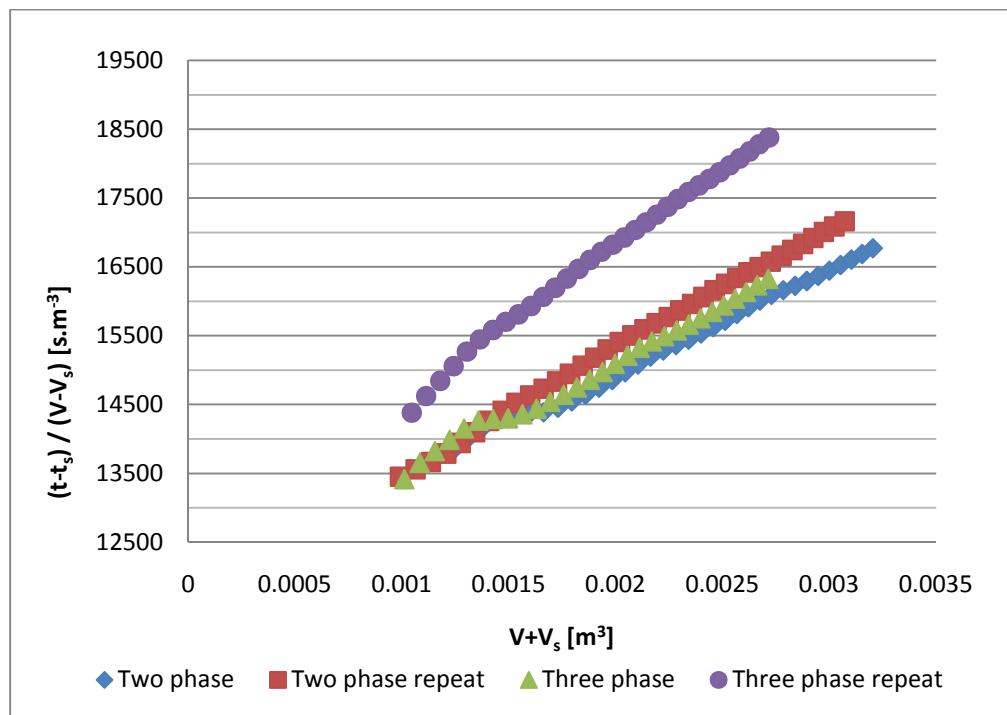
Figure 4-17: Filtration characteristic curve for a filter cloth pore size of 10µm (Runs 53, 55, 57 and 59)
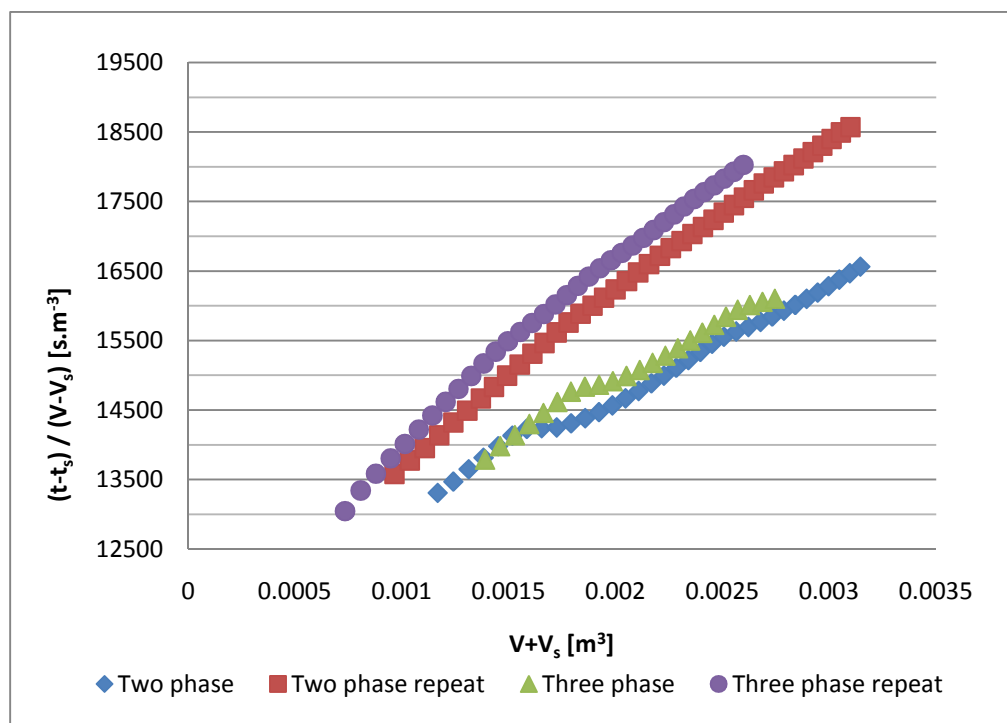
Figure 4-18: Filtration characteristic curve for a filter cloth pore size of 20µm (Runs 54, 56, 58 and 60)

4.2.1.1. Experiments in which the solids concentration was varied

From the data collected and the analysis conducted (Refer to Tables 4-1, 4-2, 4-3 and 4-4), the following observations were drawn:

- Both the two-phase and three-phase experiments produced the cake with the highest moisture content at the 5% solids concentration value. This was also visually confirmed when discharging the filter cake.

- The three-phase experiments tend to have longer filtration times. This is justified by the turbulence created by the bubbling air. The turbulence prevents immediate settling of the solid particles in the slurry, preventing a more rapid cake formation compared with the two-phase experiments.

- For both the two-phase and three-phase experiments, the air flow tends to decrease with increasing concentration even though the applied pressure was fixed for each of these experiments. This can be attributed to the increased cake height with an increase in concentration, resulting in a greater resistance to air flow, thus decreasing the air flow of the applied gaseous phase.

- Another observation made after computing the calculations was that the m_s (mass of filtrate collected that corresponds to the t_s selected) for the runs with a low concentration (Runs 1-4, 11-14, 21-24 and 31-34) was generally much higher than m_s of the runs with a higher concentration (Runs 8-10, 18-20, 28-30 and 38-40). The average m_s for the experiments of higher concentration was ±450g for the two-phase experiments and ±470g for the three-phase experiments. Whereas for the low concentration experiments, values between ±600-1000g were obtained.

- The pressure drop across the cake (ΔP_c) was seen to increase with increasing solids concentration. This is easily explained by the increase in cake height with an increase in solids concentration, providing a greater resistance to suspension flow and thus an increased pressure drop across the cake.

- Figures 4-4 to 4-13 show that the experiments with a lower solids concentration give curves that are not as linear as the curves obtained for experiments with a higher solids concentration.

- The filtration characteristic curves in Figures 4-5 to 4-13 show the general linear behaviour of the curves, as predicted by the filtration theories discussed in Chapter 2.

- Figures 4-4 to 4-13 also illustrates that for each experiment, the two-phase and three-phase tests along with their replicates, all exhibit similar filtration curves. However the experiments at

3, 5 and 6% solids concentration (Figures 4-6, 4-8 and 4-9 respectively) show distinctive deviations in the similarity of the two-phase curves as well as the three-phase curves. This could be further investigated by conducting additional repetitions at these solid concentrations and noting if the behaviour of the filtration characteristic curves for each of these experiments alters.

- The filter medium resistance (R_m) was similar for all the two-phase and three-phase investigations (see Figure 4-19). This is expected as the same type of filter cloth was used for each investigation and its resistance to filtrate flow should thus be similar whenever used.

- It was difficult to find a trend for the behaviour of the specific cake resistance either for the two-phase or three-phase experiments as the α_{av} fluctuated erratically (refer to Figure 4-20). The short cycle duration of each of the experiments which limited the time for the cake conditions to stabilise may explain the erratic behaviour of the α_{av} values obtained (Holdich, 2003). The scattered values of α_{av} could also have been attributed to the unsteady state of the behaviour of the equipment during the initial stages of experimentation.
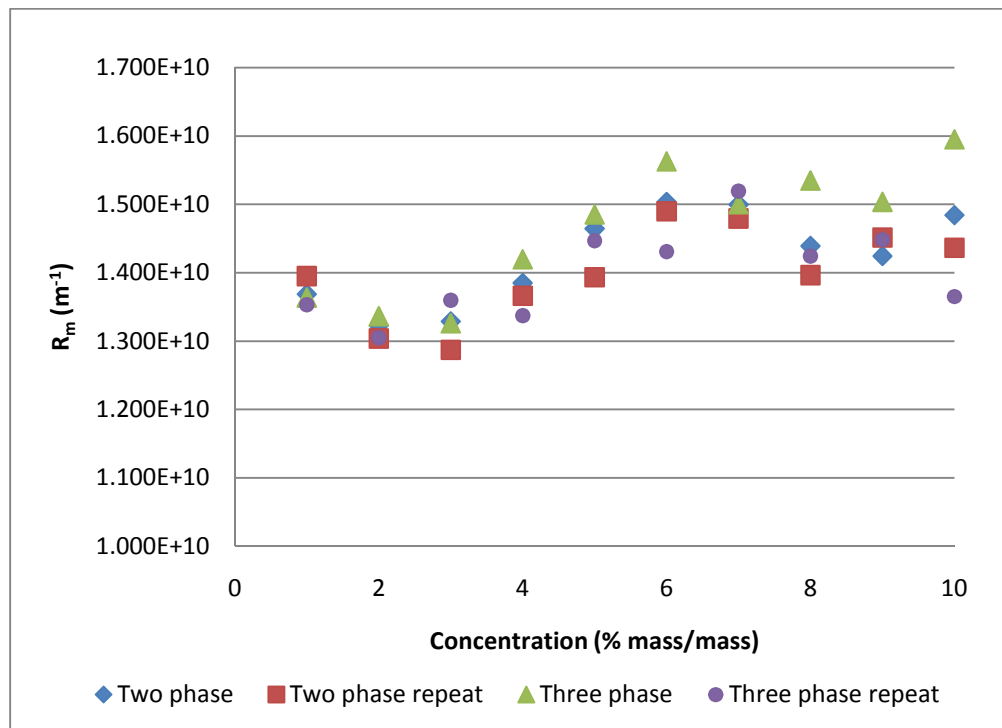
Figure 4-19: Behaviour of the filter medium resistance with changing concentration
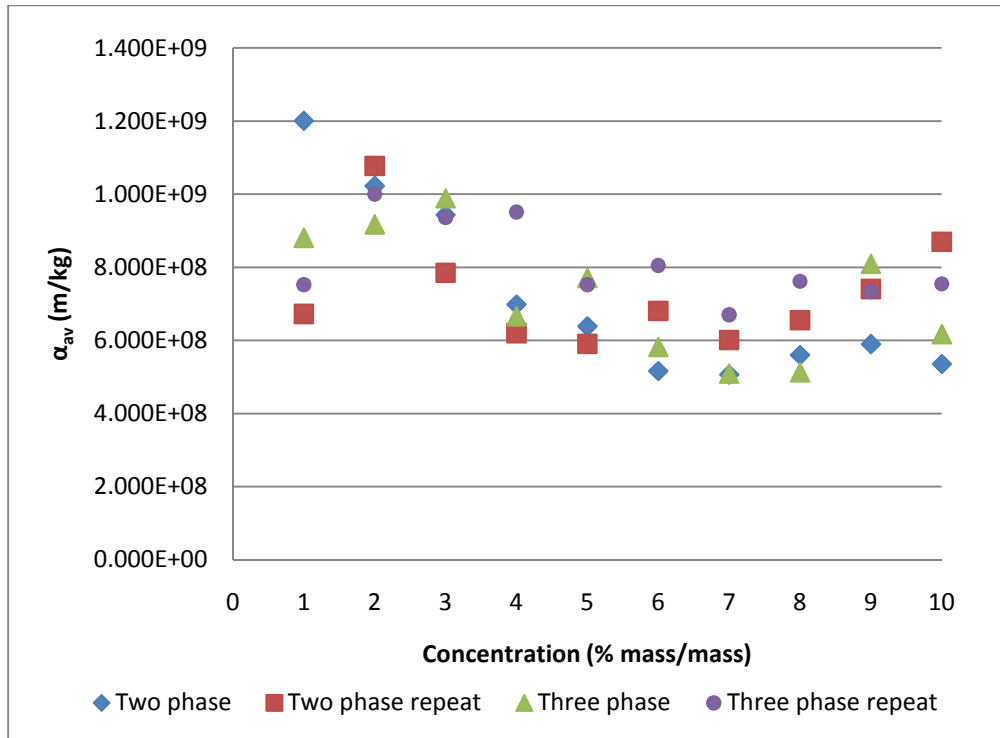
Figure 4-20: Behaviour of the specific cake resistance with changing concentration

4.2.1.2. Experiments in which the applied pressure was varied

Since there were only three pressures that were used for this analysis, definitive observations are difficult to draw. However, a few comments about the behaviour can be made (Refer to Tables 4-5, 4-6, 4-7 and 4-8):

- The lower pressure experiments (1.5bar abs - Runs 41, 44, 47 and 50) produced cakes with a higher moisture content. However, the 2.0bar abs and 2.5bar abs experiments (Runs 42, 43, 45, 46, 48, 49, 51 and 52) produced cakes with similar moisture contents.

- With an increase in pressure, the air flow rate in the system increased resulting in a decrease in the filtration time, as was visually observed during the experimental runs.

- All these experiments were conducted at a concentration 5% and the average m_s found was around 400g, with the exception being the three-phase experiment at 2.5bar (Run 49) which had a value of 605.9g. The repeat experiment for this run (Run 52) did however have a value of 487g.

- The pressure drop across the cake (ΔP_c) generally increased with increasing applied pressure.

- Figure 4-14, which displays the results from the experiments at 1.5bar abs, shows that the filtration curves for the two-phase tests and three-phase tests, along with their replicates are similar, with the same observation noted for an applied pressure of 2.0bar abs (Figure 4-15). However, the experiments conducted at the highest pressure available, being 2.5bar abs, (Figure 4-16) indicates a deviation in the behaviour of the three-phase tests to the two-phase tests. This perhaps indicates a noticeable effect of the introduction of the gaseous phase into the system at higher pressures.

- The resistance of the filter medium (R_m) remained to be more or less similar across all the experiments (see Figure 4-21). This can be expected as the same concentration of solid was used for each experiment along with the same type of filter cloth.

- Once again the values of the specific cake resistance (α_{av}) are difficult to discuss as there is no specific trend occurring (see Figure 4-22). However, the values for the three-phase tests are similar but this does not occur for the two-phase experiments. The inclusion of more pressure levels may have allowed for a more descriptive analysis to be conducted. As discussed in section 4.2.1.1, the unsteady state of the equipment during initial stages of operation and short filtration cycle times may have contributed to the inability to draw any definitive conclusions regarding the behaviour of the specific cake resistance.
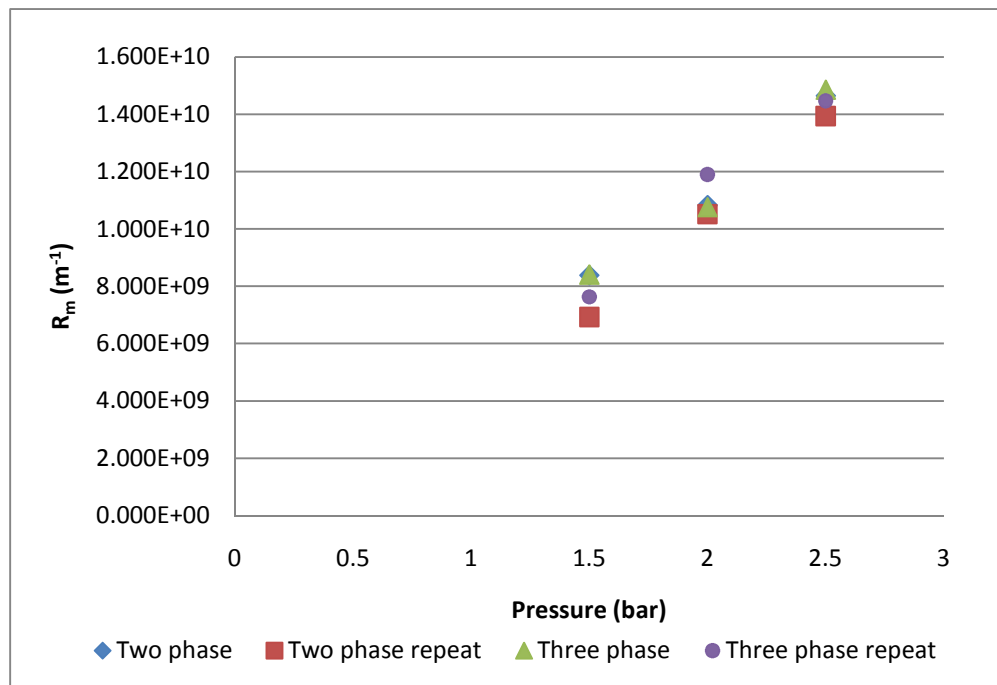
Figure 4-21: Behaviour of the filter medium resistance with changing pressure
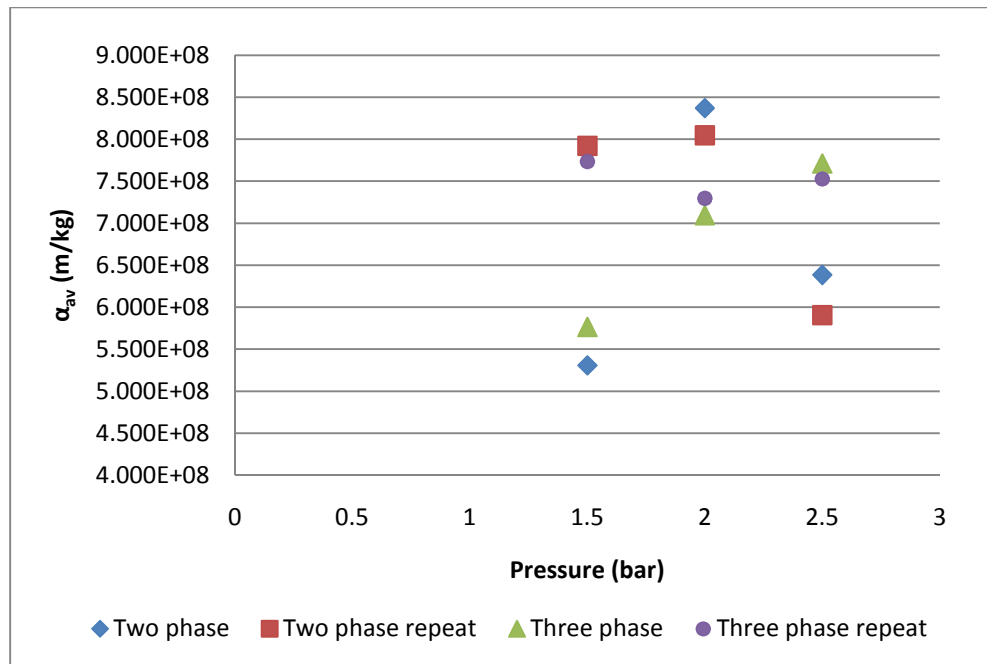
Figure 4-22: Behaviour of the specific cake resistance with changing pressure

4.2.1.3. Experiments in which the filter cloth pore size was varied

With only two types of cloth available, trends are hard to establish and behaviour hard to predict. However, the following was noted (Refer to Tables 4-9, 4-10, 4-11 and 4-12):

- The two-phase experiments (Runs 53 and 54) produced a cake with a higher moisture content when using the 20μm filter cloth. However, upon repeating these experiments (Runs 55 and 56), the values for the moisture content of the cake for the 20μm and 10μm cloths were similar. The same type of behaviour occurred for the three-phase experiments (Runs 57 and 58) and the repeat tests for these runs (Runs 59 and 60).

- The experiments (Runs 53-60) were exposed to the same applied pressure (2.5 bar abs) and solids concentration (5%) which would contribute to the similar filtration times obtained. The identical concentration levels used for the experiments also indicated similar cakes for each experiment. The similar cake heights implied that similar levels of resistance to filtrate flow were experienced. This can be used to explain why all the experiments had similar air flow rates.

- The m_s remained at values approximately in the 400g range as were obtained in previous 5% solid concentration experiments (see experiments 5, 15, 25 and 35 in Tables 4-1, 4-2, 4-3 and 4-4).

- The pressure drop across the cake (ΔP_c) increased when the 20μm filter cloths were used.

- The filtration characteristic curves for these experiments (Figures 4-17 and 4-18) did not indicate any definitive effects of using filter media of different aperture sizes. This is clearly shown by the deviations from the curves of the replicates to that of their originals.

- Little deviation between the 10μm and 20μm filter cloth occurred for the calculated values for the resistance of the medium (R_m) for the two-phase and three-phase experiments (runs 53-54 and 57-58). However, once these experiments were repeated (runs 55-56 and 59-60) the calculated values for R_m differed when using the 10μm and 20μm filter cloths (see Figure 4-23).

- Whereas, an increase in the specific resistance of the filter cake (α_{av}) was seen with the larger pore size cloth, the exception to this was the original tests for the three-phase system (Runs 57 and 58 in Table 4-11) but altered when the repeat tests (Runs 59 and 60 in Table 4-12) were conducted (see Figure 4-24).

- It is difficult to justify why the R_m and α_{av} values behaved in this manner. As mentioned previously in sections 4.2.1.1 and 4.2.1.2, the unsteady state of the initial operation of the test rig may have contributed to the results obtained. The short cycle filtration times may also have been influential on the behaviour observed. With only two types of filter cloth available, trends were hard to establish. Using additional cloths of varying pore sizes may allow for more precise conclusions to be drawn.
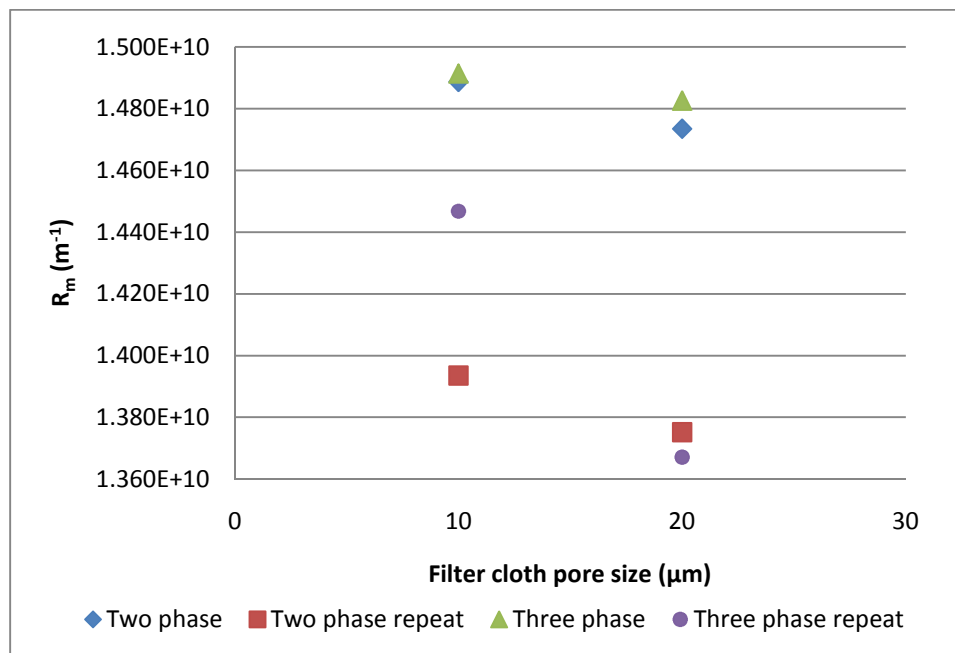
Figure 4-23: Behaviour of the filter medium resistance with changing filter cloth pore size
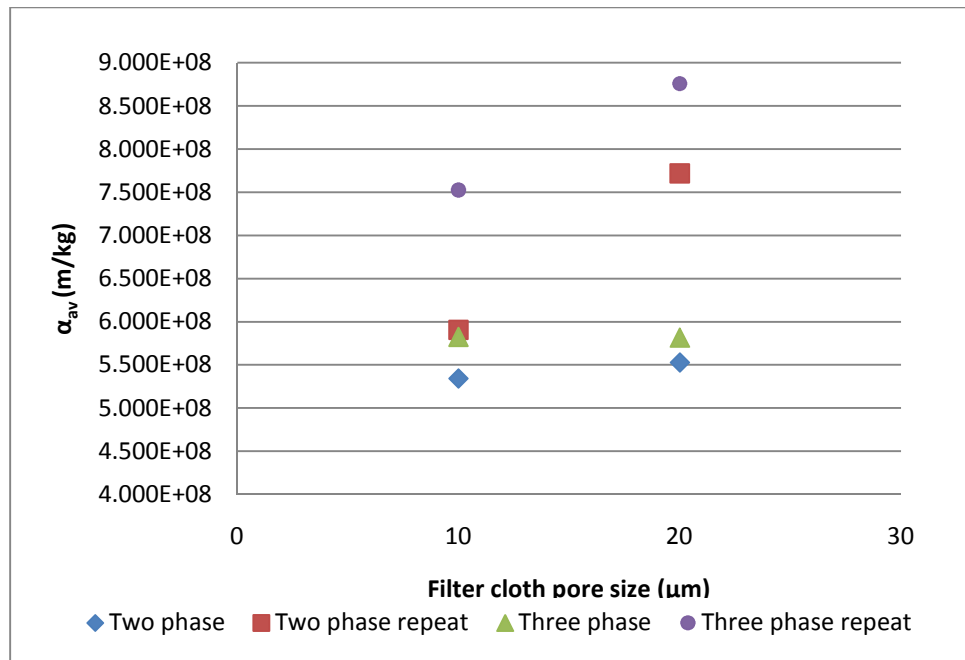
Figure 4-24: Behaviour of the filter medium resistance with changing filter cloth pore size

4.2.1.4. Overall discussion for each set of experiments

Tarleton (1998b) mentioned that generating reliable and repeatable experimental data are pre-requisites to determining the operating behaviour of any system.

Thus, all the experiments described above were conducted twice for this reason. Variations in the produced results of specific cake resistance and filter medium resistance between the original and repeatability tests for the two-phase and three-phase experiments are discussed below:

- For the two-phase tests:

    • Effect of the solids concentration in the feed:

    The difference between the α_{av} values for the original and repeated experiments ranged from 5.19%-61.27%, with α_{av} values. The greatest difference between the original and repeated experiments occurred at the experiments with lower concentrations. The difference the R_m values had a much smaller range with the difference ranging between 0.90%-4.96%.

    • Effect of the applied pressure:

    The difference in the α_{av} ranged between 3.96%-41.10% with the greatest difference occurring at the experiments at a pressure 1.5bar abs. The difference in the R_m ranged

between 2.91%-19.15%, with the highest difference occurring at the experiments at a pressure 1.5bar abs.

- • Effect of the filter cloth pore size:

  The difference in the α_{av} ranged between 10.05%-34.03% and R_m between 6.61% and 9.91%.

- For the three-phase tests:

  - • Effect of solids concentration in the feed:

    The difference in α_{av} ranged between 2.44%-36.37%and 0.78%-15.64% for R_m.

  - • Effect of the applied pressure:

    The range of difference for α_{av} was between 2.44%-29.82% and 2.63%-10.11% for R_m. The highest differences were noted at the lowest pressure, as with the two-phase experiments in which pressure was varied.

  - • Effect of the filter cloth pore size:

    The difference in α_{av} ranged between 25.89%-42.07% and 3.04%-8.12% for R_m.

It can be seen that some of the experiments when repeated, did not produce similar results with large error ranges. This could be as a result of numerous factors, Wakeman and Tarleton (1999a, pages 9-10) mention that small changes in the particle size and porosity of the cake leads to substantial changes in the specific average cake resistance when compared across numerous experiments. An alternative explanation is provided by Kantarci, et al., (2004, page 2270), the authors explained this irregularity to be as a result of the usage of tap water as the liquid in the system. The authors stated that the use of tap water in air-water two-phase studies showed significant reproducibility problems. This could be attributed to the substances present in regular tap water.

The deviations between the resistance of the filter medium (R_m) for the original and the repeated experiments is discussed by Tien (2006). The author says that this is attributed to medium clogging which can occur in two ways: interior clogging and surface clogging. Interior clogging occurs when the suspended particles are not of a uniform size, the very fine particles then penetrate into the filter medium and become deposited there. However, surface clogging occurs when the solid particles block or partially block various pore entrances of the filter medium. In either of the scenarios, the effective filter medium is increased, and can thus contribute to the inability to obtain highly accurate repeatable results. Attempts were made to determine the particle size distribution for the diatomaceous earth solid

used. This proved to be very difficult due to the large quantity of solid that was on hand for use in all the experiments, around 50kg. Particle size analysis was done by using several samples and a series of sieves. The samples were sieved in order to provide a rough idea of the grade of the diatomaceous earth. The sieves were of sizes 180μm, 125μm, 75μm and -75μm. The solids were distributed throughout the sieve size range but were concentrated in the 125μm sieve. This indicated that the particle sizes were indeed small and the theory of clogging may hold for the filter cloths used (10μm and 20μm).

Palica (1996, page 341) discussed that the beginning of cake formation is non-stationary. He used this to justify the reason why he excluded the initial stages of filtration when calculating the filtration constants. Wakeman and Tarleton (1999a, page 71) further confirmed this behaviour by stating that at the start of filtration the cake is thin and the main part of the total pressure drop is over the filter medium. The authors state that with ongoing filtration, the cake height grows and the total pressure drop is now distributed over the cake, allowing for the cake resistance to dominate over the filter medium resistance.

These justifications can be used to explain the non-linearity of the filtration curves at the start of filtration and the need to correct the t_s as indicated by Figure 4-2.

The authors' observations can also be used to explain why the t_s of the experiments of lower solids concentration would have to be moved to a time closer to the end of the filtration than for the tests with higher solids concentration. The experiments having a low solids concentration had very short filtration times, on average being 1.5 minutes. The short filtration times thus, did not allow for sufficient time for the cake formation behaviour to stabilize.

The larger t_s and thus m_s for the experiments with lower concentration can also be attributed to the small cake height obtained in these experiments. These small cake heights are within the range of 1.3mm and 1.8mm, and thus do not allow for sufficient time for the cake resistance to dominate over the filter medium resistance. Thus, the cake resistance is only found to dominate much later on in the filtration, explaining the larger t_s and larger m_s values obtained (Tables 4-1, 4-2, 4-3 and 4-4). The larger t_s resulted in a much smaller section of data points being available to construct the filtration characteristic curve (Figure 4-4) and determine the filtration constants. These observations were also mentioned by Tien (2006).

The experiments with low concentrations also exhibited erratic filtration curves (Figure 4-4) and were repeated up to 5 times but still produced the same result. The reason for this could perhaps be explained by the findings of Holdich (2003, page 78). He mentioned that although a constant pressure is applied to the system, the pressure forming the filter cake will vary, as the flow rate through the cake and medium varies. He suggested that since the filtration constants are dependent on pressure, they too will vary with the fluctuation of pressure during an experiment. Holdich found that the filtration in any system can essentially be divided into the initial stage when the pressure drop is across the medium, with the pressure drop across the cake being zero, and the second stage being the end of the filtration cycle when the pressure drop is across the cake, with this stage having a large value for the pressure drop across the cake. Holdich stated that this fluctuation is most significant in filtration cycles of a short duration and was due to the limited amount of time for the cake conditions to stabilize in short cycle filtrations. Most of the filtration tests in this investigation did not last longer than 3 minutes and consequently Holdich's explanation can be applied to the observations in this study. The fluctuation in pressure, which affects the filtration constants, could be linked to the erratic filtration curves that were occasionally obtained, especially for the experiments at lower concentration, given that they had rather short filtration times, some as fast as 1.5 minutes.

Palica (1996, page 337) also mentioned that deviations from straight line behaviour were likely to occur at low pressures. Palica used pressures within the range of 0.27-2.2MPa, whereas during this investigation the pressures used were within the range of 1.5-2.5 bar abs. Thus, the pressures available for this investigation were all relatively low as compared to those used by Palica. He justifies this non-linear behaviour to be as a result of the pore-plugging effect within the filter cloth which superimposes with sedimentation effects within the filtration column. This could also explain the deviations from linearity for some of the experiments. Palica did mention that for almost all of the experiments he has conducted and researched, an increase in filtration pressure would result in a disappearance of the pore-plugging effect.

4.2.2. Cake formation rate curves

The cake formation rate curves for the two-phase experiments were all plotted from the corrected t_s until the beginning of the cake compression stage whereas the cake formation rate curves for the three-phase experiments were graphed until the time that the three-phase behaviour transitioned into two-phase behaviour (t_{2p}) so as not to include the transition behaviour.

The cake formation rate graphs (Appendix C, Figures C-1 to C-15) showed the change in the growth rate of the cake height with time. Wakeman and Tarleton (1999a, page 82) mentioned that for constant pressure filtration processes, the rate of cake formation decreases with time, with these experiments having a maximum rate at the start of filtration. This is confirmed by Figures C-1 to C-15.

The time before the corrected t_s, was excluded from this calculation as Palica (1996, page 341) mentions that the cake formation at the start of filtration is non-stationary. The compression stage of the process was also excluded as no further cake growth occurred during this period.

Most graphs indicate that the two-phase and three-phase experiments display similar cake formation rates, with the cake formation rate for the three-phase experiments generally being slower. This can be explained by the fact that the filter cake does not form as readily in the three-phase system as it does in the two-phase system due to the constant turbulence present.

The "dips" present in all the graphs at different time intervals during the course of an experiment can be justified as occurring at the time which the electronic controller closes Valve V2 (Figure 3-1) which is used to regulate the pressure to remain at the specified set-point. This decrease in the formation rate occurs as a result of the decrease in the pressure present within the column that is used to force the filtration of the slurry, decreasing the slurry volume within the column and thus increasing the cake height simultaneously. Once this valve is then opened by the electronic controller, the cake formation rate increases until the next dip occurs, which is once again when the controller cuts off the supply of pressurized air to the column.

4.3. Experimental design for statistical analysis

Although the initial experiments did provide a rough indication about how the different factors play a role in the experimental result, no specific conclusions could be drawn about the distinctive effects of certain parameters. Montgomery and Runger (1999, page 626) states that conducting factorial experiments is favoured over the method of varying one factor at a time, as it requires a minimal amount of experiments to be conducted in order to provide information regarding the effects of a factor. The author also further justified conducting factorial experiments over the variation of a single factor at a time, as the statistical method is able to provide information about the interaction of factors. By conducting a statistical design of experiments, a logical design of the experiments to be conducted is provided which can be used to determine the effects of factors and their combinations on the output of an experiment.

Initially it was decided that four factors would be varied for the statistical analysis and each of their effects on the system would be determined. Each factor would have a high and a low level, as required for factorial experimental design (See section 2.10.2). The parameters chosen were:

- Pressure
- Solids concentration
- Filter cloth pore size
- Solid material used to create the slurry

The levels of all the parameters were easily determined apart from the alternative solid material to be used. In order to decide what material could be used, the research of others was consulted. Tarleton (2008) worked with a talc and water slurry and Holdich (2003) provided information on systems that used kaolin and water suspensions. Literature values for the filtration constants for a talc-water system could easily be obtained from electronic databases such as "www.filtration-and-separation.com" (discussed by Holdich, 2003) and would thus be used as the initial alternative solid. However, this was realised to be an unfit choice during an experimental run. Talc is a very fine, light material that does not settle rapidly. When a slurry mixture of talc and water was filled into the filtration cell and the necessary pressure applied, the filtration occurred at an extremely slow rate. The filtrate collected as droplets rather than as a rapid flow as with the case of the diatomaceous earth system. The filtration time was observed to increase to over 5 hours for a single experiment. Upon further research, it was realised that

talc systems were normally used in systems that reached much higher pressures of approximately 600kPa (Tarleton, 1998b). These higher pressures resulted in the filtration rate being much higher, preventing the scenario experienced in this system. It was then decided that this was not feasible for continued use in the current system due to the low pressure values that were available. It was then decided that an alternative material would be tested.

The next material type that was tested was the silica powder. This was also deemed to be unfit for use as the silica settled too rapidly in the filtration column. The high density of the silica resulted in it settling even before the experiment began. This sedimentation would alter the cake forming ability and provide erroneous results about the cake properties and filtration constants. The high density of the silica also limited the ability of the bubbling three-phase air to create the turbulence and buoyancy needed for this investigation.

A final selection of kaolin as the alternative solid was used but this too proved unsuitable. The kaolin particles completely blinded the filter medium and the filtrate collected in droplets, as with the talc slurry. The resistance of the kaolin cake was so large that this filtration took more than 5 hours to complete. The kaolin filter cake could not be discharged with ease and would have to be forcibly scraped of the cloth since the particles would adhere to each other and the medium so strongly, due to the fine size of the particles. Although attempts were made to obtain the kaolin in a coarser particle size grade, manufacturers only had the fine grade of particles sizes available for commercial use.

Thus, the ability to vary the type of solid material in the experiments was not viable due to the pressure limitation of the test rig. The factors that would be tested for their effect on the filtration would thus be the applied pressure, solids concentration and filter cloth pore size. These factors were selected as the literature review indicated that they all were significantly contributing effects on filtration performance (Wakeman & Tarleton, 1999a). However, by conducting a statistical design and analysis of experiments, the level of significance of each of these factors could be found. The levels of variation for each of these factors were chosen as follows:

- Pressure: As discussed in section 4.1.1(b), there were only three pressure levels available to choose from (1.5bar abs, 2.0bar abs and 2.5 bar abs), the lowest pressure (1.5bar abs) would be used for the low level value and the highest pressure (2.5bar abs) would be used for the high level value.

- Filter cloth: There were only two types of cloth available for experimentation and thus the 10µm cloth would be the low level value and the 20µm cloth was used as the high level value.

- Concentration: A large range of concentration values were available so the high and low levels of this factor had to be chosen cautiously. Fatti (2009) suggested that selecting the extreme values of a range to be the high and low levels should be avoided when a large range of levels are available (i.e. choosing the highest concentration (10%) as the high level and the lowest concentration (1%) as the low level). He said that selecting values adjacent to these as the levels would prevent an erroneous conclusion being drawn based on a tail end effect, rather than a conclusion based on the full effect of all of the values within the range. Thus, for this investigation, the 2% solids concentration was selected as the low level and the high level at 9%. This decision also prevented the use of the 1% concentration level as the low level which had shown erratic filtration behaviour and would make the statistical analysis difficult to conduct due to its irreproducibility.

A 2^3 factorial design was thus embarked upon. These experiments would be executed once for the two-phase experiments (8 runs) and once for the three-phase experiments (8 runs). In order to achieve a more accurate result, these tests were repeated. Thus, the total number of runs for the statistical analysis totalled to 32.

The experimental plan indicates in what combination the factors should be varied and in what sequence, and is provided in the form of a table of signs, as shown by Table 2-1 in section 2.10.2.1. For the statistical analysis, the applied pressure would be referred to as "*A*", the solids concentration as "*B*" and the filter cloth pore size as "*C*". The parameters would be varied in the following sequence:

Table 4-13: Sequence of the experiments for the factorial experiments

Factorial Run	Pressure (A)	Concentration (B)	Filter cloth pore size (C)	Run number			
				Two phase	Two phase repeat	Three phase	Three phase repeat
1	1.5bar	2%	10µm	61	69	77	85
2	2.5bar	2%	10µm	62	70	78	86
3	1.5bar	9%	10µm	63	71	79	87
4	2.5bar	9%	10µm	64	72	80	88
5	1.5bar	2%	20µm	65	73	81	89
6	2.5bar	2%	20µm	66	74	82	90
7	1.5bar	9%	20µm	67	75	83	91
8	2.5bar	9%	20µm	68	76	84	92

4.3.1. Filtration data analysis

The following tables (Tables 4-14 to 4-17) summarise the filtration behaviour for each of these experiments:

Table 4-14: Data summary for the two-phase factorial experiments

Factorial Run	Run number	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m^{-1})	Pressure (bar)	Concentration (%mass/mass)	Filter cloth pore size (µm)
1	61	69.08	02:42	2.503	448.9	2.383E+09	6.034E+09	1.5	2	10
2	62	52.49	01:53	8.862	651.3	2.666E+09	1.183E+10	2.5	2	10
3	63	69.76	04:16	1.182	202.7	1.443E+09	8.741E+09	1.5	9	10
4	64	62.95	02:29	4.533	183.1	1.640E+09	1.708E+10	2.5	9	10
5	65	68.76	02:35	2.560	346.1	2.505E+09	6.094E+09	1.5	2	20
6	66	52.24	01:52	9.178	593.4	2.476E+09	1.189E+10	2.5	2	20
7	67	69.57	04:40	1.070	148.1	1.713E+09	7.427E+09	1.5	9	20
8	68	62.63	02:29	4.340	192.9	1.662E+09	1.696E+10	2.5	9	20

Table 4-15: Data summary for the two-phase repeated factorial experiments

Factorial Run	Run number	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m^{-1})	Pressure (bar)	Concentration (%mass/mass)	Filter cloth pore size (µm)
1	69	68.95	02:43	2.391	288.5	2.203E+09	6.615E+09	1.5	2	10
2	70	52.87	01:51	8.781	704.0	2.701E+09	1.178E+10	2.5	2	10
3	71	69.79	04:19	1.219	143.9	1.388E+09	9.431E+09	1.5	9	10
4	72	63.21	02:32	4.413	174.4	1.710E+09	1.684E+10	2.5	9	10
5	73	67.84	02:43	2.463	360.5	2.273E+09	6.431E+09	1.5	2	20
6	74	52.99	01:50	8.805	523.4	2.520E+09	1.210E+10	2.5	2	20
7	75	69.57	04:29	1.056	120.6	1.521E+09	9.175E+09	1.5	9	20
8	76	62.93	02:32	4.671	172.5	1.732E+09	1.699E+10	2.5	9	20

Table 4-16: Data summary for the three-phase factorial experiments

Factorial Run	Run number	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m^{-1})	Pressure (bar)	Concentration (%mass/mass)	Filter cloth pore size (µm)
1	77	69.15	02:47	2.643	476.9	2.454E+09	6.207E+09	1.5	2	10
2	78	53.22	01:53	9.111	651.1	2.854E+09	1.133E+10	2.5	2	10
3	79	69.60	04:23	1.042	151.0	1.510E+09	9.010E+09	1.5	9	10
4	80	62.45	02:32	4.256	214.8	1.747E+09	1.693E+10	2.5	9	10
5	81	69.13	02:44	2.545	256.4	2.212E+09	6.785E+09	1.5	2	20
6	82	52.09	01:52	8.790	481.0	2.500E+09	1.231E+10	2.5	2	20
7	83	69.40	04:23	1.076	169.9	1.535E+09	8.965E+09	1.5	9	20
8	84	60.21	02:33	4.506	215.3	1.758E+09	1.714E+10	2.5	9	20

Table 4-17: Data summary for the three-phase repeated factorial experiments

Factorial Run	Run number	Moisture content of cake (% - wet basis)	Filtration time (mm:ss)	Airflow (l/min)	m_s (g)	α_{av} (m/kg)	R_m (m^{-1})	Pressure (bar)	Concentration (%mass/mass)	Filter cloth pore size (µm)
1	85	68.87	02:49	2.394	310.6	2.201E+09	7.023E+09	1.5	2	10
2	86	53.70	01:52	8.675	402.5	2.438E+09	1.260E+10	2.5	2	10
3	87	68.36	04:29	1.099	184.4	1.421E+09	1.051E+10	1.5	9	10
4	88	62.56	02:32	4.473	183.3	1.755E+09	1.682E+10	2.5	9	10
5	89	60.88	02:46	2.551	355.4	2.141E+09	6.913E+09	1.5	2	20
6	90	52.72	01:52	8.520	519.4	2.626E+09	1.219E+10	2.5	2	20
7	91	69.90	04:35	1.090	161.7	1.469E+09	9.995E+09	1.5	9	20
8	92	62.22	02:32	4.054	142.5	2.191E+09	1.690E+10	2.5	9	20

The filtration characteristic curves for these experiments can be found in Appendix C, Figures C-24 to C-31. The following observations were drawn from the data in the above tables (Tables 4-14, 4-15, 4-16 and 4-17):

- A higher applied pressure results in a reduced filtration time, increased airflow rate and decreased moisture content of the filter cake for both the two-phase and three-phase experiments.

- The two-phase and three-phase experiments showed an increase in pressure generally results in an increase in the specific cake resistance (α_{av}), with the exception of runs 66 and 68. However upon repeating these experiments the α_{av} was seen to increase with an increase pressure.

- The two-phase and three-phase experiments both showed a slight increase in the resistance of the filter medium (R_m) with an increase in pressure.

- An increase in concentration for both the two-phase and three-phase experiments resulted in a longer filtration time, lower air flow rate and increased moisture content of the cake.

- The specific cake resistance (α_{av}) was seen to decrease with an increase in the solids concentration of the slurry for the two-phase and three-phase experiments.

- The filter medium resistance (R_m) was seen to increase with increasing solids concentration in the two-phase and three-phase experiments.

- No definitive conclusions could be drawn regarding the effect of the filter cloth pore size on the behaviour of the filtration constants α_{av} and R_m, or on the moisture content of the filter cake, airflow rate or filtration time.

- There are no distinctive differences between the behaviour of the two-phase and three-phase systems for each of the experiments conducted.

- All the filtration curves for these experiments (Figures C-24 to C-31) exhibited linear behaviour with the curves for the two-phase and three-phase experiments, as well as their repeat experiments, being very similar in shape.

4.3.2. Statistical analysis of experiments

The impact of the parameters (pressure, concentration and filter cloth pore size) on the filtration constants is the prime objective of the statistical analysis. Thus, the statistical analysis would be done to determine the effect on the specific cake resistance (α_{av}) and then be done to determine the effect on the filter medium resistance (R_m) for each of the phases of operation. Thus, a total of four analyses would be done: two analyses would be done for α_{av} (one for two-phase operation and one for three-phase operation) and two analyses for R_m (one for two-phase operation and one for three-phase operation).

The values of the filtration constants for each of these experiments that would be used in the statistical analysis are shown below in Tables 4-18 and 4-19:

Table 4-18: Summary of the filtration constants calculated for the two-phase and two-phase repeated factorial experiments

Factorial Run	Treatment combination	Run Number	α_{av} (m/kg)	R_m (m^{-1})
1	1	61	2.383E+09	6.034E+09
1 repeat		69	2.203E+09	6.615E+09
2	a	62	2.666E+09	1.183E+10
2 repeat		70	2.701E+09	1.178E+10
3	b	63	1.443E+09	8.741E+09
3 repeat		71	1.388E+09	9.431E+09
4	ab	64	1.640E+09	1.708E+10
4 repeat		72	1.710E+09	1.684E+10
5	c	65	2.505E+09	6.094E+09
5 repeat		73	2.273E+09	6.431E+09
6	ac	66	2.476E+09	1.189E+10
6 repeat		74	2.520E+09	1.210E+10
7	bc	67	1.713E+09	7.427E+09
7 repeat		75	1.521E+09	9.175E+09
8	abc	68	1.662E+09	1.696E+10
8 repeat		76	1.732E+09	1.699E+10

Table 4-19: Summary of the filtration constants calculated for the three-phase and three-phase repeated factorial experiments

Factorial Run	Treatment combination	Run Number	α_{av} (m/kg)	R_m (m^{-1})
1	1	77	2.454E+09	6.207E+09
1 repeat		85	2.201E+09	7.023E+09
2	a	78	2.854E+09	1.133E+10
2 repeat		86	2.438E+09	1.260E+10
3	b	79	1.510E+09	9.010E+09
3 repeat		87	1.421E+09	1.051E+10
4	ab	80	1.747E+09	1.693E+10
4 repeat		88	1.755E+09	1.682E+10
5	c	81	2.212E+09	6.785E+09
5 repeat		89	2.141E+09	6.913E+09
6	ac	82	2.500E+09	1.231E+10
6 repeat		90	2.626E+09	1.291E+10
7	bc	83	1.535E+09	8.965E+09
7 repeat		91	1.469E+09	9.995E+09
8	abc	84	1.758E+09	1.714E+10
8 repeat		92	2.191E+09	1.690E+10

The differences between the values obtained for the two-phase and three-phase factorial experiments were not as large as those obtained for the experiments in which one factor was varied at a time. This could be attributed to the fact that the statistical experiments were conducted on the newly rebuilt test rig (after the original test rig had been damaged by the flood mentioned in chapter 3). The new test rig operated at peak performance as the commissioning of the original apparatus indicated all the necessary equipment modifications that were needed. The differences in the values of the filtration constants for original and repeat experiments were as follows:

- For the two-phase case with respect to α_{av}, the difference in the values from the repeat experiments (Runs 69-76) to the original experiments (Runs 61-68) ranged from 1.29%-11.88%.

- For the two-phase experiments with respect to R_m, the difference in the values between the original (Runs 61-68) and repeat experiments (Runs 69-76) ranged between 0.17%-21.30%.

- In the three-phase case, with respect to α_{av}, the difference in the values from the repeat experiments (Runs 85-92) to the original experiments (Runs 77-84) ranged from 0.43%-21.22%.

- For the three-phase experiments with respect to R_m, the difference in the values between the original (Runs 77-84) and repeat experiments (Runs 85-92) ranged between 0.70%-15.10%

The effects of each factor or the interaction of the factors on the filtration constants were determined through the application of Equations (2-21) to (2-27) (As shown in Appendix B). By comparing these values, it is possible to obtain an idea of which factors or interaction terms could or may have a significant effect on the respective filtration constants, this is further explained in the sections that follow. A summary of these effect values are shown in Tables 4-20, 4-21, 4-22 and 4-23

Table 4-20: Summary of the effects of each factor and the interaction of factors for the two-phase experiments with respect to α_{av}

	A	B	C	AB	AC	BC	ABC
Two-phase α_{av} Effect:	2.097E+08	-8.648E+08	3.310E+07	-4.017E+07	-1.152E+08	7.830E+07	2.547E+07

From the above table the values that differ significantly from the others are the effects calculated for factors *A*, *B* and *AC*. This implies that for the two-phase experiments, the α_{av} is influenced by the pressure, solids concentration and the interaction between the pressure and filter cloth pore size.

Table 4-21: Summary of the effects of each factor and the interaction of factors for the two-phase experiments with respect to R_m

	A	B	C	AB	AC	BC	ABC
Two-phase R_m Effect:	6.941E+09	3.735E+09	-1.600E+08	1.335E+09	2.631E+08	-2.257E+08	1.362E+08

Table 4-21 shows that the values which differ significantly from the others are the effects calculated for factors *A*, *B* and *AB*. This implies that for the two-phase experiments, the R_m is influenced by the pressure, solids concentration and the interaction between the both these factors.

Table 4-22: Summary of the effects of each factor and the interaction of factors for the three-phase experiments with respect to α_{av}

	A	B	C	AB	AC	BC	ABC
Three-phase α_{av} Effect:	3.658E+08	-7.551E+08	6.375E+06	1.331E+07	6.380E+07	1.236E+08	2.949E+07

Table 4-22 shows that factors *A* and *B* influence the α_{av} in the three-phase system. Thus, pressure and solids concentration are significant factors and the interaction of any factors does not seem to be significant.

Table 4-23: Summary of the effects of each factor and the interaction of factors for the three-phase experiments with respect to R_m

	A	B	C	AB	AC	BC	ABC
Three-phase R_m Effect:	6.352E+09	3.864E+09	9.607E+07	9.746E+08	1.200E+08	-1.653E+08	9.291E+07

Factors *A*, *B* and *AB* are seen to be influential on the effect of the R_m in three-phase experiments, as indicated by the effect values shown in Table 4-23. The pressure, solids concentration and their interaction are thus important parameters.

The conclusions drawn from Tables 4-20 to 4-23 provided an indication of the important factors. These effect values however, did not indicate the level of significance of the factors or their interactions on the filtration constants. This was determined by conducting an *ANOVA* analysis as explained in the section that follows.

4.3.2.1. *ANOVA* Analysis

In order to determine exactly which factors and which combination of factors affected the results of the filtration constants, an *ANOVA* analysis was conducted on both filtration constants for two-phase and three phase operation. The *ANOVA* tables were constructed for four different scenarios:

- For the two-phase factorial experiments with respect to α_{av} (Table 4-24)
- For the two-phase factorial experiments with respect to R_m (Table 4-25)
- For the three-phase factorial experiments with respect to α_{av} (Table 4-26)
- For the three-phase factorial experiments with respect to R_m (Table 4-27)

Table 4-24: *ANOVA* table for the two-phase factorial experiments with respect to α_{av}

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p-value
A	1.674E+17	1	1.674E+17	21.305	**0.001720**
B	3.027E+18	1	3.027E+18	385.402	**4.714E-08**
C	3.134E+15	1	3.134E+15	0.399	0.5453
AB	4.918E+15	1	4.918E+15	0.626	0.4516
AC	4.847E+16	1	4.847E+16	6.171	**0.0379**
BC	2.783E+16	1	2.783E+16	3.543	0.0966
ABC	1.658E+15	1	1.658E+15	0.211	0.6581
Error	6.284E+16	8	7.855E+15		
Total	3.344E+18	15			

Table 4-25: *ANOVA* table for the two-phase factorial experiments with respect to R_m

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p-value
A	1.927E+20	1	1.927E+20	753.838	**3.339E-09**
B	5.582E+19	1	5.582E+19	218.369	**4.327E-07**
C	1.024E+17	1	1.024E+17	0.401	0.5444
AB	7.132E+18	1	7.132E+18	27.901	**0.0007442**
AC	2.769E+17	1	2.769E+17	1.083	0.3284
BC	2.038E+17	1	2.038E+17	0.797	0.3980
ABC	7.424E+16	1	7.424E+16	0.290	0.6046
Error	2.045E+18	8	2.556E+17		
Total	2.583E+20	15			

Table 4-26: *ANOVA* table for the three-phase factorial experiments with respect to α_{av}

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p-value
A	5.352E+17	1	5.352E+17	18.693	**0.002534**
B	2.281E+18	1	2.281E+18	79.653	**1.971E-05**
C	1.626E+14	1	1.626E+14	0.006	0.9418
AB	7.090E+14	1	7.090E+14	0.025	0.8789
AC	1.628E+16	1	1.628E+16	0.569	0.4724
BC	6.113E+16	1	6.113E+16	2.135	0.1821
ABC	3.480E+15	1	3.480E+15	0.122	0.7364
Error	2.290E+17	8	2.863E+16		
Total	3.127E+18	15			

Table 4-27: *ANOVA* table for the three-phase factorial experiments with respect to R_m

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p-value
A	1.614E+20	1	1.614E+20	451.631	**2.527E-08**
B	5.972E+19	1	5.972E+19	167.142	**1.213E-06**
C	3.692E+16	1	3.692E+16	0.103	0.7561
AB	3.800E+18	1	3.800E+18	10.634	**0.01151**
AC	5.761E+16	1	5.761E+16	0.161	0.6985
BC	1.093E+17	1	1.093E+17	0.306	0.5953
ABC	3.453E+16	1	3.453E+16	0.097	0.7639
Error	2.859E+18	8	3.573E+17		
Total	2.280E+20	15			

From the above *ANOVA* tables the following observations can be drawn:

For the two-phase analysis with respect to the specific cake resistance (α_{av}) (Table 4-24):

- The specific cake resistance (α_{av}) is seen to be directly affected by the applied pressure to the system and the concentration of the feed slurry. The level of significance for both these parameters is quite high (0.001720 and 4.714E-08 respectively), with the concentration of the suspension having a much greater effect on α_{av}, as indicated by the higher level of significance found for this factor.

- The combination of the applied pressure and filter cloth pore size are seen to affect the α_{av}, indicated a *p-value* of 0.0379.

For the two-phase analysis with respect to the filter medium resistance (R_m) (Table 4-25):

- The applied pressure and solids concentration of the suspension are seen to have rather significant effects on the medium resistance (R_m), indicated by *p-values* of 3.339E-09 and 4.327E-07 respectively.

- R_m is also affected by a combination of the applied pressure and solids concentration (calculated *p-value* of 0.0007442).

For the three-phase analysis with respect to the specific cake resistance (α_{av}) (Table 4-26):

- The pressure and concentration are seen to have significant effects (*p-values* of 0.002534 and 1.971E-05 respectively), with the effect of the concentration being at a greater level of significance.

- There are no combination effects in this analysis.

For the three-phase analysis with respect to the filter medium resistance (R_m) (Table 4-27):

- The pressure and concentration are noted to have an influence on the resistance of the filter medium (R_m)as indicated by *p-values* of 2.527E-08 and 1.213E-06 respectively.

- R_m is also affected by a combination of the applied pressure and solids concentration (as shown by the *p-value* of 0.01151).

The significant effects found for the two-phase systems are the same as the significant effects for the three-phase systems with the only difference being that the three-phase system, with respect to α_{av}, was not influenced by any combination effects of the tested factors. The two-phase experiments with respect to α_{av} was seen to be influenced by the combined effect of the pressure and filter cloth (See Table 4-24), however, this was not indicated for the three-phase experiments with respect to α_{av}. From analysing the behaviour of the α_{av} in Tables 4-14 and 4-15, it is seen that the α_{av} is indeed separately influenced by the pressure and filter cloth. With an increase in pressure, the α_{av} is seen to increase and with a change of the filter cloth pore size from the low level (10µm) to the high level (20µm), the α_{av} decreases. This results in the "cancelling out" of the combined effects of the pressure and filter cloth pore size, since the effect of each of these factors causes the α_{av} values to change in the opposite direction (i.e. increasing pressure, increases the α_{av} and moving from a low level of the filter cloth pore size to the high level decreases the α_{av}.

The factors which were found significant in both the two-phase and three-phase systems displayed similar levels of significance (i.e. the factors had similar *p-values*).

In filtration processes the α_{av} is known to be affected by both the applied pressure and concentration of the slurry (Wakeman & Tarleton, 1999a). The observation that the α_{av} is also affected by a combination of the pressure and filter cloth pore size came to light after conducting the statistical analysis. This combination effect is however not present in the three-phase system. It was also known that the R_m is dependent on pressure and solids concentration (Wakeman & Tarleton, 1999a). The statistical analysis also showed that a combination of the pressure and solids concentration also jointly influenced the R_m of the filtration system. The *ANOVA* analysis not only indicated which factors were significant but also gave an indication of the degree of significance for each of these factors and their combinations. The solids concentration of the slurry had a significant effect on the α_{av} for both the two and three-phase experiments. The pressure and solids concentration were also found to have a significant effect on the R_m.

4.3.3. Regression analysis

In order to describe the behaviour of the significant effects (pressure, solids concentration, filter cloth pore size and their combinations) on the filtration constants (α_{av} and R_m), a regression analysis was embarked upon. Using the computed effect values for each factor (shown in Tables 4-20 to 4-23), the regression coefficients were determined, along with the intercept for the regression model (see Appendix B).

For the two-phase system, with regards to the specific cake resistance (α_{av}), the pressure, concentration and pressure-cloth effects were significant and thus the regression model contains 4 terms, including the intercept. The number of terms for the α_{av} for the three-phase systems can be determined similarly, as well as the number of terms for the two-phase and three-phase systems with respect to R_m. The regression equations for each of these scenarios are listed below with the assumption being that each system behaves in a first-order manner and that the expected error term (ε) (shown in Equation 2-46) is negligible:

For the two-phase experiments with respect to α_{av}:

$$y = 2.034x10^9 + 1.048x10^8x_1 - 4.324x10^8x_2 - 5.760x10^7x_{13}$$

Where y = the dependant variable, α_{av}

x_1 = the independent variable of the applied pressure to the system

x_2 = the independent variable of the solids concentration of the feed

x_{13} = the combination term for the effects of the applied pressure and filter cloth pore size

For the two-phase experiments with respect to R_m:

$$y = 1.096x10^{10} + 3.470x10^9x_1 + 1.868x10^9x_2 + 6.676x10^8x_{12}$$

Where y = the dependant variable, R_m

x_1 = the independent variable of the applied pressure to the system

x_2 = the independent variable of the solids concentration of the feed

x_{12} = the combination term for the effects of the applied pressure and solids concentration

For the three-phase experiments with respect to α_{av}:

$$y = 2.051x10^9 + 1.829x10^8x_1 - 3.775x10^8x_2$$

Where y = the dependant variable, α_{av}

x_1 = the independent variable of the applied pressure to the system

x_2 = the independent variable of the solids concentration of the feed

For the three-phase experiments with respect to R_m:

$$y = 1.135x10^{10} + 3.176x10^9x_1 + 1.932x10^9x_2 + 4.873x10^8x_{12}$$

Where y = the dependant variable, R_m

x_1 = the independent variable of the applied pressure to the system

x_2 = the independent variable of the solids concentration of the feed

x_{12} = the combination term for the effects of the applied pressure and solids concentration

The above regression equations were used to calculate predicted values for the filtration constants for the two-phase and three-phase systems. Once these predicted values were found, the residual values were easily obtained by finding the difference between the actual value of the dependant variable, computed from the filtration characteristic equation (Equation 2-17), and the predicted value, from the regression models. These predicted values and the residual values are shown in the tables that follow (Tables 4-28 to 4-31):

Table 4-28: Residual values for the two-phase factorial experiments with respect to α_{av}

Factorial Run	Run	Actual result	Predicted result	Residual
1	61	2.383E+09	2.304E+09	7.976E+07
1 repeat	69	2.203E+09	2.304E+09	-1.002E+08
2	62	2.666E+09	2.628E+09	3.806E+07
2 repeat	70	2.701E+09	2.628E+09	7.278E+07
3	63	1.443E+09	1.439E+09	4.634E+06
3 repeat	71	1.388E+09	1.439E+09	-5.039E+07
4	64	1.640E+09	1.764E+09	-1.234E+08
4 repeat	72	1.710E+09	1.764E+09	-5.360E+07
5	65	2.505E+09	2.419E+09	8.598E+07
5 repeat	73	2.273E+09	2.419E+09	-1.459E+08
6	66	2.476E+09	2.513E+09	-3.713E+07
6 repeat	74	2.520E+09	2.513E+09	6.629E+06
7	67	1.713E+09	1.554E+09	1.588E+08
7 repeat	75	1.521E+09	1.554E+09	-3.268E+07
8	68	1.662E+09	1.648E+09	1.339E+07
8 repeat	76	1.732E+09	1.648E+09	8.331E+07

Table 4-29: Residual values for the two-phase factorial experiments with respect to R_m

Factorial Run	Run	Actual result	Predicted result	Residual
1	61	6.034E+09	6.293E+09	-2.599E+08
1 repeat	69	6.615E+09	6.293E+09	3.211E+08
2	62	1.183E+10	1.190E+10	-7.002E+07
2 repeat	70	1.178E+10	1.190E+10	-1.226E+08
3	63	8.741E+09	8.694E+09	4.751E+07
3 repeat	71	9.431E+09	8.694E+09	7.376E+08
4	64	1.708E+10	1.697E+10	1.110E+08
4 repeat	72	1.684E+10	1.697E+10	-1.246E+08
5	65	6.094E+09	6.293E+09	-1.991E+08
5 repeat	73	6.431E+09	6.293E+09	1.379E+08
6	66	1.189E+10	1.190E+10	-1.208E+07
6 repeat	74	1.210E+10	1.190E+10	2.046E+08
7	67	7.427E+09	8.694E+09	-1.267E+09
7 repeat	75	9.175E+09	8.694E+09	4.816E+08
8	68	1.696E+10	1.697E+10	-7.759E+06
8 repeat	76	1.699E+10	1.697E+10	2.138E+07

Table 4-30: Residual values for the three-phase factorial experiments with respect to α_{av}

Factorial Run	Run	Actual result	Predicted result	Residual
1	77	2.454E+09	2.245E+09	2.090E+08
1 repeat	85	2.201E+09	2.245E+09	-4.409E+07
2	78	2.854E+09	2.611E+09	2.428E+08
2 repeat	86	2.438E+09	2.611E+09	-1.732E+08
3	79	1.510E+09	1.490E+09	1.959E+07
3 repeat	87	1.421E+09	1.490E+09	-6.961E+07
4	80	1.747E+09	1.856E+09	-1.087E+08
4 repeat	88	1.755E+09	1.856E+09	-1.013E+08
5	81	2.212E+09	2.245E+09	-3.385E+07
5 repeat	89	2.141E+09	2.245E+09	-1.044E+08
6	82	2.500E+09	2.611E+09	-1.111E+08
6 repeat	90	2.626E+09	2.611E+09	1.483E+07
7	83	1.535E+09	1.490E+09	4.461E+07
7 repeat	91	1.469E+09	1.490E+09	-2.122E+07
8	84	1.758E+09	1.856E+09	-9.840E+07
8 repeat	92	2.191E+09	1.856E+09	3.350E+08

Table 4-31: Residual values for the three-phase factorial experiments with respect to R_m

Factorial Run	Run	Actual result	Predicted result	Residual
1	77	6.207E+09	6.732E+09	-5.251E+08
1 repeat	85	7.023E+09	6.732E+09	2.909E+08
2	78	1.133E+10	1.211E+10	-7.820E+08
2 repeat	86	1.260E+10	1.211E+10	4.935E+08
3	79	9.010E+09	9.621E+09	-6.109E+08
3 repeat	87	1.051E+10	9.621E+09	8.931E+08
4	80	1.693E+10	1.695E+10	-1.278E+07
4 repeat	88	1.682E+10	1.695E+10	-1.309E+08
5	81	6.785E+09	6.732E+09	5.341E+07
5 repeat	89	6.913E+09	6.732E+09	1.809E+08
6	82	1.231E+10	1.211E+10	2.026E+08
6 repeat	90	1.219E+10	1.211E+10	8.585E+07
7	83	8.965E+09	9.621E+09	-6.559E+08
7 repeat	91	9.995E+09	9.621E+09	3.737E+08
8	84	1.714E+10	1.695E+10	1.927E+08
8 repeat	92	1.690E+10	1.695E+10	-4.904E+07

The residuals are defined as the error in the fit of the model with respect to each data point obtained from experimentation, and are used to describe the adequacy of the fitted model through use of normal probability plots. The coefficient of multiple determination (R^2) was also used to judge the adequacy of the regression models fitted. It was used to determine the correlation between the actual values and those predicted by the models. The coefficient of determination was also calculated for the trend-lines that were fitted for the normal probability plots. In this case, the coefficient served as to indicate how close the data resembled the behaviour of the trend-lines fitted.
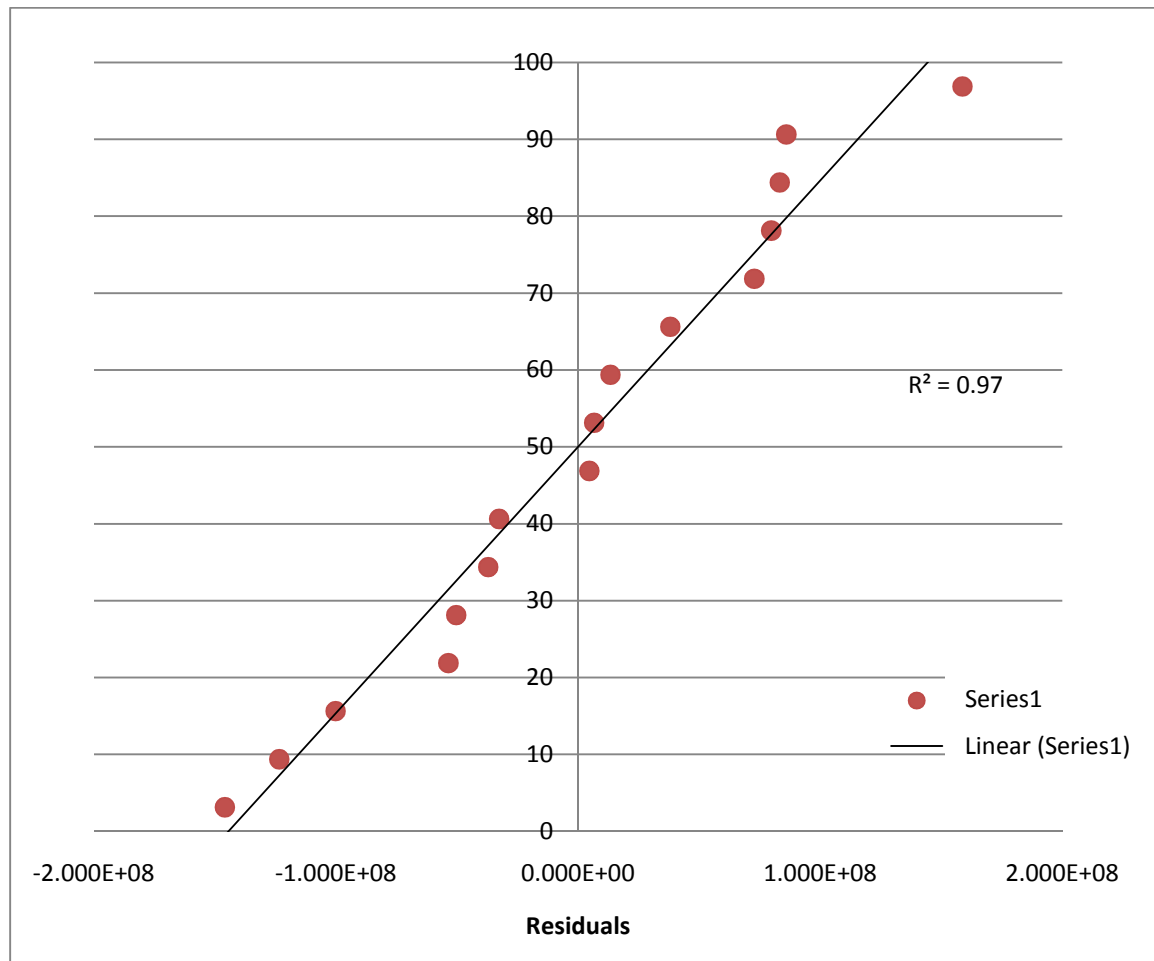
Figure 4-25: Normal probability plot for the two-phase factorial experiments with respect to α_{av}

Figure 4-25 indicates an even spread of the data point across both sides of the trend-line that was fitted. The trend-line was found to have an R^2 value of 0.97, this indicates that the data closely resembles the shape of the trend-line. The data also lies approximately along a straight line, indicating normality of the data. The correlation coefficient (R^2) between the actual and predicted values of α_{av} was calculated to be 0.97, this indicates that the regression model is a good representation of the experimental data. There is also no indication of severe outliers in the plot. All these observations combined, indicates a good fit of the regression model used.
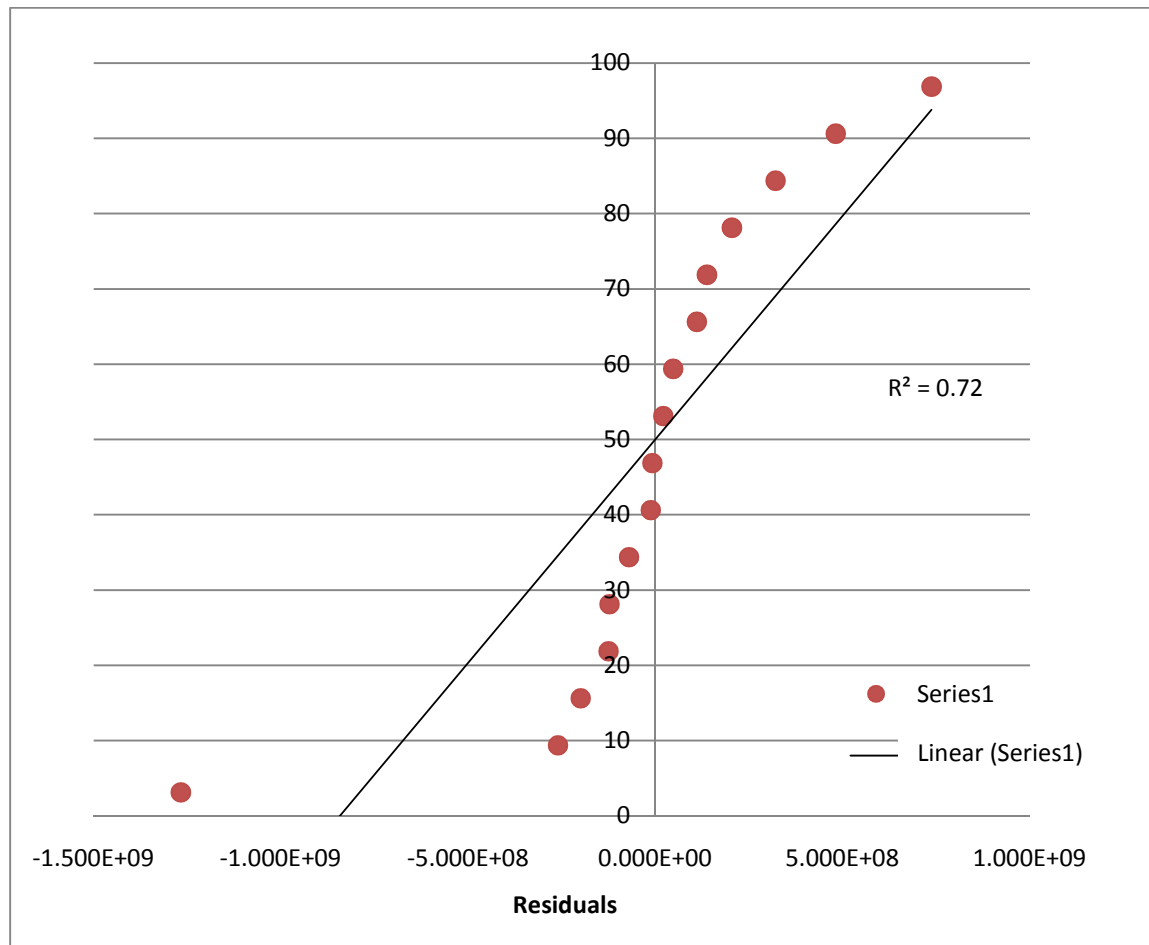
Figure 4-26: Normal probability plot for the two-phase factorial experiments with respect to R_m

Figure 4-26 shows the presence of an outlier in the plot. This may be attributed to an experimental or computational error. The outlier is not indicative of any significant effect of this run on the behaviour of the R_m because if this was so, the repeat experiment for this run would also be displayed as an outlier. The trend-line fitted to the data was found to have an R^2 of 0.72; the deviation of this value from an ideal value of 1 is attributed to the presence of the outlier mentioned above. The correlation coefficient (R^2) between the actual and predicted values of R_m was found to be 0.99; this indicates a good fit of the regression model.
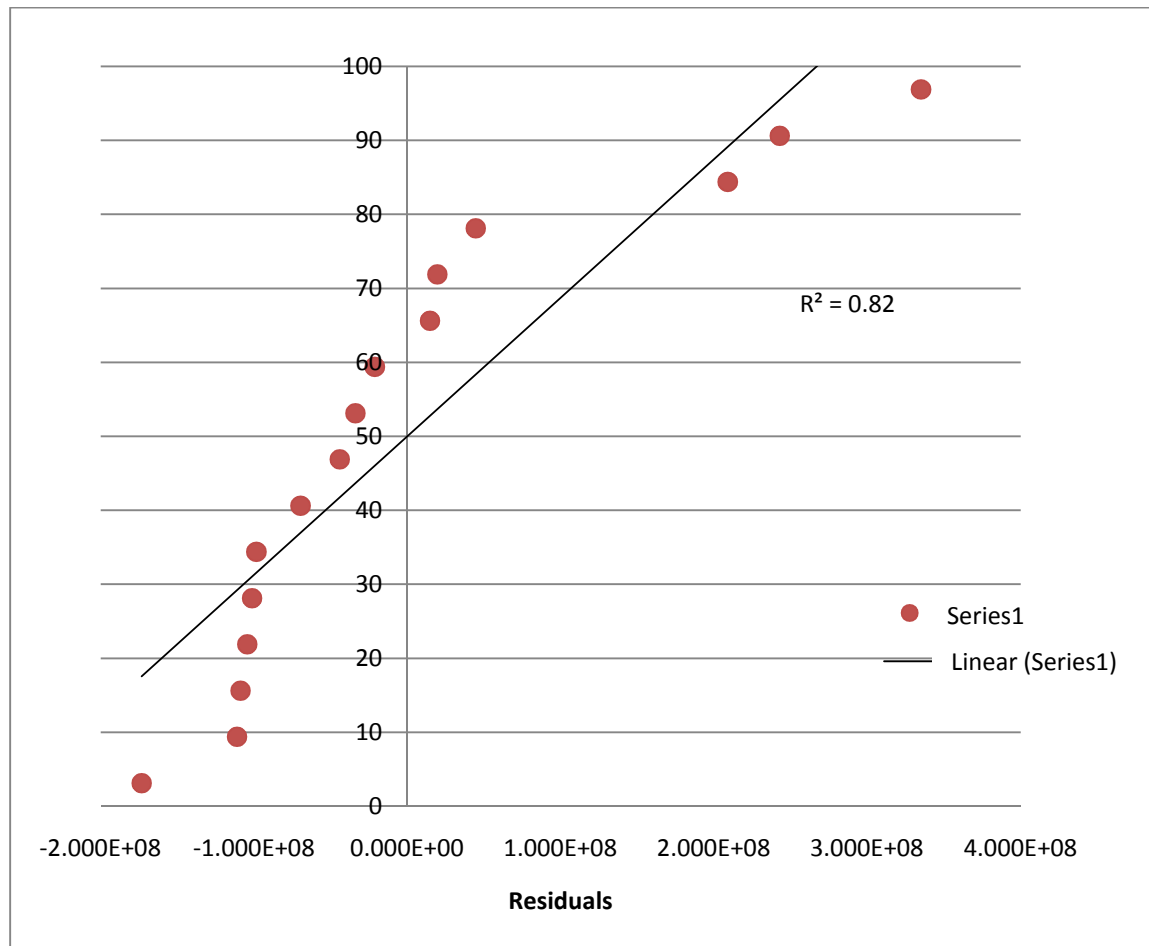
Figure 4-27: Normal probability plot for the three-phase factorial experiments with respect to α_{av}

Figure 4-27 indicates a less accurate fit of a trend-line to the data as compared to the previous plots. Here the trend-line fitted to the data has an R^2 of 0.82. The three outliers in this case may be due to experimental error and these experiments should perhaps be repeated to assess if this behaviour continues. This less accurate fit is further confirmed by an R^2 value (for the correlation between the actual and predicted α_{av}) of 0.90, which is slightly lower that the value found for the two-phase experiments shown in Figures 4-25 and 4-26.
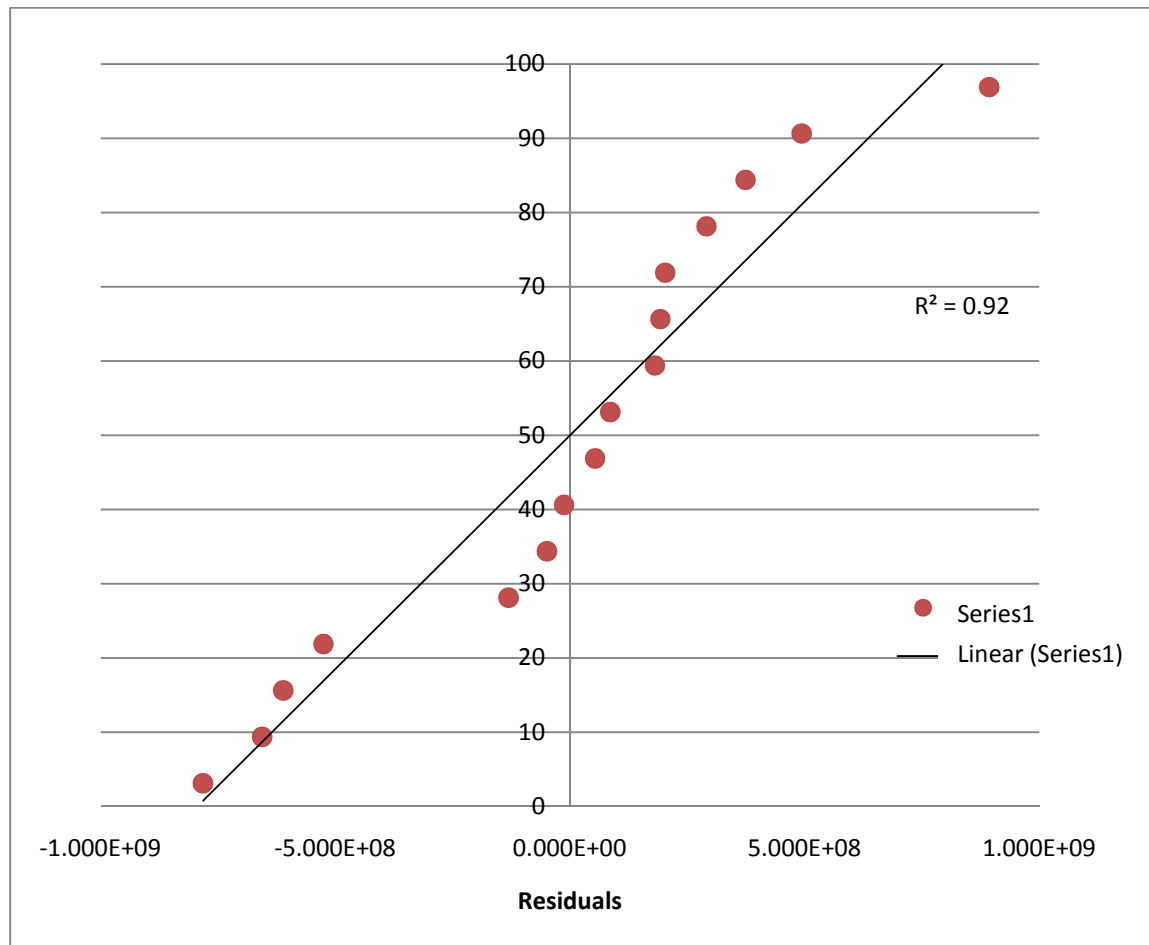
Figure 4-28: Normal probability plot for the three-phase factorial experiments with respect to R_m

Figure 4-28 also indicates a similar plot, with no significant outliers. The R^2 of the trend-line that was fitted to the data was found to be 0.92. The coefficient (R^2) calculated for the correlation between the actual and predicted R_m values was calculated as 0.98. All these observations contributed to the suitable fit of the regression model.

The general good fit of the experimental data to the regression models that were fitted indicates that the initial assumption of a first order, linear regression model to describe the behaviour of the system was indeed correct. Had the regression models exhibited poor normal probability plots, and low R^2 values for the correlation between the actual and predicted filtration constants, the assumptions made regarding the order and linearity of the regression model would have been re-assessed.

CHAPTER 5:

CONCLUSIONS AND RECOMMENDATIONS

The aim of this research project was to design and develop an operating three-phase filtration test rig. Literature in the field of three-phase filtration is sparse, as this field is not well developed. The design of the equipment was based on that obtained from the works of Tarleton (1998a, b) with the inclusion of a method to introduce the third phase to the system. The introduction of the third phase during the filtration process could have been implemented in several ways, with the most sensible occurring through the entry of a sparger in the filtration column. Upon testing the operating of the filtration device, the use of the air sparger to introduce the air was found to be more feasible. The air introduced was noticed to create a level of buoyancy within the column.

The experiments were initially conducted by varying a single factor at a time. These tests provided preliminary information on the importance of variables. These preliminary investigations also provided a better understanding regarding the behaviour of the proposed filtration system.

For the experiments in which concentration was varied whilst the applied pressure and filter cloth pore size remained fixed, the runs with low concentrations exhibited erratic filtration characteristic curves. This is attributed to the fact that the filtration times for these experiments were short (with the longest filtration time being 4.5min), which did not allow for the cake resistance to dominate over the filter medium resistance (Wakeman & Tarleton, 1999a). With increasing concentration, the behaviour of the filtration characteristic curves approached a more linear shape, agreeing with the filtration theory discussed in section 2.8 (see Equation 2-17). The experiments in which the applied pressure was varied showed that at higher pressures (2.5bar abs) the three-phase system displayed filtration characteristic curves which were different to those obtained from the two-phase experiments. This could possibly indicate that pressure indeed affects the filtration performance in a three-phase test cell. The variation of the filter cloth pore size could not provide any definitive conclusions about how this parameter affects the filtration constants. The solid material, although having particles that were very small in size (the particle size distribution showed the highest concentration of solid particles in the 125µm sieve), comprised of particles which were larger than the size of the filter cloths used (i.e. 10µm and 20µm). This could imply that using a filter medium aperture of either 10µm or 20µm did not alter the

filterability of the slurry as most of the solid particles would still be retained on the medium for either type of cloth.

The use of a statistical design and analysis of experiments provided a more efficient and effective method of experimentation, when compared to those experiments in which a single factor was varied at a time. The experiments provided conclusive results regarding the effect of the important factors (solids concentration, applied pressure and filter cloth pore size) that were tested. The α_{av} values in the two-phase system were found to be significantly dependant on the applied pressure and solids concentration of the slurry, with the combination of pressure and filter cloth pore size also affecting the α_{av}. The three-phase system exhibited dependence on the same factors with the exclusion of combination effects. The R_m in both the two and three-phase systems was found to be dependent on the applied pressure and solids concentration and the interaction of these factors.

The regression models fitted to the data showed a good fit to the data, as indicated by the normal probability plots in Chapter 4. The good fit of the models was further confirmed by the correlation coefficients that were calculated for the actual values vs. the predicted values from the model. The correlation coefficients all were close to a value of 1 and lay in the 0.90-0.99 range, as mentioned in section 2.10.2.3, indicating a good fit of the model to the experimental data.

The factors tested (solids concentration, applied pressure and filter cloth pore size) were chosen by referencing literature (Wakeman & Tarleton, 1999a). The authors mentioned that these were just some of the most important factors to consider during a filtration investigation. This investigation proved that all these factors do not necessarily affect the behaviour of this system. It was also seen that the three-phase experiments did not exhibit noticeably significant differences in their filtration behaviour when compared with their two-phase counterparts. The author proposes that future investigations into this topic should embark on monitoring the effects of other parameters, as these could potentially allow for the third phase to influence the filtration behaviour. Possible factors whose effects should be evaluated are:

- Variation of the bubbling air flow rate.

- Variation of the type of filter cloth used, perhaps the use of a woven material or mesh etc.

- Variation of the temperature of the feed by using a heating/cooling coil and variation of the temperature of the filtration column by use of a cooling/heating fluid. (This could be easily implemented due to the outer annulus present on the filtration column that can be used for circulation of a cooling or heating fluid. The slurry feed vessel also has a heating coil that was constructed, but not used during this investigation, for the slurry feed vessel.)

- Usage of an alternate fluid to water with a different viscosity.

- Conducting experiments with an alternate solid material to those tested.

- Addition of a filter aid to the system.

- Usage of particles of varying sizes and creating a particle size distribution. This can be used to determine how the particle size affects the cake formation behaviour and in turn the filtration behaviour.

- Usages of particles of varying shapes. Smooth or rough particles could also affect the filtration behaviour.

The use of a solid material with a varying particle size distribution is an important parameter to be considered. The filter cake behaviour is ultimately affected by the compacting behaviour of the solid particles within the slurry mixture. The particle size is directly linked to this, as well as the porosity and permeability of the filter cake. These factors, in turn, greatly influence the behaviour of the filtration constants. Thus, investigating the effect of the particle size on the filtration behaviour in a three-phase filtration vessel could prove to be meaningful.

Modifications to the test rig structure may possibly provide the ability to expand on the observations made. A camera may be installed to observe the bubble behaviour during the three-phase operation. The type of sparger used to distribute the third phase during the experiments may also have influenced the filtration behaviour. The base of the sparger used in the test rig was of the following structure (see Figure 5-1):
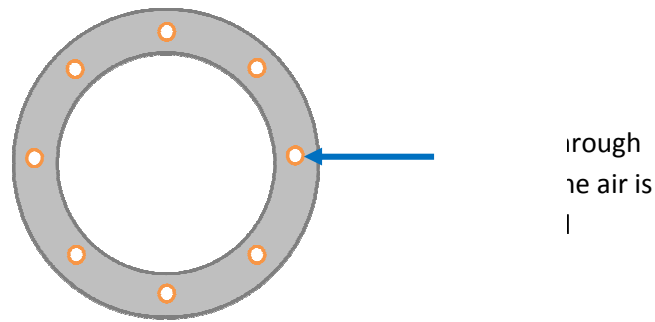
Figure 5-1: Base of air sparger, showing the distributor

Deckwer (1992, page 10) mentions that the structure of the gas distributor has a decisive influence on the behaviour within a bubble column. Alternate types of gas distributors (sintered plates, perforated plates, twin jet distributors etc.) should be considered to monitor the influence of the distributor type on the filtration behaviour during the three-phase experiments.

An alternative method of filtration may also be tested. This could be achieved through the use of a cartridge filter. These filters are easy to operate and are used when handling dilute suspensions (Tarleton and Wakeman, 2008). The structure of this type of filter is shown below (see Figure 5-2):
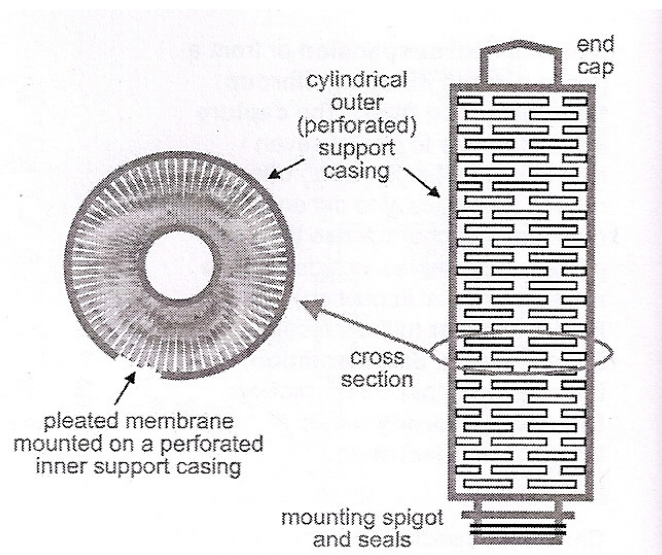
Figure 5-2: Cartridge filter (*Source: Tarleton & Wakeman, 2008, page 38)*

In this filtration device the cake is collected on the outside of the cartridge and the filtrate flows through the centre of the cartridge (Figure 5-3). Once the cartridge is fully loaded with the solid cake, the solids are removed by mechanical or physical means. Use of such a filtration device would also require alternate filtration theory to be used to analyse the filtration behaviour and determine the filtration constants. This theory is explained in great detail by Tien (2006).
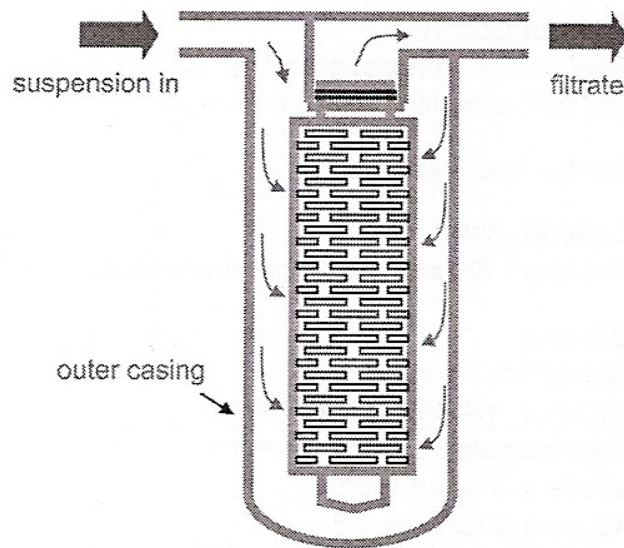
Figure 5-3: Cartridge filtration device (*Source: Tarleton & Wakeman, 2008, page 38)*

Westerterp and Wammes (2005, page 7) mention that the determination of the beginning or the end points of the filtration process are difficult to establish if there is no way of viewing the level of suspension in a filtration device. The need to view the system behaviour during the introduction of the bubbling air to the system made the use of a glass column key in this investigation. However, the use of the glass column did have a major disadvantage, the severity of which was only realised in the latter part of this investigation. The glass column had a maximum allowable operating pressure of 3bar abs, this implied that if the pressure exceeded this value, the glass column could shatter. The maximum pressure that the test rig was operated at was 2.5bar abs (the pressure could be controlled to 0.5bar).

The major modification that the author strongly encourages for future research would be the ability to create a higher pressure in the test rig. This was seen to be the major limitation when conducting the

experiments and hindered the ability to establish the effect of the buoyancy and turbulence that ought to be created by the third phase. By modifying the test rig to reach higher pressures, a larger range of experiments, at greater turbulence within the column, can be conducted. This could possibly provide more information on how the filtration process is affected by the presence of the third phase. The higher pressures will also allow for alternate solid materials, which require a greater force to promote settling of the particles, to be tested (such as talc). This increased level of turbulence may allow for more definitive conclusions to be drawn about the three-phase filtration system.

In order to attain the higher pressure within the test rig, the equipment would need to be modified. The glass column will have to be replaced by a steel column with a sight glass, and a camera can be used to observe the bubble behaviour and monitor the slurry levels within the column, thus replacing the need for a transparent glass column.

The work presented delved into the construction and smooth operation of a three-phase test rig. Drawing definitive conclusions regarding the effect of the parameters and the three-phase system behaviour may be considered to be premature, as there were operational limitations that did exist. The findings presented are preliminary to understanding the behaviour of a three-phase filtration system, with great potential in the ability to investigate numerous other factors and their effect on the three-phase filtration behaviour. The successfully designed and operated test rig can now be used for further investigations into this topic along with the knowledge of the important factors that need to be considered for further research.

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APPENDIX A:
RAW DATA

A.1. Experiments in which the solids concentration was varied whilst the applied pressure and filter cloth pore size was fixed

Two-phase experiments

Run 1: Solids concentration of 1%

Mass of solid (kg)	0.0338
Mass of water (kg)	3.004
Height of slurry (m)	0.326
Height of cake (m)	0,01
Mass of wet cake (kg)	0.0184
Mass of dry cake (kg)	0.007

Run 2: Solids concentration of 2%

Mass of solid (kg)	0.06
Mass of water (kg)	3.0026
Height of slurry (m)	0.364
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0796
Mass of dry cake (kg)	0.0334

Run 3: Solids concentration of 3%

Mass of solid (kg)	0.0904
Mass of water (kg)	3.002
Height of slurry (m)	0.367
Height of cake (m)	0.025
Mass of wet cake (kg)	0.152
Mass of dry cake (kg)	0.0508

Run 4: Solids concentration of 4%

Mass of solid (kg)	0.1194
Mass of water (kg)	3.0024
Height of slurry (m)	0.358
Height of cake (m)	0.025
Mass of wet cake (kg)	0.043
Mass of dry cake (kg)	0.0208

Run 5: Solids concentration of 5%

Mass of solid (kg)	0.1512
Mass of water (kg)	3.0036
Height of slurry (m)	0.357
Height of cake (m)	0.029
Mass of wet cake (kg)	0.0428
Mass of dry cake (kg)	0.0178

Run 6: Solids concentration of 6%

Mass of solid (kg)	0.181
Mass of water (kg)	3.003
Height of slurry (m)	0.357
Height of cake (m)	0.035
Mass of wet cake (kg)	0.0822
Mass of dry cake (kg)	0.0246

Run 7: Solids concentration of 7%

Mass of solid (kg)	0.2102
Mass of water (kg)	3
Height of slurry (m)	0.36
Height of cake (m)	0.039
Mass of wet cake (kg)	0.1428
Mass of dry cake (kg)	0.0464

Run 8: Solids concentration of 8%

Mass of solid (kg)	0.2396
Mass of water (kg)	3.0014
Height of slurry (m)	0.357
Height of cake (m)	0.048
Mass of wet cake (kg)	0.1792
Mass of dry cake (kg)	0.0622

Run 9: Solids concentration of 9%

Mass of solid (kg)	0.2708
Mass of water (kg)	3.0012
Height of slurry (m)	0.36
Height of cake (m)	0.057
Mass of wet cake (kg)	0.1748
Mass of dry cake (kg)	0.0624

Run 10: Solids concentration of 10%

Mass of solid (kg)	0.3
Mass of water (kg)	3.0012
Height of slurry (m)	0.362
Height of cake (m)	0.073
Mass of wet cake (kg)	0.1508
Mass of dry cake (kg)	0.056

Two-phase repeat experiments

Run 11: Solids concentration of 1%

Mass of solid (kg)	0.0302
Mass of water (kg)	3.0018
Height of slurry (m)	0.365
Height of cake (m)	0.012
Mass of wet cake (kg)	0.0572
Mass of dry cake (kg)	0.0194

Run 12: Solids concentration of 2%

Mass of solid (kg)	0.061
Mass of water (kg)	3.001
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0882
Mass of dry cake (kg)	0.0392

Run 13: Solids concentration of 3%

Mass of solid (kg)	0.091
Mass of water (kg)	3.005
Height of slurry (m)	0.366
Height of cake (m)	0.073
Mass of wet cake (kg)	0.0978
Mass of dry cake (kg)	0.0442

Run 14: Solids concentration of 4%

Mass of solid (kg)	0.1202
Mass of water (kg)	3.0014
Height of slurry (m)	0.36
Height of cake (m)	0.026
Mass of wet cake (kg)	0.17636
Mass of dry cake (kg)	0.0552

Run 15: Solids concentration of 5%

Mass of solid (kg)	0.1504
Mass of water (kg)	3.001
Height of slurry (m)	0.365
Height of cake (m)	0.029
Mass of wet cake (kg)	0.232
Mass of dry cake (kg)	0.0703

Run 16: Solids concentration of 6%

Mass of solid (kg)	0.1806
Mass of water (kg)	3.003
Height of slurry (m)	0.365
Height of cake (m)	0.038
Mass of wet cake (kg)	0.3342
Mass of dry cake (kg)	0.1022

Run 17: Solids concentration of 7%

Mass of solid (kg)	0.2106
Mass of water (kg)	3.0084
Height of slurry (m)	0.335
Height of cake (m)	0.042
Mass of wet cake (kg)	0.3016
Mass of dry cake (kg)	0.1062

Run 18: Solids concentration of 8%

Mass of solid (kg)	0.2402
Mass of water (kg)	3.0008
Height of slurry (m)	0.368
Height of cake (m)	0.049
Mass of wet cake (kg)	0.3444
Mass of dry cake (kg)	0.1263

Run 19: Solids concentration of 9%

Mass of solid (kg)	0.2702
Mass of water (kg)	3.001
Height of slurry (m)	0.357
Height of cake (m)	0.057
Mass of wet cake (kg)	0.406
Mass of dry cake (kg)	0.1534

Run 20: Solids concentration of 10%

Mass of solid (kg)	0.3002
Mass of water (kg)	3.0012
Height of slurry (m)	0.33
Height of cake (m)	0.065
Mass of wet cake (kg)	0.4582
Mass of dry cake (kg)	0.1792

Three-phase experiments

Run 21: Solids concentration of 1%

Mass of solid (kg)	0.0306
Mass of water (kg)	3.0002
Height of slurry (m)	0.365
Height of cake (m)	0.01
Mass of wet cake (kg)	0.0536
Mass of dry cake (kg)	0.0189
t_{2p} (mm:ss)	1:30

Run 22: Solids concentration of 2%

Mass of solid (kg)	0.0604
Mass of water (kg)	3
Height of slurry (m)	0.367
Height of cake (m)	0.015
Mass of wet cake (kg)	0.082
Mass of dry cake (kg)	0.0358
t_{2p} (mm:ss)	1:25

Run 23: Solids concentration of 3%

Mass of solid (kg)	0.09
Mass of water (kg)	3.0006
Height of slurry (m)	0.367
Height of cake (m)	0.019
Mass of wet cake (kg)	0.1108
Mass of dry cake (kg)	0.0526
t_{2p} (mm:ss)	1:31

Run 24: Solids concentration of 4%

Mass of solid (kg)	0.1202
Mass of water (kg)	3.0018
Height of slurry (m)	0.36
Height of cake (m)	0.025
Mass of wet cake (kg)	0.049
Mass of dry cake (kg)	0.0222
t_{2p} (mm:ss)	3:53

Run 25: Solids concentration of 5%

Mass of solid (kg)	0.1504
Mass of water (kg)	3.008
Height of slurry (m)	0.36
Height of cake (m)	0.029
Mass of wet cake (kg)	0.0418
Mass of dry cake (kg)	0.0184
t_{2p} (mm:ss)	4:25

Run 26: Solids concentration of 6%

Mass of solid (kg)	0.1802
Mass of water (kg)	3.0026
Height of slurry (m)	0.356
Height of cake (m)	0.039
Mass of wet cake (kg)	0.0772
Mass of dry cake (kg)	0.0284
t_{2p} (mm:ss)	3:35

Run 27: Solids concentration of 7%

Mass of solid (kg)	0.211
Mass of water (kg)	3.0028
Height of slurry (m)	0.36
Height of cake (m)	0.043
Mass of wet cake (kg)	0.067
Mass of dry cake (kg)	0.0236
t_{2p} (mm:ss)	3:38

Run 28: Solids concentration of 8%

Mass of solid (kg)	0.2412
Mass of water (kg)	3.002
Height of slurry (m)	0.365
Height of cake (m)	0.049
Mass of wet cake (kg)	0.1358
Mass of dry cake (kg)	0.0588
t_{2p} (mm:ss)	4:03

Run 29: Solids concentration of 9%

Mass of solid (kg)	0.2708
Mass of water (kg)	3.0024
Height of slurry (m)	0.368
Height of cake (m)	0.057
Mass of wet cake (kg)	0.398
Mass of dry cake (kg)	0.1612
t_{2p} (mm:ss)	1:52

Run 30: Solids concentration of 10%

Mass of solid (kg)	0.3002
Mass of water (kg)	3.0036
Height of slurry (m)	0.357
Height of cake (m)	0.061
Mass of wet cake (kg)	0.2116
Mass of dry cake (kg)	0.084
t_{2p} (mm:ss)	4:19

Three-phase repeat experiments

Run 31: Solids concentration of 1%

Mass of solid (kg)	0.03
Mass of water (kg)	3.0002
Height of slurry (m)	0.37
Height of cake (m)	0.01
Mass of wet cake (kg)	0.0552
Mass of dry cake (kg)	0.0204
t_{2p} (mm:ss)	1:24

Run 32: Solids concentration of 2%

Mass of solid (kg)	0.0602
Mass of water (kg)	3
Height of slurry (m)	0.368
Height of cake (m)	0.015
Mass of wet cake (kg)	0.0832
Mass of dry cake (kg)	0.0372
t_{2p} (mm:ss)	1:30

Run 33: Solids concentration of 3%

Mass of solid (kg)	0.0902
Mass of water (kg)	3.001
Height of slurry (m)	0.368
Height of cake (m)	0.019
Mass of wet cake (kg)	0.1262
Mass of dry cake (kg)	0.0544
t_{2p} (mm:ss)	1:30

Run 34: Solids concentration of 4%

Mass of solid (kg)	0.12
Mass of water (kg)	3.0008
Height of slurry (m)	0.36
Height of cake (m)	0.026
Mass of wet cake (kg)	0.2204
Mass of dry cake (kg)	0.0916
t_{2p} (mm:ss)	0:53

Run 35: Solids concentration of 5%

Mass of solid (kg)	0.1504
Mass of water (kg)	3.005
Height of slurry (m)	0.367
Height of cake (m)	0.03
Mass of wet cake (kg)	0.273
Mass of dry cake (kg)	0.0842
t_{2p} (mm:ss)	1:33

Run 36: Solids concentration of 6%

Mass of solid (kg)	0.0181
Mass of water (kg)	3.0004
Height of slurry (m)	0.364
Height of cake (m)	0.038
Mass of wet cake (kg)	0.3216
Mass of dry cake (kg)	0.107
t_{2p} (mm:ss)	1:37

Run 37: Solids concentration of 7%

Mass of solid (kg)	0.2104
Mass of water (kg)	3.0002
Height of slurry (m)	0.366
Height of cake (m)	0.044
Mass of wet cake (kg)	0.3426
Mass of dry cake (kg)	0.1186
t_{2p} (mm:ss)	1:38

Run 38: Solids concentration of 8%

Mass of solid (kg)	0.2404
Mass of water (kg)	3
Height of slurry (m)	0.368
Height of cake (m)	0.052
Mass of wet cake (kg)	0.3766
Mass of dry cake (kg)	0.1435
t_{2p} (mm:ss)	1:43

Run 39: Solids concentration of 9%

Mass of solid (kg)	0.2704
Mass of water (kg)	3.0024
Height of slurry (m)	0.365
Height of cake (m)	0.058
Mass of wet cake (kg)	0.387
Mass of dry cake (kg)	0.1544
t_{2p} (mm:ss)	1:35

Run 40: Solids concentration of 10%

Mass of solid (kg)	0.3012
Mass of water (kg)	3.0028
Height of slurry (m)	0.37
Height of cake (m)	0.064
Mass of wet cake (kg)	0.4612
Mass of dry cake (kg)	0.1832
t_{2p} (mm:ss)	1:47

A.2. Experiments in which the applied pressure was varied whilst the solids concentration and filter cloth pore size was fixed

Two-phase experiments

Run 41: Pressure of 1.5bar

Mass of solid (kg)	0.1524
Mass of water (kg)	3.0012
Height of slurry (m)	0.364
Height of cake (m)	0.033
Mass of wet cake (kg)	0.114
Mass of dry cake (kg)	0.034

Run 42: Pressure of 2.0bar

Mass of solid (kg)	0.1504
Mass of water (kg)	3.0024
Height of slurry (m)	0.365
Height of cake (m)	0.033
Mass of wet cake (kg)	0.28
Mass of dry cake (kg)	0.0854

Run 43: Pressure of 2.5bar

Mass of solid (kg)	0.1502
Mass of water (kg)	3.001
Height of slurry (m)	0.357
Height of cake (m)	0.033
Mass of wet cake (kg)	0.1332
Mass of dry cake (kg)	0.0444

Two-phase repeated experiments

Run 44: Pressure of 1.5bar

Mass of solid (kg)	0.1512
Mass of water (kg)	3.0036
Height of slurry (m)	0.37
Height of cake (m)	0.034
Mass of wet cake (kg)	0.2828
Mass of dry cake (kg)	0.084

Run 45: Pressure of 2.0bar

Mass of solid (kg)	0.15
Mass of water (kg)	3.0002
Height of slurry (m)	0.37
Height of cake (m)	0.034
Mass of wet cake (kg)	0.285
Mass of dry cake (kg)	0.0869

Run 46: Pressure of 2.5bar

Mass of solid (kg)	0.1504
Mass of water (kg)	3.001
Height of slurry (m)	0.365
Height of cake (m)	0.029
Mass of wet cake (kg)	0.232
Mass of dry cake (kg)	0.0703

Three-phase experiments

Run 47: Pressure of 1.5bar

Mass of solid (kg)	0.151
Mass of water (kg)	3.0036
Height of slurry (m)	0.358
Height of cake (m)	0.033
Mass of wet cake (kg)	0.1416
Mass of dry cake (kg)	0.0422
t_{2p} (mm:ss)	4:57

Run 48: Pressure of 2.0bar

Mass of solid (kg)	0.1512
Mass of water (kg)	3.0042
Height of slurry (m)	0.36
Height of cake (m)	0.033
Mass of wet cake (kg)	0.0992
Mass of dry cake (kg)	0.03
t_{2p} (mm:ss)	3:45

Run 49: Pressure of 2.5bar

Mass of solid (kg)	0.1514
Mass of water (kg)	3.0004
Height of slurry (m)	0.37
Height of cake (m)	0.036
Mass of wet cake (kg)	0.2654
Mass of dry cake (kg)	0.0824
t_{2p} (mm:ss)	1:43

Three-phase repeated experiments

Run 50: Pressure of 1.5bar

Mass of solid (kg)	0.15
Mass of water (kg)	3.0018
Height of slurry (m)	0.37
Height of cake (m)	0.034
Mass of wet cake (kg)	0.2922
Mass of dry cake (kg)	0.087
t_{2p} (mm:ss)	2:14

Run 51: Pressure of 2.0bar

Mass of solid (kg)	0.1506
Mass of water (kg)	3.0018
Height of slurry (m)	0.37
Height of cake (m)	0.034
Mass of wet cake (kg)	0.02924
Mass of dry cake (kg)	0.0896
t_{2p} (mm:ss)	1:47

Run 52: Pressure of 2.5bar

Mass of solid (kg)	0.1504
Mass of water (kg)	3.005
Height of slurry (m)	0.367
Height of cake (m)	0.03
Mass of wet cake (kg)	0.273
Mass of dry cake (kg)	0.0842
t_{2p} (mm:ss)	1:33

A.3. Experiments in which filter cloth pore size was varied whilst the solids concentration and applied pressure was fixed

<u>Two-phase experiments</u>

Run 53: Filter cloth pore size of 10μm

Mass of solid (kg)	0.1512
Mass of water (kg)	3.0036
Height of slurry (m)	0.357
Height of cake (m)	0.029
Mass of wet cake (kg)	0.0428
Mass of dry cake (kg)	0.0178

Run 54: Filter cloth pore size of 20μm

Mass of solid (kg)	0.1512
Mass of water (kg)	3.0002
Height of slurry (m)	0.36
Height of cake (m)	0.033
Mass of wet cake (kg)	0.1308
Mass of dry cake (kg)	0.0402

<u>Two-phase repeated experiments</u>

Run 55: Filter cloth pore size of 10μm

Mass of solid (kg)	0.1504
Mass of water (kg)	3.001
Height of slurry (m)	0.365
Height of cake (m)	0.029
Mass of wet cake (kg)	0.232
Mass of dry cake (kg)	0.0703

Run 56: Filter cloth pore size of 20μm

Mass of solid (kg)	0.15
Mass of water (kg)	3.0006
Height of slurry (m)	0.37
Height of cake (m)	0.033
Mass of wet cake (kg)	0.2786
Mass of dry cake (kg)	0.086

Three-phase experiments

Run 57: Filter cloth pore size of 10μm

Mass of solid (kg)	0.1504
Mass of water (kg)	3.008
Height of slurry (m)	0.36
Height of cake (m)	0.029
Mass of wet cake (kg)	0.0418
Mass of dry cake (kg)	0.0184
t_{2p} (mm:ss)	4:25

Run 58: Filter cloth pore size of 20μm

Mass of solid (kg)	0.151
Mass of water (kg)	3.003
Height of slurry (m)	0.361
Height of cake (m)	0.033
Mass of wet cake (kg)	0.1434
Mass of dry cake (kg)	0.0442
t_{2p} (mm:ss)	4:27

Three-phase repeated experiments

Run 59: Filter cloth pore size of 10μm

Mass of solid (kg)	0.1504
Mass of water (kg)	3.005
Height of slurry (m)	0.367
Height of cake (m)	0.03
Mass of wet cake (kg)	0.273
Mass of dry cake (kg)	0.0842
t_{2p} (mm:ss)	1:33

Run 60: Filter cloth pore size of 20μm

Mass of solid (kg)	0.1502
Mass of water (kg)	3.001
Height of slurry (m)	0.37
Height of cake (m)	0.033
Mass of wet cake (kg)	0.262
Mass of dry cake (kg)	0.0877
t_{2p} (mm:ss)	1:34

A.4. Factorial experiments

<u>Two-phase experiments</u>

Run 61: Statistical run 1

Mass of solid (kg)	0.0608
Mass of water (kg)	3.002
Height of slurry (m)	0.368
Height of cake (m)	0.017
Mass of wet cake (kg)	0.141
Mass of dry cake (kg)	0.0436

Run 62: Statistical run 2

Mass of solid (kg)	0.061
Mass of water (kg)	3.0014
Height of slurry (m)	0.365
Height of cake (m)	0.017
Mass of wet cake (kg)	0.0922
Mass of dry cake (kg)	0.0438

Run 63: Statistical run 3

Mass of solid (kg)	0.2704
Mass of water (kg)	3
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6052
Mass of dry cake (kg)	0.183

Run 64: Statistical run 4

Mass of solid (kg)	0.2708
Mass of water (kg)	3.0004
Height of slurry (m)	0.374
Height of cake (m)	0.067
Mass of wet cake (kg)	0.5004
Mass of dry cake (kg)	0.1854

Run 65: Statistical run 5

Mass of solid (kg)	0.061
Mass of water (kg)	3.0028
Height of slurry (m)	0.35
Height of cake (m)	0.017
Mass of wet cake (kg)	0.1434
Mass of dry cake (kg)	0.0348

Run 66: Statistical run 6

Mass of solid (kg)	0.0604
Mass of water (kg)	3.0022
Height of slurry (m)	0.36
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0892
Mass of dry cake (kg)	0.0426

Run 67: Statistical run 7

Mass of solid (kg)	0.2702
Mass of water (kg)	3.0014
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.627
Mass of dry cake (kg)	0.1908

Run 68: Statistical run 8

Mass of solid (kg)	0.2706
Mass of water (kg)	3.0018
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.5026
Mass of dry cake (kg)	0.1878

Two-phase repeated experiments

Run 69: Statistical run 1

Mass of solid (kg)	0.0604
Mass of water (kg)	3.0004
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.1409
Mass of dry cake (kg)	0.0436

Run 70: Statistical run 2

Mass of solid (kg)	0.061
Mass of water (kg)	3.0034
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0976
Mass of dry cake (kg)	0.046

Run 71: Statistical run 3

Mass of solid (kg)	0.27
Mass of water (kg)	3.0026
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6262
Mass of dry cake (kg)	0.1892

Run 72: Statistical run 4

Mass of solid (kg)	0.2706
Mass of water (kg)	3.0002
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.5262
Mass of dry cake (kg)	0.1936

Run 73: Statistical run 5

Mass of solid (kg)	0.06
Mass of water (kg)	3.0012
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.1312
Mass of dry cake (kg)	0.0422

Run 74: Statistical run 6

Mass of solid (kg)	0.06
Mass of water (kg)	3.0018
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0936
Mass of dry cake (kg)	0.044

Run 75: Statistical run 7

Mass of solid (kg)	0.2706
Mass of water (kg)	3.003
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6336
Mass of dry cake (kg)	0.1928

Run 76: Statistical run 8

Mass of solid (kg)	0.271
Mass of water (kg)	3.0026
Height of slurry (m)	0.372
Height of cake (m)	0.068
Mass of wet cake (kg)	0.5104
Mass of dry cake (kg)	0.1892

Three-phase experiments

Run 77: Statistical run 1

Mass of solid (kg)	0.0606
Mass of water (kg)	3.0036
Height of slurry (m)	0.365
Height of cake (m)	0.017
Mass of wet cake (kg)	0.142
Mass of dry cake (kg)	0.0438
t_{2p} (mm:ss)	2:05

Run 78: Statistical run 2

Mass of solid (kg)	0.0602
Mass of water (kg)	3.0036
Height of slurry (m)	0.365
Height of cake (m)	0.017
Mass of wet cake (kg)	0.0962
Mass of dry cake (kg)	0.045
t_{2p} (mm:ss)	1:30

Run 79: Statistical run 3

Mass of solid (kg)	0.2704
Mass of water (kg)	3.002
Height of slurry (m)	0.374
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6148
Mass of dry cake (kg)	0.1869
t_{2p} (mm:ss)	3:48

Run 80: Statistical run 4

Mass of solid (kg)	0.2704
Mass of water (kg)	3.0018
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.5076
Mass of dry cake (kg)	0.1906
t_{2p} (mm:ss)	2:14

Run 81: Statistical run 5

Mass of solid (kg)	0.061
Mass of water (kg)	3.0002
Height of slurry (m)	0.365
Height of cake (m)	0.017
Mass of wet cake (kg)	0.1354
Mass of dry cake (kg)	0.0418
t_{2p} (mm:ss)	2:03

Run 82: Statistical run 6

Mass of solid (kg)	0.0606
Mass of water (kg)	3.0014
Height of slurry (m)	0.365
Height of cake (m)	0.017
Mass of wet cake (kg)	0.0956
Mass of dry cake (kg)	0.0458
t_{2p} (mm:ss)	1:30

Run 83: Statistical run 7

Mass of solid (kg)	0.2708
Mass of water (kg)	3.002
Height of slurry (m)	0.37
Height of cake (m)	0.068
Mass of wet cake (kg)	0.621
Mass of dry cake (kg)	0.196
t_{2p} (mm:ss)	3:44

Run 84: Statistical run 8

Mass of solid (kg)	0.2712
Mass of water (kg)	3.0014
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.5036
Mass of dry cake (kg)	0.2004
t_{2p} (mm:ss)	2:15

Three-phase repeated experiments

Run 85: Statistical run 1

Mass of solid (kg)	0.061
Mass of water (kg)	3.0018
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.1516
Mass of dry cake (kg)	0.0472
t_{2p} (mm:ss)	2:13

Run 86: Statistical run 2

Mass of solid (kg)	0.0606
Mass of water (kg)	3.003
Height of slurry (m)	0.368
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0946
Mass of dry cake (kg)	0.0438
t_{2p} (mm:ss)	1:39

Run 87: Statistical run 3

Mass of solid (kg)	0.272
Mass of water (kg)	3.0012
Height of slurry (m)	0.374
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6012
Mass of dry cake (kg)	0.1902
t_{2p} (mm:ss)	4:01

Run 88: Statistical run 4

Mass of solid (kg)	0.2708
Mass of water (kg)	3.002
Height of slurry (m)	0.375
Height of cake (m)	0.068
Mass of wet cake (kg)	0.508
Mass of dry cake (kg)	0.1902
t_{2p} (mm:ss)	2:15

Run 89: Statistical run 5

Mass of solid (kg)	0.0602
Mass of water (kg)	3.001
Height of slurry (m)	0.365
Height of cake (m)	0.016
Mass of wet cake (kg)	0.1094
Mass of dry cake (kg)	0.0448
t_{2p} (mm:ss)	2:10

Run 90: Statistical run 6

Mass of solid (kg)	0.0612
Mass of water (kg)	3.003
Height of slurry (m)	0.37
Height of cake (m)	0.016
Mass of wet cake (kg)	0.0994
Mass of dry cake (kg)	0.047
t_{2p} (mm:ss)	1:32

Run 91: Statistical run 7

Mass of solid (kg)	0.2714
Mass of water (kg)	3.0022
Height of slurry (m)	0.373
Height of cake (m)	0.068
Mass of wet cake (kg)	0.6352
Mass of dry cake (kg)	0.1912
t_{2p} (mm:ss)	4:13

Run 92: Statistical run 8

Mass of solid (kg)	0.2716
Mass of water (kg)	3.002
Height of slurry (m)	0.37
Height of cake (m)	0.069
Mass of wet cake (kg)	0.5204
Mass of dry cake (kg)	0.1966
t_{2p} (mm:ss)	2:11

APPENDIX B:

SAMPLE CALCULATIONS

All calculations were done with respect to Run 61 (Factorial run 1 for the two-phase experiments).

B.1. General calculations

Table B-1: General data

Density of Water (ρ_{water})	998	kg/m^3
Viscosity of Water (μ_{water})	1x10^{-8}	bar.s
Bulk Density of Solid (ρ_{solid})	256.295	kg/m^3
Radius of column (r)	0.05	m
Radius of filter (R)	0.05	m

Table B-2: Parameters for Run 61

Pressure drop (ΔP)	0.527	bar
Solids concentration of slurry	2	mass/mass %
Filter cloth pore size	20	µm

Table B-3: Data for Run 61

Mass of Solid (m_{solid})	0.0608	kg
Mass of Water (m_{water})	3.002	kg
Height of Slurry (H_{slurry})	0.368	m
Height of cake (L)	0.0017	m
Mass of wet cake (m_{wc})	0.141	kg
Mass of dry cake (m_{dc})	0.0436	kg

Determining s (mass fraction of solids in the feed-kg/kg):

The volume of slurry in the column was determined by adding the volume of the solid used to the volume of the liquid used:

$$V_{solid} = {m_{solid}}/{\rho_{solid}} = (0.0608)/(256.295) = 0.0002372 \ m^3$$

$$V_{water} = {m_{water}}/{\rho_{water}} = (3.002)/(998) = 0.003008 \ m^3$$

$$V_{water} = V_{solid} + V_{water} = 0.0002372 + 0.003008 = 0.003245 \ m^3$$

The filter area was calculated:

$$A = \pi R^2 = \pi \ (0.05)^2 = 0.007854 \ m^2$$

The mass fraction of solids in the feed slurry (s) is found:

s = (0.0608) / (0.0608+3.002) = 0.01985 kg/kg

Determining m (the wet to dry cake mass ratio-kg/kg):

m = 0.141/0.0436= 3.234 kg/kg

Determining c (mass of dry cake solids per unit volume of filtrate-kg/m^3):

This was found through application of Equation (2-7):

During the filtration, only a trace amount of solid particles passed through the filter cloth and collected with the filtrate. Thus, the density of the filtrate (ρ) is assumed to be equivalent to the density of the liquid used (i.e. water) as there was a minimal amount of solid that exited the system with the filtrate.

$$c = \frac{(0.01985)(998)}{1 - (3.234)(0.01985)} = 21.17 kg/m^3$$

Determining the moisture content of the cake (calculated on a wet cake basis):

$$Moisture\ content\ of\ the\ cake = \frac{m_{wc} - m_{dc}}{m_{wc}} x\ 100 = \frac{0.141 - 0.0436}{0.141} = 69.08\%$$

B.2. The filtration characteristic curve:

A graph of $\dfrac{t - t_s}{V - V_s}$ vs. $(V + V_s)$ was plotted for each run.

The t_s and corresponding V_s needed to be selected prior to drawing the graph. The t_s was selected to occur at the point when the first drop of filtrate was collected. For this run this occurred *54 seconds* into the recording of the data and the corresponding mass was *0.1 grams*. This mass was converted to a volume by using the density of water and the V_s was obtained. This value was *1.002x10⁻⁷m³*.

The filtration characteristic curve could now be plotted and found to have the following shape:
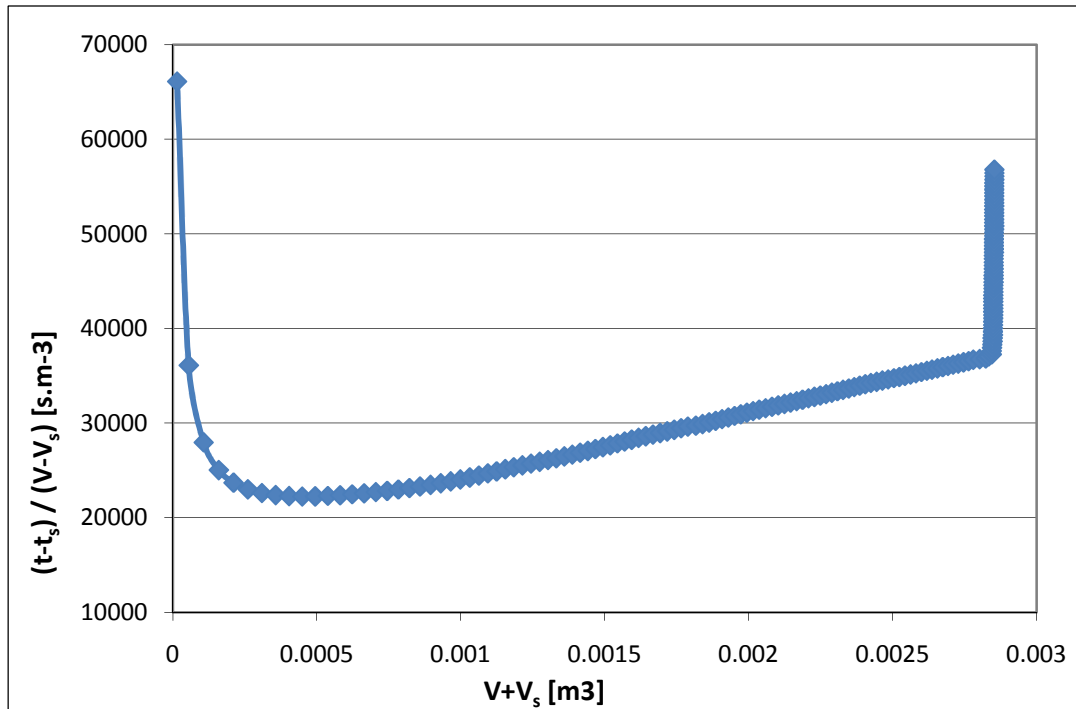
Figure B-1: Original filtration characteristic curve

The data from the cake compression stage must now be removed from this curve to eliminate the steep slope present at the end of the filtration.
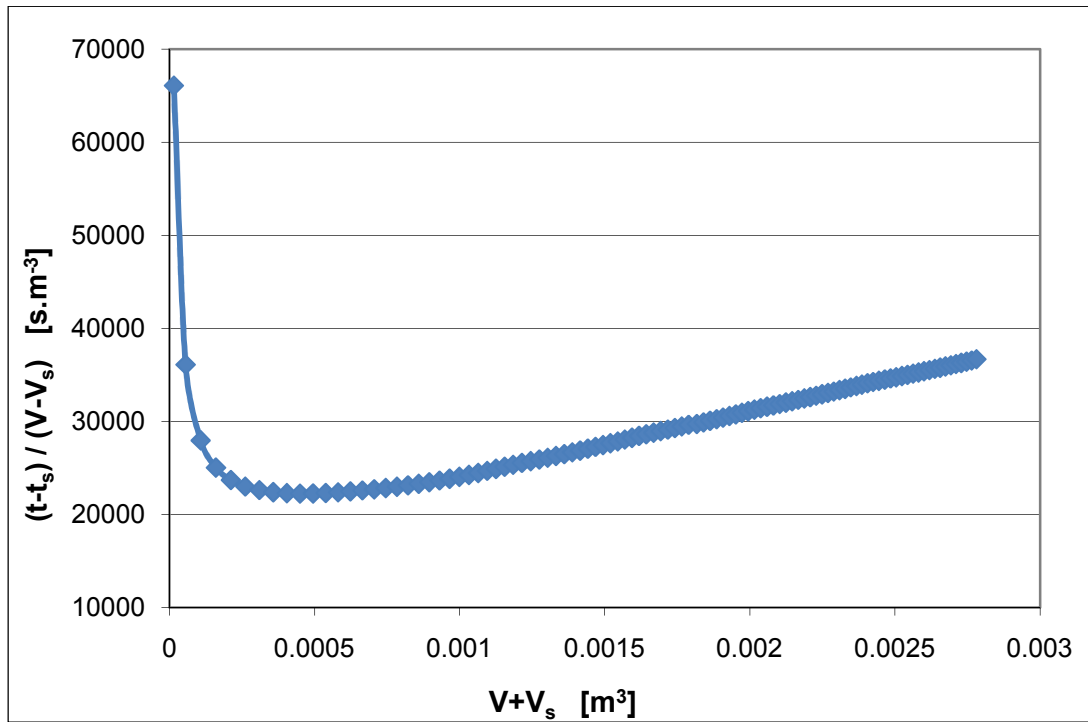
Figure B-2: Filtration characteristic curve after removing the data from the cake compression stage

The graph ought to have a linear shape, thus the t_s was then moved from the original value of *54 seconds* to the point when the $\frac{t-t_s}{V-V_s}$ began increasing again. This occurred at a time of *64 seconds*. The V_s would also have to be changed due to the use of a corrected t_s value. The corresponding mass at this time was found to be *448.9 grams*. This was converted to a volume as done previously and used as the V_s, this value was found to be *0.0004498 m³*.

The corrected filtration characteristic curve was then plotted and found to have the linear shape required, as is shown below:
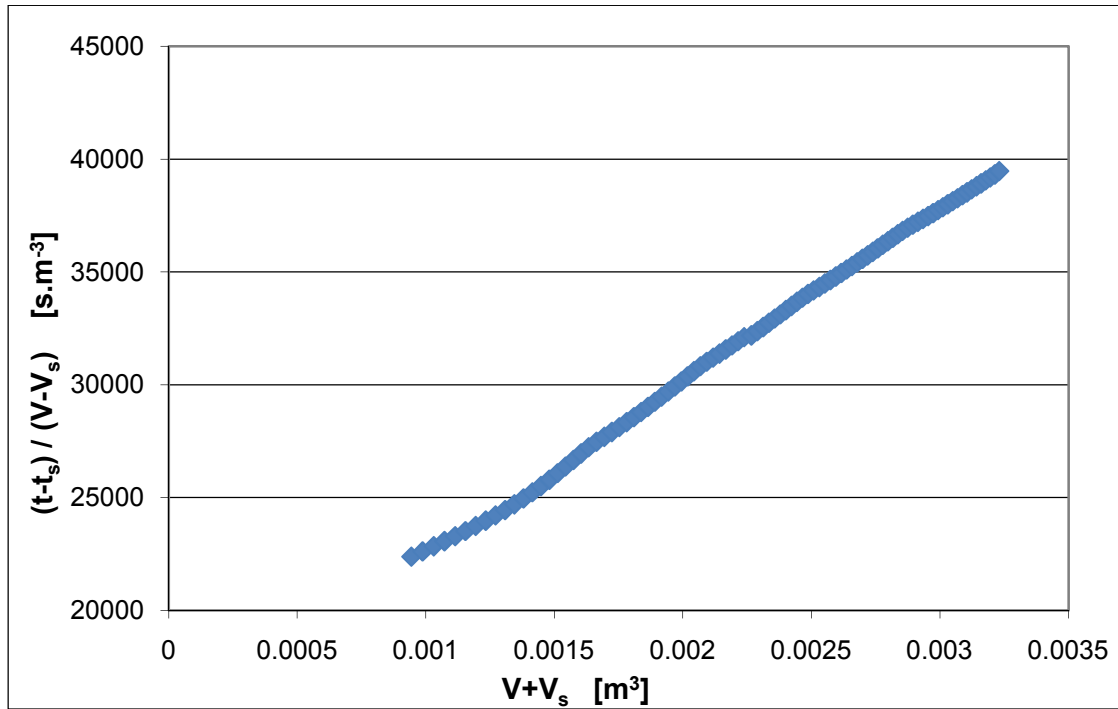
Figure B-3: Final filtration characteristic curve

B.3. Calculation of the filtration constants

The gradient of the filtration characteristic curve was obtained along with the intercept value. The gradient was found to be *7.761x10⁶* and the intercept to be *14578.63*.

These values were used in conjunction with the Filtration characteristic curve and Equation (2-17) to determine α_{av} and R_m:

$$7.761x10^6 = \frac{\alpha_{av}(1x10^{-8})(21.17)}{2(0.007854)^2(0.527)}$$

$$\alpha_{av} = 2.38x10^9\, m/kg$$

and

$$14578.63 = \frac{R_m(1x10^{-8})}{(0.007854)(0.527)}$$

$$R_m = 6.03x10^9\, m^{-1}$$

B.4. Cake formation rate curve

The cake formation rate was found by finding the cake height at each second during the filtration, this was done through use of Equation (2-9). For example the *64th second* represented the first filtration data point after correcting the initial t_s. Thus the cake height at this second is:

$$L = \frac{0.0004498}{0.007854} \frac{(0.01985)[256.295(3.234-1)+998]}{(256.295)[1-(3.234)(0.01985)]}$$
$$= 0.0007562\text{m}$$
$$= 0.7562\text{mm}$$

Once this was calculated at second, the cake formation rate could be graphed:
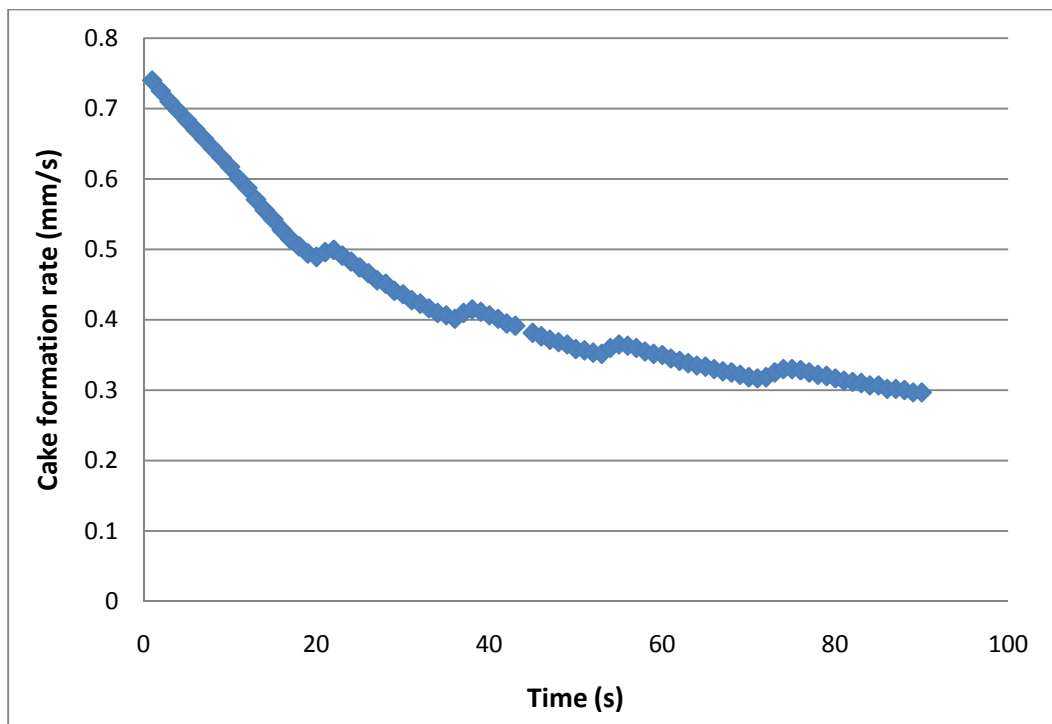
Figure B-4: Cake formation rate curve

B.5. Statistical analysis of the data

B.5.1. Determination of the effects of the factors

The table of signs for a 2 level, three factor factorial design experiment was drawn up. The three factors that were considered were the pressure (A), the concentration (B) and the filter cloth pore size (C). The interaction effects are given by the combination terms, AB, AC, BC and ABC.

Table B-4: Table of signs

Factorial Run	Treatment combination	A	B	C	AB	AC	BC	ABC
1	1	-1	-1	-1	1	1	1	-1
2	a	1	-1	-1	-1	-1	1	1
3	b	-1	1	-1	-1	1	-1	1
4	ab	1	1	-1	1	-1	-1	-1
5	c	-1	-1	1	1	-1	-1	1
6	ac	1	-1	1	-1	1	-1	-1
7	bc	-1	1	1	-1	-1	1	-1
8	abc	1	1	1	1	1	1	1

This table indicated how the factors should be varied and in what sequence for the experimental runs. A negative sign indicates that the low level should be used for that factor and a positive sign indicates that the high level of that factor should be used for that specific experiment.

Table B-5: The parameters that would be varied and their combination

Factorial Run	Treatment combination	A	B	C	Run number			
					Two phase	Two phase repeat tests	Three phase	Three phase repeat tests
1	1	1.5bar	2%	10µm	61	69	77	85
2	a	2.5bar	2%	10µm	62	70	78	86
3	b	1.5bar	9%	10µm	63	71	79	87
4	ab	2.5bar	9%	10µm	64	72	80	88
5	c	1.5bar	2%	20µm	65	73	81	89
6	ac	2.5bar	2%	20µm	66	74	82	90
7	bc	1.5bar	9%	20µm	67	75	83	91
8	abc	2.5bar	9%	20µm	68	76	84	92

Once these experiments were conducted, the filtration constants were determined. The values for these are summarized in the table below:

For the two phase tests:

Table B-6: Summary of α_{av} and R_m values for the two-phase factorial experimental runs

Factorial Run	Treatment combination	Run Number	α_{av} (m/kg)	R_m (m^{-1})
1	1	61	2.383E+09	6.034E+09
1 repeat		69	2.203E+09	6.615E+09
2	a	62	2.666E+09	1.183E+10
2 repeat		70	2.701E+09	1.178E+10
3	b	63	1.443E+09	8.741E+09
3 repeat		71	1.388E+09	9.431E+09
4	ab	64	1.640E+09	1.708E+10
4 repeat		72	1.710E+09	1.684E+10
5	c	65	2.505E+09	6.094E+09
5 repeat		73	2.273E+09	6.431E+09
6	ac	66	2.476E+09	1.189E+10
6 repeat		74	2.520E+09	1.210E+10
7	bc	67	1.713E+09	7.427E+09
7 repeat		75	1.521E+09	9.175E+09
8	abc	68	1.662E+09	1.696E+10
8 repeat		76	1.732E+09	1.699E+10

For the three phase tests:

Table B-7: Summary of α_{av} and R_m values for the three-phase factorial experimental runs

Factorial Run	Treatment combination	Run Number	α_{av} (m/kg)	R_m (m^{-1})
1	1	77	2.454E+09	6.207E+09
1 repeat		85	2.201E+09	7.023E+09
2	a	78	2.854E+09	1.133E+10
2 repeat		86	2.438E+09	1.260E+10
3	b	79	1.510E+09	9.010E+09
3 repeat		87	1.421E+09	1.051E+10
4	ab	80	1.747E+09	1.693E+10
4 repeat		88	1.755E+09	1.682E+10
5	c	81	2.212E+09	6.785E+09
5 repeat		89	2.141E+09	6.913E+09
6	ac	82	2.500E+09	1.231E+10
6 repeat		90	2.626E+09	1.291E+10
7	bc	83	1.535E+09	8.965E+09
7 repeat		91	1.469E+09	9.995E+09
8	abc	84	1.758E+09	1.714E+10
8 repeat		92	2.191E+09	1.690E+10

These values would now be used to determine the effect of each factor on the α_{av} for two-phase tests, the R_m for two-phase tests, the α_{av} for three-phase tests and the R_m for three-phase tests.

The calculation of the effects of the factors with respect to α_{av} for the two-phase tests are shown below. These effects are summarized in Table B-8 with an explanation as to how these values were computed below Table B-8:

Table B-8: Effects table for the two-phase factorial experiments, with respect to α_{av}

Factorial Run	Treatment combination	A	B	C	AB	AC	BC	ABC	Total α_{av}
1	1	-1	-1	-1	1	1	1	-1	4.587E+09
2	A	1	-1	-1	-1	-1	1	1	5.368E+09
3	B	-1	1	-1	-1	1	-1	1	2.832E+09
4	Ab	1	1	-1	1	-1	-1	-1	3.350E+09
5	C	-1	-1	1	1	-1	-1	1	4.778E+09
6	Ac	1	-1	1	-1	1	-1	-1	4.996E+09
7	Bc	-1	1	1	-1	-1	1	-1	3.234E+09
8	Abc	1	1	1	1	1	1	1	3.393E+09
Effect		2.097E+08	-8.648E+08	3.310E+07	-4.017E+07	-1.152E+08	7.830E+07	2.547E+07	

The last column in the above table is computed by summing the α_{av} values of the run concerned and the α_{av} from the repeat test for that run.

The effects for each factor and the interaction terms, shown in the last row of the above table is calculated by use of Equations (2-21)-(2-27). The values *a*, *b*, *c*, *ab*, *ac*, *bc* and *abc* that are used in these equations are obtained from the last column of the above table:

$$A = \frac{1}{4(2)}[5.368x10^9 + 3.350x10^9 + 4.996x10^9 + 3.393x10^9 - 4.628x10^9$$
$$- 2.832x10^9 - 4.778x10^9 - 3.234x10^9]$$
= 2.045 x 10^8

$$B = \frac{1}{4(2)}[2.832x10^9 + 3.350x10^9 + 3.234x10^9 + 3.393x10^9 - 4.628x10^9$$
$$- 5.368x10^9 - 4.778x10^9 - 4.996x10^9]$$
= -8.700 x 10^8

$$C = \frac{1}{4(2)}[4.778x10^9 + 4.996x10^9 + 3.234x10^9 + 3.393x10^9 - 4.628x10^9$$
$$- 5.368x10^9 - 2.832x10^9 - 3.350x10^9]$$
= 2.799 x 10^7

$$AB = \frac{1}{4(2)}[3.393x10^9 - 3.234x10^9 + 3.350x10^9 - 2.832x10^9 - 4.996x10^9$$
$$+ 4.778x10^9 - 5.368x10^9 + 4.628x10^9]$$
= -3.506 x 10^7

$$AC = \frac{1}{4(2)}[4.628x10^9 - 5.368x10^9 + 2.832x10^9 - 3.350x10^9 - 4.778x10^9$$
$$+ 4.996x10^9 - 3.234x10^9 + 3.3934x10^9]$$
= -1.101 x 10^8

$$BC = \frac{1}{4(2)}[4.628x10^9 + 5.368x10^9 - 2.832x10^9 - 3.350x10^9 - 4.778x10^9$$

$$- 4.996x10^9 + 3.234x10^9 + 3.393x10^9]$$

= 8.341 x 10^7

$$ABC = \frac{1}{4(2)}[3.393x10^9 - 3.234x10^9 - 4.996x10^9 + 4.778x10^9 - 3.350x10^9$$

$$+ 2.832x10^9 + 5.368x10^9 - 4.268x10^9]$$

= 2.036 x 10^7

From Table B-8 it is clear that the effect of A (pressure), B (solids concentration) and AC (a combination of pressure and filter cloth pore size) is quite significant on α_{av} in a two-phase system. This will be verified through the use of an *ANOVA* table.

B.5.2. *ANOVA* analysis

The *ANOVA* analysis sample calculation is computed for the two-phase experiments with regard to α_{av}.

In order to compute the sum of squares for each factor in this example with ease, the α_{av} values are set up in a table according to their run numbers as follows: in this table the first run for any given cell is the original run, and the second run is the repeat run.

Table B-9: Levels of the factors and the experiments indicating their levels

A	B			
	2%		9%	
	C		C	
	10µm	20µm	10µm	20µm
1.5bar	r61 (original)	r65	r63	r67
	r69 (repeat)	r73	r71	r75
2.5bar	r62	r66	r64	r68
	r70	r74	r72	r76

Thus, the α_{av} values are:

Table B-10: The α_{av} values for the original and repeat experiments

A	B			
	2%		9%	
	C		C	
	10µm	20µm	10µm	20µm
1.5bar	2.383E+09	2.505E+09	1.443E+09	1.713E+09
	2.203E+09	2.273E+09	1.388E+09	1.521E+09
2.5bar	2.666E+09	2.476E+09	1.640E+09	1.662E+09
	2.701E+09	2.520E+09	1.710E+09	1.732E+09

Summing the α_{av} values from the original and repeat runs for each cell:

Table B-11: The total α_{av} values for the original and repeat experiments

A	B			
	2%		9%	
	C		C	
	10µm	20µm	10µm	20µm
1.5bar	4.587E+09	4.778E+09	2.832E+09	3.234E+09
2.5bar	5.368E+09	4.996E+09	3.350E+09	3.393E+09

The BxC totals are needed for the calculation of the sum of squares. To calculate the $y_{\bullet jk \bullet}$ totals for each column, the α_{av} values are summed going down each column (From Table B-11). To calculate the $y_{i \bullet \bullet \bullet}$ totals for each row, the α_{av} values are summed going across each row (From Table B-11).

Table B-12: BxC totals

	B				
	2%		9%		
	C		C		
	10µm	20µm	10µm	20µm	$y_{i \bullet \bullet \bullet}$
1.5bar	4.587E+09	4.778E+09	2.832E+09	3.234E+09	1.543E+10
2.5bar	5.368E+09	4.996E+09	3.350E+09	3.393E+09	1.711E+10
$y_{\bullet jk \bullet}$	9.954E+09	9.774E+09	6.182E+09	6.627E+09	*3.254E+10*

The AxB totals are calculated in order to neglect the effect of factor C. These totals are calculated in a slightly different manner. From Table B-13, the cell in the second row of the second column is found by adding the first two values across the second row of Table B-11. Similarly the cell in the third column of the second row in the Table B-13 is found by adding the last two values in the second row of Table B-11.

Table B-13: AxB Totals

A	$y_{ij\bullet\bullet}$	
	B	
	2%	**9%**
1.5bar	9.364E+09	6.066E+09
2.5bar	1.036E+10	6.743E+09
$y_{\bullet j\bullet\bullet}$	1.973E+10	1.281E+10

To calculate the $y_{\bullet j\bullet\bullet}$ totals for each column in Table B-13, the α_{av} values in Table B-13 are summed going down each column.

The AxC totals are calculated in order to neglect the effect of factor B. From Table B-14, the cell in the second row of the second column is found by adding the first value from the second row in Table B-12 to the third value in the second row of Table B-12. Similarly the cell in the third column of the second row in the Table B-14 is found by adding the second value the second row in Table B-12 to the fourth value in the second row of Table B-12.

Table B-14: AxC Totals

A	$y_{i\bullet k\bullet}$	
	C	
	10µm	**20µm**
1.5bar	7.418E+09	8.011E+09
2.5bar	8.718E+09	8.389E+09
$y_{\bullet\bullet k\bullet}$	1.614E+10	1.640E+10

To calculate the $y_{\bullet\bullet k\bullet}$ totals for each column in Table B-14, the α_{av} values in Table B-14 are summed going down each column.

Now that all totals are computed, the sums of squares can be found using Equations (2-29) to (2-37).

$$SS_T = (2.382x10^9)^2 + (2.203x10^9)^2 + (2.666x10^9)^2 + (2.701x10^9)^2 + (2.505x10^9)^2$$
$$+ (2.273x10^9)^2 + (2.476x10^9)^2 + (2.520x10^9)^2 + (1.443x10^9)^2$$
$$+ (1.389x10^9)^2 + (1.640x10^9)^2 + (1.710x10^9)^2 + (1.713x10^9)^2$$
$$+ (1.521x10^9)^2 + (1.662x10^9)^2 + (1.732x10^9)^2 - \frac{(3.254x10^{10})^2}{16}$$

= 3.328 x 10^{18}

$$SS_A = \frac{(1.543x10^{10})^2 + (1.711x10^{10})^2}{(2)(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$

= 1.758 x 10^{17}

$$SS_B = \frac{(1.973x10^{10})^2 + (1.281x10^{10})^2}{(2)(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$

= 2.992 x 10^{18}

$$SS_C = \frac{(1.614x10^{10})^2 + (1.640x10^{10})^2}{(2)(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$

= 4.382 x 10^{15}

$$SS_{AB} = \frac{(9.364x10^9)^2 + (1.036x10^{10})^2 + (6.066x10^{10})^2 + (6.743x10^9)^2}{(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$
$$- 1.758x10^{17} - 2.992x10^{18}$$

= 6.456 x 10^{15}

$$SS_{AC} = \frac{(7.418x10^9)^2 + (8.718x10^9)^2 + (8.011x10^9)^2 + (8.389x10^9)^2}{(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$
$$- 1.758x10^{17} - 4.382x10^{15}$$

= 5.308 x 10^{16}

$$SS_{BC} = \frac{(9.954x10^9)^2 + (9.774x10^9)^2 + (6.182x10^9)^2 + (6.627x10^9)^2}{(2)(2)} - \frac{(3.254x10^{10})^2}{16}$$

$$- 2.992x10^{18} - 4.382x10^{15}$$

= 2.453 x 10^{16}

$$SS_{ABC} = \frac{\left[\begin{array}{c}(4.628x10^9)^2 + (5.368x10^9)^2 + (4.778x10^9)^2 + (4.996x10^9)^2 + \\ (2.832x10^9)^2 + (3.350x10^9)^2 + (3.234x10^9)^2 + (3.393x10^9)^2\end{array}\right]}{(2)}$$

$$- \frac{(3.254x10^{10})^2}{16} - 1.758x10^{17} - 2.992x10^{18} - 4.382x10^{15} - 6.456x10^{15}$$

$$- 5.308x10^{16} - 2.453x10^{16}$$

= 2.595 x 10^{15}

$$SS_E =$$
$$3.328x10^{18} - 1.758x10^{17} - 2.992x10^{18} - 4.382x10^{15} - 6.456x10^{15} - 5.308x10^{16} -$$
$$2.453x10^{16} - 2.595x10^{15}$$

= 6.936 x 10^{16}

The degrees of freedom are determined as indicated by Table 2-2, where a = 2 levels, b = 2 levels, c = 2 levels and n = 2 replicates.

Degree of freedom of A = 2-1 = 1

Degree of freedom of B = 2-1 = 1

Degree of freedom of C = 2-1 = 1

Degree of freedom of AB = (2-1)(2-1) = 1

Degree of freedom of AC = (2-1)(2-1) = 1

Degree of freedom of BC = (2-1)(2-1) = 1

Degree of freedom of ABC = (2-1)(2-1)(2-1) = 1

Degree of freedom of $Error$ = (2)(2)(2)(2-1) = 8

Degree of freedom of $Total$ = [(2)(2)(2)(2)] – 1 = 15

The mean square is calculated for each term according to Equations (2-38) to (2-45):

$$MS_A = \frac{1.758x10^{17}}{2-1}$$

$$= 1.758x10^{17}$$

$$MS_B = \frac{2.992x10^{18}}{2-1}$$

$$= 2.992x10^{18}$$

$$MS_C = \frac{4.382x10^{15}}{2-1}$$

$$= 4.382x10^{15}$$

$$MS_{AB} = \frac{6.456x10^{15}}{2-1}$$

$$= 6.456x10^{15}$$

$$MS_{AC} = \frac{5.308x10^{16}}{2-1}$$

$$= 5.308x10^{16}$$

$$MS_{BC} = \frac{2.453x10^{16}}{2-1}$$

$$= 2.453x10^{16}$$

$$MS_{ABC} = \frac{2.595x10^{15}}{2-1}$$

$$= 2.595x10^{15}$$

$$MS_E = \frac{6.936x10^{16}}{(2)(2)(2)(2-1)}$$

$$= 8.670x10^{15}$$

The F-statistic is then calculated for the terms according to the equations specified in Table 2-2 in section 2.10.2.2

$$F\ statistic\ of\ A = \frac{1.758x10^{17}}{8.670x10^{15}}$$

$$= 20.279$$

$$F\ statistic\ of\ B = \frac{2.992x10^{18}}{8.670x10^{15}}$$

$$= 345.087$$

$$F\ statistic\ of\ C = \frac{4.382x10^{15}}{8.670x10^{15}}$$

$$= 0.505$$

$$F\ statistic\ of\ AB = \frac{6.456x10^{15}}{8.670x10^{15}}$$

$$= 0.745$$

$$F\ statistic\ of\ AC = \frac{5.308x10^{16}}{8.670x10^{15}}$$

$$= 6.122$$

$$F\ statistic\ of\ BC = \frac{2.453x10^{16}}{8.670x10^{15}}$$

$$= 2.829$$

$$F\ statistic\ of\ ABC = \frac{2.595x10^{15}}{8.670x10^{15}}$$

$$= 0.299$$

The *ANOVA* table can now be constructed as indicated by Table 2-2 in Chapter 2, section 2.10. The p-values are obtained from *Microsoft Office Excel®* by using the "*FDIST*" command.

Table B-15: *ANOVA* table for the two-phase experiments with respect to α_{av}

Source of variation	Sums of squares	Degrees of freedom	Mean squares	F-statistic	p value
A	1.758E+17	1	1.758E+17	20.279	**0.001994**
B	2.992E+18	1	2.992E+18	345.087	**7.272E-08**
C	4.382E+15	1	4.382E+15	0.505	0.4973
AB	6.456E+15	1	6.456E+15	0.745	0.4133
AC	5.308E+16	1	5.308E+16	6.122	**0.0385**
BC	2.453E+16	1	2.453E+16	2.829	0.1311
ABC	2.595E+15	1	2.595E+15	0.299	0.5993
Error	6.936E+16	8	8.670E+15		
Total	3.328E+18	15			

From the *ANOVA* table shown above, the significant effects are *A*, *B* and *AC*. Thus there will be 4 terms in the regression model, including the intercept.

B.5.3. The regression model

The coefficients for each term in the regression model is found by merely dividing the total effect found for each term in section E (Table B-8) by 2.

$$\beta_0 = \frac{\begin{bmatrix} 2.383x10^9 + 2.203x10^9 + 2.666x10^9 + 2.701x10^9 + 2.505x10^9 + 2.273x10^9 + \\ 2.476x10^9 + 2.520x10^9 + 1.443x10^9 + 1.389x10^9 + 1.640x10^9 + \\ 1.710x10^9 + 1.713x10^9 + 1.521x10^9 + 1.662x10^9 + 1.732x10^9 \end{bmatrix}}{16}$$

= 2.034 x 10^9

$$\beta_1 = \frac{2.097x10^8}{2}$$

= 1.048 x 10^8

$$\beta_2 = \frac{-8.648x10^8}{2}$$

= -4.324 x 10^8

$$\beta_{13} = \frac{-1.152x10^8}{2}$$

= -5.760 x 10^7

The regression equation is thus:

$$y = 2.034x10^9 + (1.048x10^8)x_1 + (-4.324x10^8)x_2 + (-5.760x10^7)x_1x_3$$

Where y is the dependant variable, in this case α_{av}

x_1 is the term representing the effect of factor A (i.e. the applied pressure)

x_2 is the term representing the effect of factor B (i.e. the solids concentration)

x_{13} is the term representing the combination effect of factors A and C (i.e. the applied pressure and filter cloth pore size)

B.5.4. Normal probability plot

This regression equation can now be used to predict output values of α_{av}. By finding the difference between this predicted value and the actual value obtained, the residual can be found.

This range of residuals is used to construct a normal probability plot which indicates the fit of the regression model.

The regression model fitted for the case mentioned found that the significant terms were the pressure, solids concentration and, the pressure and filter cloth pore size interaction. The high levels of these terms are assigned values of +1 and the low levels of these terms are assigned values of -1. Thus for each run that was conducted, the levels of these variables can be indicated:

Table B-16: Levels of the dependant variables

Run number	Treatment combination	x_1	x_2	x_3	x_1x_3
1	1	-1	-1	-1	1
2	a	1	-1	-1	-1
3	b	-1	1	-1	1
4	ab	1	1	-1	-1
5	c	-1	-1	1	-1
6	ac	1	-1	1	1
7	bc	-1	1	1	-1
8	abc	1	1	1	1

These levels can now be substituted into the regression model to calculate a predicted value for the α_{av} value for each experiment, along with the repeat experiments.

For example, for Run 61, the predicted α_{av} value is:

$$y =$$
$$2.034x10^9 + (1.048x10^8)(-1) + (-4.324x10^8)(-1) + (-5.760x10^7)(-1)(-1)$$
$$= 2.304 \text{ x } 10^9.$$

This can be used to determine the residual value for this run. The residual is found by subtracting the predicted value from the value obtained from the actual run. This can be done for each run and its repeat:

For Run 61,
$e = 2.383 \text{ x } 10^9 - 2.304 \text{ x } 10^9 = 7.976 \text{ x } 10^7$
For the repeat run of Run 61 (i.e. Run 69):
$e = 2.203 \text{ x } 10^9 - 2.304 \text{ x } 10^9 = -1.002 \text{ x } 10^8$

These results are tabulated as follows:

Table B-17: Actual and predicted values and the residual values for the two-phase experiments with respect to α_{av}

Run Number	Actual result	Predicted result	Residual
1	2.383E+09	2.304E+09	7.976E+07
1 repeat	2.203E+09	2.304E+09	-1.002E+08
2	2.666E+09	2.628E+09	3.806E+07
2 repeat	2.701E+09	2.628E+09	7.278E+07
3	1.443E+09	1.439E+09	4.634E+06
3 repeat	1.388E+09	1.439E+09	-5.039E+07
4	1.640E+09	1.764E+09	-1.234E+08
4 repeat	1.710E+09	1.764E+09	-5.360E+07
5	2.505E+09	2.419E+09	8.598E+07
5 repeat	2.273E+09	2.419E+09	-1.459E+08
6	2.476E+09	2.513E+09	-3.713E+07
6 repeat	2.520E+09	2.513E+09	6.629E+06
7	1.713E+09	1.554E+09	1.588E+08
7 repeat	1.521E+09	1.554E+09	-3.268E+07
8	1.662E+09	1.648E+09	1.339E+07
8 repeat	1.732E+09	1.648E+09	8.331E+07

These residual values are then listed in ascending order and plotted on a normal probability scale to indicate the fit of the data. This is shown below:
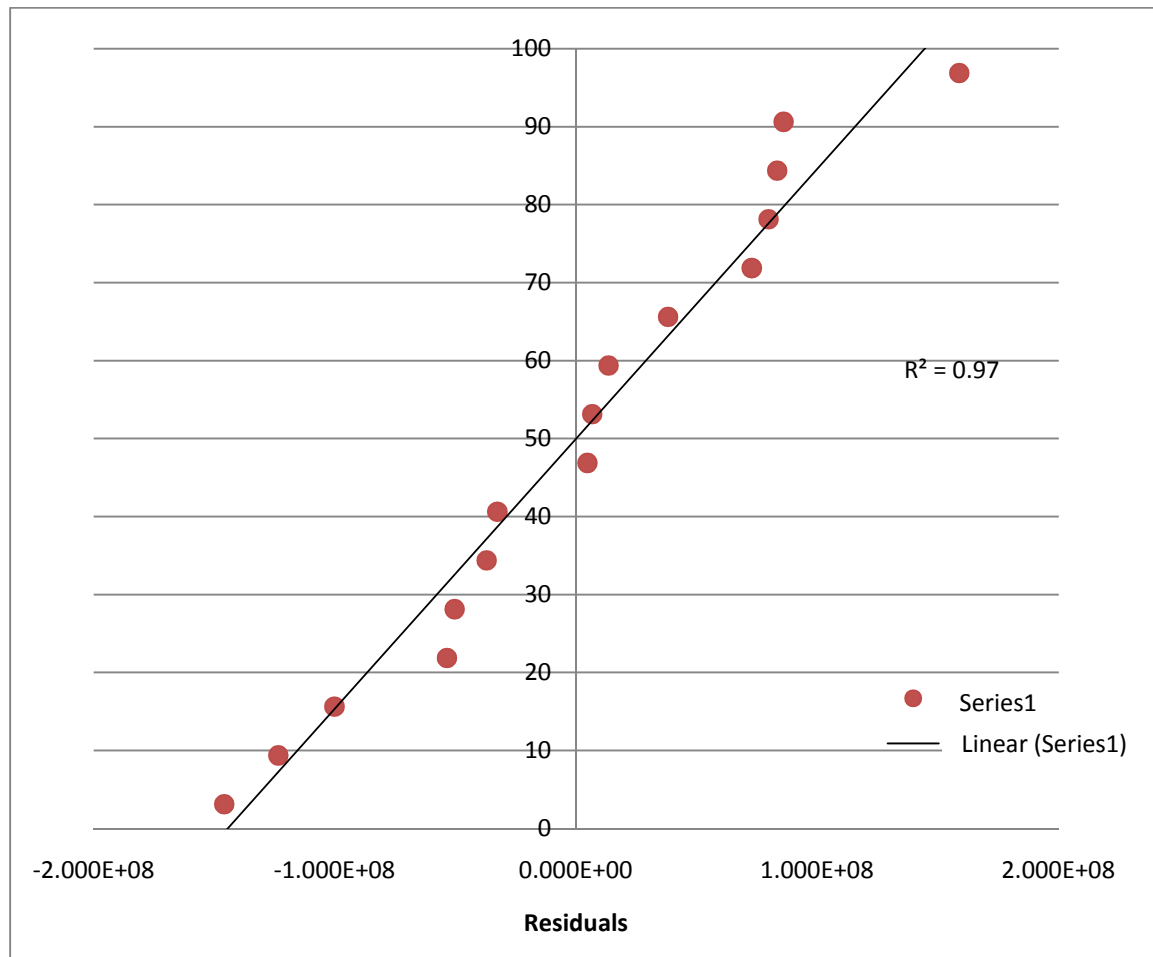
Figure B-5: Normal probability plot for the two-phase experiments with respect to α_{gv}

A correlation between the actual values and the values predicted from the model was also conducted in *Microsoft Office Excel®* by using the *"RSQ"* command. This found the correlation coefficient between these two outputs. The correlation coefficient for this case was found to be 0.97.

APPENDIX C:
GRAPHS

C.1. Cake formation rate curves

C.1.1. Experiments in which the solids concentration was varied whilst the applied pressure and filter cloth pore size was fixed
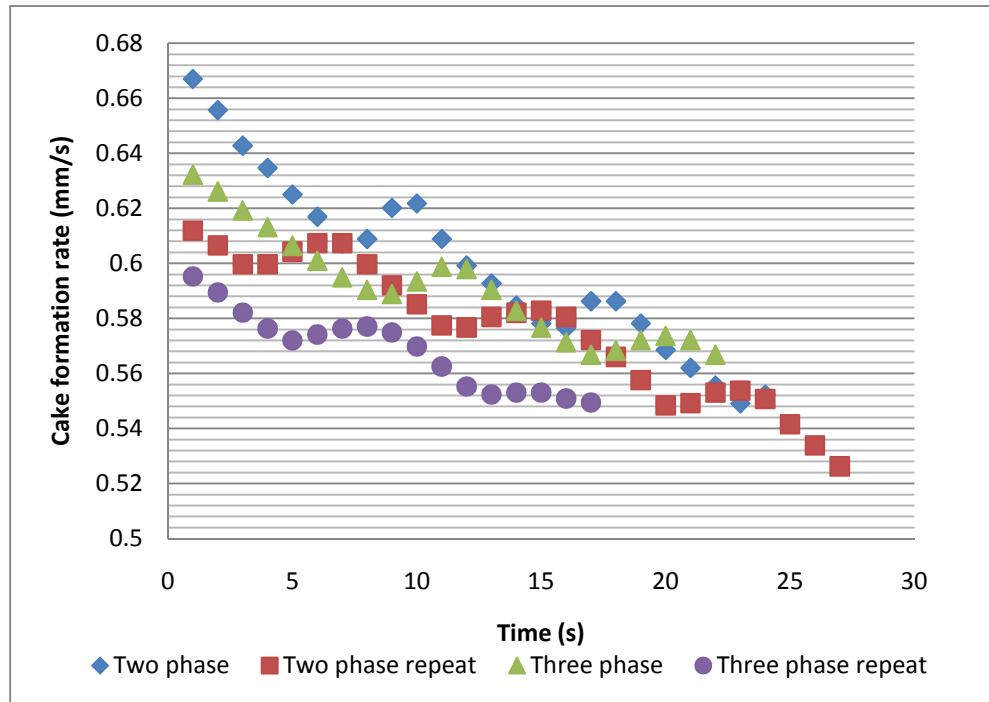
Figure C-1: Cake formation rate curve for a solids concentration of 1% (mass/mass) (Runs 1, 11, 21 and 31)
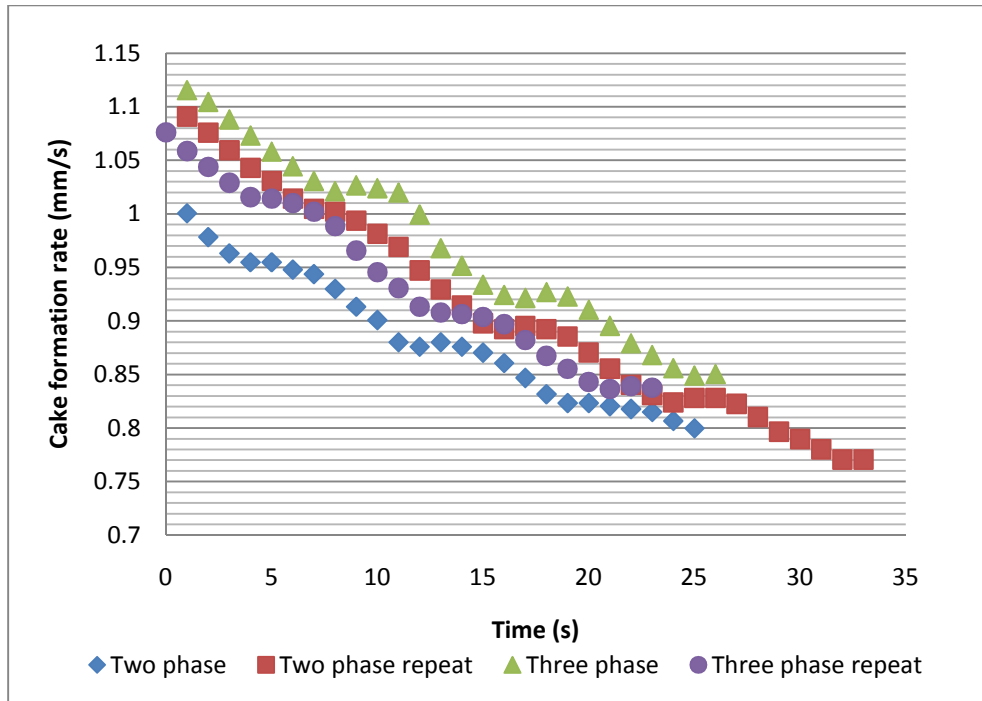
Figure C-2: Cake formation rate curve for a solids concentration of 2% (mass/mass) (Runs 2, 12, 22 and 32)
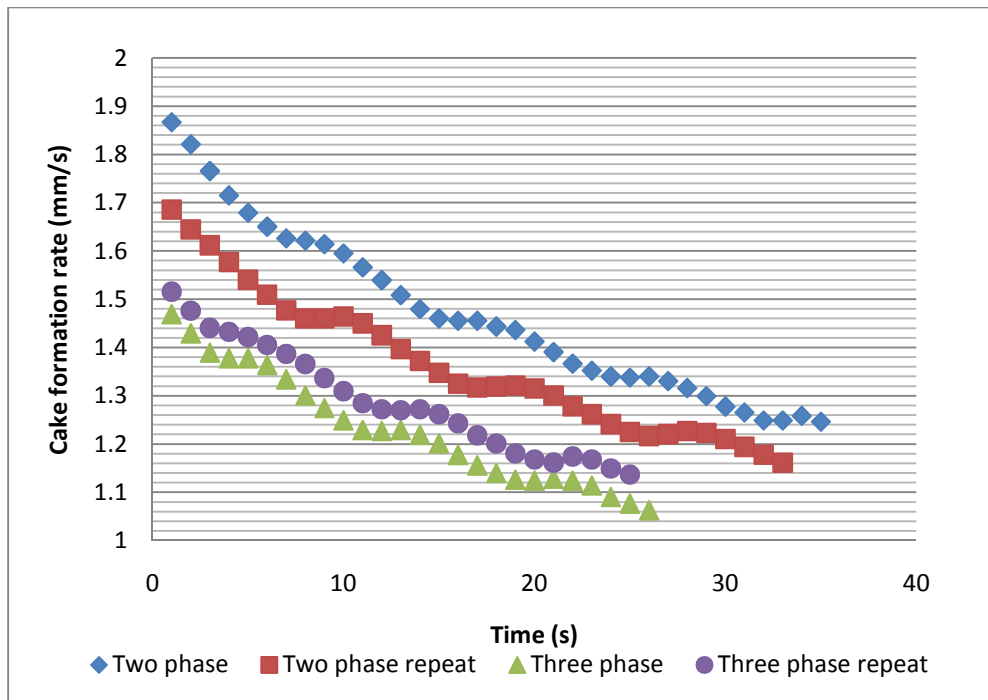
Figure C-3: Cake formation rate curve for a solids concentration of 3% (mass/mass) (Runs 3, 13, 23 and 33)
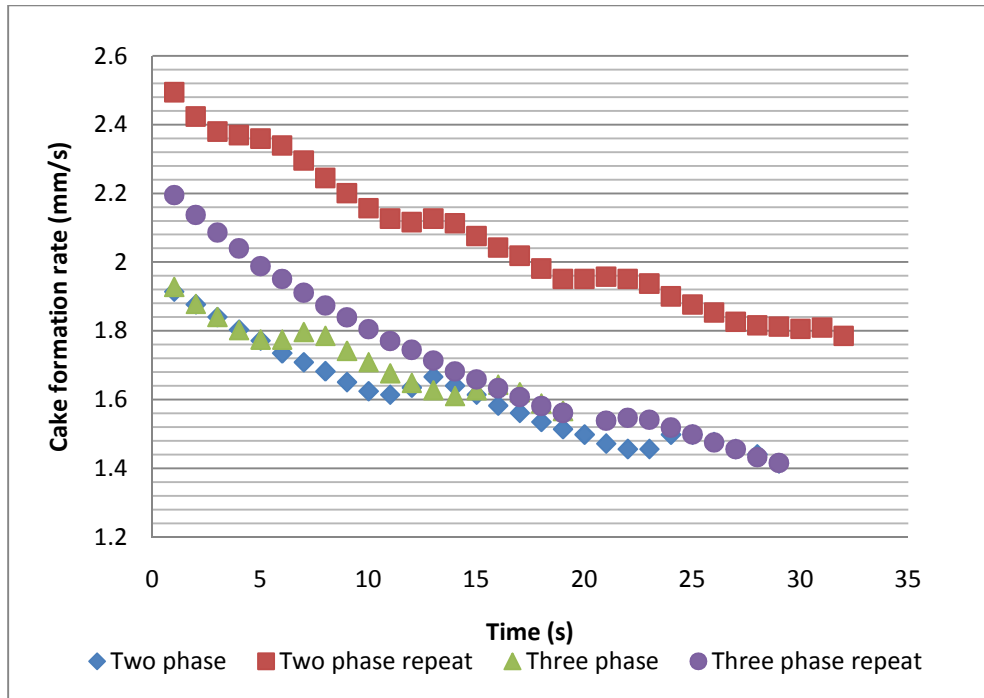
Figure C-4: Cake formation rate curve for a solids concentration of 4% (mass/mass) (Runs 4, 14, 24 and 34)
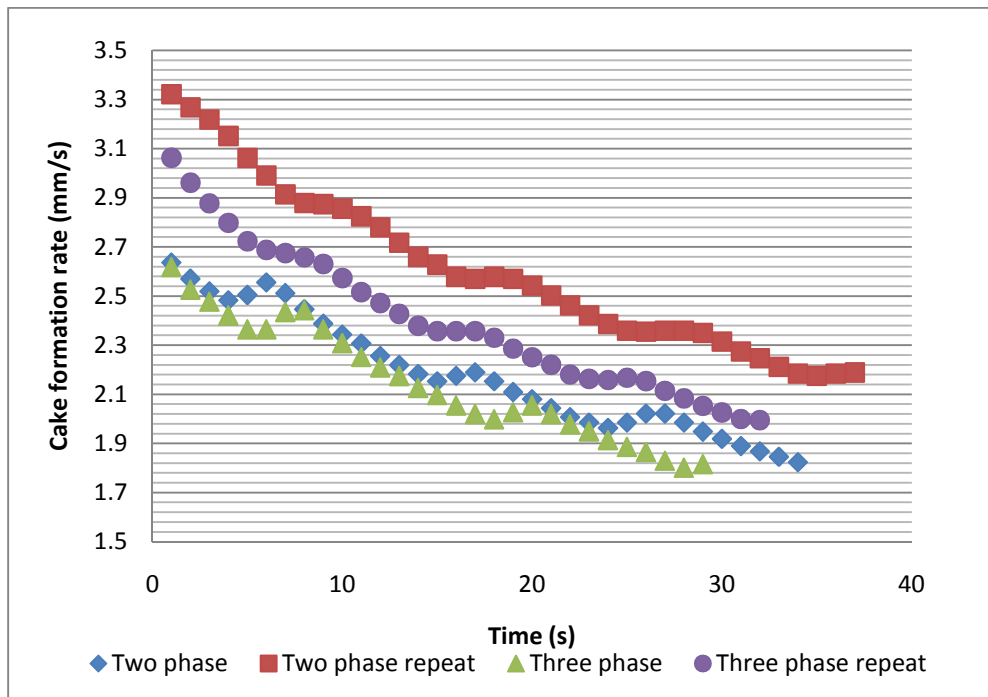
Figure C-5: Cake formation rate curve for a solids concentration of 5% (mass/mass) (Runs 5, 15, 25 and 35)
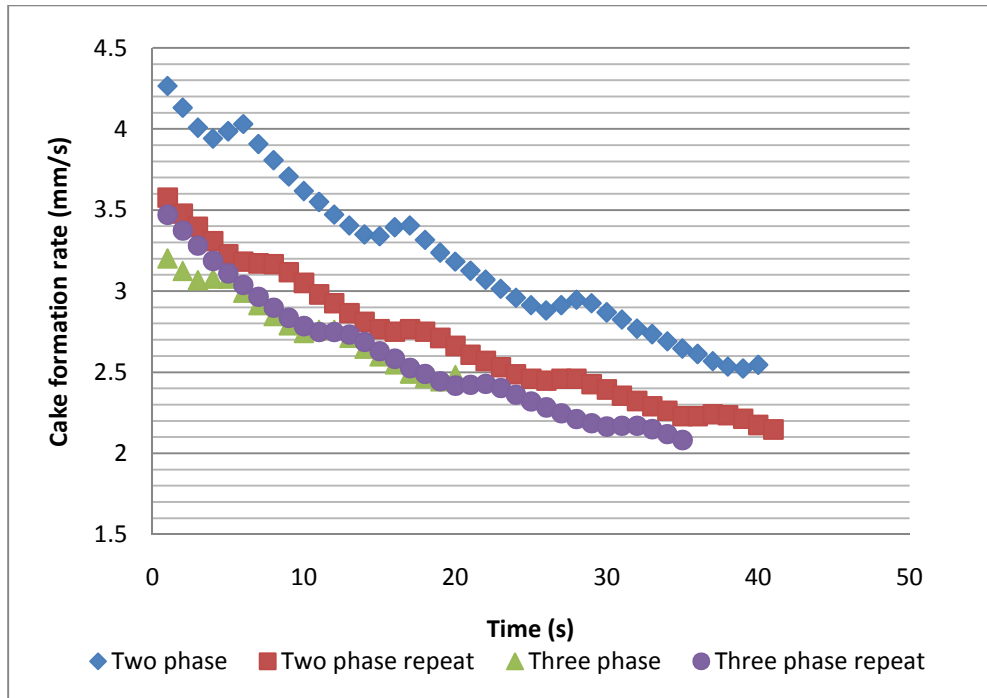
Figure C-6: Cake formation rate curve for a solids concentration of 6% (mass/mass) (Runs 6, 16, 26 and 36)
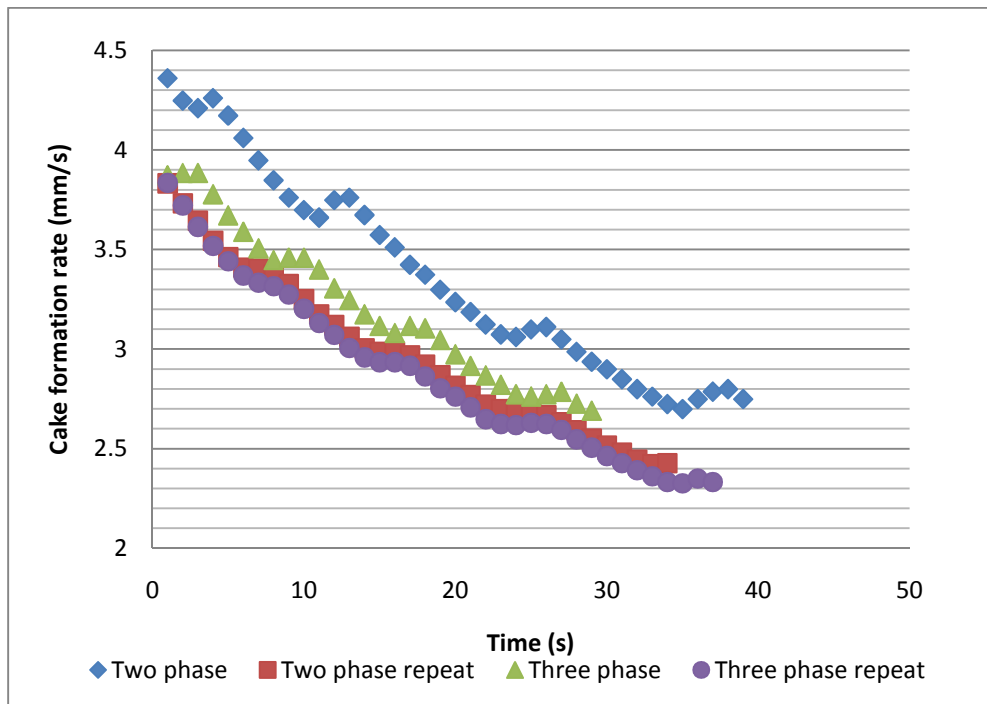
Figure C-7: Cake formation rate curve for a solids concentration of 7% (mass/mass) (Runs 7, 17, 27 and 37)
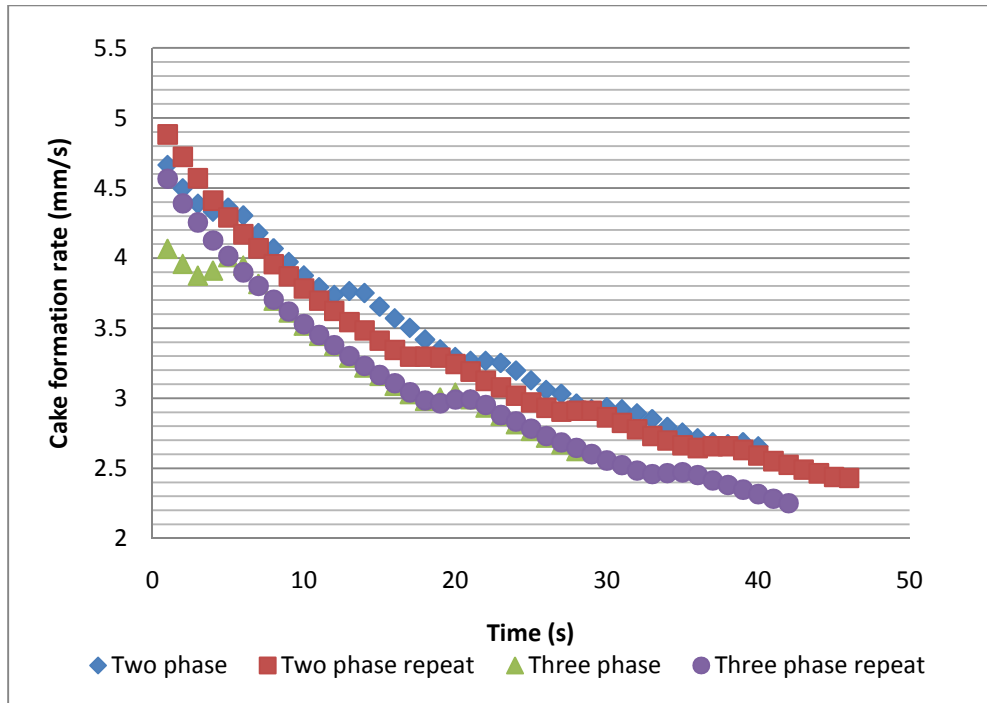
Figure C-8: Cake formation rate curve for a solids concentration of 8% (mass/mass) (Runs 8, 18, 28 and 38)
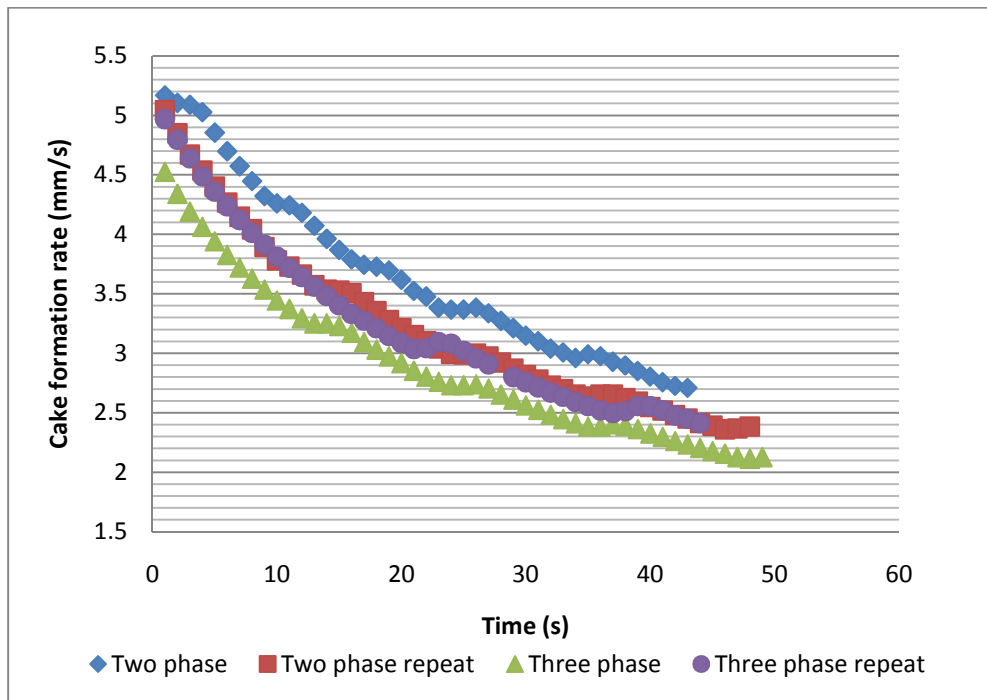
Figure C-9: Cake formation rate curve for a solids concentration of 9% (mass/mass) (Runs 9, 19, 29 and 39)
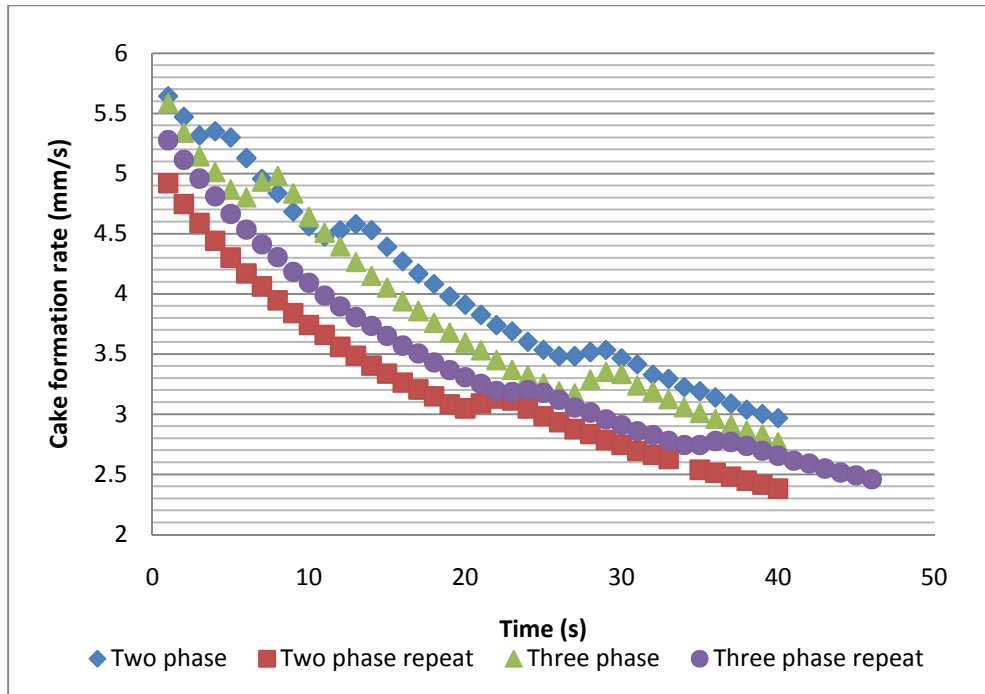
Figure C-10: Cake formation rate curve for a solids concentration of 10% (mass/mass) (Runs 10, 20, 30 and 40)

C.1.2. Experiments in which the applied pressure was varied whilst the solids concentration and filter cloth pore size was fixed
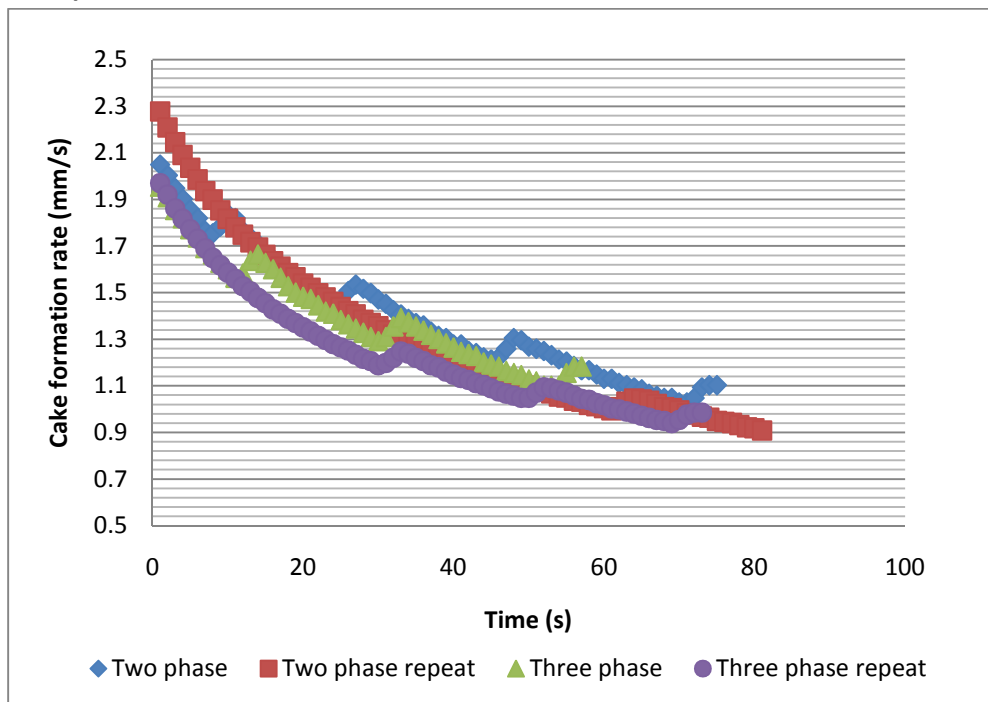
Figure C-11: Cake formation rate curve for an applied pressure of 1.5bar abs (Runs 41, 44, 47 and 50)Figure
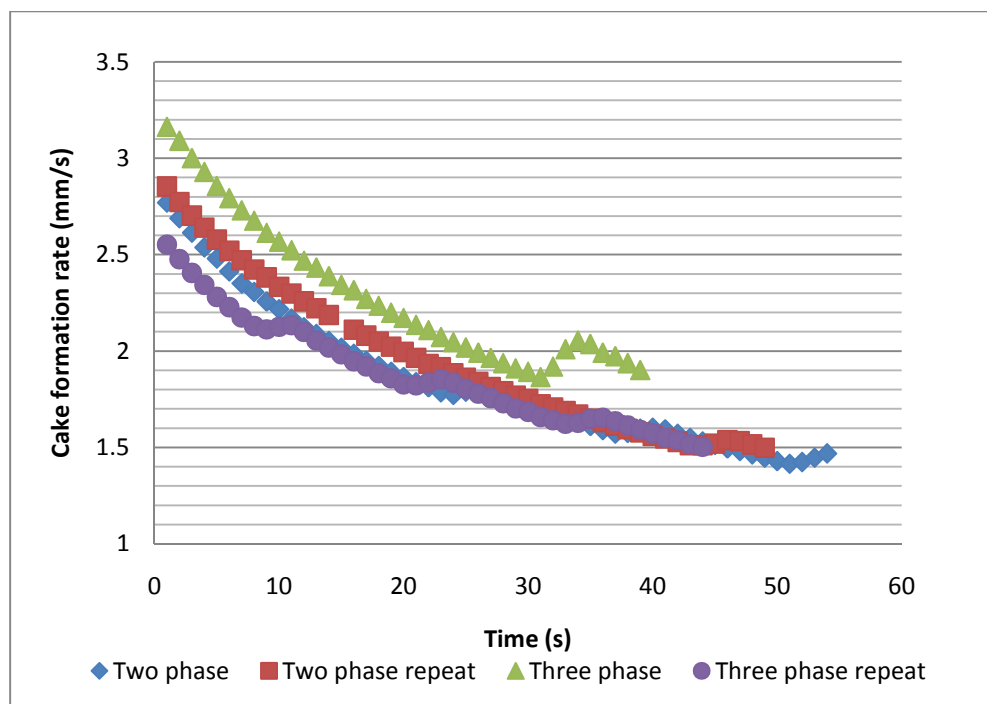
C-12: Cake formation rate curve for an applied pressure of 2.0bar abs (Runs 42, 45, 48 and 51)
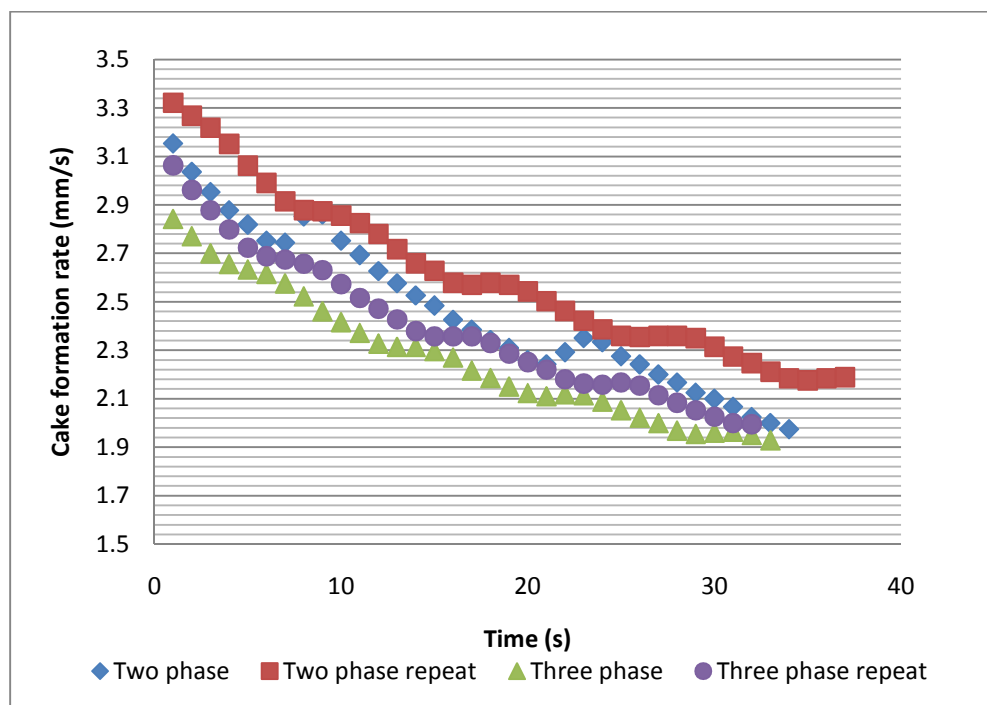
Figure C-13: Cake formation rate curve for an applied pressure of 2.5bar abs (Runs 43, 46, 49 and 52)

C.1.3. Experiments in which the filter cloth pore size was varied whilst the solids concentration and applied pressure was fixed
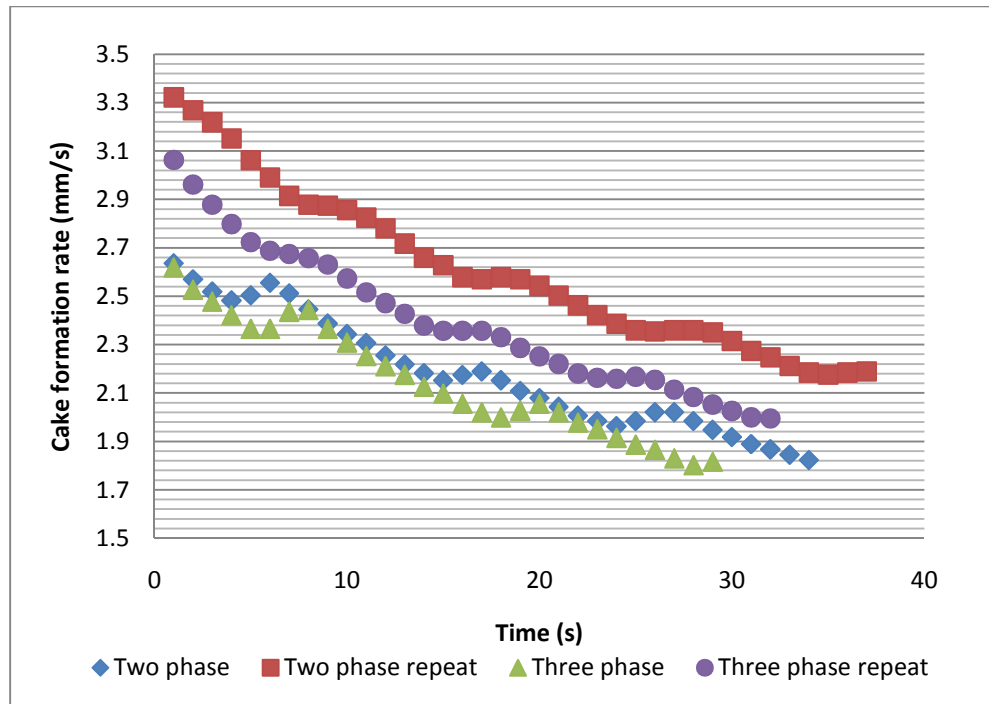
Figure C-14: Cake formation rate curve for a filter cloth pore size of 10μm (Runs 53, 55, 57 and 59)
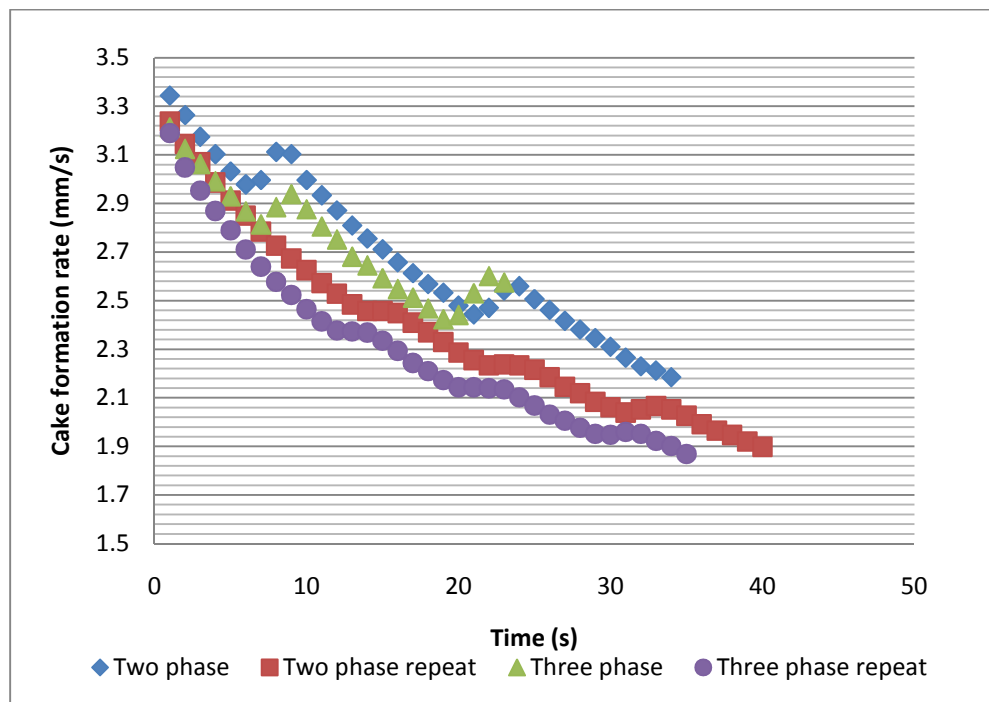
Figure C-15: Cake formation rate curve for a filter cloth pore size of 20μm (Runs 54, 56, 58 and 60)

C.1.4. Factorial experiments
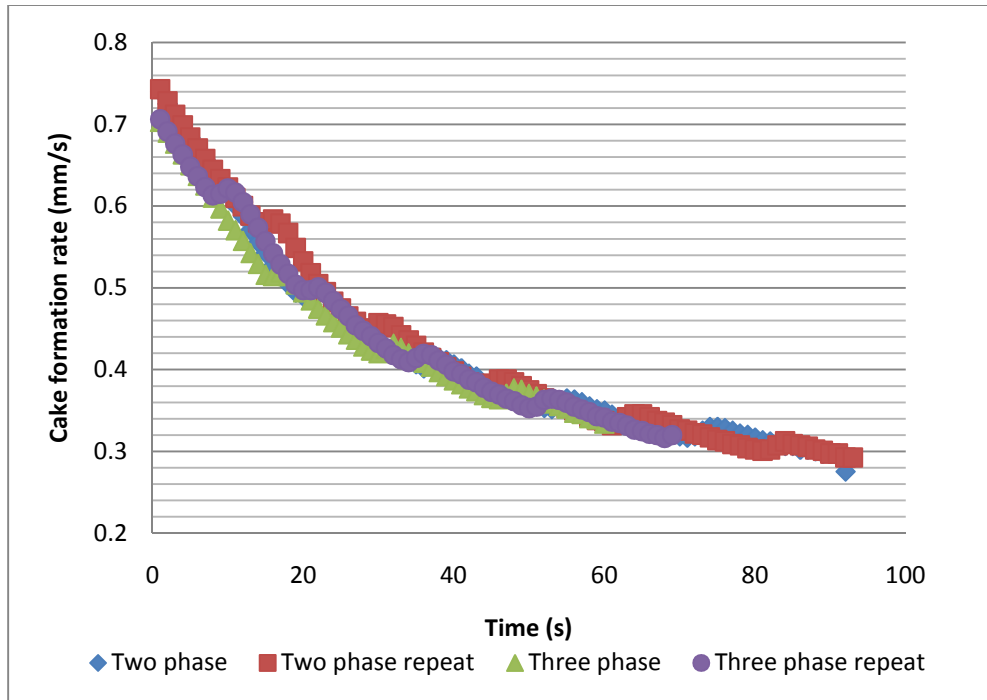
Figure C-16: Cake formation rate curve for factorial run 1 (Runs 61, 69, 77 and 85)
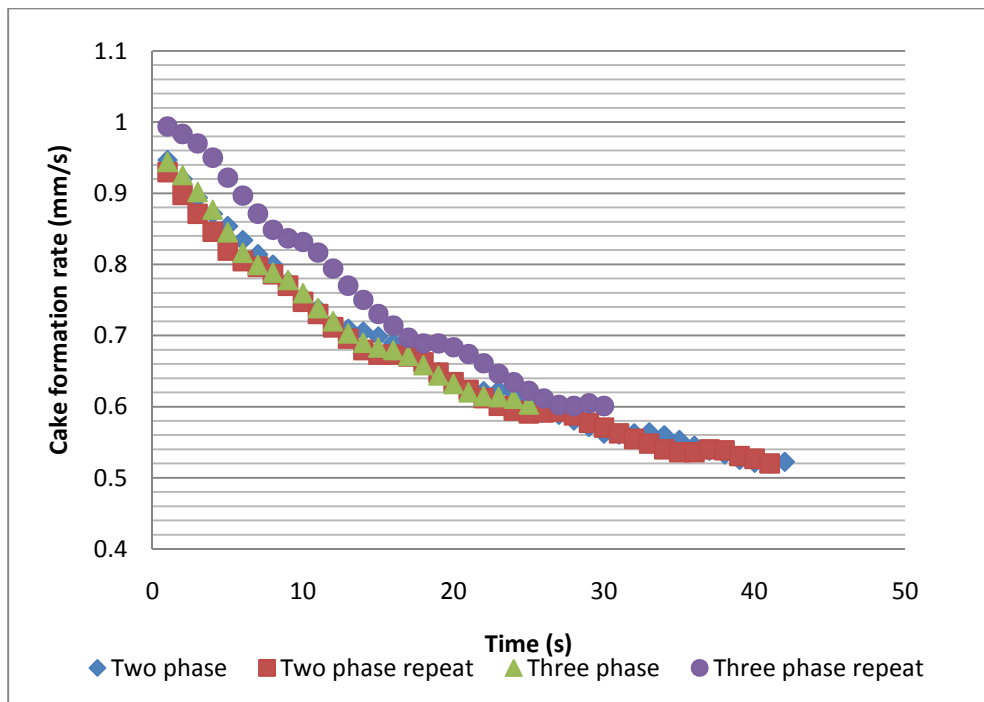
Figure C-17: Cake formation rate curve for factorial run 2 (Runs 62, 70, 78 and 86)
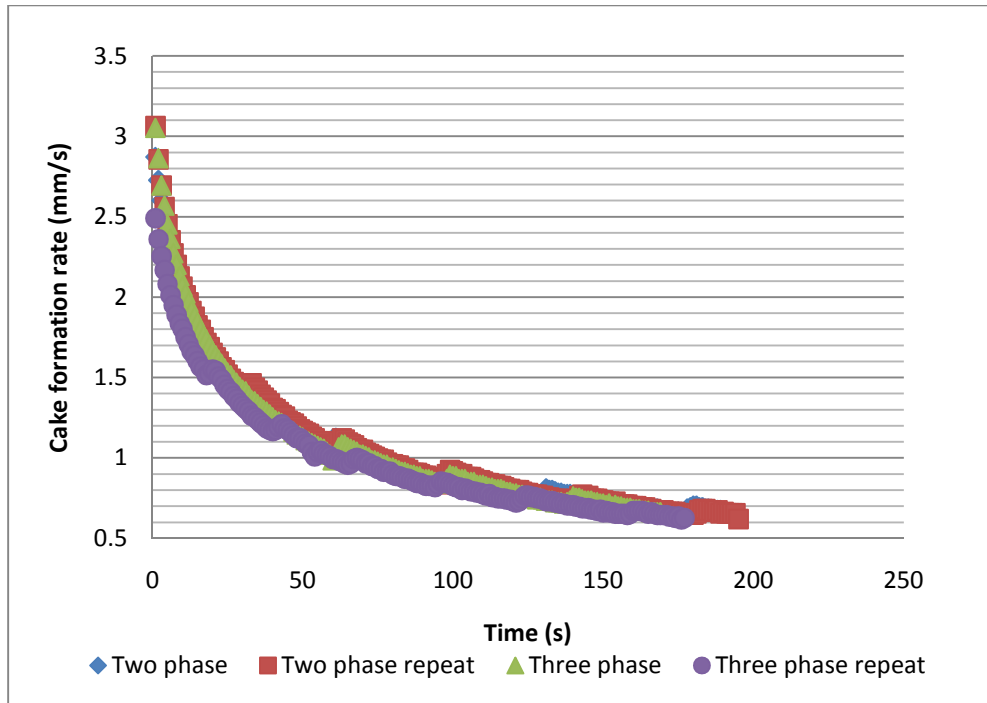
Figure C-18: Cake formation rate curve for factorial run 3 (Runs 63, 71, 79 and 87)
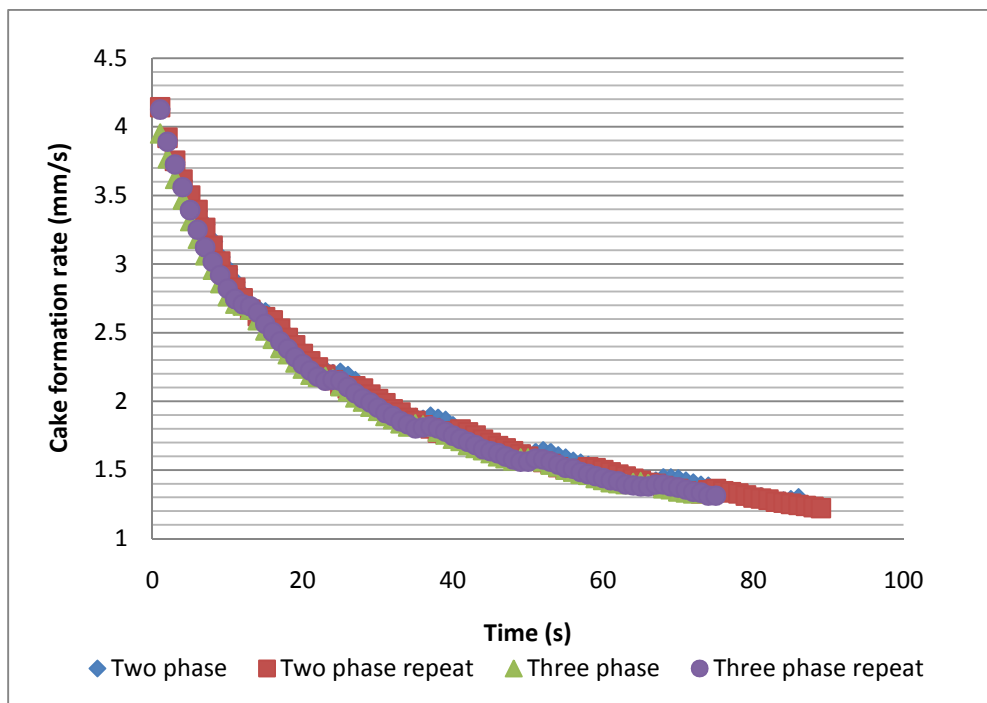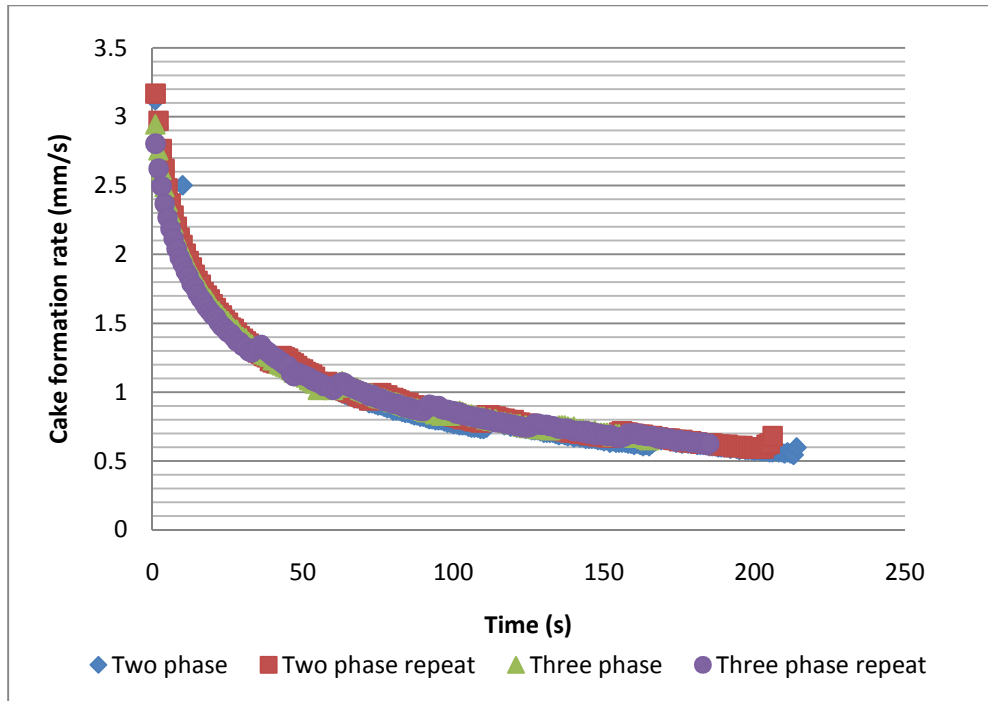

Figure C-19: Cake formation rate curve for factorial run 4 (Runs 64, 72, 80 and 88)
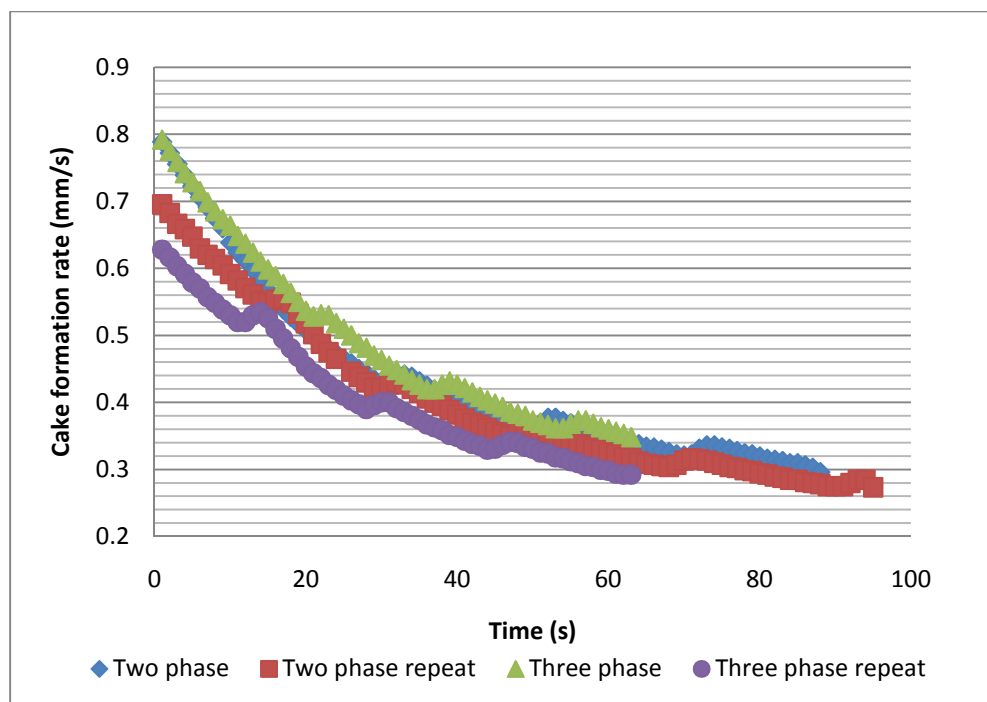
Figure C-20: Cake formation rate curve for factorial run 5 (Runs 65, 73, 81 and 89)
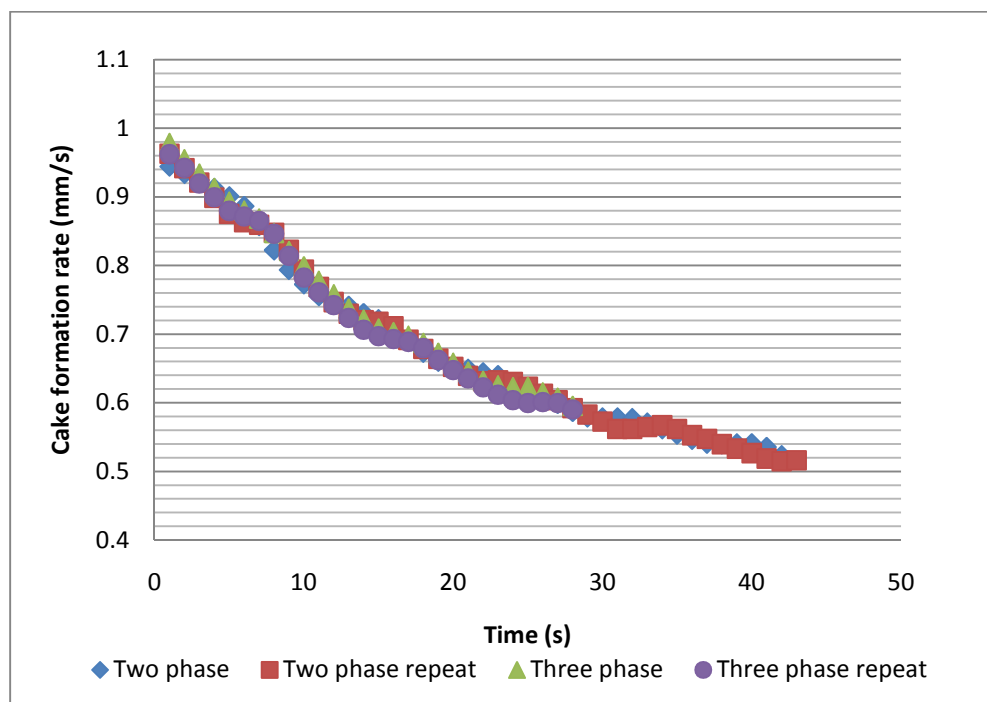
Figure C-21: Cake formation rate curve for factorial run 6 (Runs 66, 74, 82 and 90)

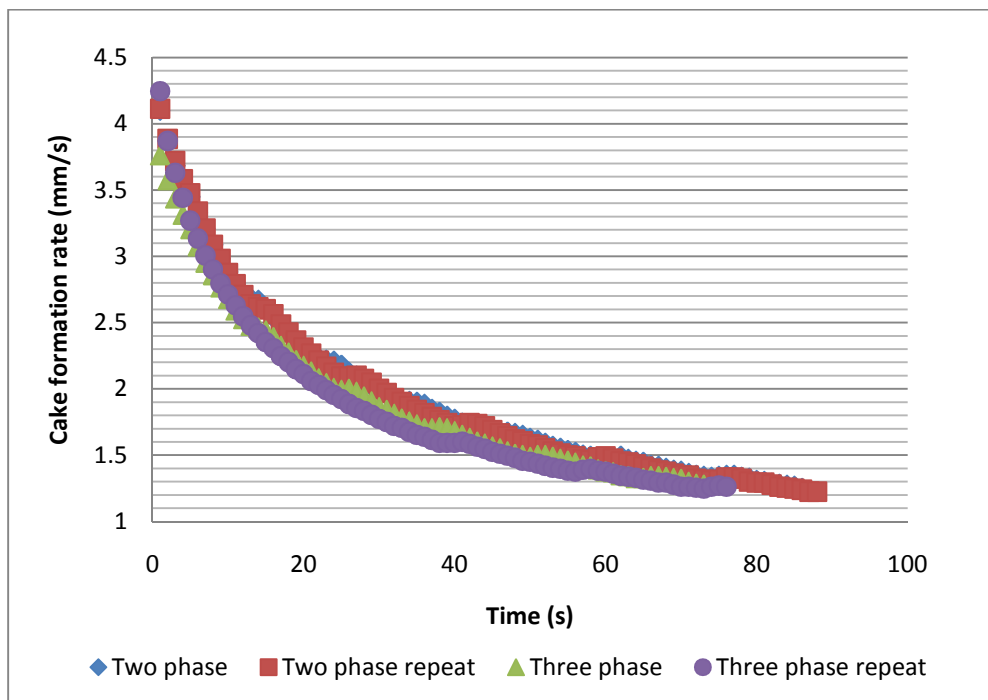Figure C-22: Cake formation rate curve for factorial run 7 (Runs 67, 75, 83 and 91)

Figure C-23: Cake formation rate curve for factorial run 8 (Runs 68, 76, 84 and 92)

C.2. Filtration characteristic curves
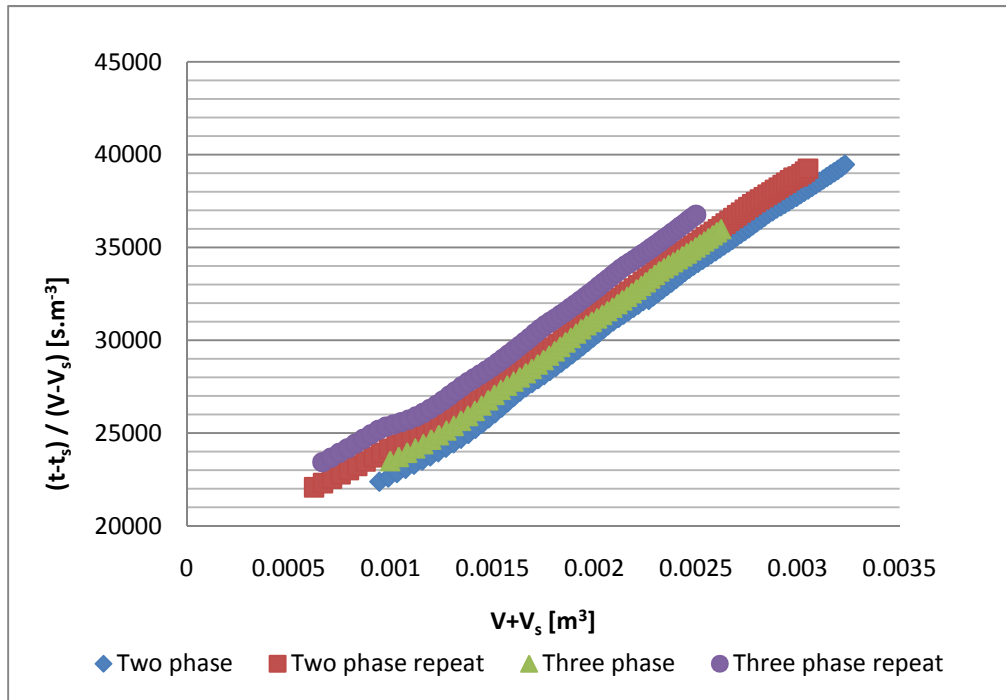
C.2.1. Factorial experiments

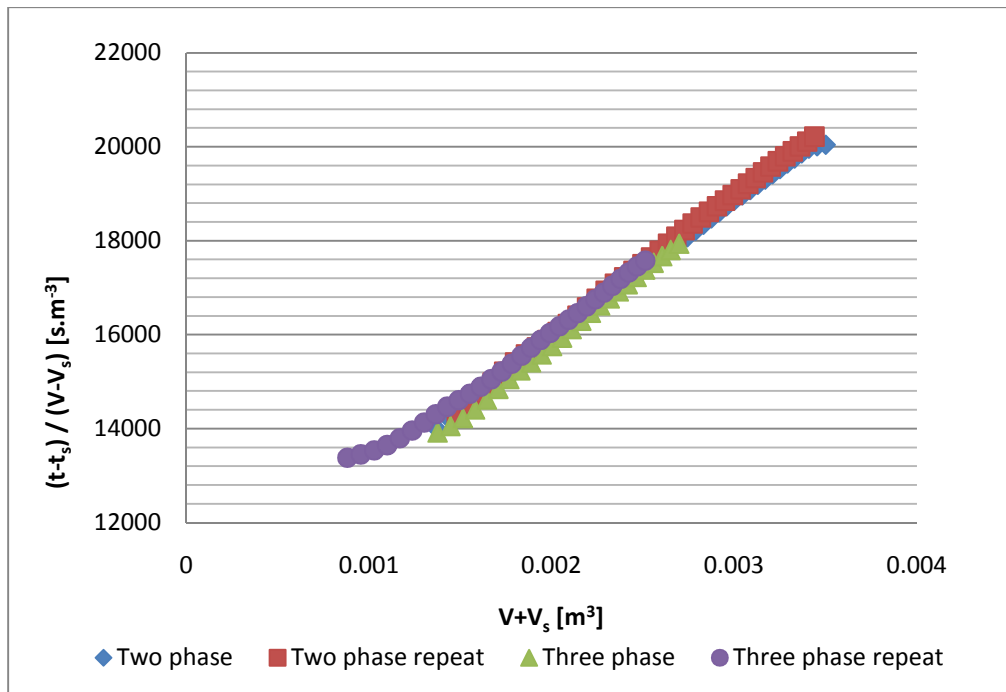Figure C-24: Filtration characteristic curve for factorial run 1 (Runs 61, 69, 77 and 85)

Figure C-25: Filtration characteristic curve for factorial run 2 (Runs 62, 70, 78 and 86)
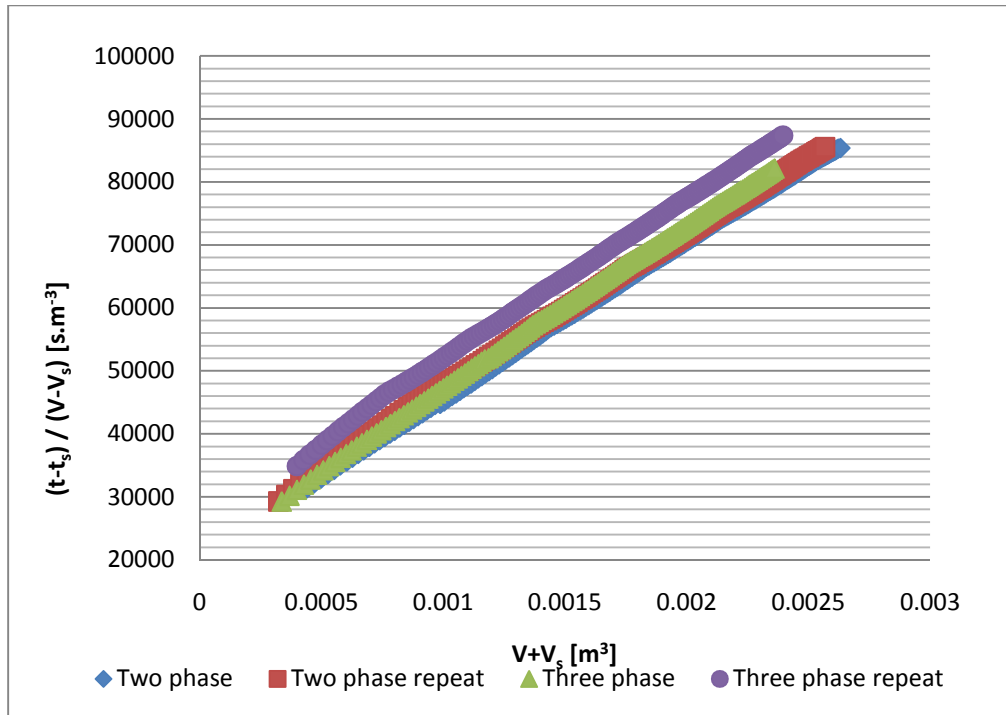
Figure C-26: Filtration characteristic curve for factorial run 3 (Runs 63, 71, 79 and 87)
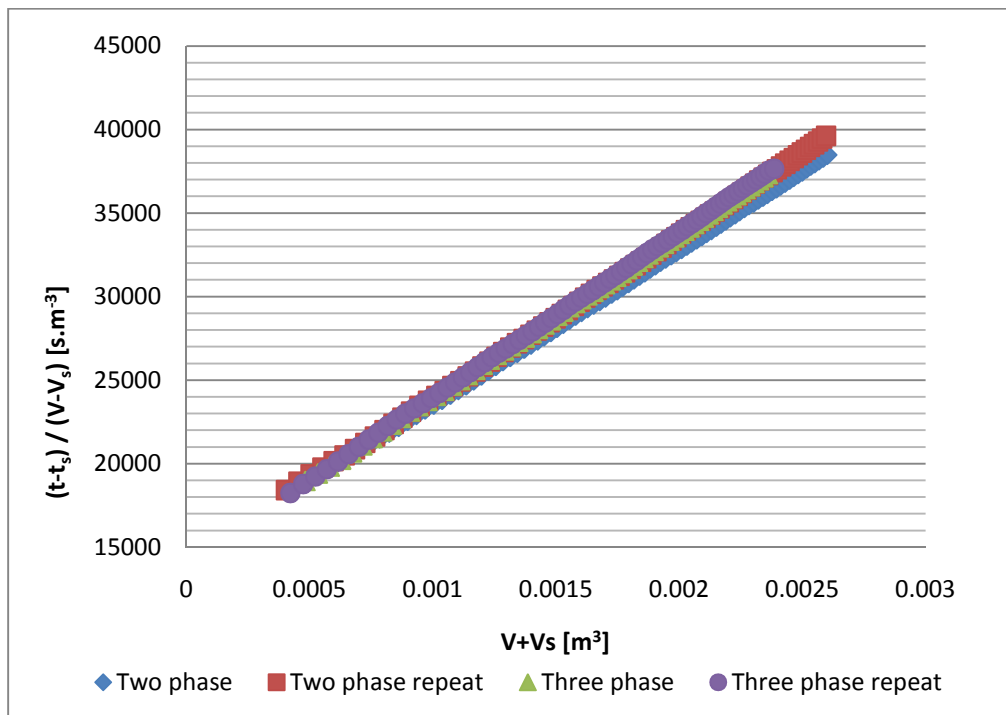

Figure C-27: Filtration characteristic curve for factorial run 4 (Runs 64, 72, 80 and 88)
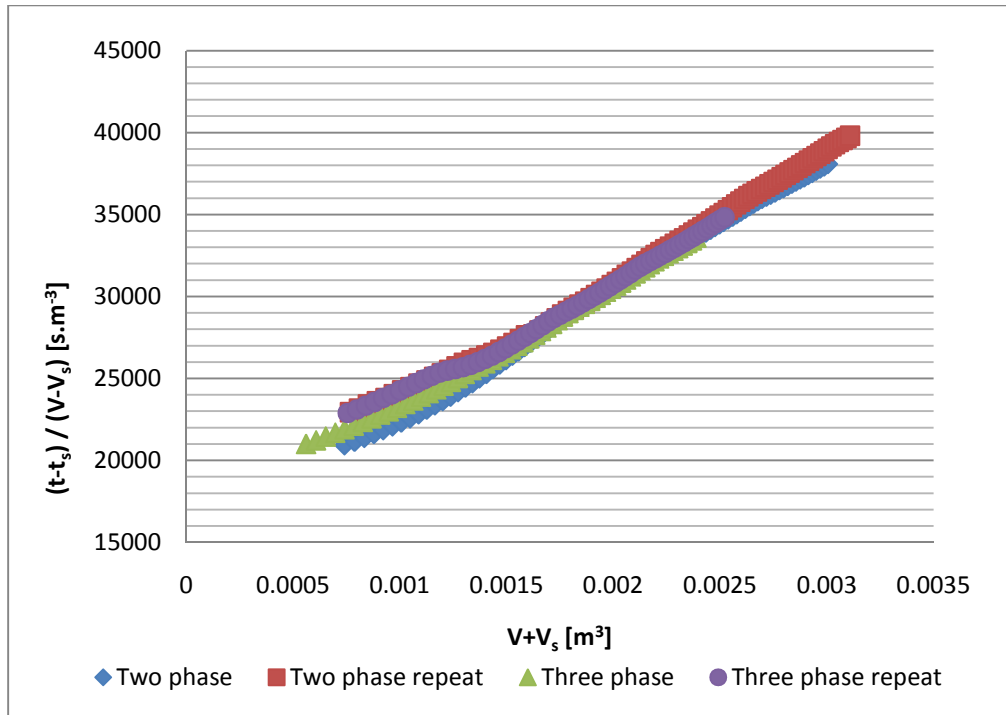
Figure C-28: Filtration characteristic curve for factorial run 5 (Runs 65, 73, 81 and 89)
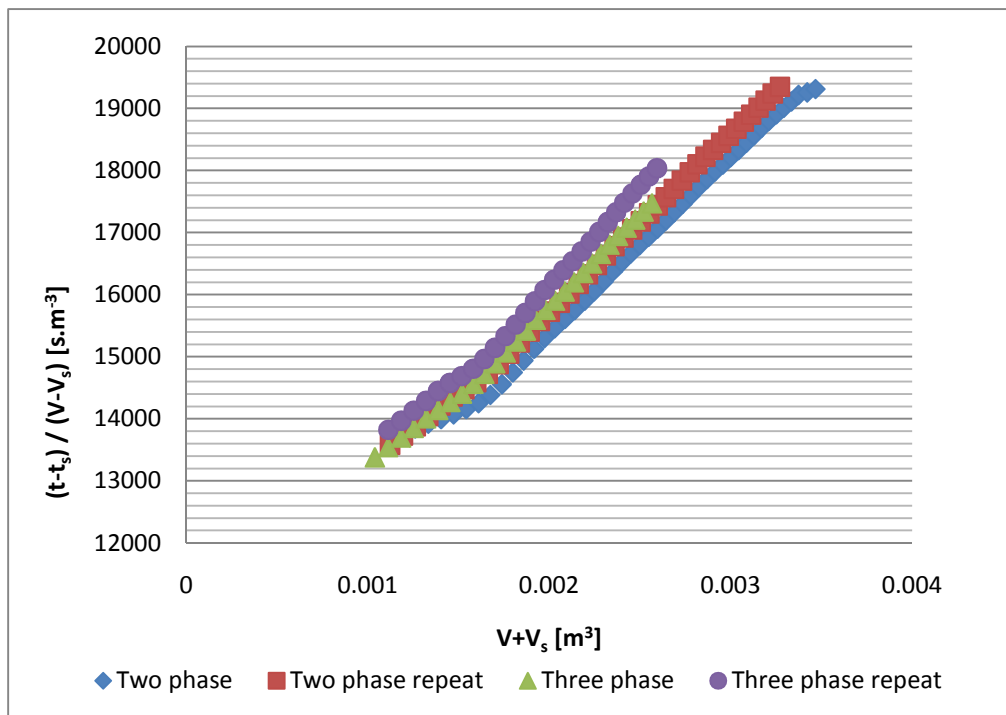

Figure C-29: Filtration characteristic curve for factorial run 6 (Runs 66, 74, 82 and 90)
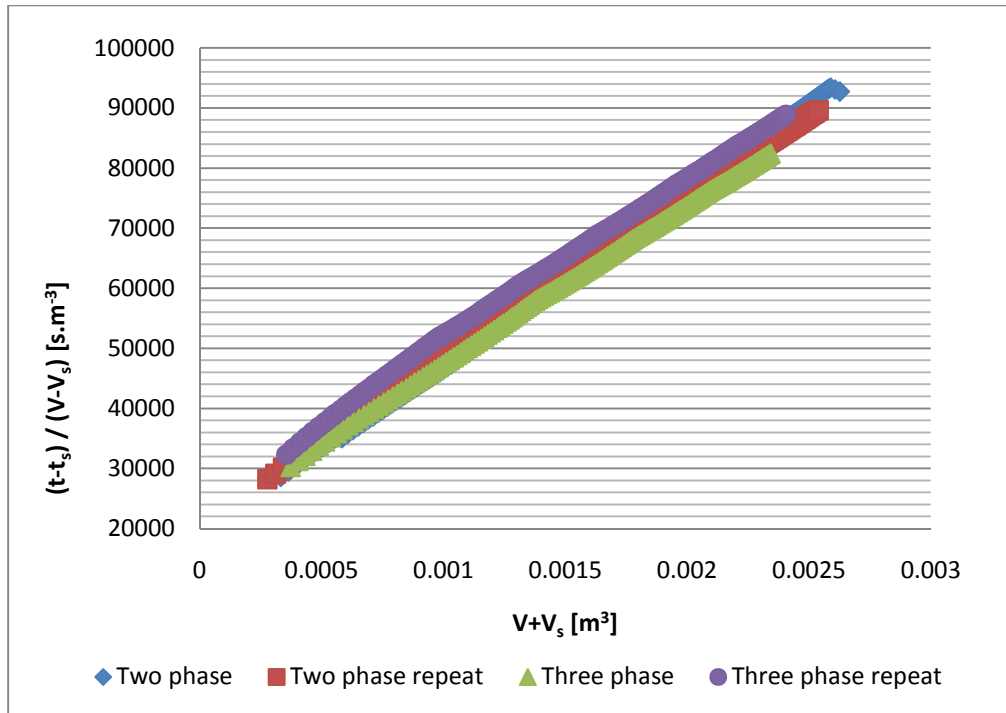
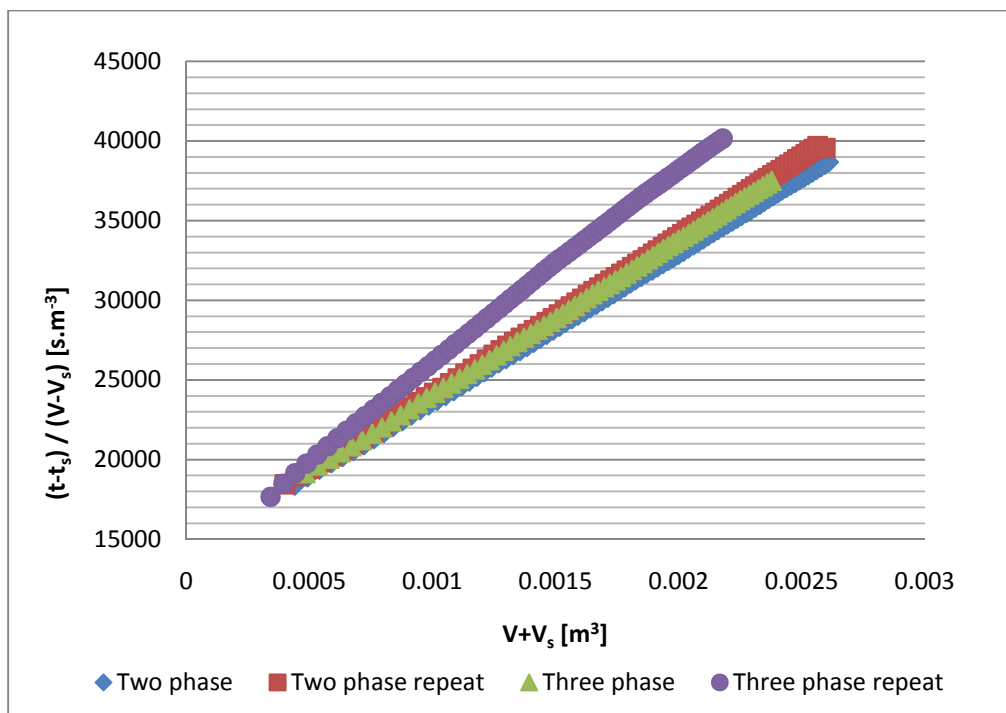Figure C-30: Filtration characteristic curve for factorial run 7 (Runs 67, 75, 83 and 91)

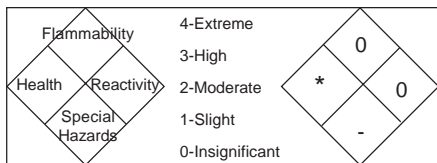Figure C-31: Filtration characteristic curve for factorial run 8 (Runs 68, 76, 84 and 92)

APPENDIX D:
MSDS AND TECHNICAL DATA

1. Material safety data sheet for Diatomaceous earth
   Source: www.gmzinc.com

2. Technical data sheet for Diatomaceous earth
   Source: www.gmzinc.com

Flammability	4-Extreme
Health / Reactivity	3-High
Special Hazards	2-Moderate
	1-Slight
	0-Insignificant

NFPA FIRE HAZARD SYMBOL˜
See NFPA 704 for detailed explanation.
* See Section 3 HAZARD IDENTIFICATION

Material Safety Data Sheet

No.: 2400   **Rev. No.: 9**

Date Revised: 11/07/2006

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Trade Name(s): (CELITE® = C) C110, C224, C226, C319, C427, C501, C513, C522, FILTER AID FOR COOKING OIL, C HSC, HYFLO® (ALL), HYFLO® SUPER CEL®, X-4, X-5

Generic Name: FLUX CALCINED DIATOMACEOUS EARTH
Chemical Name: SILICA
Manufacturer: CELITE CORPORATION
Address: P.O. BOX 519
City: LOMPOC   **State:** CA   **Zip:** 93438

CAS: 68855-54-9
EINECS: 272-489-0
Formula: SiO_2
Telephone: (805) 735-7791
Emergency: CHEMTREC - USA: (800) 424-9300
International: (703) 527-3887 (collect)

2. COMPOSITION/INFORMATION ON INGREDIENTS

INGREDIENT NAME:	CAS NUMBER:	%	PEL AND TLV (except as noted)
FLUX CALCINED DIATOMACEOUS EARTH (DE)	68855-54-9	100	SEE BELOW
THIS PRODUCT MAY CONTAIN UP TO 44% CRYSTALLINE SILICA:			
CRISTOBALITE	14464-46-1	< 40	.025mg/M^3 RESPIRABLE CRISTOBALITE, ACGIH
QUARTZ	14808-60-7	< 4	.025mg/M^3 RESPIRABLE QUARTZ, ACGIH

3. HAZARD IDENTIFICATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED INHALATION OF CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A KNOWN HUMAN CARCINO-GEN. INHALATION OF CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

Medical conditions which may be aggravated: PRE-EXISTING UPPER RESPIRATORY AND LUNG DISEASE SUCH AS, BUT NOT LIMITED TO BRONCHITIS, EMPHYSEMA AND ASTHMA.
Target Organ(s): LUNGS, EYES

Acute Health Effects: TRANSITORY UPPER RESPIRATORY OR EYE IRRITATION.
Chronic Health Effects: INHALATION OF CRYSTALLINE SILICA HAS BEEN CLASSIFIED BY IARC AS CARCINOGENIC FOR HUMANS (GROUP 1). INHALATION OF CRYSTALLINE SILICA IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE CAUSED BY EXCESSIVE EXPOSURE TO CRYSTALLINE SILICA.

Primary Entry Route(s): INHALATION, DUST CONTACT WITH EYES.

Inhalation: IRRITATION AND SORENESS IN THROAT & NOSE. IN EXTREME EXPOSURES SOME CONGESTION MAY OCCUR.
Eyes: TEMPORARY IRRITATION OR INFLAMMATION.
Skin Contact: NA   **Skin Absorption:** NA   **Ingestion:** NOT HAZARDOUS WHEN INGESTED.

4. FIRST AID MEASURES

Inhalation: REMOVE TO FRESH AIR. DRINK WATER TO CLEAR THROAT AND BLOW NOSE TO EVACUATE DUST.
Eyes: FLUSH EYES WITH LARGE QUANTITIES OF WATER. IF IRRITATION PERSISTS CONSULT A PHYSICIAN.
Skin Contact: NA   **Skin Absorption:** NA   **Ingestion:** NA

5. FIRE FIGHTING MEASURES

Flash Point (Method): NONFLAMMABLE   **NFPA Flammable/Combustible Liquid Classification:** NA
Flammable Limits: LEL: NA   **UEL:** NA   **Auto-Ignition Temperature:** NA
Extinguishing Media: NA   **Unusual Fire or Explosion Hazards:** NONE   **Special Fire-Fighting Procedures:** NONE

6. ACCIDENTAL RELEASE MEASURES

Procedures for Spill/Leak: VACUUM CLEAN DUST WITH EQUIPMENT FITTED WITH HEPA FILTER. USE A DUST SUPPRESSANT SUCH AS WATER IF SWEEPING IS NECESSARY-

7. HANDLING AND STORAGE

MINIMIZE DUST GENERATION AND ACCUMULATION. AVOID BREATHING DUST, AVOID CONTACT WITH EYES. SEAL BROKEN BAGS IMMEDIATELY. CONTINUE TO FOLLOW ALL MSDS/LABEL WARNINGS WHEN HANDLING EMPTY CONTAINERS.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Goggles: GOGGLES OR SAFETY GLASSES WITH SIDESHIELDS ARE RECOMMENDED.
Gloves: NOT NORMALLY REQUIRED.
Respirator: <10X PEL, USE AN N95 QUARTER OR HALF MASK RESPIRATOR; <50X PEL, USE A FULL FACE RESPIRATOR EQUIPPED WITH N95 FILTERS; <200X PEL, USE A POWERED AIR PURIFYING RESPIRATOR (POSITIVE PRESSURE) WITH N95 FILTERS; >200X PEL, USE A FULL FACE, TYPE C SUPPLIED AIR RESPIRATOR (CONTINUOUS FLOW MODE).
Ventilation: USE SUFFICIENT NATURAL OR MECHANICAL VENTILATION TO KEEP DUST LEVEL BELOW PEL.

Other: Special Considerations for repair/maintenance of contaminated equipment: INSURE PROPER RESPIRATORY PROTECTION.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odor: FINE WHITE POWDER, NO ODOR.

Boiling Point:	NA	**Evaporation Rate (= 1) :**	NA	**Specific Gravity (water = 1) :**	2.3
Vapor Pressure:	NA	**Melting Point:**	ND	**% Volatile by Volume:**	NIL
Water Solubility (%) :	NEGLIGIBLE	**Vapor Density (Air=1) :**	NA	**pH:**	9 - 10.5

10. STABILITY AND REACTIVITY

MATERIAL IS STABLE.   HAZARDOUS POLYMERIZATION CANNOT OCCUR.
Chemical Incompatibilities: HYDROFLUORIC ACID.   **Conditions to Avoid:** NONE IN DESIGNED USE.

11. TOXICOLOGICAL INFORMATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A KNOWN HUMAN CARCINOGEN. CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

12. ECOLOGICAL INFORMATION

GENERALLY CONSIDERED CHEMICALLY INERT IN THE ENVIRONMENT. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANT AND SHOULD BE EVALUATED ACCORDINGLY.

13. DISPOSAL CONSIDERATIONS

WASTE IS NOT HAZARDOUS AS DEFINED BY RCRA (40 CFR 261). OTHER STATE AND LOCAL REGULATIONS MAY VARY, CONSULT LOCAL AGENCIES AS NEEDED. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANTS AND SHOULD BE EVALUATED ACCORDINGLY.

14. TRANSPORTATION INFORMATION

D.O.T. Proper Shipping Name: EARTH, DIATOMACEOUS, CRUDE OR GROUND   **Hazard Classification:** NOT CLASSIFIED
Reportable Quantities: NOT APPLICABLE   **UN (United Nations), NA(North America) Number:** NOT APPLICABLE

15. REGULATORY INFORMATION

OSHA Hazard Communications Standard, 29 CFR 1910.1200: MATERIAL IS CONSIDERED HAZARDOUS, SEE SECTION 3.
RCRA: THIS MATERIAL IS NOT DEFINED AS HAZARDOUS WASTE PER 40 CFR 261.
TSCA: THIS MATERIAL IS LISTED IN THE TSCA INVENTORY, AND IS NOT OTHERWISE REGULATED BY TSCA SEC. 4, 5, 6, 7 OR 12.
CERCLA: MATERIAL IS NOT REPORTABLE UNDER CERCLA, LOCAL REQUIREMENTS MAY VARY.
SARA: 311/312 HAZARD CATAGORIES - IMMEDIATE AND DELAYED HEALTH, 313 REPORTABLE INGREDIENTS - NONE.
Canada: THIS PRODUCT IS LISTED ON THE DSL.
California Proposition 65: THIS PRODUCT CONTAINS CHEMICALS KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

16. OTHER INFORMATION

Storage Segregation Hazard Classes: NA
Special Handling/Storage: REPAIR ALL BROKEN BAGS IMMEDIATELY.
Special Workplace Engineering Controls: ADEQUATE VENTILATION TO KEEP DUST LEVEL BELOW PEL.

Prepared/Revised by: WORLD MINERALS ENVIRONMENTAL HEALTH & SAFETY SERVICES

As of the date of preparation of this document, the foregoing information is believed to be accurate and is provided in good faith to comply with applicable federal and state law(s). However, no warranty or representation with respect to such information is intended or given.

Technical Data

CELITE® 110

TYPICAL PHYSICAL PROPERTIES

Color	White
Appearance	Powder
Origin	Plankton Marine Diatomite
Description	Flux Calcined Filler
Density	
Wet (lbs/ft^3)	19.5
150 Mesh Screen Residue, %	4.0
325 Mesh Screen Analysis	17.5
Water absorption, % by weight	175.0
pH	10.0
Specific Gravity	2.3
Moisture, as shipped, %	0.1
Loss on Ignition	0.2

TYPICAL CHEMICAL ANALYSIS, %

SiO_2	89.6
Al_2O_3	4.0
Fe_2O_3	1.3
P_2O_5	0.2
TiO_2	0.2
CaO	0.5
MgO	0.6
$Na_2O + K_2O$	3.3

APPENDIX E:

SAFETY PRECAUTIONS

1. Laboratory coats, closed shoes must be worn at all times during the course of experimentation

2. Electrical hazards are present due to the nature of equipment used and care is to be taken.

3. Ensure that the Perspex shield is closed during operation of the test rig.

4. Ensure that the pressure within the column does not exceed 3bar abs.