Removal of Nickel(II) ion from Industrial Waste Water employing Ion Exchange Process

By

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ABSTRACT

The presence of Nickel (II) ion in the wastewater from the metal industry can be unfavourable to living species. Nickel ion in the water caused damaged to human being as well as to the environment. Ion exchange technology was chosen to be applied in this study to treat nickel ion and enhance the quality of the wastewater from the industry. Ion exchange is a reversible chemical reaction wherein ion from the solution is exchanged for a similarly charged ion attached to the resins. In this project, the removal of Nickel (II) ion from wastewater using a strong acid and strong base resin in fixed beds column will be investigated. The purpose of the project is to determine the optimum condition for the removal of metal ions from the waste water solution. The experiments were performed under different pH values (3, 5,and 7) and different pollutant's initial concentrations(1.8, 2.8, and 3.8 g Ni/L). Besides, the effect of regeneration of the resin is conducted and evaluated. The result of the removal efficiency and rate of removal is shown in the breakthrough curves and the kinetic for the process is calculated. The continuous study showed that the exchange isotherm is well fitted by the Thomas Model. Atomic Adsorption Spectrophotometer (AAS) is used to measure the Nickel ion concentration to be compared against the environmental limit. The effect of waste water treatment to the characterization of the resins is determined by using Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). Results from this project can help to design an appropriate heavy metal removal technique by using ion exchange process in order to minimize the negative impacts caused by industrial waste water.

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INTRODUCTION

1.1 Background Study

Based on the Malaysia Environmental Quality Report 2005, 47.5% of the water pollution is generated by industrial sector. One of the most water polluting industries in Malaysia is the metal plating industry. The discharges from metal plating industry mainly consist of many heavy metals such as copper, lead, chromium, nickel, iron and zinc [1]. As far as we know, heavy metals has many negative impact to both human and environmental as it can affects human's health as well as causing environmental pollutions. Nickel for example can cause cancer and anosmia which is lost of ability to smell [2].

Malaysia government has set a standard for Nickel in the wastewater discharge which is 1 mg/L based on the WHO water requirement [3]. Therefore, a suitable heavy metal removal method should be used to comply with the stricter regulation. A number of technologies for the removal of metal ions from aqueous solutions have been developed by other researcher and used in the industry for over the years. The methods include coagulation, chemical precipitation, floatation, ion exchange, adsorption and reverse osmosis [4,5,6].

Ion exchange technology has many advantages as it can treat a large volume of effluent at one time and more efficient in removal of ion from the wastewater [5]. In this method, ion exchange removes unwanted ions mainly Nickel (II) by transferring them to a solid material namely resins. Resins will accept ions from the solutions and giving back an equivalent number of desirable species stored on the resins.

In this process, cation which is Nickel (II) ion is exchanged with Hydrogen ion. Also, anion such as sulfates is exchanged with Hydroxyl ion. This process can remove almost 97% of Nickel in the solutions and producing pure water that save to be discharge to the drainage [4,5].

Ion exchange method is widely applied in many other industry sectors, including petroleum and chemical industries, water softening process, and separation and purification in the food industry [7]. In this project, the concept of removal of heavy metal by ion exchange will be studied and reviewed to identifying the optimum operating parameter for the project. Is it useful in order to ensure the efficiency of the effluent removal from the industrial waste water.

1.2 Problem statement

The interest in heavy metal removal especially Nickel (Ni^{2+}) from industrial wastewater has grown enormously due to stricter effluent limits in Malaysia. Malaysia discharge standards are known as “Malaysia A” and “Malaysia B” exist based on WHO water standards [3]. Therefore, an efficient separation system such as ion exchange is used for Nickel ions (Ni^{2+}) removal from industrial wastewater discharge. Although ion exchange method has been applied in the industry for many years, there appears to be very limited number of studies reported about removal of Nickel ions (Ni^{2+}) from industry employing ion exchange method [4,5]. Hence, an exploratory research is planned to be conducted to identify and quantify the optimum condition for Nickel ions (Ni^{2+}) removal. Some present work by other researcher throws some light on the probable mechanism of the process [4-6]. Therefore, this project will aim to understand the concept of removal of heavy metal by using ion exchange method and select the best operating conditions for the process.

1.3 Objective

The general objective of this project is to enhance the removal of nickel ions (Ni^{2+}) from wastewater by using ion exchange method. Specifically, this project aims to:

- To determine the effect of process parameters in terms of pollutant's concentration and pH on nickel ion removal from the waste water solution.
- To determine the effect of ion exchange process on the characteristic of Ion Exchange resin.
- To investigate the effect of regeneration on the performance of Ion Exchange resin.
- To calculate and identify the exchange kinetics of the Ion Exchange process.

1.4 Scope of study

The ion exchange study is to investigate the ability of the resins to remove heavy metal ion such as Nickel (Ni^{2+}) from aqueous solution over a range of operating conditions. The wastewater used in the experiment is prepared in the laboratory based on the typical metal plating wastewater concentration. The project starts by comparing the suitable heavy metal removal process and suitable resins for the project. The optimum wastewater treatment condition is determined and selected by variation of nickel concentration in the wastewater and pH. A comparative study is also conducted to determine the efficiency of regeneration process to the exhausted resin in removing heavy metal from the waste water in order to comply with regulation

1.5 Significant of Study

This study is focused on the removal of heavy metal which is Nickel (II) ion from the typical industrial wastewater solution. Although many researches and studies have been discussed by others regarding this issue but the details is not sufficient and further work need to be done to enhance the result. Moreover, the purpose of this project is to determine the optimum condition for the removal of heavy metal from the solution to increase the quality of wastewater from the metal plating industry.

CHAPTER 2

LITERATURE REVIEW

2.1 Wastewater from Metal Plating industry

Metal plating industry is the major source for industrial wastewater in Malaysia as 27% of toxic and hazardous wastes are from metal planting industry [8]. The metal planting process involves alkaline cleaning, acid pickling, plating and rising. During the process, water is used to cleanse the surface of the part after each process bath and copious amounts of wastewater are generated through this step [9].

Wastewater from metal plating industry contains heavy metals, oil and grease, and suspended solids, at levels which are hazardous and harmful to human and environment. Heavy metal presents in metal plating wastewater are consists of cadmium, chromium, copper, lead, nickel, silver, tin, and zinc [10]. However, nickel in particular is the main concern because of the toxicity to human and can cause various serious illness to humankind such as cancer and poisoning [2].

Hence, the wastewater is required to be treated prior to releasing it to municipal sewers or surface water based on the requirement from Department of Environment Malaysia.

2.1.1 Heavy Metal Removal Techniques

With greater quantities of wastewater produced and discharge standards becoming increasingly more stringent in Malaysia [1], there is a need for more efficient and cost-effective methods for removing heavy metals. Thus, a number of techniques for the removal of heavy metal have been developed over the years.

The heavy metal removal process can be accomplished by filtration, precipitation [10], adsorption[6], ion exchange[4,5] and reverse osmosis. All these techniques have their inherent advantages and limitation. Table 1 shows the advantages and disadvantages of different techniques in heavy metal removal.

Table 1: Comparison of Heavy Metal Removal Techniques.

Removal technique	advantages	Disadvantages
Filtration	Low cost, effective for removal of particulate.	Less effective and produced concentrated sludge.
Precipitation	Low cost	Required longer time and extensive setup. Concentrated sludge production.
Adsorption	Widely used in industry.	Ineffective for very low concentration of metal
Reverse osmosis	Effective and lowered the toxic metal level.	Expensive and sophisticated. Requiring a higher level of technical expertise to operate.
Ion exchange	Effective and can treat large volume of solution. Not producing sludge.	Ineffective to mono and divalent ion.

Comparing all techniques in the Table 1, ion exchange has the main advantages that it can remove Nickel ion from the wastewater without generating concentrated sludge. Besides, it permits rinse water to be reuse in the closed cycle and directly recover 97% of Nickel form the solution to be reuse in the process [12]. Thus, ion exchange is effective technique for Nickel removal from metal plating industry wastewater.

2.2 Ion Exchange Technique

2.2.1 Background

Ion exchange is a reversible chemical reaction wherein an ion from the solution is exchanged for a similarly charged ion attached to an immobile solid particle [11]. The immobile solid particle is referred to zeolite or synthetic cation and anion resins. The ion exchange process is a chemical reaction process between ions in liquid phase and ions in solid phase. In the process, certain ions in the solution are sorbed by the ion exchanger solid, then, the ion exchanger solid releases replacement ions back into the solution to maintain the electroneutrality. The reactions are stoichiometric and comply with the law of mass action [7,11]. Ion exchange is used extensively in wastewater treatment plant for water softening, demineralization, desalting, ammonia removal and treatment of heavy metal wastewater [5,7].

2.2.2 Ion Exchange Process

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.

$$NiSO_4\,(aq) + 2\,R.H(s) \leftrightarrow R_2.Ni\,(s) + H_2SO_4(aq) \qquad \text{(equation 1)}$$

$$Ni^{2+}\,(aq) + 2\,R.H\,(s) \leftrightarrow R_2.Ni\,(s) + 2H^+\,(aq) \qquad \text{(equation 2)}$$

Where, R represent the exchanger solid (cation resins)

Based on equation above, wastewater solution which is contains Nickel(II)Sulfate solution is fed to a hydrogen cation exchange resins 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+}) [7,13].

Because of the high concentration of hydrogen ion (H^+) generated in the first process, the acid solution is then removed by passing the effluent through an alkali regenerated anion exchange resin which replaces the anions in the solution (SO_4^{2-}) with an equivalent number of hydroxide ion (OH^-)[11]. The reaction is described as below;

$$H_2SO_4\ (aq) + 2R.OH(s) \leftrightarrow R_2.SO_4\ (s) + 2H_2O\ (aq) \qquad \text{(equation 3)}$$

The hydrogen ions generated from cation exchanger unit and hydroxide ion generated from anion exchanger unit neutralize each other to form an equivalent amount of pure water [11]. During ion exchange process in both cation and anion ion exchanger unit, the resins will become exhausted and need to be regenerated.

The cation resin is regenerated with sulfuric acid and anion resin is regenerated with Distilled water or strong base such as Sodium Hydroxide, NaOH[5]. The reaction for cation regeneration process is shown below.

$$Ni.R_2\ (s) + H_2SO_4\ (aq) \rightarrow 2\ R.H\ (s) + NiSO_4\ (aq) \qquad \text{(equation 4)}$$

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, H_2SO_4 with 5 to 10 % concentration, the reaction will drive to the left side.

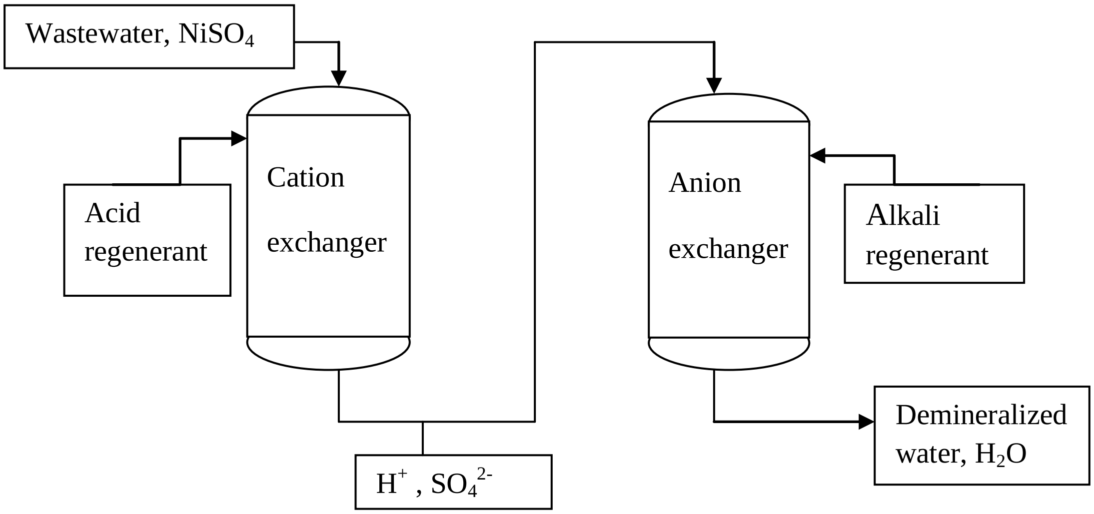


Figure 1: Typical Flow Diagram for the Removal of Heavy Metal using Ion Exchange Resins for Two-Bed Column. Adapted from Matcalf &Eddy , Wastewater Engineering Treatment and Reuse, McGrawHill, n.d[8].

2.2.3 Types of Resin

Exchange resins are usually bead or granular-shaped with size of 0.1 to 1.0 mm. Ion exchange resin beads contain many fine pores that will fill the water , Figure 2 shows the pores of the ion exchange resins. The resins can be classified to four major classes which are strong acidic cation resins, weak acidic cation resins, strong basic anion resins and weak basic anion resins. Each of these major resin classes has several physical or chemical variations within the class. The variations impart different operating properties to the resin. The selection of ion exchange resins is based on the peak efficiency of the resins and maximum cost effectiveness [15].

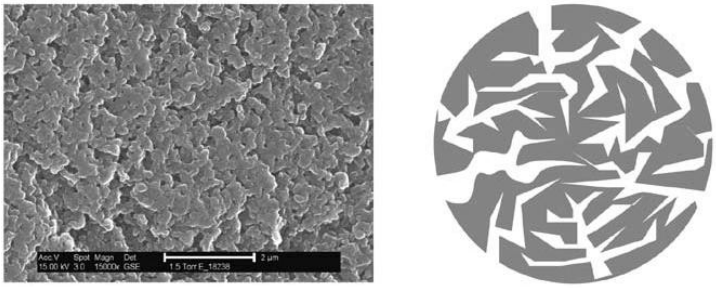


Figure 2: Ion Exchange Resins Pores. Source: Principle of Ion Exchange in Wastewater Treatment, Asian Water, March 2009[13]

The acidic resins are cation exchanger because of the hydrogen ion (H^+) contains in the resins can attached to the negatively charge exchange site[7]. The strong acidic cation resins remove all cations from solutions, where as the weak acidic cation resins will remove ions such as calcium and magnesium but have limited ability to remove ions that situated at the bottom lines in the displacement series table. However, weak acidic cation resins is highly efficient compared to the strong acidic cation resins as it only needs 110% of the stoichiometric amount of acid compared to 200 to 300 % for the weakly acidic resins[13].

For basic resins, the exchange site is positively charged to allow hydrogen ion(H^+) and other basic species such as amine group attach to it. The strong basic anion resins have an ability to remove all anions in the displacement table. Conversely, the weakly basic only removes the anions of strong acids [11].

For removal of heavy metal in the industrial wastewater, although weakly acidic cation and weakly basic anion have greater affinity for counter ion, strong resins were more favorable as the solid exchanger because of the advantage in the pH. It is because, pH is essential in the ion exchange process in wastewater in order to comply with certain environmental requirement [5].

2.2.4 Resin Selectivity

Selection of suitable resin is the most crucial parts in ion exchange process because different ion has a tendency to react differently with functional group of ion exchangers. Some ion will have great bound where as some ion will less strongly bound [14]. This situation is called resins selectivity. The selectivity of the resins is based on displacement series for ion exchange which is shown in Table 2. An ion exchanger tends to prefer higher valence ions, bigger atomic number, small solvated volume ions, ions with greater ability to polarize and ions that participate least with other ions to from complexes [7].

Table 2: Displacement Series for Ion Exchange. Source: adapted from Physical-chemical treatment of water and wastewater [12]

Cation	Anion
Pb^{2+}	SO_4^{2-}
Ca^{2+}	CrO_4^{2-}
Ni^{2+}	NO_3^-
Cd^{2+}	AsO_4^{3-}
Cu^{2+}	PO_4^{3-}
Zn^{2+}	$MoOa^{2-}$
Mg^{2+}	I^-
Ag^+	Br^-
Cs^+	Cl^-
K^+	F^-
$NH4^+$	OH^-
Na^+	
H^+	

2.2.5 Exchange Capacity

Exchange capacity is the ability of an insoluble material to undergo displacement of ions previously attached and loosely incorporated into its structure by oppositely charged ions present in the surrounding solution [16]. Resin capacity is usually expressed in terms of equivalents per liter (eq/L) or (kg/cu.ft) of resin. An equivalent is the molecular weight in grams of the compound divided by its electrical charge or valence. The capacities are strongly influenced by the quantity of acid or base used to regenerate the resin[17]. For example, weak acid and weak base systems are more efficiently regenerated during regeneration process because their capacity increases almost linearly with regenerant dosage. Thus, the resin exchange capacity will strongly affected the rate of ion exchange between heavy metal ion (Ni^{2+}) and ion in the resins. Resin capacity can be calculated by using Thomas Model [19].

2.2.6 Characterization of the resin

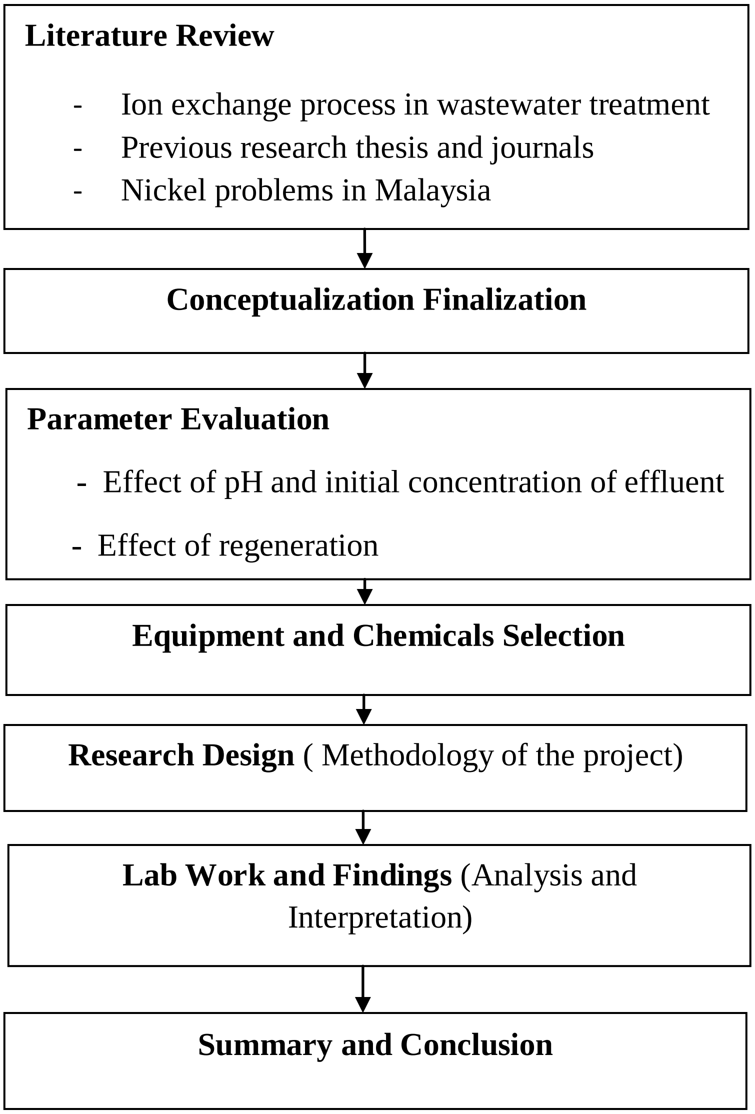
Table 3 : Method of Characterization Study for Ion Exchange Resin

Method	Purpose	Working principle
Scanning Electron Microscope (SEM)	To observe the surface structure for the particle. SEM shows very detail 3 dimension image that can be magnified up to 500 000 times.	The magnified image is produced by scanning the particle with high-energy beam of electrons in a raster scan pattern.
Fourier Transform Infrared Spectroscopy (FTIR)	To provide information regarding the amount of elements in the particle based on the functional group. To identify unknown material in the sample.	FTIR Spectrum is produced by the different in IR radiation that passed through the sample.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology



3.2 Project Methodology

3.2.1. Overview

The experiment will be conducted in continuous mode and the rinse water used in the test is prepared by dissolving nickel sulfate ($NiSO_4.6H_2O$) in distilled water. The project is divided into two main parts which are studies of removal of Nickel from wastewater solution based on different parameters and studies of regeneration effect on the removal process. All experiments were conducted at similar temperature and flowrate which are at 25^oC and $35\ cm^3/min$. The diameter of the column used for the experiments is 2 cm with 60 cm height.

For the removal of nickel from wastewater, the parameters are varied according to acquire the effects of pH and the effects of initial concentration of Nickel [19-20]. The pH of solution is manipulated for pH=3, pH=5 and pH=7 based on the pH range described in the Material and Safety Data Sheet (MSDS).The pH is adjusted accordingly using 5% NaOH and 97%H_2SO_4.

For the second parameter which is Initial Nickel Concentration, the experiments are carried out by manipulating the initial concentration at 1.8 gNi/L, 2.8 gNi/L and 3.8 gNi/L. Nickel Sulfate is weighted by using electronic balance and diluted with distilled water to obtain the preferable concentration.

In the second part of the project, the effectiveness of the resin regeneration is studied. The experiment is conducted by using 1.8 g Ni/L initial Nickel (II) concentration and the Sulfuric Acid(H_2SO_4) is allows to flow through the column for about 1 hour until the conductivity of the effluent similar with the conductivity of the influent.

The parameter of the effluent will be tested by using pH meter, and absorption atomic spectrophotometer (AAS). Characterization of the resins will be determined by using Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). Operating manual for Ion Exchange unit is attached in Appendix B.

3.2.2 Pretreatment of the Resins

Before the experiment was carried out, the resin had to be pre-treated. The purpose of pretreatment is different with the objective of the experiment. The method of pre treatment was applied for both cationic and anionic resins. For both type of resin, the resins were packed in the cation column up to 10 cm height. After that, the resin was rinsed in downward flow patten by using dionized water excessively. For cationic resin (Lewatit S 1467), the Hydrogen ion (H^+) inside the dionized water substituted with Sodium ion (Na^+) inside the resins. In other hand, anion resins (Amberlite ARA 402) substituted it's ionic form from Chloride ions to hydroxyl ions that available in the deionized water. The exact procedure for the process was attached in Appendix B.

3.2.3 Regeneration of the Resins

Resins have limited capacities and must be regenerated upon exhaustion. Therefore, regenerating cationic resin in the cation column used 5% Acid Sulfuric (H_2SO_4) solution in downward flow pattern [17]. The flowrate of the regenerant was set at 35cm^3/min and minimum contact time is allowed for 40 minutes. Then, the regenerated resin was rinsed with deionized water. For anionic resin, 5% Sodium Hydroxide (NaOH) was rinsed in downward flow pattern inside the anion column. After the 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 tubed 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 anionic column, had a diameter of 2 cm and length of 60 cm. The Schematic diagram of Ion exchange Unit is attached in the Appendix A.

Figure 3 : 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. Conductivity meter was used to quantify the total ion content in the liquid.

Figure 4 : Atomic Absorption Spectrophotometer

3.3.3 Materials

i) Resins

Synthetic resin was used in experimental studies. Cation resin used in this experiment is Lewatit S1467 , produced by Lanxess. The resin is in Na^+ ionic form and was from sulfonic acid functional group. The resin is light brown, gel type beads that have crosslinked polystyrene matric. Anion resin is a strongly basic anion exchange resin with pale yellow translucent beds. The resin is based on crosslinked polystyrene and contains Chloride (Cl^-) ionic form.

Table 4: Physical and Chemical Properties

	Lewatit S 1467	Amberlite IRA 402
Total Capacity	2.0 eq/L	1.3 eq/L
Mean Bead Size	0.60 mm	0.60 mm
Maximum Swelling	8% (Na^+ to H^+)	30%(Cl^- to OH^-)
Bulk Density	8.20 g/l	670 g/l

ii) Chemicals

Nickel (II) sulfate ($NiSO_4.6H_2O$) made by S&M Chemical Corporation to prepare the wastewater. In order to regenerate cation resin, Sulfuric Acid (H_2SO_4) manufactured by Merck chemical was used. Meanwhile, Sodium Hydroxide (NaOH) manufactured by Merck chemical was employed to regenerate the anion resins.

3.4 Key Milestone.

This project is divided into two parts and completed in Two Semester period. The research work is done in Final Year Project I, while the experimental work is been carried out in Final Year Project II. At the end of the project, the parameters will be verified accordingly and the results will be studied and analyzed thoroughly.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Resin Characterization

4.1.1 Analysis of cation resin by SEM

In order to study the surface structure of the resins, the analyses were performed by scanning electron microscope (SEM). Four samples of cation resins were used which are fresh resin, resin after treatment with concentration of 1.8gNi/L, resin after treatment with concentration of 2.8 g Ni/L concentration and resin after treatment with concentration of 3.8 g Ni/L.

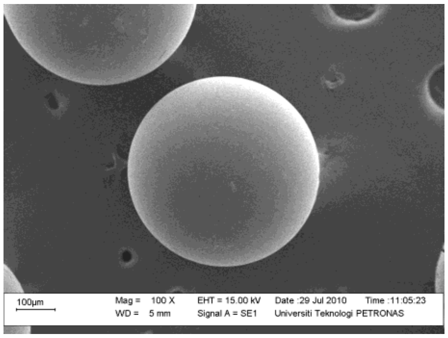


Figure 5: SEM Structure for Fresh Resin (1x100)

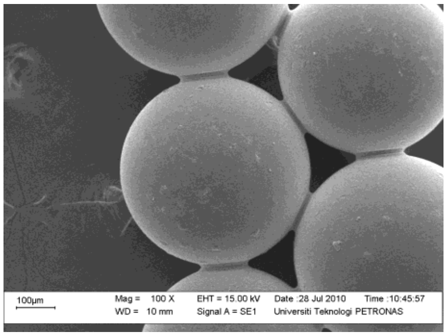


Figure 6: SEM Structure of Cation Resin after Treatment with 1.8 g Ni/L (1x100)

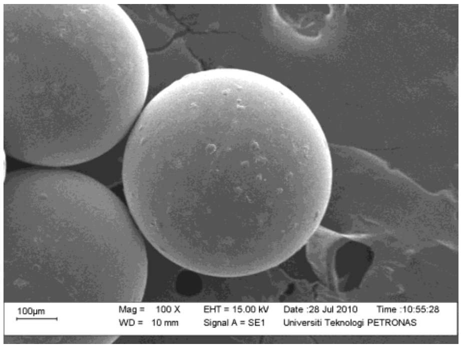


Figure 7: SEM Structure of Cation Resin after Treatment with 2.8 g Ni/L(1x100)

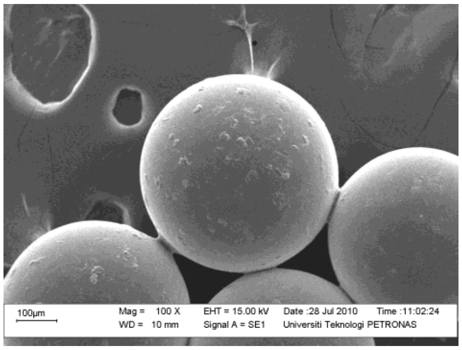
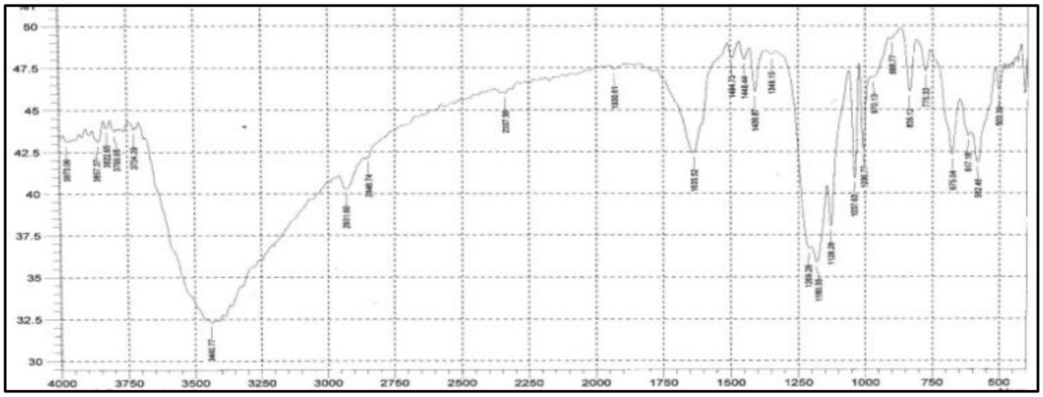


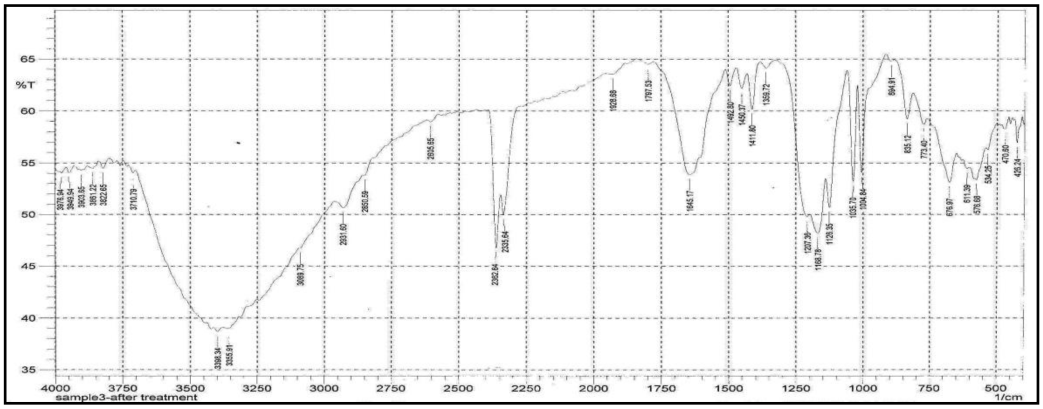
Figure 8: SEM Structure of Cation Resin after Treatment with 3.8 g Ni/L(1x100)

The SEM images in the Figure above shows the effect of different concentration to the resin morphological structure. Figure 5 shows the fresh resin before wastewater treatment. The fresh resin surface had minimum flakes because no exchange between ion occurred in the resin. As the initial concentration of the solution increases, the amount of flakes and cracks appear more visible. Figure 6 shows that the minimum flakes appeared at the pores of the resin, however, figure 8 shows that the flakes and cracks appeared more visible as the cracks and flakes was formed in the surface of the resin. The change of the surface picture is due to change of its chemistry which is proven later by using FTIR. The same result was observed by ZHU Shaomin *et. al*, they observed that the possibility for the ion exchange sites changed from being in the intraparticle space to on the surface of the resin when the Ni(II) concentration increased due to particle diffusion rate control model[25].

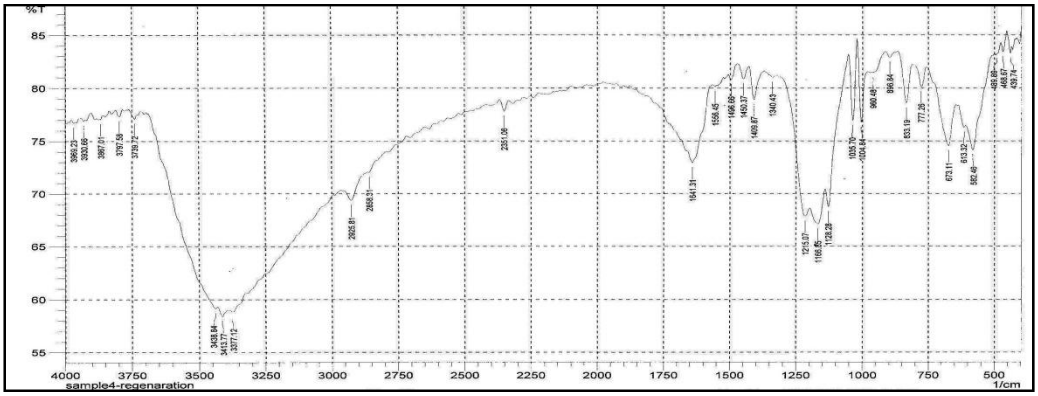
4.1.2 FTIR Analysis



(a) After Pre-Treatment with Deionized Water



(b) After Nickel Loading



(c) After Regeneration with Sulfuric Acid

Figure 9: 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 pre treatment, cation after loaded with Nickel and cation after regeneration. FTIR picture in Figure 9 shows that the the broad and intense peak at around 2400 cm^{-1} was referring to OH group due to the present of free hydroxyl group and water in the resin. The band at 2931.60cm^{-1} indicates -CH_2- stretching vibration of acyclic raised by electronegative substituent in the solution from the used of plastic laboratory ware. Peak observed at 1635 cm^{-1} is the stretching vibration of primary amine –N-H- . The peak at 1209.28 indicates there is secondary amide group (N=H) available in the resin.

FTIR spectra of Nickel ion loaded resin in Figure 9(b), shows that a sharp decrease of the peak at the range around 2335.64 cm^{-1} to 2362.63 cm^{-1} which signifies NH_3^+ region. Due to the positive ions present in the resin, the ions have a tendency to exchange with other positive ions available in the solution. Thus, after the wastewater treatment cycle, the concentration of nickel ion in the resin increased as the concentration of NH_3^+ keep on decreasing. The increasing and broad peak at 1800 cm^{-1} indicates the existent of carbonates group (C=O) in the resin. From Figure 9, the band had shifted from 3440.77, 2931.60, 2337.50, 1635.52 and 1209.28 cm-1 at Figure 9(a) to 3398.34, 2925.81, 2362.64, 1635.52 and 1215.07 cm-1 at Figure 9(b). This is due to the existence of predominant contributor for nickel uptake in the bonds.[26]

Regeneration process in the 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 is similar with the pre treatment resin which can offer the same condition to be used for practical treatment purpose. From Figure 9(c), it clearly shows that the peak at 2335.64 has increased to the initial position as Figure 9(a) indicating the NH_3^+ group is recover from the regeneration process. Besides, the regeneration process also managed to shift back the stretching band from 2931.60 cm^{-1} at Figure 9(b) to 2931.60 which is similar with the band for pretreatment resin in Figure 9(a).

4.2 Effect of Initial Concentration

Nickel ion 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 g Ni/L , 2.8 gNi/L and 3.8 gNi/L of Nickel (II) Sulfate Solution. In order to minimize the effect of other parameters to the removal of Nickel (II) ion, the wastewater solution is prepared at pH=5.6 and flows through the column at constant flow rate 35 cm^3/min. Both 2 cm column is filled with cation and anion resins respectively until the resin approaching 10 cm height in the column.

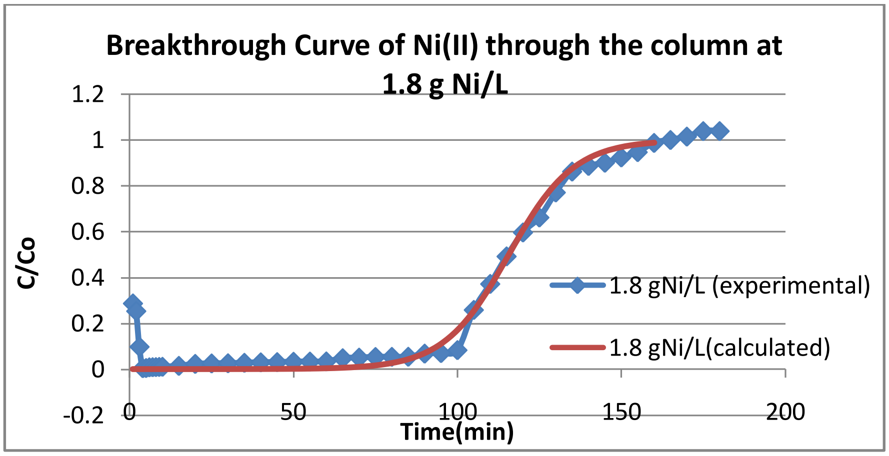


Figure 10: Breakthrough curve of Ni(II) through the resins column at 1.8 g Ni/L

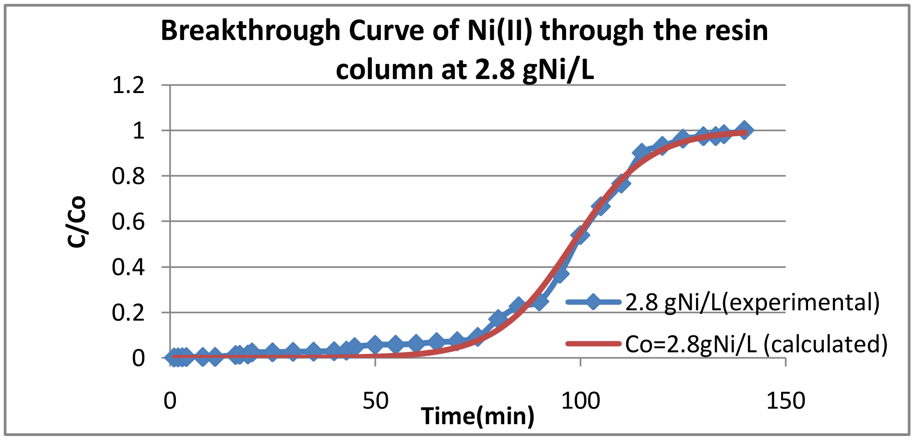


Figure 11: Breakthrough curve of Ni(II) through the resin column at 2.8 gNi/L

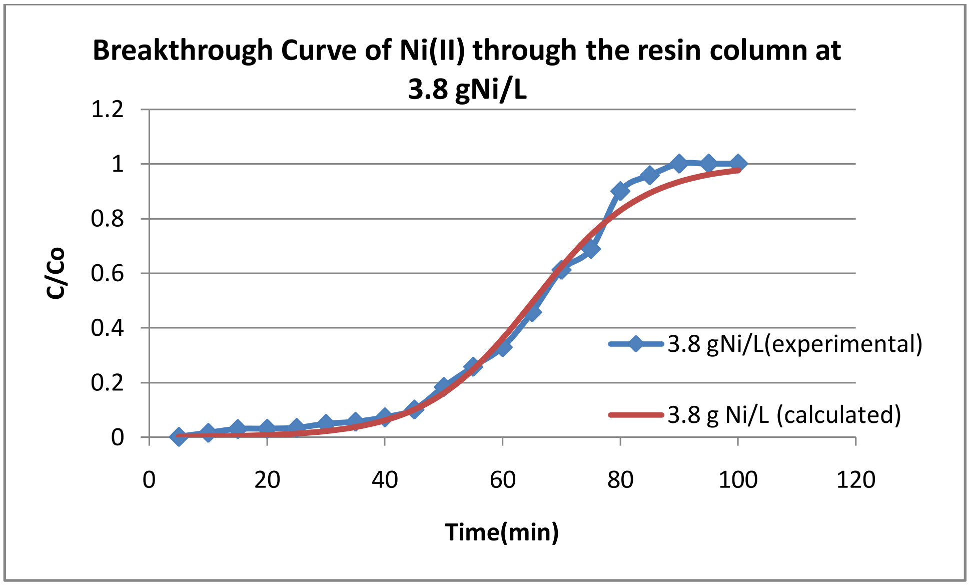


Figure 12 : Breakthrough Curve of Ni(II) through the resin column at 3.8 g Ni/L

Table 5: Breakthrough time for different initial concentration

Initial concentration (gNi/L)	Breakthrough Time(min)
1.8	60
2.8	43
3.8	15

Breakthrough curves at different initial nickel concentration (C_o) are shown in Figure 10, 11 and 12. Breakthrough time in this experiment is defined as time taken for the 1ppm of Nickel ion concentration to be detected at the outlet stream. After the breakthrough time exceeded, the resin cannot be used and regeneration is needed in order to put the resin in service cycle again. At the chosen breakpoint concentration of C_o=1ppm, the time taken for breakthrough decreases with an increase in C_o values. Figure 10 shows that the breakthrough time for initial concentration at 1.8g Ni/L is at 60 minutes, whereas Figure 12 shows the breakthrough time for initial concentration at 3.8 g Ni/L is at 15 minutes.

This situation due to the fact that for a given flow rate and quantity of resin, the exchange sites of the resin are exhausted earlier when a higher initial nickel concentration influent is encountered. The amount of Nickel ions in the solution is higher as the nickel concentration increasing, hence, Nickel ions will occupy the exchange sites faster until the column become exhausted. Therefore, the operation period until the breakthrough point is less at the higher initial concentration. Based on A.H. Norzilah *et. al*, the mass transfer between the ion in the wastewater solution and ion attached in the resin will increases as the initial concentration of the solution increasing [22]. The higher concentration gradient is the driving force for the mass transfer in the column. Thus, the ion in the solution will exchange faster with ion that attached in the resin and this situation will lead the resin exhausted fast.

The same result is achieved by L.Lv *et. al* for their study entitled Effect of operating conditions on the removal of Pb^{2+} by microporous titanosilicate ETS-10 in a fixed-bed column. Although in the study another absorbent was used, but, the trend for the breakthrough curve is similar. They concluded that a rise in the feed concentration reduced the volume treated and the breakthrough point for the system[23].

The solid curve shown in the Figure 12 indicates 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 as the model for kinetic study[24]. Thomas model is widely used in method in column performance theory because it included many parameters that affecting the ion exchange process performance. The model is represented by:

$$\frac{C}{Co} = \frac{1}{1+\exp\left(\frac{kT}{Q}\left(qo\ W - Co\ Vout\right)\right)} \qquad \text{(equation 5)}$$

The linearized form of Thomas model

$$\ln\left(\frac{Co}{C} - 1\right) = \frac{k_T\ q_o W}{Q} - \ k_T Co\ t \qquad \text{(equation 6)}$$

Where C_o is the inlet metal concentration (mol/m^3), C is the outlet concentration at time t (mol/m^3),k_T the Thomas rate constant (m^3/(mol min)),Q is the volumetric flowrate (m^3/min) ,q_o is the maximum resin-phase concentration of metal equivalent to an equilibrium liquid concentration of C_o (mol/kg),W is the amount of resin in the bed (kg),and V out is the effluent volume (m^3).

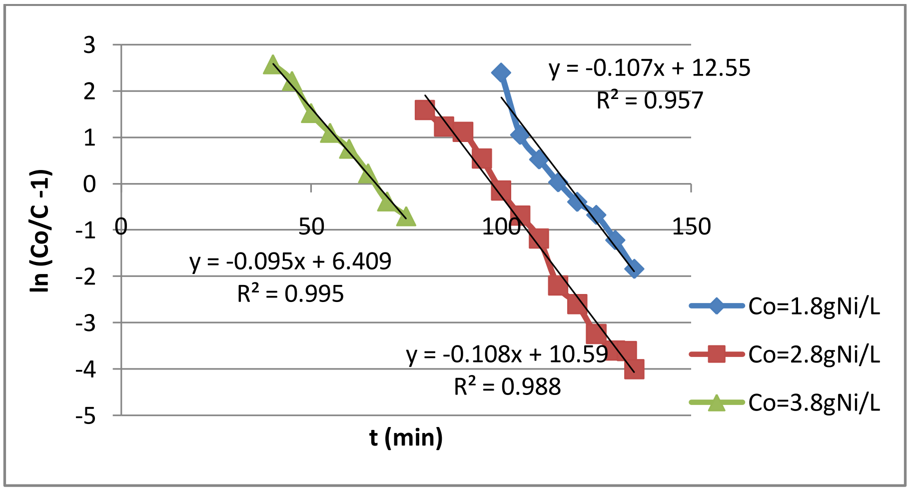


Figure 13: Graph for determination of Thomas parameters and exchange capacity for different inlet concentration

Table 6: Properties of Ion Exchange for Different Initial Concentration

Initial Concentration (gNi/L)	Exchange Capacity,q_o (mol/kg)	k_T
1.8	0.0454	0.242
2.8	0.0464	0.199
3.8	0.034	0.166

The value for exchange capacity ,q_o and Thomas coefficient is determined based on graph ln((Co/C)-1 versus time as illustrated in the Figure 13. Table 6 clearly shown that as the initial concentration increased from 1.8g Ni/L to 2.8 g Ni/L, the exchange capacity is increase from 0.0454 mol/kg to 0.0464 mol/kg which is increasing with 2.2%.

It found that R.S Juang *et. al* also reported similar outcome in their research which is the exchange capacity of the resin will increase as the initial concentration of the nickel increasing [19].Besides, from the Thomas Model, it can be concluded that the exchange capacity of the resin is inversely proportional with k_T .

Based on the k_T and q_o extracted from Figure 13, the breakthrough curve can be constructed. The differences between experimental values and calculated value for Thomas's Model are small which is in the range below 5%.

The colour of the wastewater has significant changes from before treatment and after treatment. The typical wastewater from industry which contains Nickel (II) ions is usually in green colour. After the treatment, the colour for the effluent changed to colourless and it has pH=7 which is safe for drainage and complying with regulation.

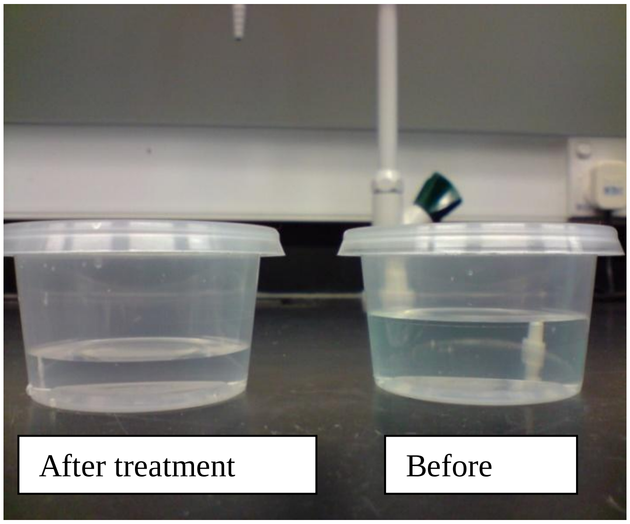


Figure 14: Different colour of solution for wastewater.

4.3 Effect of pH

The effect of pH on nickel removal is conducted with resin dosage of 31.41 cm^3 (diameter 2 cm and height 10 cm) and initial nickel concentration of 1.8 g Ni/L at room temperature. The waste water solution is continuously flowed at 35 cm^3/min. Sulfuric Acid (H_2S0_4) was used to reduce the pH to 3 and Sodium Hydroxide (NaOH) was used to increase the pH to pH=7.The range of the pH is kept below pH 8 to avoid chemical precipitation. S.Kumar reported that the participation at high pH values will affected the exchange capacity due to domination of the chemical precipitation [27]. Figure 15 to 17 show the breakthrough curve for Ni(II) ions flow through the column at different pH.

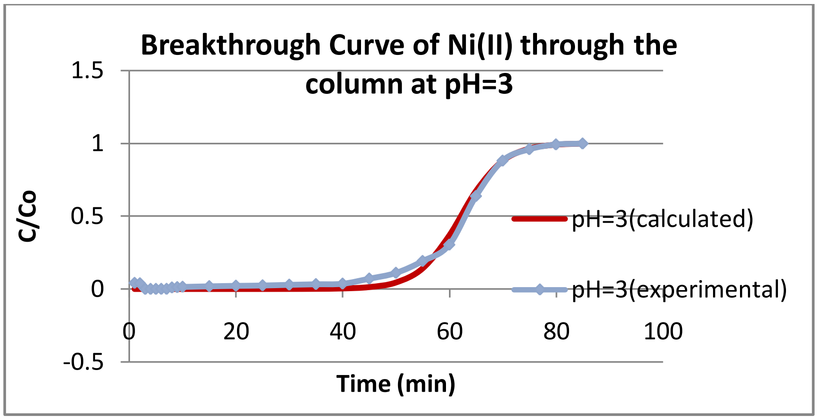


Figure 15: Breakthrough Curve of Ni(II) through the resin column at pH=3

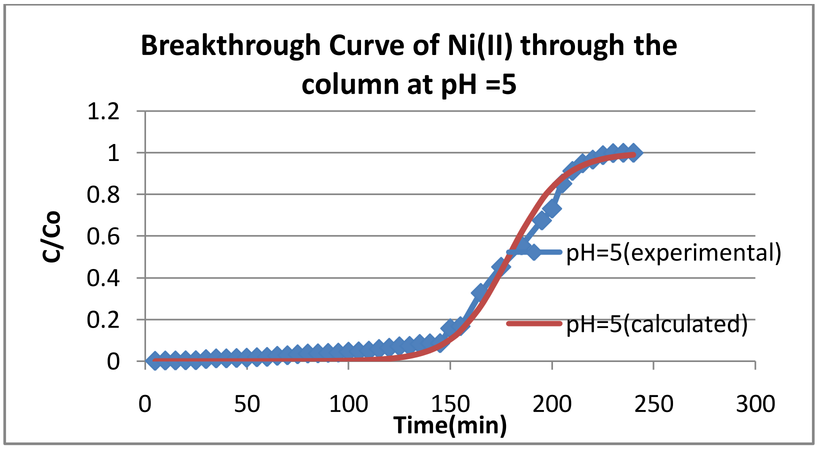


Figure 16: Breakthrough Curve of Ni(II) through the resin column at pH=5

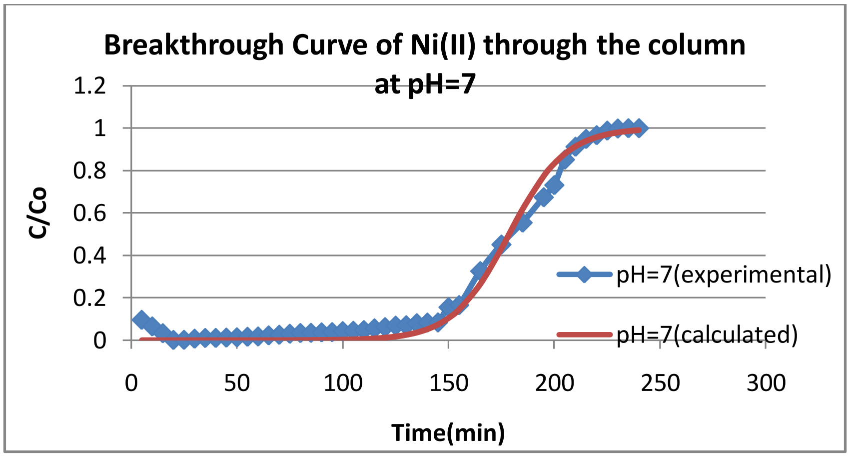


Figure 17: Breakthrough Curve of Ni(II) through the resin column at pH=7

Table 7: Breakthrough time for different pH

pH of the wastewater solution	**Breakthrough Time(min)**
3	40
5	60
7	75

The above result shows that the breakthrough time is highly dependent with the pH of the solution. Figure 17 demonstrates that the higher the pH , the longer the breakthrough time but it is only applicable in range between 3 to 7 . At pH =3, the breakthrough time for the resin is faster compared to breakthrough time at pH= 5 and 7 which are 60 and 75 minute. This is due to the facts that amount of hydrogen ion present in the solution at lower pH is higher . Thus, the Nickel ion needs to compete with the hydrogen ion during ion exchange process. Hydrogen ions occupied the vacant exchange site and exhausted the column faster. Based on the result, moderate pH values around pH 5 to 7 is acceptable to optimize the Nickel(II) removal from waste water.

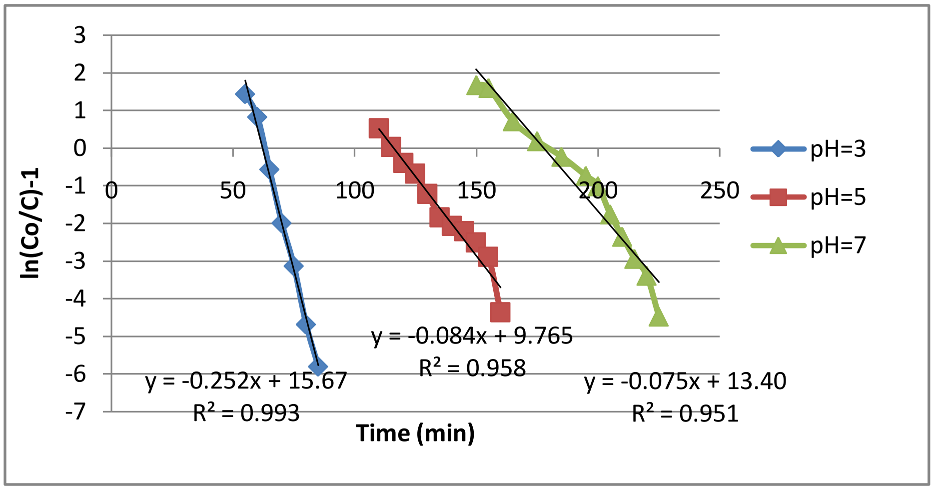


Figure 18: Graph for determination of Thomas parameters and exchange capacity for different pH.

Table 8: Properties of Ion Exchange for Different pH

pH of the wastewater solution	Exchange Capacity,q_o (mol/kg)	kT
3	0.02407	0.569
5	0.0454	0.242
7	0.0823	0.142

The value for exchange capacity,q_o and Thomas coefficient is determined based on graph ln((Co/C)-1 versus time as illustrated in the Figure 18. Table 8 clearly shown that as the pH decreasing from 7 to 3, the exchange capacity is decreases from 0.0823 mol/kg to 0.02407 mol/kg. As mention from Kumar, the excessive protonation of the ions at the exchange surface will refuses the formation of links between Ni^{2+} ion and the active ion in the resins [27]. The amount of exchanged ions is lower due to higher competition between Nickel ions and Hydrogen ions at the vacant site, thus the exchange capacity for the process is lower.

The similar patent is found at R.S Juang project which is indicates that exchange capacity of the bed increases with increasing pH. He also stated that the trend is consistent with the trend of q_{sat} from equilibrium studies[19].

4.4 Effect of Regeneration

For removal of Nickel (II) ion from waste water in plant, the regeneration of exhausted ion exchange resin is important in order to use the resin back into the service cycle. The repeated usage of resin can minimized 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 exchanged with the Nickel(II) Ni^{2+} 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 flowrate of 35 cm^3/min. Figure 19 , shows the effect of regeneration to the resin.The breakthrough time for virgin resin is 60 minutes and the time for regenerated resin to breakthrough is 50 minutes. The result indicated that the regeneration resin become exhausted first before virgin resin because of the presence of other ions in the unregenerate resin. By using Thomas Model, the exchange capacity for the resin is predicted.

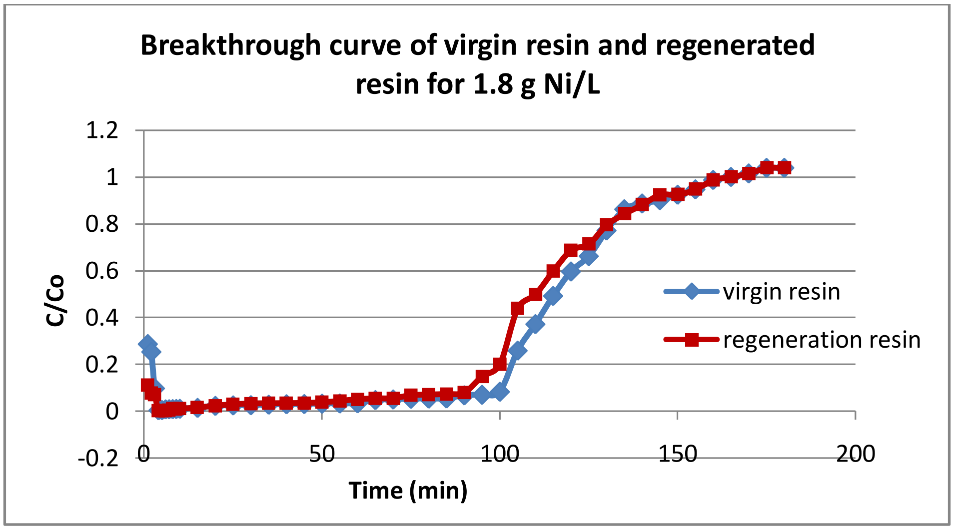


Figure 19: Breakthrough curve of virgin resin and regenerated resin for 1.8 g Ni/L

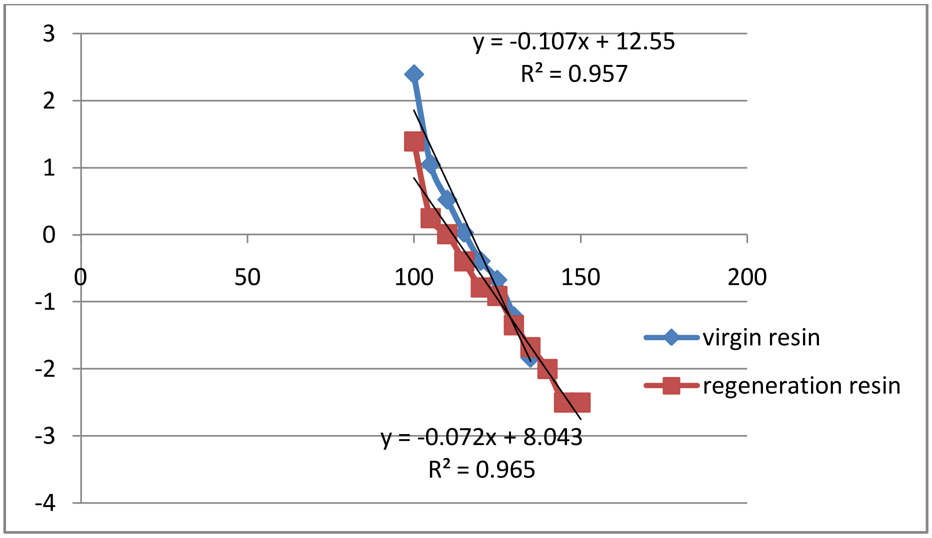


Figure 20: Graph for determination of Thomas parameters and exchange capacity for virgin resin and regeneration resin.

Table 9: Properties of Ion Exchange for Regenerated resin

	Virgin resin	Regenerated resin
k_T(m^3/mol.min)	0.24	0.162
Exchange Capacity(mol/kg)	0.045	0.043

The result in Table 9 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% percentages different. Hence, the regenerated resin can be used back in the process cycle without affecting on the capabilities of ion exchange resin. Similar trend of result can be seen in R.Ansari and F.Raofie research, they concluded that regenerated sorbent can effectively adsorb ions without losing the capacity as the loss in the capacity was only 2%[28]. Therefore, the resin can be regenerated in order to minimize the cost of purchasing new resins.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.0 Conclusion

Comparing with other heavy metal removal method used in the industry, ion exchange technology will be applied to this study to treat nickel ion from plating wastewater. This technique is used because Ion Exchange technology has many advantages as it can treat a large volume of effluent at once and more efficient in removal of ion from the wastewater. From the experiment conducted in the laboratory scale, the removal of Nickel (II) ions from waste water solutions were successfully enhanced by using Ion Exchange method.

From the different initial concentration experiments, it can be concluded that initial concentration affected the breakthrough time and exchange capacity for resin. The breakthrough time is decreases from 60 to 15 minutes with increasing initial Nickel(II) concentration in a range of 1.8,2.8 and 3.8 gNi/L. Besides, the exchange capacity of the resin is increasing from 0.0454 to 0.0464 as the initial concentration keep on increasing from 1.8 to 2.8 gNi/L. This situation is due to mass transfer behavior Based on the result, the optimum condition for the ion exchange system is at lower initial concentration as it will decreases the need to regeneration and reduce the cost of the operation. The effluent after treatment is colourless and has pH=7 which is complying with the regulation.

Besides, the optimum pH for the removal is at the middle range which is around pH 5 to 6. At pH below 2, adsorption is impossible due to the protonation meanwhile at pH above 7, chemical precipitation forms metal complex which is no longer considered removal via ion exchange process.

Based on the experimental works, the ion exchange process is optimized at the initial concentration of 1.8 gNi/L and pH 5.The effect of the resin regeneration is studied and it can be concluded that regeneration did not affect the exchange capacity, thus, the urge to buy new resin is minimized. The result shows the loss of exchange capacity between virgin resin and regenerated resin is about 4%. Results for all experiments are used for calculating kinetic of the process. The Thomas Model was applied to describe the breakthrough curve for the process. Most of calculated breakthrough curves were closely fitted with experimental results; hence, it can be used to predict the exchange capacity of the resin.

The SEM Figure shows that the exchange site can be changed from intraparticle to the surface of the resins as the concentration of Nickel ion increases. In conclusion, Ion Exchange Process is an effective way to remove heavy metal in the waste water treatment. This process appear more reliable compare to other expensive process. Results of this study can be applied for practical proposes.

5.1 Recommendation for Future Works

The project has met the expected results and goals, however, some improvement can be made for the current project. In order to improve the exchange capacity and efficiency of the process, the experiment must be carried on by using mixed bed column where cation and anion resins in put together in a single column. These properties have a significant influence on the removal of heavy metal. For instance, mixed bed column will produce higher quality water with a lower total capacity than two bed system. Hence, the amount of resins needed for every process is lower and the operational cost for the plant will be minimized. Besides, it is recommended to investigate the effect of other operating parameters such as flow rate of waste water solution, temperature and resin dosage towards Nickel removal.

CHAPTER 6

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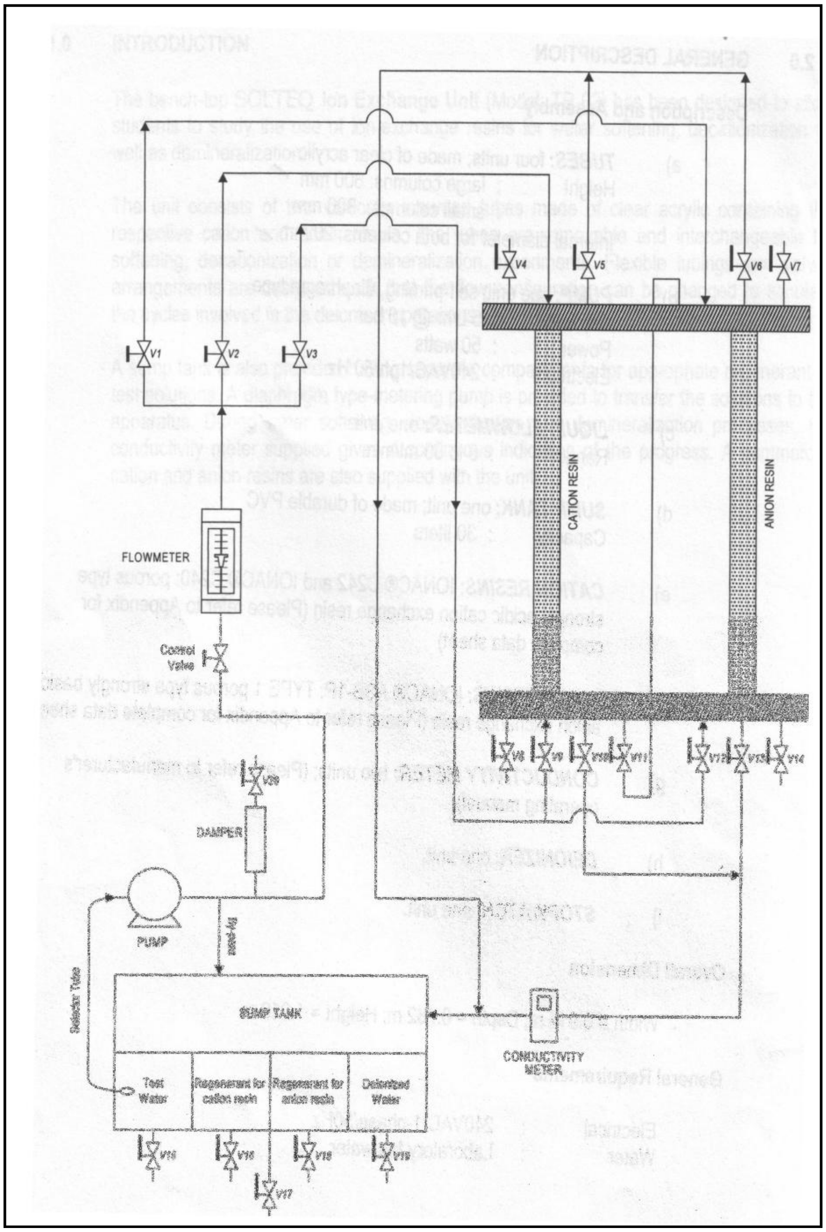
CHAPTER 7

APPENDICES

APPENDIX A: Schematic Diagram of Ion Exchange Unit



APPENDIX B: Operational Procedure for Ion Exchange Unit

Experiment 1: Removal of Nickel (II) ions from industrial wastewater by using two-bed exchange

Pre treatment for resin

1. Lewatit S 1467 type resin is filled into the cation column up to 10 cm height.
2. Amberlite IRA 402 type resin is filled into the anion column up to 10 cm height.
3. Both columns are placed to Ion Exchange Unit.
4. Deionized water is prepared in the appropriate reservoir. The conductivity meter reading of the deionized water is recorded.
5. The cation and anion resin is rinsed simultaneously in downward flow pattern by opening valves V1,V2,V10 and V13.
6. The selector tube is placed into the deionized water reservoir and pump is started.
7. The control valve is adjusted to obtained a flowrate between 35 and 40 cm^3/min
8. The conductivity meter is observed. Rinsing is stopped when conductivity meter reading is close to conductivity reading of the deionized water.
9. The deionized water is flushed out by opening valves V4,V7,V8 and V14.

Experiment 1a: Removal of Nickel ion from the solution by manipulating Nickel(II) concentration

1. 1.8 g Nickel Sulfate is dissolved in distilled water to get the concentration of Nickel ion is 1.8g Ni/L. The initial conductivity meter reading of the test water is recorded.
2. The initial concentration of heavy metal in the test water is tasted by using Atomic Adsorption Spectrophotometer.
3. Valves V2,V11 and V13 is opened.
4. The selector tube is placed into the test water reservoir and pump is started.
5. The control valve is adjusted to obtained a flowrate between 35 and 40 cm^3/min
6. Sample is taken in an interval of 5 minutes.
7. The sample test water recovered from the column is tasted by using Spectrophotometer to study the heavy metal contains in the solution.
8. Steps 1 to 7 is repeated for Nickel(II) Sulfate concentration of 2.8 gNi/L and 3.8 gNi/L.

Experiment 1b: Effects of pH to the rate of Nickel ion removal from the wastewater.

1. 1.8 g Nickel Sulfate is dissolved in distilled water. The initial conductivity meter reading of the test water is recorded.
2. The initial concentration of heavy metal in the test water is tasted by using Spectrophotometer.
3. HCl is added to the wastewater to increase the pH of the solution.
4. Valves V2,V11 and V13 is opened.
5. The selector tube is placed into the test water reservoir and pump is started.
6. The control valve is adjusted to obtained a flowrate between 35 and 40 cm^3/min
7. The sample test water recovered from the column is tasted by using Spectrophotometer to study the heavy metal contains in the solution.
8. Steps 1 to 7 is repeated for different pH . The solution pH is adjusted with H_2SO_4 and NaOH.

APPENDIX C: RAW DATA

Breakthrough Time For initial Concentration 1.8 gNi/L

Initial concentration=Co= 25.9683 ppm

Time (min)	C (ppm)	C (mol/m^3)	C/Co
1	7.4656	0.1272	0.2875
2	6.5967	0.1124	0.2540
3	2.5552	0.0435	0.0984
4	0.1308	0.0022	0.0050
5	0.1461	0.0025	0.0056
6	0.2273	0.0039	0.0088
7	0.2427	0.0041	0.0093
8	0.2536	0.0043	0.0098
9	0.2690	0.0046	0.0104
10	0.2887	0.0049	0.0111
15	0.3941	0.0067	0.0152
20	0.6157	0.0105	0.0237
25	0.6705	0.0114	0.0258
30	0.6881	0.0117	0.0265
35	0.7407	0.0126	0.0285
40	0.7934	0.0135	0.0306
45	0.8153	0.0139	0.0314
50	0.8592	0.0146	0.0331
55	0.8811	0.0150	0.0339
60	0.8987	0.0153	0.0346
65	1.2607	0.0215	0.0485
70	1.3265	0.0226	0.0511
75	1.3989	0.0238	0.0539
80	1.4143	0.0241	0.0545
85	1.4253	0.0243	0.0549
90	1.7807	0.0303	0.0686
95	1.8443	0.0314	0.0710
100	2.1772	0.0371	0.0838
105	6.7301	0.1147	0.2592
110	9.6730	0.1648	0.3725
115	12.7928	0.2179	0.4926
120	15.5000	0.2641	0.5969
125	17.2000	0.2930	0.6623
130	20.0323	0.3413	0.7714
135	22.3963	0.3815	0.8624
140	23.0323	0.3924	0.8869
145	23.3963	0.3986	0.9010
150	24.0000	0.4089	0.9242
155	24.6000	0.4191	0.9473
160	25.6392	0.4368	0.9873

Breakthrough Time For initial Concentration 2.8 gNi/L

Initial Concentration: Co= 0.540886 mol/m^3

Time(min)	C(mol/m^3)	C/Co
1	0.000688	0.001272
2	0.000922	0.001704
3	0.001215	0.002246
4	0.001671	0.00309
8	0.001872	0.003461
11	0.002	0.003698
16	0.006494	0.012006
17	0.006945	0.012841
19	0.007714	0.014261
20	0.012673	0.02343
25	0.01302	0.024072
30	0.014051	0.025978
35	0.014542	0.026885
40	0.015617	0.028872
43	0.016942	0.031323
45	0.025348	0.046863
50	0.030651	0.056668
55	0.031065	0.057433
60	0.033061	0.061124
65	0.037194	0.068765
70	0.039261	0.072586
75	0.049593	0.091688
80	0.091986	0.170066
85	0.122533	0.226542
90	0.134036	0.247808
95	0.199608	0.369039
100	0.291491	0.538913
105	0.359956	0.665493
110	0.41406	0.765521
115	0.487034	0.900438
120	0.503428	0.930746
125	0.520579	0.962457
130	0.526433	0.973279
133	0.526675	0.973726
135	0.531186	0.982066
140	0.541036	1.000277

Breakthrough Time For initial Concentration 3.8 gNi/L

Co = 3.8 gNi/L = 33.57ppm=0.57191 mol/m^3

pH = 5.6

Time(min)	C(ppm)	C(mol/m^3)	C/Co
5	0.0531	0.0009	0.0016
10	0.5227	0.0089	0.0156
15	0.9763	0.0166	0.0291
20	1.0140	0.0173	0.0302
25	1.1085	0.0189	0.0330
30	1.5931	0.0271	0.0475
35	1.8372	0.0313	0.0547
40	2.3736	0.0404	0.0707
45	3.2985	0.0562	0.0983
50	5.9955	0.1021	0.1786
55	8.3894	0.1429	0.2499
60	10.7251	0.1827	0.3195
65	14.8841	0.2536	0.4434
70	19.9200	0.3394	0.5934
75	22.4116	0.3818	0.6676
80	29.2956	0.4991	0.8727
85	31.1770	0.5311	0.9287
90	32.5700	0.5549	0.9702
95	33.5690	0.5719	1.0000
100	33.5730	0.5719	1.0001

Breakthrough Time For pH 3

Initial Concentration=1.8 g Ni/L= 25.9683ppm = 0.44239 mol/m^3

Time(min)	C(ppm)	C(mol/m^3)	C/Co
1	1.074	0.018296	0.041358
2	1.0032	0.01709	0.038632
3	0.0045	7.67E-05	0.000173
4	0.00724	0.000123	0.000279
5	0.00993	0.000169	0.000382
6	0.01837	0.000313	0.000707
7	0.019117	0.000326	0.000736
8	0.2639	0.004496	0.010162
9	0.31973	0.005447	0.012312
10	0.3871	0.006595	0.014907
15	0.4971	0.008468	0.019143
20	0.5827	0.009927	0.022439
25	0.6431	0.010956	0.024765
30	0.7631	0.013	0.029386
35	0.8621	0.014687	0.033198
40	0.953	0.016235	0.036699
45	1.853	0.031567	0.071356
50	2.875302	0.048983	0.110724
55	4.9752	0.084756	0.191587
60	7.8852	0.13433	0.303647
65	16.542	0.281806	0.637007
70	22.84	0.389097	0.879534
75	24.88	0.42385	0.958091
80	25.73	0.43833	0.990823
85	25.89	0.441056	0.996985
90	25.97	0.442419	1.000065

Breakthrough Time For pH 7

Initial Concentration=1.8 g Ni/L= 0.5267 mol/m^3 = 30.919ppm

Time(min)	C(ppm)	C(mol/m^3)	C/Co
5	3.0213	0.05147	0.097722
10	2.0676	0.035223	0.066875
15	1.0769	0.018346	0.034832
20	0.1001	0.001705	0.003238
25	0.114	0.001942	0.003687
30	0.2784	0.004743	0.009005
35	0.3873	0.006598	0.012527
40	0.3896	0.006637	0.012601
45	0.4383	0.007467	0.014177
50	0.4846	0.008256	0.015674
55	0.5564	0.009479	0.017996
60	0.6051	0.010308	0.019572
65	0.7613	0.012969	0.024624
70	0.8506	0.014491	0.027512
75	0.9951	0.016952	0.032186
80	1.1054	0.018831	0.035753
85	1.1332	0.019305	0.036653
90	1.1974	0.020399	0.038729
95	1.2375	0.021082	0.040026
100	1.3788	0.023489	0.044596
105	1.4274	0.024317	0.046168
110	1.5317	0.026094	0.049542
115	1.7842	0.030395	0.057709
120	1.9625	0.033433	0.063476
125	2.1757	0.037065	0.070372
130	2.2336	0.038051	0.072244
135	2.5324	0.043141	0.081909
140	2.6204	0.044641	0.084755
145	2.6482	0.045114	0.085654
150	4.8504	0.08263	0.156883
155	5.1677	0.088036	0.167146
165	10.1115	0.172257	0.32705
175	13.9851	0.238247	0.452339
185	17.1693	0.292492	0.55533
195	20.8716	0.355564	0.675079
200	22.6347	0.3856	0.732105
205	26.3521	0.448928	0.852342

Effect of regeneration of resin

Co= 1.8 g Ni/L =25.9683ppm = 0.442390119 mol/m^3

pH= 5

Time (min)	C (ppm)	C (mol/m^3)	C/Co
1	2.8863	0.04917	0.111147
2	1.9973	0.034026	0.076913
3	1.8632	0.031741	0.071749
4	0.0635	0.001082	0.002445
5	0.0752	0.001281	0.002896
6	0.08652	0.001474	0.003332
7	0.10752	0.001832	0.00414
8	0.27386	0.004665	0.010546
9	0.269	0.004583	0.010359
10	0.2887	0.004918	0.011117
15	0.3941	0.006714	0.015176
20	0.6157	0.010489	0.02371
25	0.7439	0.012673	0.028646
30	0.8593	0.014639	0.03309
35	0.8714	0.014845	0.033556
40	0.8914	0.015186	0.034326
45	0.9063	0.01544	0.0349
50	0.9875	0.016823	0.038027
55	1.134	0.019319	0.043669
60	1.3265	0.022598	0.051082
65	1.414	0.024089	0.054451
70	1.4395	0.024523	0.055433
75	1.7649	0.030066	0.067964
80	1.8502	0.03152	0.071248
85	1.8993	0.032356	0.073139
90	2.0628	0.035141	0.079435
95	3.8512	0.065608	0.148304
100	5.1883	0.088387	0.199794
105	11.38	0.193867	0.438227
110	12.943	0.220494	0.498415
115	15.532	0.2646	0.598114
120	17.832	0.303782	0.686683
125	18.526	0.315605	0.713408
130	20.637	0.351567	0.7947