Enhanced Removal of Nickel (II) Ions from Synthetic Wastewater employing Fixed Bed Ion Exchange Column Technique

By,

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12254

Dissertation submitted in partial fulfillment

of the requirements for the

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(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
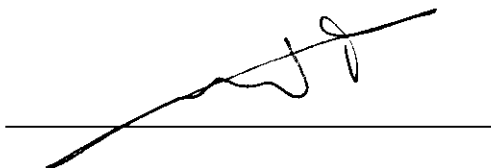
Chemical Engineering Programme

Universiti Teknologi PETRONAS

In partial fulfillment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(CHEMICAL ENGINEERING)

Approved by,

_____

(Dr. Usama Mohamed Nour Eldemerdash)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

SEPTEMBER 2011

CERTIFICATE OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Atif Ashraf

ATIF MOHAMMED ASHRAF

ABSTRACT

Ion exchange process is an exchange of ions between a solid resin and a liquid phase. This method is widely used in the extraction of heavy metals in industries like electroplating for industrial waste water treatment. Nickel present in the wastewater can cause serious harm to living beings. It is equally harmful to human beings as well as the environment. In this study, ion exchange process was used to study and enhance the removal of nickel from wastewater which in turn improves the quality of the wastewater. In this project, the removal of Nickel (II) ions from synthetic wastewater using a strong acid cation resin called Lewatit S1467 in fixed bed column is investigated. The resin is modified with Tetrabutylammonium Iodide prior to the experiments. The purpose of this project is to determine the optimum conditions for the removal of metal ions from the waste water solution and enhancing the performance of the resin through modification. The experiments were conducted under different pH values (3, 5 and 7) and at different initial concentrations of the contaminant (1.8, 2.8 and 3.8 gNi/L). The effect of regeneration on the modified resin is also studied. The removal efficiency and rate of removal is shown in breakthrough curves and the kinetic for the process is calculated. The exchange isotherm was confirmed on Thomas model. Atomic Adsorption Spectrophotometer was used to measure the Nickel ion concentration in order to compare against the environmental limit. Fourier Transform Infrared Spectroscopy was used for characterization of the resin. Results from this project will be useful in designing an appropriate heavy metal removal technique using ion exchange process to minimize the negative impacts caused on the environment by industrial wastewater.

ACKNOWLEDGEMENT

First and foremost, I would like to express my praises to God for His blessing.

My deepest appreciation and gratitude is extended to my supervisor, Dr. Usama Mohamed Nour Eldemerdash for being very encouraging, supportive and responsive throughout the whole process of completing this final year project to fulfill the university requirement. Without his constant supervision and guidance, I may not be able to complete this project successfully.

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Thank you.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Wastewater treatment is a process which is used to separate, modify and remove objectionable, hazardous and pathogenic substances carried by wastewater in solution or suspension to render the water fit and safe for disposal or other intended uses. The processes are classified as preliminary, primary, secondary and advanced wastewater treatment, depending on type, sequence, and method of removal of the harmful and unacceptable constituents. Contaminant composition, characteristic and concentration in the solution forms the basis to select which process is to be chosen to treat the wastewater. Ion exchange, adsorption and membrane filtration are the most frequently studied for the treatment of heavy metal wastewater (Fu & Wang, 2011). Table 1.1 below compares the characteristics of these methods used for heavy metal extraction.

ION EXCHANGE	ADSORPTION	MEMBRANE FILTRATION	COAGULATION - FLOCCULATION
High treatment capacity	Flexibility in design and operation	High removal efficiency	Conventional wastewater treatment process
High removal efficiency	Removal of heavy metal from wastewater containing low conc. of heavy metal	High cost	Increased sludge volume generation
Fast kinetics	High cost of AC	Process complexity	Chemical consumption

Table 1.1: Comparison between different methods used for heavy metal extraction

Ion exchange method is one of the most effective methods which have been applied in advanced wastewater treatment. Advantages like high treatment capacity and more efficient removal of ion from wastewater favors ion exchange over other methods. In this method, ion exchange removes unwanted ions mainly Nickel (II) by transferring them to a solid material called resins. Resins will accept ions from the solution and transfer the equivalent number of desirable species stored on the resin.

The resin will be modified with Tetrabutylammonium iodide (TBAI) on its surface. The modification is based on the work done by Kerodjoud (Amara & Kerdjoudj, 2002).

In this study, Nickel (Ni (ii)), a heavy metal in its nature is recognized as the contaminant in the wastewater. Ion exchange method using modified strong acid cation resin is chosen to extract the Ni (ii) impurity. Lewatit S 1467, a commercially available strong acid cation resin, modified with TBAI is used to conduct experiments to study the rate of removal of Ni (ii) ions from the wastewater.

1.2   PROBLEM STATEMENT

Effluent of industries such as electroplating contains toxic heavy metals which can be a serious threat to the environment and humans. Heavy metal like nickel can cause cancer and anosmia which is loss of ability to smell. Therefore industries have to comply with strict discharge regulation limits. Therefore an efficient separation system such as ion exchange is used for Nickel ions removal from industrial waste water discharge.

Ion exchange is widely studied in the extraction of heavy metal from waste streams in chemical industries. High rate of removal of Ni (ii) ions from effluent have been achieved by ion exchange process. But only limited number of studies focuses on fixed bed performance for metal removal. Also the breakthrough characteristic of the removal of Ni (ii) ions from waste streams still requires improvement. Thus a study is required to enhance and evaluate the rate of removal of Ni (ii) ions from wastewater by modification of the resin and achieving improved breakthrough values at the same time.

1.3   OBJECTIVES

- To investigate the performance of Lewatit S 1467 cation exchange resin modified with TBAI in removing Ni(ii) ions from wastewater

- To analyze and identify the optimum operating conditions for the ion exchange process, i.e. range of pH values and initial concentration of contaminant.

- To compare removal efficiencies for unmodified ion exchange resin and modified ion exchange resin.

- Kinetic study of the ion exchange process and evaluation of rate of removal of ions

1.4   SCOPE OF STUDY

The extraction of Ni (ii) ions from waste water using the modified resin Lewatit S 1467 is studied in this research. This study aims to determine the optimal value of process parameters which include the, range of pH values, mass transfer zones and the initial concentration of contaminant. The breakthrough characteristic of resin is also studied. The knowledge involves fundamentals of chemical equilibrium and ion exchange process, which is needed to be understood clearly in order to predict the breakthrough curves and to study the kinetics of the experiment.

1.5   SIGNIFICANCE OF THE PROJECT

This study is focused on the removal of heavy metal which is Nickel (II) ion from industrial wastewater solution. Although many researches and studies have been discussed by others regarding this issue, the details are not sufficient for further work to enhance the results. Moreover, the purpose of this project is to determine the optimum condition for the removal of heavy metal from solution to increase the quality of wastewater from the metal plating industry. A comparative study of the results between modified and unmodified resin will also be conducted.

CHAPTER 2

LITERATURE REVIEW

This chapter presents the theory on ion exchange process and consists of previous studies conducted on the extraction of heavy metals by ion exchange process using various types of resins.

2.1 WASTEWATER TREATMENT

Metallic pollutants are inevitable for industrial growth and some of them are essential nutrients both for plants and human beings. But if these species are present beyond the tolerance limits, these are reported to affect the health adversely (Yavuza, et al. 2008). Metal ions cause serious environmental problems because of their widespread usage and application. Effluent limits have become stricter in recent years causing the chemical process industries to upgrade their wastewater. To meet these limits, plants have to employ sophisticated chemical and physical processes because biological treatment alone is not adequate. Because of this removal of heavy metals from wastewater is one of the predominant portions of environment research. It is evident from the literature survey of 185 articles that ion exchange, adsorption and membrane filtration are the most frequently studied for the treatment of heavy metal wastewater (Fu and Wang 2011). Today ion exchangers are applied in the field of environmental protection processes such purification, extraction recycling etc.

2.2 ION EXCHANGE PROCESS

An ion exchange reaction can be defined as a reversible exchange of ions between a solid phase (ion exchanger) and a liquid phase (solution).Ions are either positively (cation) charged or negatively (anion) charged. The displaced ion is originally a part of an insoluble material, and the displacing ion is originally in solution. There are basically two types of ion exchange materials; naturally occurring inorganic zeolites and synthetically produced organic resins. Since the characteristics of the synthetic organic resins can be altered for specific applications, they are favored over zeolites. A resin having a cation as a mobile exchangeable ion is classified as a

cation exchanger whereas anion exchangers are those containing anions. Both types of resins are produced from the same basic organic polymers. Cation and anion exchangers differ in the ionizable group attached to the hydrocarbon network. The functional group determines the chemical behavior of the resin.

The nickel ion is removed from the metal plating wastewater by exchanging the nickel ions (Ni^{2+}) in the wastewater with cation (H^+) in the resins.

$$NiSO_4 \text{ (aq)} + 2 \text{ R.H(s)} \Leftrightarrow R_2Ni(s) + H_2SO_4 \text{ (aq)} \qquad \text{(equation 1)}$$

$$Ni^{2+} \text{ (aq)} + 2 \text{ R.H (s)} \Leftrightarrow R_2Ni(s) + 2H^+ \qquad \text{(equation 2)}$$
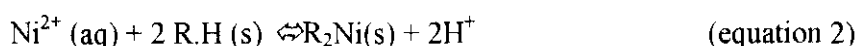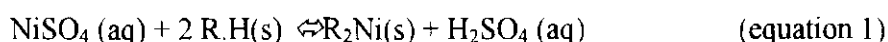
Where, R represents the exchanger solid (cation resins)

Based on the equation above, wastewater solution which contains Nickel Sulfate is fed into a hydrogen cation exchange resin which converts the influent heavy metal (Ni^{2+}) to the sulfuric acid (H_2SO_4) by exchanging an equivalent number of hydrogen (H^+) ions for the metallic cations (Ni^{2+})(T.D.Reynolds & P.A.Richards, 1996)(Neumann & Fatul, 2009)

The cation resin is regenerated with sulfuric acid. The reaction for cation regeneration process is shown below.

$$Ni.R_2 \text{ (s)} + H_2SO_4 \text{ (aq)} \rightarrow 2 \text{ R.H (s)} + NiSO_4 \text{ (aq)} \qquad \text{(equation 3)}$$
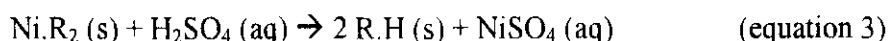
The regeneration process is based on the Law of mass action where the reaction is driven to the left by increasing the concentration of the hydrogen ion in the right. Thus, by using strong acid such as sulfuric acid with 5% to 10% concentration, the reaction will drive to the left side.

2.3   BREAKTHROUGH CURVES

In order to analyze and identify the optimum operating condition and rate of removal characteristics of resins in an ion exchange process, the study of breakthrough curves is very important. Breakthrough curves is plot between the relative concentration versus time. The relative concentration is defined as C/Co where C is the concentration at a point and Co is the source concentration.

Refer to the research article called "Removal of Ni(II) using cation exchange resins in packed bed column: Prediction of breakthrough curves" by Priya, P.G., et al., (2011), breakthrough point is defined as the the point at which the rinse water concentration at the top of the column reaches a particular concentration. Breakthrough curves is affected by both operating variables and characteristics of the resins. In this study, the experimental data was applied on models like Thomas, Yoon-Nelson, Clark and Wolborska to represent the breakthrough curves. The author found out that the performance of removal of ions was well described by the Thomas, Yoon-Nelson and Wolborska models at influent concentration ratios (C/Co) > 0.03 and <0.99. The author also concluded that the removal performance was improved with increasing bed height and decreasing the flow rate.

In 2011, Shaidan, N.H., et al. had conducted a research titled "The removal od Ni(II) ions from aqueous solutions using fixed bed ion exchange technique". The main objective of the study was to investigate the removal of Ni(ii) from synthetic wastewater and its effect on parameters like initial concentration of contaminant, the optimum range of pH for removal and regeneration effect on resins. The author found out that a contact time of 1hour can help to maintian around zero Ni(ii) outlet concentration. Through this research, the author established that the initial concentration of contaminant affected the breakthrough time of the system. Thomas model was applied to study the fixed bed dynammics and to compare the experimental data with the calculated one and the calculated breakthrough curves agreed well with measured ones. Another study conducted by Juang, R.S., et al. in 2006 investigated the column removal of Ni(II) from synthetic electroplating waste water using a strong-acid resin (Purolite NRW-100). The authours findingsshows some abnormalities in breakthrough curve charaterictics at certain pH values. Their study identified a non zero concentration of Ni(ii) at pH 5 at time equals to zero. This concluded that such non zero Ni(ii) concentration could be partly explained by the formation of non exchangable Ni(ii) species in solution. However they suggested that this can be reduced by increasing the contact time between solutions and the resins.

2.4   ION EXCHANGE IOSTHERMS

Langmuir isotherm seems more suitable for describing ion exchange studied than Freundlich isotherm. It is defined as:

$$\frac{X}{M} = \frac{abC_e}{1 + bC_e}$$

Where   X/M: Amount of contaminant adsorbed per unit mass of adsorbent
         a,b: Empirical constant
         C_e: Equilibrium concentration of adsorbate in solution after adsorption, mg/L

The constant in the Langmuir isotherm can be determine by plotting $C_e/(\frac{X}{M})$ versus C_e and making use of following equation:

$$\frac{C_e}{X/M} = \frac{1}{ab} + \frac{1}{a}C_e$$

Alyuz and Veli, in 2009 evaluated the performance of DOWEX HCR S/S cation exchange resin for removal of nickel and zinc from aqueous solutions. They published their findings in the paper titled "Kinetics and equilibrium studeis for the removal of nickel annd znic from aqueous solutions by ion exchge resins". The author conducted batch shaking asdorption experiments to examine the effects of pH, dosage of resin and contact time on removal process. The experimental equilibrium data was tested on various isotherms like Langmiur, Freundlich and Temkin. Langmiur isotherm fitted the for the data.The author managed to attain a removal efficieny of 98% under optimum operating conditions.

2.5   ION EXCHANGE MODIFICATION

Modification of ion exchange resin by adsorption of TBAI has given better results in relation to the hydrophobic and hydrophilic properties of resin. Based on the work conducted by Sata,T., et al. in 1995, modification of cation and anion exchange changed the selectivity of the resulting membrane.

The number of water molecules surrouding two monovalent cation is larger than those of one divalent ion. Since the ions are more hydrated, they are less fixed on the resin. Therfore the resin

modified with TBAI becomes more selective to elements with higher valencies compared to the parent resin. Similar work conducted by Amara.M. et al. in 2002, proves that the introduction of TBAI inside the resin phase increases hydrophobicity due to the increasing aliphatic chains bonded to the ammonium groups. Their experiment proved that the higher valence ions such as Al^{3+} and Fe^{3+} are more sorbed in the TBAI modified resin than in the parent resin because each Al^{3+} or Fe^{3+} can replace three ions of hydrogen or alkaline ion.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

Figure 3.1 below illustrate 6 steps of research procedure

1. Further literature reviews on the subject

2. Selection of resin and outlining a process to investigate the performance of the resin

3. Initial charectirization of sample

4. Modification of resin

5. Conduct ion exchange experiment with fixed bed column

6. Characterization of the samples

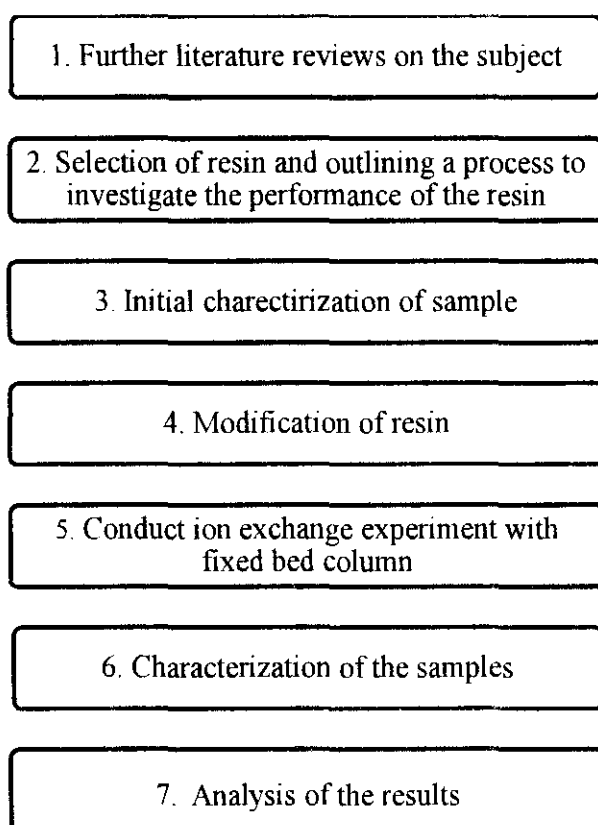7. Analysis of the results

Figure 3.1: Research process flow diagram

3.2   PROJECT METHODOLOGY

3.2.1   Overview

The experiment will be conducted in continuous mode. The rinse water used for the experiment is prepared by dissolving nickel sulfate ($NiSO_4.6H_2O$) in distilled water. The project will be divided into two main parts:

1. Studying the removal of nickel from wastewater solution based on parameters like initial concentration of contaminant and varying pH values of contaminant.

2. Studying the regeneration effect on the removal process.

All experiments will be conducted under similar temperature of $25^{\circ}C$ and at constant flow rate of $35cm^3/min$. The diameter and the height of the column used for the experiments are 2cm and 60cm respectively.

For the modification of the ion exchange resin, approximately 100 grams of resin is mixed with 40 grams of TBAI. This mixture will be immersed into a 500ml beaker containing alcohol and distilled water and is stirred for 72 hours nonstop. Magnetic stirrer will be used for stirring the solution at a constant rpm with film to cover up the beaker.

For the first part of the experiment, the removal of nickel from the wastewater is studied by manipulating the pH accordingly using 5% NaOH and 97% H_2SO_4. The rinse water is prepared for the following values; pH=3, pH=5, pH=7. This is done based on the pH range described in the Material Safety Data Sheet (MSDS). Experiments will also be carried out by preparing rinse water solutions at concentrations 1.8 gNi/L, 2.8 gNi/L and 3.8 gNi/L. This is done in order to study the effect of initial nickel concentration on the ion exchange process.

In the second part of the project, the effectiveness of the resin generation is studied. The experiment is conducted by using 1.8 gNi/L rinse water. Sulfuric acid (H_2SO_4) is allowed to flow through the column for about 1 hour until the conductivity of the effluent is similar to the conductivity of the influent.

The parameter of the effluent will be tested using the pH meter and the Absorption Atomic Spectrophotometer (AAS). Characterization of the resins will be determined by

using Fourier Transform Infrared Spectroscopy (FTIR). Operating manual for the ion exchange unit is attached in Appendix B.

3.2.2 Pretreatment of resin

Before the experiment, the resin needs to be pre-treated. The method of pretreatment was applied for cationic resins. The resin is packed in the cation column upto 10cm height. After that, the resin was rinsed downward flow pattern by using deionized water excessively. For cationic resin (Lewatit S 1467), the hydrogen ion (H+) inside the deionized water substitutes with Sodium ion (Na+) inside the resin. The downward rinsing is continued until the conductivity reading of the effluent water is equal to the initial conductivity reading of the deionized water. The exact procedure for the process attached in Appendix B.

3.2.3 Modification of resin

For the modification of ion exchange resin, TBAI will be immersed in solution of alcohol and distilled water. Approximately 70 gram of resin will be immersed into 500ml beaker containing alcohol and distilled water and is stirred for 72 hours nonstop. Magnetic stirrer is used for stirring the solution at constant rpm with film to cover the top of the beaker.

3.2.3 Regeneration of resin

Resins have limited capacities and must be regenerated upon exhaustion. Therefore, the cationic resin is regenerated in the cation column using 5% Sulfuric acid solution in a downward pattern. The flow rate of the regenerator was set at 35 cm3/min and minimum contact time of 40min is allowed. After regeneration, the resin was rinsed with deionized water.

3.3   TOOLS AND MATERIALS

3.3.1   Tools

The bench-top SOLTEQ Ion Exchange Unit (Model: TR 02) is used for this experiment. The unit consists of two vertically mounted tubes made of clear acrylic containing the respective cation and anion resins. The tubes are removable and interchangeable for softening, decationization or demineralization experiments. The size of the column composed in each column, cationic and anion column had a diameter of 2cm and length of 60cm. The schematic diagram of Ion exchange unit is attached in the Appendix A.
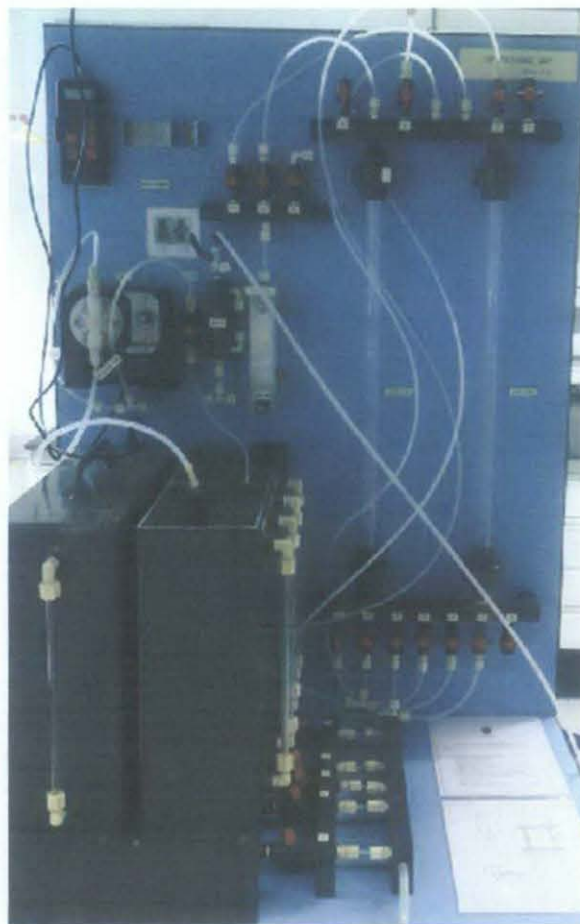
Figure 3.2: SOLTEQ Ion Exchange Unit (Model TR 02)

3.3.2 Instrument

The nickel concentration measurement was carried out using Atomic Absorption Spectrophotometer (AAS). pH meter was used to measure the pH of the initial solution and pH of the effluent after treatment.

Figure 3.3: Atomic Adsorption Spectrophotometer

3.3.3 Materials

I. Resins

Synthetic resin was used in the experimental studies. Cation resin used in this experiment is Lewatit S1467, produced by Lanxess. The resin is in Na+ ionic form and has sulfonic acid as the functional group. The resin is light brown, gel type beads that have cross-linked polystyrene matric.

Table 3.1: Physical and chemical properties of the resin

Name of Resin	Lewatit S 1467
Total Capacity	2.0 eq/L
Mean Bead Size	0.60mm
Maximum Swelling	8% (Na^+ to H^+)
Bulk Density	8.20 /L

II. Chemicals

Nickel (II) sulfate ($NiSO_4.6H_2O$) made by S&M Chemical Corporation to prepare the rinse water. In order to regenerate the cation resin, 98% sulfuric acid manufactured by Merck chemical was used. The resin modifier tetrabutylammonium Iodide is also manufactured by Merck.

3.4 KEY MILESTONE

The project is divided into two parts and completed within a period of two semesters. The research work is done in Final year project 1 and the experimental work is carried out in Final year project 2. At the end of the project, the parameters will be verified accordingly and the results will be analyzed.

CHAPTER 4

RESULT AND DISCUSSION

4.1 RESIN CHARACTERIZATION

4.1.1 FTIR analysis
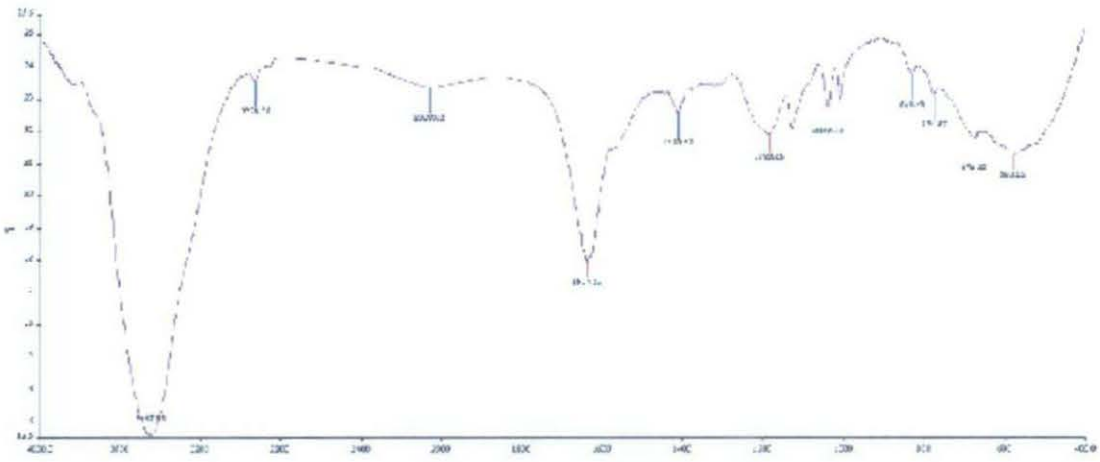
(a) After Pre-Treatment with Deionized Water
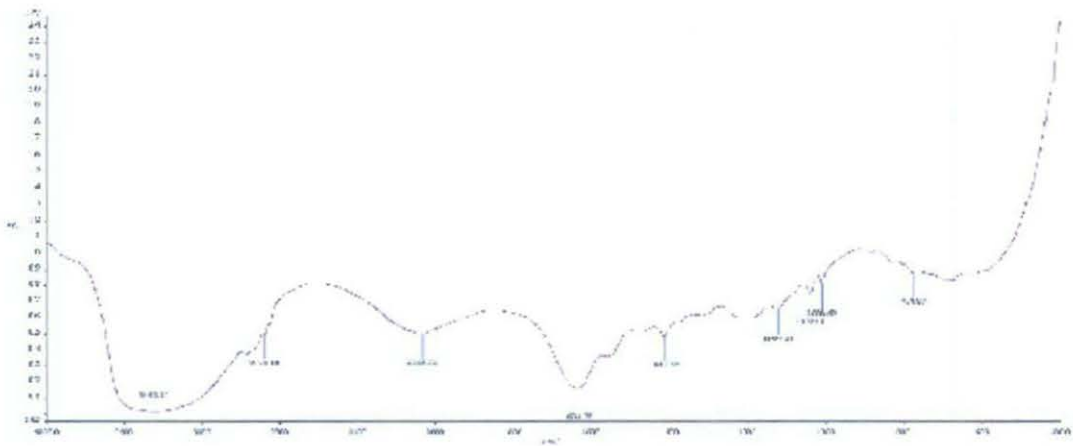
(b) Resin modified with TBAI
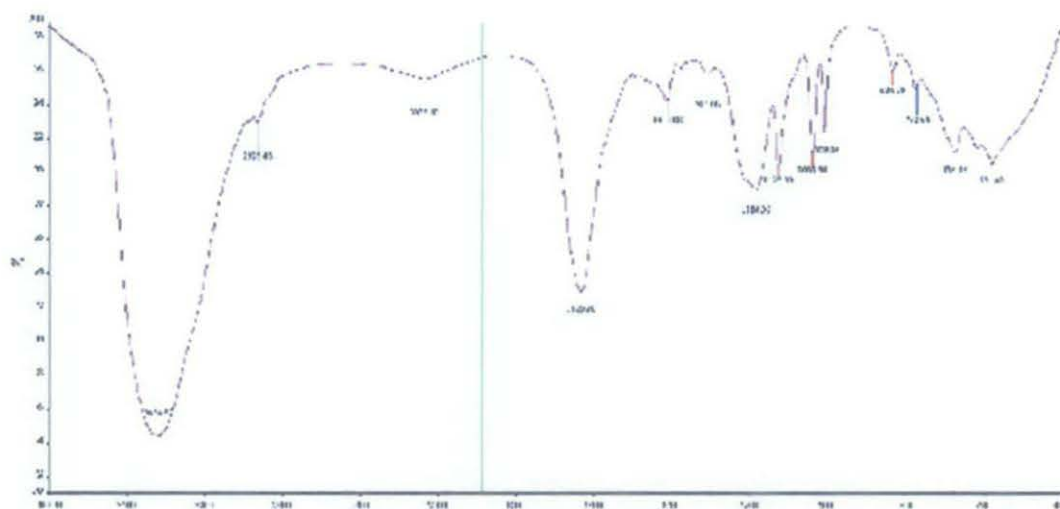
(c) After regeneration

Figure 4.1: FTIR image for Cation resins

The preliminary quantitative analysis of the functional group available in the cation resin is determined by using FTIR Spectroscopy for cation after pretreatment, cation after modification with TBAI and cation after regeneration. FTIR picture in Figure 4.1 shows that the broad and intense peak at around 2400 cm^{-1} refers to the OH group. This is due to the presence of free hydroxyl group and water in the resin. The band at 2928.43 cm^{-1} indicates -CH$_2$- stretching vibration of acyclic raised by electronegative substituent in the solution from the usage of plastic laboratory ware. Peak observed at 1633.80 cm^{-1} is the stretching vibration of primary amine –NH-. Peak at 3447.8 cm^{-1} is due to the presence of moisture in the KBr used for the FTIR analysis.

FTIR spectra of Nickel loaded resin in Figure 4.1(b) shows a sharp decrease of the peak at the range around 2878.14 cm^{-1} to 2924.56 cm^{-1} which signifies the –CH- region. The introduction of TBAI into the resin has resulted in reducing the carbon chain. The introduction of TBAI increases the hydrophobicity of the resin therefore the H$^+$ are loosely attached to the carbon chain. The broad peak at 1800 cm^{-1} indicates the existence of (C=O) in the resin. From Figure 4.1, the band has shifted from 3445.55, 2878.14, 2065.76, 1634.78, and 1412.56 at Figure (a) to 3447.38, 2924.56, 2058.02, 1637.02 and 1637.55. This is due to the existence of TBAI within the resin after modification.

Regeneration process in an ion exchange process is needed to recover back the resin before putting it back to the service cycle. Thus, the characteristic and ion available in the resin after regeneration will be similar to the resin which has undergone pretreatment. Figure (a) and Figure (c) looks very similar to each other. This shows that the Nickel ions absorbed during the ion exchange process has been replaced during the regeneration process and the exchanges site are vacant similar to the pretreated resin. A slight shift can be noticed in the regenerated resin from 3447.38, 2924.56, 2058.02, 1637.02, 1637.55 to 3434.89, 2928.43, 2073.50, 1633.80, and 1413.08. This difference is due to the presence of TBAI in the resin compared to the pretreated resin which was analyzed by FTIR before modification.

4.2 EFFECT OF INITIAL CONCENTRATION

Nickel concentration is an important parameter affecting the ion exchange process. In order to study the effect, three different initial concentrations were used. The study was conducted at 1.8 gNi/L, 2.8 gNi/L of Nickel (II) sulphate solution. To minimize the effect of other parameters on the Ni (II) ion removal, the rinse water solution was prepared at a pH value of 5.6. The flow rate was at a constant of 35cm^3/min. Cation resin was filled up to 10cm height of the column.
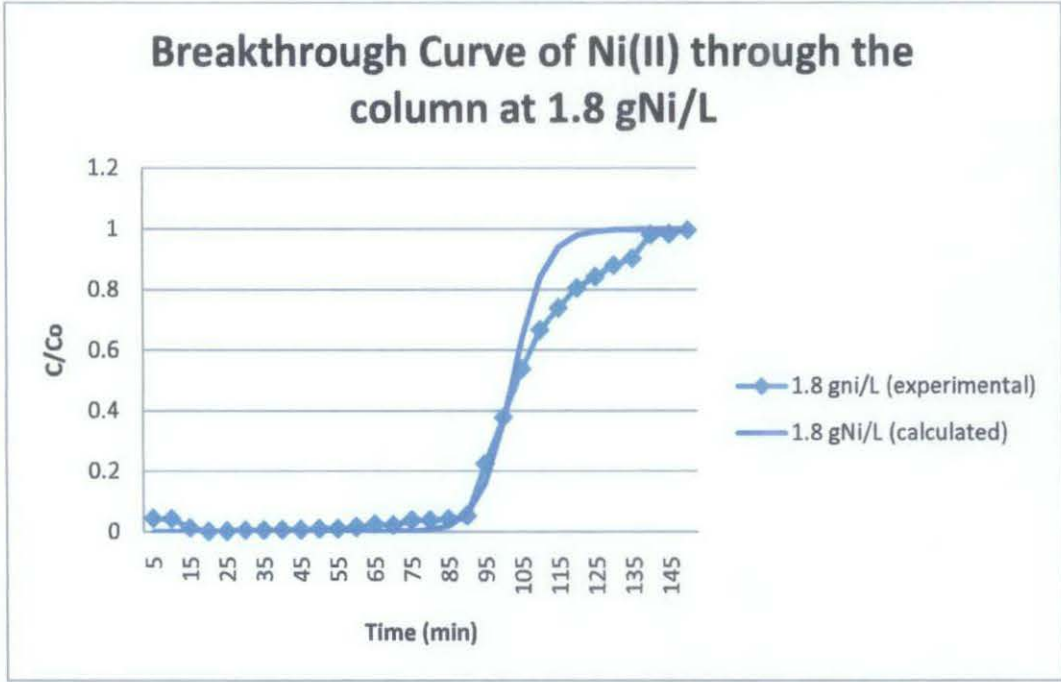
Figure 4.2: Breakthrough curve of Ni(II) at 1.8 gNi/L
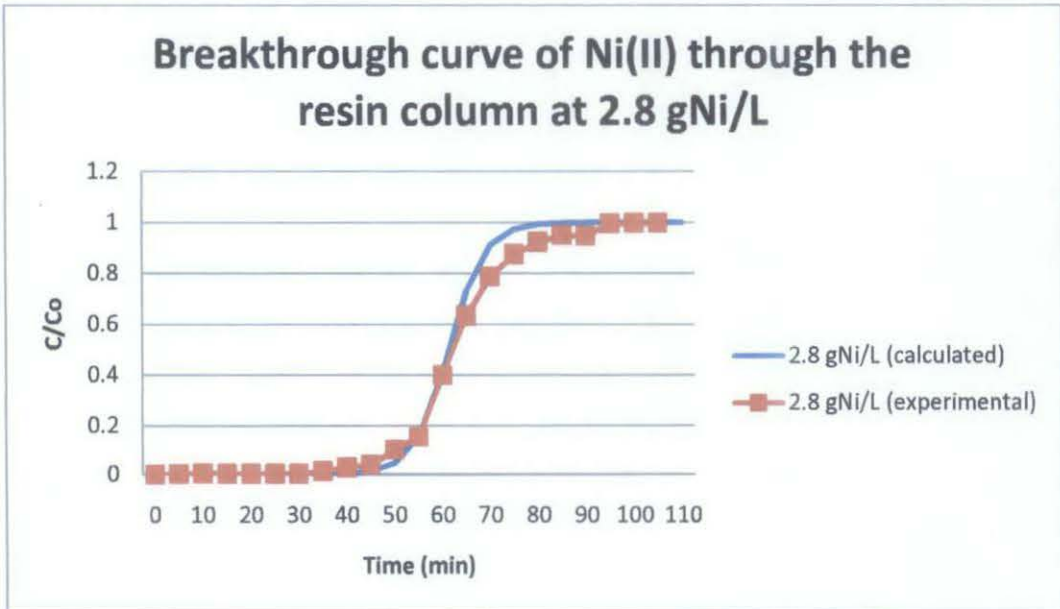
Figure 4.3: Breakthrough curve of Ni (II) at 2.8 gNi/L
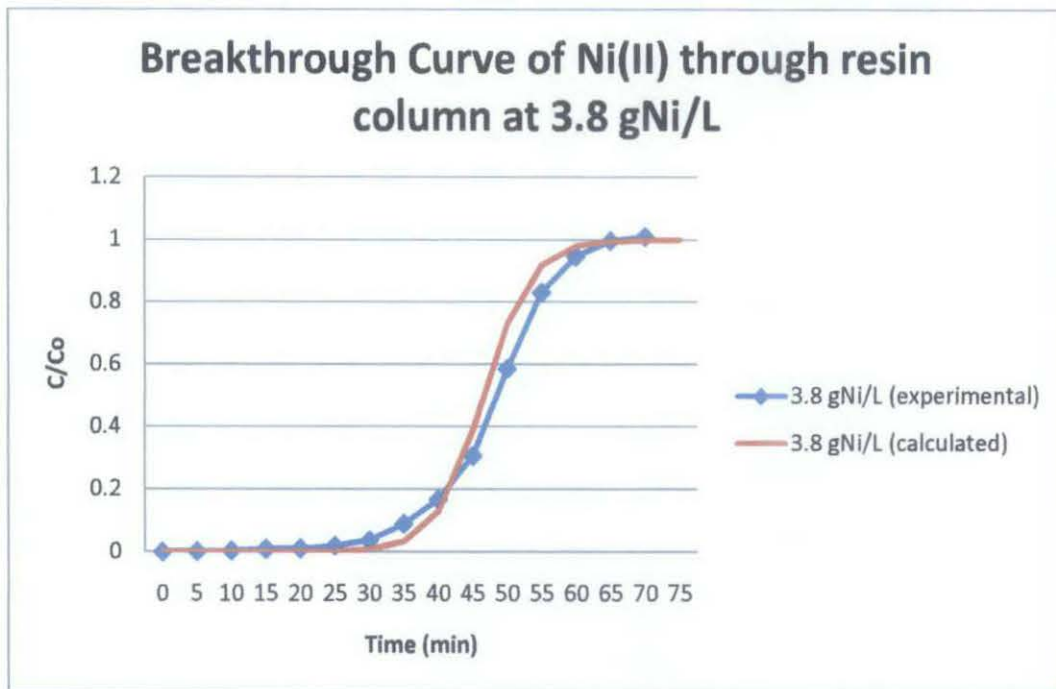
Figure 4.4: Breakthrough curve of Ni(II) at 3.8 gNi/L

Table 4.1: Breakthrough time for different initial concentration

Initial concentration (gNi/L)	Breakthrough time (min)
1.8	80
2.8	50
3.8	35

Breakthrough curves at different initial nickel concentration (C_0) are shown in Figure 4.2, 4.3 and 4.4. Breakthrough time in this experiment is defined as the time taken for 1ppm of Ni (II) ion concentration to be detected in the effluent. Once the breakthrough time exceeds, the resin can't be used and it must be regenerated before allowing the resin to be in service again. For the chosen breakthrough concentration of C_e= 1ppm, the time required to attain breakthrough point decreases with increase in C_0 values. Figure 4.2 shows that the breakthrough time for initial concentration at 1.8 gNi/L is at 80 minutes, whereas Figure4.4 show the breakthrough time for initial concentration at 3.8 gNi/L is at 35mins. The breakthrough time decreases with increase in

initial concentration due to the fact that the exchange sites of the resin gets exhausted earlier when a higher initial concentration influent is encountered. The amount of nickel ions in the solution is higher as the nickel concentration increases. Therefore Nickel ions will occupy the exchange sites faster till the column gets exhausted. Thus operation period until the resin hits breakthrough point is lesser at higher initial concentration is. Based on the study conducted by A.H. Norzilah., et al., the mass transfer between the ion in the wastewater solution and ion attached in the resin will increase as the initial concentration of the solution increases. The higher concentration gradient is the driving force for the mass transfer in the column. Hence, the ions in the solution will exchange faster with the ion that is attached to the resin thereby leading the resin into exhaustion earlier.

A similar study was conducted by Shaidan, N.H., et al., entitled Removal of Ni (II) ions from aqueous solutions using fixed-bed ion exchange column technique. Although in the study, the parent resin (unmodified Lewatit S1467) was used, the trend for the breakthrough curve is similar. Their study concluded that as the initial concentration increases the breakthrough point for the system decreased.

Initial Concentration	Breakthrough time (min)		Percentage improvement achieved
(gNi/L)	Parent resin**	Modified resin	in breakthrough value
1.8	60	80	33.33 %
2.8	43	50	14 %
3.8	15	35	133.33 %

Table 4.2: Comparison of breakthrough times between parent resin and modified resin

** Shaidan, N.H., et al., Removal of Ni (II) ions from aqueous solutions using fixed-bed ion exchange column technique

From the table 4.2, we can observe that the breakthrough time for the modified resin has significantly improved compared to the parent resin which did not undergo any modification. The improvement can be explained due to the presence of TBAI which increases hydrophobicity because of the increased aliphatic chains bonded to the NH3 groups. The introduction of TBAI into the resin makes it more selective towards ion with higher valences compared to parent resin.

Figure 4.2 shower a non-zero Ni (II) concentration at t= 0 min. According to R.S. Juang et al., the non-zero constant at the initial stage of the solid curve cannot be predicted using the present

model. Such non-zero Ni (II) concentration at t=0 implies that not all of Ni (II) ions in the solution are exchangeable. This is due to the presence of anionic ligands.(Juang, Kao, & Chen, 2006)

The solid curve shown in the Figure 4.2 indicated the breakthrough curve calculated by using Thomas's Model. For continuous flow ion exchange column, the first order reversible adsorption model such as Thomas's Model is selected for the kinetic studies (Won & S.Hun, 2006). Sorption is usually not limited by chemical reaction kinetics but is often controlled by interphase mass transfer. Therefore, this model is suitable for sorption process in which external and internal diffusion limitations are absent (Ponnaiah Gomathi Priya, 2011). At the same time, Thomas Model is widely used to study the column performance theory because it includes many parameters that affect the performance of ion exchange process. The Thomas model is given by:

$$\frac{C}{Co} = \frac{1}{1 + \exp(\frac{kT\,(qo\,W - Co\,Vout)}{Q})}$$

The linearized form of Thomas model is:

$$\ln(\frac{C}{Co} - 1) = \frac{kTqo\,W}{Q} - kTCot$$

Where,

C_o is the inlet metal concentration (mol/m$_3$),C is the outlet concentration (mol/m$_3$) at time t (min), k_T the Thomas rate constant (m$_3$/ (mol min)), Q is the volumetric flow rate (m$_3$/min), q_0 is the maximum resin-phase concentration of metal equivalent to an equilibrium liquid concentration of C_0 (mol/kg), W is the amount of resin in the bed (kg) and V_{out} is the effluent volume (m$_3$).
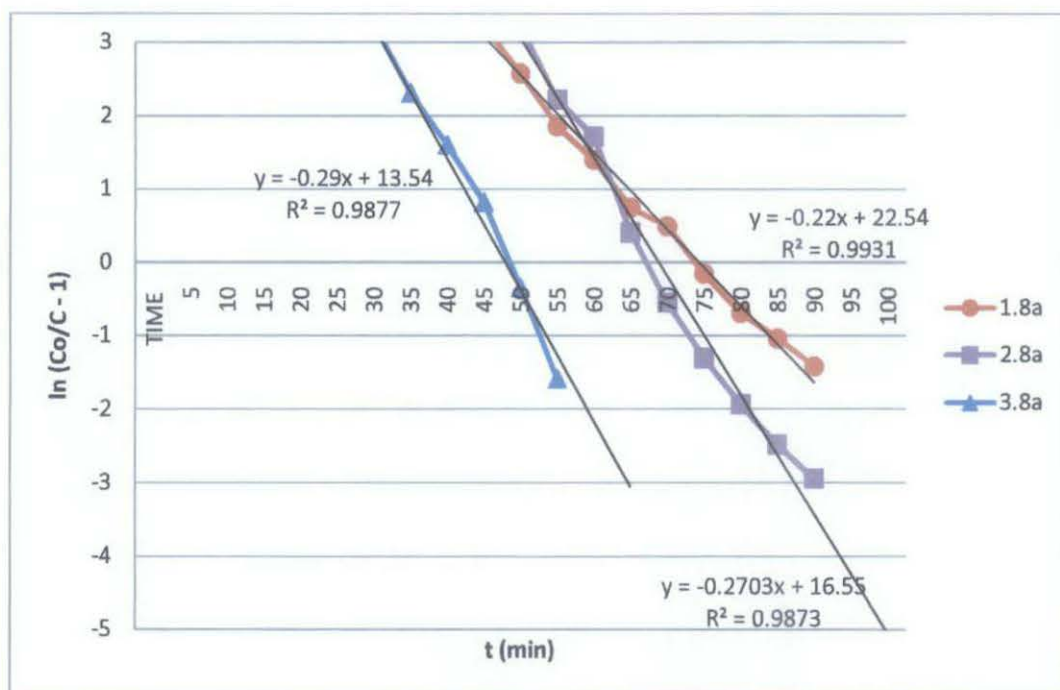
Figure 4.5: Graph for determination of Thomas parameters and exchange capacity for different inlet concentration

Initial Concentration (gNi/L)	Exchange Capacity, q_0 (mol/kg)	k_T
1.8	0.0317	0.62
2.8	0.0192	0.74
3.8	0.0141	0.837

Table 4.3: Properties of Ion exchange for different initial concentration

The value for exchange capacity q_0 and Thomas coefficient k_T is determined based on the graph ln ((C0/C) -1) vs. t as illustrated in the Figure 4.5. Table 4.3 shows that as the initial concentration increases from 1.8 gNi/L to 3.8 gNi/L, the exchange capacity decreases from .0317 mol/kg to.0141 mol/kg. Whereas the k_T values are increasing with increase in initial contaminant concentration. Thus we can conclude that the exchange capacity of the resin is inversely proportional to k_T.

Based on the k_T and q_0 values obtained from Figure 4.3, the breakthrough curve is constructed. The difference between the experimental values and calculated values for Thomas's model are small which is in the range below 5%.

The color of the wastewater has significant changes before and after treatment. The typical wastewater from industry which contains Ni (II) is usually green in color. After treatment, the effluent was colorless and had a pH value of 7. Hence we can say that it is safe for drainage and complying with the regulations.

4.3 EFFECT OF pH

Hydronium ions concentration is an important parameter affecting the ion-exchange process. This is partly because hydrogen ions themselves are strongly competing adsorbate and the solution pH influences the ionization of surface functional groups (Alyuz & Veli, 2009).

The effect of pH on nickel removal was conducted with a resin dosage of 31.41 cm^3 (diameter = 2cm, height = 10cm) and initial nickel concentration of 1.8 gNi/L. Sulfuric acid was used to reduce the pH to 3 and sodium hydroxide was used to increase the pH to 7. In order to avoid chemical precipitation, pH was kept below 8.
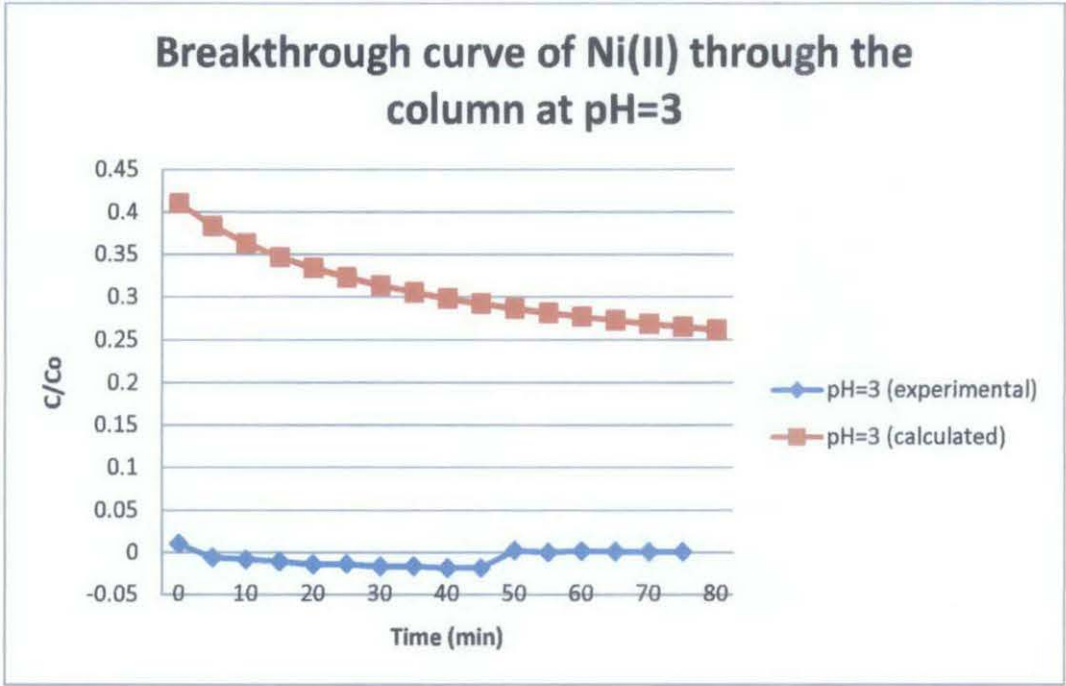
Figure 4.6: Breakthrough curve on Ni (II) through the resin column at pH=3
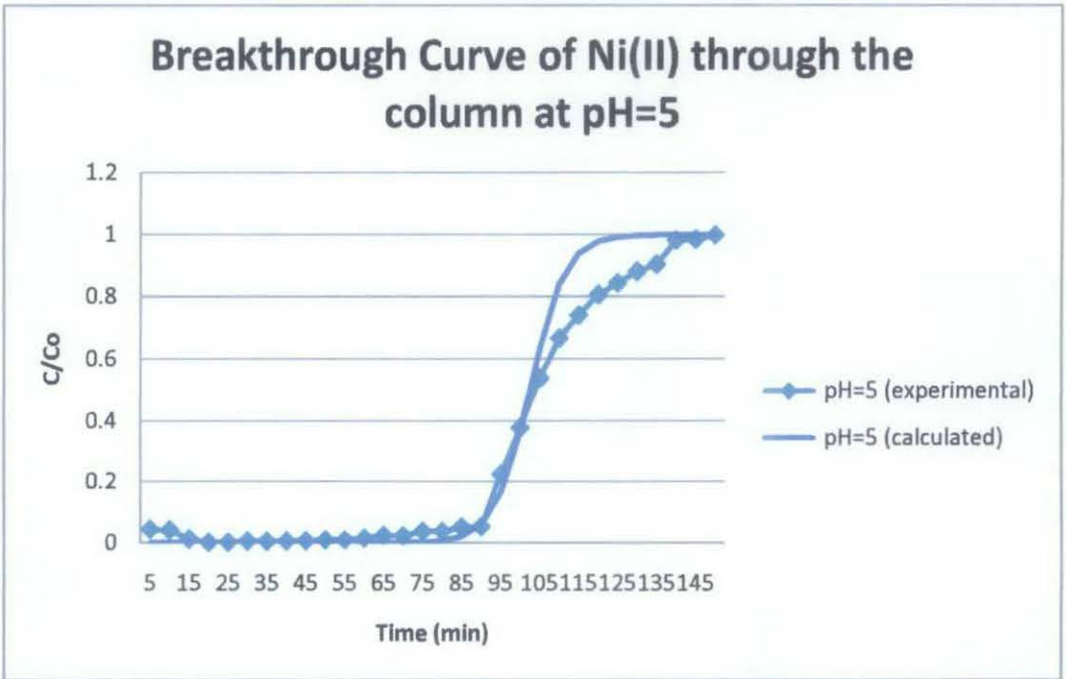
Figure 4.7: Breakthrough curve of Ni (II) through resin column at pH=5

Breakthrough curve of Ni(II) through the resin column at pH=7
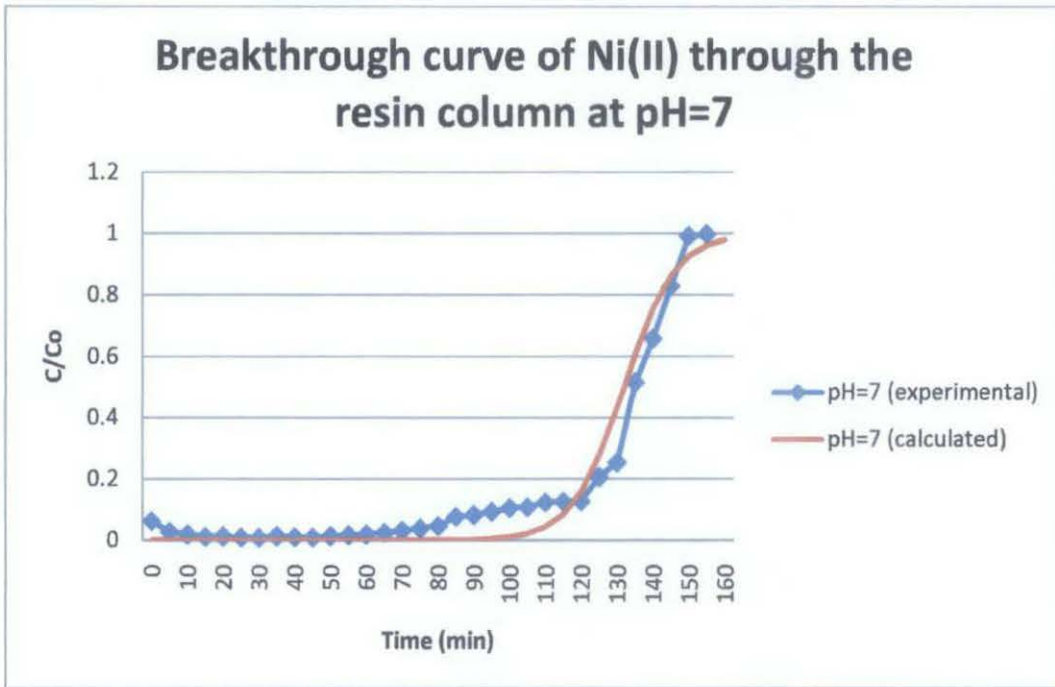
Figure 4.8: Breakthrough curve of Ni (II) through the resin column at pH=7

Table 4.4: Breakthrough time for different pH

pH of the wastewater solution	Breakthrough time (min)
3	-
5	80
7	85

The above result shows that the breakthrough time is dependent on the pH of the solution. From Figure 4.8, we can see that at a higher pH value, the breakthrough time is longer. But this is only applicable between the range of pH = 3 to 7(Shaidan, Eldemerdash, & Awad, 2011). The breakthrough curve at pH=3 is not as expected. This is due to the presence of large number of H^+ in the solution. A study conducted by Li-Chun, L., et al. on the removal of Cu (II) and Ni (II) from aqueous solutions using batch and fixed-bed ion exchange process states that when pH is below 3, an excess of H^+ compete effectively with Cu^{2+} or Ni^{2+} for binding, and whatever the ration of Cu^{2+} to Ni^{2+}, the total exchange with the resin will be low. Conversely, metal exchange

will be high. This explains why breakthrough times at higher pH like 5 and 7 we get better breakthrough time.
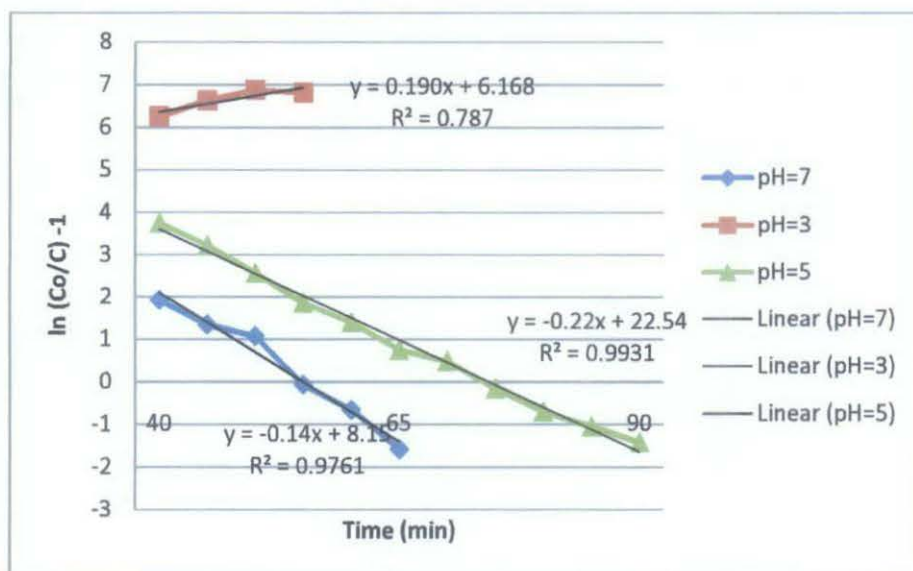
Figure 4.9: Graph for determination of Thomas parameters and exchange capacity for different pH

Table 4.5: Properties of ion exchange for different pH

pH of the wastewater solution	Exchange Capacity, q_0 (mol/kg)	k_{lt}
3	-6.82×10^{-3}	-0.538
5	0.0317	0.62
7	0.0385	0.395

The value for exchange capacity q_0 and Thomas coefficient k_T is determined based on the graph ln $((C_0/C) -1)$ vs. t as illustrated in the Figure 4.9. Table 4.5 clearly shows that as the pH increases from 3 to 7, the exchange capacity increases. The amount of exchanged ions at lower pH is lesser due to the competition between the Ni (II) ions and the H^+ ions at the vacant site which results in lower value for q_0. R.S. Juang et al., in their study of column removal of Ni(II) from synthetic electroplating wastewater using strong-acid resin verified that low q_0 values obtained at low pH values is caused by the competitive exchange of H^+ and the Na-form resins with Ni(II) ions. They also stated that the exchange capacity of the bed increases with increasing pH. The values of q_0 and k_T at different pH are obtained similarly even though the curves look

different. This is because k_T is determined merely abstracting the data in the sharp-rise part of the curve (Lin & Kiang, 2003).

4.4 EFFECT OF REGENERATION

For the removal of Ni (II) ion from the waste water in plant, the regeneration of exhausted ion exchange resin is important in order to reuse the resin back service cycle. The repeated usage of resin can minimize the cost of the operation. The regeneration of Lewatit S1467 is accomplished by using 5% Sulphuric acid as proposed by the manufacturer. The H^+ in the acid solution will be exchanged with the Ni (II) ions in the immobile particle due to the selectivity.

For this project, the sulphuric acid is allowed to flow into the column for about 1 hour with flow rate of 35 cm^3/min. Figure 4.10 shows the effect of regeneration on the resin. The breakthrough time achieved for the regenerated resin is 70 minutes while for the virgin resin is 80 minutes. Regenerated resin exhausts faster than the virgin resin due to the presence of other ions in the non-regenerated resin.
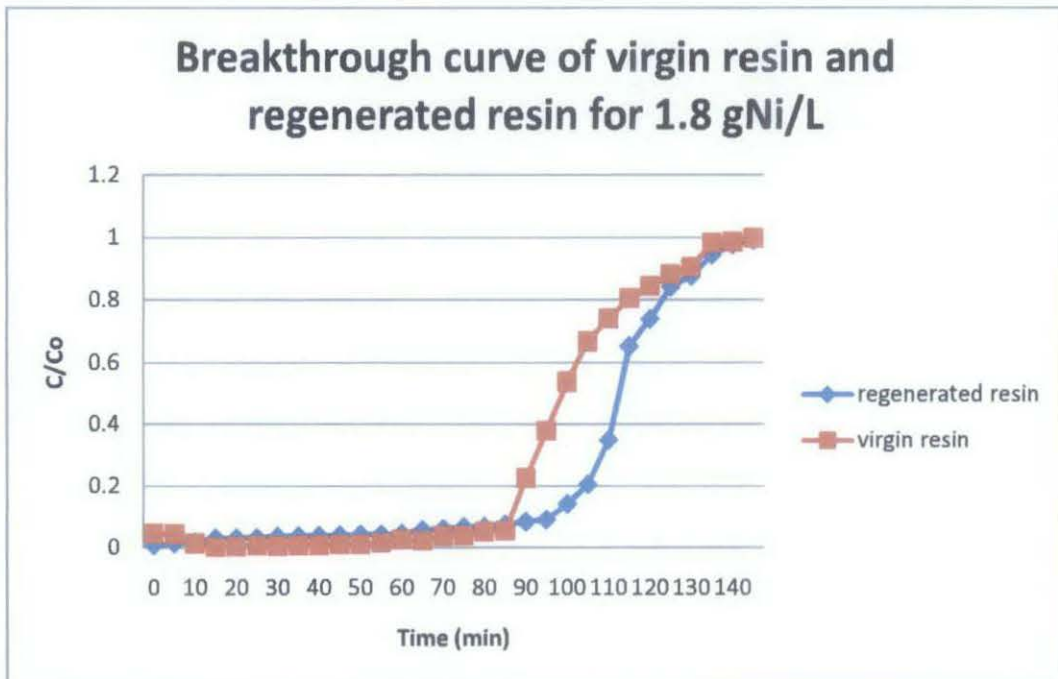
Figure 4.10: Breakthrough curve of virgin resin and regenerated resin for 1.8 gNi/L
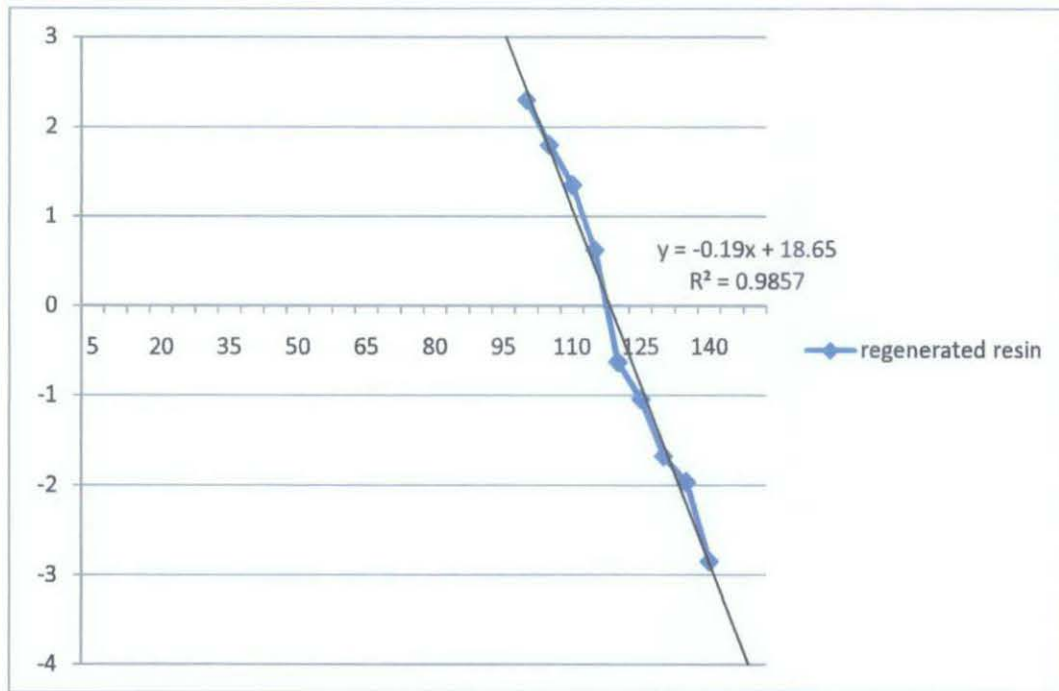
Figure 4.11: Graph for determining the Thomas parameters and exchange capacity for regenerated resin

Type of resin	K_T (m³/mol.min)	Exchange capacity, q_0(mol/kg)
Virgin	.0317	0.62
Regenerated	.0304	0.535

Table 4.6: Properties of Ion exchange for regenerated resin

The result in Table 4.6: shows the calculated value for exchange capacity based on Thomas model after first regeneration with 5% sulfuric acid. The results indicate that the ion exchange capacities of both virgin and regenerated resins are almost similar with 4% difference. Therefore the regenerated can be used back in the service cycle without affecting the performance of the resin. Thus, the resin can be regenerated and the cost of purchasing new resin can be minimized.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.0   CONCLUSION

Adsorption, membrane filtration, coagulation, flocculation, ion exchange are different types of heavy metal removal methods used currently in the industry. Among these, ion exchange process is selected in this study to remove nickel ions from synthetic wastewater. Ion exchange is chosen due to its advantages like ability to treat large volume of effluent at once and its proven efficiency in removing ions from wastewater. From the experiments conducted on a laboratory scale, the removal of Ni (II) ions from wastewater solutions were successfully enhanced using ion exchange method. On comparing the results with the parent resin, resin modified with Tetrabutylammonium Iodide has improved the performance of the resin significantly.

Based on the experiments done with different initial concentrations, it can be concluded that initial concentration affected the breakthrough time and the exchange capacity of the resin. The breakthrough time was significantly improved using the modified resin compared to the parent resin. An improvement of 33.33% on the breakthrough time was achieved at an initial concentration of 1.8 gNi/L. The breakthrough time decreases with an increase in initial concentration. This was explained based on the mass transfer characteristics and concentration gradient of ions in wastewater. The improvement accomplished in the breakthrough time was found to be the effect of TBAI on the resin. Based on the attained results, we can conclude that the optimum condition for ion exchange system is at lower initial concentration as it will decrease the need to regenerate the resin upon exhaustion and thereby decreasing the cost of the process. The effluent after treatment was found to be colorless and with a neutral pH value of 7.

With the modified resin, at pH= 3, the removal efficiency of the resin was unacceptable. This was due to the effect of excess protonation of H^+ at low pH values. For pH values above 7, chemical precipitation forms metal complexes. Thus the optimum pH value for the removal of ions is between the ranges of pH 5 to 6.

The effect of regeneration was studied and it can be concluded that regeneration of the resin did not affect the exchange capacity. This reduces the necessity to buy new resin thereby reducing the cost of the process itself.

The Thomas model was applied to describe the breakthrough curve of the process. Except for the experiment conducted at pH= 3, all other breakthrough curves closely fitted the experimental results having value of $R^2 > 0.9$.

In conclusion, ion exchange process is an effective way to remove heavy metal from wastewater. Results of this study show that the breakthrough time of the resin modified with TBAI has increased considerably. Comparing to the research conducted by N.H. Shaidan et al., using the parent resin, 95.53% removal if Ni (II) ions from wastewater with 33.3% improvement in breakthrough time was achieved by using the modified resin at 1.8 gNi/L and pH=5 (optimum values). With the enhancement in the breakthrough time, the life of the resin increases per service cycle is increased. This helps to reduce cost of the total process by reducing the frequency of procuring new resin. Therefore, the outcome of this study can be applied for practical purposes for e.g. in the electroplating industry.

5.1   RECOMMENDATION FOR FUTURE WORKS

This project has met its expected results and goals. However some improvements can be made for the current project. Experiments can be conducted to study the effects of parameters like resin dosage, flow rate of the rinse water and temperature on the ion exchange process for nickel removal. The exchange capacity and of the resin can be investigated using mixed bed columns instead of single bed.

The resin can also be modified adjusting the ratio of alcohol to water and the corresponding effect can be further investigated. Instead of using TBAI, the resin can be modified with Polyethamine iodide or neutral red solution. By using Polyethamine Iodide modified resin, transition metals as well as ion complexed with amine ligand can be eliminated. (Amara & Kerdjoudj, 2002)

CHAPTER 6

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CHAPTER 7

APPENDICES

APPENDIX A: Schematic diagram of Ion Exchange Unit
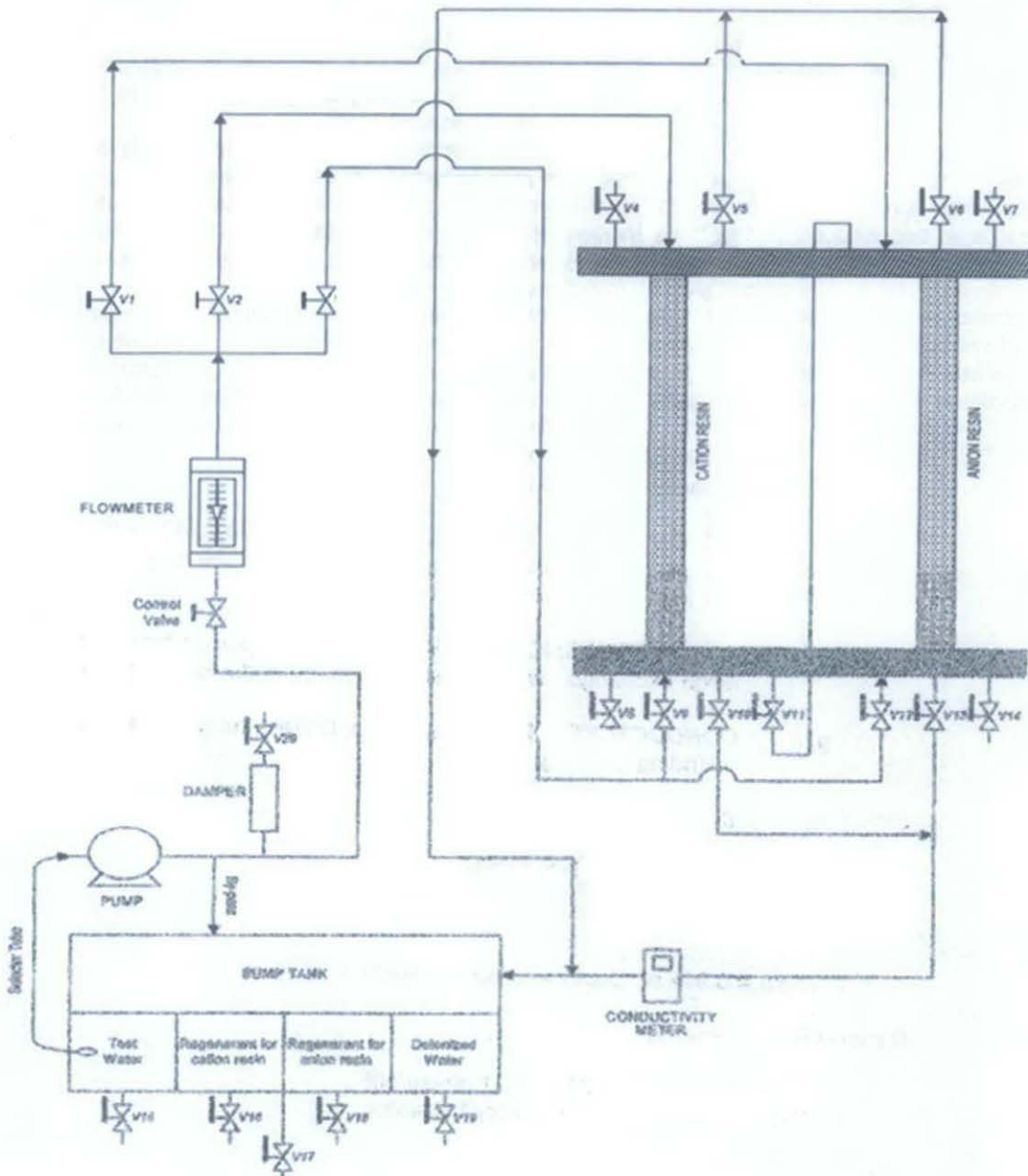
APPENDIX B: Operational Procedure for Ion Exchange Unit

The experiment is divided into three sections; Pre experiment stage which involved the rinsing of the cationic resin, the experiment itself and the regeneration stage where the cation resin is regenerated. The step by step procedure to conduct the experiment is as follows:

- Pretreatment of the resin:

  1. Fill in the modified Lewatit S 1467 type resin into the cation column up to 30 cm height.

  2. Prepare 5L deionized water in the appropriate reservoir. Record the conductivity meter reading of the deionized water.

  3. Make sure all valves are closed.

  4. Rinse the cationic resin in downward flow pattern by opening valves V2 and V10. (refer to figure)

  5. Place the selector tube into the deionized water reservoir and start the pump (25-30% opening).

  6. Adjust the control valve to obtain a flow rate between 35 and 40 cm^3/min.

  7. Observe the conductivity meter. Stop rinsing when conductivity meter reading is close to conductivity reading of the deionized water.

  8. Flush the deionized water out by opening valves V4 and V8.

  9. When the entire residue has been removed, close V4 and V8.

- Removal of Ni(II) ion from the solution by manipulating Ni(II) concentration:

  1. Prepare solution (Nickel sulphate) as test water. Record the initial conductivity meter reading of the test water.

  2. Place the selector tube into the test water ($NiSO_4.6H_2O$) reservoir and start the pump (25-39% opening).

  3. Adjust the control valve to obtain a flow rate between 35 and 40 cm^3/min.

4. Observe the conductivity meter. Record the conductivity reading every 5 s (the conductivity reading will decrease and remains at relatively low value).

   a. *(Note: Stop recording when conductivity reading starts to increase)*

5. Stop the operation by switching off the pump.

6. Determine the total time required to reach the "breakthrough" point from the plot of conductivity versus time.

7. Calculate the amount of test water that passed through the column before the "breakthrough" point.

8. Record the data obtained inside the water deionization table provided.

- Regeneration the cation resin:

  1. After decationization, regenerate the cationic resin by using 5% of hydrochloric acid (HCl) solution in downward flow pattern.

  2. Set the regenerant flow rate to 60 cm^3/min and allow a minimum contact time of 60 minutes.

  3. Then, rinse the generated resin with deionized water at 35 to 40 cm^3/min flow rate until conductivity meter reading is close to conductivity reading of deionized water.

Modification of resin with Tetrrabutylammonium Iodide (TBAI)

1. Weight TBAI to 40 grams of and put it into 500ml beaker
2. Take 240ml alcohol and 160 ml water and mix together (3/5 alcohol and 2/5 water)
3. Immerse the solution with TBAI and 100 grams of fresh resin
4. Put film to cover beaker
5. Stir the resin using magnetic stirrer. Stir nonstop for 72 hours with constant rpm
6. The modified resin is inserted in a Pyrex column and kept until used.

APPENDIX C: RAW DATA

Breakthrough time for initial concentration 1.8 gNi/L

Initial concentration Co = 20.8303

Time	C	C/C0
(min)	(ppm)	(mol/m3)
5	0.9307	0.044679
10	0.8878	0.04262
15	0.2767	0.013283
20	0.0376	0.001805
25	0.0633	0.003039
30	0.1353	0.006495
35	0.1304	0.00626
40	0.1487	0.007138
45	0.1537	0.007378
50	0.2168	0.010408
55	0.2223	0.010672
60	0.3264	0.015669
65	0.5296	0.025424
70	0.4747	0.022788
75	0.7991	0.038361
80	0.8046	0.038625
85	1.0765	0.051678
90	1.1207	0.0538
95	4.6861	0.22496
100	7.8917	0.378848
105	11.2209	0.538669
110	13.8923	0.666911
115	15.4072	0.739636
120	16.7821	0.805639
125	17.5733	0.843621
130	18.3529	0.881046
135	18.8352	0.9042
140	20.4572	0.982065
145	20.5234	0.985243
150	20.7896	0.998022

Breakthrough Time for Initial Concentration 2.8 gNi/L

Initial Concentration C0= 21.1414

Time	conc	C
min	ppm	mol/m3
0	21.1414	0.360201
5	0.0009	1.53E-05
10	0.0173	0.000295
15	0.0783	0.001334
20	0.0454	0.000774
25	0.0329	0.000561
30	0.0428	0.000729
35	0.0392	0.000668
40	0.2837	0.004834
45	0.5838	0.009947
50	0.8208	0.013985
55	2.0729	0.035317
60	3.2216	0.054889
65	8.4378	0.143761
70	13.4019	0.228337
75	16.6462	0.283613
80	18.482	0.314891
85	19.5084	0.332378
90	20.0808	0.34213
95	20.0304	0.341272
100	21.0523	0.358683
105	21.0993	0.359483
110	21.1004	0.359502

Breakthrough time for initial concentration 3.8 gNi/L

C0= 20.3406

Time	C (ppm)	C/C0
0	20.3406	
5	1.1852	0.000747
10	0.4375	0.001844
15	0.2131	0.004085
20	0.1917	0.009425
25	0.2295	0.011283
30	0.4136	0.020334
35	0.7666	0.037688
40	1.8346	0.090194
45	3.3993	0.167119
50	6.2199	0.305787
55	11.9103	0.585543
60	16.882	0.829966
65	19.2512	0.946442
70	20.2712	0.996588
75	20.5486	1.010226

Breakthrough time for pH=3

C0= .292965 mol/m3

Time	Conc (ppm)	C(mol/m3)	C/C0
0	17.1951	0.292965	1
5	0.1828	0.003114	0.010631
10	-0.0983	-0.001675	-0.00572
15	-0.1387	-0.002363	-0.00807
20	-0.179	-0.00305	-0.01041
25	-0.237	-0.004038	-0.01378
30	-0.2295	-0.00391	-0.01335
35	-0.2824	-0.004811	-0.01642
40	-0.2749	-0.004684	-0.01599
45	-0.3152	-0.00537	-0.01833
50	-0.3102	-0.005285	-0.01804
55	0.0454	0.000774	0.00264
60	0.0076	0.000129	0.000442
65	0.0328	0.000559	0.001908
70	0.0227	0.000387	0.00132
75	0.0177	0.000302	0.001029
80	0.0189	0.000322	0.001099

Breakthrough time for pH=7

C0= 20.8068ppm

Time (min)	Conc (ppm)	C(mol/m3)	C/C0
0	20.8068	0.3545	1
5	1.3003	0.022154	0.062494
10	0.5681	0.009679	0.027304
15	0.3939	0.006711	0.018931
20	0.2373	0.004043	0.011405
25	0.2664	0.004539	0.012804
30	0.1881	0.003205	0.00904
35	0.173	0.002948	0.008315
40	0.2752	0.004689	0.013226
45	0.2121	0.003614	0.010194
50	0.1868	0.003183	0.008978
55	0.2916	0.004968	0.014015
60	0.3484	0.005936	0.016745
65	0.3952	0.006733	0.018994
70	0.5151	0.008776	0.024756
75	0.6514	0.011098	0.031307
80	0.8092	0.013787	0.038891
85	1.1514	0.019617	0.055338
90	1.5945	0.027167	0.076634
95	1.7235	0.029364	0.082833
100	1.9573	0.033348	0.09407
105	2.1958	0.037411	0.105533
110	2.2358	0.038093	0.107455
115	2.583	0.044008	0.124142
120	2.6275	0.044767	0.126281
125	2.6428	0.045027	0.127016
130	4.2938	0.073156	0.206365
135	5.2745	0.089865	0.253499
140	10.7392	0.182971	0.516139
145	13.6923	0.233285	0.658069
150	17.2385	0.293704	0.828503
155	20.6703	0.352174	0.99344
160	20.7927	0.35426	0.999322

Effect of regeneration of resin

C0= 20.8308 ppm

time	C (ppm)	c/co(regen)
0	20.8308	
5	0.1482	0.007114465
10	0.2845	0.013657661
15	0.3921	0.018823089
20	0.6349	0.030478906
25	0.6724	0.032279125
30	0.6847	0.032869597
35	0.7539	0.036191601
40	0.7934	0.038087831
45	0.8145	0.039100755
50	0.8502	0.040814563
55	0.8843	0.042451562
60	0.8963	0.043027632
65	0.9538	0.045787968
70	1.2058	0.057885439
75	1.2649	0.060722584
80	1.3865	0.066560094
85	1.4326	0.068773163
90	1.5396	0.073909787
95	1.7639	0.084677497
100	1.8993	0.091177487
105	2.9547	0.141842848
110	4.2845	0.205681011
115	7.2592	0.348483976
120	13.5843	0.652125698
125	15.3962	0.739107475
130	17.539	0.841974384
135	18.2643	0.876793018
140	19.6853	0.945009313
145	20.3586	0.977331644
150	20.6386	0.990773278