UNIVERSITY OF KWAZULU-NATAL
Faculty of Engineering (Westville Campus)

Thesis Title:

A COMPARATIVE STUDY OF CONTACTING EQUIPMENT FOR THE RECOVERY OF COPPER FROM A CUPRIC SULPHATE SOLUTION

Candidate:

Natisha Sukhraj

Submitted in Fulfilment of the Degree:

Masters in Chemical Engineering

Supervisor:

Mr A.F.C Bassa

Date of Submission:

January 2005

MASTERS BY RESEARCH

A Comparative Study of Contacting Equipment for the Recovery of Copper from a Cupric Sulphate Solution

SUBMITTED BY

Natisha Sukhraj

IN FULFILMENT OF THE DEGREE

Masters in Chemical Engineering
from the University of KwaZulu-Natal (Westville Campus)

DATE OF SUBMISSION

January 2005

SUPERVISED BY

Mr A.F.C Bassa

As the candidate's supervisor, I have approved this thesis for submission.

Signed: ________________

Name: ________________________________ Date: ________________

DECLARATION

I, Natisha Sukhraj, Student Number 9901396, hereby declare that the thesis entitled **A Comparative Study of Contacting Equipment for the recovery of Copper from a Cupric Sulphate Solution** is a result of my own investigation and research, and presents my own work unless specifically referenced in the text. This work has not been submitted in part or in full for any other degree or to any other University.

ACKNOWLEDGEMENTS

I would like to acknowledge my supervisor, Mr A.F.C Bassa, for his technical and academic patronage throughout my University career, in particular, toward the completion of this MSc degree by research.

I acknowledge the academic and managerial support structure at the Department of Chemical Engineering (Westville Campus), in particular, Professor M Carsky, Mr. D. Singh and Mr. S.A. Naidoo, for their continuous conscientious and technical support for my pursuit of this degree. To my alma mater at the Faculty of Engineering at Westville Campus, thank you.

A special thanks to Mr B. Sukhraj and Mr P. Gareeb for their moral support.

Further more, I would like to acknowledge Mr. A. Bisessur and Mr. S. Naidoo from the Department of Chemistry (Westville Campus), for their assistance in aspects of chemistry matters.

Finally, I would like to thank the staff of Mintek South Africa. My postgraduate studies were financially endorsed by Mintek South Africa. I wish to recognise their support for permitting me use of their laboratories and facilities.

"RESEARCH IS TO SEE WHAT EVERYBODY ELSE SEES, AND TO THINK WHAT NOBODY ELSE HAS THOUGHT."

.... **Albert Szent-Gyorgyi**

ABSTRACT

Ion exchange for the recovery of metals from solutions is a well established process. It features significantly in terms of being able to recover valuable substances from what would otherwise be waste streams as well as recovering substances that could be harmful to the environment if left in the waste stream.

The more popular options for ion exchange processes could be batch, fixed bed, fluidized, moving bed, and chromatographic columns. Although most ion exchange processes tend to be batch processes making use of the fixed bed columns, technological developments enable the use of fluidized beds to be explored.

The main purpose of this research was to compare the performance of a fixed bed ion exchange system with a fluidized ion exchange system for the recovery of copper from a cupric sulphate solution. By experimentation the bed depth required for each type of equipment (in order to achieve a specified percentage recovery of copper from a specified feed) was determined. The comparative advantage of one type of equipment over the other ensures the correct type of system to be used for a sulphate solution of a particular concentration.

This study provides a basis for comparative studies of contacting equipment for the removal of other substances from dilute solutions.

TABLE OF CONTENTS

Title Page i
Declaration ii
Acknowledgements iii
Abstract v
List of Symbol and Abbreviations xiii
List of Figures xvi
List of Tables xxiii

Chapter One: Introduction 1
1.1 Comparative Studies Conducted 1
1.2 Study Conducted by Author 2
  1.2.1 Definition of Ion Exchange and Purpose of this Study....................2
  1.2.2 Test Work Conducted 3
    1.2.2.1 Choice of Resin 3
    1.2.2.2 Preliminary Experimental Test Work 4
    1.2.2.3 Experimental Work 4

Chapter Two: Ion Exchange Material 5
2.1 The Phenomenon 5
2.2 Resin 6
  2.2.1 Types of Resin 6
    2.2.2.1 Cation Exchange Resin 6
    2.2.2.2 Anion Exchange Resins 6
  2.2.2 Considerations When Choosing a Resin 7
    2.2.2.1 Typical Particle Shape and Size 7
    2.2.2.2 Total Capacity and Operating Capacity 7
    2.2.2.3 Bulk Density and True Wet Density 7
    2.2.2.4 Swelling and Shrinking 8
    2.2.2.5 Stability 8
    2.2.2.6 Pressure Drop 9
  2.2.3 Resin Chosen: TP 207 Weak acid Cation Resin 9
    2.2.3.1 Safety Data on Lewatit TP 207 (Weak acid Cation Resin) 9

Chapter Three: Equilibria and Kinetics 12
3.1 Ion Exchange Equilibrium 12
  3.1.1 Batch Method 12
  3.1.2 Column Method 12
3.2 Ion Exchange: Mass Balance 12
3.3 Adsorption Isotherms Model 14
3.4 Selectivity of Ion Exchange Resins 15
  3.4.1 Separation Factor 15
  3.4.2 Selectivity Coefficient 16
  3.4.3 Distribution Coefficient 17
3.5 Kinetics 17
  3.5.1 Diffusional Steps 17
  3.5.2 Integral Method 18
    3.5.2.1 Order of Reaction 18
      3.5.2.1(a) Zero Order 19
      3.5.2.1(b) First Order 19
      3.5.2.1(c) Second Order 20

Chapter Four: Breakthrough Test.......... 21
4.1 The "Mass Transfer Zone" 21
  4.1.1 Definition and Principle 21
  4.1.2 Calculations of the Mass Transfer Zone Height 23

Chapter Five: Fixed and Fluidized Beds 25
5.1 Fixed Beds 26
5.2 Fluidized Beds 26
  5.2.1 Analysis of Experimental Determination of Minimum Fluidization Velocity 27
  5.2.2 Richardson Zaki Correlation 28
    5.2.2.1 Void volume as a function of Bed Height 29

Chapter Six: Design Considerations 30
6.1 Vessel Sizing and Requirements 30
  6.1.1 Vessel Sizing 30
  6.2.1 Product Requirements 30
  6.1.3 Feed Composition 30
6.2 Schematic Representation of Designs for Ion Exchange Columns.......... 31

Chapter Seven: Determination of Copper Present in the Effluent and Eluates 34
7.1 Spectrophotometry 34
7.1.1 Spectrophotometric Analysis 34

Chapter Eight: Regeneration 36
8.1 Regeneration Procedure 36
8.2 Co-Current and Counter-Current Regeneration 36

Chapter Nine: Applications of Ion Exchange Processes 38
9.1 Waste Water Treatment.. 38
9.2 Water Softening.. 38
9.3 Applications in the Gold Industry 39
9.4 Other Applications of Ion Exchange Processes 39

Chapter Ten: Experimental Procedures 41
10.1 Tapped Wet Volume Measurement... 41
10.1.1 Procedure 41
10.1.2 Packing a Column with Resin 41
10.1.2.1 Procedure 41
10.1.3 Pre conditioning of Resins 42
10.1.3.1 Purpose 42
10.1.3.2 Resin 42
10.2 Determination of Average Particle Size of TP 207 resin 42
10.2.1 Apparatus and Equipment 42
10.2.2 Reagents 42
10.2.3 Procedure 42
10.3 Void Volume Measurement 43
10.3.1 Apparatus and Equipment 43
10.3.2 Reagents 44
10.3.3 Procedure 44
10.4 Water Retention Capacity.. 44
10.4.1 Apparatus and Equipment 44
10.4.2 Reagents 44
10.4.3 Procedure 45
10.5 pH Test 45
10.5.1 Apparatus and Equipment 45
10.5.2 Reagents 46
10.5.3 Procedure 46

10.6 Equilibrium Tests 47
  10.6.1 Apparatus and Equipment 47
  10.6.2 Reagents 47
  10.6.3 Procedure 47
10.7 Kinetic Tests 48
  10.7.1 Apparatus and Equipment 48
  10.7.2 Reagents 48
  10.7.3 Procedure 48
10.8 Effect of Swelling of TP 207 resins 49
  10.8.1 Conversion into the Hydrogen form 49
    10.8.1.1 Procedure 49
  10.8.2 Conversion into the Di Sodium 49
    10.8.2.1 Procedure 49
  10.8.3 Conversion into the Mono Sodium form 50
    10.8.3.1 Procedure 50
10.9 Mini Column Tests 51
  10.9.1 Apparatus and Equipment 51
  10.9.2 Reagents 51
  10.9.3 Procedure 51
10.10 Minimum Fluidization Velocity Measurement 52
  10.10.1 Apparatus and Equipment 52
  10.10.2 Reagents 52
  10.10.3 Procedure 52
10.11 Column Tests 53
  10.11.1 Apparatus and Equipment 53
  10.11.2 Reagents 53
  10.11.3 Procedure 53
10.12 Pilot Plant Testing 54
  10.12.1 Apparatus and Equipment 54
  10.12.2 Reagents 55
  10.12.3 Procedure 55

Chapter Eleven: Results and Discussion 60
11.1 Influent Characteristics 60
  11.1.1 Chemical Analysis 60
  11.1.2 Void Volume Determination 60

11.2 Adsorbent 60
 11.2.1 Resin Characterization 60
 11.2.2 Particle Size Analysis 60
 11.2.3 Water Retention Capacity 61
11.3 Preliminary Test Work 61
 11.3.1 pH Dependence of Metal Loadings 61
 11.3.2 Ion Exchange Equilibrium Isotherms 62
  11.3.2.1 The Langmuir Isotherm for Equilibrium Curve at pH 3 63
  11.3.2.2 The Freundlich Isotherm for Equilibrium Curve at pH 3 64
 11.3.3 Kinetic Tests 67
 11.3.4 Effect of Swelling 70
11.4 Mini Column Tests 70
11.5 Determination of Minimum Fluidization Velocity 73
11.6 Column Tests 75
11.7 Pilot Plant Testing 77
 11.7.1 Breakthrough Tests 78
 11.7.2 Full Scale Plant Design 81
  11.7.2.1 Sizing of Pilot Plant 81
 11.7.3 Cost Estimation 83
  11.7.3.1 Capital Expenditure 83
  11.7.3.2 Production Costs 85
   11.7.3.2(a) Regenerant: Sulphuric Acid 86
   11.7.3.2 (b) Operating Labour 86
  11.7.3.3 Cost Indices 86
 11.7.4 Summary 87

Chapter Twelve: Conclusion 88
12.1 Resin Properties 88
12.2 Preliminary Experimental Test Work 88
 12.2.1 Effect of pH on Resin Loading 89
 12.2.2 Ion Exchange Equilibria 89
 12.2.3 Effect on Particle Size on Kinetics 90
12.3 Mini Column Tests 91
12.4 Minimum Fluidization Velocity 92
12.5 Column Tests 92
12.6 Pilot Plant Testing 93

Chapter Thirteen: The Way Forward......95

References 96

Appendices

Appendix A

A1: Glossary of Terms A1-1

Appendix B

B1: Cost Estimation for Ion Exchange Project B1-1

Appendix C: Resin Properties

C1: Particle Size Distribution C1-1
C2: Determination of Void Volume C2-1
C3: Calculation of Minimum Fluidization and Terminal Velocity for TP 207 ...... C3-1
C4: Determination of Water Retention Capacity C4-1
C5: Determination of the Effect of Swelling on the TP 207 resin C5-1

Appendix D: pH Test

D1 pH Test Calculations D1-1

Appendix E: Equilibrium Tests

E1: Equilibrium Test for pH set at 3 ...... E1-1
E2: Equilibrium Test for pH set at 2.5 ......E2-1
E3: Equilibrium Test for pH set at 2 ......E3-1
E4: Equilibrium Test for pH set at 1.5 ......E4-1
E5: Summary of Equilibrium Plots E5-1

Appendix F: Kinetic Tests

F1: Kinetic tests for particle diameter 850 μm......F1-1
F2: Kinetic tests for particle diameter 600 μm......F2-1
F3: Comparisons of the Particle Diameters...... F3-1
F4: Predicted Kinetic Responses......F4-1

Appendix G: Mini Column Tests

G1: Hydrogen form, feed pH 3 ......G1-1
G2: Di Sodium form, feed pH 3 ......G2-1

G3: Mono Sodium form, feed pH 3 .... G3-1
G4: Summary of Results obtained from Feed pH 2.5 .... G4-1
G5: Summary of Results obtained from Feed pH 2.04 .... G5-1
G6: Summary of Results obtained from Feed pH 1.59 .... G6-1

Appendix H: Minimum Fluidization Velocity

H1: Determination of Minimum Fluidization Velocity H1-1

Appendix I: Column Tests

I1: Column test at Linear Velocity 0.13 cm/s I1-1
I2: Column test at Linear Velocity 0.19 cm/s I2-1
I3: Column test at Linear Velocity 0.41 cm/s I3-1
I4: Mass Transfer Zone versus Linear Velocity I4-1
I5: Full Scale Plant Design for 0.13 cm/s I5-1
I6: Full Scale Plant Design for 0.19 cm/s I6-1
I7: Full Scale Plant Design for 0.4 cm/s I7-1

Appendix J: Comparison of Contacting Equipment at 6 g/L

J1: Fixed Bed Ion Exchange for Concentration at 6 g/L J1-1
J2: Fluidized Bed Ion Exchange for Concentration at 6 g/L J2-1
J3: Comparison of Contacting Equipment for 6 g/L J3-1

Appendix K: Comparison of Contacting Equipment at 0.6 g/L

K1: Fixed Bed Ion Exchange Column at Concentration of 0.6 g/L K1-1
K2: Fluidized Bed Ion Exchange Column at Concentration of 0.6 g/L K2-1
K3: Comparison of Contacting Equipment for 0.6 g/L K3-1

Appendix L: Full Scale Plant Design and Cost Estimations

L1: Full scale Plant Design for Fixed Bed at 6 g/L L1-1
L2: Full scale Plant Design for Fluidized Bed at 6 g/L L2-1
L3: Full scale Plant Design for Fixed Bed at 0.6 g/L L3-1
L4: Full scale Plant Design for Fluidized Bed at 0.6 g/L L4-1
L5: Cost Estimation for Full scale Fixed Bed Design L5-1
L6: Cost Estimation for Full scale Fluidized Bed Design L6-1

LIST OF SYMBOLS AND ABBREVIATIONS

Symbol	Description	Unit
a	Langmuir constant	dimensionless
a_p	Surface area per unit volume of particle	m^2/m^3
A_{col}	Column cross-sectional area	m^2
A_{int}	Total interfacial area	m^2
b	Langmuir constant	dimensionless
C	Solution concentration	g/L
C_B	Solution concentration at time t_B	g/L
C_D	Drag coefficient	dimensionless
C_{el}	Elution Concentration of Cu^{2+}	g/L
C_o	Total solution concentration	g/L
C_e	Equilibrium solution concentration	g/L
d	Resin particle diameter	mm
D_{col}	Column diameter	cm
f_e	Zone fraction	dimensionless
g	Gravitational constant	m/s^2
H_p	Height of Packed Column	mm; cm
h	Mass Transfer Zone	cm; m
K_f	Freundlich parameter	dimensionless
L	Liquid flow rate	m^3/s
1/n	Freundlich parameter	dimensionless
q	Resin concentration	g/L
q_o	Initial resin concentration	g/L
qe	Amount of Copper loaded onto resin from absorption	g/L
Qe	Amount of Copper loaded onto resin from elution	g/L

Q	Total resin capacity (m^3 of resin beads)	g /L
Q_{op}	Operating resin capacity (m^3 of resin beads)	g /L
r	Fitting Parameter	dimensionless
Re	Reynolds number ($\rho d U_L / \mu$)	dimensionless
Sc	Schmidt number ($\mu / \rho D$)	dimensionless
Sh	Sherwood number ($k_L d / D$)	dimensionless
t	Time	min
t_B	Time to reach breakthrough concentration	min
t_T	Time to reach specified concentration at the top of breakthrough curve	min; hr
T	Temperature	K
u_F	Minimum Fluidization Velocity	cm/s
u_L	Liquid superficial velocity (L/ A_{col})	cm/s
u_o	Liquid velocity at $\varepsilon=1$ (fluidized bed)	cm/s
u_R	Resin superficial velocity (R/ A_{col})	cm/s
u_T	Resin bead terminal velocity	cm/s
V_c	Volume of resin loaded with copper	mL
V_o	Initial volume of feed	L
V_B	Liquid volume at time t_B	BV
V_{bed}	Resin bed volume including voids	mL
V_{cu}	Volume of Cu^{2+} solution from elution	L
V_e	Volume of resin after elution	mL
V_{ro}	Tapped wet volume of resin initially	mL
V_s	Volume of Cu^{2+} solution concentration after equilibrium	mL; L
V_L	Liquid volume	L
z	Distance along column	cm; m
Z	Total column height	cm; m

Greek Symbols

Greek Symbol	Description	Unit
ε	Resin bed void volume	dimensionless
ε_o	Settled bed void volume	dimensionless
ε_p	Resin pore void volume	dimensionless
μ	Liquid viscosity	kg/ms
ρ_f	Liquid density	g/L
ρ_s	Resin density	g/L
τ	Residence Time	min

List of Abbreviations

Bed Volumes	BV
Linear Velocity	LV
Mass Transfer Zone	MTZ
Not Applicable	NA
Time	t
Concentration	Conc.
Hours	hr
Hydrogen form	H^+ form

LIST OF FIGURES

CHAPTER TWO: ION EXCHANGE MATERIALS

Figure 2.1: Representation of Cation Exchange on an Ion Exchangeable Material.................. 5
Figure 2.2: Schematic Representation of Drying and Swelling of a Resin Bead.......................8

CHAPTER THREE: EQUILIBRIA AND KINETICS

Figure 3.1: Initial Conditions at Start of Operation.. 13
Figure 3.2: Conditions at the End of Operation.. 14
Figure 3.3: Plots of Adsorption Isotherm Models..14
Figure 3.4: Steps in an Ion Exchange Reaction.. 18
Figure 3.5: Zero-Order Reaction...19
Figure 3.6: First-Order Reaction... 19
Figure 3.7: Second-Order Reaction...20

CHAPTER FOUR: BREAKTHROUGH TESTS

Figure 4.1: Photograph of Resin Bed (TP 207 resin in the hydrogen form)........................... 21
Figure 4.2: Photograph of first few layers of the Resin Bed adsorbed with Copper in a Fixed Bed Ion Exchange Column...22
Figure 4.3: A Plot of the Concentration Profile of adsorbate in the fluid phase as a function of Distance along the Adsorbent Bed.. 22
Figure 4.4: A Typical plot of the ratio of Outlet solute concentration to Inlet solute concentration in the fluid as a function of Time from the start of flow...............................22
Figure 4.5: Schematic Representation of the Mass Transfer Zone in a fixed bed ion exchange column..23
Figure 4.6: Calculation of Mass Transfer Zone from Breakthrough Tests..............................24

CHAPTER FIVE: FIXED AND FLUIDIZED BEDS

Figure 5.1: Fixed Bed versus Fluidized Bed...25
Figure 5.2: Graph of Pressure drop and Bed Height versus Superficial Velocity................... 27

CHAPTER SIX: DESIGN CONSIDERATIONS

Figure 6.1: Schematic Design of Fixed Bed Ion Exchange Column...................................... 32
Figure 6.2: Schematic Design of Fluidized Ion Exchange Column...33

CHAPTER SEVEN: DETERMINATION OF COPPER PRESENT IN THE EFFLUENT AND ELUATES

Figure 7.1: Varian Spectra AA 50 B 34

CHAPTER NINE: APPLICATIONS OF ION EXCHANGE PROCESSES

Figure 9.1: Some Water Treatment Resins 38
Figure 9.2: Exchange of Hard Water Ions for Soft Water Ions 39
Figure 9.3: Down Flow Ion Exchange Columns 40
Figure 9.4: Up Flow Ion Exchange Columns 40

CHAPTER TEN: EXPERIMENTAL PROCEDURES

Figure 10.1: Photograph of a handful of TP 207 resin in the Hydrogen form 43
Figure 10.2: Photograph of a Single Particle Magnified fifteen times 43
Figure 10.3: Photograph of the Watson Marlow Peristaltic Pump 52
Figure 10.4: Photograph of the Pilot Plant 56
Figure 10.5: Photograph of the Fixed Bed Column at the Start of Operations 57
Figure 10.6: Photograph of the Fixed Bed during Operation with Copper loading onto resin 57
Figure 10.7: Photograph of the Fixed Bed at the End of Operation with resin fully loaded with Copper 58
Figure 10.8: Photograph of the Fluidized Bed during Operation with Copper loading onto resin 58
Figure 10.9: Photograph of the Fluidized Bed at the End of Operation with resin fully loaded with Copper 59
Figure 10.10: Photograph of the Regeneration of the resin with 2M Sulphuric Acid 59

CHAPTER ELEVEN: RESULTS AND DISCUSSION

Figure 11.1: Effect of pH on Copper Loading 62
Figure 11.2: Equilibrium Isotherm of TP 207 Resin [Favourable Isotherm] 62
Figure 11.3: Langmuir Isotherm for Equilibrium Curve at pH 3 64
Figure 11.4: Freundlich Isotherm for Equilibrium Curve at pH 3 65
Figure 11.5: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 3 65
Figure 11.6: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 2.5 66
Figure 11.7: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 2 66

Figure 11.8 : A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 1.5......66
Figure 11.9 : Kinetic Response for 850 μm and Initial Concentration 6 g/L......68
Figure 11.10: First Order Reaction Kinetics for 850 μm and Initial Concentration of 6 g/L...... 68
Figure 11.11: Second Order Reaction Kinetics for 850 μm and Initial Concentration of 6 g/L..68
Figure 11.12: Effect of particle diameter at initial concentration of 6 g/L...... 69
Figure 11.13: Effect of particle diameter at initial concentration of 0.6 g/L...... 70
Figure 11.14: Resin in Three forms at Initial Feed at pH of 3......71
Figure 11.15: Resin in Three forms at Initial Feed at pH of 2.5......71
Figure 11.16: Resin in Three forms at Initial Feed at pH of 2.04......71
Figure 11.17: Resin in Three forms at Initial Feed at pH 1.59...... 72
Figure 11.18: Experimental Determination of Minimum Fluidization Velocity...... 74
Figure 11.19: Comparison of Predicted Data and Experimental Data for the Graph of Void Volume versus Linear Velocity......74
Figure 11.20: Breakthrough curve at Linear Velocity of 0.13 cm/s...... 76
Figure 11.21: Breakthrough curve at Linear Velocity of 0.195 cm/s...... 76
Figure 11.22: Breakthrough curve at Linear Velocity of 0.41 cm/s...... 76
Figure 11.23: Graph of Mass Transfer Zone versus Linear Velocity...... 77
Figure 11.24: Fixed Bed Testing at 6 g/L...... 78
Figure 11.25: Fluidized Bed Testing at 6 g/L...... 78
Figure 11.26: Comparison of Fixed and Fluidized Bed at 6g/L...... 79
Figure 11.27: Fixed Bed Testing at 0.6 g/L...... 79
Figure 11.28: Fluidized Bed Testing at 0.6 g/L...... 79
Figure 11.29: Comparison of Fixed and Fluidized Bed at 0.6 g/L......80
Figure 11.30: Sizing and Operation of an Ion Exchange Pilot Plant......81
Figure 11.31: Vertical pressure vessel...... 85
Figure 11.32: Chemical Engineering Plant Cost Indices...... 86

CHAPTER TWELVE: CONCLUSION

Figure 12.1: Effect of pH on Copper Loading...... 89
Figure 12.2: Summary of Copper Ion Exchange Equilibrium Isotherms...... 89
Figure 12.3: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 6 g/L......90
Figure 12.4: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 0.6 g/L......91

Figure 12.5: Resin in Three Forms at Initial Feed pH of 3 91
Figure 12.6: Experimental determination of Minimum Fluidization Velocity 92
Figure 12.7: Graph of Mass Transfer Zone versus Linear Velocity 92
Figure 12.8: Comparison of Fixed and Fluidized bed at 6g/L 93
Figure 12.9: Comparison of Fixed and Fluidized bed at 0.6g/L 93

APPENDIX C: RESIN PROPERTIES

Figure C1.1: Cumulative Graph for Run 1 .... C1-1
Figure C1.2: Summary of Cumulative graphs for Runs 2-8 .... C1-3

APPENDIX D: pH TESTS

Figure D1.1: pH Curve for Copper Solution D1-3

APPENDIX E: EQUILIBRIUM TESTS

Figure E1.1: Equilibrium Curve at pH 3 for Adsorption E1-3
Figure E1.2: Equilibrium Curve at pH 3 for Elution E1-3
Figure E1.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 3 .... E1-4
Figure E1.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 3 .... E1-5
Figure E1.5: Comparison of Experimental and Predicted Results E1-6
Figure E2.1: Equilibrium Curve at pH 2.5 for Adsorption E2-3
Figure E2.2: Equilibrium Curve at pH 2.5 for Elution E2-3
Figure E2.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 2.5 .... E2-4
Figure E2.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 2.5 .... E2-5
Figure E2.5: Comparison of Experimental and Predicted Results E2-6
Figure E3.1: Equilibrium Curve at pH 2 for Adsorption E3-3
Figure E3.2: Equilibrium Curve at pH 2 for Elution E3-3
Figure E3.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 2 .... E3-4
Figure E3.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 2 .... E3-5
Figure E3.5: Comparison of Experimental and Predicted Results E3-6
Figure E4.1: Equilibrium Curve at pH 1.5 for Adsorption E4-3
Figure E4.2: Equilibrium Curve at pH 1.5 for Elution E4-3
Figure E4.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 1.5 .... E4-4
Figure E4.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 1.5 .... E4-5
Figure E4.5: Comparison of Experimental and Predicted Results E4-6

APPENDIX F: KINETIC TEST

Figure F1.1: Kinetic Response for 850 μm from Table F1.4 .... F1-2
Figure F1.2: Kinetic Response for Initial Concentration at 6 g/L from Table F1.4 .... F1-3
Figure F1.3: First Order Reaction Plot for 6 g/L from Table F1.5 .... F1-3
Figure F1.4: Second Order Reaction Plot for 6 g/L from Table F1.6 .... F1-4
Figure F1.5: Kinetic Response for Initial Concentration at 0.6 g/L from Table F1.4 .... F1-5
Figure F1.6: First Order Reaction Plot for 0.6 g/L from Table F1.7 .... F1-5
Figure F1.7: Second Order Reaction Plot for 0.6 g/L from Table F1.8 .... F1-6
Figure F2.1: Kinetic Response for 600 μm from Table F2.4 .... F2-2
Figure F2.2: Kinetic Response for Initial Concentration at 6 g/L from Table F2.4 .... F2-3
Figure F2.3: First Order Reaction Plot for 6 g/L from Table F2.5 .... F2-3
Figure F2.4: Second Order Reaction Plot for 6 g/L from Table F2.6 .... F2-4
Figure F2.5: Kinetic Response for Initial Concentration at 0.6 g/L from Table F2.4 .... F2-5
Figure F2.6: First Order Reaction Plot for 0.6 g/L from Table F2.7 .... F2-5
Figure F2.7: Second Order Reaction Plot for 0.6 g/L from Table F2.8 .... F2-6
Figure F3.1: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 6 g/L F3-1
Figure F3.2: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 0.6 g/L F3-2
Figure F4.1: Predicted Kinetic Response for Particle Diameter 850 μm and Concentration 6g/L F4-1
Figure F4.2: Predicted Kinetic Response for Particle Diameter 600 μm and Concentration 6g/L F4-2
Figure F4.3: Predicted Kinetic Response for Particle Diameter 850 μm and Concentration 0.6g/L F4-3
Figure F4.4: Predicted Kinetic Response for Particle Diameter 600 μm and Concentration 0.6g/L F4-4

APPENDIX G: MINI COLUMN TESTS

Figure G1.1: Breakthrough Curve with Initial Condition from Table G1.1 .... G1-3
Figure G1.2: Monitoring of Copper Concentration and Effluent pH G1-3
Figure G2.1: Breakthrough Curve with Initial Condition from Table G2.1 .... G2-3
Figure G2.2: Monitoring of Copper Concentration and Effluent pH G2-3
Figure G3.1: Breakthrough Curve obtained with Initial Condition from Table G3.1 .... G3-4
Figure G3.2: Monitoring of Copper Concentration and Effluent pH stream G3-4
Figure G3.3: Resin in Three Forms at Initial Feed pH of 3 .... G3-4

Figure G4.1: Breakthrough Curve with Initial Condition from Table G4.1 .... G4-1
Figure G4.2: Monitoring of Copper Concentration and Effluent pH G4-1 .... G4-1
Figure G4.3: Breakthrough Curve with Initial Condition from Table G4.3 .... G4-2
Figure G4.4: Monitoring of Copper Concentration and Effluent pH G4-2
Figure G4.5: Breakthrough Curve with Initial Condition from Table G4.5 .... G4-3
Figure G4.6: Monitoring of Copper Concentration and Effluent pH G4-3
Figure G4.7: Resin in Three Forms at Initial Feed pH of 2.5 .... G4-4
Figure G5.1: Breakthrough Curve with Initial Condition from Table G5.1 .... G5-1
Figure G5.2: Monitoring of Copper Concentration and Effluent pH G5-1
Figure G5.3: Breakthrough Curve with Initial Condition from Table G5.3 .... G5-2
Figure G5.4: Monitoring of Copper Concentration and Effluent pH G5-2
Figure G5.5: Breakthrough Curve with Initial Condition from Table G5.5 .... G5-3
Figure G5.6: Monitoring of Copper Concentration and Effluent pH G5-3
Figure G5.7: Resin in Three Forms at Initial Feed pH of 2.04 .... G5-4
Figure G6.1: Breakthrough Curve with Initial Condition from Table G6.1 .... G6-1
Figure G6.2: Monitoring of Copper Concentration and Effluent pH G6-1
Figure G6.3: Breakthrough Curve with Initial Condition from Table G6.3 .... G6-2
Figure G6.4: Monitoring of Copper Concentration and Effluent pH G6-2
Figure G6.5: Breakthrough Curve with Initial Condition from Table G6.5 .... G6-3
Figure G6.6: Monitoring of Copper Concentration and Effluent pH G6-3
Figure G6.7: Resin in Three Forms at Initial Feed pH of 1.59 .... G6-4

APPENDIX H: MINIMUM FLUIDIZATION VELOCITY

Figure H1.1: Graph of Bed Height versus Column Velocity H1-2
Figure H1.2: Graph of Void Fraction versus Column Velocity H1-3
Figure H1.3: Comparison of Experimental and Predicted Curves H1-3

APPENDIX I: COLUMN TESTS

Figure I1.1: Breakthrough Curve with Initial Condition from Table I1.1 .... I1-4
Figure I1.2: Monitoring of Copper Concentration and Effluent pH I1-4
Figure I2.1: Breakthrough Curve with Initial Condition from Table I2.1 .... I2-4
Figure I2.2: Monitoring of Copper Concentration and Effluent pH I2-4
Figure I3.1: Breakthrough Curve with Initial Condition from Table I3.1 .... I3-4
Figure I3.2: Monitoring of Copper Concentration and Effluent pH I3-4
Figure I4.1: Graph of Mass Transfer Zone versus Linear Velocity I4-1

APPENDIX J: COMPARISON OF CONTACTING EQUIPMENTS AT 6 g/L

Figure J1.1: Breakthrough Curve with Initial Condition from Table J1.1 .... J1-4
Figure J1.2: Monitoring of Copper Concentration and Effluent pH J1-4
Figure J2.1: Breakthrough Curve with Initial Condition from Table J2.1 .... J2-4
Figure J2.2: Monitoring of Copper Concentration and Effluent pH J2-4
Figure J3.1: Comparison of Fixed and Fluidized Breakthrough Curves at 6 g/L J3-1

APPENDIX K: COMPARISON OF CONTACTING EQUIPMENTS AT 0.6 g/L

Figure K1.1: Breakthrough Curve with Initial Condition from Table K1.1 .... K1-3
Figure K2.1: Breakthrough Curve with Initial Condition from Table K2.1 .... K2-3
Figure K3.1: Comparison of Fixed and Fluidized Breakthrough Curves at 0.6 g/L K3-1

LIST OF TABLES

CHAPTER TWO: ION EXCHANGE MATERIAL

Table 2.1: Safety Data on Lewatit TP 207 resin....9

CHAPTER FIVE: FIXED AND FLUIDIZED BEDS

Table 5.1: Determination of n (Function of Reynolds Number).... 29

CHAPTER EIGHT: REGENERATION

Table 8.1: Guidelines for Typical Regeneration Level and Corresponding Resin Operating Capacity....37

CHAPTER ELEVEN: RESULTS AND DISCUSSION

Table 11.1: Summary of Particle Size Analysis.... 61
Table 11.2: Fitting Parameters for Langmuir Isotherm at pH 3.... 64
Table 11.3: Fitting Parameters for Freundlich Isotherm at pH 3....65
Table 11.4: Final Results and Fitting Parameters for Equilibrium Curves....67
Table 11.5: Mass Transfer Coefficient (k) for Diameter 850 μm....69
Table 11.6: Mass Transfer Coefficient (k) for Diameter 600 μm....69
Table 11.7: Tabulation of Three Forms of Resin at Initial Feed pH 3.... 72
Table 11.8: Tabulation of Three Forms of Resin at Initial Feed pH 2.5.... 72
Table 11.9: Tabulation of Three Forms of Resin at Initial Feed pH 2.... 72
Table 11.10: Tabulation of Three Forms of Resin at Initial Feed pH 1.5.... 73
Table 11.11: Theoretical Determination of Minimum Fluidization and Settling Velocity....74
Table 11.12: t-test (Two samples Assuming Equal Variance).... 75
Table 11.13: Tabulation of the Mass Transfer Zones heights at respective velocity.... 76
Table 11.14: Tabulation of Mass Transfer Zone heights for 6 g/L 78
Table 11.15: Tabulation of Mass Transfer Zone heights for 0.6 g/L 80
Table 11.16: Preliminary Column Design and Operating Parameters for the Removal of Copper from a Cupric Sulphate Solution at 6 g/L....82
Table 11.17: Preliminary Column Design and Operating Parameters for the Removal of Copper from a Cupric Sulphate Solution at 0.6 g/....83
Table 11.18: Typical factors for the Estimation of Fixed Capital Cost.... 84
Table 11.19: Summary of Production Costs....85

CHAPTER TWELVE: CONCLUSION

Table 12.1: Summary of Properties of TP 207 resin........ 88
Table 12.2: Fitting Parameters and Correlation Coefficients........ 90
Table 11.3: Tabulation of the Mass Transfer Zones heights........ 92
Table 11.4: Tabulation of the Resin Bed Depth Requirement for 6 g/L 93
Table 11.5: Tabulation of Resin Bed Depth Requirement for 0.6 g/L........ 94

APPENDIX C: RESIN PROPERTIES

Table C1.1: Initial Conditions for each Run........ C1-1
Table C1.2: Cumulative Graph Data for Run 1........ C1-1
Table C1.3: Summary of Particle Size Analysis........ C1-4

APPENDIX D: pH TESTS

Table D1.1: Results obtained from pH Test........ D1-1
Table D1.2: Calculations obtained from pH Test........ D1-2

APPENDIX E: EQUILIBRIUM TESTS

Table E1.1: Results from Equilibrium Test at pH 3........ E1-1
Table E1.2: Calculations from Equilibrium Test at pH 3........ E1-2
Table E1.3: The Langmuir and Freundlich Adsorption Isotherms........ E1-4
Table E1.4: Parameters for the Langmuir Plot........ E1-4
Table E1.5: Parameters for the Freundlich Plot........ E1-5
Table E1.6: Tabulation of Results from Langmuir and Freundlich Equation........ E1-5
Table E2.1: Results from Equilibrium Test at pH 2.5........ E2-1
Table E2.2: Calculations from Equilibrium Test at pH 2.5........ E2-2
Table E2.3: The Langmuir and Freundlich Adsorption Isotherms........ E2-4
Table E2.4: Parameters for the Langmuir Plot........ E2-4
Table E2.5: Parameters for the Freundlich Plot........ E2-5
Table E2.6: Tabulation of Results from Langmuir and Freundlich Equation........ E2-5
Table E3.1: Results from Equilibrium Test at pH 2........ E3-1
Table E3.2: Calculations from Equilibrium Test at pH 2........ E3-2
Table E3.3: The Langmuir and Freundlich Adsorption Isotherms........ E3-4
Table E3.4: Parameters for the Langmuir Plot........ E3-4
Table E3.5: Parameters for the Freundlich Plot........ E3-5
Table E3.6: Tabulation of Results from Langmuir and Freundlich Equation........ E3-5
Table E4.1: Results from Equilibrium Test at pH 1.5........ E4-1
Table E4.2: Calculations from Equilibrium Test at pH 1.5........ E4-2

Table E4.3: The Langmuir and Freundlich Adsorption Isotherms..E4-4
Table E4.4: Parameters for the Langmuir Plot..E4-4
Table E4.5: Parameters for the Freundlich Plot..E4-5
Table E4.6: Tabulation of Results from Langmuir and Freundlich Equation..E4-5

APPENDIX F: KINETIC TESTS

Table F1.1: Operating Conditions at the beginning of Kinetic Test..F1-1
Table F1.2: Operating Conditions at the End of the Kinetic Test..F1-1
Table F1.3: Calculations for Kinetic Test from Experimental Data..F1-1
Table F1.4: Results obtained from Kinetic Test..F1-2
Table F1.5: First Order Reaction Kinetics for 6 g/L..F1-3
Table F1.6: Second Order Reaction Kinetics for 6 g/L..F1-4
Table F1.7: First Order Reaction Kinetics for 0.6 g/L..F1-5
Table F1.8: Second Order Reaction Kinetics for 0.6 g/L..F1-6
Table F1.9: Mass Transfer Coefficient (k) calculated from First and Second Order Reaction Curves for Diameter 850 μm..F1-6
Table F2.1: Operating Conditions at the beginning of Kinetic Test..F2-1
Table F2.2: Operating Conditions at the End of the Kinetic Test..F2-1
Table F2.3: Calculations for Kinetic Test from Experimental Data..F2-1
Table F2.4: Results obtained from Kinetic Test..F2-2
Table F2.5: First Order Reaction Kinetics for 6 g/L..F2-3
Table F2.6: Second Order Reaction Kinetics for 6 g/L..F2-4
Table F2.7: First Order Reaction Kinetics for 0.6 g/L..F2-5
Table F2.8: Second Order Reaction Kinetics for 0.6 g/L..F2-6
Table F2.9: Mass Transfer Coefficient (k) calculated from First and Second Order Reaction Curves for Diameter 850 μm..F2-6
Table F3.1: Experimental Data for 6 g/L..F3-1
Table F3.2: Experimental Data for 0.6 g/L..F3-2
Table F4.1: Predicted Kinetic Data for Particle Diameter 850 μm and Concentration 6 g/L..F4-1
Table F4.2: Predicted Kinetic Data for Particle Diameter 600 μm and Concentration 6 g/L..F4-2
Table F4.3: Predicted Kinetic Data for Particle Diameter 850 μm and Concentration 0.6 g/L..F4-1
Table F4.4: Predicted Kinetic Data for Particle Diameter 600 μm and Concentration 0.6 g/L..F4-1

APPENDIX G: MINI COLUMN TESTS

Table G1.1: Initial Conditions for Hydrogen Form at pH 3 .... G1-1
Table G1.2: End Conditions for Hydrogen Form, Feed pH 3 .... G1-1
Table G1.3: Calculations for Mini Column Test with Initial Conditions from Table G1.1..G1-2
Table G2.1: Initial Conditions for Di Sodium Form, Feed pH 3 .... G2-1
Table G2.2: End Conditions for Di Sodium Form, Feed pH 3 .... G2-1
Table G2.3: Calculations for Mini Column Test with Initial Conditions from Table G2.1..G2-2
Table G3.1: Initial Conditions for Mono Sodium Form, Feed pH 3 .... G3-1
Table G3.2: End Conditions for Mono Sodium Form, Feed pH 3 .... G3-1
Table G3.3: Calculations for Mini Column Test with Initial Conditions from Table G3.1..G3-2
Table G4.1: Initial Conditions for Hydrogen Form, Feed pH 2.5 .... G4-1
Table G4.2: End Conditions Hydrogen Form, Feed pH 2.5 .... G4-1
Table G4.3: Initial Conditions for Di Sodium Form, Feed pH 2.5 .... G4-2
Table G4.4: End Conditions for Di Sodium Form, Feed pH 2.5 .... G4-2
Table G4.5: Initial Conditions for Mono Sodium Form, Feed pH 2.5 .... G4-3
Table G4.6: End Conditions for Mono Sodium Form, Feed pH 2.5 .... G4-3
Table G5.1: Initial Conditions for Hydrogen Form, Feed pH 2.04 .... G5-1
Table G5.2: End Conditions for Hydrogen Form, Feed pH 2.04 .... G5-1
Table G5.3: Initial Conditions for Di Sodium Form, Feed pH 2.04 .... G5-2
Table G5.4: End Conditions for Di Sodium Form, Feed pH 2.04 .... G5-2
Table G5.5 Mono Sodium Form, Feed pH 2.04 .... G5-3
Table G5.6: End Conditions for Mono Sodium Form, Feed pH 2.04 .... G5-3
Table G6.1: Initial Conditions for Hydrogen Form, Feed pH 1.59 .... G6-1
Table G6.2: End Conditions for Hydrogen Form, Feed pH 1.59 .... G6-1
Table G6.3: Initial Conditions for Di Sodium Form, Feed pH 1.59 .... G6-2
Table G6.4: End Conditions for Di Sodium Form, Feed pH 1.59 .... G6-2
Table G6.5: Initial Conditions for Mono Sodium Form, Feed pH 1.59 .... G6-3
Table G6.6: End Conditions for Mono Sodium Form, Feed pH 1.59 .... G6-3

APPENDIX H: MINIMUM FLUIDIZATION VELOCITY

Table H1.1: Experimental Calculation of Minimum Fluidization Velocity H1-1
Table H1.2: Theoretical Determination of Minimum Fluidization and Settling Velocity....H1-2
Table H1.3: t-Test (Two-Sample Assuming Equal Variances) H1-3

APPENDIX I: COLUMN TESTS

Table I1.1: Initial Conditions for Linear Velocity 0.13 cm/s I1-1

Table I1.2: End Conditions for Linear Velocity 0.13 cm/s I1-1

Table I1.3: Calculations for Column Tests with Initial Conditions from Table I1.1 .... I1-2

Table I2.1: Initial Conditions for Linear Velocity 0.195 cm/s I2-1

Table I2.2: End Conditions for Linear Velocity 0.195 cm/s I2-1

Table I2.3: Calculations for Column Tests with Initial Conditions from Table I2.1 .... I2-2

Table I3.1: Initial Conditions for Linear Velocity 0.41 cm/s I3-1

Table I3.2: End Conditions for Linear Velocity 0.41 cm/s I3-1

Table I3.3: Calculations for Column Tests with Initial Conditions from Table I3.1 .... I3-2

Table I4.1: Summary of Results obtained from Column Tests I4-1

APPENDIX J: COMPARISON OF CONTACTING EQUIPMENTS AT 6 g/L

Table J1.1: Initial Conditions for Fixed Bed at 6 g/L J1-1

Table J1.2: End Conditions for Fixed Bed at 6 g/L J1-1

Table J1.3: Calculations for Fixed Bed Test with Initial Conditions from Table J1.1 .... J1-2

Table J2.1: Initial Conditions for Fluidized Bed at 6 g/L J2-1

Table J2.2: End Conditions for Fluidized Bed at 6 g/L J2-1

Table J2.3: Calculations for Fluidized Bed Test with Initial Conditions from Table J2.1 .... J2-2

APPENDIX K: COMPARISON OF CONTACTING EQUIPMENTS AT 0.6 g/L

Table K1.1: Initial Conditions for Fixed Bed at 0.6 g/L K1-1

Table K1.2: End Conditions for Fixed Bed at 0.6 g/L K1-1

Table K1.3: Calculations for Fixed Test with Initial Conditions from Table K1.1 .... K1-2

Table K2.1: Initial Conditions for Fluidized Bed at 0.6 g/L K2-1

Table K2.2: End Conditions for Fluidized Bed at 0.6 g/L K2-1

Table K2.3: Calculations for Fluidized Bed Test with Initial Conditions from Table K2.1. K2-2

CHAPTER ONE: INTRODUCTION

Extensive studies for various applications have been conducted on the use of ion exchange processes. The literature is extensive and can be broadly categorized:

- Fundamentals of Ion Exchange
- Applications of Ion Exchange

The "fundamentals of ion exchange" deals with the theory behind ion exchange principles and involve the selection of an ion exchange material (resin), equilibrium and kinetics whereas an "application of ion exchange" deals with ion exchange at an industrial level.

1.1 Comparative Studies Conducted

Resins utilized in ion exchange processes have received extensive attention. The selection of the correct resin is of extreme importance to ensure that the recovery of the metal of interest is maximized. Research has been done on ion exchange materials and an effective resin was chosen. A number of comparative studies have been done.

An interesting study conducted by **[Ulmanu]** [35], examined a wide range of materials with adsorbent for use in the recovery of copper from solutions. The objective of the study was to identify a cheap, naturally occurring and non-polluting substance to utilize in the removal process. The synthetic zeolite ZSM-5 showed extensive promise.

A comparative study has been done by **[Raghavan and Bhatt]** [30] to determine the most suitable resin for the recovery of copper, by comparing chelating resins with different functional groups in the presence of other metal ions. The need for the study was due to there being no comparative performance data available for different commercially available resins.

A recent study conducted by **[Wyethe]** [37] examined the removal of copper and zinc from a cobalt electrolyte. The research incorporated a comparative study of two types of resin by batch contact with synthetic solution to determine the optimum resin for this application.

From theses studies it is evident that the effect of pH (on weak acid cation resin) and feed flow rate are important to find the optimum. The column studies that formed a significant part of all work mentioned above was performed using fixed bed columns.

Studies are available that describes contacting equipment for ion exchange processes and the literature available for the fixed bed system is extensive.

Articles published by **[Vermeulem]** [38] and **[Smith]** [33] discuss factors that effect ion exchange. **[Schmelzer]** [31] discusses the purpose behind fixed bed system.

Research conducted by **[Moison]** [26] using the *Michaels Concept* for "exchange zone heights" reveals a method for calculating heights of transfer units from ion exchange. **[Moison]** [26] findings led to the conclusion that the results of one or two fixed bed experiments will serve as a good basis for designing equipment (for operation involving unfavourable equilibria).

[Coulson and Richardson] [2] provide information on types of contacting equipment. **[Craig]** [21] also provides information on types of contacting equipment available but states that a comparative analysis on the different types of contacting equipment would require "comparative data from operations where each ion exchange system has been applied to almost identical feed waters". The author refers that there is insufficient data in literature for a quantitative comparison of the different types of equipment.

However, some work has been done by researchers on comparisons of different types of contacting equipment. **[Slater]** [15] provides information on fixed bed and counter current systems. **[Celik]** [20] compares a fixed bed ion exchange column and a fluidized column for the removal of of ammonia by natural clays.

1.2 Study Conducted by Author

The following represents a brief summary of the theoretical and practical research covered by the author.

1.2.1 Definition of Ion Exchange and Purpose of this Study

Ion exchange is a reversible chemical reaction where an ion from a solution is exchanged for a similarly charged ion attached to a stationary medium (that is, ion exchange material). The resin is held in a column through which solution is passed, allowing exchange to occur **[Internet]** [59]. The resin bed can be operated either in:

a down flow, in which case the bed is fixed, and solution is passed through the top of the column at a constant flow rate, or

b up flow, at a liquid velocity such that the particles are fluidized.

A fixed bed is when a liquid (for this research cupric sulphate solution was used) is passed through a bed of fine particles at a low flow rate so that the liquid just percolates through the void spaces between stationary particles. If the liquid was passed upward and the flow rate was increased, the particles would move apart. At a still higher velocity, a point is reached where all the particles are just suspended by a upward flowing liquid. This is considered to be a fluidized bed.

When the resin capacity is reached, the exchanged ions can be removed by elution. Elution is the desorption of the adsorbed solute by a solvent. The desorption solvent is the eluant, and the effluent stream containing the desorbed solute and eluting solvent is the eluate.

Although most ion exchange operations whether in the laboratory or in plant-scale processes are carried out in columns using fixed bed, developments enable the use of fluidized beds to be established.

The main purpose of this research was to compare the performance of a fixed bed ion exchange column with a fluidized ion exchange column for the recovery of copper from a cupric sulphate solution. The need for this study arises because although the literature on ion exchange is extensive, comparative work on contacting equipment is limited.

1.2.2 Test Work Conducted

A separate chapter was dedicated to all the experimental procedures conducted during experimentation. This is located in Chapter Ten. To ensure that the objectives of this research were being met the following were conducted:

1.2.2.1 Choice of Resin

Research had been conducted to determine the most suitable resin for this application. This is discussed in great detail in chapter two. An in depth analysis of the resin was performed to determine the characteristics experimentally.

The following were determined experimentally:

- Average particle size of the resin.
- Water retention capacity.
- The percentage swelling of the resin converted into the mono sodium and di sodium forms.
- Void volume packed in a column.

1.2.2.2 **Preliminary Experimental Test Work**

Since pH has an effect on weak cation resins, a pH test was included to determine the effect of pH on the loading of copper onto the resin.

Preliminary work was conducted to determine the equilibrium isotherms and kinetic response. Equilibrium isotherms were established at four different pH values to determine the maximum exchange capacity of the resin. The experimental data obtained for each equilibrium isotherm was modelled with a Langmuir and Freundlich isotherm **(Reference is made to Appendix E).**

Kinetic response was determined by measuring the concentration as a function of time. Two particle sizes were used to determine the kinetic response. These are discussed in full detail in Appendix F.

1.2.2.3 **Experimental Work**

Mini column tests were conducted which involved the conversion of the resin into two other resin forms.

To enable efficient comparisons of the fixed and fluidized bed, one linear velocity was used to operate both contacting equipment. The choice of the linear velocity required the following considerations to be taken into account:

- Minimum fluidization, so that the particles are fluidized in the fluidized bed system. [The Richardson and Zaki correlations enabled to compare experimental data with theoretical data].
- Tests were performed using three other linear velocities to determine an optimum linear velocity for fixed bed system.

To enable a pilot plant to be designed a series of design considerations needed to be taken into account together with the preliminary experimental test work. The pilot plant for both contacting equipment are discussed together with a cost estimation located in Appendix B **(Reference is made to Chapter Six)**. For each test the copper present in the effluent stream was determined by spectrophotometry principles **(Reference is made to Chapter Seven)**. When the resin became exhausted, analysis was done to determine how much copper was loaded onto the resin by elution. The results from the pilot plant testing enabled designs of a full scale plant for each contacting equipment together with cost estimations.

CHAPTER TWO: ION EXCHANGE MATERIALS

Ion exchange materials comprise of two main groups that is organic and inorganic exchangers. Both groups comprise of synthetic and natural materials. Ion exchangers form a very heterogeneous group of materials. Their only common feature is that they contain a fixed electric charge, which can bind counter ions with an opposite charge.

2.1 The Phenomenon

An ion exchange reaction may be defined as the reversible interchange of ions between a solid phase (the ion exchanger) and a solution phase; the ion exchanger is usually insoluble in the medium in which the exchange is carried out. If an ion exchanger $M^- A^+$, carrying cation A^+ as the exchanger ions, is placed in an aqueous solution phase containing B^+, an ion exchange reaction takes place which may be represented by Reaction 2.1.

$$\underset{\textbf{solid}}{\mathbf{M^-A^+}} + \underset{\textbf{solution}}{\mathbf{B^+}} \rightleftharpoons \underset{\textbf{solid}}{\mathbf{M^-B^+}} + \underset{\textbf{solution}}{\mathbf{A^+}} \qquad \textbf{(Reaction 2.1)}$$

The equilibrium represented by the above equation is an example of cation exchange. In much the same way, anions can be exchanged provided that an anion receptive medium is employed. An analogous representation of an anion exchange reaction may be written:

$$\underset{\textbf{solid}}{\mathbf{M^+A^-}} + \underset{\textbf{solution}}{\mathbf{B^-}} \rightleftharpoons \underset{\textbf{solid}}{\mathbf{M^+B^-}} + \underset{\textbf{solution}}{\mathbf{A^-}} \qquad \textbf{(Reaction 2.2)}$$

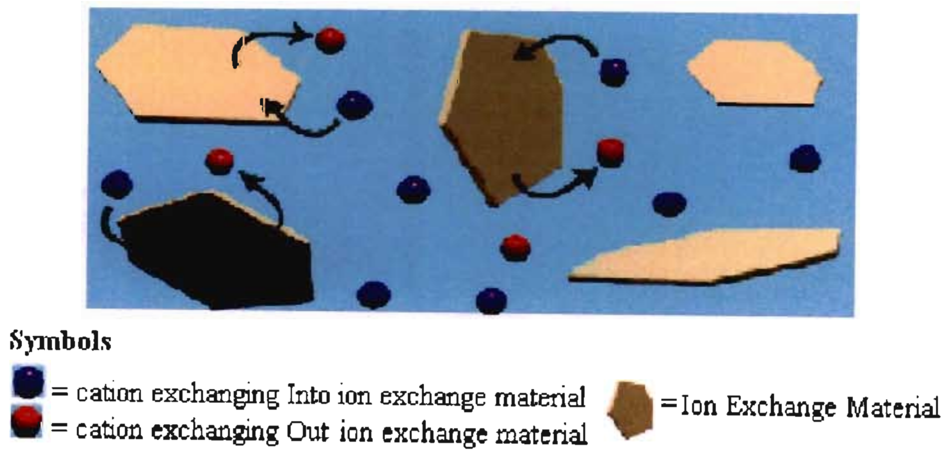


Figure 2.1: Representation of Cation Exchange on an Ion Exchangeable Material [Adapted from Internet] [66]

2.2 Resin

Ion exchange resins are a type of synthetic resin manufactured by introducing functional groups into a three dimensional crosslinked polymer matrix. Ion exchange resins are classified according to their function as a cation exchanger, which have positively charged mobile ions available for exchange and anion exchangers, whose exchangeable ions are negatively charged. Both anion and cation resins are produced from the same basic organic polymers **[Internet]** [59].

2.2.1 Types of Resin [Marcus] [8]

The types of resin available are discussed below.

2.2.1.1 Cation Exchange Resins

Cation exchange resins can be separated into two classes according to their functional groups:

2.2.1.1(a) Strong Acid Cation Resins

Strong acid resins are named because their chemical behaviour is similar to that of strong acids. The resins are highly ionized in both the acid ($R\text{-}SO_3H$) and salt ($R\text{-}SO_3Na$) form.

2.2.1.1(b) Weak Acid Cation Resins

In a weak acid resin, the exchangeable groups are carboxylic acid (COOH) as opposed to the sulfonic acid group (SO_3H) used in strong acid resins. These resins behave similarly to weak organic acids that are weakly dissociated. Weak acid resins exhibit a much higher affinity for hydrogen ions than do strong acid resins. This characteristic allows for regeneration to the hydrogen form with significantly less acid than is required for strong acid resins. Almost complete regeneration can be accomplished with stoichiometric amounts of acid. The degree of dissociation of a weak acid resin is strongly influenced by the solution pH.

2.2.1.2 Anion Exchange Resins

Anion exchange resins can be separated into two classes.

2.2.1.2(a) Strong Base Anion Resins

Like strong acid resins strong base resins are highly ionized and can be used over the entire pH range. These resins are used in the hydroxide (OH) form for water deionization. These resins react with anions in solution and can convert an acid solution to pure water.

2.2.1.2(b) Weak Base Anion Resins

Weak base resins are like weak acid resins, in that the degree of ionization is strongly influenced by pH. Consequently, weak base resins exhibit minimum exchange capacity above a pH of 7.0.

The costs of modern ion exchange resins are relatively high. For large-scale application, special factors to minimize the quantity of resin required for a specific duty (example: high capacity), and to prolong the resin lifetime (example: chemical and physical stability) are immensely important **[Marcus]** [8].

2.2.2 Considerations When Choosing a Resin

Typically when choosing a resin for a specific purpose the following considerations need to be taken into account:

2.2.2.1 Typical Particle Shape and Size

Generally the resins are supplied water wet in the form of spherical beads having a particle diameter between the range of 0.30 and 1.2 mm. Powder resins are also available for specific requirements.

2.2.2.2 Total Capacity and Operating Capacity

The total available capacity or total exchange capacity of a resin, expressed in equivalents per unit volume, is a measure of all the functional groups on a resin **[Rohm and Haas]** [12]. Test methods are available to measure the total exchange capacity of each type of ion exchange resin. The total exchange capacity is not necessarily the same as the operating capacity. The actual operating capacity is usually lower than the total capacity for an ion exchange process such as in recovery or purification applications. Some of these factors affecting the operating capacity are total and relative ion concentrations, charge density of ions, flow rate, temperature, pH, regeneration efficiency and equipment design.

2.2.2.3 Bulk Density and True Wet Density

Resin density is an important property because it determines the hydrodynamic behaviour in counter flow systems **[Rohm and Haas]** [12]. The *bulk density* is the weight of wet resin per unit volume. This density is measured in a calibrated glass column after backwashing the resin, allowing it to settle and draining off the water. The bulk density is specific for each resin and is dependent on the type of resin and ionic form.

The *true wet density* or specific gravity is determined on the wet resin. The resin sample is weighed and the volume is found by water displacement using a pycnometer. Values range from about 1.04 to about 1.25. Cation exchangers have a greater true wet density than anion exchangers.

2.2.2.4 Swelling and Shrinking

Ion exchange resins carry both fixed and mobile ions which are always surrounded by water molecules located in the interior of the resin beads. Water wet ion exchange resins shrink or swell when they change from one ionic form to another and they shrink when they are in contact with non-polar solvents. This research looked at the water retention capacity (how much water is in the pores of the resin) and the conversion of resin from the hydrogen form to the di sodium and mono sodium forms of the TP 207 resin.

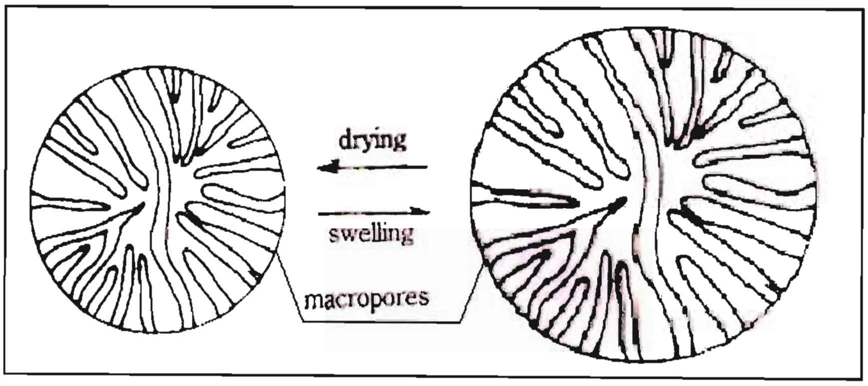


Figure 2.2: Schematic Representation of Drying and Swelling of a Resin Bead [Mitsubishi] [11]

2.2.2.5 Stability

The resin bead needs to be stable when it is exchanging ions. Two types of stability are discussed below:

2.2.2.5(a) Physical Stability

The polymeric beads of resins should be physically very stable. The sulfonic group of cation-exchange resins are extremely stable. Anion exchange resins are temperature sensitive.

2.2.2.5(b) Chemical Stability

Ion exchange resins are stable over the full range of pH. They are virtually stable to most inorganic or organic chemicals except for strong oxidants such as dissolved chlorine, ozone or peroxides.

2.2.2.6 Pressure Drop

The pressure drop is directly proportional to the flow rate, the viscosity of the feed, and the depth of the resin bed, and is inversely proportional to the square of the diameter of the resin beads. Usually one bar of pressure drop across a bed of ion exchange resin should not be exceeded.

2.2.3 Resin Chosen: Lewatit TP 207 Weak acid Cation Resin

Studies were conducted by **[Wyethe]** [37] to determine the most selective and cost effective resin for copper recovery. The study involved the use of two other resins to select the best resin for copper recovery. The resin that was selected by **[Wyethe]** [37] was a Lewatit TP 207 resin. Therefore a TP 207 resin was chosen for all experimental work covered in this research.

2.2.3.1 Safety Data on Lewatit TP 207 (Weak acid Cation Resin)

TP 207 is a chelating weak acid cation resin. The resin contains functional groups which form chelates (complexes) with metal ions, binding them to the resin in the process. The functional group of the carboxylic type gives high chemical efficiency in many applications. This resin is used to selectively recover transitional metals from aqueous solutions. TP 207 is insoluble in acids, alkalis, and all common solvents. The ion exchange behaviour of the resins is chiefly determined by the fixed ionic groups. The number of groups determines the ion exchange capacity. The chemical nature of the groups greatly affects ion exchange equilibria. An important factor is the acid or base strength of the groups. Weak acid groups such as -COO- are ionized at high pH. At low pH they combine with H^+, forming an undissociated -COOH. The weak acid resin is pH dependant.

TABLE 2.1: Safety Data on Lewatit TP 207 Resin

General Description

Ionic form, as shipped	Na^+
Functional Group	iminodiacetic acid
Matrix	cross linked polystyrene
Structure	macroporous
Appearance	beige, opaque

Chemical and Physical Properties

Bead size* min. 90%		mm	0.4-1.25
Effective Size*		mm	0.55 (± 0.05)
Uniformity coefficient*		max	1.7
Bulk weight (± 5%)		g/L	800
Density		approx. g/ml	1.18
Water Retention		%wt	50-55%
Total capacity* (H^+ - form)		min.eq/L	2.4
Volume-change ($Na^+ \rightarrow H^+$)		approx. %	-30
Stability	temperature range	°C	-20 up to 80
	pH range		0-14
Storability	of product	min years	2
	temperature range	°C	-20 up to 40

*Recommended Operating Conditions***

Operating temperature		max. °C	80	
Operating pH range			1.5-9	
Bed depth		min mm	1000	
Pressure loss(15 °C) perm/h		approx. kPa/m	1.1	
Pressure loss		max. kPa	250	
Flow velocity	exhaustion	max.m/h	40	
	backwash (20 °C)	approx. m/h	10	
Regeneration with			HCL	H_2SO_4
Co-Current	Level (form as shipped)	approx. g/L	140	200
	concentration	%	4-10	5-15
Flow velocity	regeneration	approx. m/h	5	
	Rinsing	max. m/h	5	
Conditioning with			Mono-Na:	Di-Na:
Co-Current	Level (form as shipped)	g/L	40-48	80-96
	concentration	approx. %	4	
Flow velocity	conditioning	approx. m/h	5	
	rinsing	min. m/h	5	
Rinse water requirement		approx. bed vol.	5	
Bed Expansion (20 °C) per m/h		approx. %	4	
Freeboard (as % of resin volume)		approx.	80	

*These values are specification values and are subject to continuous monitoring.

** The information given here refers to use of the product under normal operating conditions and is based on tests in pilot plant and data obtained from industrial installations. However, additional data are needed to calculate the resin volumes required for ion exchange units. These are to be found in our Technical Information Sheets.

Note:

Safety data was obtained from the suppliers of the resin (Bayer).

CHAPTER THREE: EQUILIBRIA AND KINETICS

3.1 Ion Exchange Equilibrium

When a resin bead containing a mobile ion is placed in water containing a different ion of the same charge sign, an equilibrium situation will be achieved when the thermodynamic chemical potential for each ion is the same inside and outside the resin. A plot of solute concentration in the resin against concentration in the solution is called an isotherm if temperature is held constant **[Slater]** [15].

Two methods could be used for the equilibria tests namely:

- Batch Method
- Column Method

3.1.1 Batch Method

Aliquots of resin in a given form must be weighed. The resin is then added to known volumes of solution of constant ionic concentration. After mechanical agitation for several hours, samples of solution may be analyzed. This method is only suitable when the volume of the resin is much smaller than that of the solution, in the case of treating dilute solutions (that is 0.1 M or less). Resin densities will be required for representation of concentrations on a resin volume basis.

3.1.2 Column Method

A quantity of known form resin is placed in a small column. A solution of known composition is then passed through the resin bed until the effluent has the same composition as the feed. The resin is then rinsed and then totally eluted. A sample of elute collected is used to calculate the resin composition. The batch method was used to do equilibrium testing on the resin.

3.2 Ion Exchange: Mass Balance

(The following analysis was used for pH, equilibrium and column testing.)

Initially: The resin is loaded into a column or batch **(Refer to Figure 3.1)**. The volume of a chelating resin depends on the pH of the resin. Thus, a resin volume to be used for a specific test is measured as a tapped volume at a specific pH. The resin has an initial volume referred to as V_{ro}.

Excess copper solution is added to the resin. The copper solution has total copper concentration C_o and an initial volume V_o.

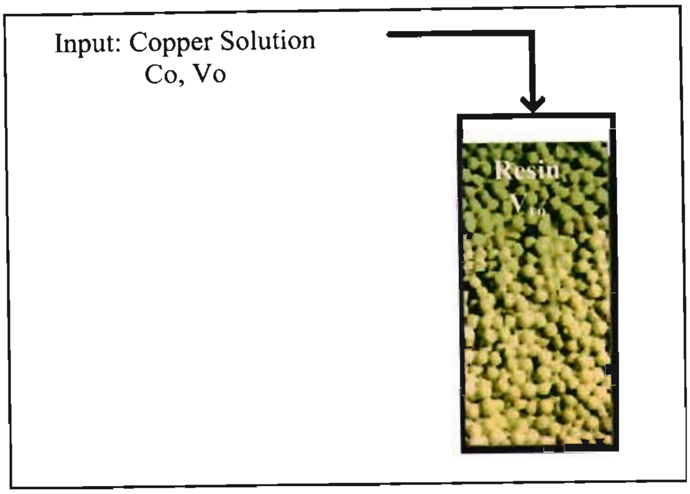


Figure 3.1: Initial Conditions at the Start of Operation

When the copper comes into contact with the resin, ion exchange occurs. When the resin is fully loaded with copper the process is stopped.

At the end of the process:

- Some of the copper is adsorbed on the resin (The resin is saturated with copper.) and,
- the remainder of the copper is in solution.

The volume of resin which is adsorbed with copper V_C, is separated from the copper solution that has a volume V_s and a concentration C_e.

The resin that is loaded with copper is eluted with H_2SO_4 solution **(Refer to Figure 3.2)**. The copper solution that is recovered has a solution volume V_{cu} and concentration C_{el}. The resin thus gets back to its initial form V_e. The accountability of the copper was calculated for all test work which is represented in Equation 3.1.

$$\text{Accountability} = \frac{\text{Amount of copper from solution} + \text{Amount of copper from elution}}{\text{Total amount of copper}}$$

.... **(Equation 3.1)**

The above hypothesis was used to obtain the adsorbent concentration q_e from adsorption which was obtained by mass balance **(Reference is made to Equation 3.2)** and compared to Q_e from elution calculated as (mass of copper that was recovered / volume of resin that was eluted) for all tests.

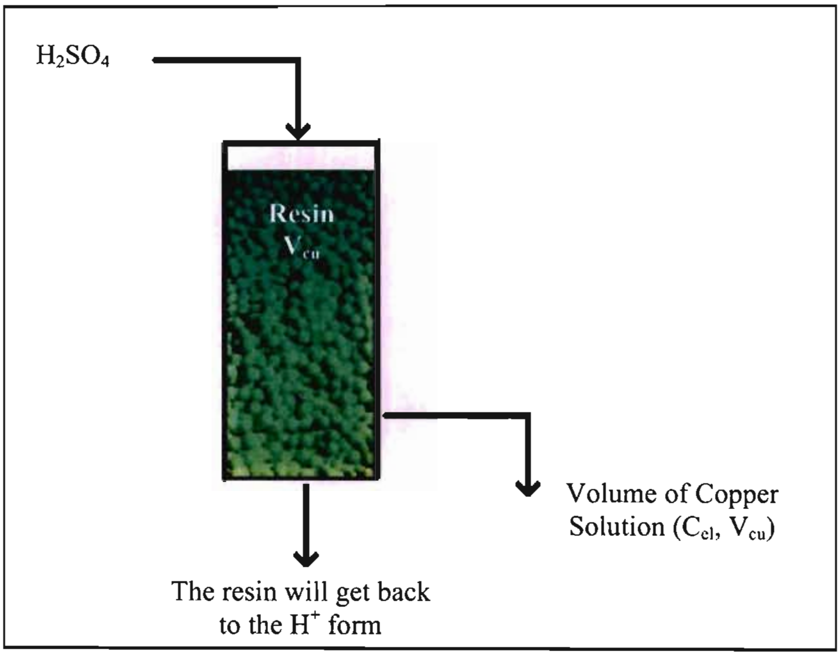


Figure 3.2: Conditions at the End of Operation

$$q_e = \frac{(C_o - C_e) * V}{V_{ro}} \quad \text{...} \quad \textbf{(Equation 3.2)}$$

Where

q_e : is the adsorbate adsorbed per volume of adsorbent

C_o : initial concentration of Cu^{2+} solution (adsorbate)

C_e : final equilibrium concentration of adsorbate

V : volume of liquid

V_{ro} : volume of adsorbent (mL)

3.3 Adsorption Isotherm Models

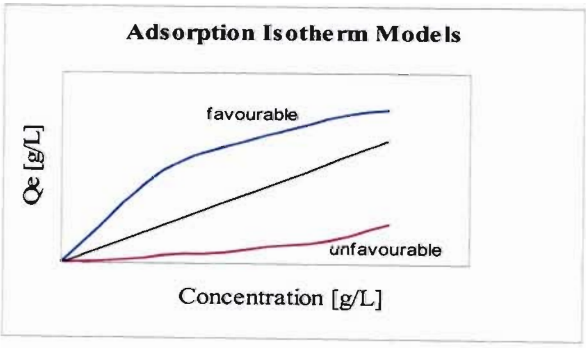


Figure 3.3: Plots of Adsorption Isotherm Models (Adapted from [Internet] [39])

The shape of the curves in Figure 3.3 is significant and factors into design. "Favourable" isotherms permit higher solid loadings at lower solution concentrations. These tend to start out steep and level out.

Isotherms that start out flat are "unfavourable", because they only work well at high concentrations of solute. Several fits have been proposed for isotherms. A linear isotherm seems to work for very dilute solutions, but not for many others. The *Freundlich isotherm* describes physical adsorption from liquids.

The results from the equilibrium data will be used to fit two different adsorption models.

- Freundlich Isotherm
- Langmuir Isotherm

Freundlich isotherm adsorption model is of the form:

$$q_e = K_f C_e^{\ 1/n} \quad \text{...}\textbf{(Equation 3.3)}$$

(Adapted from [Internet] [68])

Langmuir isotherm adsorption model is of the form:

$$q_e = \frac{ab * C_e}{(1 + bC_e)} \quad \text{...}\textbf{(Equation 3.4)}$$

(Adapted from [Internet] [69])

Where

K_f, $1/n$, a, b, are the coefficients of interest (Fitting parameters).

3.4 Selectivity of Ion Exchange Resins

The following parameters describe the selectivity of copper onto the resin.

3.4.1 Separation Factor [Vermeulem] [20]

In binary exchange involving only one exchanging ionic species [Cu^{2+}] in the feed solution and one other species [H^+] initially on the resin, the separation factor is defined as:

$$r_{H^+}^{Cu^{2+}} = \frac{C_{Cu^{2+}}(Q - q_{Cu^{2+}})}{q_{Cu^{2+}}(C_o - C_{Cu^{2+}})} = \frac{x(1-y)}{y(1-x)} \quad \text{.....................}\ \textbf{(Equation 3.5)}$$

Where

q_{Cu} : is the concentration of Cu^{2+} on the resin

Q : is the total resin concentration of both $[Cu^{2+}]$ and $[H^+]$

C_o : initial concentration of $[Cu^{2+}]$

$C_{Cu^{2+}}$: Concentration of copper in solution

The available solutions for breakthrough curves may be classified into the following categories of equilibrium behaviour:

- Strongly favourable (with $r \leq 0.3$); in the limiting case, irreversible (r=0)
- Linear (r =1)
- Non Linear ($0.3 < r < 10$)
- Strongly unfavourable ($r > 10$)

3.4.2 Selectivity Coefficient [Helfferich] [6]

Selectivity is a characteristic of an ion exchanger, which makes the exchanger prefer one counter ion to another; thus selectivity drives the reaction either to the left hand or right hand side.

For any exchange reaction, such as:

$$2\overline{H^+} + Cu^{2+} \Leftrightarrow \overline{Cu^{2+}} + 2H^+ \text{ ....}\textbf{(Reaction 3.1)}$$

Where: —— Denotes a counter ion in the resin

A corresponding selectivity coefficient is found:

$$K_{H^+}^{Cu^{2+}} = \frac{[\overline{Cu}][H]^2}{[\overline{H}]^2[Cu]} \text{ .... }\textbf{(Equation 3.6)}$$

The greater the selectivity coefficient, the greater the preference for the ion exchanger.

K increases with:

- Ionic valence
- Inverse of hydrated ionic radius
- Degree of polarization
- Inversely with degree of complexation in solution

Cationic Preference Series

$$Ba^{2+} > Pb^{2+} > Sr^{2+} > Ca^{2+} > Ni^{2+} > Cd^{2+} > Cu^{2+} > Co^{2+} > Zn^{2+} > Mg^{2+}$$

$$Ag^{+} > Cs^{+} > K^{+} > NH_4^{+} > H^{+}$$

Anionic Preference Series

$$SO4^{2-} > I^{-} > NO_3^{-} > HCrO_4^{-} > Br^{-} > Cl^{-} > OH^{-}$$

3.4.3 Distribution Coefficient [Mitsubishi] [17]

The distribution coefficient may be defined as the amount of copper present on the resin divided by the amount of copper in solution **(Refer to Equation 3.7)**.

$$\alpha = \frac{\overline{[Cu^{2+}]}}{[Cu^{2+}]} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad \textbf{(Equation 3.7)}$$

3.5 Kinetics

A batch system was used to determine the rate law parameters for the ion exchange process. This determination is achieved by measuring concentration as a function of time. The integral method was then used to determine the reaction order α, and specific reaction rate, k.

3.5.1 Diffusional Steps

The effective rate of exchange is determined by one or more of several alternate diffusional steps. Appreciable differences in the shapes of breakthrough curves are encountered only with strongly favourable equilibrium.

The diffusional steps are:

Step 1: Movement of the ions from bulk of solution.
Step 2: Diffusion of the ions through the laminar film.
Step 3: Diffusion of the ions through the pores.
Step 4: Ion exchange.
Step 5: Diffusion of the exchanged ions outward.
Step 6: Diffusion of the exchanged ions through laminar layer.
Step 7: Movement of exchanged ions into bulk of solution.

Mechanisms for step two or step three tend to control exchange rate.

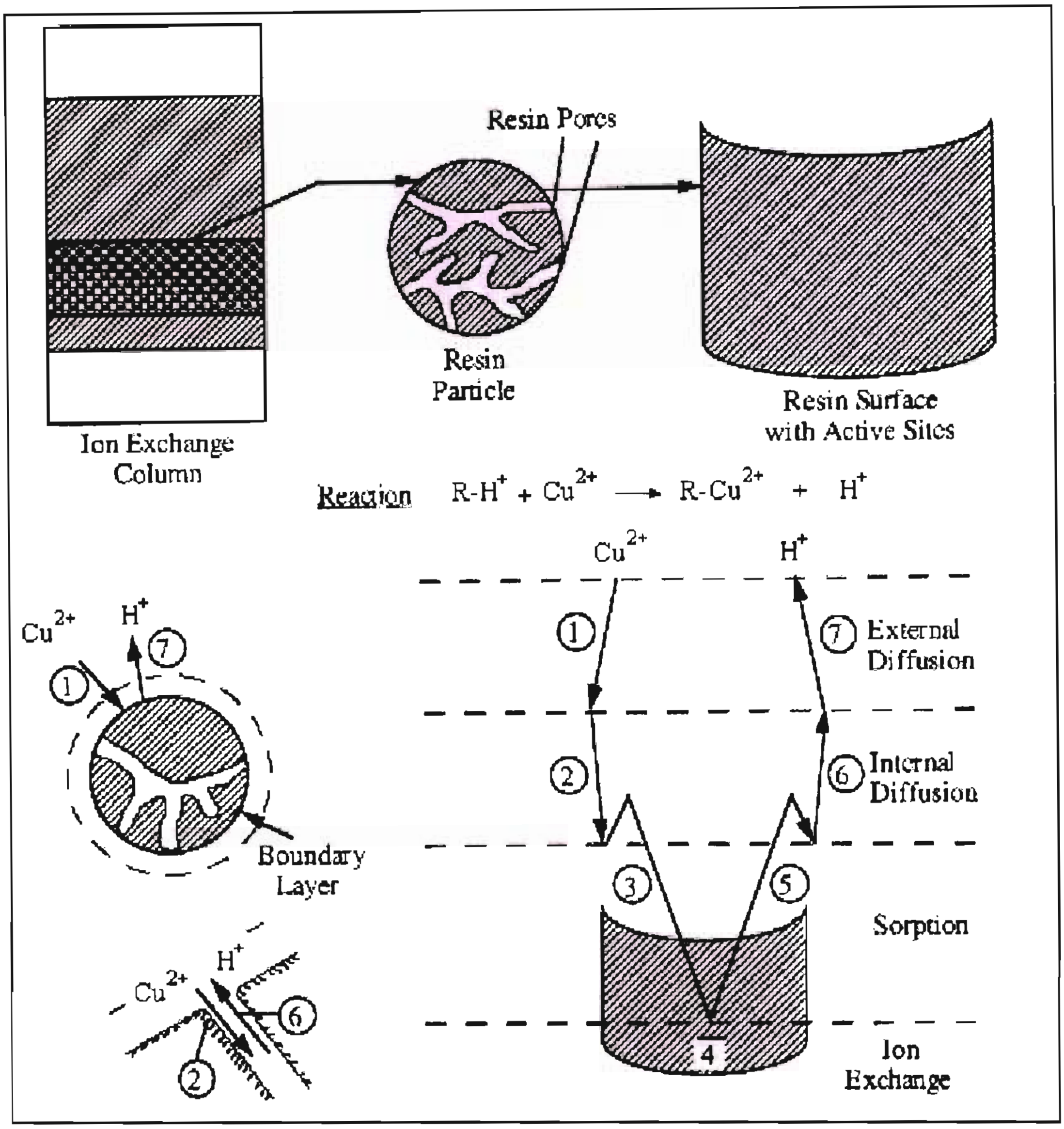


Figure 3.4: Steps in an Ion Exchange Reaction (Adapted from [Environex] ^{3})

3.5.2 Integral Method [Fogler] ^{10}

The integral method of analysis of rate data looks for the appropriate function of concentration corresponding to a particular rate law that is linear with time.

3.5.2.1 Order of Reaction

The integral method uses a trial and error procedure to find the reaction order. The zero, first and second order reaction is discussed below.

3.5.2.1(a) Zero Order

For a zero order reaction the combined rate law and mole balance is:

$C_{Cu} = C_{Cu_o} - kt$ .... **(Equation 3.8)**

A plot of C_{CU} as a function of time will be linear with slope (-k) for a zero order reaction carried out in a constant volume batch system.

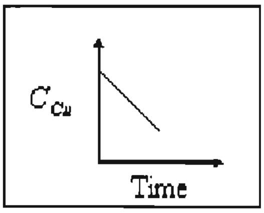


Figure 3.5: Zero-Order Reaction

3.5.2.1(b) First Order

If the reaction is first order, integration of the combined mole balance and the rate law:

$-\frac{dC_{Cu}}{dt} = kC_{Cu}$ .... **(Equation 3.9)**

With the limits Ccu = Ccu_o at t=0 gives:

$\mathrm{In}\left(\frac{C_{Cu_o}}{C_{Cu}}\right) = kt$ .... **(Equation 3.10)**

The slope of a plot of $\mathrm{In}\left(\frac{C_{Cu_o}}{C_{Cu}}\right)$ as a function of time is linear with slope k.

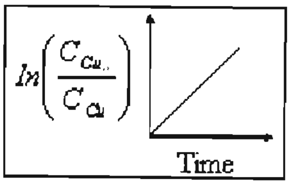


Figure 3.6: First-Order Reaction

3.5.2.1(c) Second Order

If the reaction is second order, then

$$-\frac{dC_{Cu}}{dt} = kC_{Cu}^{2} \quad \text{...} \quad \textbf{(Equation 3.11)}$$

$$\frac{1}{C_{Cu}} - \frac{1}{C_{Cu_o}} = kt \quad \text{...} \quad \textbf{(Equation 3.12)}$$

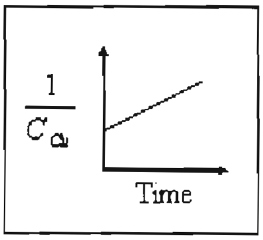


Figure 3.7: Second-Order Reaction

The above method was used to determine the order of the reaction and the mass transfer coefficient k **(Refer to Appendix F for results on Kinetic Tests)**.

CHAPTER FOUR: BREAKTHROUGH TEST

4.1 The "Mass Transfer Zone"

The following below illustrate the principles and calculations of the height of the resin bed required for design purposes.

4.1.1 Definition and Principle

A component can be separated from a mixture if the component is selectively adsorbed onto a solid surface (that is ion exchange resin). Ion Exchange occurs mainly on the pore walls "inside" the particles. The amount of material exchanged within a resin bed depends both on position and time **[Internet]**[39]. The time dependence is considered below.

When fluid enters the resin bed **(Refer to Figure 4.1)**, it comes in contact with the first few layers of weak acid cation resin. Solute (Cu^{2+} from the $CuSO_4.5H_2O$) adsorbs, filling up some of the available sites. Soon, the adsorbent near the entrance is saturated and the fluid penetrates further into the bed before all solute is removed **(Refer to Figure 4.2)**. Thus the active region shifts down through the bed as time goes on. This region is referred to as the mass transfer zone. It is a specific section of the bed in which the reaction between the resin and the copper solution occurs **(Refer to Figure 4.3)**.

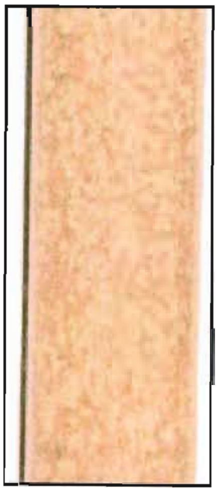


Figure 4.1: Photograph of Resin Bed (TP 207 resin in the hydrogen form)

The fluid that emerges from the bed will have little or no solute remaining at least until the bulk of the bed becomes saturated.

Figure 4.2: Photograph of first few layers of Resin Bed adsorbed with Copper in a Fixed Bed Ion Exchange Column

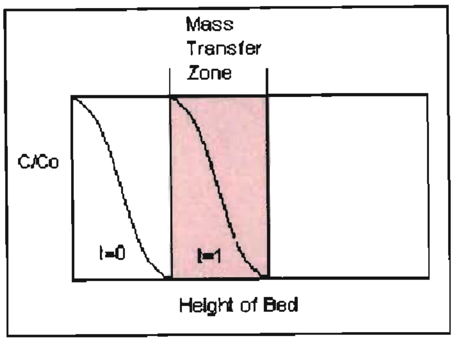


Figure 4.3: A Plot of the Concentration Profile of adsorbate in the fluid phase as a function of Distance along the Adsorbent Bed [Internet] [39]

The breakthrough point occurs when the concentration of the solution leaving the bed spikes as unabsorbed and solute begins to emerge. The bed has become exhausted **(Refer to Figure 4.4)**.

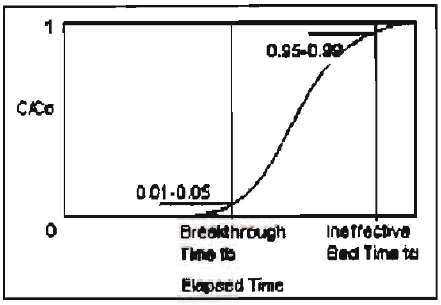


Figure 4.4: A Typical plot of the ratio of Outlet solute concentration to Inlet solute concentration in the fluid as a function of Time from the start of flow [Internet] [39]

Usually, a breakthrough point composition is set to be the maximum amount of solute that can be acceptably lost, typically between 1 and 5 percent **[Internet]** [39].

As the concentration wave moves through the bed, most of the mass transfer is occurring in a small region. This mass transfer zone moves down the bed until it "breaks through". The shape of the mass transfer zone depends on the adsorption isotherm (equilibrium expression), flow rate, and the diffusion characteristics.

4.1.2 Calculations of the Mass Transfer Zone Height

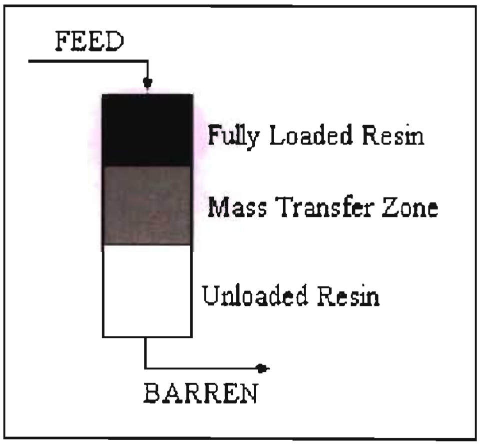


Figure 4.5: Schematic Representation of the Mass Transfer Zone in a fixed bed ion exchange column (Adapted from [Wyethe] [24])

Refer to Figure 4.6 and Equation 4.1:

$$h = \left[\frac{Z_2 - Z_1}{Z_2}\right] * H$$ ....**(Equation 4.1) [Wyethe]** [24]

Where

h : mass transfer zone height

Z_1 : Run time at maximum tolerable impurity

Z_2 : Run time at 80% breakthrough

H : Total height of resin bed used for evaluation

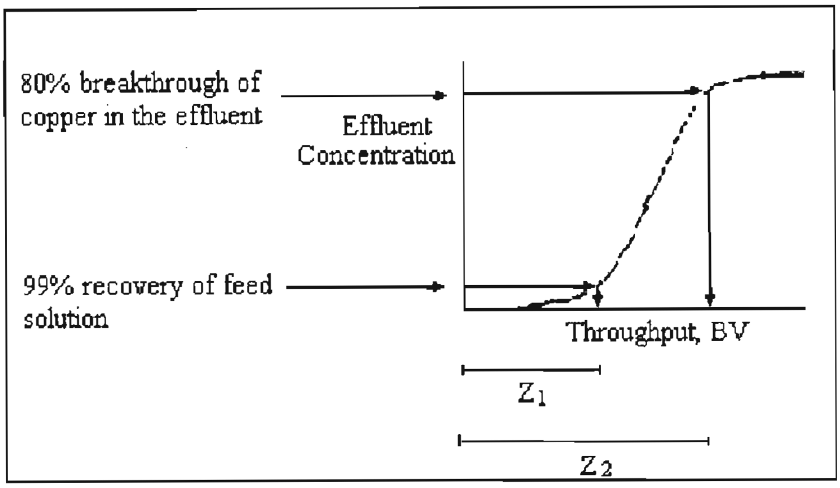


Figure 4.6: Calculation of Mass Transfer Zone from Breakthrough Tests

Breakthrough tests were conducted at three different superficial linear velocities to determine the effect of an increase in linear velocity on the height of the mass transfer zone and during pilot plant operation.

For comparative purposes the following was used as a basis for all calculations.
For all breakthrough tests conducted, the mass transfer zone height was based on the feed throughput required at maximum tolerable impurity and was chosen at 99% recovery of initial feed solution, that was required to obtain 80 % breakthrough of copper in the effluent.

CHAPTER FIVE: FIXED AND FLUIDIZED BEDS

To compare the performance of the fixed and fluidized bed systems, background knowledge of both systems needed to be understood first. Therefore this section was dedicated to understanding the principles behind both contacting equipment.

If a liquid is passed through a bed of fine particles, at a low flow rate, the fluid just percolates through the void spaces between the stationary particles. This is known as a fixed bed.

If the liquid was passed upward and the flow rate was increased, the particles would move apart. At a higher velocity, a point is reached where all the particles are just suspended by an upward flowing liquid. This is a point when the frictional forces between the particles and the fluid counterbalance the weight of the particles. The bed is considered to be just fluidized and is referred to as a bed at minimum fluidization **[Kunii and Levenspiel]** [7]. The main difference between a fixed bed system and a fluidized bed system is the direction of flow **(Refer to Figure 5.1 below)**.

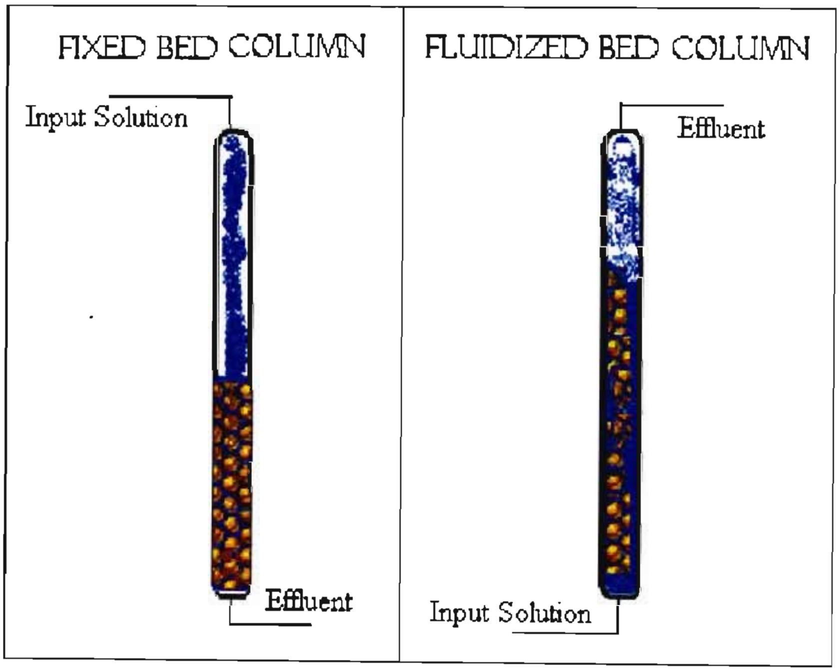


Figure 5.1: Fixed Bed versus Fluidized Bed

5.1 **Fixed Beds**

A typical fixed bed is a cylindrical column that is filled with a suitable material for the purpose that it must perform. The liquid is distributed as uniformly as possible at the top of the column and flows downward, wetting this material. From a fluid mechanical perspective, the most important issue is the pressure drop required for the liquid to flow through the column at a specified flow rate.

5.2 **Fluidized Beds**

Fluidization is the interaction between a liquid and solid in which the solid propels freely throughout the bed. Important parameters for the analysis of the fluidized bed include properties of both the liquid and solids stream. All the parameters listed below effect fluidization. These include:

- Particle diameter
- Particle shape
- Liquid density (in this case – cupric sulphate)
- Particle density
- Porosity / Void volume
- Liquid viscosity

The properties of both the liquid and solids stream effect fluidization behaviour. This research deals with liquid-solid fluidization, also known as particulate fluidization. Particulate fluidization utilizes an upward flow of liquid to fluidize a bed of solid particles. Fluidization is the point at which a packed bed of solids begins to exhibit fluid like properties, such as movement of the solid particles in the bed and the expansion of the bed. In particulate fluidization, the solid particles are continuously agitated and mixed. The most common reason for fluidizing a bed is to obtain vigorous agitation of the solids in contact with the fluid, leading to excellent contact of:

- the solid and the fluid
- and the solid and the wall

The particles act independently from one another in a liquid-solid system as opposed to a gas-liquid system, where the particles tend to stick together. Once the liquid supply is shut off, the solids settle back to their original position. If the solid particles are forced to the top of the column due to fluid flowing faster than the particles settling velocity, the particles at this point are no longer fluidized but are acting as a packed bed.

5.2.1 Analysis of Experimental Determination of Minimum Fluidization Velocity [Internet] [54]

The behaviour of a bed of particles when the upward superficial fluid velocity is slowly increased from zero past the point of minimum fluidization and back to zero was considered. The representation is present in Figure 5.2.

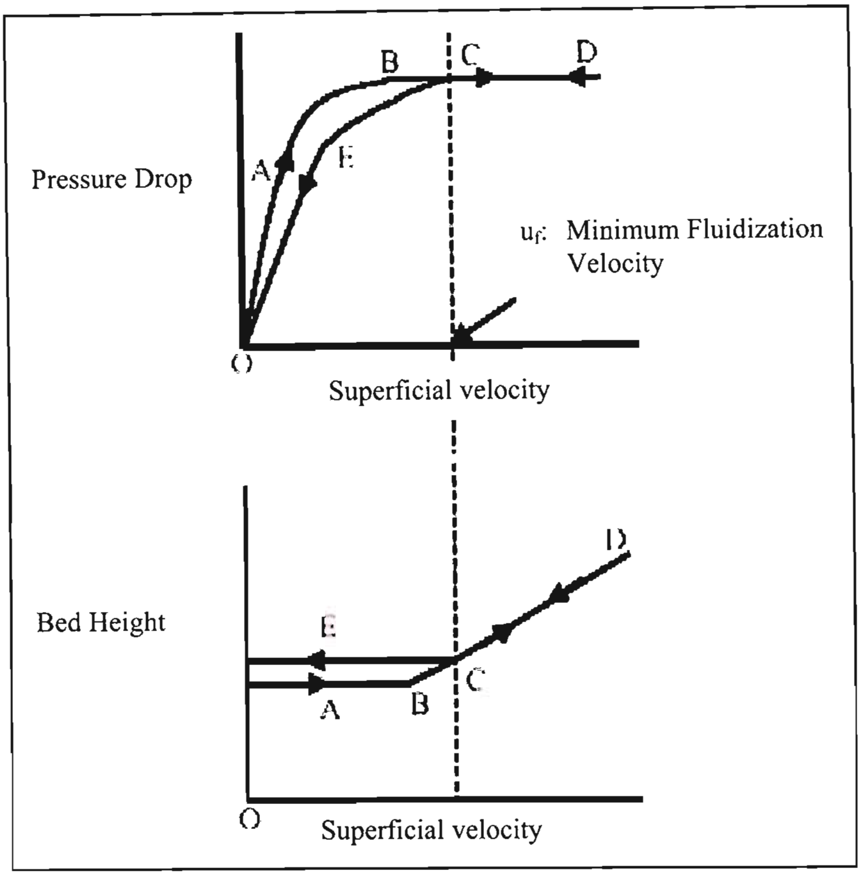


Figure 5.2: Graph of Pressure drop and Bed Height versus Superficial Velocity (Adapted from [Internet] [54])

When there is no flow, the pressure drop is zero, and the bed has a certain height. Proceeding along the right arrow in the direction of increasing superficial velocity, and tracing the path ABCD, the pressure drop gradually increases while the bed height remains fixed.

When point B is reached, the bed starts expanding in height while the pressure drop levels off and does not increase further as the superficial velocity is increased. This occurs when the upward force exerted by the fluid on the particles is sufficient to balance the net weight of the bed and the particles begin to separate from each other and float in the fluid. As the velocity is increased further, the bed continues to expand in height, but the pressure drop remains constant.

If a path is traced backward, gradually decreasing the superficial velocity, in the direction of the reverse arrows in Figure 5.2, the behaviour of the bed follows the curves DCE. At first, the pressure drop remains fixed while the bed settles back down, and then begins to decrease when point C is reached. The bed height no longer decreases while the pressure drop follows the curve CEO. If the procedure is repeated by increasing the superficial velocity from zero, the set of curves ECD will be followed in both directions. Because of this reason, the velocity at the point C in the Figure 5.2 is defined as the *minimum fluidization velocity* u_f.

For Reynolds number that is relatively small ($Re \leq 10$) the Kozeny-Carman Equation can be used to determine the minimum fluidization velocity. This is applicable to the viscous flow regime, for establishing the point of onset of fluidization:

$$u_f = \frac{(\rho_P - \rho_f) * g \; D_P^2}{150\mu} \frac{\varepsilon^3}{1-\varepsilon} \quad \text{.................................}\textbf{(Equation 5.1)}$$

(Adapted from [Internet] ^{54})

Equation 5.2 refers to superficial velocity so that particles are not carried out with the fluid at the exit. This would occur if the superficial velocity were equal to the terminal (settling) velocity of the particles.

$$u_T = \frac{(\rho_P - \rho_f) \; g \; D_P^2}{18\mu} \quad \text{..}\textbf{(Equation 5.2)}$$

(Adapted from [Internet] ^{54})

5.2.2 Richardson Zaki Correlation

The Richardson Zaki equation models fluidization velocity as a function of void volume based on several particles.

$$u = u_T \varepsilon^n \quad \text{..}\textbf{(Equation 5.3) [Carsky]}^{70}$$

Where

u_T : Terminal velocity

n : Constant which is related to Reynolds number **(Refer to Table 5.1)**

ε : Void volume which is related to linear velocity **(Refer to Equation 5.3)**

u : Linear velocity

Due to n being a factor based on Reynolds' number, by holding the Reynolds number and terminal velocity constant, a variety of projected velocities and void fractions can be examined.

TABLE 5.1: Determination of n (Function of Reynolds Number) [Carsky] [70]

Re_t	n
0-0.2	4.6
0.2-1	$4.4\ Re_t^{-0.33}$
1-500	$4.4\ Re_t^{-0.1}$
>500	2.4

5.2.2.1 Void volume as a function of Bed Height

Void volume can be related to bed height with the following principles:

$$V = V_s + V_{voids} \quad \textbf{(Equation 5.4)}$$

$$V_{mf} = V_s + V_{voidsmf} \quad \textbf{(Equation 5.5)}$$

Where

V : Total volume of packed bed resin ($V = A*h$)

V_s : Volume of resin solids

V_{voids} : Volume of voids [$V*\varepsilon$]

$$V - V_{voids} = V_{mf} - V_{voidsmf} \quad \textbf{(Equation 5.6)}$$

$$V(1-\varepsilon) = V_{mf}(1-\varepsilon_{mf}) \quad \textbf{(Equation 5.7)}$$

$$H(1-\varepsilon) = H_{mf}(1-\varepsilon_{mf}) \quad \textbf{(Equation 5.8)}$$

$$\varepsilon = 1-(1-\varepsilon_{mf})\frac{H_{mf}}{H} \quad \textbf{(Equation 5.9)}$$

The Kozeny-Carman Equation **(Equation 5.1)** was used to calculate the minimum fluidization velocity and detailed calculations are present in Appendix C3. The minimum fluidization velocity was also determined experimentally, using the above explanation **(5.2.1 Analysis of experimental determination of minimum fluidization velocity)**. The Richardson Zaki correlations were used to model predicted results with experimental results and are located in Appendix H.

CHAPTER SIX: DESIGN CONSIDERATIONS

6.1 Vessel Sizing and Requirements

The following are considerations taken into account when the pilot plant for the contacting equipment was constructed.

6.1.1 Vessel Sizing

The vessels were made from typical, well-known materials. Sight-glasses for the columns was advisable to use in order to check resin levels and separation that occurs inside the columns, therefore perspex was chosen to construct the ion exchange columns. For good distribution of the solution in the fixed bed column, a sieve was installed on the top of the column so that the solution entering the resin bed will be evenly distributed.

The design of the vessels should give a maximum resin bed depth, whilst limiting the pressure drop across the resin bed to approximately one bar. The optimum column diameter must be a balance between the resin bed height, the ratio of resin bed height to diameter (H/D) and the linear velocity. H/D should be in the range 2/3 to 3/2 **[Internet]** [37].

To eliminate wall effects the column diameter of the pilot plant must be at least twenty times that of the resin particle diameter **[Wyethe]** [37]. For this reason the inner column diameter was chosen for the fixed and fluidized bed operation at 2.4 cm. Vessel sizing should be adjusted to allow for resin expansion for backwashing (80-100% of the settled resin bed height), resin swelling during service and for regeneration purposes. The height of the column was chosen at 1 m for the fixed bed system and 2m for the fluidized bed system.

Industrial size columns use a maximum vessel diameter of 3.5m (11.5 feet) and typical resin bed depth is 1.2 m (4 ft) for co-current and block regeneration systems and 2 m (6.5 ft) for counter-current packed bed systems **[Internet]** [37].

6.1.2 Product Requirements

The required product quality helps define the regeneration system. For high quality, a counter-current regeneration system should be used **(Refer to Chapter Eight)**.

6.1.3 Feed Composition

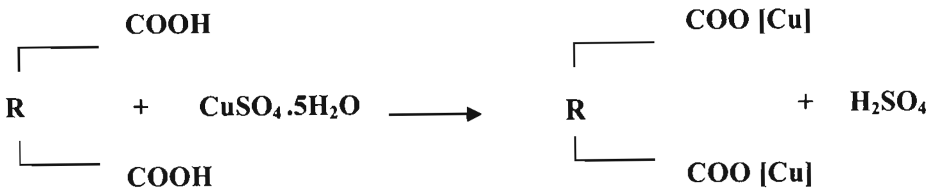
Feed composition and temperature are known parameters. Feed compositions of 6 and 0.6 g/L copper was used at room temperatures.

6.2 Schematic Representation of Designs for Ion Exchange Columns

A solution of Cu^{2+} ions as $CuSO_4 .5H_2O$ (blue solution) was passed through a column containing a polymer matrix of cross linked polystyrene (TP 207 resin), for exchanging positively charged ions, in the form of spherical beads at a constant flow rate **(Refer to Figures 6.1 and 6.2)**. The resin was a weak acid cation resin and the exchange group is the carboxylic acid (COOH) present.

The resin can adsorb Cu^{2+} ions from solution and return in exchange the same number of H^+ ions, so that the solution maintains electrical neutrality.

The process can be represented by:



$$R\begin{bmatrix}COOH\\ \\ COOH\end{bmatrix} + CuSO_4.5H_2O \longrightarrow R\begin{bmatrix}COO\,[Cu]\\ \\ COO\,[Cu]\end{bmatrix} + H_2SO_4$$

………… **[Reaction 6.1]**

Where

R : Polystyrene

As the solution passes through the column, it becomes more dilute in Cu^{2+} and more concentrated in H^+, a change that can be followed by sampling the H^+ concentration in the exit stream with a pH electrode.

Eventually the resin becomes saturated with Cu^{2+} ions and further exchange ceases. The ion exchange process is reversible, so that the resin can be regenerated by allowing it to come in contact with a strong acid solution (H_2SO_4).

The exchange of Cu^{2+} ion for a H^+ ion is not instantaneous, since it takes time for Cu^{2+} ion to diffuse from the bulk of the liquid to the surface of the resin and then to the same position inside the resin where it can exchange for H^+ ion. And conversely for the H^+ ion, which has to migrate back to the bulk solution. The rate of exchange is expected to become slower as the Cu^{2+} ion concentration in the resin approaches saturation.

The bed can be operated either in:

- **a)** down flow, in which case the bed is fixed, and solution is passed through the top of the column at a constant flow rate **(Refer to Figure 6.1)**, or
- **b)** up flow, at a liquid velocity such that the particles are fluidized **(Refer to Figure 6.2)**.

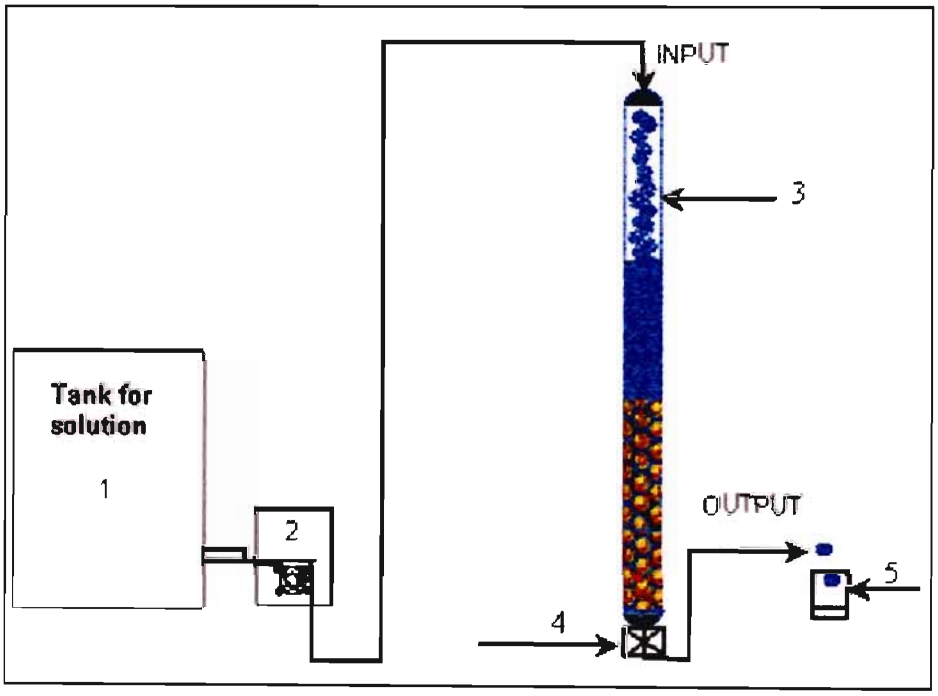


Figure 6.1: Schematic Design of Fixed Bed Ion Exchange Column

1 - Tank for input solution
2 - Peristaltic Pump
3 - Fixed bed column (2.4 cm id and 1 m long)
4 - Valve
5 - Collection cylinder

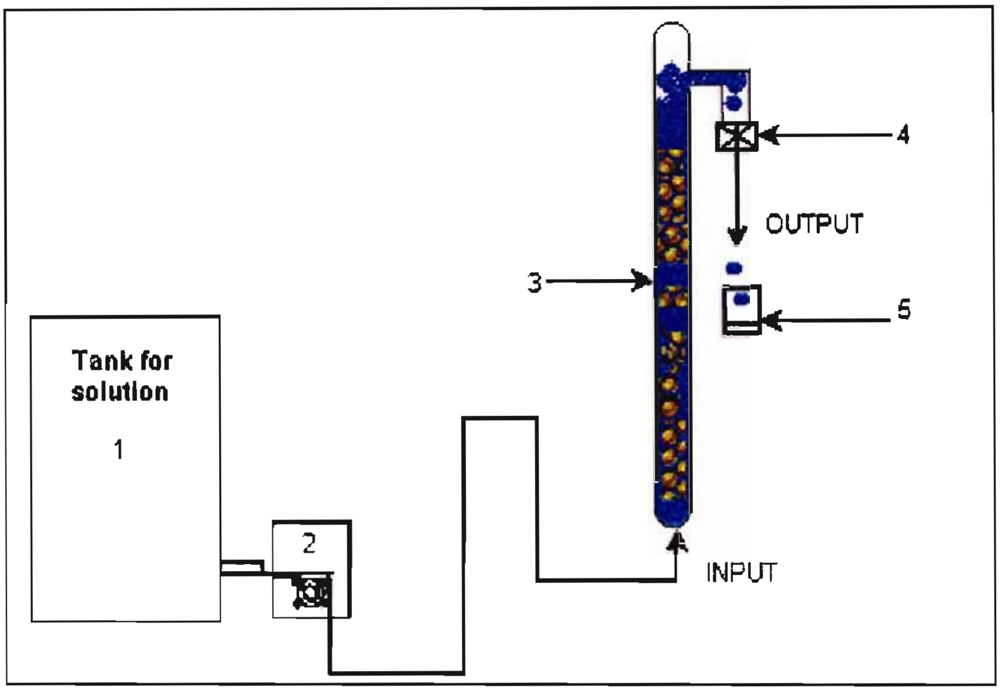


Figure 6.2: Schematic Design of Fluidized Bed Ion Exchange Column

1 - Tank for input solution

2 - Peristaltic Pump

3 - Fluidized bed column (2.4 cm id and 2 m long)

4 - Valve

5 - Collection cylinder

Note:

Refer to Appendix B1 for the cost estimation of the above experimental designs.

CHAPTER SEVEN: DETERMINATION OF COPPER PRESENT IN THE EFFLUENT AND ELUATES

An atomic absorption spectrometer was used to determine the amount of copper present in the effluent streams and the amount of copper from elution **(Refer to Figure 7.1)**.

Figure 7.1: Varian Spectra AA 50 B

7.1 Spectrophotometry

The principal of spectrophotometry uses the intensity of colour to measure the amount of material in solution. The colour seen in a sample of solution is due to the selective adsorption of certain wavelengths of visible light and transmittance of the remaining wavelengths.

7.1.1 Spectrophotometric Analysis

a) Choice of wavelength

For a particular concentration range a set wavelength was used and this wavelength is determined in the Varian Spectra AA 50 B manual.

b) Plotting Calibration Graphs

A set of standard copper solutions are prepared for the determination of a calibration graph. Again the manual of the Varian Spectra AA 50 B will state the standard range to prepare depending in which concentration range that needed to be determined.

The calibration curve is a plot of concentration versus absorbance and is determined manually by the Varian Spectra AA 50 B using the standard copper solutions. The machine was calibrated before analysis of unknown samples at all times.

Once the Varian Spectra AA 50 B was calibrated for a specific metal of interest, the machine could determine the unknown concentration of copper in the effluent and elution streams.

CHAPTER EIGHT: REGENERATION

8.1 Regeneration Procedure

After the feed solution was processed to the extent that the resin becomes totally exhausted and cannot exchange any more ions. The resin must be eluted so that it can be reused. In normal column operation, for a cation system being converted first to the hydrogen form, regeneration employs the following steps.

8.1.1 Step One

The column was backwashed to remove any suspended solids collected by the resin bed during the service cycle and also to eliminate any channelling that may have formed during the cycle. (Channelling occurs only if the columns are not properly aligned. The columns were vertical to avoid channelling of the feed solution.)

The back-wash flow fluidizes the bed, releases any trapped particles and reorients the resin according to size. During backwash usually the larger, denser particles accumulates at the base and the particle size will decrease moving toward the top of the column.

8.1.2 Step Two

The resin bed was brought in contact with sulphuric acid (H_2SO_4). In the case of the cation resin, acid elutes the collected ions and converts the resin bed to the hydrogen form.

8.1.3 Step Three

A slow water rinse was applied to the eluted resin, this removes any acid residual.

The column was then ready for service.

8.2 Co-Current and Counter-Current Regeneration

Columns are designed to use either co-current or counter-current regeneration systems. In co-current units, both feed and regenerant solutions make contact with the resin in a down flow movement. These units are the less expensive of the two in terms of initial equipment cost. Co-current flow uses regenerant chemicals less efficiently than counter-current flow: it has higher leakage concentrations (the concentration of the feed solution ion being removed in the column effluent), and cannot achieve as high a product concentration in the regenerant.

TABLE 8.1: Guidelines for Typical Regeneration Level and Corresponding Resin Operating Capacity [Internet] [62]

Regeneration System	Regenerant Level [g/l]	Typical Operating Capacity [eq/L]
Co-Current Regeneration:		
HCl	80 - 120	0.8 - 1.2
H_2SO_4	150 - 200	0.5 - 0.8
NaOH	80 - 120	0.4 - 0.6
Counter-Current Regeneration:		
HCl	40 - 55	0.8 - 1.2
H_2SO_4	60 - 80	0.5 - 0.8
NaOH	30 - 45	0.4 - 0.6

CHAPTER NINE: APPLICATIONS OF ION EXCHANGE PROCESSES

The observations of the phenomenon on ion exchange date from ancient times. The mechanism of the reaction was established in 1850 by two English chemists, H.S. Thompson and J.T. Way. But it was only in the last few decades that the subject has expanded to become a true science from which extensive industrial applications have emerged **[Helfferich]** [6].

Applications of ion exchange processes have many current forms (of which some are discussed below), and some of these could expand as increased awareness of ion exchange continues.

9.1 Waste Water Treatment

In industry, there are many situations where solutions containing low concentrations of metals are produced. Such metals are removed from solution by chemical precipitation before the waste water is discharged to drain. For example, in the metal finishing industry, rinse effluents drag out tank solutions and floor spillage all contribute to aqueous streams requiring treatment. Water treatment procedures needs to be reviewed and ion exchange technology will be applied, as more industrial organizations aim at zero waste water discharge.

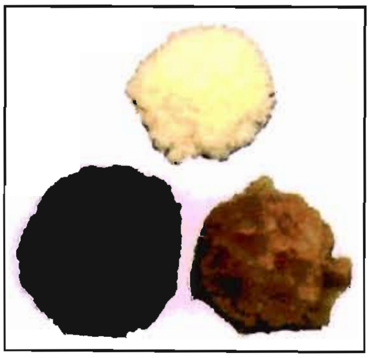


Figure 9.1: Some Water Treatment Resins [Internet] [67]

9.2 Water Softening

Water that contains calcium and magnesium ions are called "hard water" because those ions can combine with other ions or compounds, to leave a hard scale on the surfaces they get in contact with. An ion exchange water softener can reduce or eliminate such problems.

A typical water softener has a tank partially filled with an ion exchange resin **(Reference is made to Figure 9.2).** As water passes through, the resin's stronger attraction for the hardness ions causes the resin to take on the hardness ions and give up its sodium ions. Iron and manganese are considered hardness and they are removed also, provided they are in solution. Ion exchange cannot remove suspended matter.

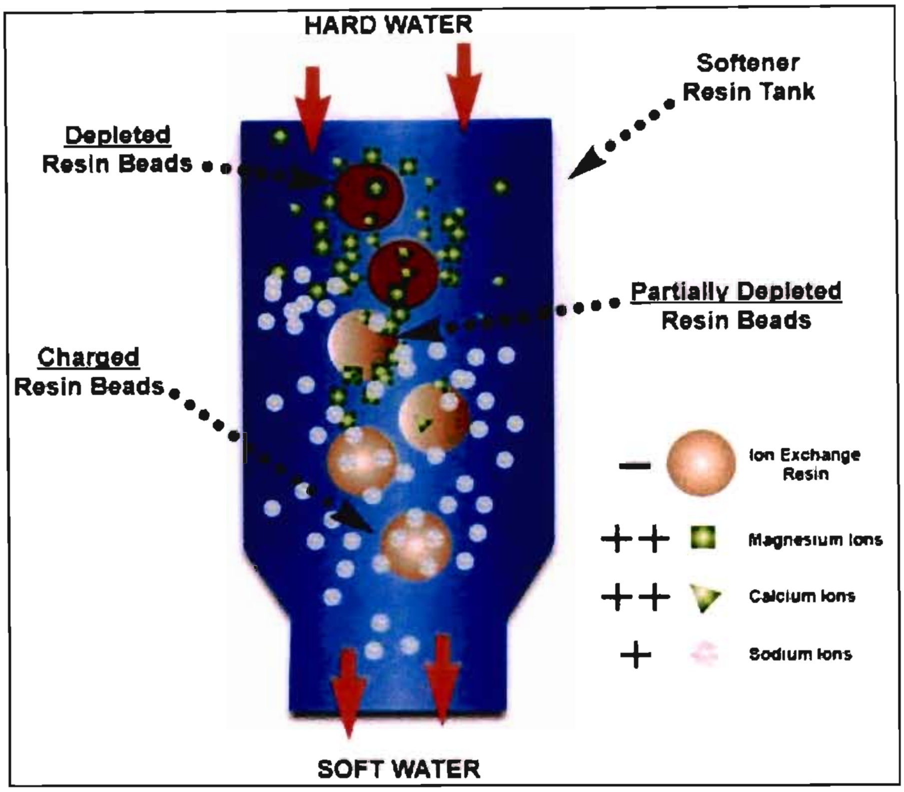


Figure 9.2: Exchange of Hard Water Ions for Soft Water Ions [Internet] [64]

9.3 Applications in the Gold Industry

Gold is now almost universally recovered from their ores by being dissolved in solutions of alkaline cyanide and then being precipitated from cyanide solution onto zinc or being adsorbed onto the ion exchange resin beads from which, the gold is taken back into solution and finally electro-deposited.

The gold is present in the cyanide solution as the complex $Au\ (CN)_2$ anion at concentrations typically a few parts per million. The cyanide concentration is usually about 100 to 300 parts per million.

9.4 Other Applications of Ion Exchange Processes

- Preparation of various acids, bases, salts, and solutions.
- Analytical chemistry uses ion exchange in chromatography.
- The recovery of valuable metals is also possible with resins.

- Industrial drying of treatment of gases.
- The food industry uses ion exchange in a variety of ways, ranging from winemaking to sugar manufacture.
- Ion exchange is also useful in death, as it plays a role in the treatment of formaldehyde.

Figure 9.3: Down Flow Ion Exchange Columns [Internet] [65]

Figure 9.4: Up Flow Ion Exchange Columns [Internet] [65]

CHAPTER TEN: EXPERIMENTAL PROCEDURES

10.1 Tapped Wet Volume Measurement

The tapped wet volume measurements of the resin ensure volume reproducibility. When ever a volume of resin was required for test work, this procedure was employed.

10.1.1 Procedure:

A measuring cylinder is used with a solid glass. To obtain very accurate measurements a measuring cylinder with a capacity closest to the volume of resin must be used. The resin was placed into a measuring cylinder with de-ionized water to approximately the desired value. The cylinder was filled to the rim with de-ionised water. No space for air was left.

The palm of the hand was placed over the mouth of the measuring cylinder and the cylinder was tapped gently on a hard surface. The resin volume decreases during tapping. The tapping must continue until the volume of the resin is constant. More resin is added, this process must continue until resin volume is half a resin bead size over the required volume.

10.1.2 Packing a Column with Resin

A well packed column is required for efficient operation. The columns were packed at all times using the procedure below.

10.1.2.1 Procedure:

The volume of resin required for test work must be measured out (using the above method).The column must be clamped securely. The tap of the column must be opened. Using a wash-bottle, demonized water is sprayed onto the resin. The resin was swirled and poured into the column with the tap open to drain any excess water from the bottom of the column. The level of the water was not allowed to go below the resin bed height as this introduces air bubbles into the resin bed. The tap must be closed when the water level approaches the level of the packed resin.

If the water level dropped below the resin level, the air bubble was removed by moving a thin wire up and down the resin bed. If there were too many air bubbles in the column the resin was emptied out and reloaded into the column

10.1.3 Pre conditioning of Resins

10.1.3.1 Purpose

Many of the properties of an ion exchange resin are affected to some degree by the ionic form of the functional groups, it is important to covert the resin sample into some known ionic form or 'standard state' before use.

10.1.3.2 Resin

The TP 207 resin that was obtained from Mintek was in the Cu^{2+} form. The resin had to be stripped and washed first before any test work could be carried out. The stripping of the resin was done in a column with of 2M H_2SO_4 solution.

2M H_2SO_4 was passed down flow through the packed bed of resin at a flow rate of 21.13 mL/min. The resin was stripped and converted to the hydrogen form and then washed down flow with water at a flow rate of 21.13 mL/min to remove any excess acid. The pH of the resin was 2.18 at a temperature of 20 ^{0}C. The resin in the hydrogen form was used for all the experimental work.

10.2 Determination of Average Particle Size of TP 207 resin

10.2.1 Apparatus and Equipment:

Fritsch vibrating screen (wet screen shaker) with sieve sizes 850, 710, 600, 500, 355, 212 µm.

10.2.2 Reagents:

TP 207 Iminodiacetic acid

10.2.3 Procedure:

A volume of 10 mL of TP 207 resin in the hydrogen form at pH 2.18 was measured out using the tapped wet volume method.

The sieves of 850, 710, 600, 500, 355, 212 µm were arranged in descending order over the pans, and the 10 mL of resin was passed through the 850 µm screen. Water was poured gently over the resin from a rubber tube connected to a tap so that smaller particles can pass through the sieve. The vibrations of the resin in the Fritsch vibrating screen were programmed for ten minutes. This caused the machine to agitate up and down so as to sieve smaller particles through. At each screen size the resin was collected and tapped volumes of the resin for each screen was noted. This procedure was repeated eight times using different resin samples from the top, bottom and various parts of the resin drum. The average particle size was calculated using the cumulative oversize and cumulative undersize plots. The average particle size was required for determining the minimum fluidization and settling velocity for the TP 207 resin.

Figure 10.1: Photograph of a handful of TP 207 resin in the Hydrogen form

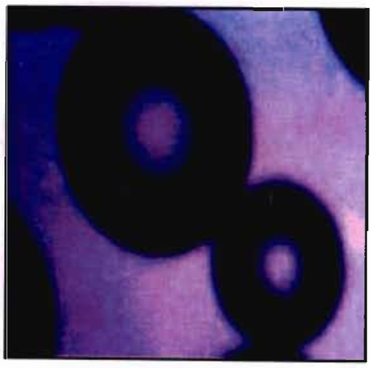


Figure 10.2: Photograph of a Single Particle Magnified fifteen times

10.3 Void Volume Measurement

10.3.1 Apparatus and Equipment:

Perspex column with dimensions:

Height = 1 m

Inner Diameter = 2.4 cm

Measuring cylinder (100 mL)

10.3.2 **Reagents:**

TP 207 Iminodiacetic acid
Distilled water

10.3.3 **Procedure:**

Void volume means the volume of the spaces between the resin particles when the resin is packed in a column. A simple method was used to determine the void volume of the resin. 100 mL of TP 207 was tapped in a 100 mL measuring cylinder. The resin was poured into a perspex column and the resin height was measured. Water was run to the top surface of the resin bed. All the water from the resin layer was drained off and the volume of water was collected in a measuring cylinder at the bottom of the column. The void volume of the resin was determined using the following formulae. The test was repeated to ensure reliability.

$$\text{Void volume } \% = \left(\frac{V'}{V}\right) * 100 \quad \text{..} \textbf{(Equation 10.1)}$$

Where

V' : Volume of water drained
V : Bed volume of swollen resin ($V = \pi r^2 * Hp$)

Note: The above value was used to calculate the minimum and settling velocities for fluidization **(Reference is made to Appendix C2)**.

10.4 **Water Retention Capacity**

10.4.1 **Apparatus and Equipment:**

Sintered glass funnel
Cloth
Rubber band
Spatula

10.4.2 **Reagents:**

TP 207 Iminodiacetic acid
Distilled water

10.4.3 **Procedure:**

The mass of a small dish was determined. Using the tapped wet volume method of resin, an accurate volume of the resin was measured out. The resin was washed into a sintered glass funnel. The funnel was covered with damp, wrung out cloth and held in place with a rubber band. Mild suction was applied for three minutes. The funnel was removed and wiped at the sides with the cloth to remove any water droplets.

The funnel was turned upside down into a pre-weighed dish and the resin was lightly tapped out. A spatula was used to remove the remainder of the resin from the funnel. The mass of the dish and the wet resin was determined. The dish containing the resin was placed overnight into an oven at 65 ^{0}C.

The sample was allowed to cool. The mass of the dish and dried resin was determined. The water retention capacity of the resin was determined using Equation 10.2. A duplicate test for water retention capacity was determined to ensure reliability.

$$WRC = \left[\frac{M_W - M_D}{M_w}\right] * 100 \ldots\ldots\ldots\ldots \textbf{(Equations 10.2)}$$

Where

WRC : The water retention capacity

M_W : Mass of wet resin

M_W = (Mass of dish + wet rein) – Mass of dish

M_D : Mass of dried resin

M_D = (Mass of dish + dried rein) – Mass of dish

10.5 **pH Test**

The dependence of copper loadings on the TP 207 resin, as a function of pH was derived.

10.5.1 **Apparatus and Equipment:**

Radiometer Copenhagen TTT 80 Titrator

Radiometer Copenhagen PHM 82 standard pH meter

Spatula

Stirrer

Plastic containers

Overhead Stirrer

10.5.2 **Reagents:**

TP 207 Iminodiacetic acid

Cupric Sulphate ($CuSO_4.5H_2O$)

Distilled water

Sodium Hydroxide (NaOH)

Sulphuric Acid (H_2SO_4)

10.5.3 **Procedure:**

A 6 g/L of Copper [Cu^{2+}] stock solution was prepared using 117.8617 g of $CuSO_4.5H_2O$ in 5L of distilled water. Six portions of 10 mL resin in the hydrogen [H^+] form were measured out and placed in plastic containers.

The Radiometer Copenhagen TTT 80 Titrator was set at pH set points for 1, 1.5, 2, 2.5, 3, 4 and a Radiometer Copenhagen PHM 82 standard pH meter was placed within the plastic container to monitor the pH at all times.

500 mL volumes of the copper solution were placed into plastic containers with the resin. Each plastic container with the resin/copper solution had an overhead stirrer which was set at 180 rpm. The stop watch was then started.

For each plastic container 2M NaOH or 2M H_2SO_4 solution was added automatically using the Radiometer Copenhagen TTT 80 Titrator to keep the pH set at the respective pH values. After 24 hours the overhead stirrers were stopped and values for 2M NaOH and 2M H_2SO_4 were noted depending on the tests.

The resin was separated from the copper solution. The resin was tapped to determine the swelling of the resin saturated with copper. Copper solution remaining was measured with a measuring cylinder to compare values with the volume and then analyzed with an Atomic Absorption Spectroscopy (AA spectrometer).

The resin was eluted with 10 BV of 2M H_2SO_4 in 100 mL volumetric cylinders and the eluates were analyzed with an AA spectrometer. This was done to determine the accountability for each test.

From the data obtained the copper loading against pH was plotted.

10.6 **Equilibrium Tests**

10.6.1 **Apparatus and Equipment:**

Radiometer Copenhagen TTT 80 Titrator
Radiometer Copenhagen PHM 82 standard pH meter
Spatula
Stirrer
Plastic containers
Overhead Stirrer

10.6.2 **Reagents:**

TP 207 Iminodiacetic acid
$CuSO_4.5H_2O$
Distilled water
NaOH
H_2SO_4

10.6.3 **Procedure:**

Four equilibrium tests were done at pH values set at 1.5, 2, 2.5 and 3. The pH of the resin was measured before and after equilibrium tests.

For each tests a 6 g/L of Cu^{2+} stock solution was prepared using 117.8617 g of $CuSO_4.5H_2O$ in 5L of distilled water. Six portions of 10 mL of TP 207 resin in the H^+ form were measured out for each equilibrium tests and placed in plastic containers.

The Radiometer Copenhagen TTT 80 Titrator was set at pH set points for the different tests and a Radiometer Copenhagen PHM 82 standard pH meter was placed within the plastic container to monitor the pH at all times.

Volumes ranging from 50 mL to 1000 mL of Copper Sulphate Solution were prepared and placed into plastic containers with the resin. Each plastic container with the resin/copper solution had an overhead stirrer which was set at 180 rpm. The stop watch was then started.

For each plastic container 2M NaOH or 2M H_2SO_4 solution was added automatically using the Radiometer Copenhagen TTT 80 Titrator to keep the pH set at the respective pH values. After 24 hours the overhead stirrers were stopped and values for 2M NaOH and 2M H_2SO_4 were noted depending on the tests. The resin was separated from the copper solution.

The resin was tapped to determine the swelling of the resin saturated with copper. Copper solution remaining was measured with a measuring cylinder to compare values with the volume balance. Samples of the solution were analyzed with an atomic absorption spectroscopy (AA spectrometer). The resin was eluted with 10 BV of 2M H_2SO_4 in 100 mL volumetric cylinders and the eluates were analyzed with an AA spectrometer. These were determined for the accountability purposes.

From the data obtained the equilibrium curve was plotted in terms of concentration of copper ion adsorbed on the resin (g of copper ion adsorbed per L of resin) versus the concentration of copper ion in solution (g/L) for the respective pH testing.
Langmuir and Freundlich parameters were also determined for each test.

10.7 **Kinetic Tests**

10.7.1 **Apparatus and Equipment:**

Radiometer Copenhagen TTT 80 Titrator
Radiometer Copenhagen PHM 82 standard pH meter
Spatula
Stirrer
Plastic containers
Overhead Stirrer

10.7.2 **Reagents:**

TP 207 Iminodiacetic acid
$CuSO_4.5H_2O$
Distilled water
NaOH

10.7.3 **Procedure:**

Two solutions were prepared 6 and 0.6 g/L for testing. From equilibrium data the amount of resin that was used for kinetic tests were calculated using 0.0322 g/L as the finial concentration for loading. The following amounts 609.89 and 58.03 mL was obtained respectively as the amount of resin to use for the concentrations mentioned above. Two particle size distributions were used for testing, 850 μm and 600 μm.
The two solutions were placed in plastic containers and were attached to a mechanical stirrer. The water was removed from the resin using a pipette and placed with the respective solutions.

The Radiometer Copenhagen TTT 80 Titrator was set at a pH of 3 and a Radiometer Copenhagen PHM 82 standard pH meter was placed within the plastic container to monitor the pH at all times. The speed of the mechanical stirrer was set at 310 rpm and the stopwatch was started.

A sample of 5 mL of solution was taken at set-intervals for 24 hours. The resin was tapped to determine the swelling of the resin saturated with copper. Copper solution remaining was measured with a measuring cylinder to compare values with the volume balance. Samples of the solution were analyzed with an AA spectrometer.

10.8 **Effect of Swelling of TP 207 resins**

For the mini column tests resins in three different forms were required. The hydrogen, di sodium and the mono sodium forms.

10.8.1 **Conversion into the Hydrogen form**

10.8.1.1 **Procedure:**

Three litres of resin in the H^+ form was required. The conversion of the resin into the hydrogen form was followed from Section 10.1. Two litres of this resin was used for the conversion to the other forms and one litre was to be kept in the hydrogen form.

The following were the results obtained from the hydrogen form:
Volume of resin required [H^+ form] = 1000 mL.
pH of resin at the end of conversion [H^+ form] = 2.5

10.8.2 **Conversion into the Di Sodium form**

10.8.2.1 **Procedure:**

One litre of resin in the H^+ form was required. The resin was loaded into a column. A solution of 2M NaOH was prepared using 239.94g of NaOH in 3L distilled water. A Watson Marlow peristaltic pump **(refer to Figure 10.3)** was used to set the flow rate at 1 L/hr. 2 M NaOH solution was passed *up flow* through the inlet of the column at the required flow rate. The resin was water washed *down flow* with four litres of distilled water for two hours. The resin was tapped (using the tapped wet volume method) and the pH was measured.

The final resin was in the Di Na^+ form with the following result:
Volume of resin [Di Na^+ form] = 1349 mL
pH of resin [Di Na^+ form] =11.50

10.8.3 Conversion into the Mono Sodium form

10.8.3.1 Procedure:

One litre of resin in the H^+ form was required. The resin was loaded into a column and water washed for two hours with four litres of water until the pH of the resin was greater than 2.5. The excess water was sucked out with a pipette. A solution of 0.475 M NaOH was prepared using 37.99g of NaOH in 2L distilled water. The 0.475 M NaOH solution and the one litre of resin in the H^+ form were placed in a bucket with an over head stirrer. The speed of the mechanical stirrer was set at 310 rpm. The resin was tapped and the pH was measured.

The resin was in the Mono Na^+ form with the following result:
Volume of resin [Mono Na^+ form] = 1250 mL
pH of resin [Mono Na^+ form] =6.71

Note:

- **Di Sodium form**

The resin that was converted to the Di sodium form must be converted using *upward flow* movement for the following reason:
The resin is in the H^+ form and is exchanging $2H^+$ for $2Na^+$. As the resin is converted to the Di sodium form the resin swells. This implies that the resin increases in size. If the process was operated down flow the resin which would expand might crack the bottom of the column. Therefore the flow needs to be upwards to allow the resin to expand.

- **Mono Sodium form**

The resin that was converted to the mono sodium form needs to be converted using the batch method because if it were done using up flow, only the bottom half of the column would be converted to the Na^+ form and the top part of the column would still be in the H^+ form. The converse is also true (down flow).
Using the batch system allows the Na^+ to be evenly distributed into the resin. The batch system allows for well mixing of the resin and the solution.

10.9 Mini Column Tests

10.9.1 Apparatus and Equipment:

6 glass columns:
Inner Diameter = 2.08 cm
Measuring cylinder (100 mL)
Watson Marlow peristaltic pump
pH meter
Spatula
Stirrer
Pill vials

10.9.2 Reagents:

TP 207 Iminodiacetic acid
Distilled water
H_2SO_4
$CuSO_4.5H_2O$

10.9.3 Procedure:

The TP 207 resin was converted into three forms namely hydrogen; mono sodium and di sodium form **(Refer to procedures in Section 10.8 for the conversion)**. The hydrogen form was used as a reference for calculations. All three resins had the same functional group content depending on the form it was converted to **(Reference is made to Appendix C5)**. 50 mL of TP 207 resin in the H^+ form was tapped and the pH was measured. This was repeated for the resin in the di sodium and mono sodium form at 67 mL and 62 mL respectively. The resin was loaded into a 2.08 cm inner diameter column and 6 g/L of Cu^{2+} from a copper solution was made up.

For each test the feed pH was varied. Four different feed pH values were tested to find the effect of feed pH on the residence time. Therefore 98 % of concentrated H_2SO_4 solution was added for each test to drop the pH to the required levels of 3, 2.5, 2 and 1.5.

A Watson Marlow peristaltic pump was used to set the required flow rates. Copper solution was passed down flow through the inlet of the column at the required flow rate. 25 mL of the effluent was collected at the bottom of the column with a measuring cylinder. The pH and the flow rate of the effluent were monitored throughout the test. The effluent was sampled and analyzed for Cu^{2+} to determine breakthrough through the resin bed. The sample was analyzed using the Atomic Absorption Spectrometry.

The resin loaded with copper was tapped to determine the volume and eluted with 2M H_2SO_4 to determine the amount of copper that was adsorbed onto the resin. This was done to determine the accountability for each test. The resin converted to hydrogen form was tapped and the pH was measured.

10.10 **Minimum Fluidization Velocity Measurement**

10.10.1 **Apparatus and Equipment:**

Perspex Column:
  Height = 1 m
  Inner Diameter = 2.4 cm
Measuring cylinder (100 mL)
Watson Marlow peristaltic pump

10.10.2 **Reagents:**

TP 207 Iminodiacetic acid
Distilled water

10.10.3 **Procedure:**

100 mL of tapped TP 207 resin was loaded into a perspex column and the static resin bed height of the column was measured using a ruler. It was determined at 203 mm. Water was used as the fluidizing liquid and the Watson Marlow peristaltic pump was started.

A volume of liquid was collected for one minute to determine the flow rate. The speed was slowly increased and the volume was collected for that time interval. The bed height was measured with a ruler.

At new speed intervals the procedure was repeated to determine the minimum fluidization velocity.

Figure 10.3: Photograph of the Watson Marlow Peristaltic Pump

10.11 **Column Tests**

The column tests were conducted at three different superficial linear velocities to determine the effect of an increase in linear velocity on the height of the mass transfer zone. The heights of the resin bed was varied to accommodate the mass transfer zone at each linear velocity. From the mini column tests one pH solution and one form of resin was chosen for testing.

10.11.1 **Apparatus and Equipment:**

3 perspex columns:
  Height = 1 m
  Inner Diameter = 2.4 cm
Measuring cylinder (500, 1000, 2000 mL)
Watson Marlow peristaltic pump
pH meter
Spatula
Stirrer
Pill vials

10.11.2 **Reagents:**

TP 207 Iminodiacetic acid
Distilled water
H_2SO_4
$CuSO_4.5H_2O$

10.11.3 Procedure:

Three volumes of resin were used for the test namely 406, 814, and 1626 mL. The TP 207 resin in the H^+ form was tapped to the above mentioned volumes and the pH measured.

For Column test 1
The 406 mL resin was loaded into a 2.4 inner diameter and 1m height column. 3 g/L of Cu^{2+} from a copper sulphate solution was made up. Concentrated H_2SO_4 solution was added to the feed solution to drop the pH to 3. A Watson Marlow peristaltic pump was used to set the required flow rates.

Copper solution was passed down flow through the inlet of the column at the required flow rate. 200 mL of the effluent was collected at the bottom of the column with a measuring cylinder. The pH and the flow rate of the effluent were monitored throughout the test. The effluent was sampled and analyzed for Cu^{2+} to determine breakthrough through the resin bed. The samples were analyzed using the Atomic Absorption Spectrometry. The resin loaded with copper was tapped to determine the volume of the resin and was eluted with 2M H_2SO_4 to determine the amount of copper that was adsorbed onto the resin. This was done to determine the accountability for each test.

The resin converted to H^+ form was tapped and the pH was measured. The similar procedure was used for the 814 and 1626 mL of resin except for the 814 mL of resin two perspex columns were required and for the 1626 mL of resin three perspex columns were required. The columns were run in series for the last two tests.

10.12 **Pilot Plant Testing**

The pilot plant testing involved operations of a fixed bed ion exchange system and a fluidized ion exchange system **(Refer to Figure 10.4)**.

For the fixed bed column the feed solution was passed through the top of the resin bed and for the fluidized bed the feed solution was passed through the bottom of the resin bed.

Two concentrations namely 6 g/L and 0.6 g/L of Cu^{2+} solution were used for testing and comparing the contacting equipment.

10.12.1 **Apparatus and Equipment:**

Fixed Bed Dimensions:
One Perspex Column
    Height = 1 m
    Inner Diameter = 2.4 cm

Fluidized Bed Dimensions:
One Perspex Column
    Height = 2 m
    Inner Diameter = 2.4 cm
Measuring cylinder (1000 mL)
Watson Marlow peristaltic pump
pH meter
Spatula
Stirrer
Pill vials

10.12.2 **Reagents:**

TP 207 Iminodiacetic acid
Distilled water
H_2SO_4
$CuSO_4.5H_2O$

10.12.3 **Procedure:**

One volume of resin was used for all test work and both contacting equipment were run at the same linear velocity so that comparisons could be made.
406 mL of TP 207 resin in the hydrogen form was tapped and the pH measured.

For the Fixed bed column:
406 mL resin was loaded into a 2.4 inner diameter and 1m height column **(Refer to Figure 10.5)**. 6 g/L of Cu^{2+} from a cupric sulphate solution was made up. Concentrated H_2SO_4 solution was added to the feed solution to drop the pH to 3. A Watson Marlow peristaltic pump was used to set the required flow rates. Copper solution was passed down flow through the inlet of the column at a linear velocity of 0.13 cm/s.

200 mL of the effluent was collected at the bottom of the column with a measuring cylinder **(Refer to Figure 10.6)**. The pH and the flow rate of the effluent were monitored throughout the test. The effluent was sampled and analyzed for Cu^{2+} to determine breakthrough through the resin bed. The samples were analyzed using the Atomic Absorption Spectrometry.

The resin loaded with copper was tapped to determine the volume of the resin and was eluted with 2M H_2SO_4 to determine the amount of copper that was adsorbed onto the resin. This was done to determine the accountability for each test **(Refer to Figure 10.10)**. The resin converted to hydrogen form was tapped and the pH was measured. The similar procedure was used for the fluidized bed except the resin was loaded into a 2m long column and the direction of flow of the input solution was up flow.

The above was repeated using both contacting equipment at 0.6 g/L solution to find the effects of smaller concentrations. Again the same amount of resins was used and the same linear velocity.

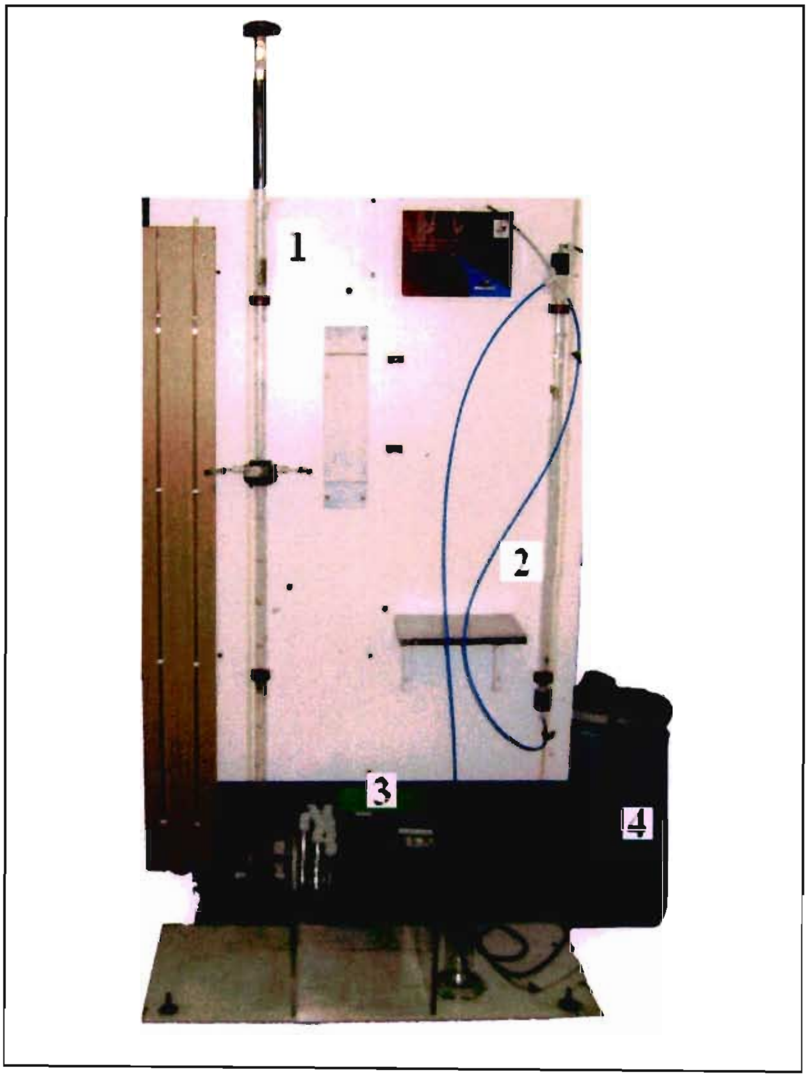


Figure 10.4: Photograph of the Pilot Plant

1 - Fluidized bed column (2.4 cm id and 2 m long)
2 - Fixed bed column (2.4 cm id and 1 m long)
3 - Peristaltic Pump
4 - Tank for input solution

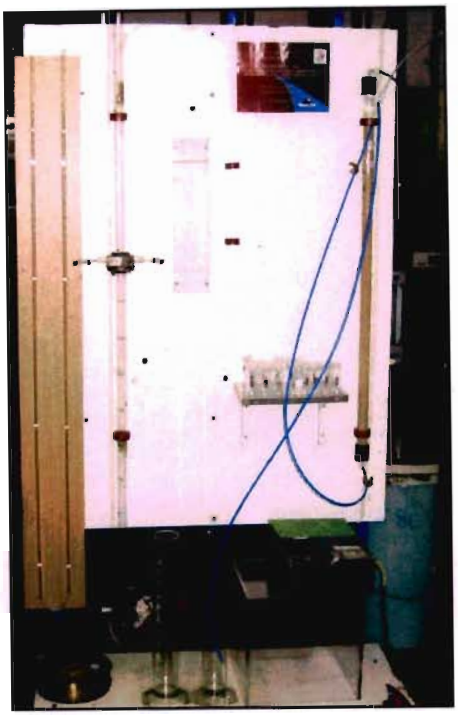


Figure 10.5: Photograph of the Fixed Bed Column at the Start of Operations

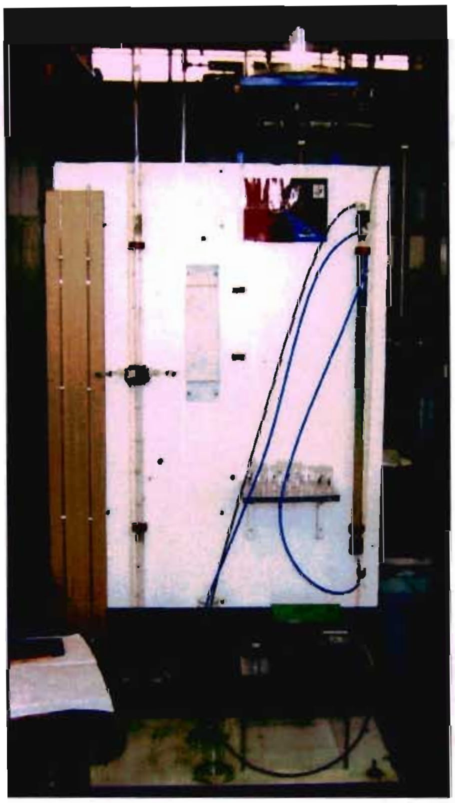


Figure 10.6: Photograph of the Fixed Bed during Operation with Copper loading onto the resin

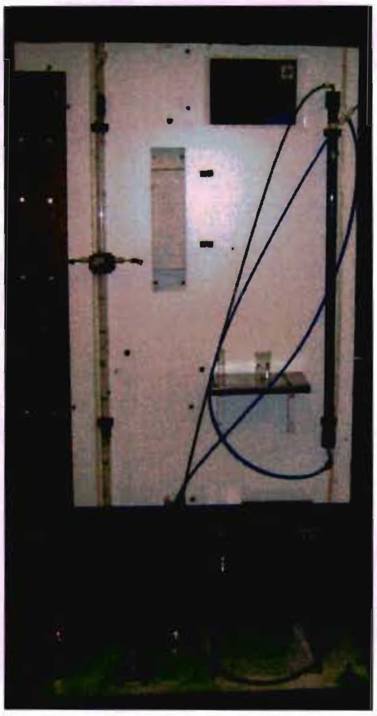


Figure 10.7: Photograph of the Fixed Bed at the End of Operation with resin fully loaded with copper

Figure 10.8: Photograph of the Fluidized Bed during Operation with Copper loading onto the resin

Figure 10.9: Photograph of the Fluidized Bed at the End of Operation with resin fully loaded with Copper

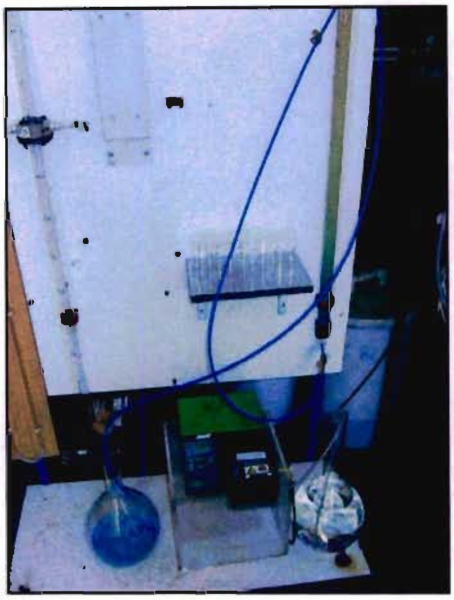


Figure 10.10: Photograph of the Regeneration of the resin with 2M Sulphuric Acid

CHAPTER ELEVEN: RESULTS AND DISCUSSION

Preliminary experimental test work was done in order to determine the start up conditions for the column testing. The test work included:

- the determination of the effect of pH on the copper loading,
- the establishment of equilibrium profiles set at four pH set points,
- kinetic tests using two particle sizes of the TP 207 resin,
- mini column tests which involved testing based on two forms of the TP 207 resin,
- experimental determination of u_f and using three linear velocities to choose one linear, velocity to operate the fixed and fluidized ion exchange columns.

11.1 **Influent Characteristics**

11.1 .1 **Chemical Analysis**

The influent solution was a pure cupric sulphate solution. It was decided that for the purpose of this test work, feed concentrations of 6 and 0.6 g/L copper would be used because these concentration range are mostly encountered for copper, in industry.

11.1.2 **Void Volume Determination**

The void volume (spaces between the resin particles when the resin is packed in a column) was determined. The procedure of the measurement was discussed in Section 10.3 and the calculations are present in Appendix C2. A duplicate test was performed and the average value was used to ensure reliability. The void volume was experimentally determined to be 29.23 %. This value was used in Appendix C3 to calculate the minimum and settling velocity for fluidization.

11.2 **Adsorbent**

11.2 .1 **Resin Characterization**

The batch of resin that was used for the purpose of this study had been used in previous investigations by Mintek. Although the theoretical capacity of the fresh resin is 1.2 mol/L (for divalent ions), the capacity of the used resin was experimentally determined to be 1 mol/L from equilibrium tests.

11.2.2 **Particle Size Analysis**

The procedure of the particle size analysis was discussed in Section 10.2 and the graphs for cumulative undersize and oversize distribution of the pre-screened TP 207 resin is present in Appendix C1 for each run. The summary of the results are tabulated below.

TABLE 11.1: Summary of Particle Size Analysis

Run Number	Average per Run [μm]
1	720
2	716
3	740
4	725
5	730
6	735
7	730
8	730
Average particle size	**728.25**
Maximum	**740**
Minimum	**716**
Standard Deviation	**7.759786448**

Eight particle size distributions were determined for the TP 207 resin in the hydrogen form **(Refer to Table 11.1)**. The finial average particle size was determined by adding all the average particle sizes for each run and dividing by the number of runs. This was determined to be 0.72825 mm. The value obtained was used in Appendix C3 to calculate the minimum and settling velocity for fluidization.

11.2.3 **Water Retention Capacity**

The water retention capacity is the amount of water that is retained inside the resin pores. The procedure of the measurement was discussed in Section 10.4 and the calculations are presented in Appendix C4. A duplicate test for water retention capacity was determined and the average value was taken to ensure reliability. The water retention capacity was experimentally determined to be 44.8 %.

11.3 **Preliminary Test Work**

Preliminary test work was done to determine the effect of pH on copper loading onto the resin, as well as establish equilibrium isotherms at four different pH set points. Results from this phase of test work were used to plan the tests for the pilot plant more effectively.

11.3.1 **pH Dependence of Metal Loadings**

The experimental procedures for determining the effect of pH on the resin performance is discussed in Section 10.5. Detailed experimental data and mass balance are present in Appendix D The effect of pH on copper loading onto TP 207 is given in Figure 11.1

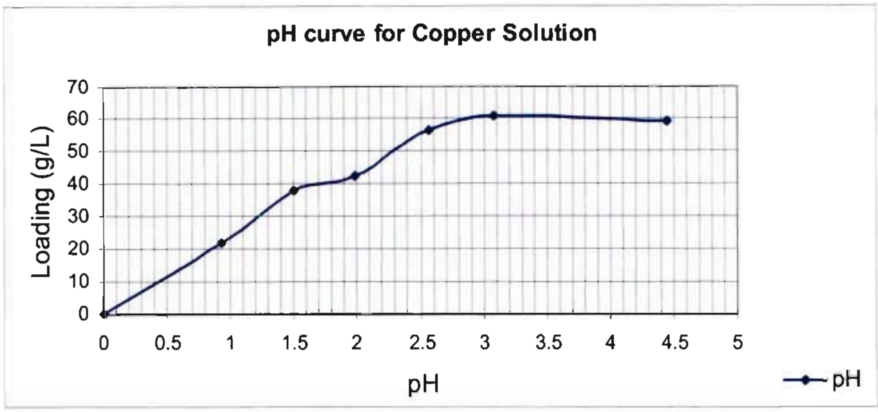


Figure 11.1: Effect of pH on Copper Loading

The pH dependence curve for copper loading was measured by contacting TP 207 resin (in the hydrogen form), with the same amount of copper solution, at different pH values. It is evident from the optimum pH curve that the maximum copper loading may be achieved at a pH of above 3. Thus the pH of 3.0 would probably be the maximum pH that can be considered for copper extraction.

11.3.2 **Ion Exchange Equilibrium Isotherms**

Ion exchange equilibrium isotherms were established for copper on the TP 207 resin at four different pH set points **(Refer to Figure 11.2)**. The ion exchange equilibrium experiments were conducted according to the method outlined in Section 10.6. The experimental data and detailed results of the mass balance may be found in Appendix E for each equilibrium isotherm.

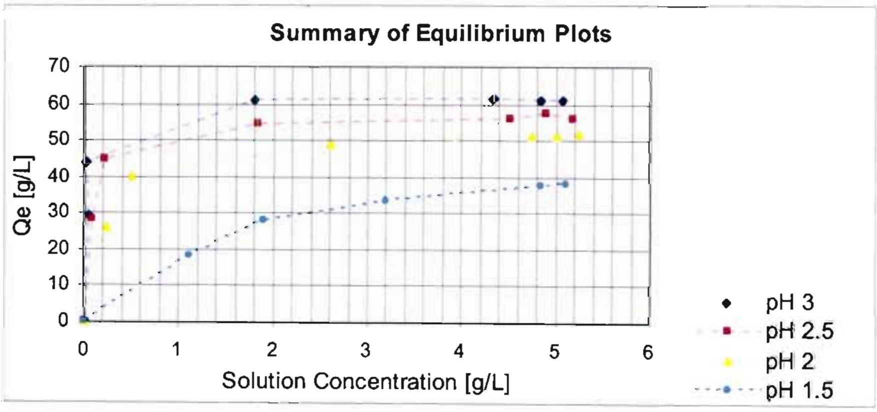


Figure 11.2: Equilibrium Isotherm of TP 207 Resin [Favourable Isotherm]

The equilibrium curve that was determined showed that pH does have an effect on the TP 207 weak acid resin. It is evident from the curve that when the pH was maintained at 1.5, the maximum loading of copper onto the TP 207 resin was 37 g/L. But when the pH was maintained at

3, the maximum copper loaded onto the TP 207 resin was 61g/L. This confirms the pH tests that the optimum loading for copper on the TP 207 is at pH 3.
The separation factor is defined in Equation 11.1.

$$r_{H^+}^{Cu^{2+}} = \frac{C_A(Q - q_A)}{q_a(C_o - C_A)} = \frac{x(1-y)}{y(1-x)} \quad \text{.......................... (Equation 11.1)}$$

Equation 11.1 can be used to classify the equilibrium behaviour into the following categories:

- Strongly favourable (with $r \leq 0.3$); in the limiting case, irreversible ($r=0$)
- Linear ($r = 1$)
- Non Linear ($0.3 < r < 10$)
- Strongly unfavourable ($r > 10$)

From the results calculated for this parameter at pH 3 and pH 2.5, the separation factor was less than 0.3 therefore from above criteria the equilibrium is strongly favourable. The distribution factor was also calculated. For each of the equilibrium curve Langmuir and Freundlich equilibrium plots were determined **(Refer to Appendix E for detailed calculations).**

11.3.2.1 The Langmuir Isotherm for Equilibrium Curve at pH 3

From the Langmuir expression and taking the reciprocals:

$$q_e = \frac{ab * C_e}{(1 + bC_e)} \quad \text{.. (Equation 11.2)}$$

$$\frac{1}{q_e} = \frac{1 + b * C_e}{ab * C_e} \quad \text{.. (Equation 11.3)}$$

$$\frac{1}{q_e} = \frac{1}{ab * C_e} + \frac{b * C_e}{ab * C_e} \quad \text{................................ (Equation 11.4)}$$

$$\frac{1}{q_e} = \frac{1}{ab * C_e} + \frac{1}{a} \quad \text{.. (Equation 11.5)}$$

Likening Equation 11.5 to a "y = mx + c" straight line plot, a graph of ($1/q_e$) against ($1/C_e$) will generate a y-axis intercept = (1/a) and a gradient = (1/ab intercept), if the equilibrium conforms to this relationship.

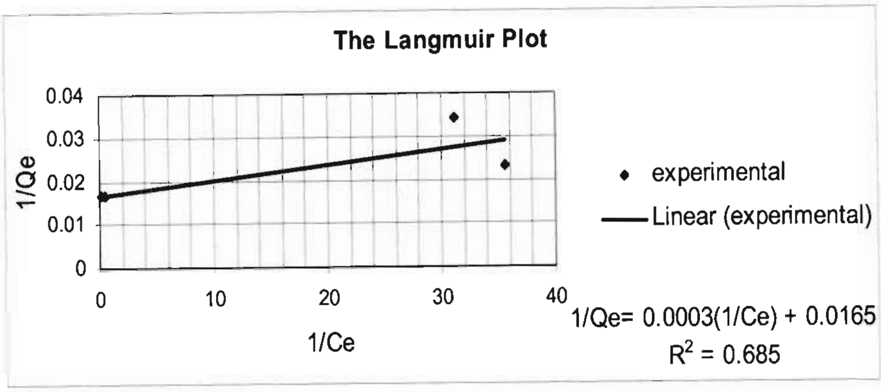


Figure 11.3: Langmuir Isotherm for Equilibrium Curve at pH 3

Fitting Parameters for the Langmuir Isotherm are present in Table 11.2.

TABLE 11.2: Fitting Parameters for Langmuir Isotherm at pH 3

a	60.606
b	55
r	0.827

Langmuir Model:

$$q_e = \frac{ab * C_e}{(1 + bC_e)} = \frac{3333.33 * C_e}{(1 + 55C_e)} \text{ .... } \textbf{(Equation 11.6)}$$

11.3.2.2 The Freundlich Isotherm for Equilibrium Curve at pH 3

From the Freundlich expression and taking logs:

$$q_e = K_f C_e^{1/n} \text{ .... } \textbf{(Equation 11.7)}$$

$$\log(q_e) = \log(K_f) + \log(C_e^{1/n}) \text{ .... } \textbf{(Equation 11.8)}$$

$$\log(q_e) = \log(K_f) + (1/n)\log C_e \text{ .... } \textbf{(Equation 11.9)}$$

Likening Equation 11.9 to a "y = mx + c" a straight-line plot, a graph of (log q_e) against (log C_e) will generate a gradient = 1/n and y-axis intercept = log (K_f), if the equilibrium conforms to this relationship.

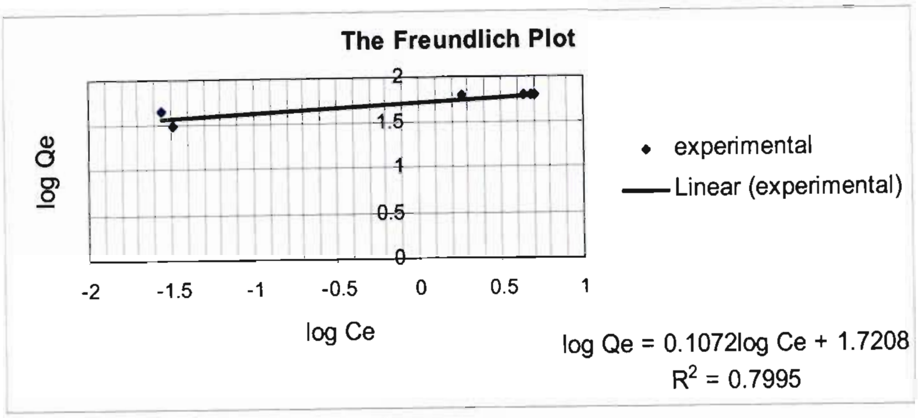


Figure 11.4: Freundlich Isotherm for Equilibrium Curve at pH 3

Fitting Parameters for the Freundlich Isotherm are present in Table 11.3

TABLE 11.3: Fitting Parameters for Freundlich Isotherm at pH 3

1/n	0.1072
K_f	52.578
r	0.89

Freundlich Model:

$$q_e = K_f C_e^{1/n} = 52.6 C_e^{0.1} \text{ ....} \textbf{(Equation 11.10)}$$

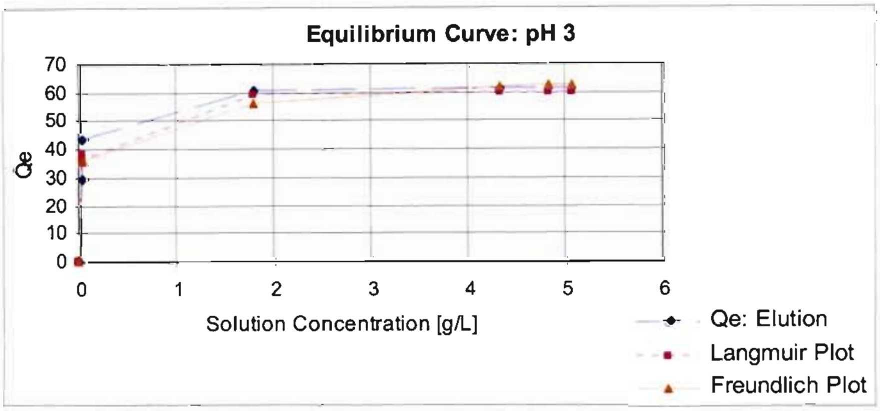


Figure 11.5: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 3

The above analysis was repeated for the three other pH values and the final results are summarized in the graphs below with a tabulation of the final models in Table 11.4.

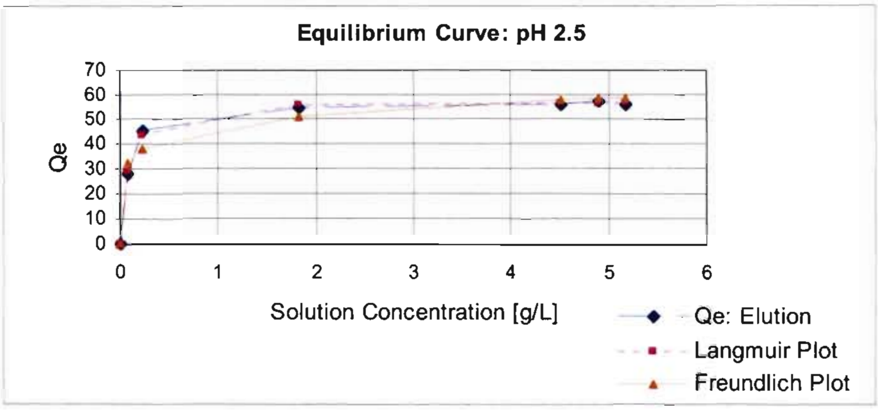


Figure 11.6: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 2.5

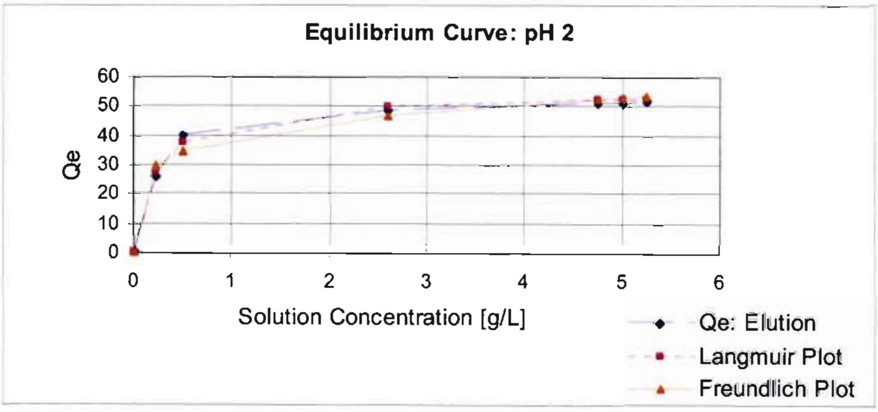


Figure 11.7: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 2

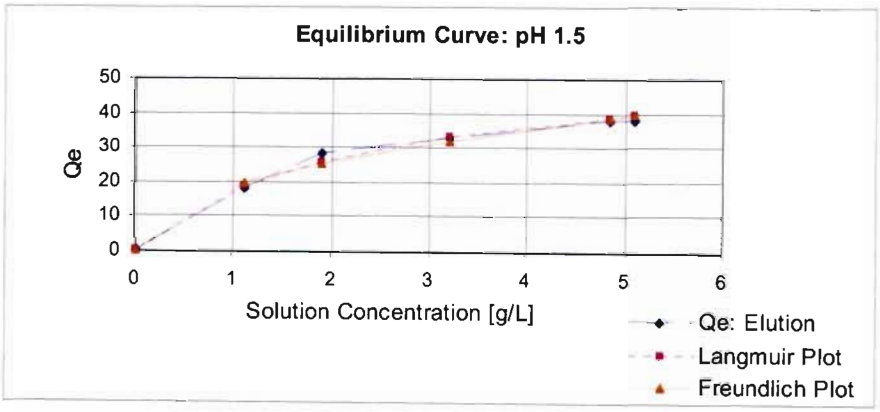


Figure 11.8: A Comparison of Experimental Data with the Langmuir and Freundlich Plots at pH 1.5

From the Langmuir and Freundlich plots, it is evident that both adsorption models resulted in a reasonably good correlation between statistical fit and the experimental data, with the Langmuir model fitting the experimental data slightly better than the Freundlich model.

TABLE 11.4: Final Results and Fitting Parameters for Equilibrium Curves

pH	Fitting Parameter:		Langmuir Curve	Freundlich Curve
	Langmuir	Freundlich		
3	a=60.606 b=55 r=0.82	1/n =0.10 Kf=52.6 r =0.89	$qe = \frac{3333.33 * C_e}{(1+55C_e)}$	$q_e = 52.6\ C_e^{0.1}$
2.5	a=57.47 b=14.5 r=0.99	1/n =0.1386 Kf=46.76 r =0.92	$qe = \frac{833.32 * C_e}{(1+14.5C_e)}$	$q_e = 46.76\ C_e^{0.14}$
2	a=54.348 b=4.279 r=0.99	1/n =0.1869 Kf=38.9 r =0.9	$qe = \frac{232.56 * C_e}{(1+4.28C_e)}$	$q_e = 38.9\ C_e^{0.187}$
1.5	a=56.497 b=0.44 r=0.99	1/n =0.46 Kf=18.625 r =0.97	$qe = \frac{24.86 * C_e}{(1+0.44C_e)}$	$q_e = 18.6\ C_e^{0.46}$

11.3.3 Kinetic Tests

Kinetic tests were conducted with feed solution concentrations of 6 and 0.6 g/L. The resin was screened and two particle sizes 850 μm and 600 μm were used to find the effect of particle size on the rate of adsorption of copper onto the TP 207 resin, at a pH set point of 3. The experimental procedure employed for the kinetic tests is described in Section 10.7. The experimental data and detailed results of the mass balance may be found in Appendix F.

Figure 11.9 is the kinetic response for particle size 850 micrometers and initial solution concentration of 6 g/L. The procedure was repeated for concentration of 0.6 g/L and particle diameter 600 micrometers **(Refer to Appendix F)**.

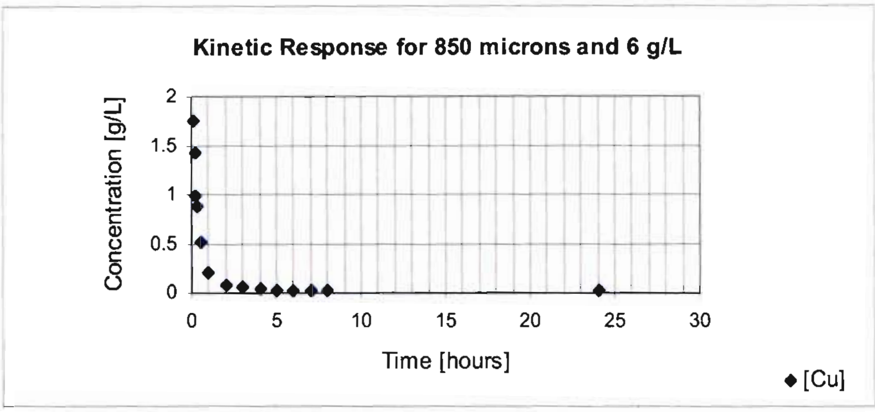


Figure 11.9: Kinetic Response for 850 μm and Initial Concentration 6 g/L

The integral method **(Refer to Chapter Three, Section 3.5.2)**, uses trial and error procedures to find the reaction order. This method was used to determine the order of the reaction for each kinetic response. Figure 11.9 was modelled into a first and second order reaction curve.

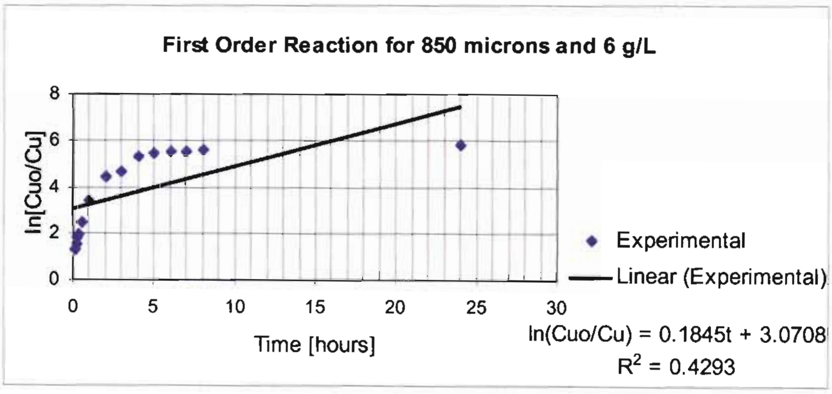


Figure 11.10: First Order Reaction Kinetics for 850 μm and Initial Concentration 6 g/L

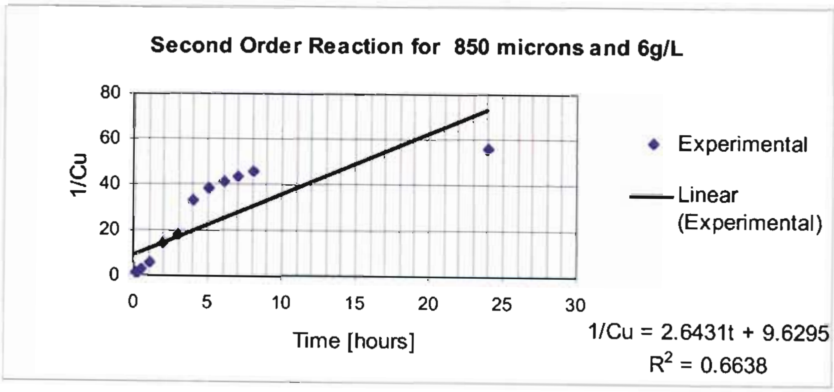


Figure 11.11: Second Order Reaction Kinetic for 850 μm and Initial Concentration 6 g/L

By plotting 1/Cu versus time (for second order reactions only), gave a better fit than the first order reaction **(Refer to Figure 11.10 and Figure 11.11)** therefore the order of the reaction was determined experimentally to be 2. The procedure was repeated for both concentrations and particle sizes. The slope of the linear plot rendered the mass transfer coefficient k. The k value was used to predict the [Cu] and compare it to experimental [Cu]. These values correlate well **(Refer to Appendix F)**. The mass transfer coefficient (k) values are summarized in Table 11.5 and Table 11.6.

TABLE 11.5: Mass Transfer Coefficient (k) for Diameter 850 μm

Mass Transfer Coefficient (k) at 6 and 0.6 g/L		
Concentration	**6 g/L**	**0.6 g/L**
k: 1st order	0.1845	0.1769
k: 2nd order	2.6431	4.857

TABLE 11.6: Mass Transfer Coefficient (k) for Diameter 600 μm

Mass Transfer Coefficient (k) at 6 and 0.6 g/L		
Concentration	**6 g/L**	**0.6 g/L**
k: 1st order	0.2068	0.2496
k: 2nd order	5.6317	23.96

From the graphs below, one can compare how particle size has an effect on concentration, these results are shown in Figure 11.12 and Figure 11.13. The effect of particle size for the TP 207 resin at 850 μm and 600 μm are represented in Figure 11.12 for concentration 6 g/L. From this graph it is clearly seen that particle size has no effect for an initial concentration of 6 g/L.

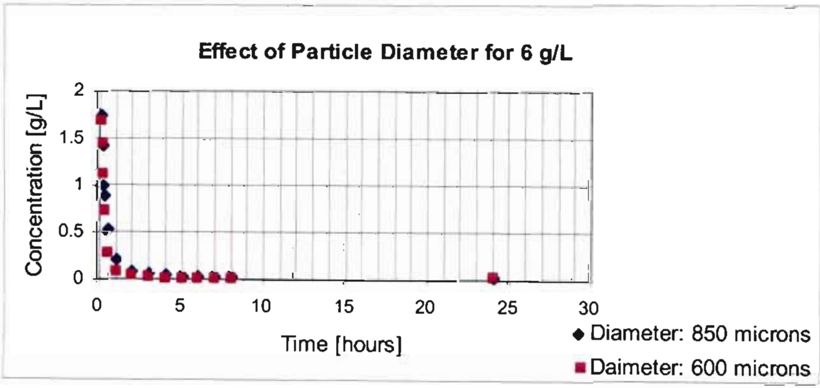


Figure 11.12: Effect of particle diameter at initial concentration of 6 g/L

But from Figure 11.13 the 850 μm particle diameter curve is slightly higher than the 600 μm curve. This implies that the smaller diameter particle (that is 600 μm) absorbs copper faster onto the TP 207 resin than the bigger diameter particle (that is 850 μm) at concentration 0.6 g/L.

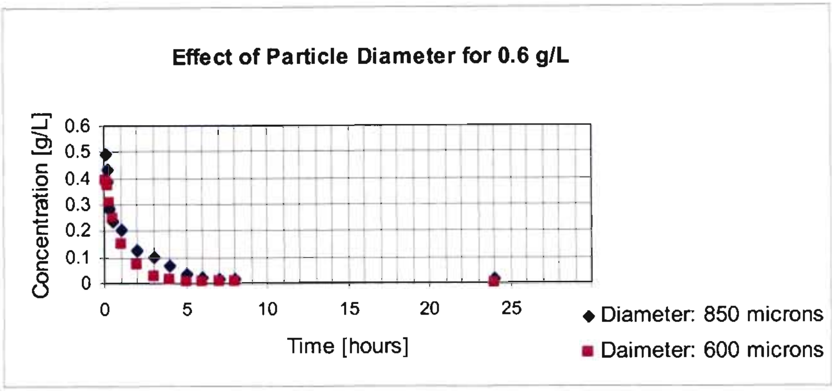


Figure 11.13: Effect of particle diameter at initial concentration of 0.6 g/L

Due to the fact that the resin and solution are in a batch system and are being constantly stirred, bulk and film diffusion can be neglected therefore particle diffusion is the rate determining step.

11.3.4 Effect of Swelling

The experimental procedure employed is described in Section 10.8. Resin in the hydrogen form was used as a reference to calculate the percentage swelling of the resin from one form to another. The calculations may be found in Appendix C5.

The percentage swelling is important to calculate because:
The resin is usually in the hydrogen form but the manufactures would rather spend more money to convert the resin into the di sodium form using sodium hydroxide, because the resin swells to 34.9 % in the di sodium form compared to the hydrogen form.

Therefore the manufactures of the resin sells 34.9% more resin in the di sodium form. If it was sold in the hydrogen form the manufactures would lose out 34.9 % of resin.

11.4 Mini Column Tests

The mini column tests were conducted to find the effects of feed pH on the residence time. Three forms of resin (i.e. hydrogen, di sodium, and mono sodium form) and four 6 g/L initial copper solution concentration set at four pH values (that is 3, 2.5, 2, and 1.5) were used for each mini column test.

The test enables to choose one form of resin for future test work. The experimental procedure employed for the mini column tests is described in Section 10.9. A sample calculation and summarized experimental data may be found in Appendix G.

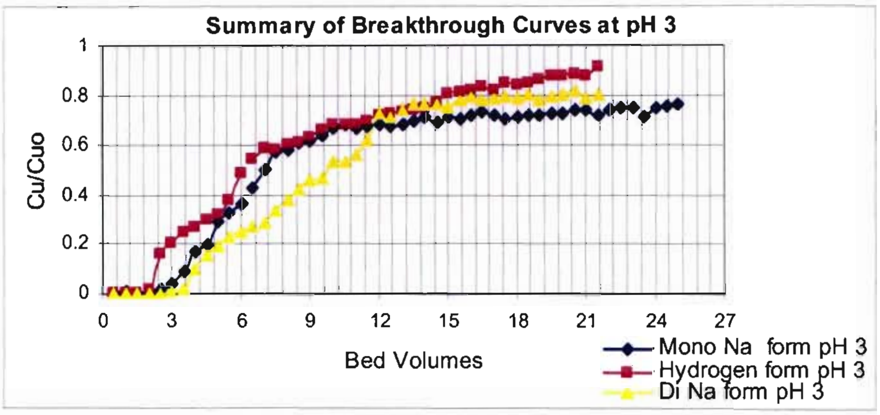


Figure 11.14: Resin in Three forms at Initial Feed pH of 3

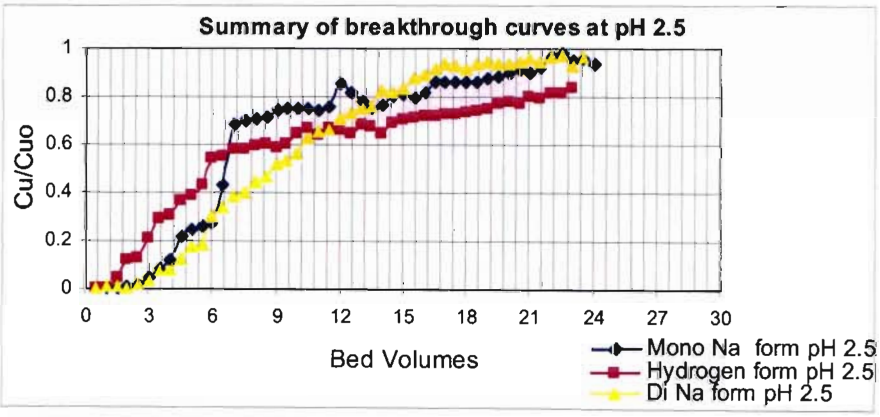


Figure 11.15: Resin in Three forms at Initial Feed pH of 2.5

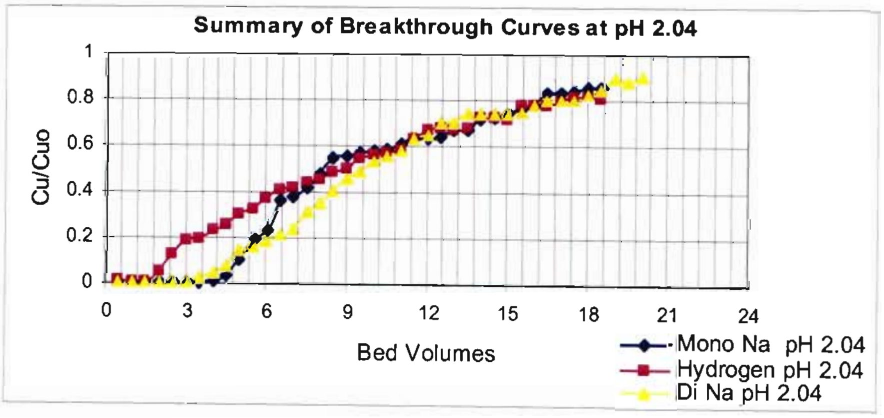


Figure 11.16: Resin in Three forms at Initial Feed pH of 2.04

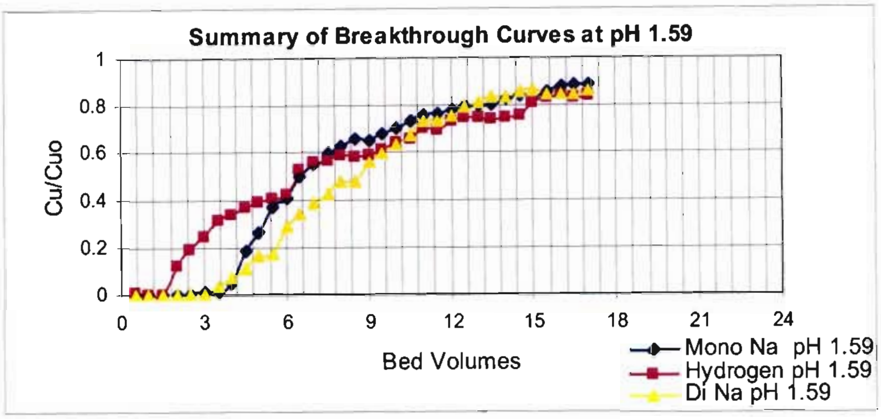


Figure 11.17: Resin in Three forms at Initial Feed pH of 1.59

The results of the mini column tests are summarized in the tables below. The mass transfer zone (MTZ) heights and residence time at the four initial copper solution concentration set at four pH values were 3, 2.5, 2, and 1.5, are shown in Tables 11.7 to Tables 11.10.

The mass transfer zone height was based on the feed throughput required at maximum tolerable impurity and was chosen at 99% recovery of initial feed solution, that required to obtain 80 % of breakthrough of copper in the effluent. This was used as basis for all calculations for comparative purposes and hydrogen was used as a reference to calculate the mass transfer zones for each pH because of the swelling that occurs when the resin is converted to the other forms.

TABLE 11.7: Tabulation of Three Forms of Resin at Initial Feed pH 3

Form of resin	MTZ [cm]	Residence Time [min]
Hydrogen	12.97	6.36
Di sodium	12.45	6.1
Mono Sodium	13.36	6.57

TABLE 11.8: Tabulation of Three Forms of Resin at Initial Feed pH 2.5

Form of resin	MTZ [cm]	Residence Time [min]
Hydrogen	12.97	6.36
Di sodium	12.4	6.1
Mono Sodium	12.35	6.07

TABLE 11.9: Tabulation of Three Forms of Resin at Initial Feed pH 2

Form of resin	MTZ [cm]	Residence Time [min]
Hydrogen	13.47	6.54
Di sodium	12	5.83
Mono Sodium	11.83	5.74

TABLE 11.10: Tabulation of Three Forms of Resin at Initial Feed pH 1.59

Form of resin	MTZ [cm]	Residence Time [min]
Hydrogen	13.34	6.48
Di sodium	11.28	5.48
Mono Sodium	11.09	5.38

The residence time (τ) is the contact time that the initial solution has with the resin in the column. Since the initial feed pH was set at 3, 2.5, 2 and 1.5 it was determined from the above results that the initial feed pH has little effect on the residence time and one feed pH set at 3 for all solutions was used for future test work.

The resin in the di sodium form would be more expensive than the Hydrogen because:

Conversion to the di sodium form:
When the resin is saturated with copper, the resin would have to be converted to the hydrogen form (to recover the copper due to selectivity) and then to the sodium form (Higher Operating Cost). That is also the case for the mono sodium form.

Conversion to the Hydrogen form:
The resin would just have to be converted to the hydrogen form to recover the copper.

Also the resin in the di sodium form swells compared to the hydrogen form **(Refer to Section 11.3.4)** this implies that a longer column is required for the Di sodium form of resin than the hydrogen form (Higher Capital Cost). Therefore the resin in the hydrogen form was used to conduct further studies for fixed and fluidized column tests.

11.5 **Determination of Minimum Fluidization Velocity**

This test was carried out to find the minimum velocity at which to operate the fluidized bed. The column test and the minimum fluidization velocity test were used to determine one velocity to operate the fixed and fluidized columns thus allowing comparisons of the two columns to be determined.

The standard method of finding u_f is of plotting pressure drop versus velocity and looking for an intercept of the two straight portions of the experimental curves. However the standard method was not used, but plotting of the bed height versus column velocity was determined **(Refer to Figure 11.18)**. The above was performed to confirm that the resin in the fluidized ion exchange column was in fact being fluidized. The experimental procedure employed for the minimum fluidization velocity is described in Section 10.10. The experimental data and detailed results may be found in Appendix H.

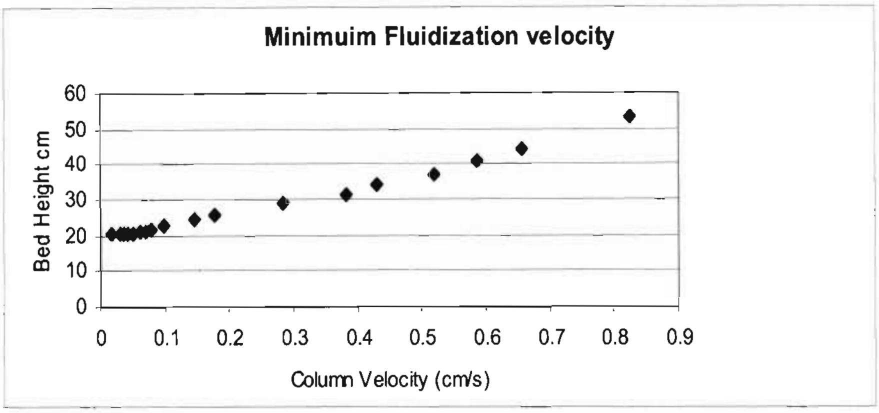


Figure 11.18: Experimental Determination of Minimum Fluidization Velocity

Minimum fluidization velocity was determined at the intersection between the straight line for the bed been fluidized and the horizontal line for packed beds **(Refer to Figure 11.18)**. The minimum fluidization velocity was experimentally determined to be 0.079 cm/s.

The experimental determination was compared to the predicted minimum fluidization value and is present in Table 11.11. The Kozeny-Carman Equation **(Refer to Chapter Five, Equation 5.1)** was used to determine the predicted value. The experimental value is 4.5 times greater than the predicted value. The Kozeny-Carman Equation does not correlate well with the experimental determination.

TABLE 11.11: Theoretical Determination of Minimum Fluidization and Settling Velocity

Minimum Fluidization Velocity	0.0175 cm/s [Refer to Appendix C3]
Terminal Velocity	4.136 cm/s [Refer to Appendix C3]

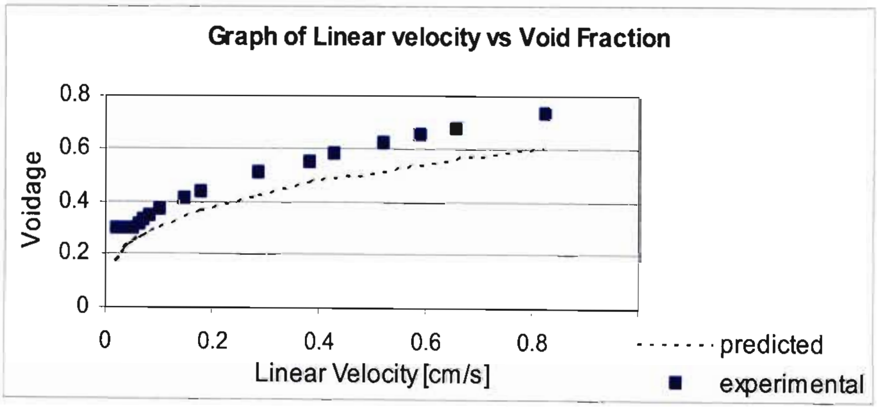


Figure 11.19: Comparison of Predicted Data and Experimental Data for the Graph of Void Volume versus Linear Velocity

The Richardson and Zaki correlations were used to predict the void volume versus linear velocity. The experimental curves were compared to the Richardson and Zaki (predicted) curve **(Refer to Figure 11.19)**.

The "Students t-test" was performed **(Refer to Table 11.12)** using computer package Microsoft Excel, to calculate the standard deviation for the curves. A 95 % level of accuracy was used, t Statistic must fall within the region of ± t Critical two-tailed.

TABLE 11.12: t -Test (Two-Sample Assuming Equal Variances)

	Experimental	Predicted
Mean	0.437820813	0.358253445
Variance	0.02362467	0.018533028
Observations	18	18
Pooled Variance	0.021078849	
Hypothesized Mean Differ-	0	
df	34	
t Statistic	1.644117321	
P(T<=t) one-tail	0.054684268	
t Critical one-tail	1.690923455	
P(T<=t) two-tail	0.109368537	
t Critical two-tail	2.032243174	

11.6 Column Tests

Column tests were performed to determine one linear velocity to operate at for future testing on the fixed and fluidized ion exchange beds. The experimental procedure employed for the column testing is described in Section 10.11.

The experimental data and detailed results of the mass balance may be found in Appendix I. The breakthrough curves for copper on the TP 207 resin are shown in Figure 11.20 to Figure 11.22. The mass transfer zone (MTZ) heights, determined for copper at the three different superficial linear velocities, are shown in Table 11.13.

The mass transfer zone height was based on the feed throughput required at maximum tolerable impurity and was chosen at 99% recovery of initial feed solution, that required to obtain 80 % of breakthrough of copper in the effluent.

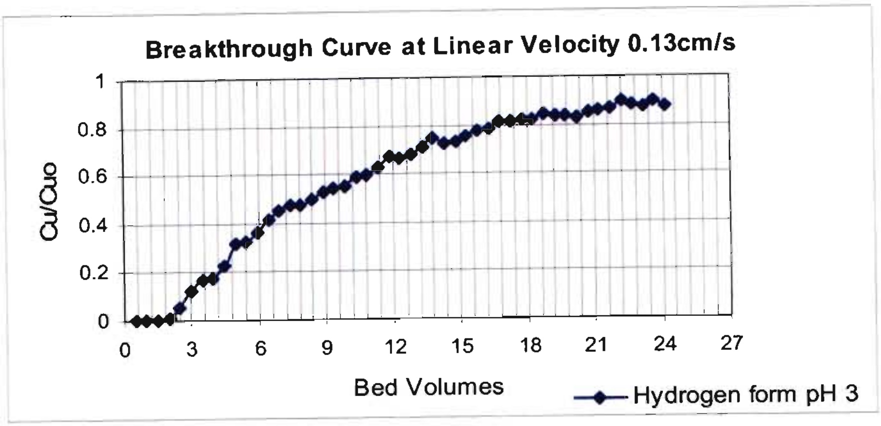


Figure 11.20: Breakthrough curve at Linear Velocity of 0.13 cm/s

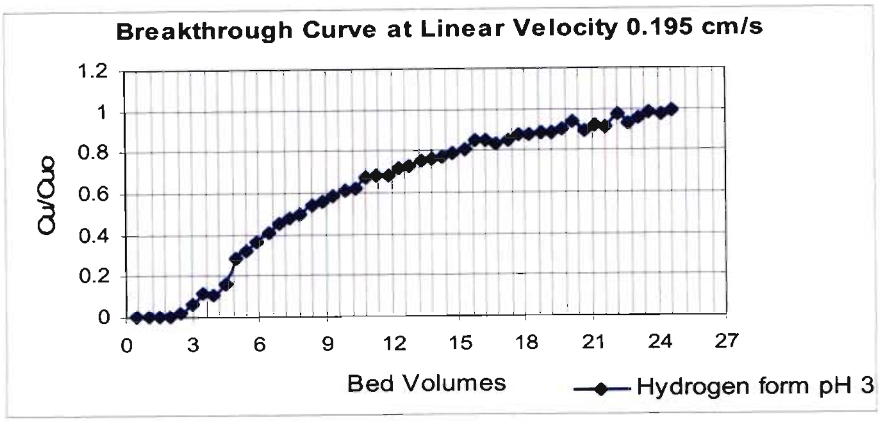


Figure 11.21: Breakthrough curve at Linear Velocity of 0.195 cm/s

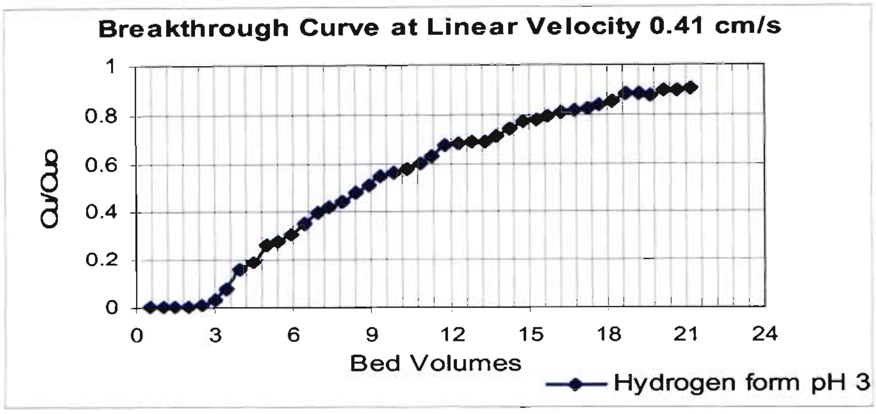


Figure 11.22: Breakthrough curve at Linear Velocity of 0.41 cm/s

TABLE 11.13: Tabulation of the Mass Transfer height obtained at respective velocity

Linear velocity [cm/s]	MTZ [cm]	Residence Time [min]
0.13	80	10.3
0.195	153.998	13.2
0.41	304.533	12.38

From the results obtained in Table 11.13, a graph of linear velocity versus the mass transfer zone height can be plotted. Refer to Figure 11.23, using the equation MTZ = 779.65 LV - 11.904, for linear velocities ranging from 0.13 cm/s to 0.41 cm/s the mass transfer height can be predicted.

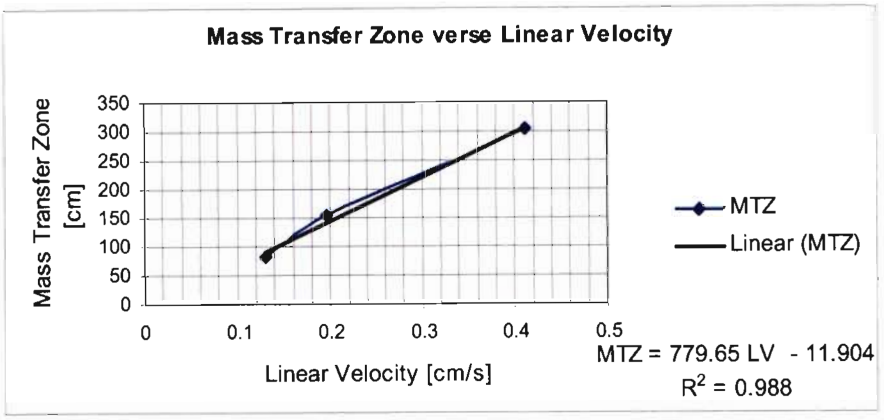


Figure 11.23: Graph of Mass Transfer Zone versus Linear Velocity

From the breakthrough test results the columns were sized at each linear velocity. Refer to Appendix I5 to I7 for the full scale plant design for each linear velocity.

The optimum linear velocity for future column testing was chosen from the full scale plant design and the minimum fluidization velocity tests. The design was based on the H:D (height to diameter) ratio. H:D should be in the range 2/3 to 3/2 **[Internet]** [37]. The linear velocity was chosen at 0.13 cm/s which is equal to 1.57 H:D (height to diameter) ratio. This linear velocity is above the minimum fluidization velocity and is 1.65 u_f.

11.7 Pilot Plant Testing

The main purpose of this research was to compare the performance of a fixed bed ion exchange column with a fluidized ion exchange column for the recovery of copper from a cupric sulphate solution. By experimentation the bed depth for each type of equipment (in order to achieve a specified percentage recovery of copper from a specified feed) was determined at two concentration values of 6 and 0.6 g/L.

The experimental procedure employed for the pilot plant testing is described in Section 10.12. The experimental data and detailed results of the mass balance may be found in Appendix J and K.

11.7.1 Breakthrough Tests

From all the experimental work obtained thus far, plans for running the pilot plants at optimum conditions could be enabled. The breakthrough curves obtained from pilot plant testing for copper on the TP 207 resin are shown from Figures 11.24 to Figures 11.26 for feed concentration of 6 g/L and from Figures 11.27 to Figures 11.29 for feed concentration of 0.6 g/L. The mass transfer zone (MTZ) heights, determined for copper are shown in Table 11.14 and Table 11.15.

Again the mass transfer zone height was based on the feed throughput required at maximum tolerable impurity and was chosen at 99% recovery of initial feed solution, that required to obtain 80 % of breakthrough of copper in the effluent.

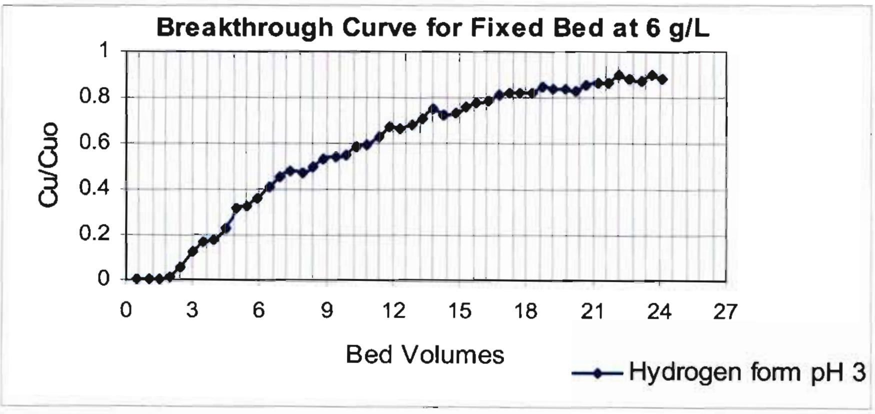


Figure 11.24: Fixed Bed Testing at 6 g/L

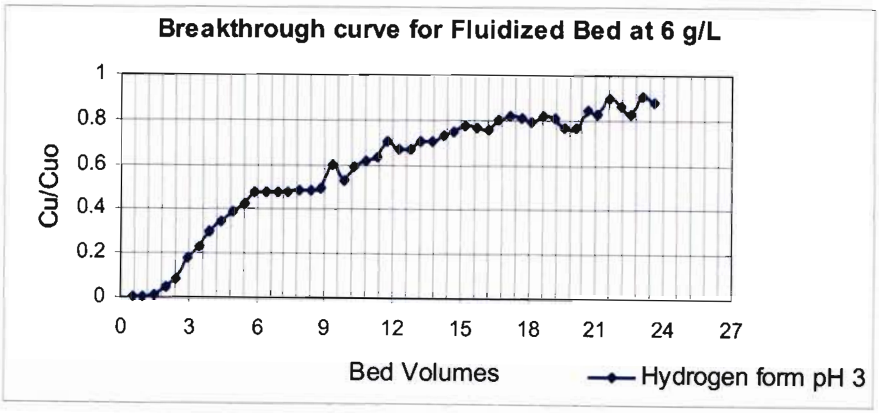


Figure 11.25: Fluidized Bed Testing at 6 g/L

TABLE 11.14: Tabulation of Mass Transfer Zone heights for 6 g/L

Contacting Equipment	MTZ [cm]	Residence Time [min]
Fixed Bed	80	10.3
Fluidized Bed	82	9.95

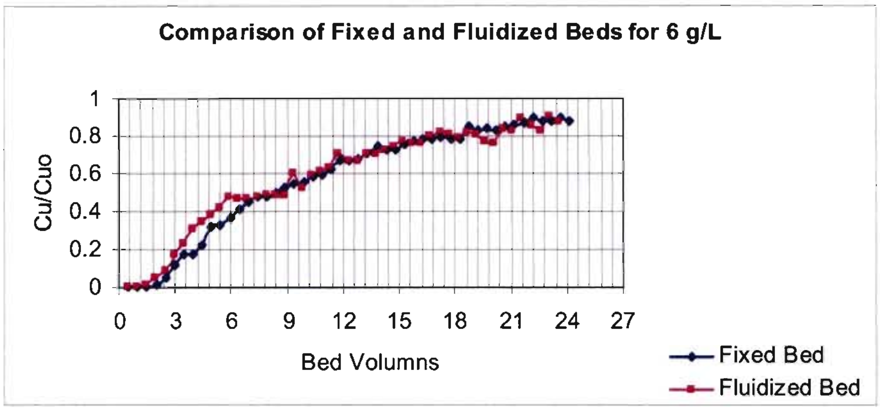


Figure 11.26: Comparison of Fixed and Fluidized bed at 6g/L

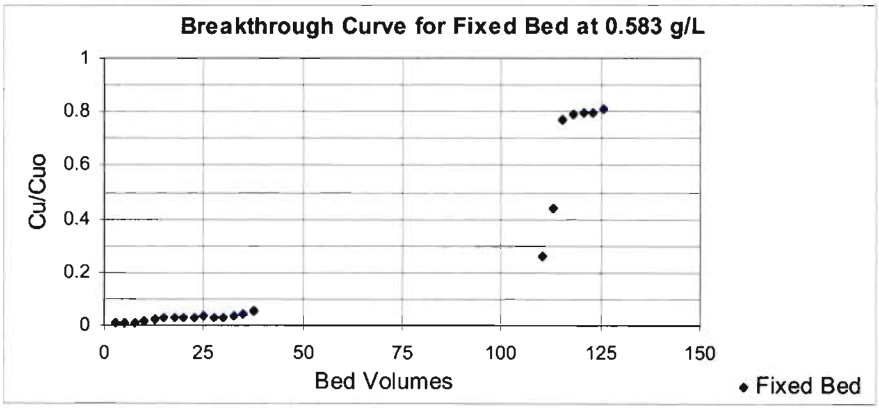


Figure 11.27: Fixed Bed Testing at 0.6 g/L

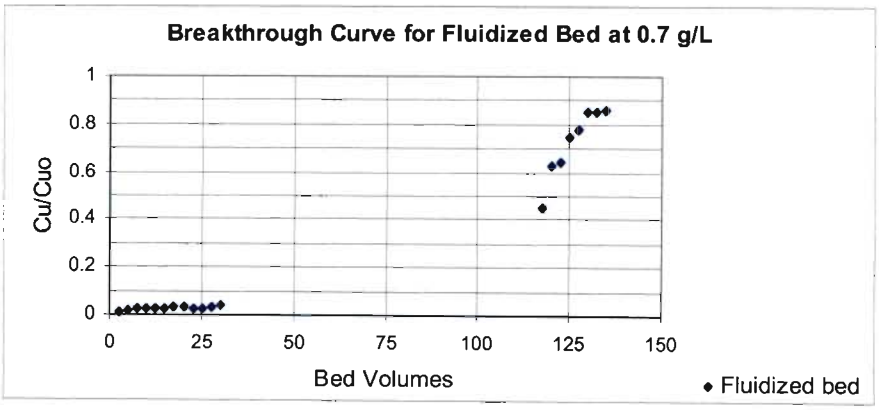


Figure 11.28: Fluidized Bed 0.7 g/L

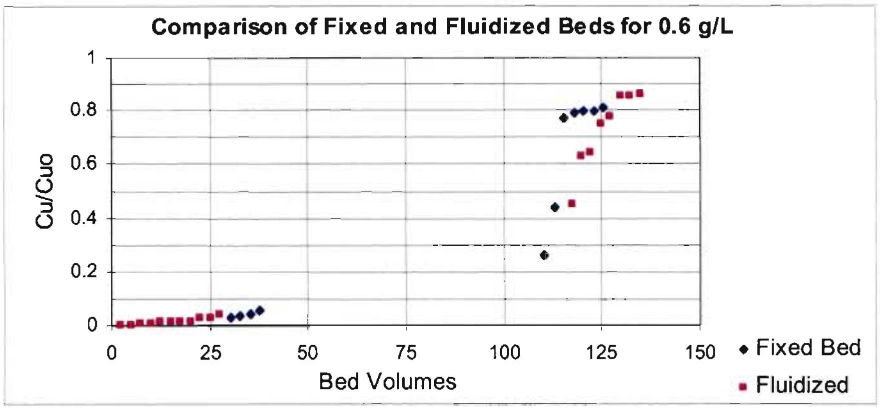


Figure 11.29: Comparison of Fixed and Fluidized bed at 0.6 g/L

TABLE 11.15: Tabulation of Mass Transfer Zone heights for 0.6 g/L

Contacting Equipment	MTZ [cm]	Residence Time [min]
Fixed Bed	81	9.58
Fluidized Bed	81	9.66

The breakthrough curves were determined for two concentrations at 6 and a 0.6 g/L of copper solution for both contacting equipment **(Refer to Figure 11.24 – Figure 11.29)**. The same linear velocity and the same amount of resin were used for both contacting equipment.

For the 6 g/L concentration:

Fixed Bed: Copper started appearing in the effluent stream after twenty seven minutes of operation.

Fluidized Bed: Copper started appearing in the effluent stream after twenty five minutes of operation.

For the 0.6 g/L concentration:

Fixed Bed: Copper started appearing in the effluent stream after eight and a half hours of operation.

Fluidized Bed: Copper started appearing in the effluent stream after five and a half hours of operation.

The same amount of resin was used, so the resin had the same functional sites available. At a higher concentration breakthrough occurs much faster than at a lower concentration therefore the effect of concentration of the feed solution is that it would take much longer to reach breakthrough at a lower concentration than at a higher concentration.

In terms of time, breakthrough for a fluidized bed at both conditions occurs slightly faster than the fixed bed, this could be due to the resin having better contact with the liquid during operation. A comparison on the resin bed depth required for each contacting equipment from the full scale plant design is located below.

11.7.2 **Full Scale Plant Design**

11.7.2.1 **Sizing of Pilot Plant**

During the piloting of the ion exchange systems, the design was based on a longitudinal cross section of the full scale plant and was evaluated at the superficial linear velocity to be employed for the full scale operation. This is illustrated in Figure 11.30.

The size of the full scale plant was based on a total feed flow rate of 5 m^3/h. Again, the design was based on the H:D ratio. H:D should be in the range 2/3 to 3/2 **[Internet]** [37]. A summary of the results for the sizing of the columns can be seen in Table 11.16 for 6 g/L and Table 11.17 for 0.6 g/L **(Refer to Appendix L for the detailed results of the full scale plant design)**.

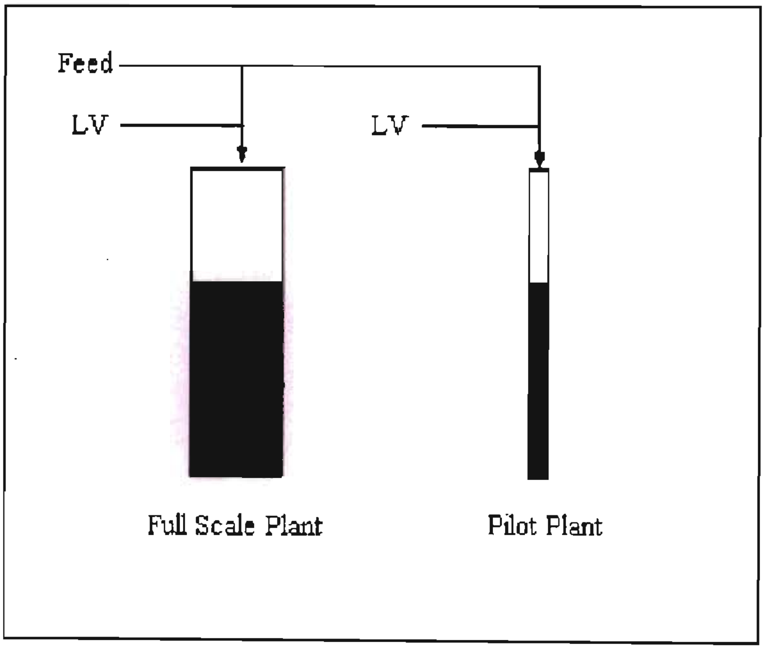


Figure 11.30: Sizing and Operation of an Ion Exchange Pilot Plant [Wyethe] [37]

From the summarized results obtained in Table 11.16 and Table 11.17 a comparison of the resin bed depths was required to compare both contacting equipment.

TABLE 11.16: Preliminary Column Design and Operating Parameters for the Removal of Copper from a Cupric Sulphate Solution at 6 g/L

FULL SCALE PLANT DESIGN OF CONTACTING EQUIPMENT AT 6 g/L				
Input :			FIXED BED	FLUIDIZED BED
	Feed: Flow rate	m^3/h	**5**	**5**
	Feed: Linear velocity	m/hr	**4.68**	**5.04**
	Feed: Copper Concentration	g/L	**6**	**6**
	Resin Copper loading	g/L	**60**	**60**
	Overall Resin Bed Depth Required	m^3	2.3	2.3
Plant Size	Column: Diameter	m	**1.1**	**1.1**
	Column: Resin Bed Height	m	2.2	2.3
	H:D		1.8	2
PILOT PLANT DESIGN				
Plant Size	Column Dimensions:			
	Column: Diameter	cm	2.4	2.4
	Column: Height	m	1	2
Input:	Feed: Flow rate	mL/min	36.6	37.97
		BV/hr	5.4	5.58
	Feed: Linear velocity	cm/s	0.13	0.14
		m/hr	4.68	5.04

At a concentration of 6 g/L an overall resin bed depth of 2.3m^3 was required for the fixed bed and 2.3m^3 for the fluidized bed.

At a concentration of 0.6 g/L an overall resin bed depth of 0.95 m^3 was required for the fixed bed and 0.95 m^3 for the fluidized bed.

Therefore in terms of the volume of resin required, there is no difference if either contacting equipment is employed. A cost estimation was performed for each contacting equipment and the assumptions for the estimates are located below.

TABLE 11.17: Preliminary Column Design and Operating Parameters for the Removal of Copper from a Cupric Sulphate Solution at 0.6 g/L

FULL SCALE PLANT DESIGN OF CONTACTING EQUIPMENT AT 0.6 g/L				
Input :			FIXED BED	FLUIDIZED BED
	Feed: Flow rate	m^3/h	5	5
	Feed: Linear velocity	m/hr	**5.112**	**5.076**
	Feed: Copper Concentration	g/L	**0.6**	**0.6**
	Resin Copper loading	g/L	**60**	**60**
	Overall Resin Bed Depth Required	m^3	0.95	0.95
Plant Size	Column Dimensions:			
	Column: Diameter	m	**1.1**	**1.1**
	Column: Resin Bed Height	m	0.97	0.97
	H:D		0.86	0.86
PILOT PLANT DESIGN				
Plant Size	Column Dimensions:			
	Column: Diameter	cm	2.4	2.4
	Column: Height	m	1	2
Input:	Feed: Flow rate	mL/min	38.635	38.243
		BV/hr	5.8	5.7
	Feed: Linear velocity	cm/s	0.142	0.141
		m/hr	5.112	5.076

11.7.3 Cost Estimation

The cost for the fixed bed and a fluidized bed were estimated for the 6 g/L **(Refer to Appendix L for cost estimations for the full scale plant designs)**.

The following represents the considerations that were taken into account for the estimation. The process is continuous, requiring two columns, one online while the other is being regenerated.

11.7.3.1 Capital Expenditure

The factorial method was used to estimate the costing for the equipment obtained from the full scale plant design. The costing was based on the estimate of the purchase cost of the major equipment required for the process and the other costs were estimated as factors **(Refer to Table 11.18)** of the equipment cost.

The bare vessel for the ion exchange columns was estimated from Figure 11.31, using the values obtained from the full scale plant design.

The resin was taken as part of the capital cost because the resin can be purchased once, thereafter can be regenerated.

TABLE 11.18: Typical factors for the Estimation of Fixed Capital Cost [Sinnot] [13]

Item	**Process Type**		
	Fluids	**Fluid-Solids**	**Solids**
1. Major Equipment, total purchase cost	PCE	PCE	PCE
f_1 Equipment Erecting	0.4	0.45	0.50
f_2 Piping	0.7	0.45	0.20
f_3 Instrumentation	0.2	0.15	0.10
f_4 Electrical	0.1	0.10	0.10
f_5 Building, process	0.15	0.10	0.05
• f_6 Utilities	0.50	0.45	0.25
• f_7 Storages	0.15	0.20	0.25
• f_8 Site development	0.05	0.05	0.05
• f_9 Ancillary buildings	0.15	0.20	0.30
2 Total Physical Plant Cost (PPC)			
$PPC = PCE\ (1 + f_1 + f_2 \ldots . f_9)$			
=PCE*	3.40	3.15	2.80
f_{10}	0.30	0.25	0.20
f_{11}	0.05	0.05	0.05
f_{12}	0.10	0.10	0.10
Fixed Capital = $PPC(1 + f_{10} + f_{11} + f_{12})$			
=PPC*	1.45	1.40	1.35
• Omitted for minor extensions or addition to existing sites.			

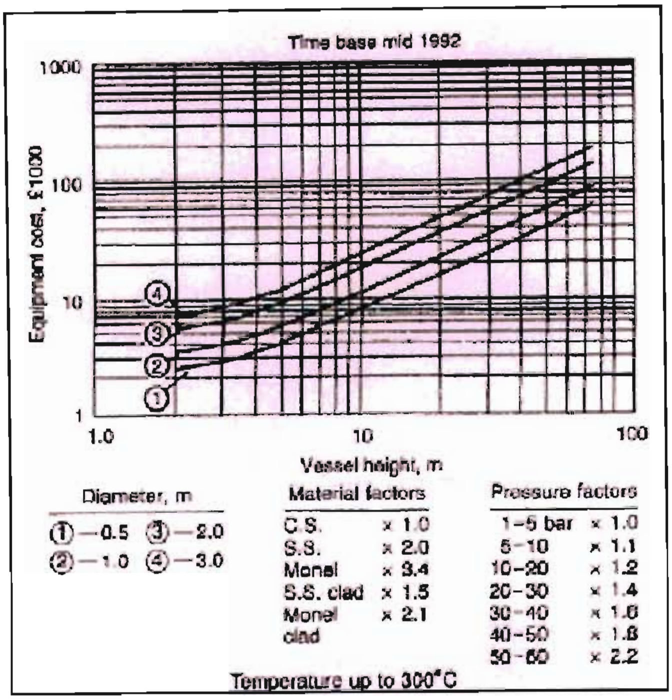


Figure 11.31: Vertical pressure vessel [Sinnot] [13]

11.7.3.2 Production Costs

TABLE 11.19: Summary of Production Costs [Sinnot] [13]

Variable Costs	*Typical Values*
1 Raw Material	From flow sheet
2 Miscellaneous Materials	10 per cent of item (5)
3 Utilities	From flow sheet
4 Shipping and Packing	Usually negligible
Sub-total A	
Fixed Costs	
5 Maintenance	5-10 per cent of fixed capital costs
6 Operating Labour	From manning estimates
7 Labour Costs	20-23 per cent of item (6)
8 Supervision	20 per cent of item (6)
9 Plant Overheads	50 per cent of item (6)
10 Capital charges	15 per cent of the fixed capital cots
11 Insurance	1 per cent of the fixed capital cots
12 Local Taxes	2 per cent of the fixed capital cots
13 Royalties	1 per cent of the fixed capital cots
Sub-total B	
Direct Production Costs A + B	
Sales Expense	20-30 per cent of the direct
General Overheads	production cost
Research and Development	
Sub-total C	
Annual Production Costs = A + B + C	

11.7.3.2(a) **Regenerant: Sulphuric Acid**

If the columns are run continuously, one online while the other is been regenerated then the columns needs to be regenerated five times a day. Therefore for 365 days the columns needs to be regenerated 1825 times. A two bed volumes of 2M sulphuric acid is sufficient to regenerate the columns using at a flow rate of one BV/h.

11.7.3.2 (b) **Operating Labour**

From estimates based on other ion exchange plants in industry. Consequently, this plant would be able to operate with one operator and half a shift supervisor per eight hour shift, working on a rotating shift basis, for a single plant.

The labour component of the operating costs assumes three eight hour shifts per day, and is based on a staff of four operators. The current wage rates per hour in the United Kingdom chemical industry are eight pounds, to which a fifty percent must be added for various allowances **[Sinnot]** [13].

11.7.3.3 **Cost Indices**

Using the cost indices from Figure 11.32 the financial values that were obtained could be inflated and forecasted for 2004.

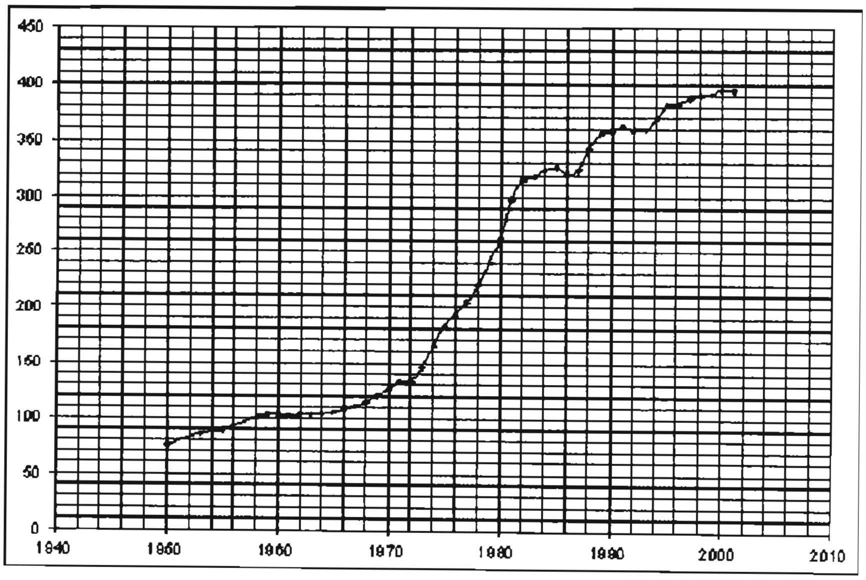


Figure 11.32: Chemical Engineering Plant Cost Indices [Internet] [70]

11.7.4 Summary

Breakthrough for a fluidized bed at both conditions occurs slightly faster than the fixed bed, this is due to the resin having better contact with the liquid during operation.

From the full scale plant design the following conclusion was determined: the same volume of resin was required for both contacting equipment. Therefore in terms of volume requirement, no contacting equipment is advantageous over the other **(Reference is made to Appendix L for full scale plant designs and cost estimations)**.

But from the cost estimation, the fixed bed column would be more advantageous in terms of capital cost investment because the fluidized bed would require a taller column to account for fluidization.

CHAPTER TWELVE: CONCLUSION

The main objective of this project was to measure the performance of fixed bed ion exchange column with a fluidized bed column at identical feed water conditions.

A comprehensive laboratory test work program was conducted in order to generate the necessary data for the planning of the fixed and fluidized ion exchange columns.

To ensure that the aims of the project were met the following was carried out:

12.1 Resin Properties were determined experimentally:

- Calculating the average particle size of the resin.
- Determining the water retention capacity of the resin.
- The effects of swelling on the TP 207 resin.
- Determining the void volume that is present in a column of the packed resin.

TABLE 12.1: Summary of Properties of TP 207 resin [Determined Experimentally]

Average Particle Size	mm	0.728
Water Retention Capacity	%	44.841
Swelling*		
Di Sodium Form	%	34.9
Mono Sodium Form	%	25
Void Volume	%	29.23

*Hydrogen was used has a reference

For detailed calculations refer to Appendix C.

12.2 Preliminary Experimental Test Work consisted of the following:

- Determination of the effect of pH on the loading of copper onto the Lewatit TP 207 , an iminodiacetic acid resin.
- Establishment of ion exchange equilibrium isotherms, at four different pH values.
- Two particle sizes (850 μm and 600 μm) were used for measurement of the kinetic response at a 6 g/L and a 0.6 g/L.

12.2.1 Effect of pH on Resin Loading

pH has an effect on copper loading onto the TP 207 resin. The results are evident from Figure 12.1. An optimum pH was determined at 3.

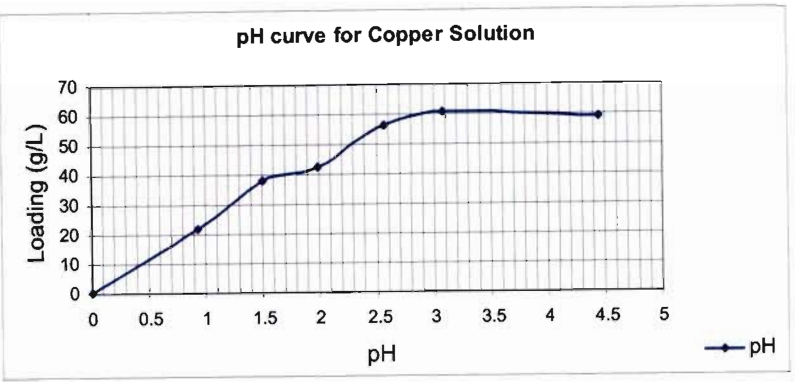


Figure 12.1: Effect of pH on Copper Loading

12.2.2 Ion Exchange Equilibria

Ion exchange equilibrium isotherms were established. A summary of the copper isotherms are presented in Figure 12.2.

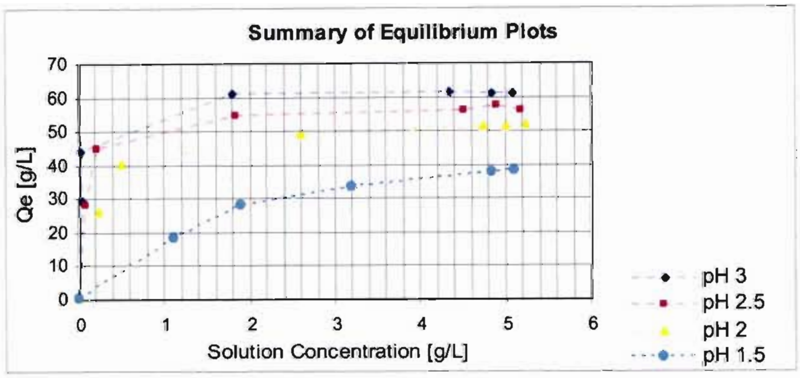


Figure 12.2: Summary of Copper Ion Exchange Equilibrium Isotherms

Langmuir and Freundlich isotherms models were fitted to the experimental data, and the fitting parameters and correlation coefficients are summarized in Table 12.2.

TABLE 12.2: Fitting Parameters and Correlation Coefficients

pH	Fitting Parameter:		Langmuir Curve	Freundlich Curve
	Langmuir	Freundlich		
3	a=60.606 b=55 r=0.82	1/n =0.10 Kf=52.6 r =0.89	$qe=\frac{3333.33*C_e}{(1+55C_e)}$	$q_e = 52.6\ C_e^{0.1}$
2.5	a=57.47 b=14.5 r=0.99	1/n =0.1386 Kf=46.76 r =0.92	$qe=\frac{833.32*C_e}{(1+14.5C_e)}$	$q_e = 46.76\ C_e^{0.14}$
2	a=54.348 b=4.279 r=0.99	1/n =0.1869 Kf=38.9 r =0.9	$qe=\frac{232.56*C_e}{(1+4.28C_e)}$	$q_e = 38.9\ C_e^{0.187}$
1.5	a=56.497 b=0.44 r=0.99	1/n =0.46 Kf=18.625 r =0.97	$qe=\frac{24.86*C_e}{(1+0.44C_e)}$	$q_e = 18.6\ C_e^{0.46}$

12.2.3 Effect of Particle Size on Kinetics

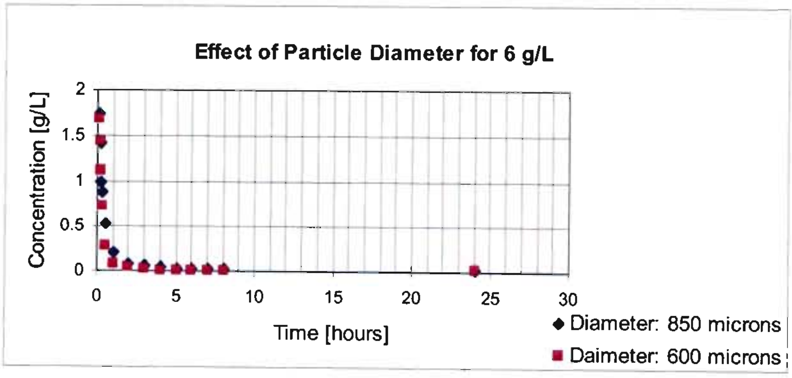


Figure 12.3: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 6 g/L

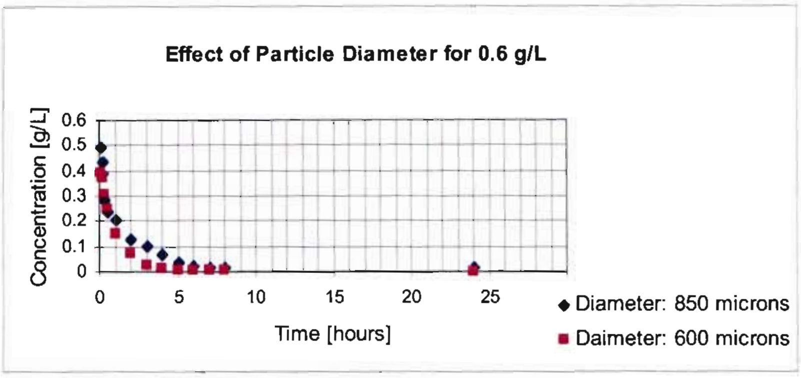


Figure 12.4: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 0.6 g/L

The integral method was used to determine the order of the reaction which was experimentally determined at 2. The slope of the linear plot determined the mass transfer coefficient k.

Particle size has an effect on concentration. These values are shown better in Figure 12.3 and Figure 12.4. Particle size has no effect on concentration at 6 g/L but for the 0.6 g/L the 850 μm diameter particle curve is slightly higher than the 600 μm curve (Figure 12.4). This implies the smaller diameter of the resin particle absorbs copper faster onto the resin than the bigger diameter particle.

12.3 Mini Column Tests

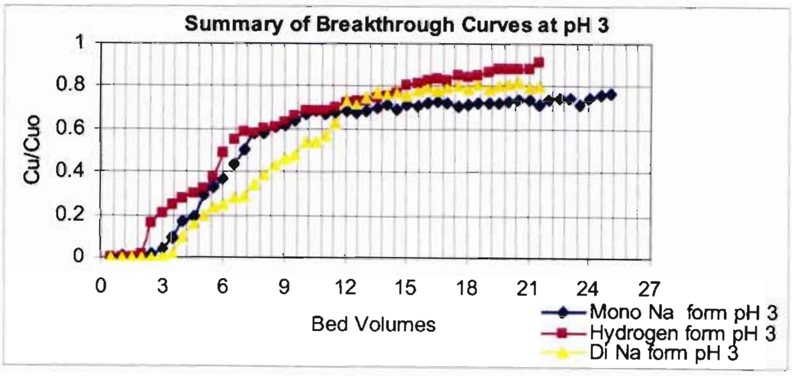


Figure 12.5: Resin in Three Forms at Initial Feed pH of 3

It was determined from the mini columns that feed pH has little effect on residence time.

Also the resin in the di sodium form swells compared to the hydrogen form **(Section 11.3.4)** this implies that a longer column is required for the di sodium form of resin than the hydrogen form. Therefore the resin in the hydrogen form was used to conduct further studies.

12.4 Minimum Fluidization Velocity

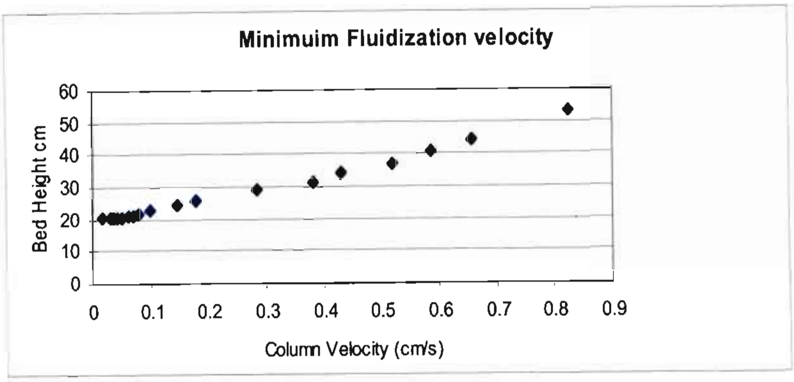


Figure 12.6: Experimental Determination of Minimum Fluidization Velocity

Minimum fluidization velocity was determined at the intersection between the straight line for the bed been fluidized and the horizontal line for packed beds **(Refer to Figure 12.6)**.
The minimum fluidization velocity was experimentally determined to be 0.079 cm/s.

12.5 Column Tests

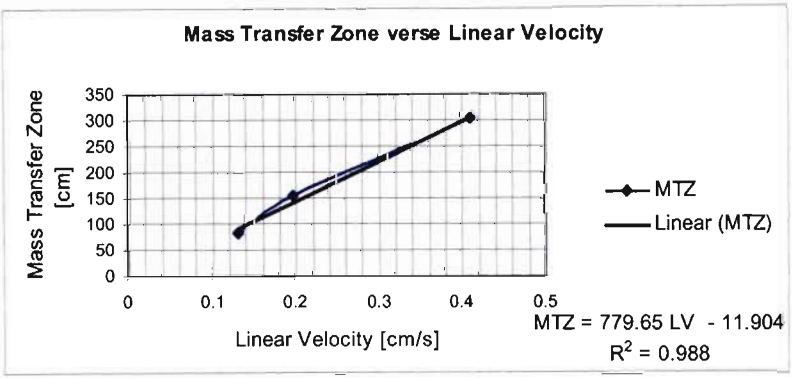


Figure 12.7: Graph of Mass Transfer Zone versus Linear Velocity

TABLE 12.3: Tabulation of the Mass Transfer Zone heights

Linear velocity [cm/s]	MTZ [cm]	Residence Time [min]
0.13	80	10.3
0.195	153.998	13.2
0.41	304.533	12.38

From the results obtained in Table 12.3 a graph of linear velocity versus the mass transfer zone height can be plotted. Refer to Figure 12.7, using the equation MTZ = 779.65 LV -11.904, for any linear velocities ranging from 0.13 cm/s to 0.41 cm/s the mass transfer zone height can be predicted.

12.6 Pilot Plant Testing

The mass transfer zone can be determined by generating the breakthrough curves for each column at the same feed conditions.

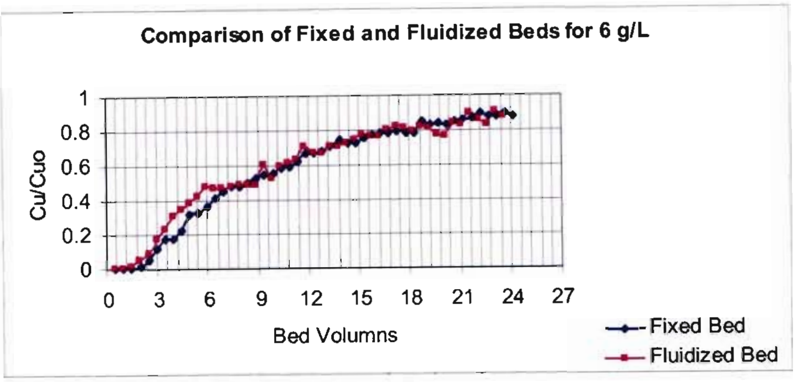


Figure 12.8: Comparison of Fixed and Fluidized bed for 6g/L

TABLE 12.4: Tabulation of the Resin Bed Depth Requirement for 6 g/L

Contacting Equipment	MTZ [cm]	Residence Time [min]	Resin Bed Depth [m^3]
Fixed Bed	80	10.3	2.3
Fluidized Bed	82	9.95	2.3

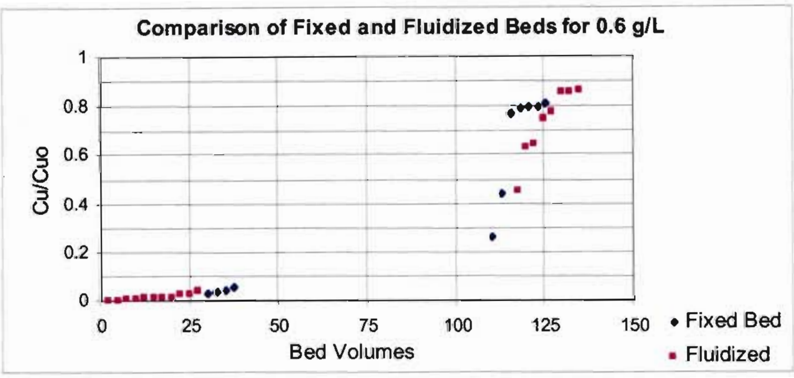


Figure 12.9: Comparison of Fixed and Fluidized bed for 0.6g/L

From Figure 12.8 and 12.9, the breakthrough for a fluidized bed at both conditions occurs slightly faster than the fixed bed; this is due to the resin having better contact with the liquid during operation.

TABLE 12.5: Tabulation of Resin Bed Depth Requirement for 0.6 g/L

Contacting Equipment	MTZ [cm]	Residence Time [min]	Resin Bed Depth [m^3]
Fixed Bed	81	9.58	0.95
Fluidized Bed	81	9.66	0.95

Comparison of fixed bed ion exchange column with the fluidized bed column was determined by calculating the volume of resin bed required for each contacting equipment at similar feed conditions.

A full scale plant design for each contacting equipment at 6 and 0.6 g/L together with cost estimation are present in Appendix L. From the full scale plant design it was determined that the same volume of resin was required for both contacting equipment. Therefore in terms of volume requirement, no contacting equipment is advantageous over the other.

But from the cost estimation, the fixed bed column would be more advantageous in terms of capital cost investment because the fluidized bed would require a taller column to account for the extra head required for fluidization.

Therefore from the design and the cost estimation, although no difference is made from the volume requirement, in terms of capital cost investment the fixed bed would be more advantageous.

CHAPTER THIRTEEN: THE WAY FORWARD

Ion exchange technology encompasses the sciences of equilibria, kinetics, and ion chemistry. Understanding the finer points of ion exchange helps to determine whether or not ion exchange will be useful for a particular application.

The pilot plant that has been designed for this research can be used to do analysis on other precious metals on the periodic table using both types of contacting equipment. Thus enabling a comparison on both types of contacting equipment for other metals of interest.

This research examined cation exchange for copper recovery from a concentration range of 6 and 0.6 g/L. Therefore further studies can be done at a different concentration range for copper using the same resin.

Also the pilot plant is sufficient to evaluate both cation and anion exchange. Therefore studies can be carried out on anions such as iodide, bromide, and chloride. A selective resin can be chosen for the anion of interest and further studies can be conducted on the comparison of the types of contacting equipment.

Furthermore, new computer modelling capabilities are used to find new ion exchange substances with more specificity. The word specificity is a key word in ion exchange, as ion exchangers are created with one unique (specific) ion in mind, which will maximize efficiency, quality, and cost effectiveness.

I therefore recommend that future studies be conducted using ion exchange.

REFERENCES

[1] Allen D, *A Guide to the Economic Evaluation of Projects*, The Institute of Chemical Engineers, Warwick Printing Company Ltd, 1980, pgs 128-131

[2] Coulson and Richardson, *Particle Technology and Separation Processes*, Chemical Engineering Volume 2 (4th Edition), Pergamon Press, 1991, Chapter six and eighteen.

[3] Environex Short Course, *Ion Exchange*, Gencor Instructor: Dr. Robert Albright and Dr. E. Robert Becker, Magaliesberg Conference Centre, February 1996, Section E and Section F

[4] Fogler Scott H, *Elements of Chemical Reaction Engineering* (3th Edition), McGraw-Hill 1999, Chapter five

[5] Geankoplis, Christie J.; *Transport Processes and Unit Operations* (3rd Edition); Prentice-Hall 1993; pgs 697-704.

[6] Helfferich, F., *Ion Exchange*, McGraw-Hill, 1962 pgs 72-94,421-499

[7] Kunii, D. and O. Levenspiel, *Fluidization Engineering* (2nd Edition), Butterworth Heinemanne, 1923, pgs, 1, 68-92

[8] Marcus Y., Kertes A.S, *Ion exchange and solvent extraction of metal complexes*, Wiley-Interscience, 1969, pgs 239-359

[9] McCabe, W.L., J.C. Smith, and P. Harriott, *Unit Operations of Chemical Engineering* (6th Edition), McGraw-Hill, 2001, pgs 812-21.

[10] Meer and Piet Andries, *On counter-current fluidized ion exchange columns*, theses, Mintek Library

[11] Mitsubishi Chemical Corporation, *Dianion Manual of Ion Exchange Resins and Synthetic Adsorbents*, Separation Material Department 1995, pgs 133-148

[12] Rohm and Haas, *Ion Exchange Principles and Applications*, Encyclopaedia of Technical Chemistry, Weinhem W Germanly, 1989

[13] Sinnot, R.K., *Coulson and Richardson's Chemical Engineering: An Introduction to Chemical Engineering Design*, Chemical Engineering Volume 6, Revised 2nd Edition, Butterworth-Heinemann Ltd, 1996.

[14] Seader, J.D. and E.J. Henley, *Separation Process Principles*, John Wiley, 1998, pgs 841.

[15] Slater, M.J, *The principles of ion exchange technology*; Bilings and Sons Ltd, 1991; pgs 54-90,118-131,133-159

[16] Skoog Douglas A., West Donald M., Holler James F., *Fundamentals of Analytical Chemistry* (7th Edition), Saunders College Publishing1997, pgs 100-118,839,866

[17] Perry Robert H, Green Don W., *Perry's Chemical Engineering Handbook*, McGraw-Hill 1999, Section 16 pgs 1-66

Journals

[18] Amsterdam Elsevier, *Fluid particle technology analysis and application*, American Institute of Chemical Engineering, Symposium Series No. 30, 1994

[19] Boyce and Thomas D, *The relationship between ion exchange resin properties and uranium recovery in fluidized bed columns*, Society of Petroleum Engineers, 1980

[20] Celik M.S, Turan M, Ozdemir B and Sarikays H.Z, *Removal of ammonia by natural clay minerals using fixed and fluidized bed column reactors*, Water Science and Technology Supply Vol 1, 2001

[21] Craige W.M, *Ion Exchange Contacting Equipment*, Mintek SAIMM Hydrometallurgy School, Mintek publication.

[22] Ernest M, Whitley R, Zidu M, and Wang L, *Effects of mass action equilibria on Fixed-bed Multi component Ion-Exchange Dynamics*, Industrial Engineering Chemical Reserch Volume 36, Number 1, 1997

[23] Hamid A, *Fluidization and fluid particle systems: recent research and development*, American Institute of Chemical Engineering, 1998

[24] Howard L, Fluidization: *Fundamental Studies, Solid – fluid reactions and applications*, American Institute of Chemical Engineering, 1971

[25] Knowlton, *Fluidization and fluid-particle systems*: theories and application, American Institute of Chemical Engineering, 1983

[26] Moison R.L and O' Hern. H Jr, *Ion Exchange Kinetics*, Chemical Engineering Progress Symposium Series No 24, Volume 55, Adsorption, Ion exchange, and Dialysis

[27] Mozzttoi M, Neri B, Gelosa D, Kruglou A, and Morbidelli M, *Kinetics of Liquid-Phase EsterificationCatalyzed by Acidic Resins*, Industrial Engineering Chemical Reserch Volume 36, Number 1 1997

[28] Palmer G.R , Nichols I.L and Seidel D.C, *Uranium ion exchange from low grade acidic solutions in a fluidized system*, United States Bureau of Mines, Washington,1980

[29] Prudhomme P and Lucas B.H, *Construction and operation of a continuous ion exchange pilot plant using fluidized- bed columns*, Canada Centre for Mineral and Energy Technology, Ottawa, 1979

[30] Raghavan R, Bhatt CV, *Comparative study of Ion Exchange resins for application in Copper Bearing Process Solutions*, Hydrometallurgy, 1998

[31] Schmelze ER. And Hagerty, *Selective adsorption of toluene from n- Heptane by Silica gel*, Chemical Engineering Progress Symposium Series No 24, Volume 55, Adsorption, Ion exchange, and Dialysis.

[32] Slater M.J and Prudhomme, *Continuous Ion Exchange in Fluidized Beds*, Technical Bulletin 158, Canada Mines Branch, Ottawa

[33] Smith S. B, Hiltgen A and Juhola A, *Kinetics of Batch Adsorption of Di chlorophenol on Activated Carbon*, Chemical Engineering Progress Symposium Series No 24, Volume 55 , Adsorption, Ion exchange, and Dialysis.

[34] Traut D.E, *Design Requirements for uranium ion exchange from ammonium bicarbonate solutions in fluidized bed systems*, U.S Bureau Mines, Washington 1978

[35] Ulmanu M, *Removal of Copper from dilute and aqueous solutions by Adsorbent and Ion Exchange Materials*, Development and Application, Editor Greig.

[36] Wyethe .J.P, *Ion Exchange for Electrolyte Purification*, Mintek publication

[37] Wyethe .J.P, Kotze M.H, *Impurity removal by fixed bed ion exchange from cobalt electrolyte derived from the Kakanda tailings dump*, Mintek publication

[38] Vermeulem T and Hiester N, *Kinetic Relationships for Ion Exchange Process*, Chemical Engineering Progress Symposium Series No 24, Volume 55, Adsorption, Ion exchange, and Dialysis.

Internet

[39] http://www.cbu.edu/~rprice/lectures/adsorb.html
[40] http://www.ipt.ntnu.no/nordic/Papers/6th_Nordic_Reinik.pdf
[41] http://www.infras.gov.ab.ca/Content/doctype306/production/wte.pdf
[42] http://www.rpi.edu/dept/chem-eng/Biotech-Environ/IONEX/resin.html
[43] http://www.lplc.com/misc/ionex.htm
[44] http://www.lenntech.com/ion_exchanger.htm
[45] http://www.frtr.gov/matrix2/section4/4-49.html
[46] http://www.resintech.com/content/TechArticles/Essential%20of%20Ion%20Exchange.pdf
[47] http://www.thefreedictionary.com/ion%20exchange
[48] http://www.chemlin.de/chemistry/ion_exchange.htm
[49] http://www.swbic.org/education/env-engr/ion_exchange /.html
[50] http://www.isr.umd.edu/CELS/research/watersim/guidance/educational%20module/ion_echange.htm
[51] http://www.iupac.org/publications/analytical_compendium/cha09sec30.pdf

[52] http://www.oceta.ca/profiles/met-tech/metals.html

[53] http;//www.dartmouth.edu/~chemlab/chem3-5/ionx1/fulltext/intro.html.

[54] http://engine.umich.edu/class/che360/coursepack/ch6-ionexchange.pdf

[55] http://ethesis.helsinki.fi/julkaisut/mat/kemia

[56] http://www.fh-nuernberg.de/tc/lab-aust/lim/wasser2_ionexchange.pdf

[57] http://www.chem.csustan.edu/chem1102/bluepennies.html

[58] http://www.eng.nus.edu.sg/ChemEngstaff/hidajat.html

[59] http://www.remco.com/#33

[60] http://www.serva.de/products/knowledge/071110.shtml

[61] http:// www.bayer.com/en/index.php#

[62] http:// www.dow.com/liquidsep/design/#sizing

[63] http://www.separationprocesses.com/Adsorption/AD_Chp04c.htm

[64] http://www.systemsaver.com

[65] http://www.wma-minelife.com/ uranium/insitu/items014.htm

[66] http://www.unm.edu/coursinf/esp462/page2.htm

[67] http://www.gocctech.com/default2/resin.htm

[68] http://www.rpi.edu/dept/chem-eng/Biotech-Environ/Adsorb/langmuir.htm

[69] http:wwwbrr.cr.usgs.gov/projects/GW_Unsat/vs2di/hlp/solute/Freundlich.html

Lecture Notes

[70] Lecture Notes, Solid fluid Systems, Professor Carsky (contact number 260 7668)

Appendices

APPENDIX A

A1: GLOSSARY OF TERMS A1-1

APPENDIX A1: GLOSSARY OF TERMS

Acidity: The concentration of hydrogen ion present in solution.

Alkalinity: The total anions present in the solution representing the carbonate and bicarbonate salts, which will react with water to produce hydroxyl groups.

Anion: A negatively charged ion.

Attrition: The physical rubbing of particles in the resin bed causing wear and the loss of ion exchange resin.

Backwash: The up flow passage of water through a resin bed and reclassify the bed after exhaustion.

Bed: The ion exchange resin contained in the bed.

Bed Depth: The height of the ion exchange resin in the vessel after regeneration and settling.

Bed Expansion: The lifting and the separation of the resin beads during backwash.

Breakthrough: the point at which the ions begin to appear in the effluent from the ion exchange bed, signalling exhaustion of the bed and regeneration is required.

Capacity: The ability of an ion exchange material to absorb ions. Usually expressed in grams per litre or mill equivalents per millilitre.

Channelling: The flow of the influent solution along the path of least resistance through channels in the exchange bed causing the solution to pass without contacting the active groups of the bed.

Density: The weight of a given volume of exchange material.

Effluent: The solution that comes out of the ion exchange column.

Exhaustion: The depletion of available ions on the ion exchange resin and corresponding reduction in the quality of the effluent solution indicating the need for regeneration.

Freeboard: The void area above the ion exchange resin in the column for expansion of the resin during backwash.

Influent: The solution flowing into the ion exchange column.

Leakage: The passage of influent ions through an ion exchange resin bed.

pH: The measurement of the acidity of a solution where 1 is very acidic, 7 is neutral and 14 is very basic.

Regenerant: The solution of the concentrated ions used to restore the activity of the ion exchange media, typically acids or brine for cation resins and alkaline solutions for anion resins.

Regeneration: The process of restoring ion exchange activity of the bed by passing the regenerant solution through the exchange bed thus replacing the exhausting ions with ions to be used in the treatment process.

Rinse: The flushing of the regenerant solution from the ion exchange bed following regeneration and prior to placing the unit back into service.

Swelling: The expansion of the ion exchange bead when the reactive groups on the bead are converted from one form to another.

Throughput: The amount of solution treated prior to the exhaustion of the ion exchange material.

APPENDIX B

B1: COST ESTIMATION FOR ION EXCHANGE PROJECT B1-1

APPENDIX B1: COST ESTIMATION FOR ION EXCHANGE PROJECT

	Rands	**Rands**	**Rands**
1. Capital Cost			**9725**
1.1 Cost of Equipment (Fixed and Fluidized Ion Exchange Columns)		8600	
1.1.1 Two Perspex Columns			
1.1.1.1 Fixed Bed Column (2.4 cm i.d and 1m long)	200		
1.1.1.2 Fluidized Bed Column (2.4 cm i.d and 2 m long)	400		
1.1.2 Pump	6000		
1.1.3 Tank	500		
1.1.4 Valves	300		
1.1.5 Plastic pipes + domes + metallic sieve	1200		
1.2 Quantity of Resin (TP 207 Iminodiacetic Acid: 1L @ R45)		1125	
2. Maintenance Costs			**8000**
2.1 Reagent (Cupric Sulphate : 500g @ R600)		3000	
2.2 Regeneration of resin with H_2SO_4 (2.5L @ R390)		5000	
TOTAL COST			**17725**

APPENDIX C: RESIN PROPERTIES

C1: PARTICLE SIZE DISTRIBUTION C1-1

C2: CALCULATION OF VOID VOLUME C2-1

C3: CALCULATION OF MINIMUM FLUIDIZATION AND SETTLING VELOCITY FOR TP 207 C3-1

C4: CALCULATION OF WATER RETENTION CAPACITY C4-1

C5: CALCULATIONS OF THE EFFECT OF SWELLING ON THE TP 207 RESIN C5-1

APPENDIX C1: PARTICLE SIZE DISTRIBUTION

TABLE C1.1: Initial Conditions for each Run

Resin [H^+ form]	TP 207
Tapped Volume	10 mL
Shaking Time	10 min
pH	2.18

TABLE C1.2: Cumulative Graph Data for Run 1

Screen Size [μm]	Volume mL	Volume fraction	Cumulative oversize	Cumulative undersize
850	2.9	0.29145729	0.291457	1
710	5	0.50251256	0.79396956	0.70854271
600	2	0.20100503	0.99497459	0.20603015
500	0.05	0.00502513	0.99999971	0.0050125
355	0	0	NA	0
Total	**9.95**			

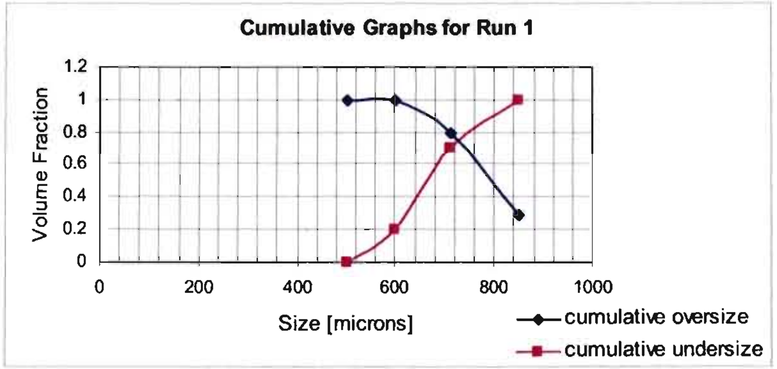
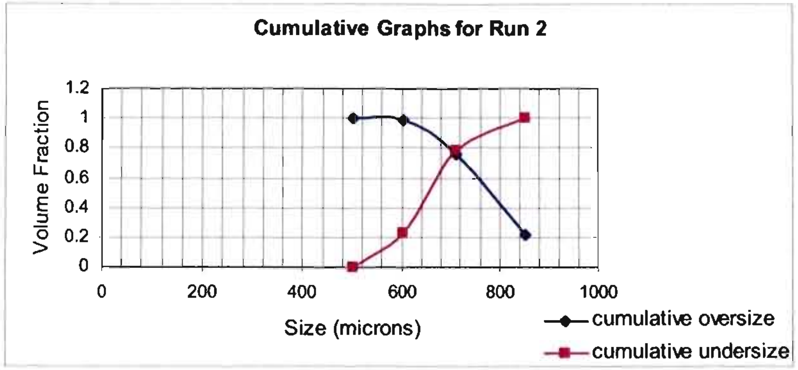


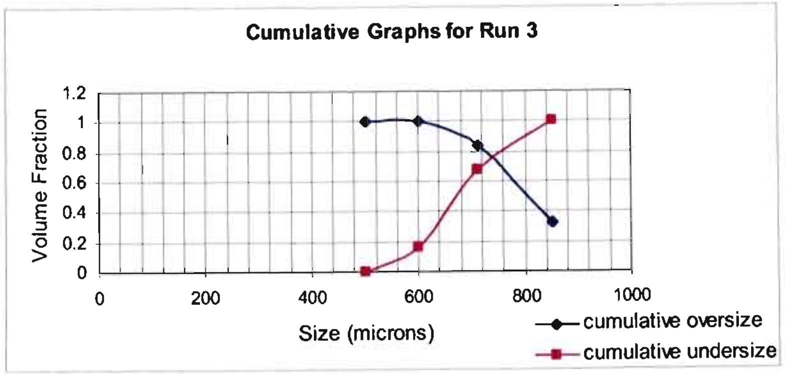
Figure C1.1: Cumulative Graph for Run 1

Average Particle Size for Run 1 was determined to be 720 microns.

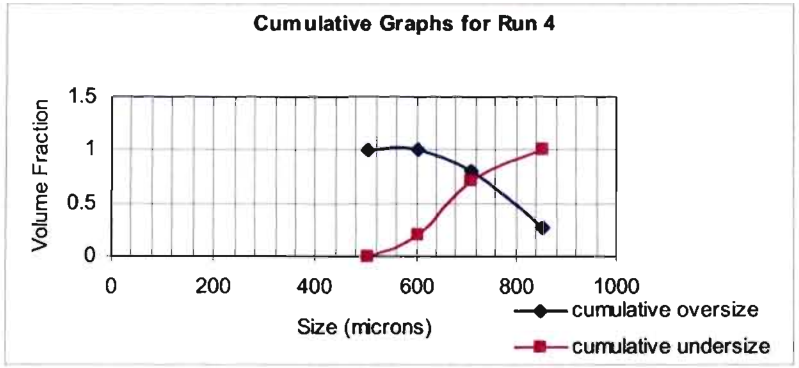
The above procedure was repeated seven more times and the final results are summarized in the graphs below.



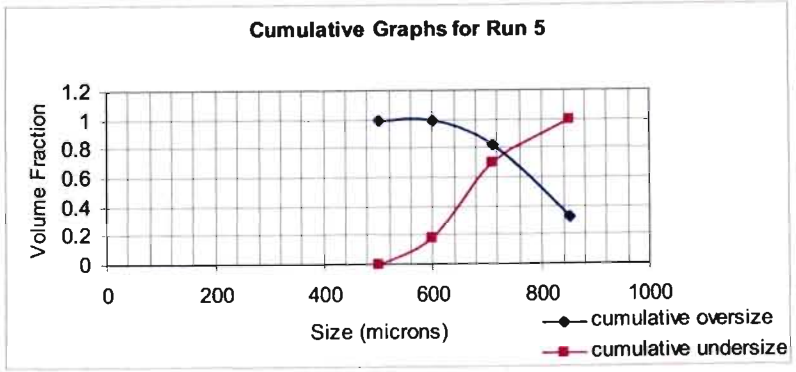
Run 2



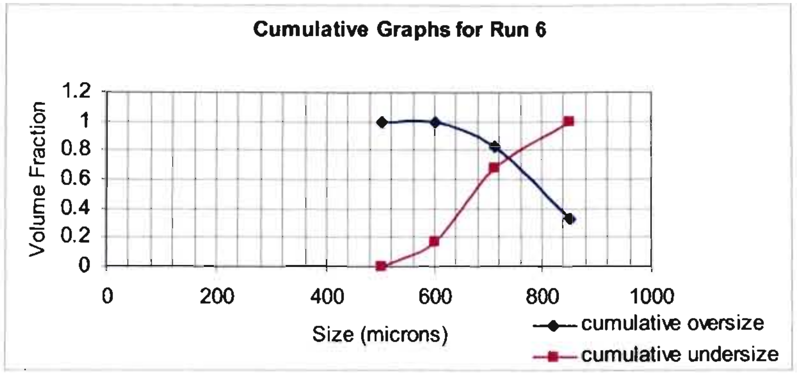
Run 3



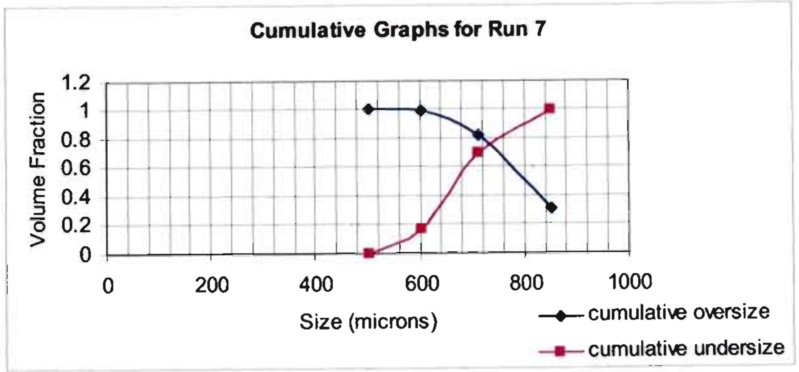
Run 4



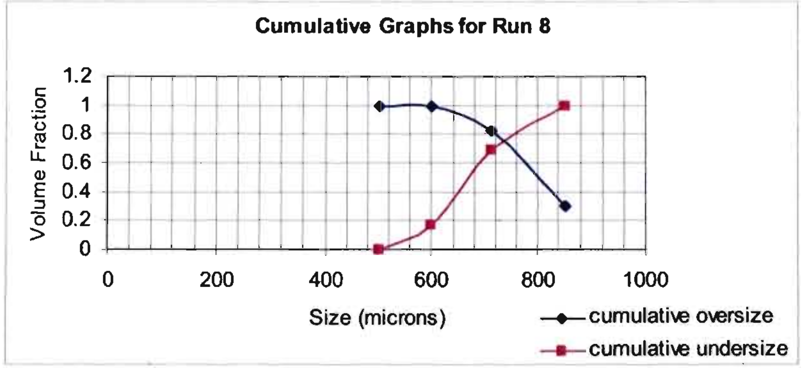
Run 5



Run 6



Run 7



Run 8

Figure C1.2: Summary of Cumulative Graphs for Runs 2-8

TABLE C1.3: Summary of Particle Size Analysis

Run Number	Average per Run [μm]
1	720
2	716
3	740
4	725
5	730
6	735
7	730
8	730
Average particle size	**728.25**
Maximum	**740**
Minimum	**716**
Standard Deviation	**7.759786448**

The finial average particle size was experimentally determined to be 728.25 microns.

APPENDIX C2: DETERMINATION OF VOID VOLUME

C2.1 **Sample 1**

Height of packed column (H_p) = 200 mm

Bed volume = (πr^2) * H_p

= $\pi(2.4/2)^2$ * 20.0

=90.48 cm^3

Volume of water drained = 25 mL

$$\text{Void volume\%} = \left(\frac{V'}{V}\right) * 100$$

$$= \left(\frac{25}{90.48}\right) * 100$$

= 27.63 %

C2.2 **Sample 2**

Height of packed column (H_p) = 208 mm

Bed volume = (πr^2) * H_p

= $\pi(2.4/2)^2$ * 20.8

=94.1 cm^3

Volume of water drained = 29 mL

$$\text{Void volume\%} = \left(\frac{V'}{V}\right) * 100$$

$$= \left(\frac{29}{94.1}\right) * 100$$

= 30.82 %

C2.3 **Average**

$$\text{Average \%} = \left[\frac{\text{sample 1} + \text{sample 2}}{2}\right]$$ ....**(Equation C2.1)**

The average void volume was calculated by Equation C2.1

$$\text{Average \%} = \left[\frac{27.63 + 30.82}{2}\right]$$

= 29.23 %

The void volume of resin packed in a column was experimentally determined to be 29.23 %.

APPENDIX C3: CALCULATION OF MINIMUM FLUIDIZATION AND TERMINAL VELOCITY FOR TP 207 RESIN

Kozeny-Carman Equation for small particles:

$$u_f = \frac{(d_p)^2(\rho_s - \rho_f)g}{150\mu} * \frac{\varepsilon_{mf}{}^3\Phi_s{}^2}{(1-\varepsilon_{mf})} \quad , \text{ Re} < 10\text{..........}\textbf{(Equation C3.1)}$$

(Equation C3.1 is reproduced from Chapter Five for convenience).

C3.1 **Parameters for Fluidization**

ε_{mf}=0.29

Particle shape: For spherical particles Φ_s=1

Gravitational acceleration: $g = 9.81$ m/s^2

Liquid viscosity: $\mu = 0.00089$ Pa.s

Liquid density ρ_f: = 1000 kg/m^3

Particle diameter: d_p = 0.728 mm

Minimum fluidization: u_f

Bulk density of resin = 800 g/L =800 kg/m^3 (Shipping weight: density including voids)

Therefore density of resin is:

$$\text{Density} = \frac{\text{Bulk density}}{(1-\varepsilon)} \text{ ....}\textbf{(Equation C3.2)}$$

$$= \frac{800}{(1-0.29)}$$

$$=1126.76 \text{ kg/m}^3$$

C3.2 **Minimum Fluidization Velocity:**

Substituting in the Kozeny-Carman Equation **(Equation C3.2)**.

$$u_f = \frac{(d_p)^2(\rho_s - \rho_f)g}{150\mu} * \frac{\varepsilon_{mf}{}^3\Phi_s{}^2}{(1-\varepsilon_{mf})}$$

$$= \frac{(0.728 * 10^{-3})^2(1126.76 - 1000\text{ kg/m3}) * 9.81}{150 * (0.00089)} * \frac{0.29^3 1^2}{(1-0.29)}$$

$= 1.7 * 10^{-4}$ m/s

$= 0.61$ m/hr $= 0.017$ cm/s

Area of column $= \pi r^2 = \pi(2.4/2)^2 = 4.52389\ \text{cm}^2$

$= 4.5 * 10^{-4}\ \text{m}^2$

Volumetric flow $= u_f *$ Area $= (0.61\ \text{m/hr}) * 4.5*10^{-4}\ \text{m}^2$

$=2.7 * 10^{-4}\ \text{m}^3/\text{hr}$

$=0.27$ L/hr

$$\text{Re}_{\text{min}} = \frac{(0.728*10^{-3})(1.7*10^{-4})(1000)}{(1-0.29)(0.00089)}$$

$= 0.196 \qquad \text{Re} < 10$

C3.3 **Terminal Velocity**

$$u_T = \frac{(d_p)^2(\rho_s - \rho_f)g}{18\mu} = \frac{(0.728*10^{-3})^2(1126.76-1000)*9.81}{18(0.00089)}$$

$=0.041$ m/s $= 148$ m/hr $= 4.1$ cm/s

Volumetric flow at terminal velocity $= u_T *$ Area $= (148\ \text{m/hr}) * 4.5 * 10^{-4}\ \text{m}^2$

$=6.7 * 10^{-2}\ \text{m}^3/\text{hr}$

$= 67$ L/hr

Operate fluidized bed below 67 L/hr to avoid particles been carried out at the exit with the fluid that is leaving.

Therefore the specification for the pump is:
Speed in the range of [0.017 cm/s - 4.1 cm/s].

APPENDIX C4: DETERMINATION OF WATER RETENTION CAPACITY

C4.1 **Sample 1**

Mass of dish 1 = 59.0734 g

Mass of dish 1 + Wet resin = 66.5109 g

Mass of dish 1 + Dry resin = 63.1929 g

$$WRC = \left[\frac{M_W - M_D}{M_w}\right] * 100 \quad \text{.............................} \quad \textbf{(Equation C4.1)}$$

M_W = (Mass of dish 1 + Wet rein) – Mass of dish 1

= 66.5109g – 59.0734

= 7.4375g

M_D = (Mass of dish 1 + Dry resin) – Mass of dish 1

= 63.1929g – 59.0734g

= 4.1195g

Substituting the appropriate values into Equation C4.1.

$$WRC = \left[\frac{7.4375 - 4.1195}{7.4375}\right] * 100$$

= 44.6117 %

C4.2 **Sample 2**

Mass of dish 2 = 59.5114 g

Mass of dish 2 + Wet resin = 67.2262 g

Mass of dish 2 + Dry resin = 63.74892 g

M_W = (Mass of dish 2 + Wet rein) – Mass of dish 2

= 67.2262 g – 59.5114 g

= 7.7148 g

M_D = (Mass of dish 2 + Dry resin) – Mass of dish 2

= 63.74892 g – 59.5114 g

= 4.23752 g

Substituting the appropriate values into Equation C4.1.

$$\text{WRC} = \left[\frac{7.7148 - 4.23752}{7.7148}\right] * 100$$

$$= 45.072\ \%$$

C4.3 **Average**

$$\text{Average}\ \% = \left[\frac{\text{sample 1} + \text{sample 2}}{2}\right] \quad \text{..............}\textbf{(Equation C4.2)}$$

The average water retention capacity is calculated using Equation C4.2.

$$\text{Average}\ \% = \left[\frac{44.61176 + 45.072}{2}\right]$$

$$= 44.841188\ \%$$

The water retention capacity of the Lewatit TP 207 resin was experimentally determined to be 44.841 %.

APPENDIX C5: DETERMINATION OF THE EFFECT OF SWELLING FOR THE TP 207 RESIN

C5.1 Determination of Swelling for TP 207 resin from the Hydrogen to the Di and Mono Sodium forms

From Section 10.8, the following were determined:

- Conversion into the Hydrogen form

Volume of resin [H^+ form] = 1000 mL.
pH of resin [H^+ form] =2.5

- Conversion into the Di sodium form

Volume of resin [Di Na^+ form] = 1349 mL
pH of resin [Di Na^+ form] =11.50

- Conversion into the Mono sodium form

Volume of resin [Mono Na^+ form] = 1250 mL
pH of resin [Mono Na^+ form] =6.71

The resin in the Hydrogen form was used as a reference for the other two forms.

For the Di Sodium form percentage swelling was determined by Equation C5.1:

$$\text{Percentage Swelling} = \frac{V_{\text{di-sodium}} - V_{H^+}}{V_{H^+}} * 100 \text{ .... } \textbf{(Equation C5.1)}$$

$$= \frac{1349 - 1000}{1000} * 100$$

$$= 34.9\ \%$$

For the Mono Sodium form percentage swelling was determined by Equation C5.2:

$$\text{Percentage Swelling} = \frac{V_{\text{mono-sodium}} - V_{H^+}}{V_{H^+}} * 100 \text{ .... } \textbf{(Equation C5.2)}$$

$$= \frac{1250 - 1000}{1000} * 100$$

$$= 25\ \%$$

APPENDIX D: PH TESTS

D1: pH TESTS CALCULATIONS D1-1

APPENDIX D1: pH TEST CALCULATIONS

The following symbols have been reproduced from List of Symbols and Abbreviations for convenience. Please note that the following symbols will be used for all calculations thus forward. The meanings of these symbols have been discussed in Chapter 3, Section 3.2, titled Ion Exchange: Material Balance. The same symbols will follow in the equilibrium and kinetic study.

Initial Cu^{2+} Concentration : Co
Initial Cu^{2+} Volume : Vo
Initial Volume of resin : Vro

Volume of Cu^{2+} solution concentration after equilibrium : Vs
Volume of resin loaded with copper : Vc
Volume of eluted resin: Ve
Concentration of Cu^{2+} after equilibrium (solution): Ce
Volume of Cu^{2+} solution from elution : Vcu
Concentration of Cu^{2+} from elution : Cel

Amount of Copper loaded onto resin from adsorption : qe
Amount of Copper loaded onto resin from elution : Qe

TABLE D1.1: Results obtained from pH Test

Readings	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Set at	pH End
1	500	10.1	2.07	1	0.92
2	500	10	2.07	1.5	1.5
3	500	10	2.07	2	1.98
4	500	10	2.07	2.5	2.56
5	500	10	2.07	3	3.08
6	500	10	2.07	4	4.45

TABLE D1.1: ...continued...

Readings	NaOH Used [mL]	H_2SO_4 Used [mL]	Solution [Cu] remaining	Volume of resin with [Cu]	Volume of Eluted resin	pH resin	Conc. of Solution [g/L]	Volume of [Cu] from eluate	Conc. of Elution
1	14	29.5	434	10.5	10	2.2	4.9	101	2.172
2	3.4	16	515	10.5	10.2	2	4.84	100	3.83
3	7	3.5	435	10.4	10	2.07	4.6	100	4.203
4	15	-	510	10.6	10.2	2.1	4.51	100	5.7
5	16	-	445	10.2	10	2.04	4.4	100.9	6.027
6	14	-	460	10.6	10	2.06	4.3	100	5.9

TABLE D1.2: Calculations obtained from pH Test

Readings	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention [mL]	NaOH Used [mL]	H_2SO_4 Used [mL]
1	500	5.8	2.9	10.1	4.5248	14	29.5
2	500	5.8	2.9	10	4.48	3.4	16
3	500	5.8	2.9	10	4.48	7	3.5
4	500	5.8	2.9	10	4.48	15	-
5	500	5.8	2.9	10	4.48	16	-
6	500	5.8	2.9	10	4.48	14	-

Readings	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
1	434	10.5	10	4.9	101	2.172
2	515	10.5	10.2	4.84	100	3.83
3	435	10.4	10	4.6	100	4.203
4	510	10.6	10.2	4.51	100	5.7
5	445	10.2	10	4.4	100.9	6.027
6	460	10.6	10	4.3	100	5.9

Table D1.2: ...continued...

Readings	Mass of solution[g]	Mass of Eluates [g]	Qe: Elution [g/L]	Mass of Copper on resin	Accountability	Volume Balance	Volume out
1	2.47217152	0.219372	21.9372	0.219372	92.81184552	543.5	480
2	2.4416832	0.383	37.54902	0.383	97.40286897	519.4	500
3	2.320608	0.4203	42.03	0.4203	94.51406897	510.5	492
4	2.2752048	0.57	55.882353	0.57	98.11051034	515	510
5	2.219712	0.6081243	60.81243	0.6081243	97.51159655	516	498
6	2.169264	0.59	59	0.59	95.14703448	514	470

Readings	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption	Qe: Elution [g/L]	pH End
1	2.9	2.47217152	10.1	42.3592554	21.9372	0.92
2	2.9	2.4416832	10	45.83168	37.5490196	1.5
3	2.9	2.320608	10	57.9392	42.03	1.98
4	2.9	2.2752048	10	62.47952	55.8823529	2.56
5	2.9	2.219712	10	68.0288	60.81243	3.08
6	2.9	2.169264	10	73.0736	59	4.45

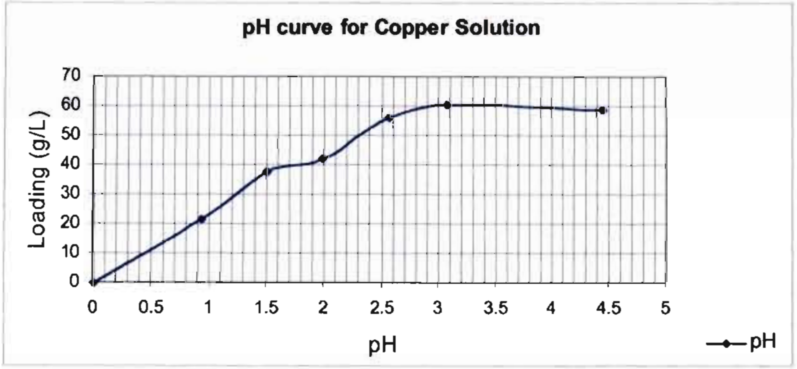


Figure D1.1: pH Curve for Copper Solution

APPENDIX E: EQUILIBRIUM TESTS

E1: EQUILIBRIUM TEST FOR pH SET AT 3 E1-1

E2: EQUILIBRIUM TEST FOR pH SET AT 2.5 E2-1

E3: EQUILIBRIUM TEST FOR pH SET AT 2 E3-1

E4: EQUILIBRIUM TEST FOR pH SET AT 1.5 E4-1

E5: SUMMARY OF EQUILIBRIUM PLOTS E5-1

APPENDIX E1: EQUILIBRIUM TEST FOR PH SET AT 3

TABLE E1.1: Results from Equilibrium Test at pH 3

Readings	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
1	50	10	2.1	1.6	3.16
2	80	10	2.1	1.72	3.12
3	150	10	2.1	1.92	3.09
4	500	10	2.1	2.16	3.03
5	700	10	2.1	2.26	3.03
6	1000	10	2.1	2.32	3.03

Readings	NaOH Used [mL]	Solution [Cu] remaining	Volume of resin with [Cu]	Volume of Eluted resin	pH of Resin	Conc. of Solution [g/L]	Volume of [Cu] from eluates	Conc. from Elution
1	11.6	59	10.5	10	2.2	0.0322	103	2.85
2	21.4	96	10.5	10	1.9	0.0281	101	4.31
3	10.8	146	10.8	10	1.9	1.805	100	6.06
4	14.5	510	11	10.2	2.3	4.34	100	6.23
5	12.6	700	11	10.2	1.9	4.84	102.6	6.06
6	16	1000	11	10.2	1.9	5.07	100	6.2

TABLE E1.2: Calculations from Equilibrium Test at pH 3

Readings	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	Volume NaOH [mL]
1	50	5.8	0.29	10	4.48	11.6
2	80	5.8	0.464	10	4.48	21.4
3	150	5.8	0.87	10	4.48	10.8
4	500	5.8	2.9	10	4.48	14.5
5	700	5.8	4.06	10	4.48	12.6
6	1000	5.8	5.8	10	4.48	16

Readings	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
1	59	10.5	10	0.0322	103	2.85
2	96	10.5	10	0.0281	101	4.31
3	146	10.8	10	1.805	100	6.06
4	510	11	10.2	4.34	100	6.23
5	700	11	10.2	4.84	102.6	6.06
6	1000	11	10.2	5.07	100	6.2

Readings	Mass of solution[g]	Mass of Eluates [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance	Volume Measured
1	0.001754256	0.29355	29.355	0.29355	101.8290538	61.6	59
2	0.002373888	0.43531	43.531	0.43531	94.32842414	101.4	96
3	0.2788364	0.606	60.6	0.606	101.7053333	160.8	146
4	2.1894432	0.623	61.0784314	0.623	96.9808	514.5	510
5	3.4096832	0.621756	60.9564706	0.621756	99.29653202	712.6	700
6	5.0927136	0.62	60.7843137	0.62	98.49506207	1016	1000

Readings	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]	Qe: Elution [g/L]	Ce [g/L]	$r_{H+}^{Cu\ 2+}$	α
1	0.29	0.001754256	10	28.8245744	29.355	0.0322	0.006033	911.646
2	0.464	0.002373888	10	46.1626112	43.531	0.0281	0.001962	1549.146
3	0.87	0.2788364	10	59.11636	60.6	1.805	0.003567	33.57341
4	2.9	2.1894432	10	71.05568	61.0784314	4.34	0	14.07337
5	4.06	3.4096832	10	65.03168	60.9564706	4.84	0.010087	12.59431
6	5.8	5.0927136	10	70.72864	60.7843137	5.07	0.033606	11.98902

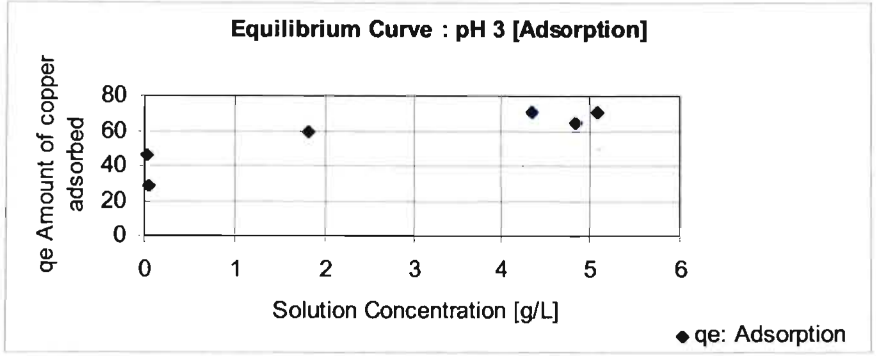


Figure E1.1: Equilibrium Curve at pH 3 for Adsorption

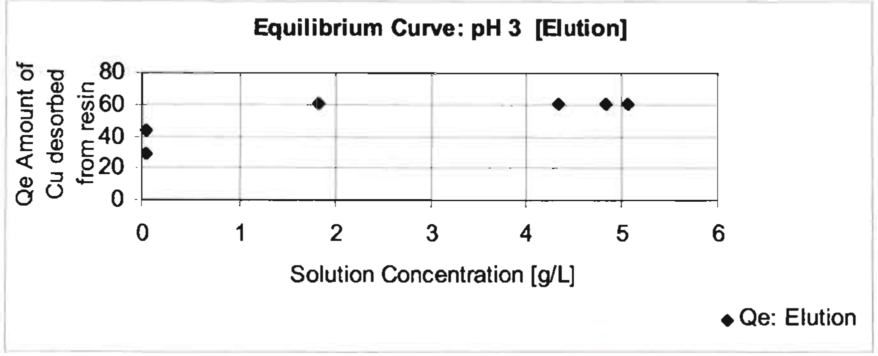


Figure E1.2: Equilibrium Curve at pH 3 for Elution

TABLE E1.3: The Langmuir and Freundlich Adsorption Isotherms

Readings	Ce [g/L]	Qe: Elution [g/L]	log Qe	log Ce	1/Qe	1/Ce
1	0.0322	29.355	1.4676821	-1.4921441	0.03406575	31.0559006
2	0.0281	43.531	1.6387986	-1.5512937	0.02297213	35.5871886
3	1.805	60.6	1.7824726	0.2564772	0.01650165	0.55401662
4	4.34	61.0784314	1.7858879	0.6374897	0.01637239	0.23041475
5	4.84	60.9564706	1.7850198	0.6848454	0.01640515	0.20661157
6	5.07	60.7843137	1.7837915	0.705008	0.01645161	0.19723866

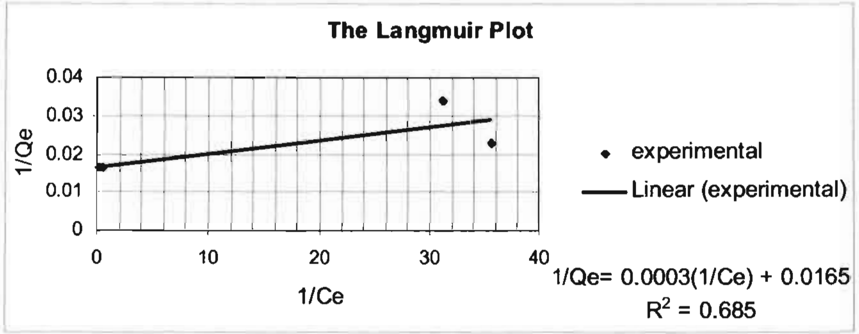


Figure E1.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 3

TABLE E1.4: Parameters for the Langmuir Plot

a	60.606
b	55
r	0.8276

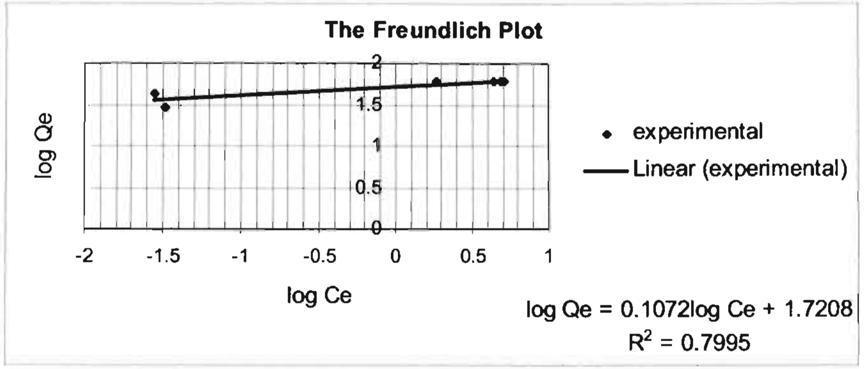


Figure E1.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 3

TABLE E1.5: Parameters for the Freundlich Plot

1/n	0.1072
K_f	52.578
r	0.89

TABLE E1.6: Tabulation of Results obtained from Langmuir and Freundlich Equation

Readings	Ce [g/L]	Langmuir Qe	Freundlich Qe
1	0.0322	38.3858977	36.37861926
2	0.0281	36.4657856	35.85133891
3	1.805	59.4616411	56.01423623
4	4.34	59.8100343	61.537941
5	4.84	59.8358223	62.26148643
6	5.07	59.8459829	62.5721266

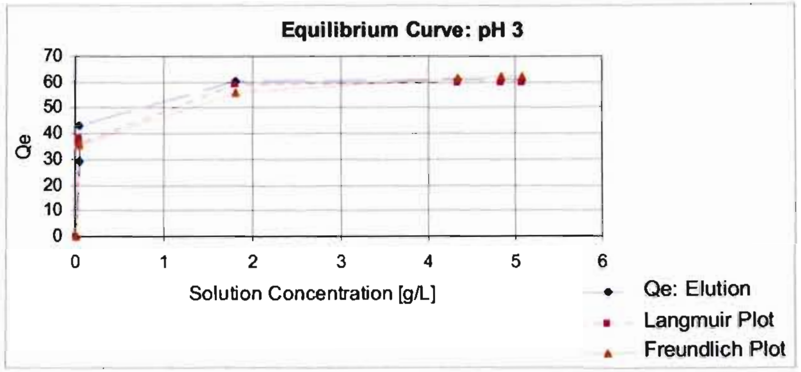


Figure E1.5: Comparison of Experimental and Predicted Results

APPENDIX E2: EQUILIBRIUM TEST FOR PH SET AT 2.5

TABLE E2.1: Results from Equilibrium Test at pH 2.5

Readings	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
1	50	10	2.1	1.89	2.57
2	80	10	2.1	1.89	2.54
3	150	10	2.1	1.93	2.59
4	500	10	2.1	2.07	2.56
5	700	10	2.1	2.31	2.52
6	1000	10	2.1	2.19	2.53

Readings	NaOH[mL] used	Solution [Cu] remaining	Volume of resin with Cu	Volume of Eluted resin	pH resin	Con of Solution[g/L]	Volume of Cu from eluates	Con of Elution
1	5	49	10.5	10	2	0.0674	100	2.8
2	3.4	84	10.5	9.8	1.9	0.218	100	4.4
3	6	146	10.5	10	2	1.83	102.6	5.3
4	15	510	10.6	10.2	2.1	4.51	100	5.7
5	10	694	10.6	10.2	2.1	4.9	100	5.83
6	10	1000	10.5	10.1	2.2	5.17	100	5.65

TABLE E2.2: Calculations from Equilibrium Test at pH 2.5

Readings	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	Volume NaOH [mL]
1	50	5.8	0.29	10	4.48	5
2	80	5.8	0.464	10	4.48	3.4
3	150	5.8	0.87	10	4.48	6
4	500	5.8	2.9	10	4.48	15
5	700	5.8	4.06	10	4.48	10
6	1000	5.8	5.8	10	4.48	10

Readings	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
1	49	10.5	10	0.0674	100	2.8
2	84	10.5	9.8	0.218	100	4.4
3	146	10.5	10	1.83	102.6	5.3
4	510	10.6	10.2	4.51	100	5.7
5	694	10.6	10.2	4.9	100	5.83
6	1000	10.5	10.1	5.17	100	5.65

Readings	Mass of solution[g]	Mass of Eluates [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance	Volume Measured
1	0.003671952	0.28	28	0.28	97.81791448	55	49
2	0.01841664	0.44	44.8979592	0.44	98.79668966	83.4	84
3	0.2826984	0.54378	54.378	0.54378	94.99751724	156	146
4	2.2752048	0.57	55.8823529	0.57	98.11051034	515	510
5	3.451952	0.583	57.1568627	0.583	99.38305419	710	700
6	5.1931616	0.565	55.9405941	0.565	99.27864828	1010	1000

Readings	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]	Qe: Elution [g/L]	Ce [g/L]	$r_{H+}^{Cu\ 2+}$	α
1	0.29	0.003671952	10	28.6328048	28	0.0674	0.456455	415.4303
2	0.464	0.01841664	10	44.558336	44.8979592	0.218	0.438542	205.9539
3	0.87	0.2826984	10	58.73016	54.378	1.83	0.895202	29.71475
4	2.9	2.2752048	10	62.47952	55.8823529	4.51	0.540963	12.39077
5	4.06	3.451952	10	60.8048	57.1568627	4.9	0.302573	11.66467
6	5.8	5.1931616	10	60.68384	55.9405941	5.17	0.299147	10.82023

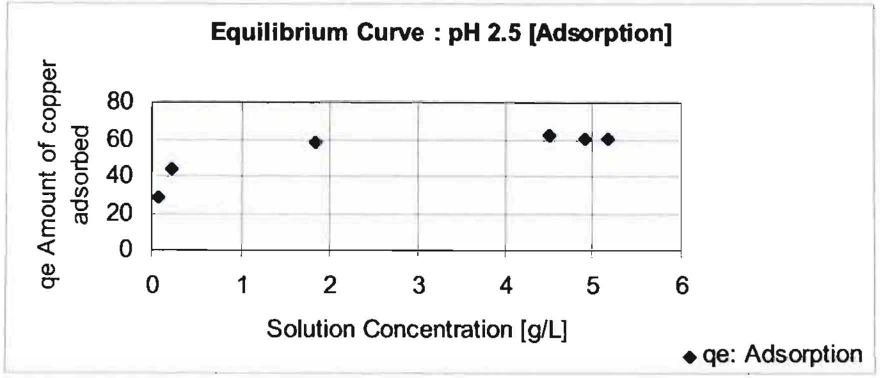


Figure E2.1: Equilibrium Curve at pH 2.5 for Adsorption

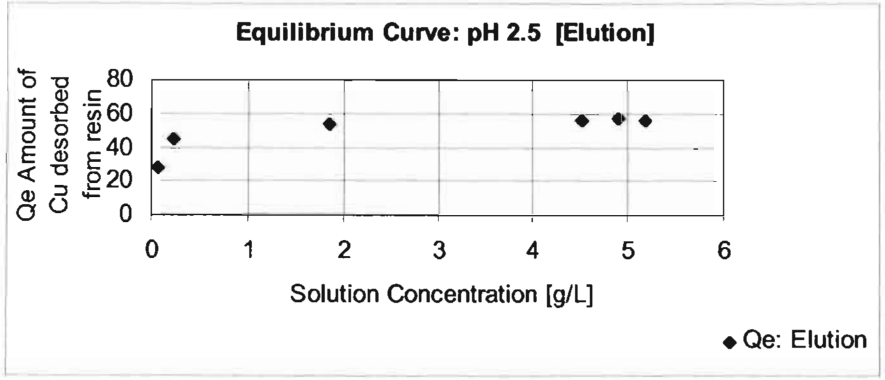


Figure E2.2: Equilibrium Curve at pH 2.5 for Elution

TABLE E2.3: The Langmuir and Freundlich Adsorption Isotherms

Readings	Ce [g/L]	Qe: Elution [g/L]	log Qe	log Ce	1/Qe	1/Ce
1	0.0674	28	1.447158	-1.1713401	0.03571429	14.8367953
2	0.218	44.8979592	1.6522266	-0.6615435	0.02227273	4.58715596
3	1.83	54.378	1.7354232	0.2624511	0.01838979	0.54644809
4	4.51	55.8823529	1.7472747	0.6541765	0.01789474	0.22172949
5	4.9	57.1568627	1.7570684	0.6901961	0.01749571	0.20408163
6	5.17	55.9405941	1.7477271	0.7134905	0.01787611	0.1934236

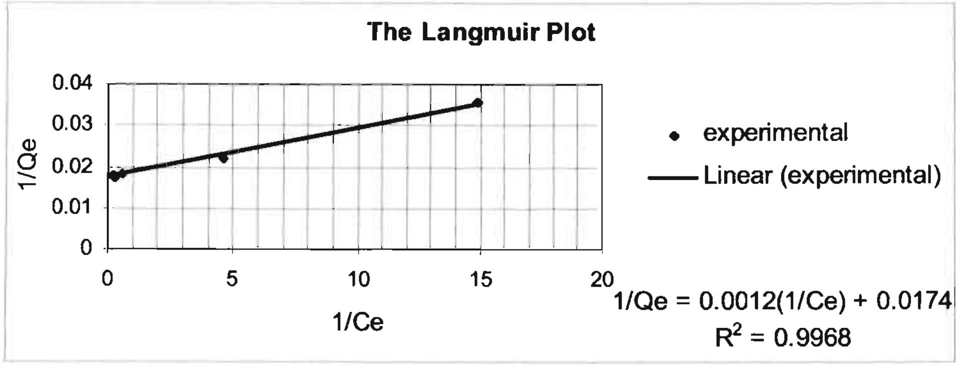


Figure E2.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 2.5

TABLE E2.4: Parameters for the Langmuir Plot

a	57.47
b	14.5
r	0.998

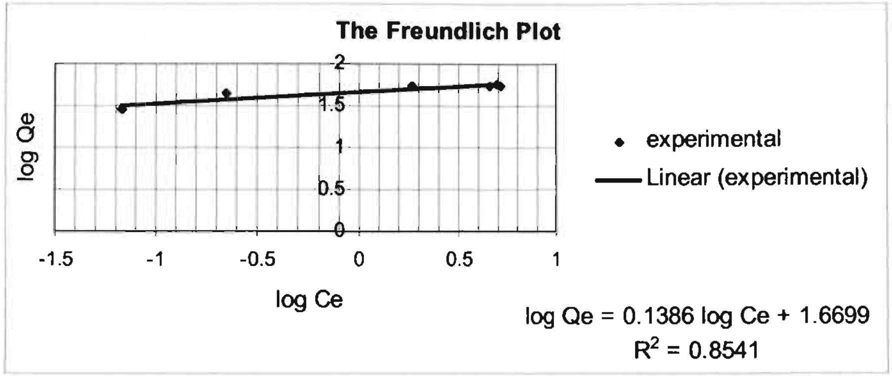


Figure E2.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 2.5

TABLE E2.5: Parameters for the Freundlich Plot

1/n	0.1386
Kf	46.76
r	0.92

TABLE E2.6: Tabulation of Results obtained from Langmuir and Freundlich Equation

Readings	Ce [g/L]	Langmuir Qe	Freundlich Qe
1	0.0674	28.4051135	32.17560814
2	0.218	43.6584162	37.86034547
3	1.83	55.3828382	50.84523086
4	4.51	56.6044228	57.61603544
5	4.9	56.6723595	58.28216542
6	5.17	56.7134674	58.71705861

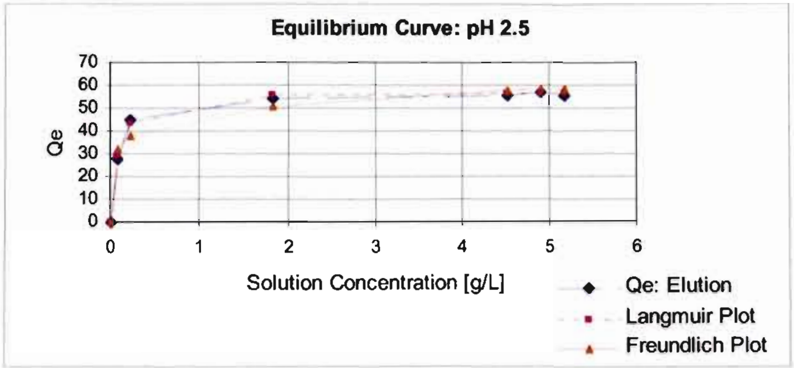


Figure E2.4: Comparison of Experimental and Predicted Results

APPENDIX E3: EQUILIBRIUM TEST FOR PH SET AT 2

TABLE E3.1: Results from Equilibrium Test at pH 2

Readings	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
1	50	10	2.1	2.07	2
2	80	10	2.1	2.13	2
3	150	10	2.1	2.1	2.01
4	500	10	2.1	2.33	2
5	700	10	2.1	2.35	2
6	1000	10	2.1	2.2	2

Readings	NaOH Used [mL]	H_2SO_4 Used [mL]	Solution [Cu] remaining	Volume of resin loaded Cu	Volume of Eluted resin	pH resin	Con of Solution [g/L]	Volume of Cu from eluates	Concentration of Elution
1	5	10.6	59	10.5	10.2	2	0.225	103.5	2.56
2	2.2	3.6	84	10.5	10	1.9	0.503	100	4
3	5.4	0	150	10.5	10	2	2.6	100.5	4.84
4	5.6	5.2	510	10.7	10.2	2.2	4.74	103	5.04
5	3.2	2.6	705	11	10.2	2.1	5	100.2	5.2
6	3	6.6	1005	10.6	10.1	2.2	5.24	103	5.07

TABLE E3.2: Calculations from Equilibrium Test at pH 2

Readings	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	NaOH [mL]	H_2SO_4 [mL]
1	50	5.85	0.2925	10	4.48	5	10.6
2	80	5.85	0.468	10	4.48	2.2	3.6
3	150	5.85	0.8775	10	4.48	5.4	0
4	500	5.85	2.925	10	4.48	5.6	5.2
5	700	5.85	4.095	10	4.48	3.2	2.6
6	1000	5.85	5.85	10	4.48	3	6.6

Readings	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
1	59	10.5	10.2	0.225	103.5	2.56
2	84	10.5	10	0.503	100	4
3	150	10.5	10	2.6	100.5	4.84
4	510	10.7	10.2	4.74	103	5.04
5	705	11	10.2	5	100.2	5.2
6	1005	10.6	10.1	5.24	103	5.07

Readings	Mass of solution[g]	Mass of Eluates [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance	Volume Measured
1	0.012258	0.26496	25.9764706	0.26496	94.77538462	65.6	59
2	0.04249344	0.4	40	0.4	94.54988034	85.8	84
3	0.401648	0.48642	48.642	0.48642	101.2043305	155.4	150
4	2.3912352	0.51912	50.8941176	0.51912	99.49932308	510.8	510
5	3.5224	0.52104	51.0823529	0.52104	98.74090354	705.8	705
6	5.2634752	0.52221	51.7039604	0.52221	98.90060171	1009.6	1005

Appendix E3: Equilibrium Test for pH set at 2

Readings	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]	Qe: Elution [g/L]	Ce [g/L]	$r_{H^+}^{Cu\ 2+}$	α
1	0.2925	0.012258	10	28.0242	25.9764706	0.225	0.054052	115.451
2	0.468	0.04249344	10	42.550656	40	0.503	0.049572	79.52286
3	0.8775	0.401648	10	47.5852	48.642	2.6	0.204538	18.70846
4	2.925	2.3912352	10	53.37648	50.8941176	4.74	0.854515	10.73716
5	4.095	3.5224	10	57.26	51.0823529	5	1.151091	10.21647
6	5.85	5.2634752	10	58.65248	51.7039604	5.24	1.557487	9.867168

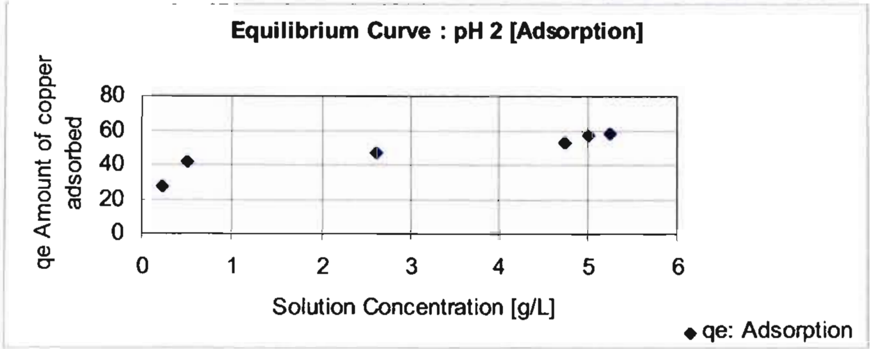


Figure E3.1: Equilibrium Curve at pH 2 for Adsorption

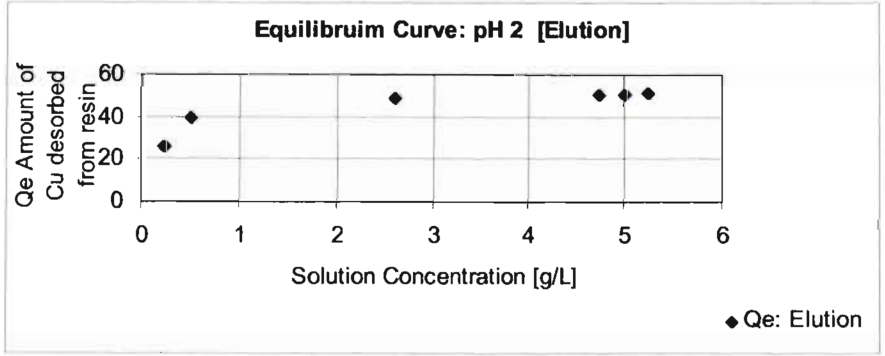


Figure E3.2: Equilibrium Curve at pH 2 for Elution

TABLE E3.3: The Langmuir and Freundlich Adsorption Isotherms

Readings	Ce [g/L]	Qe: Elution [g/L]	log Qe	log Ce	1/Qe	1/Ce
1	0.225	25.9764706	1.4145801	-0.6478175	0.03849638	4.44444444
2	0.503	40	1.60206	-0.298432	0.025	1.98807157
3	2.6	48.642	1.6870114	0.4149733	0.02055837	0.38461538
4	4.74	50.8941176	1.7066676	0.6757783	0.01964864	0.21097046
5	5	51.0823529	1.7082709	0.69897	0.01957623	0.2
6	5.24	51.7039604	1.7135238	0.7193313	0.01934088	0.19083969

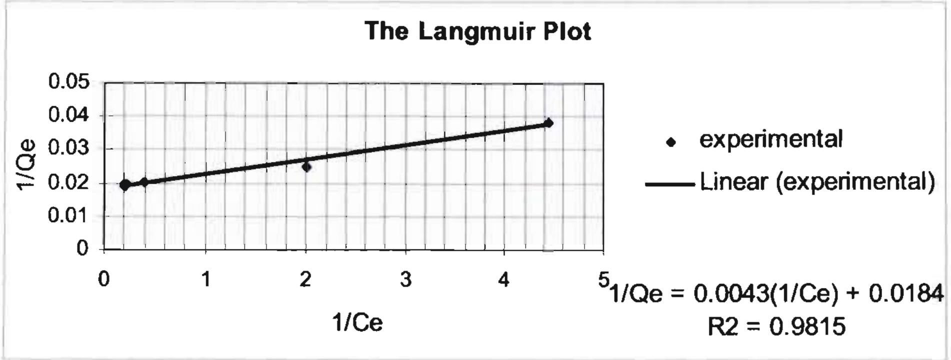


Figure E3.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 2

TABLE E3.4: Parameters for the Langmuir Plot

a	54.348
b	4.279
r	0.99

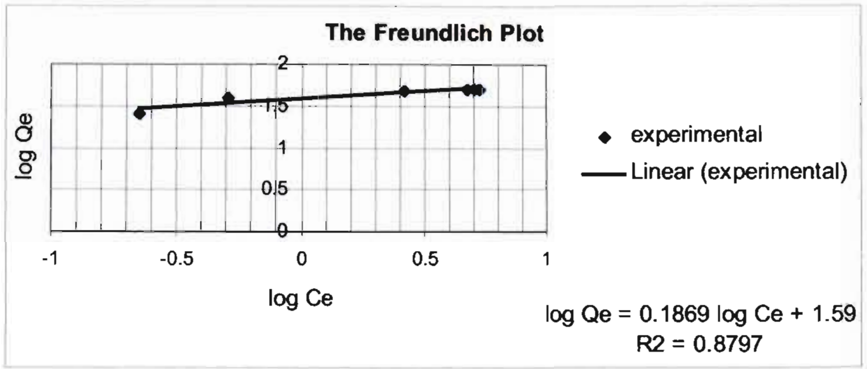


Figure E3.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 2

TABLE E3.5: Parameters for the Freundlich Plot

1/n	0.1869
Kf	38.9
r	0.9

TABLE E3.6: Tabulation of Results obtained from Langmuir and Freundlich Equation

Readings	Ce [g/L]	Langmuir Qe	Freundlich Qe
1	0.225	26.6586316	29.43557433
2	0.503	37.1074575	34.21154266
3	2.6	49.8658386	46.50590714
4	4.74	51.7943478	52.02984994
5	5	51.9212083	52.55173939
6	5.24	52.0276135	53.01424965

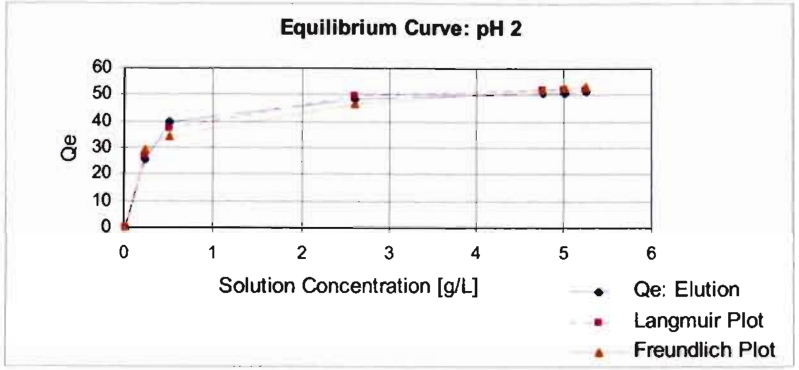


Figure E3.5: Comparison of Experimental and Predicted Results

APPENDIX E4: EQUILIBRIUM TEST FOR PH SET AT 1.5

TABLE E4.1: Results from Equilibrium Test at pH 1.5

Readings	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
1	40	10	2.1	1.7	1.5
2	80	10	2.1	1.9	1.5
3	150	10	2.1	2	1.49
4	500	10	2.1	2.4	1.5
5	700	10	2.1	2.38	1.48

Readings	NaOH [mL] used	H_2SO_4 [mL] used	Solution [Cu] remaining	Volume of resin loaded Cu	Volume of Eluted resin	pH resin	Con of Solution[g/L]	Volume of Cu from eluates	Con of Elution
1	3	1.5	40	10.5	10	2.2	1.1	100	1.8
2	4.5	1	79	10.5	10.2	2.2	1.9	100	2.84
3	3	1.2	140	10.5	10.1	1.9	3.2	100	3.32
4	6.6	17	515	10.5	10.2	2	4.84	100	3.83
5	4.3	11	702	10.5	10.2	2	5.1	103.5	3.74

TABLE E4.2: Calculations from Equilibrium Tests at pH 1.5

Readings	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	NaOH [mL]	H_2SO_4 [mL]
1	40	5.85	0.234	10	4.48	3	1.5
2	80	5.85	0.468	10	4.48	4.5	1
3	150	5.85	0.8775	10	4.48	3	1.2
4	500	5.85	2.925	10	4.48	6.6	17
5	700	5.85	4.095	10	4.48	4.3	11

Readings	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
1	40	10.5	10	1.1	100	1.8
2	79	10.5	10.2	1.9	100	2.84
3	140	10.5	10.1	3.2	100	3.32
4	515	10.5	10.2	4.84	100	3.83
5	702	10.5	10.2	5.1	103.5	3.74

Readings	Mass of solution[g]	Mass of Eluates [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance	Volume Measured
1	0.048928	0.18	18	0.18	97.83247863	44.5	40
2	0.160512	0.284	27.8431373	0.284	94.98119658	85.5	79
3	0.494336	0.332	32.8712871	0.332	94.16934473	154.2	140
4	2.4416832	0.383	37.5490196	0.383	96.57036581	523.6	515
5	3.592848	0.38709	37.95	0.38709	97.19018315	715.3	702

Readings	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]	Qe: Elution [g/L]	Ce [g/L]	$r_{H+}^{Cu\ 2+}$	α
1	0.234	0.048928	10	18.5072	18	1.1	0.554225	16.36364
2	0.468	0.160512	10	30.7488	27.8431373	1.9	0.574167	14.65428
3	0.8775	0.494336	10	38.3164	32.8712871	3.2	1.036207	10.27228
4	2.925	2.4416832	10	48.33168	37.5490196	4.84	3.002869	7.758062
5	4.095	3.592848	10	50.2152	37.95	5.1	4.144225	7.441176

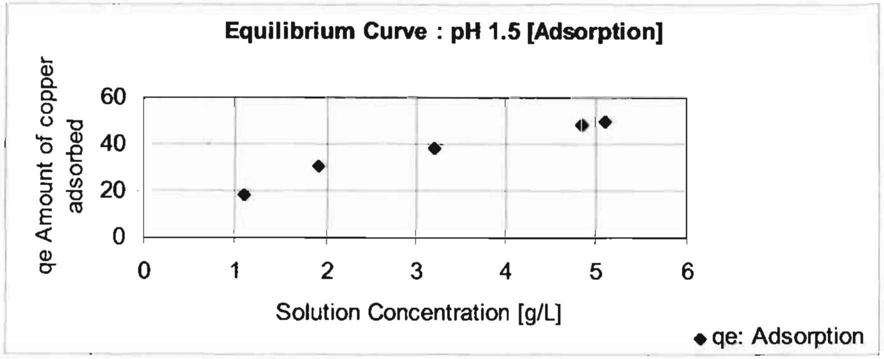


Figure E4.1: Equilibrium Curve at pH 1.5 for Adsorption

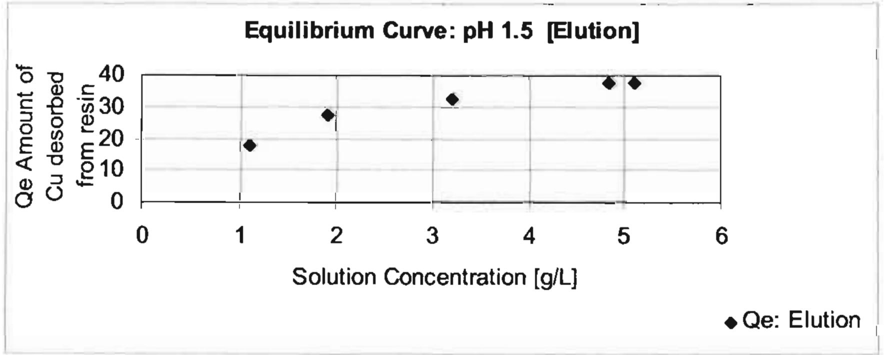


Figure E4.2: Equilibrium Curve at pH 1.5 for Elution

TABLE E4.3: The Langmuir and Freundlich Adsorption Isotherms

Readings	Ce [g/L]	Qe: Elution [g/L]	log Qe	log Ce	1/Qe	1/Ce
1	1.1	18	1.2552725	0.0413927	0.05555556	0.90909091
2	1.9	27.8431373	1.4447182	0.2787536	0.03591549	0.52631579
3	3.2	32.8712871	1.5168167	0.50515	0.03042169	0.3125
4	4.84	37.5490196	1.5745986	0.6848454	0.02663185	0.20661157
5	5.1	37.95	1.5792118	0.7075702	0.02635046	0.19607843

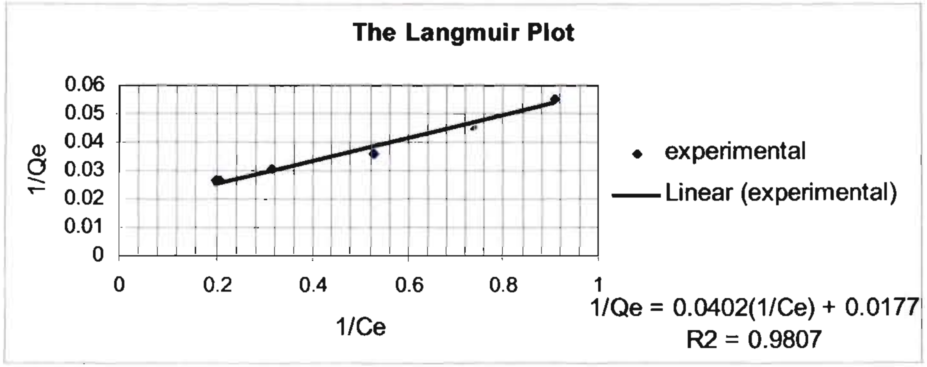


Figure E4.3: Linear Regression to obtain Parameters for the Langmuir Plot at pH 1.5

TABLE E4.4: Parameters for the Langmuir Plot

a	56.497
b	0.44
r	0.99

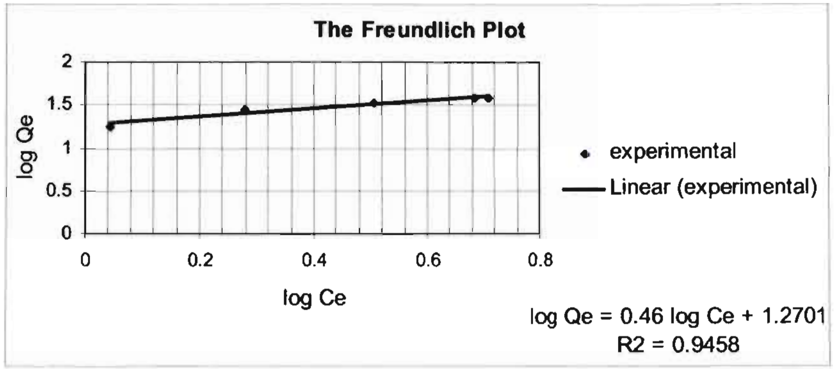


Figure E4.4: Linear Regression to obtain Parameters for the Freundlich Plot at pH 1.5

TABLE E4.5: Parameters for the Freundlich Plot

1/n	0.46
Kf	18.625
r	0.97

TABLE E4.6: Tabulation of Results obtained from Langmuir and Freundlich Equation

Readings	Ce [g/L]	Langmuir Qe	Freundlich Qe
1	1.1	18.4262453	19.45973477
2	1.9	25.7252135	25.0220529
3	3.2	33.0347907	31.80279366
4	4.84	38.4445332	38.47026233
5	5.1	39.0811554	39.4074708

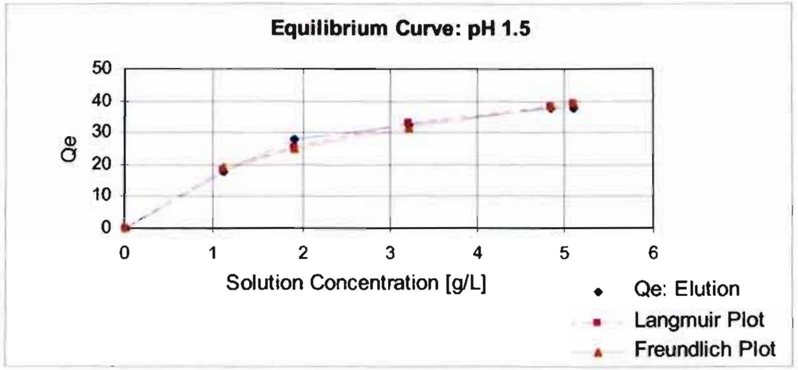


Figure E4.5: Comparison of Experimental and Predicted Results

APPENDIX E5: SUMMARY OF EQUILIBRIUM PLOTS

The following represents a summary of all the equilibrium curves at the respective pH settings. Again Qe is the equilibrium elution loading in g/L and Ce is equilibrium solution obtained after twenty four hours.

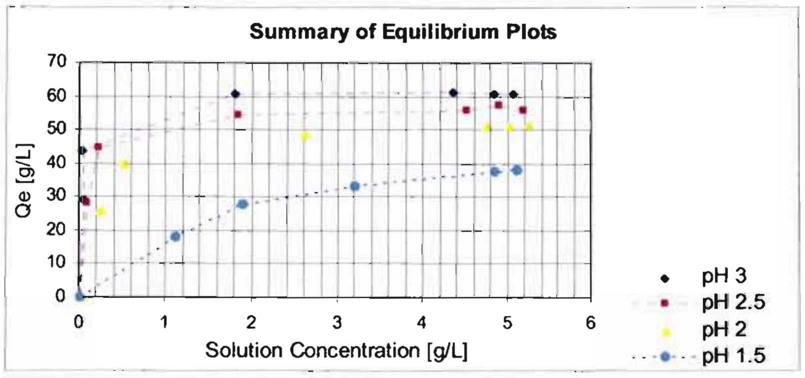


Figure E5.1: Summary of Equilibrium Plots

APPENDIX F: KINETIC TESTS

F1: KINETIC TEST FOR PARTICLE DIAMETER 850 μm F1-1

F2: KINETIC TEST FOR PARTICLE DIAMETER 600 μm F2-1

F3: COMPARISON OF THE PARTICLE DIAMETERS F3-1

F4: PREDICTED KINETIC RESPONSE F4-1

APPENDIX F1: KINETIC TEST FOR PARTICLE DIAMETER 850 μm

TABLE F1.1: Operating Conditions at the Beginning of the Kinetic Test

Concentration	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
6 g/L	3000	617	1.94	3	3.3
0.6 g/L	3000	58.9	2	3.08	3.1

TABLE F1.2: Operating Conditions at the End of the Kinetic Test

Concentration	NaOH[mL] used	Solution [Cu] remaining	Volume of resin with Cu	Volume of Eluted resin	pH resin	Conc. of Solution: End [g/L]	Volume of Cu from eluates [mL]	Conc. of Elution[g/L]
6 g/L	80	3041	640	610	2.1	0.0179	6005	2.94
0.6 g/L	55.6	3010	63	56	2.5	0.01	1010	1.71

TABLE F1.3: Calculations for Kinetic Test from Experimental Data

Concentration	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	Volume NaOH [mL]
6 g/L	3000	5.9	17.7	617	276.416	80
0.6 g/L	3000	0.61	1.83	58.9	26.3872	55.6

Concentration	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
6 g/L	3041	640	610	0.0179	6005	2.94
0.6 g/L	3010	63	56	0.01	1010	1.71

Concentration	Mass of solution[g]	Mass of Elute [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance [mL]	Volume Measured [mL]
6 g/L	0.0586478	17.6547	28.94213115	17.6547	100.0754116	3080	3041
0.6 g/L	0.0302639	1.7271	30.84107143	1.7271	96.03081268	3055.6	3010

Concentration	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]
6 g/L	17.7	0.058647846	617	28.59214287
0.6 g/L	1.83	0.030263872	58.9	30.55579165

TABLE F1.4: Results obtained from Kinetic Test

Time [hours]	Initial Concentrations	
	6 g/L	0.6 g/L
0.08333333	1.74	0.492
0.16666667	1.41	0.433
0.25	0.98	0.385
0.33333333	0.87	0.281
0.5	0.51	0.234
1	0.202	0.2
2	0.071	0.121
3	0.057	0.096
4	0.0309	0.063
5	0.0265	0.0329
6	0.0245	0.02
7	0.0232	0.0138
8	0.0222	0.0103
24	0.0179	0.01

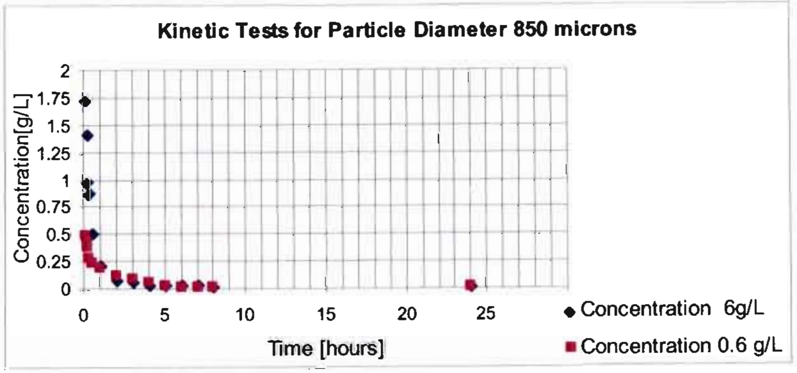


Figure F1.1: Kinetic Response for 850 μm from Table F1.4

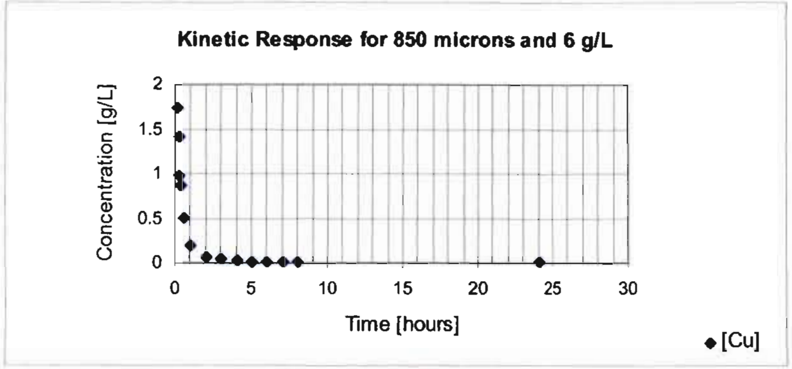


Figure F1.2: Kinetic Response for Initial Concentration at 6 g/L from Table F1.4

TABLE F1.5: First Order Reaction Kinetics for 6 g/L

Time [hours]	[Cu] 6 g/L	ln[Cuo/Cu]
0.08333333	1.74	1.22106724
0.16666667	1.41	1.43136265
0.25	0.98	1.79515506
0.33333333	0.87	1.91421442
0.5	0.51	2.4482969
1	0.202	3.37443993
2	0.071	4.42002775
3	0.057	4.63965636
4	0.0309	5.25195145
5	0.0265	5.4055629
6	0.0245	5.48403451
7	0.0232	5.53855535
8	0.0222	5.58261534
24	0.0179	5.79790692

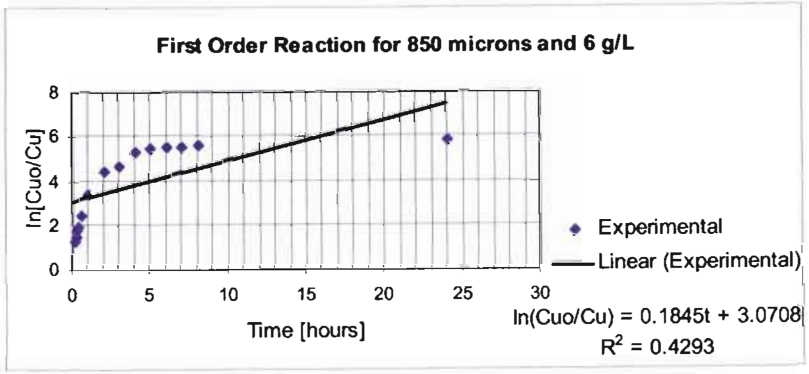


Figure F1.3: First Order Reaction Plot for 6 g/L from Table F1.5

TABLE F1.6: Second Order Reaction Kinetics for 6 g/L

Time [hours]	[Cu] 6 g/L	[1/Cu]
0.083333	1.74	0.57471264
0.166667	1.41	0.70921986
0.25	0.98	1.02040816
0.333333	0.87	1.14942529
0.5	0.51	1.96078431
1	0.202	4.95049505
2	0.071	14.084507
3	0.057	17.5438596
4	0.0309	32.3624595
5	0.0265	37.7358491
6	0.0245	40.8163265
7	0.0232	43.1034483
8	0.0222	45.045045
24	0.0179	55.8659218

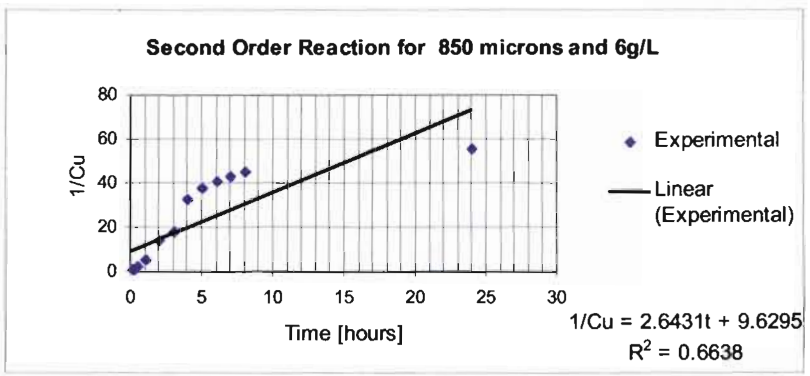


Figure F1.4: Second Order Reaction Plot for 6 g/L from Table F1.6

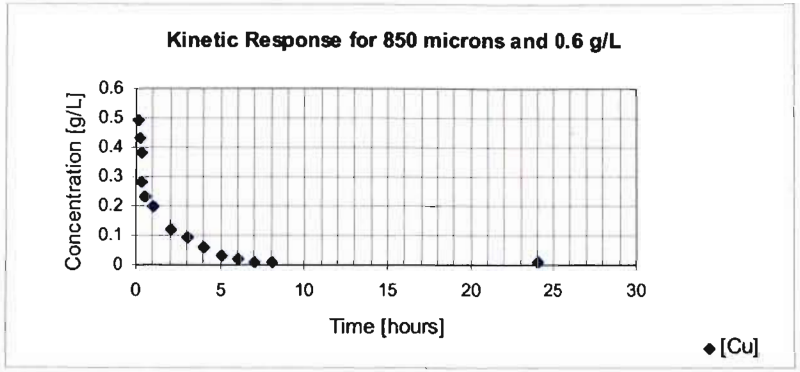


Figure F1.5: Kinetic Response for Initial Concentration at 0.6 g/L from Table F1.4

TABLE F1.7: First Order Reaction Kinetics for 0.6 g/L

Time [hours]	[Cu] 6 g/L	ln[Cuo/Cu]
0.08333333	0.492	0.21498024
0.16666667	0.433	0.34272123
0.25	0.385	0.46021562
0.33333333	0.281	0.77510429
0.5	0.234	0.95813784
1	0.2	1.11514159
2	0.121	1.61766841
3	0.096	1.84911077
4	0.063	2.27032423
5	0.0329	2.9199863
6	0.02	3.41772668
7	0.0138	3.78879037
8	0.0103	4.08131506
24	0.01	4.11087386

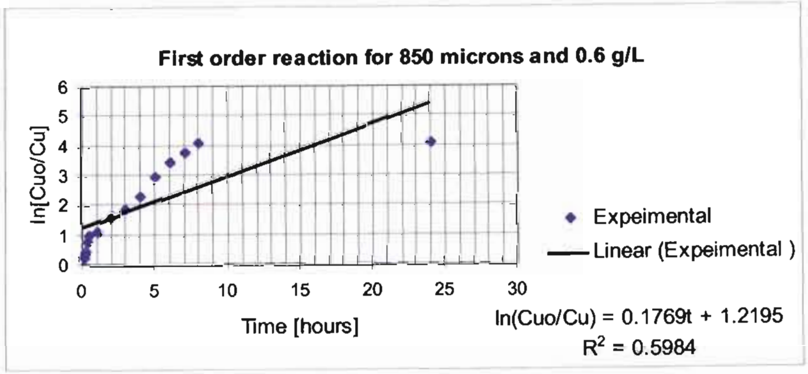


Figure F1.6: First Order Reaction Plot for 0.6 g/L from Table F1.7

TABLE F1.8: Second Order Reaction Kinetics for 0.6 g/L

Time [hours]	[Cu] 0.6 g/L	[1/Cu]
0.083333	0.492	2.03252033
0.166667	0.433	2.30946882
0.25	0.385	2.5974026
0.333333	0.281	3.55871886
0.5	0.234	4.27350427
1	0.2	5
2	0.121	8.26446281
3	0.096	10.4166667
4	0.063	15.8730159
5	0.0329	30.3951368
6	0.02	50
7	0.0138	72.4637681
8	0.0103	97.0873786
24	0.01	100

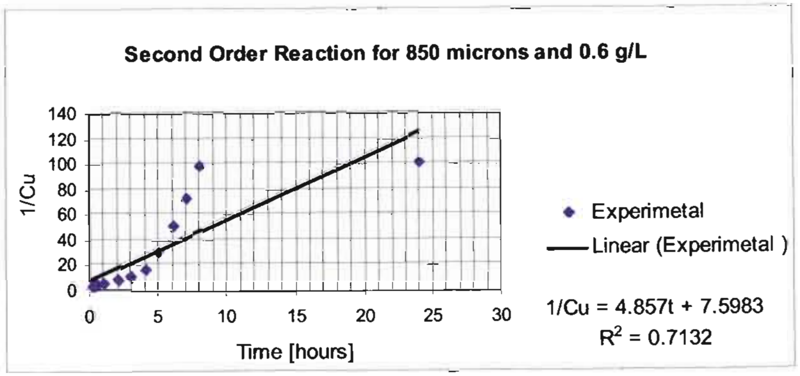


Figure F1.7: Second Order Reaction Plot for 0.6 g/L from Table F1.8

TABLE F1.9: Mass Transfer Coefficient (k) calculated from First and Second Order Reaction Curves for Diameter 850 μm:

Mass Transfer Coefficient (k) at 6 and 0.6 g/L		
Concentration	**6 g/L**	**0.6 g/L**
k: 1st order	0.1845	0.1769
k: 2nd order	2.6431	4.857

APPENDIX F2: KINETIC TEST FOR PARTICLE DIAMETER 600 μm

TABLE F2.1: Operating Conditions at the Beginning of the Kinetic Test

Concentration	Solution Volume [mL]	Resin Volume [mL]	pH resin	pH solution Beginning	pH End
6 g/L	3000	620	2.04	3.01	3.19
0.6 g/L	3000	60	1.98	3.1	3.3

TABLE F2.2: Operating Conditions at the End of the Kinetic Test

Concentration	NaOH[mL] used	Solution [Cu] remaining	Volume of resin with Cu	Volume of Eluted resin	pH resin	Conc. of Solution: End [g/L]	Volume of Cu from eluates [mL]	Conc. of Elution[g/L]
6 g/L	12.8	3010	650	615	2	0.0093	5900	3.24
0.6 g/L	35.8	2800	64	58	1.98	0.0019	1010	1.69

TABLE F2.3: Calculations for Kinetic Test from Experimental Data

Concentration	Vo [mL]	Co [g/L]	Co * Vo Mass of Cu	Vro [mL]	Water Retention[mL]	Volume NaOH [mL]
6 g/L	3000	5.9	17.7	620	277.76	12.8
0.6 g/L	3000	0.61	1.83	60	26.88	35.8

Concentration	Vs [mL]	Vc [mL]	Ve [mL]	Ce [g/L]	Vcu [mL]	Cel [g/L]
6 g/L	3010	650	615	0.0093	5900	3.24
0.6 g/L	2800	64	58	0.0019	1010	1.69

Concentration	Mass of solution[g]	Mass of Elute [g]	Qe: Elution [g/L]	Mass of Cu on resin	Accountability	Volume Balance [mL]	Volume Measured [mL]
6 g/L	0.0304832	19.116	31.08292683	19.116	108.1722213	3012.8	3010
0.6 g/L	0.0057511	1.7069	29.42931034	1.7069	93.58749027	3035.8	2800

Concentration	Co * Vo	Mass of solution[g]	Vro [mL]	qe: Adsorption[g/L]
6 g/L	17.7	0.030483168	620	28.4992207
0.6 g/L	1.83	0.005751072	60	30.4041488

TABLE F2.4: Results obtained from Kinetic Test

Time [hours]	Initial Concentrations	
	6 g/L	0.6 g/L
0.08333333	1.68	0.39
0.16666667	1.42	0.38
0.25	1.1	0.372
0.33333333	0.72	0.304
0.5	0.266	0.249
1	0.066	0.153
2	0.033	0.075
3	0.025	0.0256
4	0.0086	0.0124
5	0.0077	0.0064
6	0.0084	0.0075
7	0.0078	0.0036
8	0.0087	0.0033
24	0.0093	0.0019

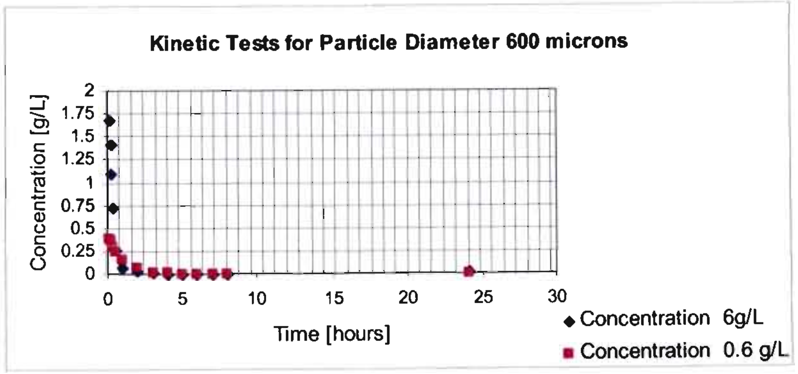


Figure F2.1: Kinetic Response for 600 μm from Table F2.4

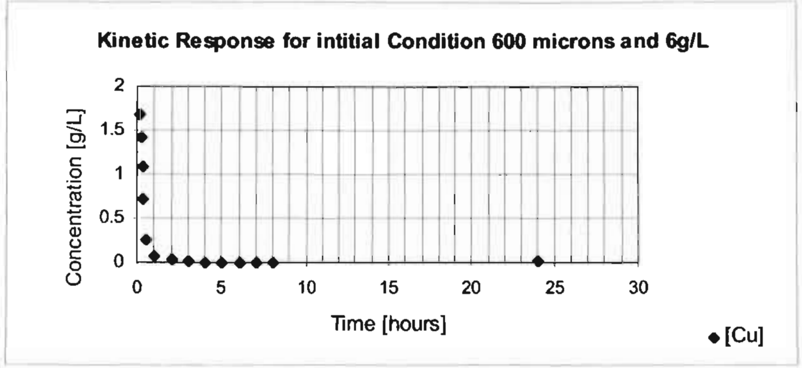


Figure F2.2 Kinetic Response for Initial Concentration at 6 g/L from Table F2.4

TABLE F2.5: First Order Reaction Kinetics for 6 g/L

Time [hours]	[Cu] 6 g/L	ln[Cuo/Cu]
0.08333333	1.68	1.25615856
0.16666667	1.42	1.42429548
0.25	1.1	1.67964217
0.33333333	0.72	2.10345642
0.5	0.266	3.09921132
1	0.066	4.49305289
2	0.033	5.18620007
3	0.025	5.46383181
4	0.0086	6.53094543
5	0.0077	6.6414873
6	0.0084	6.55447592
7	0.0078	6.6285839
8	0.0087	6.5193846
24	0.0093	6.45269323

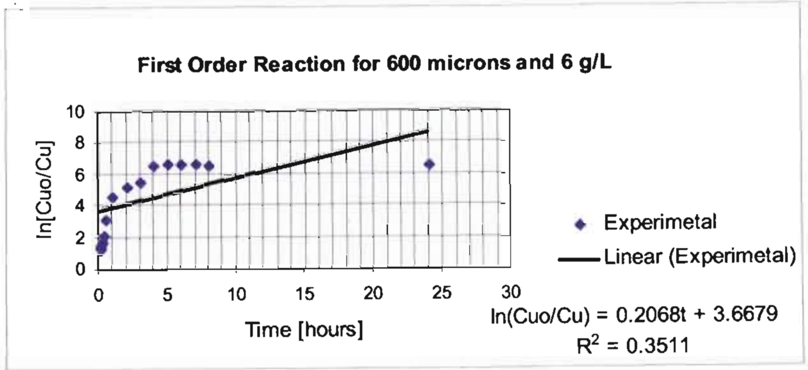


Figure F2.3: First Order Reaction Plot for 6 g/L from Table F2.5

TABLE F2.6: Second Order Reaction Kinetics for 6 g/L

Time [hours]	[Cu] 6 g/L	[1/Cu]
0.083333	1.68	0.5952381
0.166667	1.42	0.70422535
0.25	1.1	0.90909091
0.333333	0.72	1.38888889
0.5	0.266	3.7593985
1	0.066	15.1515152
2	0.033	30.3030303
3	0.025	40
4	0.0086	116.27907
5	0.0077	129.87013
6	0.0084	119.047619
7	0.0078	128.205128
8	0.0087	114.942529
24	0.0093	107.526882

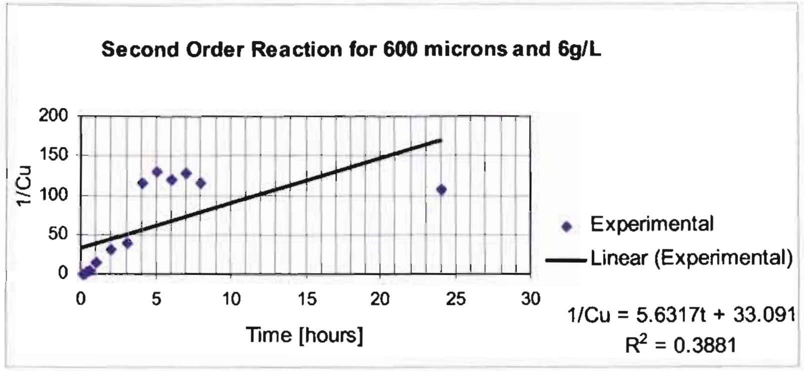


Figure F2.4: Second Order Reaction Plot for 6 g/L from Table F2.6

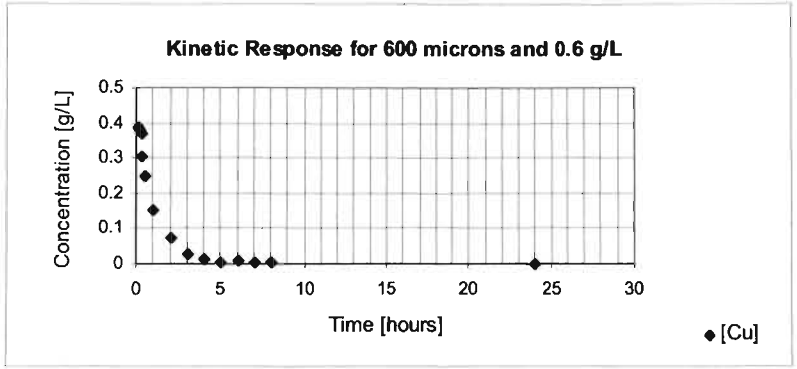


Figure F2.5: Kinetic Response for Initial Concentrations at 0.6 g/L from Table F2.4

TABLE F2.7: First Order Reaction Kinetics for 0.6 g/L

Time [hours]	[Cu] 0.6 g/L	ln[Cuo/Cu]
0.08333333	0.39	0.43078292
0.16666667	0.38	0.4567584
0.25	0.372	0.4780358
0.33333333	0.304	0.67990195
0.5	0.249	0.87947676
1	0.153	1.36649173
2	0.075	2.07944154
3	0.0256	3.1543373
4	0.0124	3.87923318
5	0.0064	4.54063166
6	0.0075	4.38202663
7	0.0036	5.11599581
8	0.0103	4.08131506
24	0.01	4.11087386

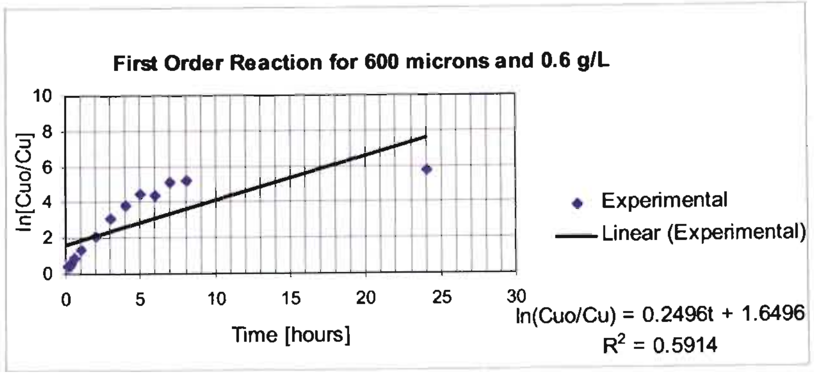


Figure F2.6: First Order Reaction Plot for 0.6 g/L from Table F2.7

TABLE F2.8: Second Order Reaction Kinetics for 0.6 g/L

Time [hours]	[Cu] 0.6 g/L	[1/Cu]
0.083333	0.39	2.56410256
0.166667	0.38	2.63157895
0.25	0.372	2.68817204
0.333333	0.304	3.28947368
0.5	0.249	4.01606426
1	0.153	6.53594771
2	0.075	13.3333333
3	0.0256	39.0625
4	0.0124	80.6451613
5	0.0064	156.25
6	0.0075	133.333333
7	0.0036	277.777778
8	0.0033	303.030303
24	0.0019	526.315789

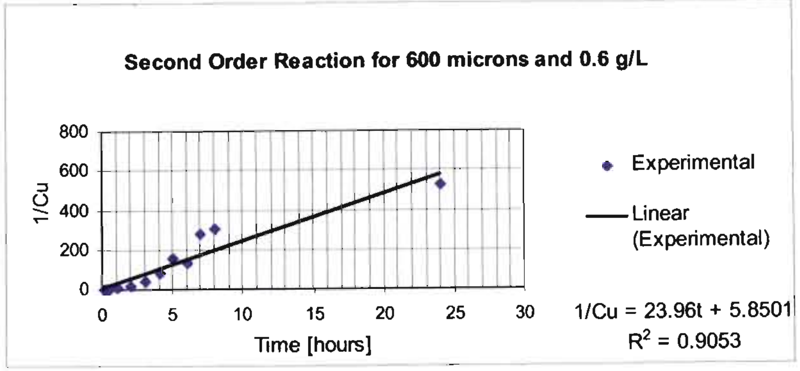


Figure F2.7: Second Order Reaction Plot for 0.6 g/L from Table F2.8

TABLE F2.9: Mass Transfer Coefficient (k) calculated from First and Second Order Reaction Curves for Diameter 600 μm:

Mass Transfer Coefficient (k) at 6 and 0.6 g/L		
Concentration	**6 g/L**	**0.6 g/L**
k: 1st order	0.2068	0.2496
k: 2nd order	5.6317	23.96

APPENDIX F3: COMPARISON OF THE PARTICLE DIAMETERS

F3.1 Comparison of Particle Diameter 850 and 600 μm for Concentration 6 g/L

TABLE F3.1: Experimental Data for 6 g/L

Time [hours]	[Cu] 6 [g/L]: 850	[Cu] 6 [g/L]: 600
0.083333333	1.74	1.68
0.166666667	1.41	1.42
0.25	0.98	1.1
0.333333333	0.87	0.72
0.5	0.51	0.266
1	0.202	0.066
2	0.071	0.033
3	0.057	0.025
4	0.0309	0.0086
5	0.0265	0.0077
6	0.0245	0.0084
7	0.0232	0.0078
8	0.0222	0.0087
24	0.0179	0.0093

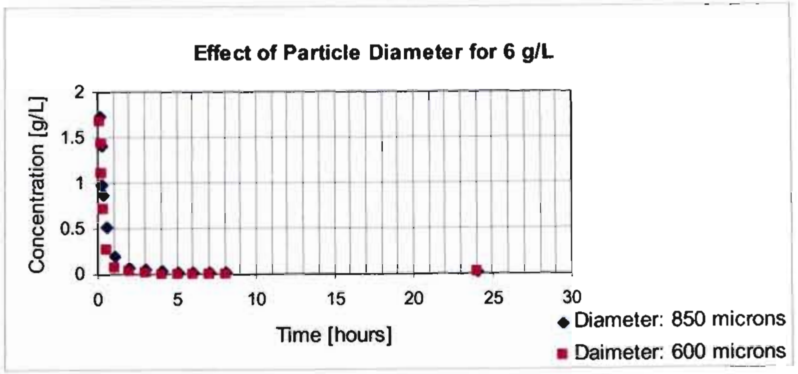


Figure F3.1: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 6 g/L

F3.2 Comparison of Particle Diameter 850 and 600 μm for Concentration 0.6 g/L

TABLE F3.2: Experimental Data for 0.6 g/L

Time [hours]	[Cu] 0.6 [g/L]: 850	[Cu] 0.6 [g/L]: 600
0.083333333	0.492	0.39
0.166666667	0.433	0.38
0.25	0.385	0.372
0.333333333	0.281	0.304
0.5	0.234	0.249
1	0.2	0.153
2	0.121	0.075
3	0.096	0.0256
4	0.063	0.0124
5	0.0329	0.0064
6	0.02	0.0075
7	0.0138	0.0036
8	0.0103	0.0033
24	0.01	0.0019

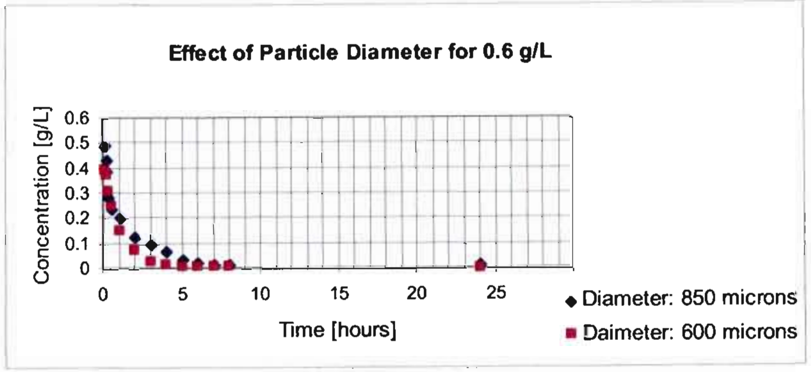


Figure F3.2: Comparison of Particle Diameter 850 μm and 600 μm at Initial Concentration 0.6 g/L

APPENDIX F4: PREDICTED KINETIC RESPONSES

TABLE F4.1: Predicted Kinetic Data for Particle Diameter 850 μm and Concentration 6g/L

Time [hours]	[Cu] 6 [g/L]	[1/Cu] k=2.6431	[Cu] predicted
0.083333333	1.74	0.389749859	2.565748204
0.166666667	1.41	0.610008192	1.639322247
0.25	0.98	0.830266525	1.204432516
0.333333333	0.87	1.050524859	0.951905128
0.5	0.51	1.491041525	0.670672133
1	0.202	2.812591525	0.355543985
2	0.071	5.455691525	0.183294821
3	0.057	8.098791525	0.123475212
4	0.0309	10.74189153	0.093093474
5	0.0265	13.38499153	0.074710544
6	0.0245	16.02809153	0.06239046
7	0.0232	18.67119153	0.053558446
8	0.0222	21.31429153	0.046916877
24	0.0179	63.60389153	0.015722308

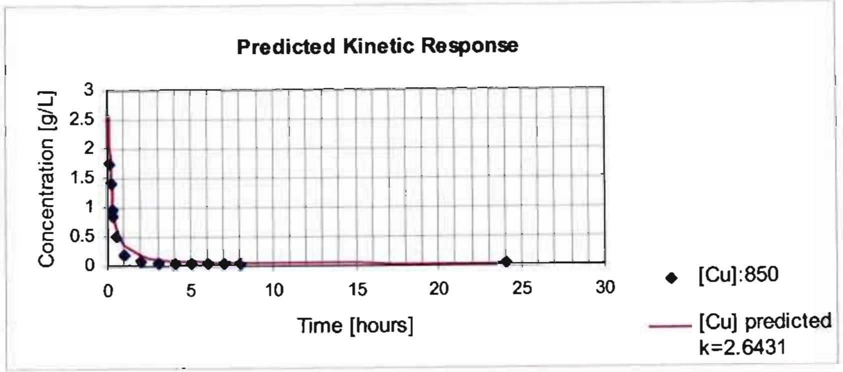


Figure F4.1: Predicted Kinetic Response for Particle Diameter 850 μm and Concentration 6g/L (Refer to Table F4.1)

TABLE F4.2: Predicted Kinetic Data for Particle Diameter 600 μm and Concentration 6g/L

Time [hours]	[Cu] 6 [g/L]	[1/Cu] k=5.6317	[Cu] predicted
0.083333333	1.68	0.638799859	1.565435537
0.166666667	1.42	1.108108192	0.902438956
0.25	1.1	1.577416525	0.633947967
0.333333333	0.72	2.046724859	0.488585457
0.5	0.266	2.985341525	0.33497005
1	0.066	5.801191525	0.17237838
2	0.033	11.43289153	0.087466937
3	0.025	17.06459153	0.058600875
4	0.0086	22.69629153	0.044060061
5	0.0077	28.32799153	0.035300773
6	0.0084	33.95969153	0.029446675
7	0.0078	39.59139153	0.025258016
8	0.0087	45.22309153	0.022112597
24	0.0093	135.3302915	0.007389329

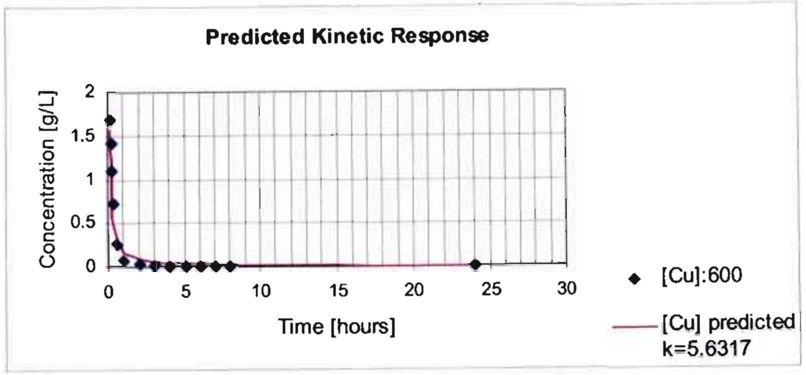


Figure F4.2: Predicted Kinetic Response for Particle Diameter 600 μm and Concentration 6g/L (Refer to Table F4.2)

TABLE F4.3: Predicted Kinetic Data for Particle Diameter 850 μm and Concentration 0.6g/L

Time [hours]	[Cu] 0.6 [g/L]	[1/Cu] k=4.857	[Cu] predicted
0.083333333	0.492	2.044094262	0.48921423
0.166666667	0.433	2.448844262	0.408355899
0.25	0.385	2.853594262	0.350435243
0.333333333	0.281	3.258344262	0.306904341
0.5	0.234	4.067844262	0.245830454
1	0.2	6.496344262	0.153932729
2	0.121	11.35334426	0.088079774
3	0.096	16.21034426	0.061689004
4	0.063	21.06734426	0.047466828
5	0.0329	25.92434426	0.038573782
6	0.02	30.78134426	0.03248721
7	0.0138	35.63834426	0.028059665
8	0.0103	40.49534426	0.024694197
24	0.01	118.2073443	0.008459711

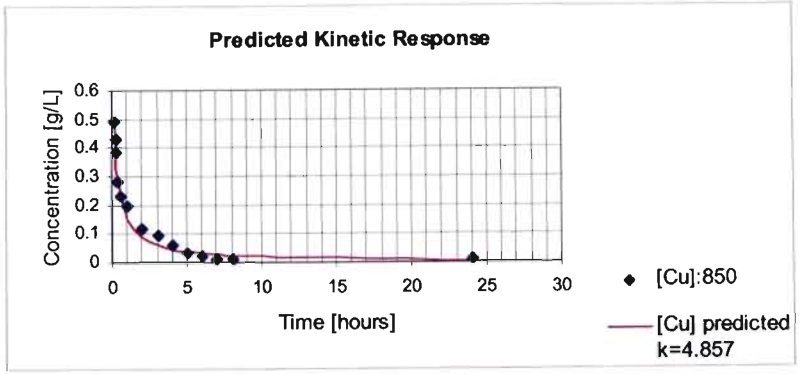


Figure F4.3: Predicted Kinetic Response for Particle Diameter 850 μm and Concentration 0.6g/L (Refer to Table F4.3)

TABLE F4.4: Predicted Kinetic Data for Particle Diameter 600 μm and Concentration 0.6g/L

Time [hours]	[Cu] 0.6 [g/L]	[1/Cu] k=23.96	[Cu] predicted
0.083333333	0.39	3.636010929	0.275026676
0.166666667	0.38	5.632677596	0.177535459
0.25	0.372	7.629344262	0.131072864
0.333333333	0.304	9.626010929	0.103885193
0.5	0.249	13.61934426	0.073424974
1	0.153	25.59934426	0.039063501
2	0.075	49.55934426	0.02017783
3	0.0256	73.51934426	0.013601862
4	0.0124	97.47934426	0.010258584
5	0.0064	121.4393443	0.008234564
6	0.0075	145.3993443	0.00687761
7	0.0036	169.3593443	0.005904605
8	0.0033	193.3193443	0.005172788
24	0.0019	576.6793443	0.001734066

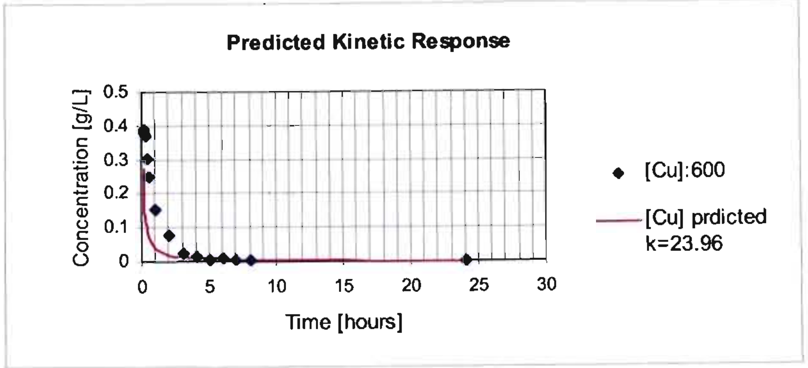


Figure F4.4: Predicted Kinetic Response for Particle Diameter 600 μm and Concentration 0.6g/L (Refer to Table F4.4)

APPENDIX G: MINI COLUMN TESTS

G1: HYDROGEN FORM, FEED pH 3 G1-1

G2: DI SODIUM FORM, FEED pH 3 G2-1

G3: MONO SODIUM FORM, FEED pH 3 G3-1

G4: SUMMARY OF RESULTS OBTAINED FROM FEED pH 2.5 G4-1

G5: SUMMARY OF RESULTS OBTAINED FROM FEED pH 2.04 G5-1

G6: SUMMARY OF RESULTS OBTAINED FROM FEED pH 1.59 G6-1

APPENDIX G1: HYDROGEN FORM, FEED PH 3

TABLE G1.1: Initial Conditions for Hydrogen Form at pH 3

Input
Column Diameter = 2.08 cm
Resin Volume = 50 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.12 [Hydrogen Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 5.35 g/L at pH = 3
Overall Mass of Copper Input = 5.75125 g

TABLE G1.2: End Conditions for Hydrogen Form, Feed pH 3

Output
Resin Volume = 52 mL [Loaded with Copper]
Volume of Eluate = 526 mL
Eluate Concentration = 3.78 g/L
Mass of Copper from Eluate = 1.98828 g

Elution
Resin Volume = 50 mL [Hydrogen Form]
pH Resin = 2.01 [Hydrogen Form]
Loading from Elution = 38.236154 g/L

TABLE G1.3: Calculations for Mini Column Test with Initial Conditions from Table G1.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.0015	0.000280374	0.5	3.6208	1.98	0.0000375	0.13375	0.1337125	2.67425	2.67425
0.006	0.001121495	1	7.2416	2.09	0.00015	0.13375	0.1336	2.672	5.34625
0.0155	0.002897196	1.5	10.8624	1.79	0.0003875	0.13375	0.1333625	2.66725	8.0135
0.062	0.011588785	2	14.4832	1.17	0.00155	0.13375	0.1322	2.644	10.6575
0.84	0.157009346	2.5	18.104	0.84	0.021	0.13375	0.11275	2.255	12.9125
1.08	0.201869159	3	21.7248	0.82	0.027	0.13375	0.10675	2.135	15.0475
1.31	0.244859813	3.5	25.3456	0.77	0.03275	0.13375	0.101	2.02	17.0675
1.45	0.271028037	4	28.9664	0.76	0.03625	0.13375	0.0975	1.95	19.0175
1.6	0.299065421	4.5	32.5872	0.76	0.04	0.13375	0.09375	1.875	20.8925
1.7	0.317757009	5	36.208	0.77	0.0425	0.13375	0.09125	1.825	22.7175
2	0.373831776	5.5	39.8288	0.84	0.05	0.13375	0.08375	1.675	24.3925
2.6	0.485981308	6	43.4496	1.13	0.065	0.13375	0.06875	1.375	25.7675
2.9	0.542056075	6.5	47.0704	1.12	0.0725	0.13375	0.06125	1.225	26.9925
3.13	0.585046729	7	50.6912	1.12	0.07825	0.13375	0.0555	1.11	28.1025
3.1	0.579439252	7.5	54.312	1.11	0.0775	0.13375	0.05625	1.125	29.2275
3.2	0.598130841	8	57.9328	1.11	0.08	0.13375	0.05375	1.075	30.3025
3.24	0.605607477	8.5	61.5536	1.13	0.081	0.13375	0.05275	1.055	31.3575
3.38	0.631775701	9	65.1744	1.13	0.0845	0.13375	0.04925	0.985	32.3425
3.54	0.661682243	9.5	68.7952	1.1	0.0885	0.13375	0.04525	0.905	33.2475
3.64	0.680373832	10	72.416	1.1	0.091	0.13375	0.04275	0.855	34.1025
3.63	0.678504673	10.5	76.0368	1.11	0.09075	0.13375	0.043	0.86	34.9625
3.65	0.682242991	11	79.6576	1.16	0.09125	0.13375	0.0425	0.85	35.8125
3.72	0.695327103	11.5	83.2784	1.16	0.093	0.13375	0.04075	0.815	36.6275
3.85	0.719626168	12	86.8992	1.19	0.09625	0.13375	0.0375	0.75	37.3775
3.89	0.727102804	12.5	90.52	1.21	0.09725	0.13375	0.0365	0.73	38.1075
3.9	0.728971963	13	94.1408	1.23	0.0975	0.13375	0.03625	0.725	38.8325
3.96	0.740186916	13.5	97.7616	1.23	0.099	0.13375	0.03475	0.695	39.5275
4	0.747663551	14	101.3824	1.25	0.1	0.13375	0.03375	0.675	40.2025
4.1	0.76635514	14.5	105.0032	1.27	0.1025	0.13375	0.03125	0.625	40.8275
4.3	0.803738318	15	108.624	1.3	0.1075	0.13375	0.02625	0.525	41.3525

Table G1.3: ...continued...

4.3	0.803738318	15	108.624	1.3	0.1075	0.13375	0.02625	0.525	41.3525
4.354	0.813831776	15.5	112.2448	1.33	0.10885	0.13375	0.0249	0.498	41.8505
4.4	0.822429907	16	115.8656	1.34	0.11	0.13375	0.02375	0.475	42.3255
4.47	0.835514019	16.5	119.4864	1.38	0.11175	0.13375	0.022	0.44	42.7655
4.4	0.822429907	17	123.1072	1.42	0.11	0.13375	0.02375	0.475	43.2405
4.54	0.848598131	17.5	126.728	1.4	0.1135	0.13375	0.02025	0.405	43.6455
4.49	0.839252336	18	130.3488	1.43	0.11225	0.13375	0.0215	0.43	44.0755
4.53	0.846728972	18.5	133.9696	1.44	0.11325	0.13375	0.0205	0.41	44.4855
4.63	0.865420561	19	137.5904	1.47	0.11575	0.13375	0.018	0.36	44.8455
4.71	0.880373832	19.5	141.2112	1.51	0.11775	0.13375	0.016	0.32	45.1655
4.7	0.878504673	20	144.832	1.53	0.1175	0.13375	0.01625	0.325	45.4905
4.72	0.882242991	20.5	148.4528	1.57	0.118	0.13375	0.01575	0.315	45.8055
4.71	0.880373832	21	152.0736	1.58	0.11775	0.13375	0.016	0.32	46.1255
4.87	0.910280374	21.5	155.6944	1.58	0.12175	0.13375	0.012	0.24	46.3655
Cumulative Total					**3.432975**			**46.3655**	

Accountability = 94.2622 %
Resin Loading [Adsorption] = 46.36 g/L
Mass Transfer Zone = 12.97 cm
Residence Time = 6.36 min

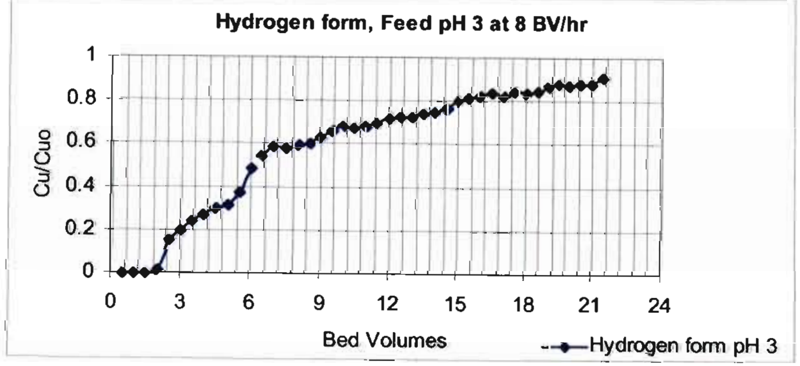


Figure G1.1: Breakthrough Curve with Initial Condition from Table G1.1

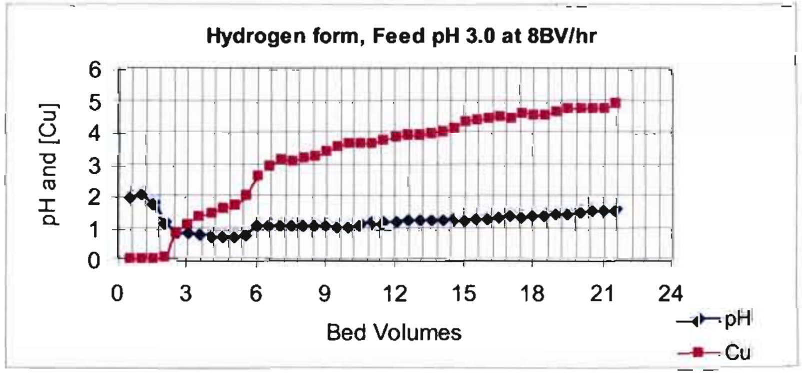


Figure G1.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX G2: DI SODIUM FORM, FEED PH 3

TABLE G2.1: Initial Conditions for Di Sodium Form, Feed pH 3

Input
Column Diameter = 2.08 cm
Resin Volume = 67.9 mL of TP 207 [Di Sodium Form]
pH of Resin = 10.85 [Di Sodium Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 5.35 g/L at pH = 3
Overall Mass of Copper Input = 5.75125 g

TABLE G2.2: End Conditions for Di Sodium Form, Feed pH 3

Output
Resin Volume = 57.9 mL [Loaded with Copper]
Volume of Eluate = 500 mL
Eluate Concentration = 4.29 g/L
Mass of Copper from Eluate = 2.145 g

Elution
Resin Volume = 51 mL [Hydrogen Form]
pH Resin = 2.09 [Hydrogen Form]
Loading from Elution = 37.046632 g/L

TABLE G2.3: Calculations for Mini Column Test with Initial Conditions from Table G2.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.0015	0.000280374	0.5	3.6208	11.24	0.0000375	0.13375	0.1337125	1.969256259	1.969256259
0.0023	0.000429907	1	7.2416	10.8	0.0000575	0.13375	0.1336925	1.968961708	3.938217968
0.0028	0.000523364	1.5	10.8624	10.34	0.00007	0.13375	0.13368	1.968777614	5.906995582
0.0027	0.000504673	2	14.4832	9.66	0.0000675	0.13375	0.1336825	1.968814433	7.875810015
0.0018	0.000336449	2.5	18.104	10.6	0.000045	0.13375	0.133705	1.969145803	9.844955817
0.0202	0.003775701	3	21.7248	6.33	0.000505	0.13375	0.133245	1.962371134	11.80732695
0.088	0.016448598	3.5	25.3456	5.17	0.0022	0.13375	0.13155	1.937407953	13.7447349
0.5	0.093457944	4	28.9664	5.05	0.0125	0.13375	0.12125	1.785714286	15.53044919
0.8	0.14953271	4.5	32.5872	4.91	0.02	0.13375	0.11375	1.675257732	17.20570692
1	0.186915888	5	36.208	4.79	0.025	0.13375	0.10875	1.601620029	18.80732695
1.2	0.224299065	5.5	39.8288	4.69	0.03	0.13375	0.10375	1.527982327	20.33530928
1.3	0.242990654	6	43.4496	4.62	0.0325	0.13375	0.10125	1.491163476	21.82647275
1.44	0.269158879	6.5	47.0704	4.37	0.036	0.13375	0.09775	1.439617084	23.26608984
1.5	0.280373832	7	50.6912	4.4	0.0375	0.13375	0.09625	1.417525773	24.68361561
1.77	0.330841121	7.5	54.312	4.39	0.04425	0.13375	0.0895	1.318114875	26.00173049
2.02	0.377570093	8	57.9328	4.32	0.0505	0.13375	0.08325	1.226067747	27.22779823
2.26	0.422429907	8.5	61.5536	4.21	0.0565	0.13375	0.07725	1.137702504	28.36550074
2.43	0.454205607	9	65.1744	4.02	0.06075	0.13375	0.073	1.075110457	29.44061119
2.5	0.46728972	9.5	68.7952	3.75	0.0625	0.13375	0.07125	1.049337261	30.48994845
2.84	0.530841121	10	72.416	3.45	0.071	0.13375	0.06275	0.924153166	31.41410162
2.84	0.530841121	10.5	76.0368	3.09	0.071	0.13375	0.06275	0.924153166	32.33825479
3	0.560747664	11	79.6576	2.83	0.075	0.13375	0.05875	0.865243004	33.20349779
3.31	0.618691589	11.5	83.2784	2.2	0.08275	0.13375	0.051	0.751104566	33.95460236
3.89	0.727102804	12	86.8992	1.94	0.09725	0.13375	0.0365	0.537555228	34.49215758
3.8	0.710280374	12.5	90.52	2.01	0.095	0.13375	0.03875	0.570692194	35.06284978
3.97	0.742056075	13	94.1408	1.88	0.09925	0.13375	0.0345	0.508100147	35.57094993
4.08	0.762616822	13.5	97.7616	1.82	0.102	0.13375	0.03175	0.467599411	36.03854934
4.07	0.760747664	14	101.3824	1.76	0.10175	0.13375	0.032	0.471281296	36.50983063
4.08	0.762616822	14.5	105.0032	1.7	0.102	0.13375	0.03175	0.467599411	36.97743004

Table G2.3: ...continued...

4	0.747663551	15	108.624	1.65	0.1	0.13375	0.03375	0.497054492	37.47448454
4.13	0.771962617	15.5	112.2448	1.62	0.10325	0.13375	0.0305	0.449189985	37.92367452
4.23	0.790654206	16	115.8656	1.55	0.10575	0.13375	0.028	0.412371134	38.33604566
4.13	0.771962617	16.5	119.4864	1.52	0.10325	0.13375	0.0305	0.449189985	38.78523564
4.2	0.785046729	17	123.1072	1.5	0.105	0.13375	0.02875	0.423416789	39.20865243
4.24	0.792523364	17.5	126.728	1.49	0.106	0.13375	0.02775	0.408689249	39.61734168
4.19	0.78317757	18	130.3488	1.48	0.10475	0.13375	0.029	0.427098675	40.04444035
4.28	0.8	18.5	133.9696	1.48	0.107	0.13375	0.02675	0.393961708	40.43840206
4.16	0.777570093	19	137.5904	1.48	0.104	0.13375	0.02975	0.43814433	40.87654639
4.24	0.792523364	19.5	141.2112	1.49	0.106	0.13375	0.02775	0.408689249	41.28523564
4.28	0.8	20	144.832	1.49	0.107	0.13375	0.02675	0.393961708	41.67919735
4.34	0.811214953	20.5	148.4528	1.51	0.1085	0.13375	0.02525	0.371870398	42.05106775
4.2	0.785046729	21	152.0736	1.72	0.105	0.13375	0.02875	0.423416789	42.47448454
4.27	0.798130841	21.5	155.6944	1.72	0.10675	0.13375	0.027	0.397643594	42.87212813
Cumulative Total					**2.8402325**			**42.87212813**	

Accountability = 86.68.58 %
Resin Loading [Adsorption] = 42.87 g/L
Mass Transfer Zone = 12.45 cm
Residence Time = 6.1 min

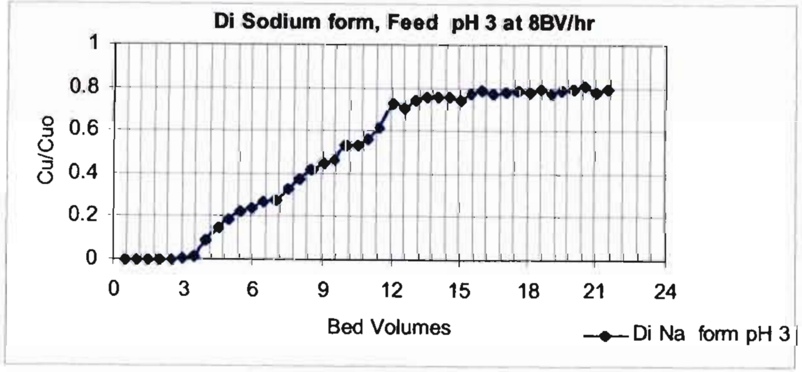


Figure G2.1: Breakthrough Curve with Initial Condition from Table G2.1

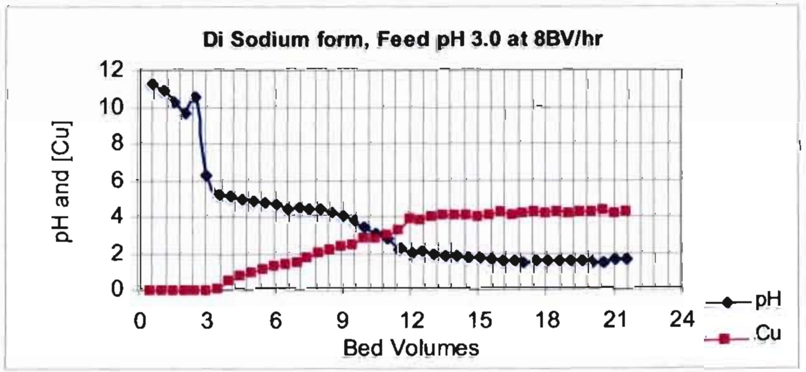


Figure G2.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX G3: MONO SODIUM FORM, FEED PH 3

TABLE G3.1: Initial Conditions for Mono Sodium Form, Feed pH 3

Input
Column Diameter = 2.08 cm
Resin Volume = 63.4 mL of TP 207 [Mono Sodium Form]
pH of Resin = 10.85 [Mono Sodium Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 5.35 g/L at pH = 3
Overall Mass of Copper Input = 6.6875 g

TABLE G3.2: End Conditions for Mono Sodium Form, Feed pH 3

Output
Resin Volume = 56 mL [Loaded with Copper}
Volume of Eluate = 536 mL
Eluate Concentration = 3.4 g/L
Mass of Copper from Eluate = 1.8224 g

Elution
Resin Volume = 51 mL [Hydrogen Form]
pH Resin = 2.14 [Hydrogen Form]
Loading from Elution = 32.542857 g/L

TABLE G3.3: Calculations for Mini Column Test with Initial Conditions from Table G3.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.009	0.001682243	0.5	3.6208	5.09	0.000225	0.13375	0.133525	2.106072555	2.106072555
0.0255	0.004766355	1	7.2416	4.55	0.0006375	0.13375	0.1331125	2.099566246	4.205638801
0.0131	0.002448598	1.5	10.8624	5.24	0.0003275	0.13375	0.1334225	2.104455836	6.310094637
0.0102	0.001906542	2	14.4832	4.62	0.000255	0.13375	0.133495	2.105599369	8.415694006
0.072	0.013457944	2.5	18.104	4.49	0.0018	0.13375	0.13195	2.081230284	10.49692429
0.209	0.039065421	3	21.7248	4.35	0.005225	0.13375	0.128525	2.027208202	12.52413249
0.475	0.088785047	3.5	25.3456	4.04	0.011875	0.13375	0.121875	1.922318612	14.4464511
0.89	0.16635514	4	28.9664	3.71	0.02225	0.13375	0.1115	1.758675079	16.20512618
1.03	0.192523364	4.5	32.5872	3.38	0.02575	0.13375	0.108	1.703470032	17.90859621
1.56	0.291588785	5	36.208	3.14	0.039	0.13375	0.09475	1.494479495	19.40307571
1.74	0.325233645	5.5	39.8288	2.84	0.0435	0.13375	0.09025	1.423501577	20.82657729
1.95	0.364485981	6	43.4496	2.51	0.04875	0.13375	0.085	1.340694006	22.16727129
2.3	0.429906542	6.5	47.0704	2.15	0.0575	0.13375	0.07625	1.202681388	23.36995268
2.67	0.499065421	7	50.6912	1.87	0.06675	0.13375	0.067	1.056782334	24.42673502
3.08	0.575700935	7.5	54.312	1.64	0.077	0.13375	0.05675	0.89511041	25.32184543
3.1	0.579439252	8	57.9328	1.42	0.0775	0.13375	0.05625	0.887223975	26.2090694
3.25	0.607476636	8.5	61.5536	1.3	0.08125	0.13375	0.0525	0.82807571	27.03714511
3.3	0.61682243	9	65.1744	1.22	0.0825	0.13375	0.05125	0.808359621	27.84550473
3.4	0.635514019	9.5	68.7952	1.18	0.085	0.13375	0.04875	0.768927445	28.61443218
3.55	0.663551402	10	72.416	1.16	0.08875	0.13375	0.045	0.70977918	29.32421136
3.63	0.678504673	10.5	76.0368	1.15	0.09075	0.13375	0.043	0.678233438	30.00244479
3.55	0.663551402	11	79.6576	1.15	0.08875	0.13375	0.045	0.70977918	30.71222397
3.59	0.671028037	11.5	83.2784	1.15	0.08975	0.13375	0.044	0.694006309	31.40623028
3.66	0.68411215	12	86.8992	1.16	0.0915	0.13375	0.04225	0.666403785	32.07263407
3.6	0.672897196	12.5	90.52	1.17	0.09	0.13375	0.04375	0.690063091	32.76269716
3.65	0.682242991	13	94.1408	1.19	0.09125	0.13375	0.0425	0.670347003	33.43304416
3.71	0.693457944	13.5	97.7616	1.2	0.09275	0.13375	0.041	0.646687697	34.07973186
3.8	0.710280374	14	101.3824	1.21	0.095	0.13375	0.03875	0.611198738	34.6909306

Table G3.3: ...continued...

3.69	0.689719626	14.5	105.0032	1.21	0.09225	0.13375	0.0415	0.654574132	35.34550473
3.81	0.712149533	15	108.624	1.23	0.09525	0.13375	0.0385	0.607255521	35.95276025
3.77	0.704672897	15.5	112.2448	1.25	0.09425	0.13375	0.0395	0.623028391	36.57578864
3.84	0.717757009	16	115.8656	1.27	0.096	0.13375	0.03775	0.595425868	37.17121451
3.9	0.728971963	16.5	119.4864	1.28	0.0975	0.13375	0.03625	0.571766562	37.74298107
3.85	0.719626168	17	123.1072	1.31	0.09625	0.13375	0.0375	0.59148265	38.33446372
3.75	0.700934579	17.5	126.728	1.31	0.09375	0.13375	0.04	0.630914826	38.96537855
3.79	0.708411215	18	130.3488	1.33	0.09475	0.13375	0.039	0.615141956	39.5805205
3.85	0.719626168	18.5	133.9696	1.35	0.09625	0.13375	0.0375	0.59148265	40.17200315
3.84	0.717757009	19	137.5904	1.37	0.096	0.13375	0.03775	0.595425868	40.76742902
3.86	0.721495327	19.5	141.2112	1.39	0.0965	0.13375	0.03725	0.587539432	41.35496845
3.88	0.725233645	20	144.832	1.39	0.097	0.13375	0.03675	0.579652997	41.93462145
3.94	0.736448598	20.5	148.4528	1.41	0.0985	0.13375	0.03525	0.555993691	42.49061514
3.95	0.738317757	21	152.0736	1.43	0.09875	0.13375	0.035	0.552050473	43.04266562
3.83	0.71588785	21.5	155.6944	1.45	0.09575	0.13375	0.038	0.599369085	43.6420347
3.94	0.736448598	22	159.3152	1.47	0.0985	0.13375	0.03525	0.555993691	44.19802839
3.99	0.745794393	22.5	162.936	1.49	0.09975	0.13375	0.034	0.536277603	44.73430599
3.99	0.745794393	23	166.5568	1.51	0.09975	0.13375	0.034	0.536277603	45.2705836
3.79	0.708411215	23.5	170.1776	1.54	0.09475	0.13375	0.039	0.615141956	45.88572555
3.99	0.745794393	24	173.7984	1.57	0.09975	0.13375	0.034	0.536277603	46.42200315
4.04	0.755140187	24.5	177.4192	1.59	0.101	0.13375	0.03275	0.516561514	46.93856467
4.08	0.762616822	25	181.04	1.61	0.102	0.13375	0.03175	0.500788644	47.43935331
4.2	0.785046729	25.5	184.6608	1.6	0.105	0.13375	0.02875	0.453470032	47.89282334
4.25	0.794392523	26	188.2816	1.62	0.10625	0.13375	0.0275	0.433753943	48.32657729
4.3	0.803738318	26.5	191.9024	1.61	0.1075	0.13375	0.02625	0.414037855	48.74061514
Cumulative Total					**3.799095**			**47.66805994**	

Accountability = 84.0597 %

Resin Loading [Adsorption] = 48.74 g/L

Mass Transfer Zone = 13.36 cm

Residence Time = 6.57 min

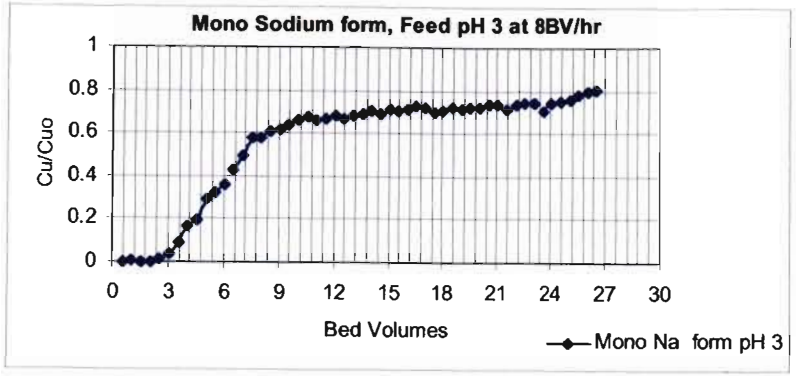


Figure G3.1: Breakthrough Curve with Initial Condition from Table G3.1

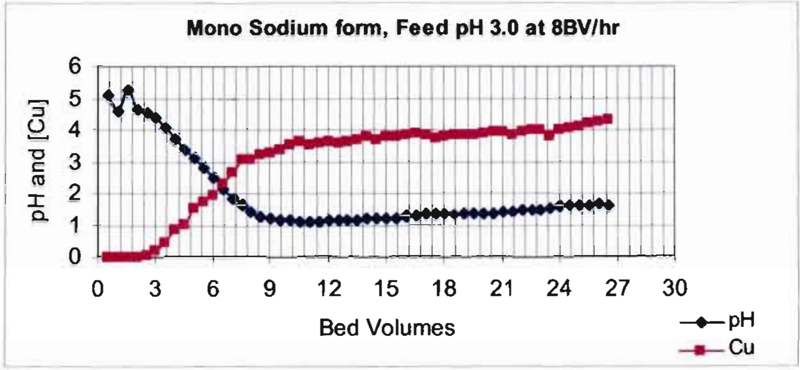


Figure G3.2: Monitoring of Copper Concentration and Effluent pH

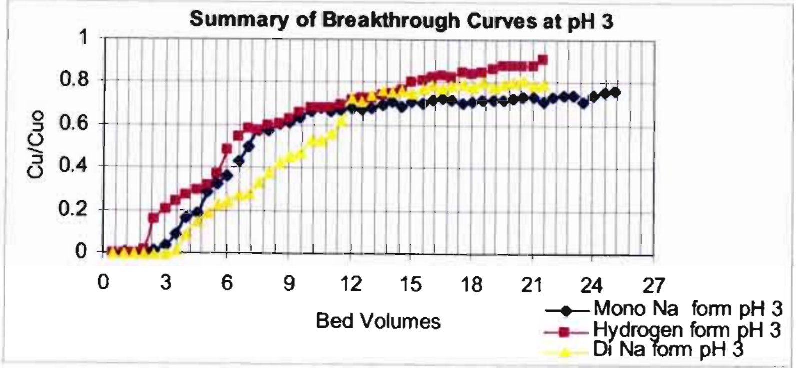


Figure G3.3: Resin in Three Forms at Initial Feed pH of 3

APPENDIX G4: SUMMARY OF RESULTS OBTAINED FROM FEED PH 2.5

The similar procedures and calculations that was performed in Appendix G1 to G3, were applied and the results for the mini column tests for feed pH of 2.5 with the three forms of resin are summarized in the following tables and graphs below.

G4.1 Hydrogen Form, Feed pH 2.5

TABLE G4.1: Initial Conditions for Hydrogen Form, Feed pH 2.5

Input
Column Diameter = 2.08 cm
Resin volume = 50.3 mL of TP 207 [Hydrogen Form]
pH of resin = 2.58 [Hydrogen Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 4.67 g/L pH = 2.5
Overall Mass of Copper Input = 5.3705 g

TABLE G4.2: End Conditions Hydrogen Form, Feed pH 2.5

Output
Resin volume = 53 mL [Loaded with Copper]
Volume of Eluate = 519.5 mL
Eluate Concentration = 3.61 g/L
Mass of copper from Eluate =1.875395 g

Elution
Resin volume = 50.2 mL [Hydrogen Form]
pH resin = 2.10 [Hydrogen Form]
Loading from Elution =35.054112 g/L

Accountability = 92.31948 %
Resin Loading [Adsorption] = 45.48 g/L
Mass Transfer Zone = 12.97 cm
Residence Time = 6.36 min

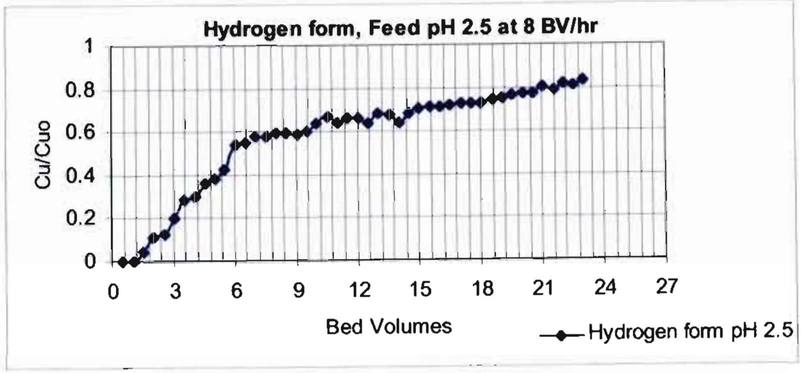


Figure G4.1: Breakthrough Curve with Initial Condition from Table G4.1

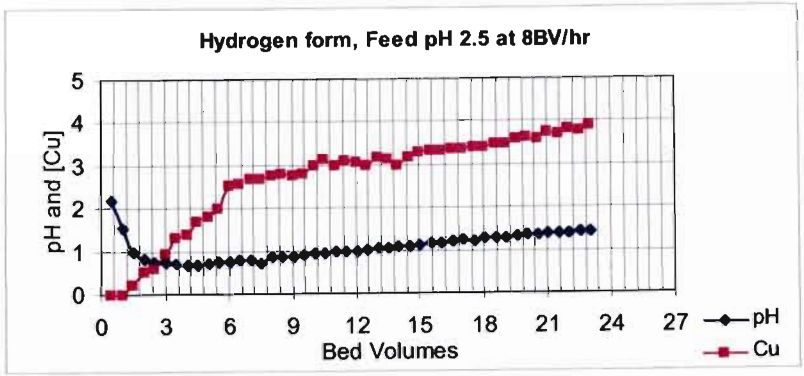


Figure G4.2: Monitoring of Copper Concentration and Effluent pH

G4.2 Di Sodium Form, Feed pH 2.5

TABLE G4.3: Initial Conditions for Di Sodium Form, Feed pH 2.5

Input
Column Diameter = 2.08 cm
Resin Volume = 67.8 mL of TP 207 [Di Sodium Form]
pH of Resin = 10.82 [Di Sodium Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 4.67 g/L at pH = 2.5
Overall Mass of Copper Input = 5.47825 g

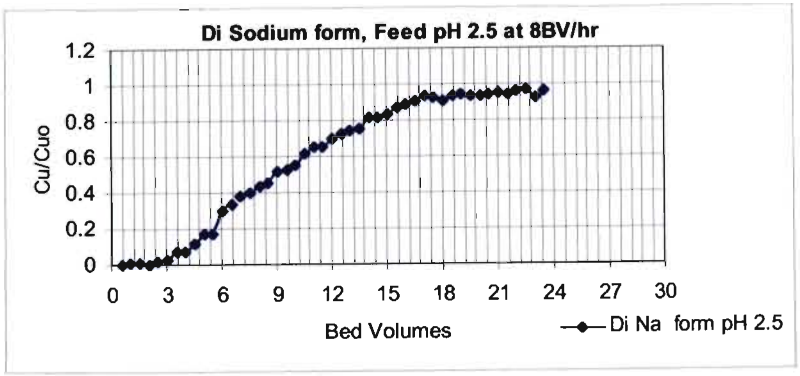


Figure G4.3: Breakthrough Curve with Initial Condition from Table G4.3

TABLE G4.4: End Conditions for Di Sodium Form, Feed pH 2.5

Output
Resin Volume = 57.9 mL [Loaded with Copper]
Volume of Eluate = 500 mL
Eluate Concentration = 4.25 g/L
Mass of Copper from Eluate = 2.125 g

Elution
Resin Volume = 50 mL [Hydrogen Form]
pH Resin = 2.28 [Hydrogen Form]
Loading from Elution =36.701209 g/L

Accountability = 97.81261 %
Resin Loading [Adsorption] = 33.11 g/L
Mass Transfer Zone = 12.4 cm
Residence Time = 6.1 min

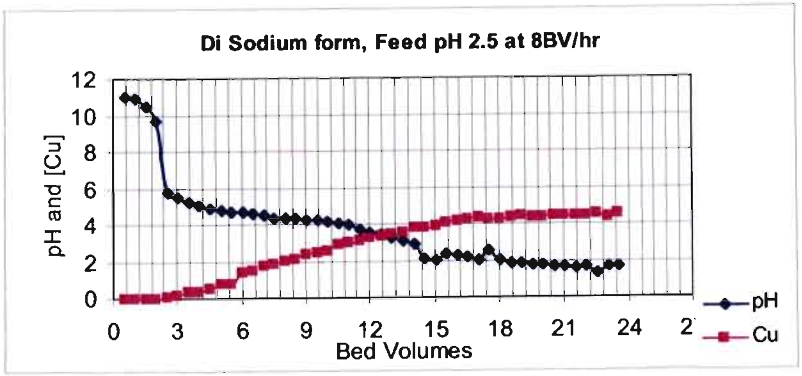


Figure G4.4: Monitoring of Copper Concentration and Effluent pH

G4.3 Mono Sodium Form, Feed pH 2.5

TABLE G4.5: Initial Conditions for Mono Sodium Form, Feed pH 2.5

Input
Column Diameter = 2.08 cm
Resin Volume = 63 mL of TP 207 [Mono Sodium Form]
pH of Resin = 6.30 [Mono Sodium Form]
Flow rate = 414.269 mL/hr = 8.285 BV/hr
Linear Velocity = 0.0339 cm/s
Feed Solution = 4.67 g/L at pH = 2.5
Overall Mass of Copper Input = 5.604 g

TABLE G4.6: End Conditions for Mono Sodium Form, Feed pH 2.5

Output
Resin Volume = 57 mL [Loaded with Copper]
Volume of Eluate = 523 mL
Eluate Concentration = 3.76 g/L
Mass of Copper from Eluate =1.96648 g

Elution
Resin Volume = 51 mL [Hydrogen Form]
pH Resin = 2.17 [Hydrogen Form]
Loading from Elution = 34.499649 g/L

Accountability = 98.77574 %
Resin Loading [Adsorption] = 32.30 g/L
Mass Transfer Zone = 12.35 cm
Residence Time = 6.07 min

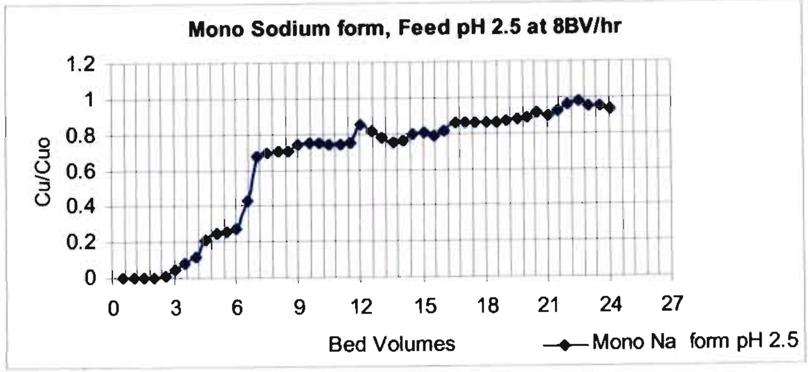


Figure G4.5: Breakthrough Curve with Initial Condition from Table G4.5

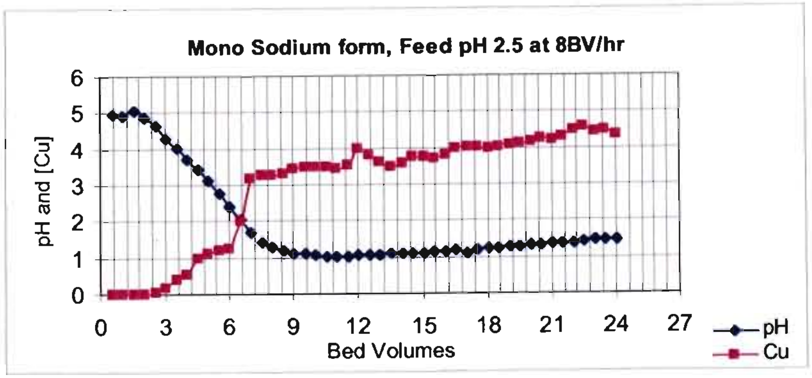


Figure G4.6: Monitoring of Copper Concentration and Effluent pH

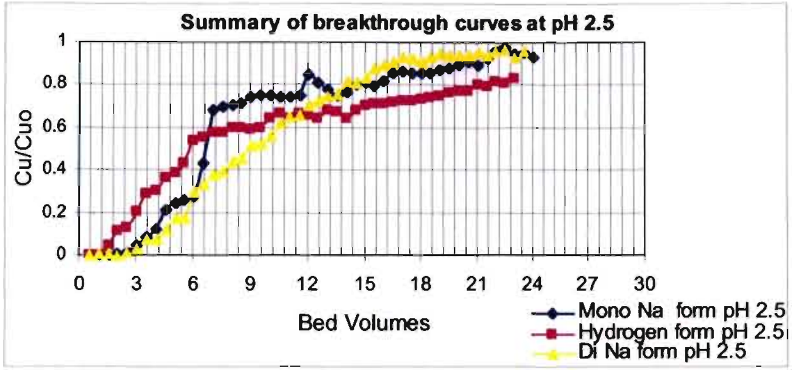


Figure G4.7: Resin in Three Forms at Initial Feed pH of 2.5

APPENDIX G5: SUMMARY OF RESULTS OBTAINED FROM FEED PH 2.04

The similar procedures and calculations that was performed in Appendix G1 to G3, were applied and the results for the mini column tests for feed pH of 2.04 with the three forms of resin are summarized in the following tables and graphs below.

G5.1 Hydrogen Form, Feed pH 2.04

TABLE G5.1: Initial Conditions for Hydrogen Form, Feed pH 2.04

Input
Column Diameter = 2.08 cm
Resin Volume = 50.3 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.39 [Hydrogen Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.262 g/L at pH = 2.04
Overall Mass of Copper Input = 4.86735 g

TABLE G5.2: End Conditions for Hydrogen Form, Feed pH 2.04

Output
Resin Volume = 53 mL [Loaded with Copper]
Volume of Eluate = 500.5 mL
Eluate Concentration = 3.35 g/L
Mass of Copper from Eluate =1.676675 g

Elution
Resin Volume = 51 mL [Hydrogen Form]
pH Resin = 2.30 [Hydrogen Form]
Loading from Elution = 31.635377 g/L

Accountability = 83.171438 %
Resin Loading [Adsorption] = 49.61 g/L
Mass Transfer Zone = 13.47 cm
Residence Time = 6.54 min

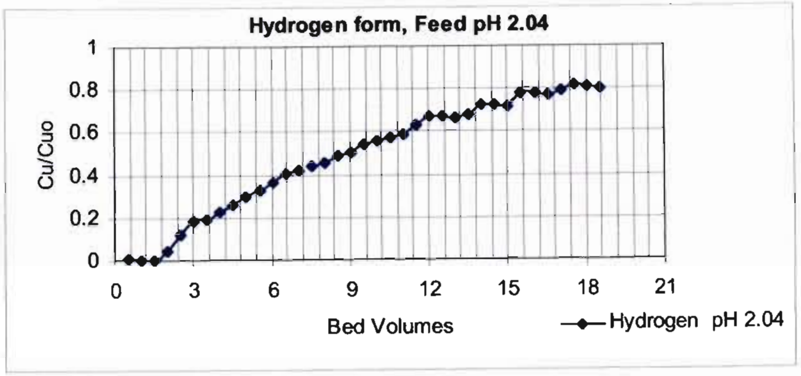


Figure G5.1: Breakthrough Curve with Initial Condition from Table G5.1

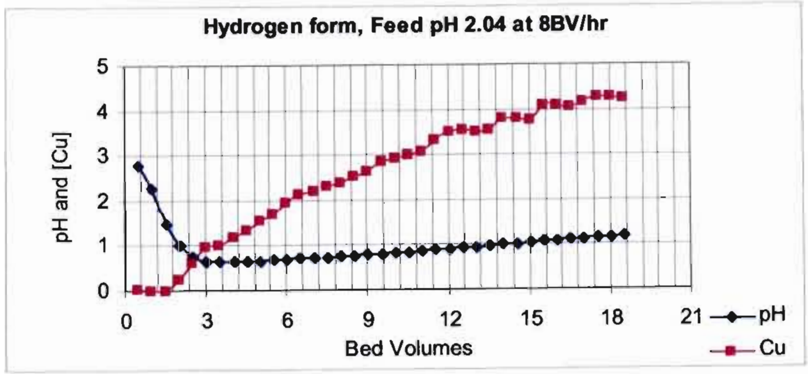


Figure G5.2: Monitoring of Copper Concentration and Effluent pH

G5.2 Di Sodium Form, Feed pH 2.04

TABLE G5.3: Initial Conditions for Di Sodium Form, Feed pH 2.04

Input
Column Diameter = 2.08 cm
Resin Volume = 67.5 mL of TP 207 [Di Sodium Form]
pH of Resin = 11.21 [Di Sodium Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.262 g/L at pH = 2.04
Overall Mass of Copper Input = 5.262 g

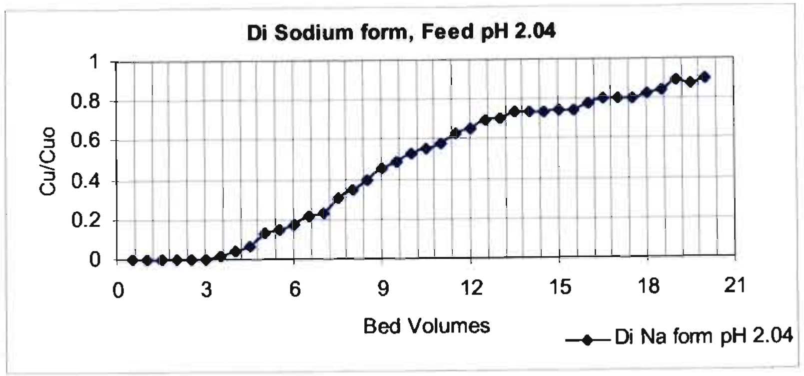


Figure G5.3: Breakthrough Curve with Initial Condition from Table G5.3

TABLE G5.4: End Conditions for Di Sodium Form, Feed pH 2.04

Output
Resin Volume = 57.2 mL [Loaded with Copper]
Volume of Eluate = 500 mL
Eluate Concentration = 4.06 g/L
Mass of Copper from Eluate = 2.03 g

Elution
Resin Volume = 50.8 mL [Hydrogen Form]
pH Resin = 2.08 [Hydrogen Form]
Loading from Elution = 35.48951 g/L

Accountability = 84.968025 %
Resin Loading [Adsorption] = 41.79 g/L
Mass Transfer Zone = 12 cm
Residence Time = 5.83 min

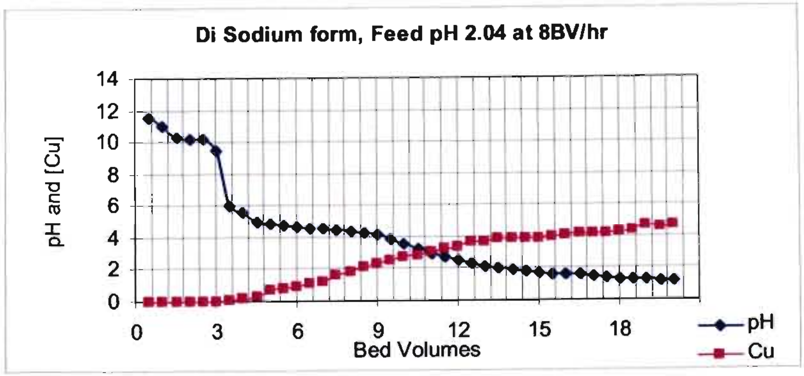


Figure G5.4: Monitoring of Copper Concentration and Effluent pH

G5.5 Mono Sodium Form, Feed pH 2.04

TABLE G5.5: Initial Conditions for Mono Sodium Form, Feed pH 2.04

Input
Column Diameter = 2.08 cm
Resin Volume = 62.8 mL of TP 207 [Mono Sodium Form]
pH of Resin = 6.39 [Mono Sodium Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.262 g/L at pH = 2.04
Overall Mass of Copper Input = 4.86735 g

TABLE G5.6: End Conditions for Mono Sodium Form, Feed pH 2.04

Output
Resin Volume = 55.2 mL [Loaded with Copper]
Volume of Eluate = 536 mL
Eluate Concentration = 3.65 g/L
Mass of Copper from Eluate =1.9564 g

Elution
Resin Volume = 50.3 mL [Hydrogen Form]
pH Resin = 2.17 [Hydrogen Form]
Loading from Elution = 35.442029 g/L

Accountability = 85.570845 %
Resin Loading [Adsorption] = 42.33 g/L
Mass Transfer Zone = 11.83 cm
Residence Time = 5.74 min

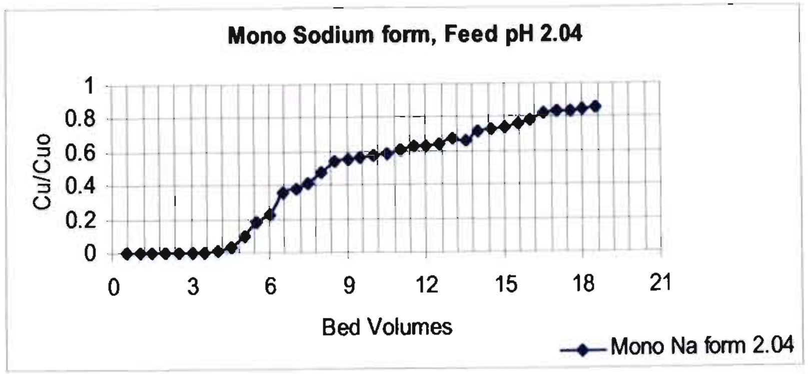


Figure G5.5: Breakthrough Curve with Initial Condition from Table G5.5

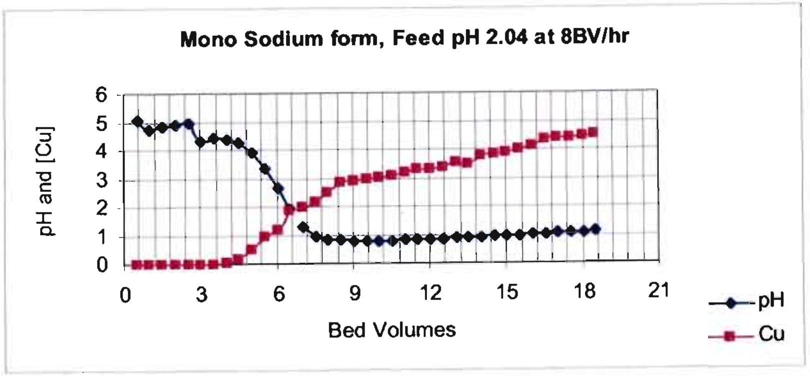


Figure G5.6: Monitoring of Copper Concentration and Effluent pH

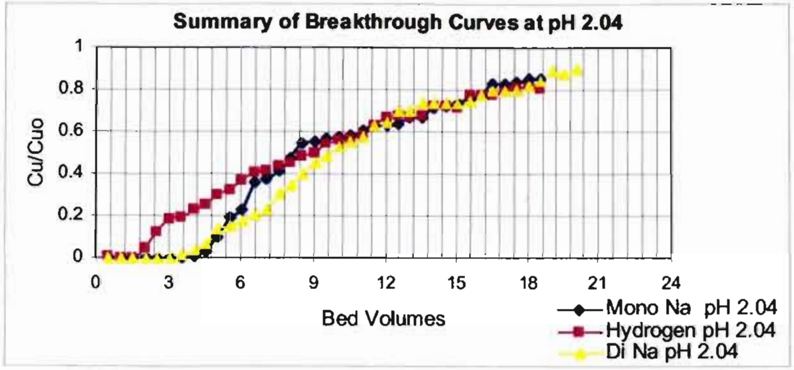


Figure G5.7: Resin in Three Forms at Initial Feed pH of 2.04

APPENDIX G6: SUMMARY OF RESULTS OBTAINED FROM FEED pH 1.59

The similar procedures and calculations that was performed in Appendix G1 to G3, were applied and the results for the mini column tests for feed pH of 1.59 with the three forms of resin are summarized in the following tables and graphs below.

G6.1 Hydrogen Form, Feed pH 1.59

TABLE G6.1: Initial Conditions for Hydrogen Form, Feed pH 1.59

Input
Column Diameter = 2.08 cm
Resin Volume = 50 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.5 [Hydrogen Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.453 g/L at pH = 1.59
Overall Mass of Copper Input = 4.63505 g

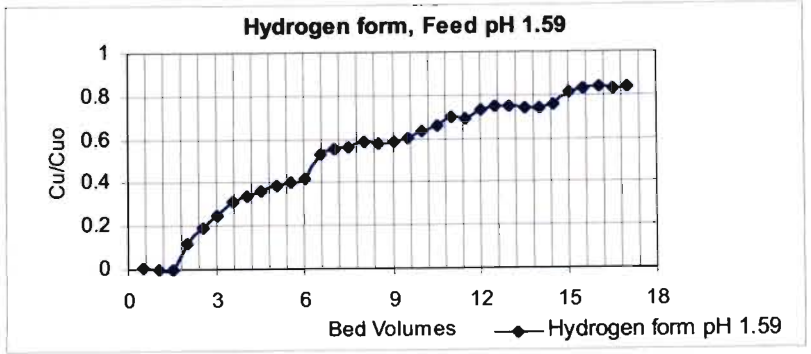


Figure G6.1: Breakthrough Curve with Initial Condition from Table G6.1

TABLE G6.2: End Conditions for Hydrogen Form, Feed pH 1.59

Output
Resin Volume = 52 mL [Loaded with Copper]
Volume of Eluate = 503 mL
Eluate Concentration = 3.2 g/L
Mass of Copper from Eluate = 1.6096g

Elution
Resin Volume = 50 mL [Hydrogen Form]
pH Resin = 2.35 [Hydrogen Form]
Loading from Elution = 30.953846 g/L

Accountability = 88.072836 %
Resin Loading [Adsorption] = 43.24 g/L
Mass Transfer Zone = 13.34 cm
Residence Time = 6.48 min

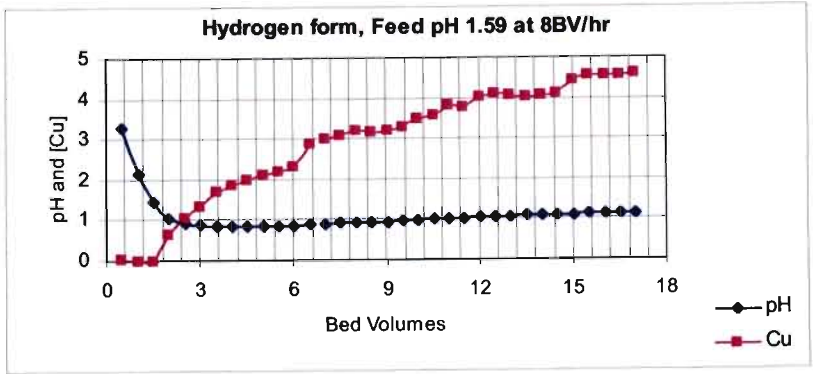


Figure G6.2: Monitoring of Copper Concentration and Effluent pH

G6.2 Di Sodium Form, Feed pH 1.59

TABLE G6.3: Initial Conditions for Di Sodium Form, Feed pH 1.59

Input
Column Diameter = 2.08 cm
Resin Volume = 67.8 mL of TP 207 [Di Sodium Form]
pH of Resin = 11.43 [Di Sodium Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.453 g/L at pH = 1.59
Overall Mass of Copper Input = 4.63505 g

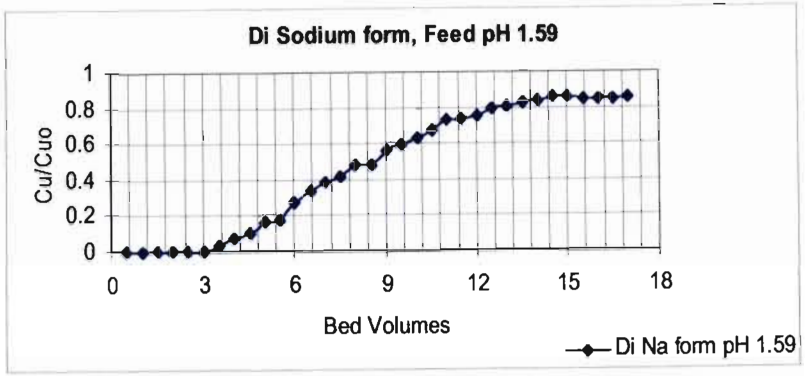


Figure G6.3: Breakthrough Curve with Initial Condition from Table G6.3

TABLE G6.4: End Conditions for Di Sodium Form, Feed pH 1.59

Output
Resin Volume = 56 mL [Loaded with Copper]
Volume of Eluate = 502.9 mL
Eluate Concentration = 3.3 g/L
Mass of Copper from Eluate = 1.65957 g

Elution
Resin Volume = 50.4 mL [Hydrogen Form]
pH Resin = 1.98 [Hydrogen Form]
Loading from Elution = 29.635179 g/L

Accountability = 82.811944 %
Resin Loading [Adsorption] = 36.22 g/L
Mass Transfer Zone = 11.28 cm
Residence Time = 5.48 min

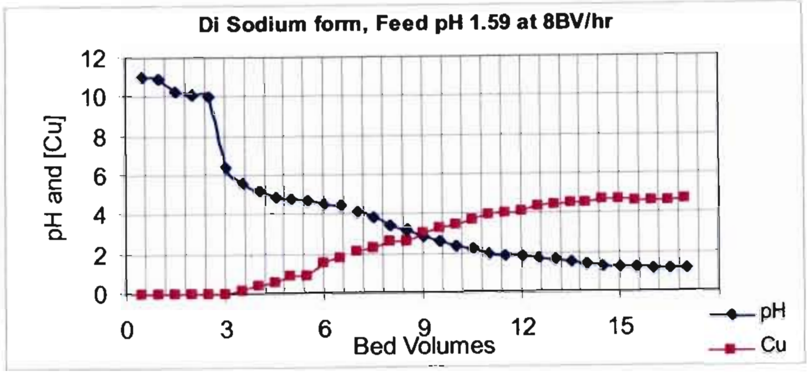


Figure G6.4: Monitoring of Copper Concentration and Effluent pH

G6.3 Mono Sodium Form, Feed pH 1.59

TABLE G6.5: Initial Conditions for Mono Sodium Form, Feed pH 1.59

Input
Column Diameter = 2.08 cm
Resin Volume = 62.9 mL of TP 207 [Mono Sodium Form]
pH of Resin = 6.37 [Mono Sodium Form]
Flow rate = 418.118 mL/hr = 8.3623 BV/hr
Linear Velocity = 0.034 cm/s
Feed Solution = 5.453 g/L at pH = 1.59
Overall Mass of Copper Input = 4.63505 g

TABLE G6.6: End Conditions for Mono Sodium Form, Feed pH 1.59

Output
Resin Volume = 55.3 mL [Loaded with Copper]
Volume of Eluate = 521.5 mL
Eluate Concentration = 2.9 g/L
Mass of Copper from Eluate = 1.51235 g

Elution
Resin Volume = 50.1 mL [Hydrogen Form]
pH Resin = 1.94 [Hydrogen Form]
Loading from Elution = 27.348101 g/L

Accountability = 84.55384 %
Resin Loading [Adsorption] = 35.42 g/L
Mass Transfer Zone = 11.09 cm
Residence Time = 5.38 min

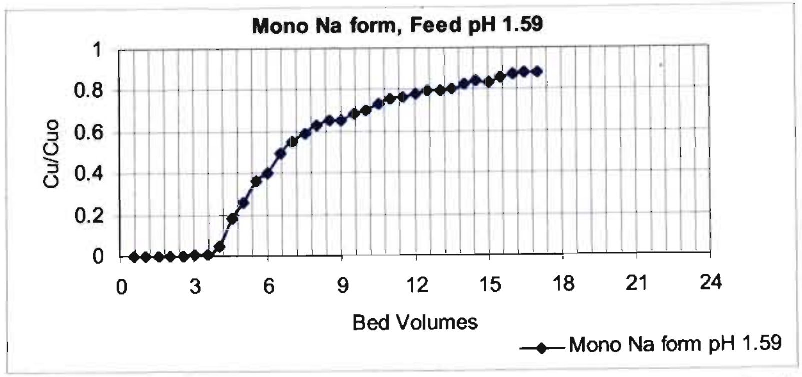


Figure G6.5: Breakthrough Curve with Initial Condition from Table G6.5

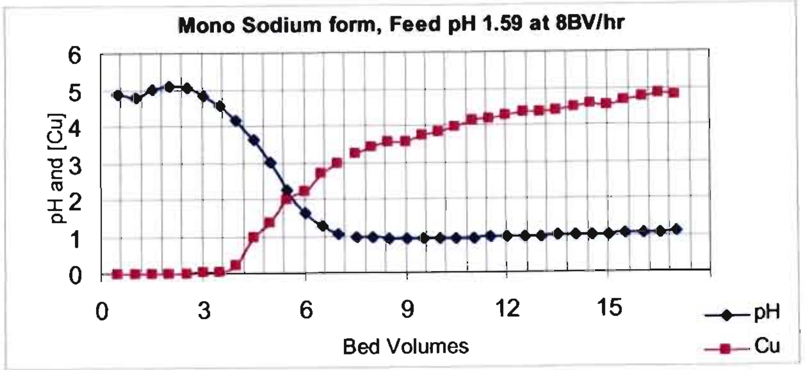


Figure G6.6: Monitoring of Copper Concentration and Effluent pH

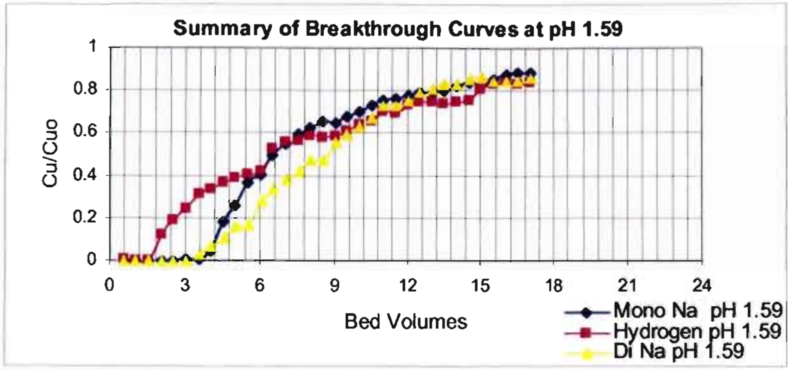


Figure G6.7: Resin in Three Forms at Initial Feed pH of 1.59

APPENDIX H: MINIMUM FLUIDIZATION VELOCITY

H1: DETERMINATION OF MINIMUM FLUIDIZATION VELOCITY H1-1

APPENDIX H1: DETERMINATION OF MINIMUM FLUIDIZATION VELOCITY

The analysis of this determination was based on the discussion present in Chapter Five, Section 5.1.2.

TABLE H1.1: Experimental Calculation of Minimum Fluidization Velocity

Volume [mL]	Time [sec]	Volume Flow Rate [cm^3/sec]	Column ID [cm]	Column Area	Void Fraction	Bed Void Area [cm^2]	Column Velocity [cm/s]	Bed Height [cm]
5	61.58	0.081195193	2.4	4.52389342	0.29	1.311929092	0.017948078	20.3
8.3	60.83	0.136445833	2.4	4.52389342	0.29	1.311929092	0.030161151	20.3
10	60.76	0.164581962	2.4	4.52389342	0.29	1.311929092	0.036380601	20.3
12	61.13	0.196302961	2.4	4.52389342	0.29	1.311929092	0.043392481	20.3
14.1	60.95	0.231337162	2.4	4.52389342	0.29	1.311929092	0.05113674	20.3
17.2	61.17	0.281183587	2.4	4.52389342	0.31	1.389139773	0.062155219	20.8
19	60.96	0.31167979	2.4	4.52389342	0.32	1.448286068	0.06889636	21.2
22	**60.95**	**0.3609516**	**2.4**	**4.52389342**	**0.34**	**1.519152597**	**0.079787821**	**21.7**
27	60.43	0.446797948	2.4	4.52389342	0.37	1.659092196	0.098764031	22.76
39.9	60.49	0.659613159	2.4	4.52389342	0.40	1.818379899	0.145806521	24.1
49	60.84	0.80539119	2.4	4.52389342	0.43	1.946704651	0.17803054	25.3
77	60	1.283333333	2.4	4.52389342	0.50	2.259904675	0.283678949	28.8
105	60.65	1.731244847	2.4	4.52389342	0.54	2.434057656	0.382689132	31.2
117	60.47	1.934843724	2.4	4.52389342	0.58	2.606161778	0.427694365	34
142	60.67	2.34053074	2.4	4.52389342	0.61	2.761653533	0.517370884	37
160	60.29	2.653839774	2.4	4.52389342	0.64	2.913945869	0.586627386	40.5
180	60.67	2.966869952	2.4	4.52389342	0.67	3.042009878	0.655822248	44
225	60.43	3.723316234	2.4	4.52389342	0.73	3.29365048	0.823033588	53

The void fraction was calculated using the Equation 5.9 present in Chapter Five, Section 5.2.2.1. From the results obtained from Table H1.1, the graph of bed height versus column velocity was plotted **(Refer to Figure H1.1).**

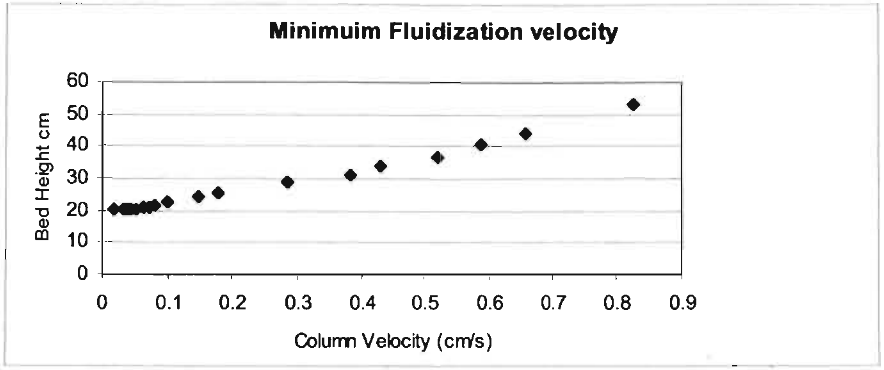


Figure H1.1: Graph of Bed Height versus Column Velocity

Minimum fluidization velocity was determined at the intersection between the straight line for the bed been fluidized and the horizontal line for the packed bed. From Figure H1.1, the minimum fluidization velocity was determined to be 0.079 cm/s. The theoretical determination of minimum fluidization and terminal velocity is present on Table H1.2.

TABLE H1.2: Theoretical Determination of Minimum Fluidization and Settling Velocity

Minimum Fluidization Velocity	0.0175 cm/s [Refer to Appendix C3]
Terminal Velocity	4.136 cm/s [Refer to Appendix C3]

From the experimental results a graph of void fraction versus column velocity can be plotted **(Figure H1.2)**.

The Richardson Zaki Correlation present in Chapter Five, Section 5.2.2, Equation 5.3, was used to model fluidization velocity as a function of void volume theoretically (Equation H1.1).

$$u = 4.1\varepsilon^{3.093} \quad \text{...} \textbf{(Equation H1.1)}$$

Figure H1.3 shows the comparison of experimental and predicted results. The "Students t-test" was performed using computer package Microsoft Excel, to calculate the standard deviation for the curves **(Refer to Table H1.3)**

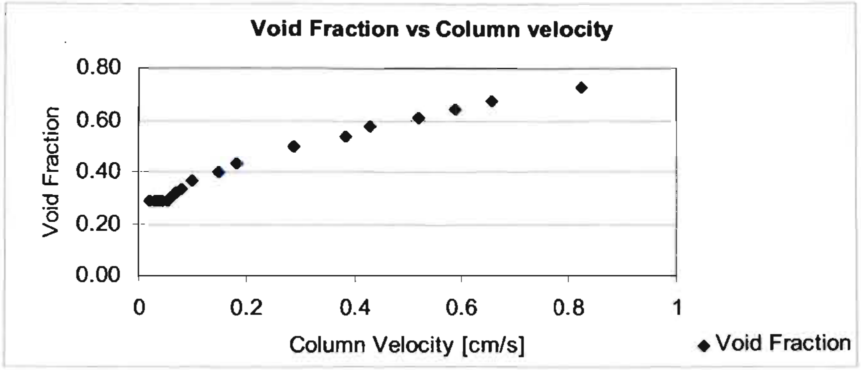


Figure H1.2: Graph of Void Fraction versus Column Velocity

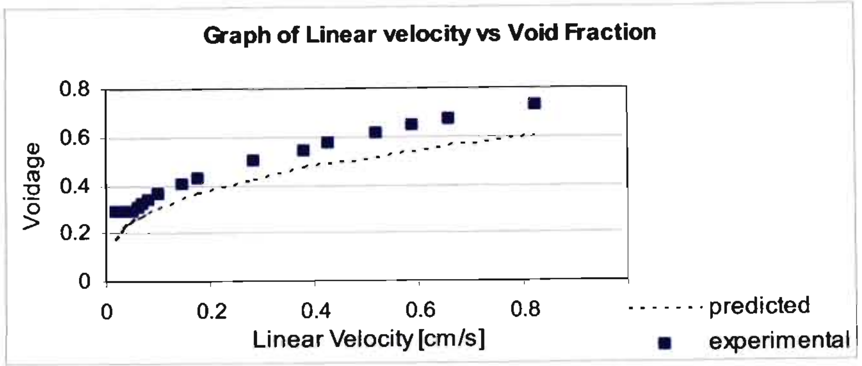


Figure H1.3: Comparison of Experimental and Predicted Curves

TABLE H1.3: t-Test (Two-Sample Assuming Equal Variances)

	Experimental	Predicted
Mean	0.437820813	0.358253445
Variance	0.02362467	0.018533028
Observations	18	18
Pooled Variance	0.021078849	
Hypothesized Mean Difference	0	
df	34	
t Statistic	1.644117321	
P(T<=t) one-tail	0.054684268	
t Critical one-tail	1.690923455	
P(T<=t) two-tail	0.109368537	
t Critical two-tail	2.032243174	

APPENDIX I: COLUMN TESTS

I1: COLUMN TEST AT LINEAR VELOCITY 0.13 cm/s G1-1

I2: COLUMN TEST AT LINEAR VELOCITY 0.19 cm/s G2-1

I3: COLUMN TEST AT LINEAR VELOCITY 0.41 cm/s G3-1

I4: MASS TRANSFER ZONE VERSES LINEAR VELOCITY G4-1

I5: FULL SCALE PLANT DESIGN FOR 0.13 cm/s G5-1

I6: FULL SCALE PLANT DESIGN FOR 0.195 cm/s G6-1

I7: FULL SCALE PLANT DESIGN FOR 0.41 cm/s G7-1

APPENDIX I1: COLUMN TEST AT LINEAR VELOCITY 0.13 CM/S

TABLE I1.1: Initial Conditions for Linear Velocity 0.13 cm/s

Input
Column Diameter = 2.4 cm
Column Height = 1 m
Resin Volume = 406 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.0 [Hydrogen Form]
Flow rate = 2196 mL/hr = 5.4 BV/hr
Linear Velocity = 0.13 cm/s
Feed Solution = 3.47 g/L at pH = 3
Overall Mass of Copper Input = 34.006 g

TABLE I1.2: End Conditions for Linear Velocity 0.13 cm/s

Output
Resin Volume = 433 mL [Loaded with Copper]
Volume of Eluate = 5100 mL
Eluate Concentration = 3.2 g/L
Mass of Copper from Eluate = 16.32 g

Elution
Resin Volume = 405 mL [Hydrogen Form]
pH Resin = 2.1 [Hydrogen Form]
Loading from Elution = 40.2962963 g/L

TABLE I1.3: Calculations for Column Tests with Initial Conditions from Table I1.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.0013	0.00037464	0.49261084	5.42	2.91	0.00026	0.694	0.69374	1.708719212	1.708719212
0.0016	0.0004611	0.98522184	10.84	2.78	0.00032	0.694	0.69368	1.708571429	3.41729064
0.0016	0.0004611	1.47783284	16.26	1.94	0.00032	0.694	0.69368	1.708571429	5.125862069
0.018	0.00518732	1.97044384	21.68	1.38	0.0036	0.694	0.6904	1.700492611	6.82635468
0.17	0.04899135	2.46305484	27.1	1.1	0.034	0.694	0.66	1.625615764	8.451970443
0.413	0.11902017	2.95566584	32.52	1	0.0826	0.694	0.6114	1.50591133	9.957881773
0.581	0.16743516	3.44827684	37.94	0.96	0.1162	0.694	0.5778	1.423152709	11.38103448
0.59	0.17002882	3.94088784	43.36	0.95	0.118	0.694	0.576	1.418719212	12.79975369
0.77	0.22190202	4.43349884	48.78	0.95	0.154	0.694	0.54	1.330049261	14.12980296
1.086	0.3129683	4.92610984	54.2	1.05	0.2172	0.694	0.4768	1.174384236	15.30418719
1.1174	0.32201729	5.41872084	59.62	1.03	0.22348	0.694	0.47052	1.158916256	16.46310345
1.247	0.35936599	5.9113318	65.04	1.02	0.2494	0.694	0.4446	1.095073892	17.55817734
1.428	0.41152738	6.4039428	70.46	1.03	0.2856	0.694	0.4084	1.00591133	18.56408867
1.563	0.45043228	6.8965538	75.88	1.04	0.3126	0.694	0.3814	0.939408867	19.50349754
1.645	0.4740634	7.3891648	81.3	1.06	0.329	0.694	0.365	0.899014778	20.40251232
1.643	0.47348703	7.8817758	86.72	1.07	0.3286	0.694	0.3654	0.9	21.30251232
1.726	0.49740634	8.3743868	92.14	1.09	0.3452	0.694	0.3488	0.8591133	22.16162562
1.833	0.52824207	8.8669978	97.56	1.1	0.3666	0.694	0.3274	0.806403941	22.96802956
1.879	0.54149856	9.3596088	102.98	1.13	0.3758	0.694	0.3182	0.783743842	23.7517734
1.904	0.54870317	9.8522198	108.4	1.15	0.3808	0.694	0.3132	0.771428571	24.52320197
2.022	0.58270893	10.344831	113.82	1.18	0.4044	0.694	0.2896	0.713300493	25.23650246
2.065	0.59510086	10.837442	119.24	1.2	0.413	0.694	0.281	0.692118227	25.92862069
2.162	0.62305476	11.330053	124.66	1.22	0.4324	0.694	0.2616	0.644334975	26.57295567
2.328	0.67089337	11.822664	130.08	1.24	0.4656	0.694	0.2284	0.562561576	27.13551724
2.304	0.66397695	12.315275	135.5	1.27	0.4608	0.694	0.2332	0.574384236	27.70990148
2.357	0.67925072	12.807886	140.92	1.29	0.4714	0.694	0.2226	0.548275862	28.25817734
2.441	0.70345821	13.300497	146.34	1.33	0.4882	0.694	0.2058	0.506896552	28.76507389
2.581	0.74380403	13.793108	151.76	1.35	0.5162	0.694	0.1778	0.437931034	29.20300493

Table I1.3: ...continued...

2.508	0.72276657	14.285719	157.18	1.38	0.5016	0.694	0.1924	0.473891626	29.67689655
2.526	0.72795389	14.77833	162.6	1.41	0.5052	0.694	0.1888	0.465024631	30.14192118
2.616	0.75389049	15.270941	168.02	1.43	0.5232	0.694	0.1708	0.420689655	30.56261084
2.678	0.77175793	15.763552	173.44	1.45	0.5356	0.694	0.1584	0.390147783	30.95275862
2.709	0.78069164	16.256163	178.86	1.47	0.5418	0.694	0.1522	0.374876847	31.32763547
2.818	0.81210375	16.748774	184.28	1.5	0.5636	0.694	0.1304	0.321182266	31.64881773
2.825	0.81412104	17.241385	189.7	1.53	0.565	0.694	0.129	0.31773399	31.96655172
2.836	0.81729107	17.733996	195.12	1.56	0.5672	0.694	0.1268	0.312315271	32.278867
2.85	0.82132565	18.226607	200.54	1.58	0.57	0.694	0.124	0.305418719	32.58428571
2.935	0.84582133	18.719218	205.96	1.61	0.587	0.694	0.107	0.263546798	32.84783251
2.889	0.83256484	19.211829	211.38	1.64	0.5778	0.694	0.1162	0.286206897	33.13403941
2.898	0.8351585	19.70444	216.8	1.66	0.5796	0.694	0.1144	0.281773399	33.41581281
2.873	0.82795389	20.197051	222.22	1.68	0.5746	0.694	0.1194	0.29408867	33.70990148
2.948	0.84956772	20.689662	227.64	1.7	0.5896	0.694	0.1044	0.257142857	33.96704433
2.975	0.8573487	21.182273	233.06	1.73	0.595	0.694	0.099	0.243842365	34.2108867
3.001	0.8648415	21.674884	238.48	1.75	0.6002	0.694	0.0938	0.231034483	34.44192118
3.103	0.8942363	22.167495	243.9	1.78	0.6206	0.694	0.0734	0.180788177	34.62270936
3.055	0.8804035	22.660106	249.32	1.8	0.611	0.694	0.083	0.204433498	34.82714286
3.032	0.8737752	23.152717	254.74	1.83	0.6064	0.694	0.0876	0.215763547	35.0429064
3.116	0.8979827	23.645328	260.16	1.85	0.6232	0.694	0.0708	0.174384236	35.21729064
3.036	0.874928	24.137939	265.58	1.87	0.6072	0.694	0.0868	0.213793103	35.43108374
Cumulative Total					**19.62098**			**35.43108374**	

Accountability = 105.69011 %
Resin Loading [Adsorption] = 35.43108 g/L
Mass Transfer Zone = 80 cm
Residence Time = 10.3 min

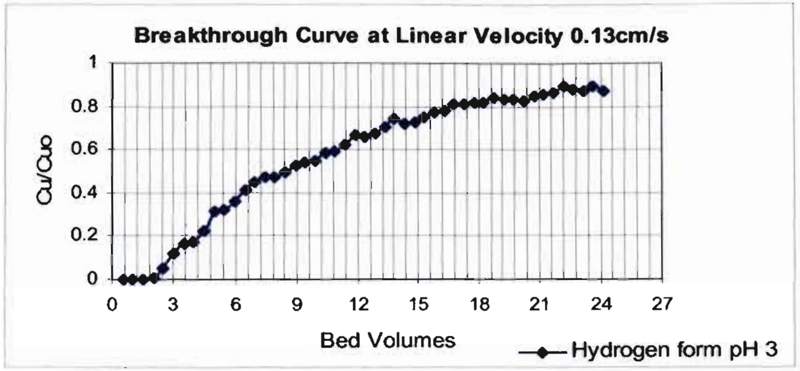


Figure I1.1: Breakthrough Curve with Initial Condition from Table I1.1

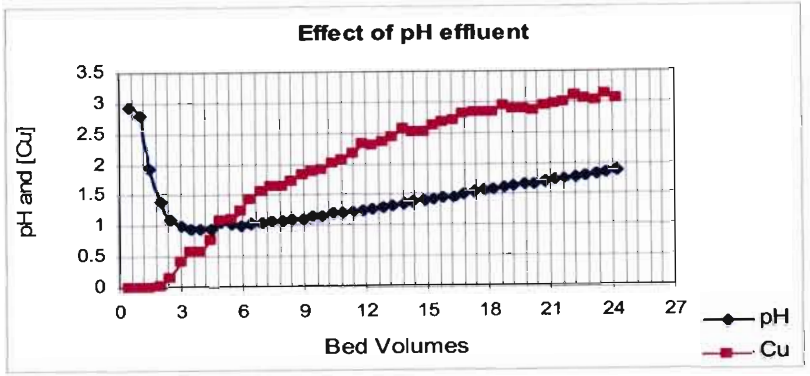


Figure I1.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX I2: COLUMN TEST AT LINEAR VELOCITY 0.195 CM/S

TABLE I2.1: Initial Conditions for Linear Velocity 0.195 cm/s

Input
Column Diameter = 2.4 cm
Column Height = 1 m
Number of Columns = 2 in Series
Resin Volume = 814 mL of TP 207 [Hydrogen Form]
pH of Resin = 1.88 [Hydrogen Form]
Flow rate = 3183.5 mL/hr = 3.9 BV/hr
Linear Velocity = 0.195 cm/s
Feed Solution = 3.603 g/L at pH = 2.86
Overall Mass of Copper Input = 72.06 g

TABLE I2.2: End Conditions for Linear Velocity 0.1 cm/s

Output
Resin Volume = 865 mL [Loaded with Copper]
Volume of Eluate = 9928 mL
Eluate Concentration = 3.40 g/L
Mass of Copper from Eluate = 33.752 g

Elution
Resin Volume = 810 mL [Hydrogen Form]
pH Resin = 2.27 [Hydrogen Form]
Loading from Elution = 41.67309 g/L

TABLE I2.3: Calculations for Column Tests with Initial Conditions from Table I2.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.0013	0.0003608	0.4914	7.54	3.13	0.00052	1.4412	1.44068	1.76987715	1.76987715
0.0016	0.0004441	0.9828	15.08	2.8	0.00064	1.4412	1.44056	1.76972973	3.53960688
0.0017	0.0004718	1.4742	22.62	1.75	0.00068	1.4412	1.44052	1.76968059	5.309287469
0.0004	0.000111	1.9656	30.16	1.19	0.00016	1.4412	1.44104	1.77031941	7.07960688
0.0775	0.0215099	2.457	37.7	0.86	0.031	1.4412	1.4102	1.732432432	8.812039312
0.216	0.05995	2.9484	45.24	0.73	0.0864	1.4412	1.3548	1.664373464	10.47641278
0.406	0.1126839	3.4398	52.78	0.7	0.1624	1.4412	1.2788	1.571007371	12.04742015
0.38	0.1054677	3.9312	60.32	0.69	0.152	1.4412	1.2892	1.583783784	13.63120393
0.57	0.1582015	4.4226	67.86	0.69	0.228	1.4412	1.2132	1.49041769	15.12162162
1.033	0.2867055	4.914	75.4	0.68	0.4132	1.4412	1.028	1.262899263	16.38452088
1.149	0.3189009	5.4054	82.94	0.7	0.4596	1.4412	0.9816	1.205896806	17.59041769
1.296	0.3597002	5.8968	90.48	0.71	0.5184	1.4412	0.9228	1.133660934	18.72407862
1.475	0.4093811	6.3882	98.02	0.71	0.59	1.4412	0.8512	1.045700246	19.76977887
1.607	0.4460172	6.8796	105.56	0.73	0.6428	1.4412	0.7984	0.980835381	20.75061425
1.707	0.473772	7.371	113.1	0.75	0.6828	1.4412	0.7584	0.931695332	21.68230958
1.769	0.49098	7.8624	120.64	0.77	0.7076	1.4412	0.7336	0.901228501	22.58353808
1.931	0.535942	8.3538	128.18	0.79	0.7724	1.4412	0.6688	0.821621622	23.40515971
2.013	0.558701	8.8452	135.72	0.82	0.8052	1.4412	0.636	0.781326781	24.18648649
2.1	0.582848	9.3366	143.26	0.85	0.84	1.4412	0.6012	0.738574939	24.92506143
2.204	0.611712	9.828	150.8	0.87	0.8816	1.4412	0.5596	0.687469287	25.61253071
2.23	0.618929	10.3194	158.34	0.89	0.892	1.4412	0.5492	0.674692875	26.28722359
2.424	0.672773	10.8108	165.88	0.91	0.9696	1.4412	0.4716	0.579361179	26.86658477
2.451	0.680266	11.3022	173.42	0.94	0.9804	1.4412	0.4608	0.566093366	27.43267813
2.452	0.680544	11.7936	180.96	0.96	0.9808	1.4412	0.4604	0.565601966	27.9982801
2.588	0.71829	12.285	188.5	0.99	1.0352	1.4412	0.406	0.498771499	28.4970516
2.599	0.721343	12.7764	196.04	1.02	1.0396	1.4412	0.4016	0.493366093	28.99041769
2.701	0.749653	13.2678	203.58	1.05	1.0804	1.4412	0.3608	0.443243243	29.43366093
2.73	0.757702	13.7592	211.12	1.08	1.092	1.4412	0.3492	0.428992629	29.86265356

Table I2.3: ...continued...

2.763	0.766861	14.2506	218.66	1.11	1.1052	1.4412	0.336	0.412776413	30.27542998
2.825	0.784069	14.742	226.2	1.16	1.13	1.4412	0.3112	0.382309582	30.65773956
2.898	0.80433	15.2334	233.74	1.19	1.1592	1.4412	0.282	0.346437346	31.0041769
3.062	0.849847	15.7248	241.28	1.22	1.2248	1.4412	0.2164	0.265847666	31.27002457
3.047	0.845684	16.2162	248.82	1.25	1.2188	1.4412	0.2224	0.273218673	31.54324324
2.978	0.826533	16.7076	256.36	1.28	1.1912	1.4412	0.25	0.307125307	31.85036855
3.064	0.850402	17.199	263.9	1.31	1.2256	1.4412	0.2156	0.264864865	32.11523342
3.156	0.875937	17.6904	271.44	1.33	1.2624	1.4412	0.1788	0.21965602	32.33488943
3.132	0.869276	18.1818	278.98	1.36	1.2528	1.4412	0.1884	0.231449631	32.56633907
3.164	0.878157	18.6732	286.52	1.38	1.2656	1.4412	0.1756	0.215724816	32.78206388
3.169	0.879545	19.1646	294.06	1.42	1.2676	1.4412	0.1736	0.213267813	32.9953317
3.247	0.901193	19.656	301.6	1.45	1.2988	1.4412	0.1424	0.174938575	33.17027027
3.358	0.932001	20.1474	309.14	1.48	1.3432	1.4412	0.098	0.12039312	33.29066339
3.211	0.891202	20.6388	316.68	1.53	1.2844	1.4412	0.1568	0.192628993	33.48329238
3.309	0.918401	21.1302	324.22	1.55	1.3236	1.4412	0.1176	0.144471744	33.62776413
3.262	0.905357	21.6216	331.76	1.57	1.3048	1.4412	0.1364	0.167567568	33.7953317
3.502	0.971968	22.113	339.3	1.6	1.4008	1.4412	0.0404	0.04963145	33.84496314
3.329	0.923952	22.6044	346.84	1.62	1.3316	1.4412	0.1096	0.134643735	33.97960688
3.443	0.955593	23.0958	354.38	1.64	1.3772	1.4412	0.064	0.078624079	34.05823096
3.515	0.9755759	23.5872	361.92	1.67	1.406	1.4412	0.0352	0.043243243	34.1014742
3.483	0.9666944	24.0786	369.46	1.69	1.3932	1.4412	0.048	0.058968059	34.16044226
3.553	0.9861227	24.57	377	1.71	1.4212	1.4412	0.02	0.024570025	34.18501229
Cumulative Total					**44.2334**			**34.18501229**	

Accountability = 108.2273 %
Resin Loading [Adsorption] = 34.18 g/L
Mass Transfer Zone = 153.998 cm
Residence Time = 13.2 min

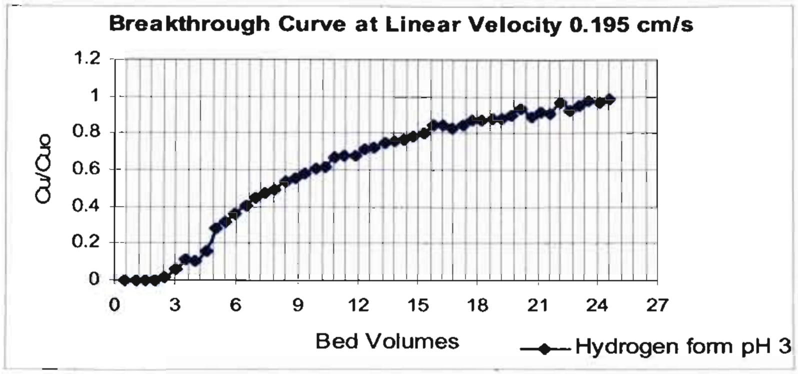


Figure I2.1: Breakthrough Curve with Initial Condition from Table I2.1

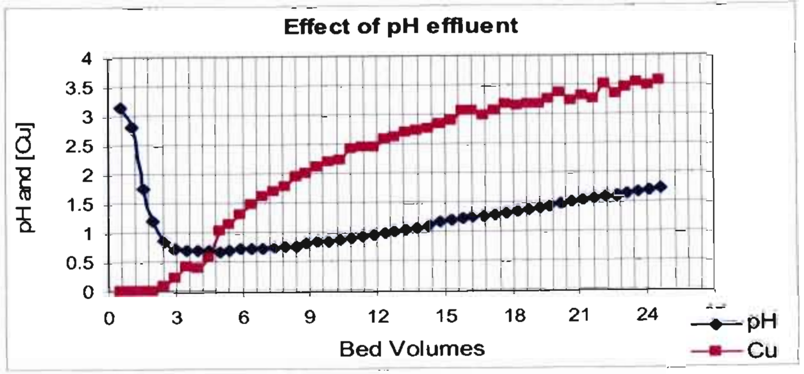


Figure I2.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX I3: COLUMN TEST AT LINEAR VELOCITY 0.41 CM/S

TABLE I3.1: Initial Conditions for Linear Velocity 0.41 cm/s

Input
Column Diameter = 2.4 cm
Column Height = 4 m
Number of Columns = 4 in Series
Resin Volume = 1626 mL of TP 207 [Hydrogen Form]
pH of Resin = 1.8 [Hydrogen Form]
Flow rate = 6652.17 mL/hr = 3.9 BV/hr
Linear Velocity = 0.41 cm/s
Feed Solution = 3.792 g/L at pH = 2.86
Overall Mass of Copper Input = 130.4448 g

TABLE I3.2: End Conditions for Linear Velocity 0.41 cm/s

Output
Resin Volume = 1750 mL [Loaded with Copper]
Volume of Eluate = 18040 mL
Eluate Concentration = 3.493 g/L
Mass of Copper from Eluate = 63.041 g

Elution
Resin Volume = 1620 mL [Hydrogen Form]
pH Resin = 2.52 [Hydrogen Form]
Loading from Elution = 38.89753086 g/L

TABLE I3.3: Calculations for Column Tests with Initial Conditions from Table I3.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.00063	0.00016614	0.49200492	7.22	1.78	0.000504	3.0336	3.033096	1.865372694	1.865372694
0.00125	0.00032964	0.98400992	14.44	1.85	0.001	3.0336	3.0326	1.865067651	3.730440344
0.00028	7.384E-05	1.47601492	21.66	1.87	0.000224	3.0336	3.033376	1.865544895	5.59598524
0.00119	0.00031382	1.96801992	28.88	1.48	0.000952	3.0336	3.032648	1.865097171	7.461082411
0.03341	0.00881065	2.46002492	36.1	1.02	0.026728	3.0336	3.006872	1.849244772	9.310327183
0.1234	0.03254219	2.95202992	43.32	0.96	0.09872	3.0336	2.93488	1.80496925	11.11529643
0.2891	0.07623945	3.44403492	50.54	0.84	0.23128	3.0336	2.80232	1.723444034	12.83874047
0.591	0.15585443	3.93603992	57.76	0.92	0.4728	3.0336	2.5608	1.574907749	14.41364822
0.721	0.19013713	4.42804492	64.98	0.9	0.5768	3.0336	2.4568	1.510947109	15.92459533
0.983	0.25922996	4.92004992	72.2	0.88	0.7864	3.0336	2.2472	1.38204182	17.30663715
1.043	0.27505274	5.41205492	79.42	0.94	0.8344	3.0336	2.1992	1.352521525	18.65915867
1.169	0.30828059	5.90405992	86.64	0.97	0.9352	3.0336	2.0984	1.290528905	19.94968758
1.316	0.3470464	6.3960649	93.86	0.93	1.0528	3.0336	1.9808	1.218204182	21.16789176
1.506	0.3971519	6.8880699	101.08	1	1.2048	3.0336	1.8288	1.124723247	22.29261501
1.594	0.4203586	7.3800749	108.3	1.01	1.2752	3.0336	1.7584	1.081426814	23.37404182
1.679	0.4427743	7.8720799	115.52	1.04	1.3432	3.0336	1.6904	1.039606396	24.41364822
1.815	0.4786392	8.3640849	122.74	1.05	1.452	3.0336	1.5816	0.972693727	25.38634194
1.931	0.50923	8.8560899	129.96	1.08	1.5448	3.0336	1.4888	0.915621156	26.3019631
2.056	0.5421941	9.3480949	137.18	1.08	1.6448	3.0336	1.3888	0.854120541	27.15608364
2.134	0.5627637	9.8400999	144.4	1.08	1.7072	3.0336	1.3264	0.815744157	27.9718278
2.187	0.5767405	10.332105	151.62	1.11	1.7496	3.0336	1.284	0.789667897	28.76149569
2.259	0.5957278	10.82411	158.84	1.13	1.8072	3.0336	1.2264	0.754243542	29.51573924
2.381	0.6279008	11.316115	166.06	1.15	1.9048	3.0336	1.1288	0.694218942	30.20995818
2.54	0.6698312	11.80812	173.28	1.19	2.032	3.0336	1.0016	0.61599016	30.82594834
2.589	0.6827532	12.300125	180.5	1.21	2.0712	3.0336	0.9624	0.591881919	31.41783026
2.595	0.6843354	12.79213	187.72	1.24	2.076	3.0336	0.9576	0.588929889	32.00676015
2.596	0.6845992	13.284135	194.94	1.28	2.0768	3.0336	0.9568	0.588437884	32.59519803
2.695	0.7107068	13.77614	202.16	1.3	2.156	3.0336	0.8776	0.539729397	33.13492743

2.797	0.7376055	14.268145	209.38	1.33	2.2376	3.0336	0.796	0.489544895	33.62447232
2.928	0.7721519	14.76015	216.6	1.36	2.3424	3.0336	0.6912	0.425092251	34.04956458
2.95	0.7779536	15.252155	223.82	1.39	2.36	3.0336	0.6736	0.414268143	34.46383272
2.988	0.7879747	15.74416	231.04	1.41	2.3904	3.0336	0.6432	0.395571956	34.85940467
3.049	0.8040612	16.236165	238.26	1.45	2.4392	3.0336	0.5944	0.365559656	35.22496433
3.09	0.8148734	16.72817	245.48	1.47	2.472	3.0336	0.5616	0.345387454	35.57035178
3.107	0.8193565	17.220175	252.7	1.5	2.4856	3.0336	0.548	0.33702337	35.90737515
3.183	0.8393987	17.71218	259.92	1.53	2.5464	3.0336	0.4872	0.299630996	36.20700615
3.232	0.8523207	18.204185	267.14	1.57	2.5856	3.0336	0.448	0.275522755	36.48252891
3.335	0.8794831	18.69619	274.36	1.59	2.668	3.0336	0.3656	0.224846248	36.70737515
3.326	0.8771097	19.188195	281.58	1.6	2.6608	3.0336	0.3728	0.229274293	36.93664945
3.306	0.8718354	19.6802	288.8	1.66	2.6448	3.0336	0.3888	0.239114391	37.17576384
3.394	0.8950422	20.172205	296.02	1.69	2.7152	3.0336	0.3184	0.195817958	37.3715818
3.386	0.8929325	20.66421	303.24	1.73	2.7088	3.0336	0.3248	0.199753998	37.57133579
3.423	0.9026899	21.156215	310.46	1.74	2.7384	3.0336	0.2952	0.181549815	37.75288561
Cumulative Total					**69.05860**			**37.75288561**	

Accountability = 101.2454 %
Resin Loading [Adsorption] = 37.75 g/L
Mass Transfer Zone = 304.533 cm
Residence Time = 12.38 min

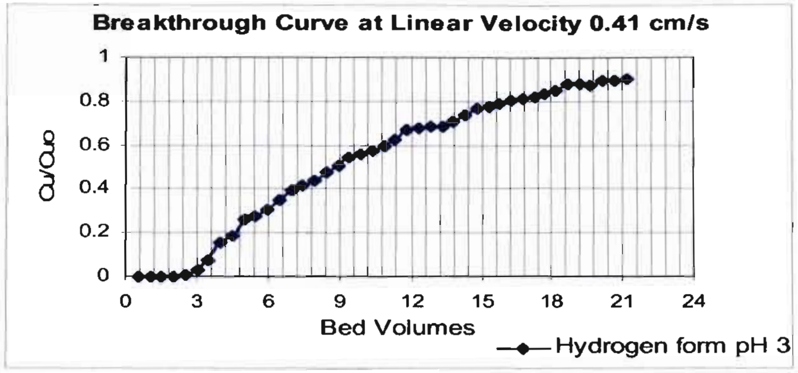


Figure I3.1: Breakthrough Curve with Initial Condition from Table I3.1

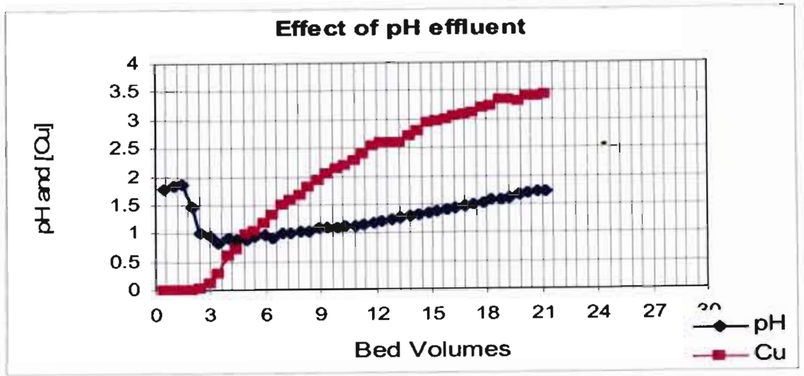


Figure I3.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX I4: MASS TRANSFER ZONE VERSES LINEAR VELOCITY

TABLE I4.1: Summary of Results obtained from Column Tests

Mass Transfer Zone [cm]	Linear Velocity [cm/s]
80	0.13
153.998	0.195
304.533	0.41

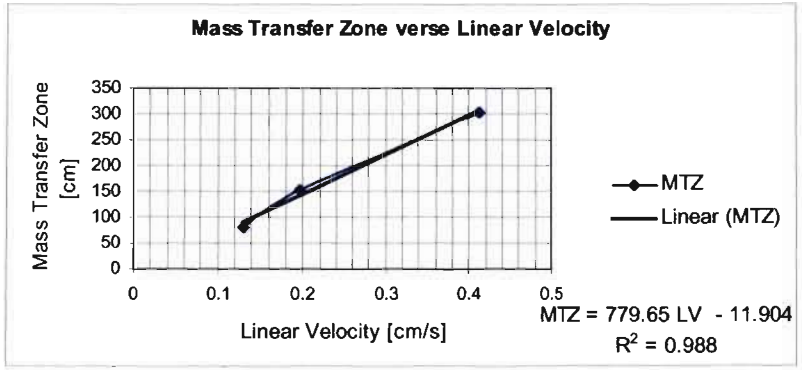


Figure I4.1: Graph of Mass Transfer Zone versus Linear Velocity

APPENDIX I5: FULL SCALE PLANT DESIGN FOR 0.13 CM/S

	Full Scale Plant Design		
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	4.68
	Feed: Copper Concentration	g/L	3
	Resin Copper Loading	g/L	40
	Residence Time	min	10.2
		hours	0.17
	Resin Exhaustion Rate	m^3/hr	0.375
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer Height	m	**0.788**
	Resin Bed Depth	m^3	0.833333
	Resin Elution	m^3	1.125
	Overall Resin Bed Depth Required	m^3	1.958333
Plant Size:	Column: Diameter	m	1.166318
	Column: Area	m^2	1.068376
	Column: Resin Bed Height	m	1.833
	Column: Height	m	2.0163
	H:D		1.571612
Adsorption:	Estimated Transfer Time	hours	5.25
	Estimated Number of Bed Volumes Treated	BV	13.35
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
	Pilot Plant Design		
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	1
Input:	Feed: Flow rate	ml/min	36.6
		BV/hr	5.4
	Feed: Linear Velocity	cm/s	0.13
		m/hr	4.68

APPENDIX I6: FULL SCALE PLANT DESIGN FOR 0.195 CM/S

Full Scale Plant Design			
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	7.02
	Feed: Copper Concentration	g/L	3
	Resin Copper Loading	g/L	40
	Residence Time	min	13.2
		hours	0.22
	Resin Exhaustion Rate	m^3/hr	0.375
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**1.54**
	Resin Bed Depth	m^3	1.096866
	Resin Elution	m^3	1.125
	Overall Resin Bed Depth Required	m^3	2.221866
Plant Size:	Column: Diameter	m	0.952295
	Column: Area	m^2	0.712251
	Column: Resin Bed Height	m	3.1195
	Column: Height	m	3.43145
	H:D		3.275771
Adsorption:	Estimated Transfer Time	hours	5.92
	Estimated Number of Bed Volumes Treated	BV	13.3
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
Pilot Plant Design			
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	2
Input:	Feed: Flow rate	ml/min	53.06
		BV/hr	3.9
	Feed: Linear Velocity	cm/s	0.195
		m/hr	7.02

APPENDIX I7: FULL SCALE PLANT DESIGN FOR 0.41 CM/S

Full Scale Plant Design			
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	14.76
	Feed: Copper Concentration	g/L	3
	Resin Copper Loading	g/L	40
	Residence Time	min	12.38
		hours	0.20633333
	Resin Exhaustion Rate	m^3/hr	0.375
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**3.05**
	Resin Bed Depth	m^3	1.03161585
	Resin Elution	m^3	1.125
	Overall Resin Bed Depth Required	m^3	2.15661585
Plant Size:	Column: Diameter	m	0.65674516
	Column: Area	m^2	0.33875339
	Column: Resin Bed Height	m	6.36633
	Column: Height	m	7.002963
	H:D		9.69376002
Adsorption:	Estimated Transfer Time	hours	5.92
	Estimated Number of Bed Volumes Treated	BV	13.333
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
Pilot Plant Design			
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	4
Input:	Feed: Flow rate	ml/min	110.9
		BV/hr	4.1
	Feed: Linear Velocity	cm/s	0.41
		m/hr	14.76

APPENDIX J: COMPARISON OF CONTACTING EQUIPMENT AT 6 g/L

J1: FIXED BED ION EXCHANGE FOR CONCENTRATION AT 6 g/L J1-1

J2: FLUIDIZED BED ION EXCHANGE FOR CONCENTRATION AT 6 g/L J2-1

J3: COMPARISON OF CONTACTING EQUIPMENT FOR 6 g/L J3-1

APPENDIX J: COMPARISON OF CONTACTING EQUIPMENT AT 6 g/L

J1: FIXED BED ION EXCHANGE FOR CONCENTRATION AT 6 g/L J1-1

J2: FLUIDIZED BED ION EXCHANGE FOR CONCENTRATION AT 6 g/L J2-1

J3: COMPARISON OF CONTACTING EQUIPMENT FOR 6 g/L J3-1

APPENDIX J1: FIXED BED ION EXCHANGE FOR CONCENTRATION 6 g/L

TABLE J1.1: Initial Conditions for Fixed Bed at 6 g/L

Input
Column Diameter = 2.4 cm
Column Height = 1 m
Resin Volume = 406 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.0 [Hydrogen Form]
Flow rate = 2196 mL/hr = 5.4 BV/hr
Linear Velocity = 0.13 cm/s
Feed Solution = 6.3 g/L at pH = 3
Overall Mass of Copper Input = 61.74 g

TABLE J1.2: End Conditions for Fixed Bed at 6 g/L

Output
Resin Volume = 433 mL [Loaded with Copper]
Volume of Eluate = 5100 mL
Eluate Concentration = 5.1 g/L
Mass of Copper from Eluate = 26.01 g

Elution
Resin Volume = 405 mL [Hydrogen Form]
pH Resin = 2.1 [Hydrogen Form]
Loading from Elution = 64.22 g/L

TABLE J1.3: Calculations for Fixed Bed Test with Initial Conditions from Table J1.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.0023602	0.00037464	0.4926108	5.42	2.91	0.000472	1.26	1.259527954	3.102285601	3.102285601
0.0029049	0.000461095	0.9852216	10.84	2.78	0.000581	1.26	1.25941902	3.102017291	6.204302892
0.0029049	0.000461095	1.4778324	16.26	1.94	0.000581	1.26	1.25941902	3.102017291	9.306320183
0.0326801	0.00518732	1.9704432	21.68	1.38	0.006536	1.26	1.253463977	3.087349697	12.39366988
0.3086455	0.048991354	2.463054	27.1	1.1	0.0617291	1.26	1.198270893	2.951406141	15.34507602
0.7498271	0.119020173	2.9556648	32.52	1	0.1499654	1.26	1.110034582	2.734075325	18.07915135
1.0548415	0.167435159	3.4482756	37.94	0.96	0.2109683	1.26	1.0490317	2.583821922	20.66297327
1.0711816	0.170028818	3.9408864	43.36	0.95	0.2142363	1.26	1.045763689	2.575772632	23.2387459
1.3979827	0.221902017	4.4334972	48.78	0.95	0.2795965	1.26	0.980403458	2.414786843	25.65353274
1.9717003	0.3129683	4.926108	54.2	1.05	0.3943401	1.26	0.865659942	2.132167346	27.78570009
2.0287089	0.322017291	5.4187188	59.62	1.03	0.4057418	1.26	0.854258213	2.104084269	29.88978436
2.2640058	0.359365994	5.9113296	65.04	1.02	0.4528012	1.26	0.807198847	1.988174501	31.87795886
2.5926225	0.411527378	6.4039406	70.46	1.03	0.5185245	1.26	0.741475504	1.826294346	33.7042532
2.8377233	0.450432277	6.8965516	75.88	1.04	0.5675447	1.26	0.692455331	1.705555003	35.40980821
2.9865994	0.474063401	7.3891626	81.3	1.06	0.5973199	1.26	0.662680115	1.632217033	37.04202524
2.9829683	0.473487032	7.8817736	86.72	1.07	0.5965937	1.26	0.66340634	1.634005764	38.676031
3.1336599	0.49740634	8.3743846	92.14	1.09	0.626732	1.26	0.633268012	1.559773427	40.23580443
3.3279251	0.528242075	8.8669956	97.56	1.1	0.665585	1.26	0.594414986	1.464076319	41.69988075
3.4114409	0.541498559	9.3596066	102.98	1.13	0.6822882	1.26	0.577711816	1.422935506	43.12281626
3.45683	0.54870317	9.8522176	108.4	1.15	0.691366	1.26	0.568634006	1.400576369	44.52339263
3.6710663	0.582708934	10.344829	113.82	1.18	0.7342133	1.26	0.525786744	1.29504124	45.81843387
3.7491354	0.595100865	10.83744	119.24	1.2	0.7498271	1.26	0.510172911	1.256583524	47.07501739
3.925245	0.623054755	11.330051	124.66	1.22	0.785049	1.26	0.474951009	1.169830071	48.24484746
4.2266282	0.670893372	11.822662	130.08	1.24	0.8453256	1.26	0.414674352	1.021365398	49.26621286
4.1830548	0.663976945	12.315273	135.5	1.27	0.836611	1.26	0.423389049	1.04283017	50.30904303
4.2792795	0.67925072	12.807884	140.92	1.29	0.8558559	1.26	0.404144092	0.995428799	51.30447183
4.4317867	0.703458213	13.300495	146.34	1.33	0.8863573	1.26	0.373642651	0.920302097	52.22477392
4.6859654	0.743804035	13.793106	151.76	1.35	0.9371931	1.26	0.322806916	0.795090927	53.01986485
4.5534294	0.722766571	14.285717	157.18	1.38	0.9106859	1.26	0.349314121	0.860379608	53.88024446

Table J1.3: ...continued...

4.5861095	0.72795389	14.778328	162.6	1.41	0.9172219	1.26	0.342778098	0.84428103	54.72452549
4.7495101	0.75389049	15.270939	168.02	1.43	0.949902	1.26	0.310097983	0.763788135	55.48831362
4.8620749	0.771757925	15.76355	173.44	1.45	0.972415	1.26	0.287585014	0.708337474	56.1966511
4.9183573	0.780691643	16.256161	178.86	1.47	0.9836715	1.26	0.27632853	0.680612143	56.87726324
4.9201729	0.780979827	16.748772	184.28	1.5	0.9840346	1.26	0.275965418	0.679717778	57.55698102
4.9564841	0.786743516	17.241383	189.7	1.53	0.9912968	1.26	0.26870317	0.661830468	58.21881149
4.9383285	0.783861671	17.733994	195.12	1.56	0.9876657	1.26	0.272334294	0.670774123	58.88958561
4.9201729	0.780979827	18.226605	200.54	1.58	0.9840346	1.26	0.275965418	0.679717778	59.56930339
5.3286744	0.845821326	18.719216	205.96	1.61	1.0657349	1.26	0.19426513	0.478485541	60.04778893
5.2451585	0.832564841	19.211827	211.38	1.64	1.0490317	1.26	0.2109683	0.519626354	60.56741528
5.2614986	0.835158501	19.704438	216.8	1.66	1.0522997	1.26	0.207700288	0.511577064	61.07899235
5.2161095	0.82795389	20.197049	222.22	1.68	1.0432219	1.26	0.216778098	0.533936202	61.61292855
5.3522767	0.849567723	20.68966	227.64	1.7	1.0704553	1.26	0.189544669	0.46685879	62.07978734
5.4012968	0.857348703	21.182271	233.06	1.73	1.0802594	1.26	0.179740634	0.442710921	62.52249826
5.4485014	0.864841499	21.674882	238.48	1.75	1.0897003	1.26	0.170299712	0.419457418	62.94195568
5.6336888	0.894236311	22.167493	243.9	1.78	1.1267378	1.26	0.133262248	0.328232138	63.27018782
5.5465418	0.880403458	22.660104	249.32	1.8	1.1093084	1.26	0.150691643	0.371161681	63.6413495
5.5047839	0.873775216	23.152715	254.74	1.83	1.1009568	1.26	0.159043228	0.391732088	64.03308159
5.6572911	0.897982709	23.645326	260.16	1.85	1.1314582	1.26	0.128541787	0.316605386	64.34968697
5.5120461	0.874927954	24.137937	265.58	1.87	1.1024092	1.26	0.157590778	0.388154626	64.7378416
Cumulative Total					**35.456436**			**64.7378416**	

Accountability = 99.55691 %
Resin Loading [Adsorption] = 64.33 g/L
Mass Transfer Zone = 80 cm
Residence Time = 10.3 min

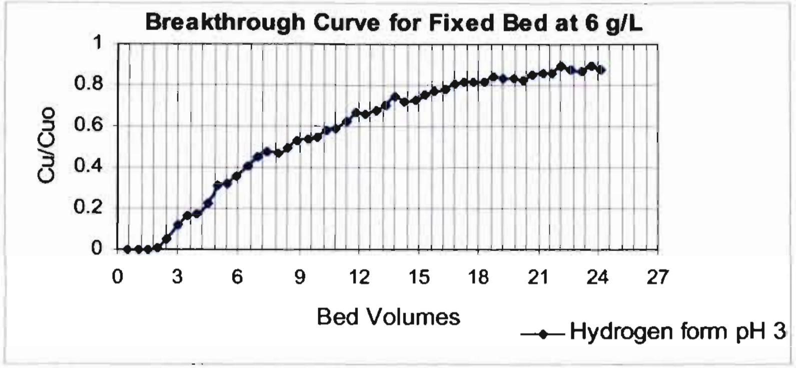


Figure J1.1: Breakthrough Curve with Initial Condition from Table J1.1

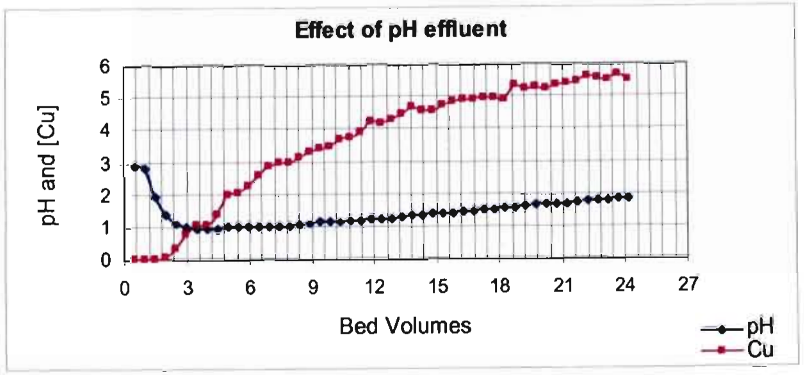


Figure J1.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX J2: FLUIDIZED BED ION EXCHANGE FOR CONCENTRATION 6 g/L

TABLE J2.1: Initial Conditions for Fluidized Bed at 6 g/L

Input
Column Diameter = 2.4 cm
Column Height = 2 m
Resin Volume = 408 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.04 [Hydrogen Form]
Static Bed Height = 872 mm
Bed Height at 0.139 cm/s = 1045 mm
Flow rate = 2307.7 mL/hr = 5.6 BV/hr
Linear Velocity = 0.139 cm/s
Feed Solution = 5.84 g/L at pH = 2.97
Overall Mass of Copper Input = 55.46 g

TABLE J2.2: End Conditions for Fluidized Bed at 6 g/L

Output
Resin Volume = 420 mL [Loaded with Copper]
Volume of Eluate = 3973 mL
Eluate Concentration = 5.9 g/L
Mass of Copper from Eluate = 23.4407 g

Elution
Resin Volume = 405 mL [Hydrogen Form]
pH Resin = 2.1 [Hydrogen Form]
Loading from Elution = 57.452696 g/L

TABLE J2.3: Calculations for Fluidized Bed Test with Initial Conditions from Table J2.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.001026	0.00017568	0.4901961	5.12	3.66	0.0002052	1.168	1.167794805	2.86224217	2.86224217
0.0110644	0.001894592	0.9803922	10.24	2.04	0.0022129	1.168	1.165787117	2.857321365	5.719563535
0.0492869	0.008439545	1.4705882	15.36	1.35	0.0098574	1.168	1.158142611	2.838584831	8.558148366
0.2506593	0.042921116	1.9607843	20.48	1.05	0.0501319	1.168	1.117868136	2.739872883	11.29802125
0.4807992	0.082328626	2.4509804	25.6	0.88	0.0961598	1.168	1.071840165	2.627059229	13.92508048
1.0118911	0.173269032	2.9411765	30.72	0.77	0.2023782	1.168	0.965621771	2.366720026	16.2918005
1.3498588	0.2311402	3.4313725	35.84	0.72	0.2699718	1.168	0.898028247	2.201049624	18.49285013
1.7622597	0.301756803	3.9215686	40.96	0.7	0.4511385	1.168	0.716861509	1.757013502	20.24986363
2.011712	0.344471237	4.4117647	46.08	0.7	0.4023424	1.168	0.765657596	1.876611754	22.12647538
2.2430589	0.384085429	4.9019608	51.2	0.7	0.4486118	1.168	0.719388219	1.763206419	23.8896818
2.4583121	0.420943851	5.3921569	56.32	0.71	0.4916624	1.168	0.676337582	1.657690151	25.54737195
2.7902446	0.477781605	5.8823529	61.44	0.72	0.5580489	1.168	0.609951085	1.49497815	27.0423501
2.7500103	0.470892181	6.372549	66.56	0.73	0.5500021	1.168	0.617997933	1.514700817	28.55705092
2.7419635	0.469514296	6.8627451	71.68	0.74	0.5483927	1.168	0.619607303	1.51864535	30.07569627
2.7842094	0.476748192	7.3529412	76.8	0.75	0.5568419	1.168	0.611158112	1.49793655	31.57363282
2.8244437	0.483637616	7.8431372	81.92	0.77	0.5648887	1.168	0.603111264	1.478213883	33.0518467
2.8264554	0.483982087	8.3333333	87.04	0.79	0.5652911	1.168	0.602708922	1.47722775	34.52907445
2.8586428	0.489493627	8.8235294	92.16	0.81	0.5717286	1.168	0.596271443	1.461449616	35.99052407
3.5023906	0.599724423	9.3137255	97.28	0.83	0.7004781	1.168	0.467521874	1.145886946	37.13641101
3.0799311	0.527385463	9.8039216	102.4	0.86	0.6159862	1.168	0.552013779	1.352974948	38.48938596
3.4400276	0.589045815	10.294118	107.52	0.88	0.6880055	1.168	0.479994488	1.17645708	39.66584304
3.5788357	0.61281433	10.784314	112.64	0.91	0.7157671	1.168	0.452232863	1.108413879	40.77425692
3.6935033	0.63244919	11.27451	117.76	0.93	0.7387007	1.168	0.429299346	1.052204278	41.8264612
4.1139511	0.704443679	11.764706	122.88	0.94	0.9462087	1.168	0.22179125	0.543606006	42.37006721
3.8866276	0.665518429	12.254902	128	0.96	0.7773255	1.168	0.390674475	0.957535477	43.32760268
3.9168033	0.670685498	12.745098	133.12	0.99	0.7833607	1.168	0.384639339	0.942743477	44.27034616
4.0898105	0.700310024	13.235294	138.24	1.01	0.8179621	1.168	0.350037892	0.857936009	45.12828217
4.0918223	0.700654495	13.72549	143.36	1.03	0.8183645	1.168	0.349635549	0.856949876	45.98523205
4.2487358	0.727523252	14.215686	148.48	1.05	0.8497472	1.168	0.318252842	0.780031475	46.76526352

Table J2.3: ...continued...

4.3392628	0.743024457	14.705882	153.6	1.08	0.8678526	1.168	0.300147434	0.735655475	47.500919
4.5122701	0.772648984	15.196078	158.72	1.11	0.902454	1.168	0.265545987	0.650848007	48.151767
4.4318016	0.758870134	15.686274	163.84	1.14	0.8863603	1.168	0.281639683	0.690293341	48.84206034
4.421743	0.757147778	16.176471	168.96	1.16	0.8843486	1.168	0.283651395	0.695224008	49.53728435
4.6671719	0.799173269	16.666667	174.08	1.18	0.9334344	1.168	0.234565622	0.57491574	50.11220009
4.7878746	0.819841543	17.156863	179.2	1.21	0.9575749	1.168	0.210425078	0.515747739	50.62794783
4.7275233	0.809507406	17.647059	184.32	1.25	0.9455047	1.168	0.22249535	0.545331739	51.17327957
4.6249259	0.791939373	18.137255	189.44	1.28	0.9249852	1.168	0.243014812	0.59562454	51.76890411
4.7637341	0.815707888	18.627451	194.56	1.3	0.9527468	1.168	0.215253186	0.527581339	52.29648545
4.7214881	0.808473992	19.117647	199.68	1.33	0.9442976	1.168	0.223702377	0.548290139	52.84477559
4.4800827	0.767137444	19.607843	204.8	1.35	0.8960165	1.168	0.271983465	0.666626141	53.51140173
4.4418601	0.760592491	20.098039	209.92	1.37	0.888372	1.168	0.279627971	0.685362674	54.1967644
4.9206476	0.842576645	20.588235	215.04	1.4	0.9841295	1.168	0.183870479	0.450662938	54.64742734
4.8361557	0.828108853	21.078431	220.16	1.42	0.9672311	1.168	0.20076886	0.492080539	55.13950788
5.2103341	0.892180503	21.568627	225.28	1.44	1.0420668	1.168	0.125933173	0.308659737	55.44816762
5.0312918	0.861522563	22.058824	230.4	1.47	1.0062584	1.168	0.161741647	0.396425604	55.84459322
4.8180503	0.825008612	22.54902	235.52	1.49	0.9636101	1.168	0.204389941	0.500955739	56.34554896
5.2686738	0.902170169	23.039216	240.64	1.51	1.0537348	1.168	0.114265243	0.28006187	56.62561083
5.1278539	0.878057182	23.529412	245.76	1.53	1.0255708	1.168	0.142429211	0.349091204	56.97470203
Cumulative Total					**32.818322**			**56.97470203**	

Accountability =101.4407 %
Resin Loading [Adsorption] = 56.97 g/L
Mass Transfer Zone = 82.12 cm
Residence Time = 9.95 min

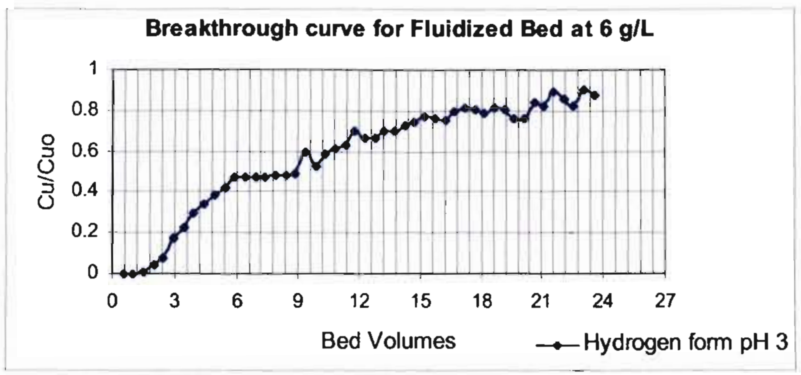


Figure J2.1: Breakthrough Curve with Initial Condition from Table J2.1

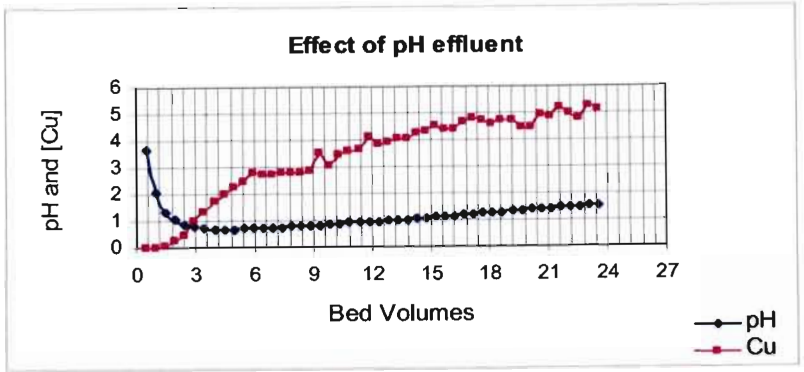


Figure J2.2: Monitoring of Copper Concentration and Effluent pH

APPENDIX J3: COMPARISON OF CONTACTING EQUIPMENT AT 6 g/L

The fixed bed breakthrough curve from Appendix J1 and the fluidized bed breakthrough curve were placed together on the same graph for comparison purposes.

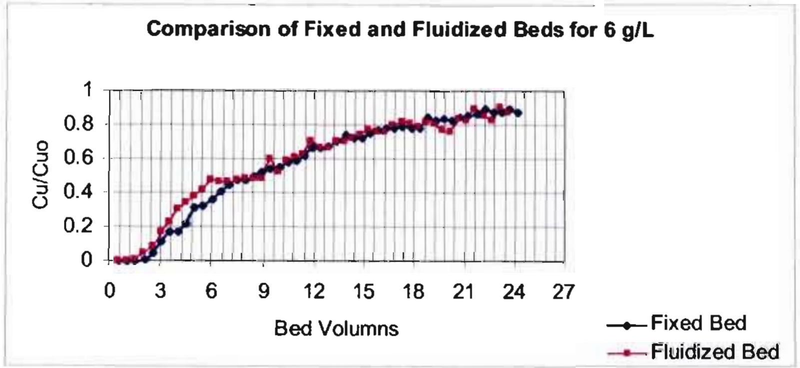


Figure J3.1: Comparison of Fixed and Fluidized Breakthrough Curves at 6 g/L

APPENDIX K: COMPARISON OF CONTACTING EQUIPMENT AT 0.6 g/L

K1: FIXED BED ION EXCHANGE FOR CONCENTRATION AT 0.6 g/L K1-1

K2: FLUIDIZED BED ION EXCHANGE FOR CONCENTRATION AT 0.6 g/L K2-1

K3: COMPARISON OF CONTACTING EQUIPMENT FOR 0.6 g/L K3-1

APPENDIX K1: FIXED BED ION EXCHANGE FOR CONCENTRATION 0.6 g/L

TABLE K1.1: Initial Conditions for Fixed Bed at 0.6 g/L

Input
Column Diameter = 2.4 cm
Column Height = 1 m
Resin Volume = 400 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.1 [Hydrogen Form]
Flow rate = 2318.094 mL/hr = 5.795 BV/hr
Linear Velocity = 0.142 cm/s
Feed Solution = 0.58 g/L at pH = 3.01
Overall Mass of Copper Input = 31.578195 g

TABLE K1.2: End Conditions for Fixed Bed at 0.6 g/L

Output
Resin Volume = 425 mL [Loaded with Copper]
Volume of Eluate = 5105 mL
Eluate Concentration = 4.4 g/L
Mass of Copper from Eluate = 22.462 g

Elution
Resin Volume = 398 mL [Hydrogen Form]
pH Resin = 2.0 [Hydrogen Form]
Loading from Elution = 56.437186 g/L

TABLE K1.3: Calculations for Fixed Test with Initial Conditions from Table K1.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.003	0.005145798	2.5	25.8833	1.84	0.003	0.58	0.577	1.4425	1.4425
0.004	0.006861063	5	51.7666	1.83	0.004	0.58	0.576	1.44	2.8825
0.0045	0.007718696	7.5	77.6499	1.82	0.0045	0.58	0.5755	1.43875	4.32125
0.0053	0.009948542	10	103.5332	1.8	0.0058	0.58	0.5742	1.4355	5.75675
0.012	0.02058319	12.5	129.4165	1.8	0.012	0.58	0.568	1.42	7.17675
0.015	0.025728988	15	155.2998	1.8	0.015	0.58	0.565	1.4125	8.58925
0.014	0.024013722	17.5	181.1831	1.79	0.014	0.58	0.566	1.415	10.00425
0.014	0.024013722	20	207.0664	1.79	0.014	0.58	0.566	1.415	11.41925
0.017	0.02915952	22.5	232.9497	1.78	0.017	0.58	0.563	1.4075	12.82675
0.019	0.032590051	25	258.833	1.77	0.019	0.58	0.561	1.4025	14.22925
0.016	0.027444254	27.5	284.7163	1.77	0.016	0.58	0.564	1.41	15.63925
0.016	0.027444254	30	310.5996	1.78	0.016	0.58	0.564	1.41	17.04925
0.019	0.032590051	32.5	336.4829	1.78	0.019	0.58	0.561	1.4025	18.45175
0.025	0.042881647	35	362.3662	1.77	0.025	0.58	0.555	1.3875	19.83925
0.03	0.051457976	37.5	388.2495	1.77	0.03	0.58	0.55	1.375	21.21425
0.1508	**0.258662093**	**110.4125**	**1108.25**	**1.78**	**4.398082**	**16.9157**	**12.517618**	**31.294045**	52.508295
0.2565249	0.440008405	112.9125	1134.133	2.07	0.2565249	0.58	0.3234751	0.80868775	53.31698275
0.44766	0.767855918	115.4125	1160.016	2.07	0.44766	0.58	0.13234	0.33085	53.64783275
0.4576	0.78490566	117.9125	1185.899	2.09	0.4576	0.58	0.1224	0.306	53.95383275
0.4627	0.793653516	120.4125	1211.783	2.12	0.4627	0.58	0.1173	0.29325	54.24708275
0.4627	0.793653516	122.9125	1237.666	2.12	0.4627	0.58	0.1173	0.29325	54.54033275
0.47	0.806174957	125.4125	1263.549	2.14	0.47	0.58	0.11	0.275	54.81533275
Cumulative Total					**7.1695669**			**54.81533275**	

Accountability = 93.835531 %

Resin Loading [Adsorption] = 54.81 g/L

Mass Transfer Zone = 81.62 cm

Residence Time = 9.58 min

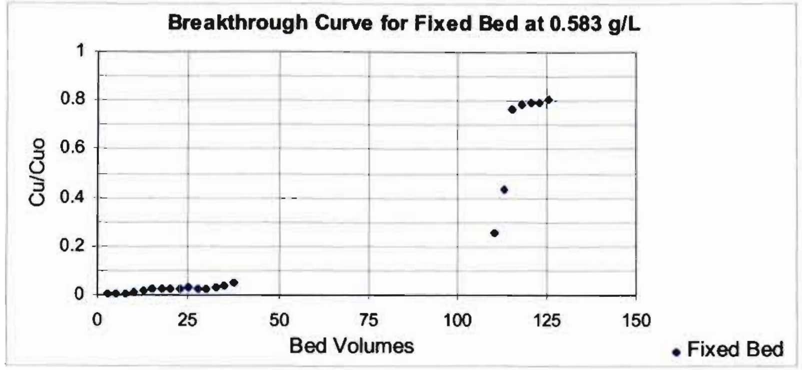


Figure K1.1: Breakthrough Curve with Initial Condition from Table K1.1

APPENDIX K2: FLUIDIZED BED ION EXCHANGE FOR CONCENTRATION 0.6 g/L

TABLE K2.1: Initial Conditions for Fluidized Bed at 0.6 g/L

Input
Column Diameter = 2.4 cm
Column Height = 2 m
Resin Volume = 401 mL of TP 207 [Hydrogen Form]
pH of Resin = 2.5 [Hydrogen Form]
Static Bed Height = 856 mm
Bed Height at 0.141 cm/s = 1030 mm
Flow rate = 2294.60 mL/hr = 5.7 BV/hr
Linear Velocity = 0.141 cm/s
Feed Solution = 0.719 g/L at pH = 3.01
Overall Mass of Copper Input = 38.86195 g

TABLE K2.2: End Conditions for Fluidized Bed at 0.6 g/L

Output
Resin Volume = 415 mL [Loaded with Copper]
Volume of Eluate = 5000 mL
Eluate Concentration = 4.2 g/L
Mass of Copper from Eluate = 21 g

Elution
Resin Volume = 405 mL [Hydrogen Form]
pH Resin = 2.1 [Hydrogen Form]
Loading from Elution = 52.369077 g/L

TABLE K2.3: Calculations for Fluidized Bed Test with initial conditions from Table K2.1

[Cu] [g/L]	Cu/Cuo	BV	Time [min]	pH Effluent	Cu OUT [g]	Cu IN [g]	Cu (resin) [g]	Cu Loading/Period [g/L]	Cumulative Cu Loading [g/L]
0.001	0.001390821	2.4937656	26.14833	1.81	0.001	0.719	0.718	1.790523691	1.790523691
0.0018	0.002503477	4.9875312	52.29666	1.72	0.0018	0.719	0.7172	1.788528678	3.579052369
0.004	0.005563282	7.4812968	78.44499	1.71	0.004	0.719	0.715	1.783042394	5.362094763
0.007	0.009735744	9.9750623	104.5933	1.74	0.007	0.719	0.712	1.775561097	7.13765586
0.008	0.011126565	12.468828	130.7417	1.76	0.008	0.719	0.711	1.773067332	8.910723192
0.009	0.012517385	14.962594	156.89	1.79	0.009	0.719	0.71	1.770573566	10.68129676
0.008	0.011126565	17.456359	183.0383	1.81	0.008	0.719	0.711	1.773067332	12.45436409
0.009	0.012517385	19.950125	209.1866	1.82	0.009	0.719	0.71	1.770573566	14.22493766
0.018	0.025034771	22.44389	235.335	1.84	0.018	0.719	0.701	1.748129676	15.97306733
0.018	0.025034771	24.937656	261.4833	1.85	0.018	0.719	0.701	1.748129676	17.72119701
0.019	0.026425591	27.431421	287.6316	1.88	0.019	0.719	0.7	1.74563591	19.46683292
0.029	0.040333797	29.925187	313.78	1.89	0.029	0.719	0.69	1.720698254	21.18753117
0.3219136	0.447724061	**117.33167**	**1213.78**	**1.98**	**11.283072**	**25.20095**	**13.91787832**	**34.70792599**	**55.89545716**
0.4528106	0.629778303	119.82544	1239.928	2.06	0.4528106	0.719	0.2661894	0.663813965	56.55927112
0.4627508	0.643603338	122.3192	1266.077	2.16	0.4627508	0.719	0.2562492	0.639025436	57.19829656
0.5381993	0.748538665	124.81297	1292.225	2.2	0.5381993	0.719	0.1808007	0.450874564	57.64917112
0.5583189	0.776521419	127.30674	1318.373	2.22	0.5583189	0.719	0.1606811	0.400700998	58.04987212
0.6136478	0.853473992	129.8005	1344.522	2.23	0.6136478	0.719	0.1053522	0.262723691	58.31259581
0.613289	0.852974965	132.29427	1370.67	2.26	0.613289	0.719	0.105711	0.263618454	58.57621426
0.6184983	0.860220167	134.78803	1396.818	2.28	0.6184983	0.719	0.1005017	0.250627681	58.82684195
Cumulative Total					**15.272386**			**58.82684195**	

Accountability = 93.336506 %
Resin Loading [Adsorption] = 58.82 g/L
Mass Transfer Zone = 81.73 cm
Residence Time = 9.66 min

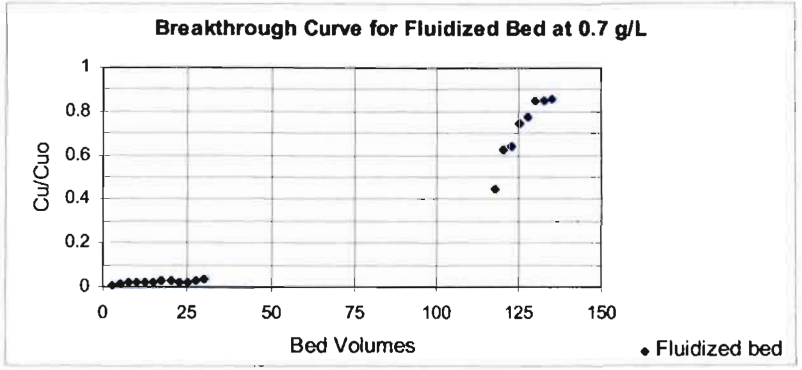


Figure K2.1: Breakthrough Curve with Initial Condition from Table K2.1

APPENDIX K3: COMPARISON OF CONTACTING EQUIPMENT

The fixed bed breakthrough curve from Appendix K1 and the fluidized bed breakthrough curve were placed together on the same graph for comparison purposes.

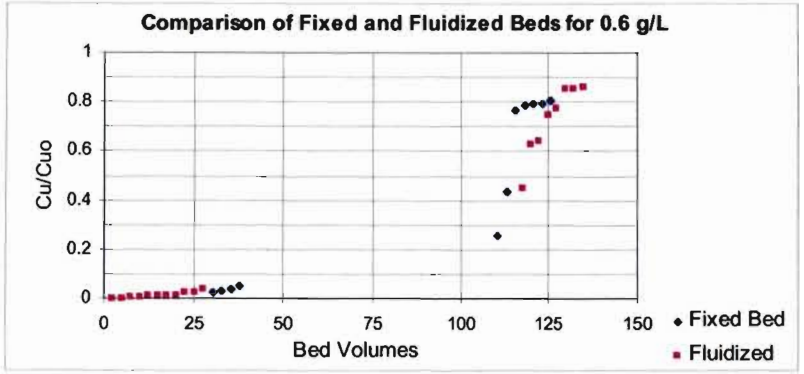


Figure K3.1: Comparison of Fixed and Fluidized Breakthrough Curves at 0.6 g/L

APPENDIX L: FULL SCALE PLANT DESIGN AND COST ESTIMATIONS

L1: FULL SCALE PLANT DESIGN FOR FIXED BED AT 6 g/L L1-1

L2: FULL SCALE PLANT DESIGN FOR FLUIDIZED BED AT 6 g/L L2-1

L3: FULL SCALE PLANT DESIGN FOR FIXED BED AT 0.6 g/L L3-1

L4: FULL SCALE PLANT DESIGN FOR FLUIDIZED BED AT 0.6 g/L L4-1

L5: COST ESTIMATION FOR FULL SCALE FIXED BED DESIGN L5-1

L6: COST ESTIMATION FOR FULL SCALE FLUIDIZED BED DESIGN L6-1

APPENDIX L1: FULL SCALE PLANT DESIGN FOR FIXED BED AT 6 g/L

Full Scale Plant Design			
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	4.68
	Feed: Copper Concentration	g/L	6
	Resin Copper Loading	g/L	60
	Residence Time	min	10.3
		hours	0.171667
	Resin Exhaustion Rate	m^3/hr	0.5
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**0.8**
	Resin Bed Depth	m^3	0.854701
	Resin Elution	m^3	1.5
	Overall Resin Bed Depth Required	m^3	2.354701
Plant Size:	Column: Diameter	m	1.166318
	Column: Area	m^2	1.068376
	Column: Resin Bed Height	m	2.204
	Column: Height	m	2.4244
	H:D		1.889707
Adsorption:	Estimated Transfer Time	hours	4.8
	Estimated Number of Bed Volumes Treated	BV	10
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
Pilot Plant Design			
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	1
Input:	Feed: Flow rate	ml/min	36.6
		BV/hr	5.4
	Feed: Linear Velocity	cm/s	0.13
		m/hr	4.68

APPENDIX L2: FULL SCALE PLANT DESIGN FOR FLUIDIZED BED AT 6 g/L

	Full Scale Plant Design		
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	5.04
	Feed: Copper Concentration	g/L	6
	Resin Copper Loading	g/L	60
	Residence Time	min	9.95
		hours	0.161
	Resin Exhaustion Rate	m^3/hr	0.5
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**0.82**
	Resin Bed Depth	m^3	0.813492
	Resin Elution	m^3	1.5
	Overall Resin Bed Depth Required	m^3	2.313492
Plant Size:	Column: Diameter	m	1.123893
	Column: Area	m^2	0.992063
	Column: Resin Bed Height	m	2.332
	Column: Head Required for Fluidization	m	0.4
	Column: Height	m	2.9652
	H:D		2.074931
Adsorption:	Estimated Transfer Time	hours	4.6
	Estimated Number of Bed Volumes Treated	BV	10
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
	Pilot Plant Design		
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	2
Input:	Feed: Flow rate	ml/min	37.97
		BV/hr	5.58
	Feed: Linear Velocity	cm/s	0.14
		m/hr	5.04

APPENDIX L3: FULL SCALE PLANT DESIGN FOR FIXED BED AT 0.6 g/L

Full Scale Plant Design			
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	5.112
	Feed: Copper Concentration	g/L	0.6
	Resin Copper Loading	g/L	60
	Residence Time	min	9.58
		hours	0.159667
	Resin Exhaustion Rate	m^3/hr	0.05
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**0.8162**
	Resin Bed Depth	m^3	0.798318
	Resin Elution	m^3	0.15
	Overall Resin Bed Depth Required	m^3	0.948318
Plant Size:	Column: Diameter	m	1.11595
	Column: Area	m^2	0.978091
	Column: Resin Bed Height	m	0.96956
	Column: Height	m	1.066516
	H:D		0.86882
Adsorption:	Estimated Transfer Time	hours	19
	Estimated Number of Bed Volumes Treated	BV	100
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
Pilot Plant Design			
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	1
Input:	Feed: Flow rate	ml/min	38.635
		BV/hr	5.8
	Feed: Linear Velocity	cm/s	0.142
		m/hr	5.112

APPENDIX L4: FULL SCALE PLANT DESIGN FOR FLUIDIZED BED AT 0.6 g/L

Full Scale Plant Design			
Input:	Feed: Flow rate	m^3/hr	5
	Feed: Linear Velocity	m/hr	5.076
	Feed: Copper Concentration	g/L	0.6
	Resin Copper Loading	g/L	60
	Residence Time	min	9.66
		hours	0.161
	Resin Exhaustion Rate	m^3/hr	0.05
	Elution Time	hours	3
	Operating Temperature	Ambient	
	Resin Mass Transfer height	m	**0.8173**
	Resin Bed Depth	m^3	0.805063
	Resin Elution	m^3	0.15
	Overall Resin Bed Depth Required	m^3	0.955063
Plant Size:	Column: Diameter	m	1.1199
	Column: Area	m^2	0.985028
	Column: Resin Bed Height	m	0.96958
	Column: Head Required for fluidization	m	1.1
	Column: Height	m	2.166538
	H:D		0.865774
Adsorption:	Estimated Transfer Time	hours	19
	Estimated Number of Bed Volumes Treated	BV	100
Elution:	Eluant	H_2SO_4	
	Eluant: Concentration	2 M	
	Eluant: Volume	2 BV	
	Eluant: Flow rate	1 BV/hr	
	Elution: Time	2 hr	
	Water Wash: Volume	2 BV	
	Water Wash: Flow rate	2 BV/h	
	Water Wash: Time	1 hr	
Pilot Plant Design			
Plant Size:	Column Dimensions		
	Column: Diameter	cm	2.4
	Column: Height	m	2
Input:	Feed: Flow rate	ml/min	38.243
		BV/hr	5.7
	Feed: Linear Velocity	cm/s	0.141
		m/hr	5.076

APPENDIX L5: COST ESTIMATION FOR FULL SCALE FIXED BED DESIGN

Fixed Bed				
		Rands	**Rands**	**Rands**
Fixed Capital Cost				2246892.3
Equipment: Total Purchase Cost (PCE)			383.84	
Ion Exchange Column				
Bare vessel(Design Parameters H=2.5m, D=1m)		3800		
Total Cost of Vessel (MF=1 and PF =1)		3800		
f_1 Equipment	0.4			
f_2 Piping	0.7			
f_3 Instrumentation	0.2			
f_4 Electrical	0.1			
f_5 Building, process	0.15			
f_6 Utilities	0.5			
f_7 Storages	0.15			
f_8 Site Development	0.05			
f_9 Ancillary buildings	0.15			
Total physical plant cost = PCE(1+ f1....+f9)		1305.056		
Design and Engineering	0.3			
Contractors Fee	0.05			
Contingency	0.1			
Peristaltic Pump			20000	
Resin (Iminodiacetic Acid : 1L @ R45)			**2225000**	
Production Costs				
Direct Production Costs				149307961
Variable Cost			148635869	
Sulphuric Acid (2M) at 2 BV (2.5L @ R390)		**148613400**		
Miscellaneous materials		22468.923		
Fixed Costs			672091.86	
Maintenance		224689.23		
Operating Labour		10618.18		
Laboratory Costs		2442.1814		
Supervision		2123.636		
Plant overheads		5309.09		
Capital Charges		337033.85		
Insurance		22468.923		
Local Taxes		44937.847		
Royalties		22468.923		
Sales expense				44792388
TOTAL COSTS				**196347241**

Where

MF : Material Factor from Figure 11 .31

PF : Pressure Factor from Figure 11.31

APPENDIX L6: COST ESTIMATION FOR FULL SCALE FLUIDIZED BED DESIGN

Fixed Bed				
			Rands	**Rands**
Fixed Capital Cost				2247290.7
Equipment: Total Purchase Cost (PCE)			464.65	
Ion Exchange Column				
Bare vessel(Design Parameters H=3m, D=1m)		4000		
Total Cost of Vessel (MF=1 and PF =1)		4000		
f1 Equipment	0.4			
f2 Piping	0.7			
f3 Instrumentation	0.2			
f4 Electrical	0.1			
f5 Building, process	0.15			
f6 Utilities	0.5			
f7 Storages	0.15			
f8 Site Development	0.05			
f9 Ancillary buildings	0.15			
Total physical plant cost = PCE(1+ f1....+f9)		1579.81		
Design and Engineering	0.3	0.3		
Contractors Fee	0.05	0.05		
Contingency	0.1	0.1		
Peristaltic Pump			20000	
Resin (Iminodiacetic Acid : 1L @ R45)			**2225000**	
Production Costs				
Direct Production Costs				149308080
Variable Cost			148635873	
Sulphuric Acid (2M) at 2 BV (2.5L @ R390)		**148613400**		
Miscellaneous materials		22472.907		
Fixed Costs			672207.4	
Maintenance		224729.07		
Operating Labour		10618.18		
Laboratory Costs		2442.1814		
Supervision		2123.636		
Plant overheads		5309.09		
Capital Charges		337093.61		
Insurance		22472.907		
Local Taxes		44945.814		
Royalties		22472.907		
Sales expense				44850509
TOTAL COSTS				**196347795**

Where

MF : Material Factor from Figure 11.31

PF : Pressure Factor from Figure 11.31