Research Article

Assessment of new cation exchange resin for nickel (II) ion continuous removal from aquatic environment using fixed bed technique

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1. Abstract

Nickel removal on ion exchange resin using fixed bed technique has proven to be very efficient. However, limited studies focused on continuous removal in fixed bed reactor. The breakthrough time of the resin still not well understood. New strong acid cation exchange resin was investigated experimentally in this study for theremoval of nickel ions from the aquatic environment using fixed bed technique. Significantly related attributes were studied, such as initial concentration of nickel ion, pH values, and regeneration. The nickel ion concentration in the aqueous environment was measured using atomic absorption spectrophotometer (AAS). The breakthrough curves demonstrates the results of the kinetic study of ion exchange process. Therefore, Thomas model was employed to examine the experimental results to show high correlations. However, the results of this study show that the optimum conditions of nickel (II) ion removal existed at pH (6–7) and the nickel (II) ion concentration of 1.8gNi/L. Additionally, the regeneration of the resin gives a significant improvement to the breakthrough time and exchange capacity. A comparison of the breakthrough time of the new material in this study to other resin material shows a significant difference of the exchange capacity of the new resin.

3. Introduction

Few studies focused the removal of hazardous ion metal from wastewater using fixed bed technique which can contribute towards industrial wastewater treatment. Many technologies used for nickel ion removal from wastewater; this includes adsorption [1,2] membrane [3], coagulation-flocculation [4], electrochemical methods [5]. However, each one of these possesses has their inherent advantages and limitations during application. Table 1 shows a comparison of chemical technologies used for heavy metals ion removal. The most efficient way to extract positively charged ions is ion exchange technique [6-11]. The ion-exchange technology gives large advantages over other methodsregarding recovery of metal value, selectivity, lesser sludge volume produced and meeting the requirements of stringent discharge specifications [12]. synthetic resins are remarkable against the materials used in ion exchange processes [13]. The pros of ion exchange such as the high capacity of the treatment, high efficiency of heavy metal removal and selectivity, and a wide range of operating temperatures. This study aims to investigate the enhancement of the removal of nickel ion from aquatic environment through using a new type of strong acid cation exchange resin and fixed bed technique. The objectives of this study are; to investigate the breakthrough time of the removal of nickel (II) ion from theaqueous environment at different conditions of concentration and pH, to study the effect of regeneration of resin on the exchange capacity, andexamine the kinetics of the ion exchange process. Cation exchange resin is typically functioning through sulfonic acid groups (-SO_3H) which are usually attached to positively charged sodium ions. Whenever the water containing dissolved metal ions specifically cations contacts the resin, the cations are transferred to the active places with sodium ions on the resin. These activated groups on the resin can also be carboxylic, phosphonic or phosphinic. The rules controlthe cation exchange process are; the high charge ions have a preference to be exchanged;ions of the small hydrated volume are preferred, and ions, which interact strongly with the functional groups of the exchangers are preferred [14].The most common cation exchangers are strongly acidic resins with sulfonic acid groups (-SO_3H) and weakly acid resins with carboxylic acid groups (-COOH) [15]. Hydrogen ions present in the sulfonic group and carboxylic group of

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the resin are exchangeable ions with metal cations. When the ion exchange resin exists in the aquatic environment, metal ions in the aquatic environment are exchanged with the hydrogen ions on the resin as shown in equations 1 and 2.

$$nR - SO3H + Mn+ \rightarrow (R - SO3-)nMn+ + nH+ \qquad (1)$$

$$nR - COOH + Mn+ \rightarrow (R - COO-)nMn+ + nH+ \qquad (2)$$
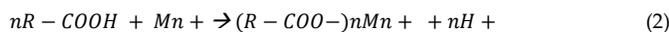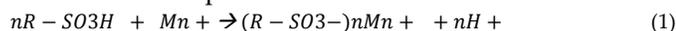
Among the materials that used in ion exchange processes, synthetic resins are commonly preferred as they are efficient and inexpensive. Moreover, cation exchange resins had been tested for their sorption capacity towards Nickel ion removal. Variables such as pH, temperature, the initial concentration of heavy metal and contact time are affecting on the removal of heavy metal ions by ion-exchange resins [16]. The solution pH level has a significant influence on the amount of ion exchanged. At low pH, the amount of hydrogen ion present in the mixture is higher. Therefore, the competition between nickel ion and hydrogen ion during ion exchange process in the aqueous solution becomes greater [17]. Furthermore, the removal of Ni(II) from wastewater produced by electroplating using strong acid resin (Purolite NRW-100) showed the good results and indicated that ion exchange technique is the efficient method to extract Ni(II) ion from wastewater aqueous solution [18]. The selectivity towards ions of the ion exchange resins increases with increasing valence or charge [19]. However, for ions with the same charge the affinities are raising with the increase of atomic number [20]. Ion exchange isotherm is a mathematical equation which is used to analyze the performance of cation exchange resin. However, Thomas model was used to examine the experimental results, as shown in equations 3-6. As a rule, the loading behavior of adsorbate to be removed from aqueous solution in a fixed-bed is expressed regarding C/C_o and can be described by the following expression:

$$\frac{C_t}{C_O} = \frac{1}{1 + \left[\left(\frac{K_{th}}{Q}\right)(q_{max}M - C_o V_{tot})\right]} \qquad (3)$$

Equation (3) is rewritten as the following linear equation:

$$\ln\left(\frac{C_o}{C_t} - 1\right) = \frac{K_{th}q_{max}M}{Q} - \frac{K_{th}C_o}{Q}V_{tot} \qquad (4)$$

$$\ln\left(\frac{C_o}{C_t} - 1\right) = \frac{K_{th}q_{max}M}{Q} - K_{th}C_o t = K_{th}C_o\left(\frac{q_{max}M}{C_o Q} - t\right) \qquad (5)$$

$$\ln\left(\frac{C_o}{C_t} - 1\right) = k\,(\tau - t) \qquad (6)$$

Where C_t, C_o are the effluent and inlet solute concentrations consequently; q_{max} is the maximum adsorption capacity; M is the total mass of the adsorbent; Q is the volumetric flow rate; V_{tot} the throughput volume; K_{th} is the Thomas rate constant, colume/mass time; t is the time in minutes and τ is characteristic time in minutes. In equation (5), at a constant flow rate, a plot of (ln ((C_o/

C_t)-1) verus time (t) was constructed. Then the kinetic parameter (k) and fixed-bed exchange q_{max} from the mathematical model can be determined from the obtained slope and intercept, and parameter t can be acquired at $C_t = C_o/2$. Then, the breakthrough curve will be obtained and be plotted (Shaidan, 2012). Furthermore, Thomas model can also be referred as bed-depth-service-time (BDST) model. This simplifies-design model excluded both intra-particles (solid). There have been different studies and investigations about removal of heavy metals by ion exchange resins. In the present study, Purolite NRW-100 with sulfonic acid ($-SO_3H$) group for removal of nickel (II) from aquatic environment was evaluated. In this study, batch studies showed that the exchange isotherms were well fitted with the reference equation. Columns experiments were also performed, however different values of pH, metal concentrations, volumetric flow rates and bed volumes were tested. This Thomas model which consists of two parameters was applied to describe the breakthrough curves. It had proved that the calculated breakthrough curve agreed well with the measured one (standard deviation 6%), except the curves before break point obtained at pH value more than 3.0 [18].

4. Experimental

4.1. Material and methods

4.1.1. Cation exchange resin

The removal of nickel ion from the aqueous solution will involve a new strong acid cation exchange resin with sulfonic acid groups ($-SO_3H$). (Figure 1) shows the resin beads of Lewatit C-249 that used in the experimental work. (Table 1) shows the chemical and physical properties of the resin.


Figure 1: Strong cation exchange resin (Lewatit C 249)

Table 1: The Chemical and Physical Properties of Resin (Lewatit C 249) [21]

| Properties	Value/Name
Functional Group	Sulphonic Acid
Structure	Gel Type
Appearance	Brown/Black Transluncent
Bead Size	0.4-1.25 mm
Effective Size	0.4
	± 0.006
Uniformity Coefficient	1.6 max
Bulk Weight	832 g/l
Density	1.26 approximately g/ml
Water Retention	45-48
Total Capacity	2 minimum eq/L

4.1.2. Pretreatment of Resin

The procedure of pretreatment was applied for the cationic resins before conducting the experiments. The resin was packed into the cation exchange column up to 30 cm height. Then, the resin was rinsed downward flow pattern by using deionized water excessively. For cationic resin, (Lewatit C-249), the hydrogen ion (H⁺) in the deionized water substitutes the sodium ion (Na⁺) on the resin and can be represented by the following equation:

$$H2O(aq) + Na.R(s) \longleftrightarrow H.R(s) + NaOH(aq) \qquad (7)$$
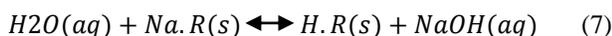
4.1.3. Regeneration of Resin

Resins have limited capacities and must be regenerated upon exhaustion. Therefore, the cationic resin was regenerated in the cation column using 5% sulphuric acid solution in a downward pattern. The flow rate of the regenerator was set at 30-35cm³/min and minimum contact time of 40 minutes. After regeneration, the resin was rinsed with deionized water for pretreatment of the resin until the conductivity of the effluent and the influent were almost similar.

$$H2SO4(aq) + Ni.R2(s) \leftrightarrow 2H.R(s) + NiSO4 \qquad (8)$$
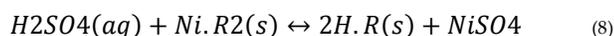
4.2. Equipment and Tools

The tool used in this study is bench-top ion exchange unit (SOLTEQ - Model: TR 02) as shown in (Figure 2). This unit comprises of two vertically mounted tubes which contain the respective cation and anion resins. The tubes are removable and interchangeable for softening, decationization and demineralization experiments. This equipment emulates the industrial operation of such unit, including monitoring breakthrough and regeneration cycles.

Figure 2: Ion exchange unit

4.3. Ion Exchange experiment

fixed-bed column which has the capability to work in a continuous operation mode was employed in this study. The ion exchange column of diameter 2 cm and height 60 cm respectively was employed in this research. The sample water solution was prepared by dissolving nickel sulfate (NiSO₄.6H₂O) in distilled water; S&M Chemical

Corporation supplied nickel sulfate. Tests will be carried out at 25°C and constant flow rate of 30cm³/min. There are three main parts of experimental work accomplished which are; the study of removal nickel (II) ion from wastewater with emphasis on controlling parameters such as initial concentration and pH values and to investigate the effect of ion exchange process on the resin material. In this research study, nickel sulfate was used to prepare the synthetic wastewater at different concentrations which are 1.8 g Ni/L, 2.8 g Ni/L and 3.8 g Ni/L based on typical metal plating industrial wastewater. Furthermore, pH values are also varied within the range (3-11) at a constant initial concentration of 2.8g Ni/L solution. The pH values are adjusted and controlled by using 5% sodium hydroxide (NaOH) and 97% sulphuric acid (H_2SO_4), sodium hydroxide and sulphuric acid (H2SO4) manufactured by Merck chemical was used. The effectiveness of regeneration of resin was further studied by using 2.8 g Ni/L concentration of the solution. 5% mass concentration of H_2SO_4 was flown downward through the resin bed until the conductivity of the effluent was similar to the conductivity of the influent. The sample of water effluents was collected at a specified interval and stored in the standard test tube. Then, they were further tested using pH meter and atomic absorption spectrophotometer (AAS) to identify the pH value and the concentration of nickel (II) ion respectively. The characterization of the resin is another important part of the experiment where Fourier Transform Infrared Spectroscopy (FTIR) and Variable Pressure Field Emission Scanning Electron Microscope (VPFESEM) were employed for characterization of the resins.

5. Results and discussion

5.1. Effect of initial concentration on breakthrough curves

The effect of initial concentration of solution on the ion exchange process was studied for 1.8, 2.8 and 3.8g Ni/L of solution via the breakthrough curve. The graph of a breakthrough fraction, C/C_o versus time treated at constant flow rate of 30cm³/min is plotted for every experiment. The 2 cm width column is filled with cation resins until it reaches 30 cm height. To neglect the effect of other parameters, the solution is prepared at constant pH of 5.6 to the column downwards. The green color of test water is fading when the concentration of Ni (II) decreases. From the results, the breakthrough curves become steeper when the initial concentration is getting higher. The sharper breakthrough curve at higher concentration shows a shortened mass transfer zone. The breakthrough curves at an initial concentration of 1.8g Ni/L, 2.8g Ni/L, and 3.8g Ni/L are shown in (Figure 3,4 and 5) respectively.

Results show that for the breakthrough curve, experimental data obtained fits well with calculated theoretical Thomas Model values. Breakthrough time, t_b in this study is defined as a period taken for the effluent from the outlet stream to reach 1 ppm of nickel ion concentration. Samples that were taken for every 5 minutes' interval were testing using Atomic Absorption Spectrophotometer

to verify the concentration of nickel ion in each particular time. (Table 2), shows the breakthrough time at different initial concentrations.
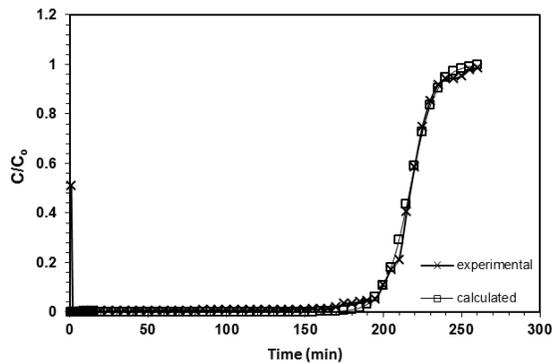

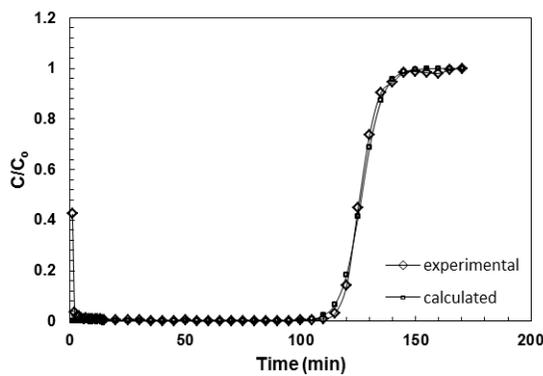**Figure 3**: Breakthrough curve of Ni (II) through time at 1.8 g Ni/

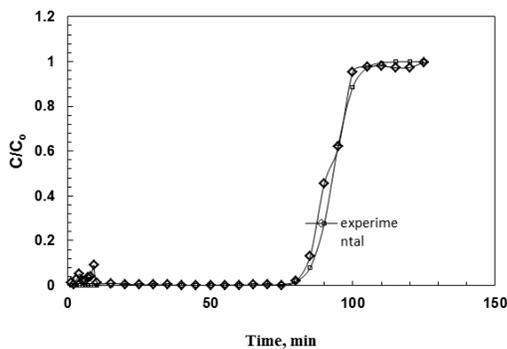**Figure 4**: Breakthrough curve of Ni (II) through time at 2.8 g Ni/L

Figure 5: Breakthrough curve of Ni (II) time at 3.8 g Ni/L

Table 2: Breakthrough Time at Different Initial Concentrations.

Initial Concentration of Nickel (g/l)	Breakthrough Time (min)
1.8	160
2.8	115
3.8	80

After a breakthrough time, the curve increases rapidly as the breakthrough curve, C/C_o is reaching 1. The resin was no longer used anymore after the breakthrough time surpassed. Regeneration of resin is required to extract nickel ion that is fully loaded into the resin and put the resin into service cycle again. At a higher initial concentration of nickel ion, the time taken for the breakthrough point is increasing at the selected breakpoint concentration of 1 ppm of effluent. (Table 3) shows the Thomas parameters obtained

by calculating values from the graph in (Figure 6) at different initial concentration.

Breakthrough time differs because for constant flow rate, and quantity of resin, the exchange sites of the resin is getting exhausted earlier when the initial concentration increases [17]. Higher concentration means that the amount of nickel ion in the solution is much greater, so ion exchange rate between nickel ion from the solution and sodium ion at the resin will increase. Therefore, time taken to reach breakthrough point becomes shorter.

The green color of nickel (II) sulfate gives a significant signal before and after treatment. Wastewater effluent that contains nickel is typically green and it will turn into colorless after it flows through the column.

Table 3: Exchange Capacity of Resin and Thomas Constant for Different Initial Concentration.

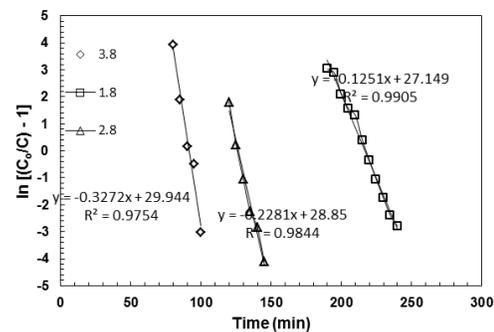| Initial Concentration of Solution (g Ni/L)	Exchange Capacity, q_{max} (mol/kg)	Thomas Constant, K_{Th}
1.8	0.8354	0.0109
2.8	0.4138	0.0234
3.8	0.3608	0.026

Figure 6: Thomas parameters and exchange capacity for various initial concentration.

5.2. Effect of pH on breakthrough curves

The effect of pH on nickel ion removal from solution was conducted at constant flow rate of 30 cm³/min in a column of diameter 2 cm, resin height of 10 cm while the initial concentration of nickel ion in the aqueous solution is maintained at 2.8 g Ni/L. Sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) were used for pH adjustment. The range of pH varied at different values of 3, 5, 7, 9 and 11. (Figure 7, 8 and 9) show the breakthrough curve at pH 3, pH 5 and pH 7 respectively. However, these experimental breakthrough curves demonstrate a good correlation with Thomas model.

In the experiments at constant pH values of 9 and 11, there is a critical phenomenon of chemical precipitation observed. Precipitation took place at the top of the column which resulted in blockage of

the flow of solution downward. Therefore, the precipitation could lead to a deterioration of the adsorption capacity. (Figure 10and 11) demonstrate unacceptable results for pH 9 and pH 11 respectively because, at pH higher than 7, precipitation starts to occur. Figure 4 shows the precipitation that occurred in the column. Kumar (2010) mentioned briefly that at pH values higher than 6, the precipitation is dominant or both ion exchange and aqueous metal hydroxide formation may become important mechanisms in the metal removal process. This condition is often not desirable as the metal precipitation could lead to a misunderstanding for the adsorption capacity. However, in this study the researcher explains this phenomenon of precipitationabout the chemical behavior of the system. When the sodium hydroxide concentration raises up with the aim to increase the pH level which results in an increase of sodium ion concentration in the aqueous solution. The equilibrium state of hydrogen ion on the resin will be disturbed according to the following reaction.

$$H2O(aq) + Na.R(s) \leftrightarrow H.R(s) + NaOH(aq) \quad (9)$$
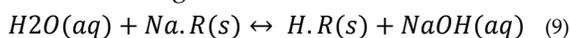
Due to the increase of sodium ion concentration, the reaction works in the backward direction which resulted in the liberation of the hydrogen ion in the solution. The liberated hydrogen ion reacts with the excess sodium hydroxide to form a precipitate.

(Table 4) shows the breakthrough time at different pH of the solution. (Figure 12) illustrates the graph used to determine Thomas parameters and exchange capacity for various pH. The value of exchange capacity of resin, t_o and Thomas constant rate was determined from the graph of $\ln[(C_o/C) - 1]$ versus time for different pH as shown in (Table5).

Results show that exchange capacity decreases from 0.5788 to 0.3570 when the value of the pH solution is ascending from 3 to 7. Besides, hydrogen ion(H^+) is released from the active sites, and adsorbed amount of nickel (II) ions is found to increase at the moderate pH from 3 until6.

Table 4: Breakthrough Time for Different pH.

pH of solution	Breakthrough Time (min)
3	20
5	25
7	40

Table 5: Exchange Capacity of Resin and Thomas Constant for Different pH

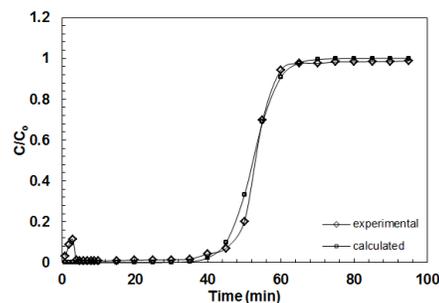| pH of solution	Exchange capacity, q_{max} (mol/kg)	Thomas constant, K_{th}
3	0.5788	0.02589
5	0.4591	0.01902
7	0.357	0.01116

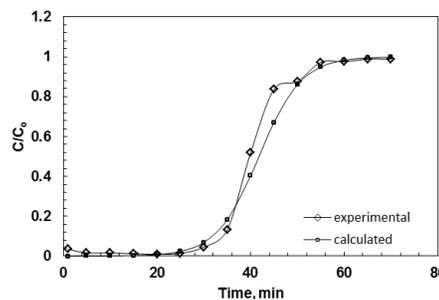**Figure 7**: Breakthroughcurve of Ni (II) Ion through time at pH 3.

Figure 8: Breakthroughcurve of Ni (II) ion through time at pH 5.

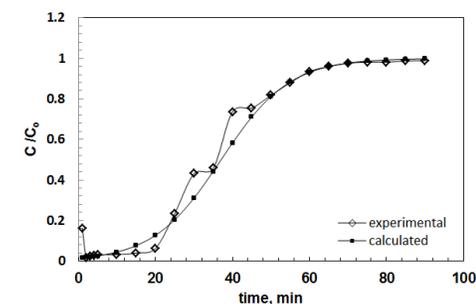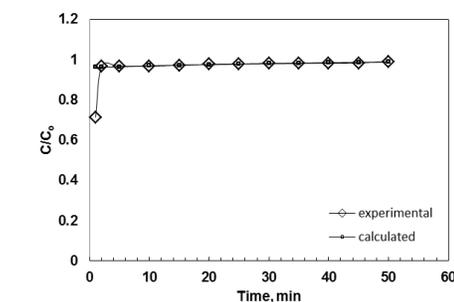**Figure 9**: Breakthroughcurve of Ni (II) ion through time at pH 7.

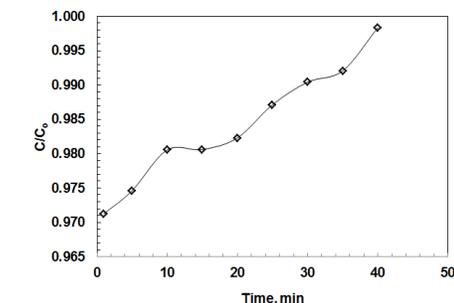**Figure 10**: Breakthroughcurve of Ni (II) ion through time at pH 9.

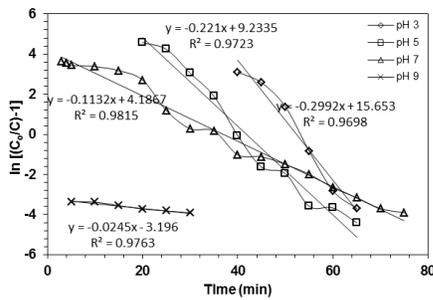**Figure 11**: Breakthroughcurve of Ni (II) ion through time at pH 11.

Figure 12: Thomas Parameters and Exchange Capacity for Different pH.

5.3. Effect of regeneration on breakthrough curves

Regeneration of exhausted resin after treatment with nickel (II) ion solution is another essential element for removal of nickel ion from wastewater. For this experiment, the regeneration of Lewatit C 249 resin is conducted by using 5% H_2SO_4. Regeneration of resin is an economical way to reduce the cost of operation because the resin can be used repeatedly. H+ in the sulphuric acid will replace nickel (II) ion in the resins when sulphuric acid flows downward through the column because of ion selectivity order.

The color of preliminary downstream effluent is typically green which indicates the presence of nickel (II) ion. Therefore, when sulphuric acid keeps flowing through the column, the downstream effluent will change from green to colorless because there is no more nickel (II) ion in the resin. To determine the effect of regeneration, the regenerated resin is then treated again using NiSO$_4$.6H$_2$O with a concentration of 2.8g Ni/L.

(Figure 13) shows that ion exchange performance through regenerated resin fits well with analytical data. Meanwhile, (Figure 14) illustrates the comparison of the breakthrough curve through parent resin and regenerated resin.

From the previous experiment, the breakthrough time for parent resin using concentration of 2.8g Ni/L solution is 115 min. However, for regenerated resin, the selected breakthrough point at a concentration of 1 ppm is not applicable since the concentration of downstream effluent is already more than 1 ppm from beginning. Based on (Figure 15), it shows that the efficiency of the regenerated resin in removing Ni^{2+} ion from solution before the breakthrough point is lower than the virgin resin due to the presence of Ni^{2+} at the regenerated resin.

However, results show that the exhausted time of the regenerated resin had delayed much more ahead from the virgin resin. This situation can be related to the fact that the recurred ion exchange between ion from resin and ion from the solution after several treatments might increase the surface area of the resin. In this instance, solute transport is also delayed due to the adsorption process. So, it makes the breakthrough time of regenerated resin longer than the virgin resin.

Calculated Thomas parameters based on Thomas Model after first regeneration are shown in (Table 6). Thomas constant rate of virgin resin differs to the regenerated resin with only 4.2 %. This result proves that regenerated resin is potentially recycled back into operation without affecting too much exchange capacity of the resin. Based on research accomplished by R. Ansari and F. Raofie, they found out that regenerated resins can effectively adsorb ions without losing the capacity, and the loss in capacity was 18%. So, regeneration of resins leads to an economical way of overall operation cost saving.
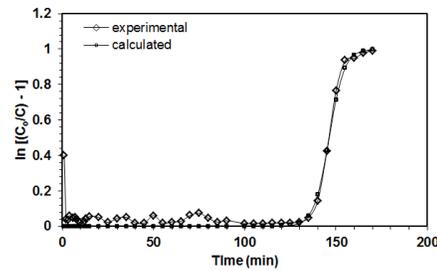

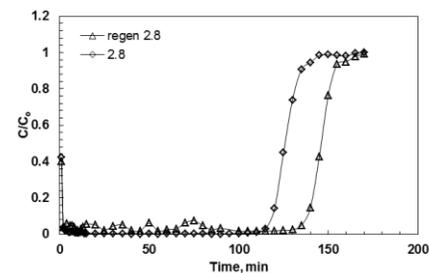**Figure 13**: Breakthrough curve of Ni (II) through regenerated resin.

Figure 14: Comparison of break through curve of Ni (II) Ion through parent resin and regenerated resin.
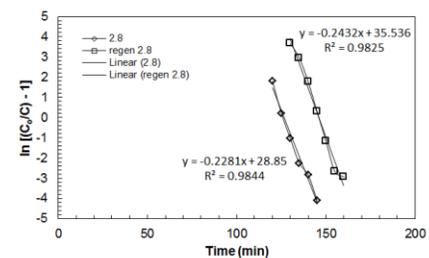

Figure 15: Thomas parameters and exchange capacity for virgin and regenerated resin.

Table 6: Thomas Parameters of Ion Exchange for Virgin and Regenerated Resin.

pH of solution	Exchange Capacity, q_{max} (mol/kg)	Thomas Constant, K_{th}
Virgin resin	0.4138	0.02438
Regenerated resin	0.4892	0.0234

5.4. Comparison to previous research work

A comparison of the breakthrough time at different initial concentration and different resin type is shown in (Table 7). The breakthrough time of the new resin of Lewatit C-249 is much higher compared to Lewatit S-1467 the same conditions of initial concentration. Which can offer a huge advantage for the real application of nickel ion removal.

Table 7: Comparison of breakthrough time and resin type.

Type of resin	Breakthrough Time (min)			Exchange capacity, q_{max} (mol/kg)			Reference
Initial concentration	1.8	2.8	3.8	1.8	2.8	3.8	
Lewatit C-249	160	115	80	0.8354	0.4138	0.3608	This study
Lewatit S-1467	60	43	15	0.045	0.046	0.034	[17]

6. Conclusion

Experimental work was conducted using a strong acid cation exchange resin and fixed bed column. Results showed that initial concentration of the solution is a significant parameter to be considered when removing nickel ion from aqueous solutions. The initial concentration affects on the breakthrough time, exchange capacity as well as Thomas constant rate. At increasing initial concentration of nickel (II) at values of 1.8g Ni/L, 2.8 and 3.8g Ni/L, the breakthrough time decreased from 160 to 80 minutes. Subsequently, the exchange capacity of the resin declined from 0.8354 mol/kg to 0.3608 mol/kg. Therefore, the lower the initial concentration, the lesser the cycles of regeneration process since the breakthrough time is longer. However the cost of operation can be reduced as well. The results showed that at different pH values, results show that the optimum pH parameter is within the range of 6 to **7.** For pH value above 7, chemical precipitation occurs and effects on the metal ion removal from wastewater. So, this condition of pH above 7 is recommended not to be considered no more. At pH below than 3, excessive H^+ ion in the solution makes the removal process difficult due to great competition between hydrogen and nickel (II) ion. However, from the results of this study, the optimum conditions of nickel (II) ion removal are existed at pH from 6 to 7 and the initial concentration of 1.8g Ni/L. Additionally, the regeneration of the resin gives a significant improvement to the breakthrough time and exchange capacity. Furthermore, The experimental results fit well with Thomas model data. The analysis of effluent samples using AAS showed that the concentration of the nickel ion after treatment is within the standard allowable limit of the Malaysian environmental regulations.

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