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Removal of nickel from water using rotating packed bed contactor: Parametric studies and mode of operations

Yee-Sern Ng^{1,*}, Yee-Tong Tan², Adeline Seak May Chua², Mohd Ali Hashim², Bhaskar Sen Gupta³

¹Department of Chemical Engineering, Lee Kong Chian Faculty of Engineering and Science, Universiti Tunku Abdul Rahman, Jalan Sungai Long, Bandar Sungai Long, 43000 Kajang, Selangor, Malaysia

²Department of Chemical Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia

³Water Academy, Institute of Infrastructure and Environment, Heriot-Watt University, Edinburgh Campus, EH14 4AS Scotland, UK

* Corresponding author: ngys@utar.edu.my

Abstract

Performance of rotating packed bed (RPB) contactor in the removal of nickel from water was investigated from two aspects: i) effect of operating parameters, and ii) effect of mode of operation. The results showed that the performance of RPB was highly governed by the rotational speed and activated carbon dosage. The increase in centrifugal force generated by the RPB from 200 rpm – 875 rpm was found to displace the activated carbon to the outer side of the RPB, which reduced the liquid flow path length and contact time between the packed bed and Ni solution and this reduced the removal efficiency. Nevertheless, a further increase in rotational speed from 875 rpm to 1200 rpm improved the removal efficiency as the enhancement in mass transfer process had outperformed the effect given by reduced path length. Higher activated carbon dosage also improved RPB performance by providing higher number of adsorption sites, as well as improving the packing density of the packed bed which minimized the reduced path length effect. The study also revealed that the mode of operation for a RPB contactor has crucial impact on the nickel adsorption. The removal efficiency was found to follow the sequence of multiple cycle mode operation > batch mode operation > continuous mode operation, as a result of better contact time and consistent contact between the solution and the packed bed.

Keywords: *nickel, rotating packed bed, operating parameters, mode of operation, rotating speed*

1. Introduction

Nickel is one of the hazardous heavy metals which can enter the environment through two ways: i) natural resources such as weathering of minerals and rocks, and ii) geothermal emissions and anthropogenic activities. Nickel can be released into the water bodies via effluent from various activities such as nickel-electroplating industry, battery industry, mining industry and refineries(1–3). The presence of low amount of nickel in natural water can cause high toxicity to human (4) and lead to chronic disorder such as gastrointestinal distress, skin dermatitis, renal edema, pulmonary fibrosis and lung and kidney damage (5). According to World Health Organization (6), the tolerance limit for nickel is 0.02 mg/L. Therefore, a proper treatment method for removing nickel from water is necessary before the industrial effluent is discharged to the environment.

As a divalent heavy metal, nickel can be removed from water/wastewater through a number of techniques such as solvent extraction (7), chemical precipitation(8), membrane separation (9,10), electrolysis (11), and etc. However, it is worth noting that these removal methods are normally suffer from drawbacks such as high operational cost, high energy consumption, membrane fouling issue and the disposal of toxic metal sludge (12,13). In contrast, adsorption, which is a process that an adsorbate is attracted and retained on the surface of an adsorbent, is a preferred method due to its simplicity, reduced operational cost and minimum waste disposal (2). Adsorption process is one of the most effective methods for the removal of heavy metals and organic compounds in water/wastewater treatment. However, as the volume of water to be treated in a treatment plant is generally large, huge number of activated carbon is often required (14), which serves as the main drawback for adsorption process. Therefore, another technique that can provide good adsorption for high volume of effluent at smaller size is to be investigated.

It is reported that enhanced mass transfer can be achieved with the aid of a centrifugal force field through high gravimetric technology (HIGEE). The mass transfer process is carried out in a doughnut shaped bed, as known as rotating packed bed (RPB). The RPB, designed by Ramshaw and Mallinson (15), is rotated at high speed to achieve high centrifugal force that is several times higher in value than the gravitational force. Due to the elevated force, very tiny droplets and/or thin liquid films are created in the packing. Therefore, the gas-liquid or liquid-liquid interfaces are renewed constantly, and this provides considerable intensification of mass transfer between the phases through mixing (16). Several advantages are reported for this system, such as higher throughput of thinner liquid film over the packing, which in turn results in improved external mass transfer, lower static holdup and better distribution of liquid over the packing (14). These enhancements aid in reducing the size of the processing equipment significantly in comparison to a conventional packed bed. Therefore, it is likely to reduce the overall cost of the process (15,17). The use of RPB contactor has been reported to give promising results for liquid-gas processes such as CO₂ capture (18–20), H₂S absorption (21), volatile organic compounds removal from air stream (22) and degradation of nitrobenzene (23).

Several studies also reported that the application of RPB in water/wastewater treatment is feasible for the removal of ammonium (24), phenol (25), dyes (26,27), pesticides (28) and heavy metals (14,29). Nevertheless, it is worth noting that the investigation on the application of this process in liquid-solid system is still relatively scarce. The objective of this study was to investigate the performance of RPB contactor in the removal of nickel from water using commercial activated carbon. The effect of different operating parameters, such as initial nickel concentration, feed rate, rotating speed, and activated carbon dosage on the adsorption of nickel in RPB contactor was evaluated. In addition, the effect of three modes of operation for RPB contactor on nickel adsorption, namely batch mode, continuous mode and multiple cycle mode was also investigated in this study.

2. Methodology

2.1 Materials

Commercial granular coconut shell activated carbon pellets of size 0.5 cm were used as the adsorbent in this study. Nickel nitrate hexahydrate, nitric acid and sodium hydroxide were of analytical grades and were supplied by R&M Chemicals. The nickel solution was prepared by dissolving nickel nitrate hexahydrate into deionized water. Depending on the experiments, the pH of the solution was adjusted using nitric acid or sodium hydroxide.

2.2 Rotating packed bed contactor

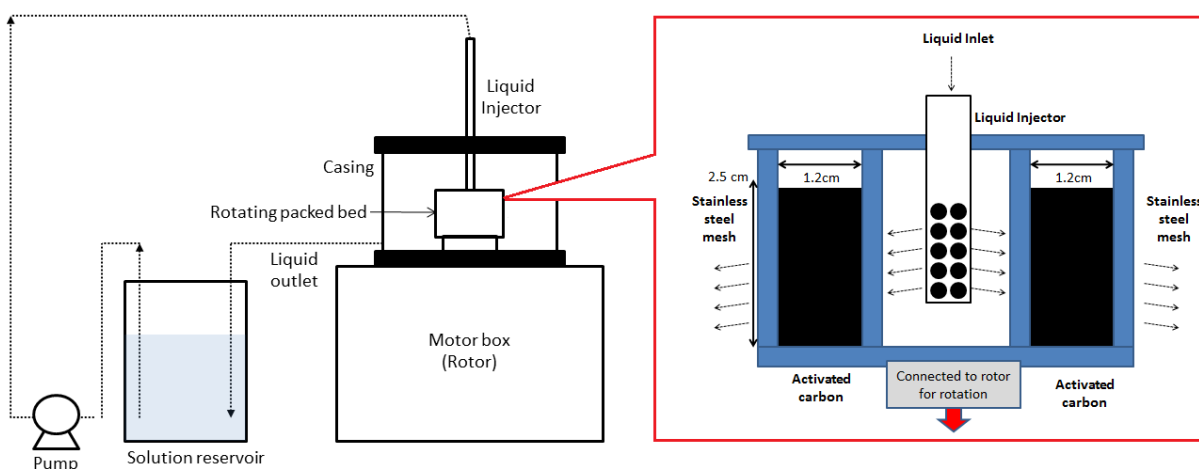


Figure 1: Schematic diagram of a rotating packed bed contactor set up

Figure 1 shows the setup of the RPB contactor. The doughnut shaped packed bed reactor is connected to a 0.5 hp motor for the rotating process/gravitational force generation on the basis of the arithmetic mean radius. The height of the reactor bed is 2.5 cm with an annulus space of 1.2 cm wide for the placement of the activated carbon, as shown in Figure 1. The sides of the inner and outer walls are made of 50 µm opening stainless steel mesh for nickel solution to flow out from the contactor while retaining the activated carbon bed.

2.3 Experimental Procedures

2.3.1 Effect of operating parameters

A mass of activated carbon was weighed and was transferred into the RPB contactor. The contactor was then connected to the motor box, as shown in Figure 1. The motor was switched on for the rotation of the contactor. Then, 1 L of nickel solution was placed in a reservoir and the solution was transferred into the contactor via the liquid injector through a peristaltic pump at specific feed rate. The solution was passing through the RPB and was recycled back to the reservoir. This operation was conducted for one hour. After the experiment, a volume of 1 mL aqueous sample was taken from the reservoir and the nickel concentration was analyzed.

The effect of operating parameters was investigated via two sections. Firstly, the effect of solution pH was evaluated at a range of 2 to 12 while the other operating parameters were held constant at level 0, as shown in Table 1. The solution pH that provided the best nickel adsorption was further applied in the second section, which was to evaluate the performance of RPB at different operating parameters, namely :i) initial concentration, ii) feed rate, iii) rotating speed and iv) activated carbon dosage. This was evaluated using response surface methodology – Box Behnken Design.

Table 1: Parameters for rotating packed bed contactor that are investigated

Parameters	Level -1	Level 0	Level +1
Initial concentration, mg/L	50	100	150
Feed rate, L/h	20	35	50
Rotational speed, rpm	200	700	1200
Activated carbon dosage, g	3	8	13

Response Surface Methodology (RSM) is a statistical technique for the analysis and modeling of problems in which the response/outcome is influenced by several variables (30). RSM requires less number of experiments in comparison to factorial experiment design and the Analysis of Variance (ANOVA) can provide an approximation for the true functional relationship between the independent variables and the response in the form of polynomial equation, as shown in Equation (1) where y is the response, β is

the regression coefficient and x is the independent parameter (30). Based on the ranges and levels of the parameters in Table 1, 29 experiments were suggested for Box-Behnken Design. The experimental sequence and the results are as shown in Table 2.

$$y = \beta_0 + \sum_{i=1}^3 \beta_i x_i + \sum_{i=1}^3 \beta_{ii} x_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^3 \beta_{ij} x_i x_j \quad (1)$$

Table 2: Experimental sequence for Box-Behnken Design

Run	Initial concentration, mg/L	Feed rate, L/h	Rotating speed, rpm	Activated carbon dosage, g	Removal efficiency, %
1	150	35	1200	8	12.62
2	100	50	200	8	27.44
3	150	35	700	3	8.13
4	100	50	1200	8	19.33
5	50	35	1200	8	33.16
6	100	35	200	3	10.96
7	50	35	700	13	41.48
8	50	35	200	8	48.50
9	150	35	700	13	17.98
10	50	35	700	3	8.82
11	100	35	700	8	13.94
12	100	20	200	8	28.96
13	100	35	1200	3	8.51
14	50	20	700	8	22.86
15	100	50	700	3	10.45
16	100	35	200	13	48.19
17	100	35	700	8	13.70
18	100	35	700	8	17.34
19	100	35	700	8	13.42
20	150	50	700	8	13.75
21	100	50	700	13	26.22
22	150	35	200	8	20.22
23	100	20	700	13	15.62
24	100	20	700	3	7.63
25	100	35	1200	13	24.61
26	100	35	700	8	18.09
27	100	20	1200	8	15.16
28	50	50	700	8	27.52
29	150	20	700	8	7.34

2.3.2 Effect of mode of operation

The effect of mode of operation on the removal of nickel from water using RPB was carried out using the optimum parameters that were obtained from the study in Section 2.3.1. Three operation modes were investigated, namely i) batch mode, ii) continuous mode, and iii) multiple cycle mode. The procedure for batch mode operation was elaborated in Section 2.3.1.

For continuous mode operation, the activated carbon dosage was weighed and was transferred into the RPB contactor. The reactor was then connected to the motor box and was rotated. Then, 1 L of nickel solution was transferred from the reservoir into the contactor via the liquid injector through a peristaltic pump. The solution was passed through the RPB and was collected in another beaker instead of recycling back to the reservoir. The experiment ended when all the solution in the reservoir had passed through the RPB. A volume of 1 mL aqueous sample was taken and the nickel concentration was analyzed.

For multiple cycle mode operation, the nickel solution was passed through the RPB and was collected in another beaker until all the solution in the reservoir had passed through the RPB. Then, 1 mL of aqueous sample was taken. After that, the solution was directed into the RPB again for another cycle of adsorption operation. This procedure was repeated for five cycles.

2.4 Analytical method

The aqueous samples which were taken after the experiment were diluted and acidified with diluted HNO_3 solution. The concentrations of nickel in the samples were then analyzed by Inductive Coupled Plasma – Optical Emission Spectrometry (ICP-OES). The measurement was conducted triplicate and the mean was recorded. From the nickel concentrations obtained from the aqueous samples, the removal efficiency can be calculated using Equation (2), where C_i is the initial Ni concentration in the aqueous sample (mg/L), C_f is the final Ni concentration in the aqueous sample (mg/L)

$$\text{Removal efficiency, \%} = \frac{C_i - C_f}{C_i} \times 100 \quad (2)$$

3. Results and Discussion

3.1 Effect of solution pH

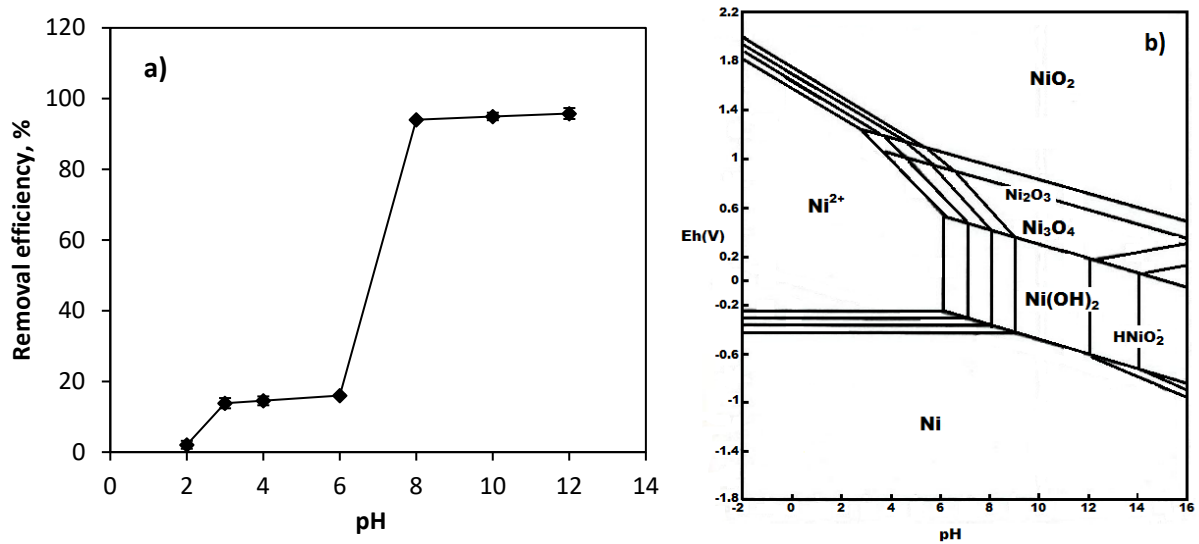


Figure2: a) Effect of pH on the removal of nickel from water using RPB contactor; b) Eh-pH diagram for nickel-water system at 25°C (31)

The investigation on the effect of solution pH on nickel adsorption using RPB was conducted at level – 0 operating conditions (Table 1) using batch mode operation. The pH range investigated was set from acidic condition (2-6) to alkaline condition (8-12). The results as shown in Figure 2a illustrate that the increase in solution pH from 2 to 4 increases the removal efficiency from 2% to 14%. Nevertheless, it is worth noting that further increase of solution pH from 4 to 6 only increases the removal efficiency slightly to 16%. This observation can be explained by the Eh-pH diagram for nickel, as shown in Figure 2b (31) where nickel is normally present in divalent cationic form at low pH condition (pH: 2-6). The cationic nickel is favorable to adsorb on the negative charge activated carbon. However, the adsorption was found poor at highly acidic condition (pH: 2), mainly due to the competitive adsorption between hydrogen ions and

nickel ions, as well as the protonation of the activated carbon surface which hinders the adsorption of cations (32,33). These mechanisms were mitigated at a higher pH condition of 4-6 as a result of low competition by the hydrogen ions on the adsorption, thereby the adsorption of nickel was improved. In contrast, a further increase in solution pH to alkaline condition (pH: 8-12) is found to achieve nearly 100% removal efficiency. Nevertheless, it is worth noting the main removal mechanism at these pHs is precipitation of water insoluble nickel hydroxide instead of adsorption (32,33). This is well supported by the Eh-pH diagram in Figure 2b that nickel is mainly in the form of water insoluble compounds at high pH condition (31). Therefore, it can be concluded that the nickel adsorption was best at the pH range of 4-6. The subsequent study was investigated at solution pH of 4.5-5.5.

3.2 Effect of operating parameters on the performance of rotating packed bed contactor

Four operating parameters were investigated using the Response Surface Methodology – Box Behnken Design, which were: i) initial concentration, ii) feed rate of the solution into the RPB, iii) rotating speed of the RPB, and iv) activated carbon dosage. The experimental results were analyzed statistically using ANOVA and the results were evaluated based on the mathematical model generated from RSM.

3.2.1 Analysis of Variance (ANOVA)

The ANOVA for the experimental results is as shown in Table 3. The model has an F-value of 28.85 with Probability > F of less than 0.0001. This indicates that the mathematical model generated has significant confidence level of > 99% as there is only 0.01% chance that the F-value this large could occur due to noise. The lack of fit for this model is not significant as there is 12.23% chance that the F-value this large could occur due to noise, which further indicates that the experimental data fits the model well. Among the parameters, initial concentration (A), rotating speed (C) and activated carbon dosage (D) show relatively higher effect in comparison to feed rate (B) as they have lower Probability > F value (<0.0001). Table 3

also shows that the goodness-of-fit of the data is justified via a close predicted R^2 and adjusted R^2 with a difference of less than 0.2. Lastly, an adequate precision of 19.734 for this model further confirms the adequacy of the model. The validity of the model is further strengthened by linear normal probability plot and close predicted results and actual results, as shown in Figures 3a and 3b, respectively. Thus, the mathematical model generated is valid and is suitable to be used for navigation on the effect of operating parameters on the removal efficiency. The mathematical model is as shown in Equation (3), where A, B, C and D are the coded factors for initial concentration, feed rate, rotating speed and activated carbon dosage, respectively, with the level range of -1 to 1.

$$\begin{aligned} \text{Removal efficiency, \%} = & 14.74 - 8.45A + 2.26B - 5.91C + 9.90D + 4.32A^2 \\ & + 8.63C^2 - 5.91AD - 5.28CD \end{aligned} \quad (3)$$

Table 3: ANOVA for the experimental results

Source	Sum of Squares	DF	Mean Square	F Value	Prob> F	
Model	3347.35	8	418.42	28.85	< 0.0001	significant
A	857.83	1	857.83	59.14	< 0.0001	
B	61.38	1	61.38	4.23	0.0529	
C	418.46	1	418.46	28.85	< 0.0001	
D	1175.33	1	1175.33	81.03	< 0.0001	
A ²	128.85	1	128.85	8.88	0.0074	
C ²	513.55	1	513.55	35.4	< 0.0001	
AD	139.95	1	139.95	9.65	0.0056	
CD	111.64	1	111.64	7.70	0.0117	
Residual	290.08	20	14.50			
Lack of Fit	270.23	16	16.89	3.40	0.1223	not significant
Pure Error	19.85	4	4.96			
Cor Total	3637.43	28				
Std. Dev.		3.81		R-Squared	0.9203	
Mean		20.10		Adj R-Squared	0.8884	
C.V.		18.95		Pred R-Squared	0.7995	
PRESS		729.23		Adeq Precision	19.734	

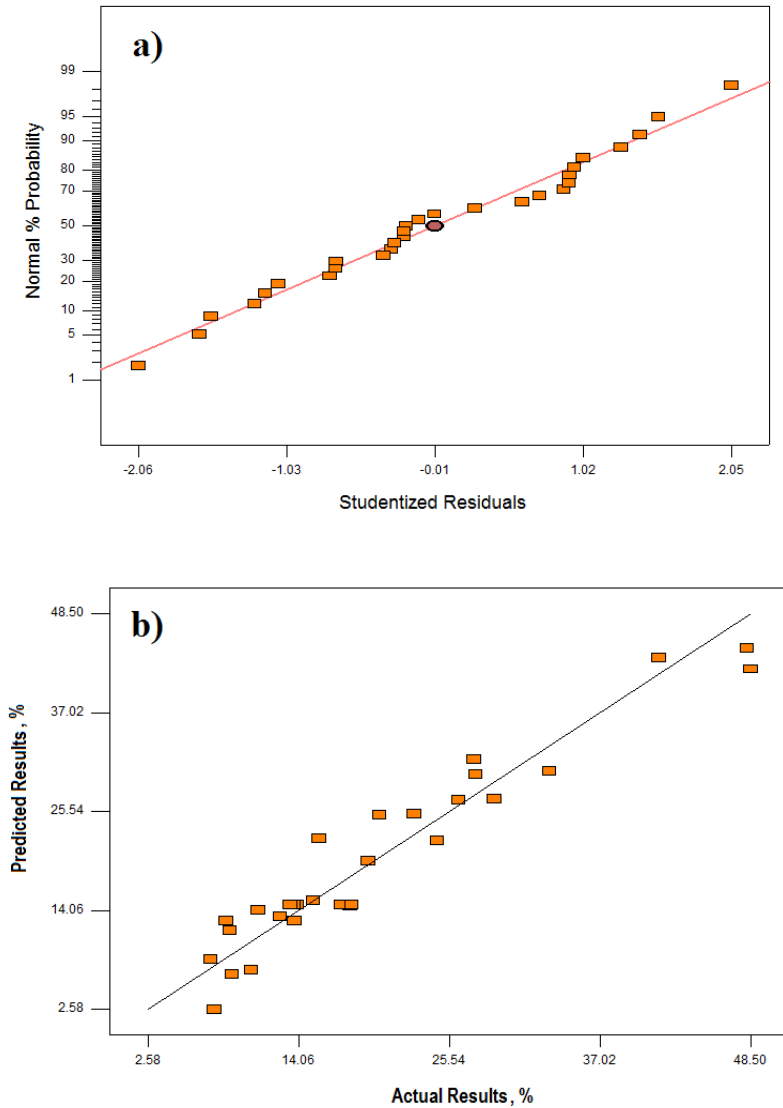


Figure3: a) Normal probability plot for the mathematical model generated, and b) Predicted results versus actual results

3.2.2 Effect of operating parameters on the removal efficiency

Figure 4a illustrates the effect of initial concentration on nickel removal efficiency. The results show that the increase in initial concentration from 50 mg/L to 150 mg/L reduces the removal efficiency from 27.51% to 10.61%. This was mainly due to higher competition between the amount of nickel to be adsorbed on limited adsorption sites at higher initial concentration (25). The percentage of nickel that can be adsorbed was reduced and this reduced the removal efficiency. Nevertheless, it was worth noting that the nickel

uptake by the activated carbon in RPB was increased from 1719.38 mg/kg to 1989.38 mg/kg when the initial concentration was increased from 50 mg/L to 150 mg/L, as more nickel was readily adsorbed.

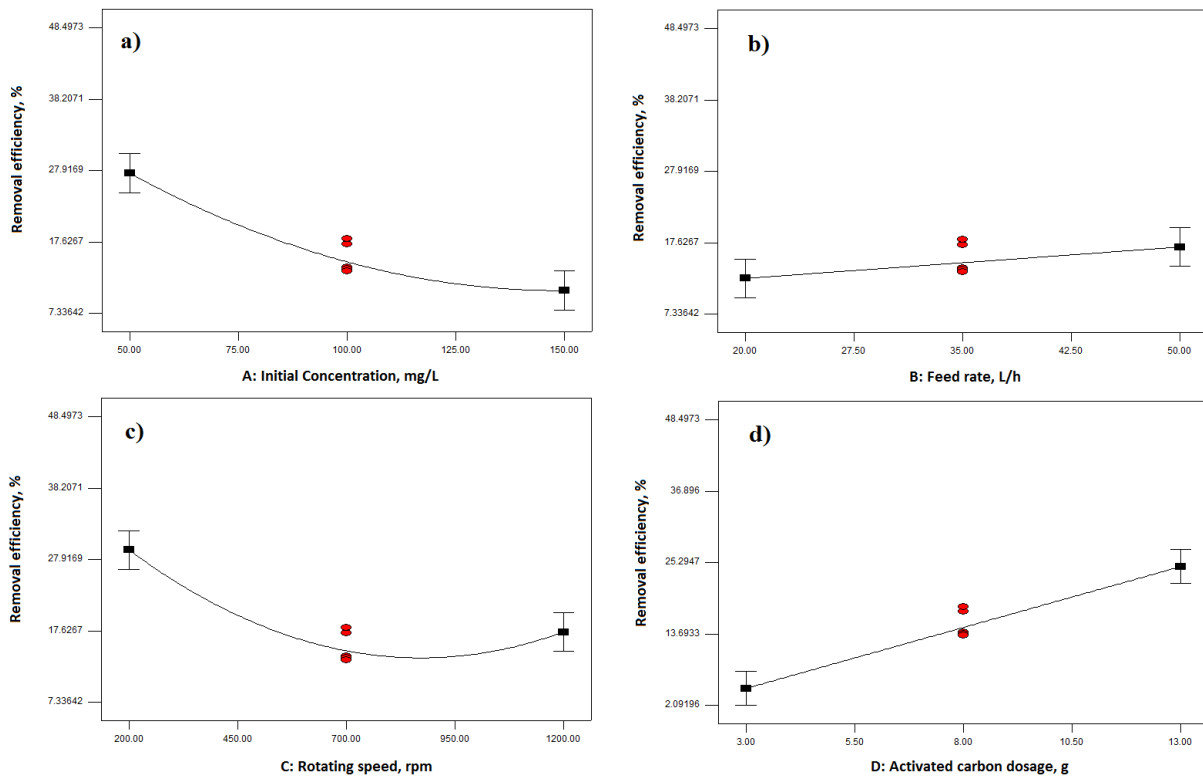


Figure 4: Effect of operating parameters on the removal efficiency: a) Initial concentration; b) Feed rate; c) Rotating speed; d) Activated carbon dosage

In contrast, the increase in feed rate from 20 L/h to 50 L/h is found to slightly improve the removal efficiency from 12.48 % to 17.00 %, as shown in Figure 4b. This observation was in line with the works of Chang and Lee(28) and Kundu et al. (14), which also operated the RPB at batch mode operation. As the system was operated with flow circulation, the increase in feed rate will improve the total contact time and mixing between the nickel solution and the activated carbon, which enhanced the rate of effective collision for better adsorption efficiency (14,28). Nevertheless, it is worth noting that the effect of feed rate was relatively lower in comparison to other parameters, as shown in Figure 4.

The use of centrifugal force in an RPB is often reported to improve mass transfer diffusion rate by forming tiny liquid droplets which not only decreases the diffusion distance between the packed bed and

the solution (24), but also enlarges the liquid-solid area for enhancing effective intraparticle diffusion and wetting efficiency of the system (25,28). However, different trend was observed in this study. Figure 4c shows that the increase of rotating speed from 200 rpm to 875 rpm reduces the removal efficiency from 29.27% to 13.72%. Then, a further increase from 875 rpm to 1200 rpm improves the removal from 13.72% to 17.46%. This could be due to the effect of rotating speed on two aspects: i) the reduce in liquid flow path length and contact time for adsorption due to the displacement of activated carbon in the packed bed, and ii) enhancement in mass transfer by centrifugal force (14,25,29). The exert of centrifugal force to the RPB system displaced the activated carbon bed to the outer wall of the RPB. The formation of the “cone” in the RPB bed caused uneven thickness of the activated carbon bed and this reduced the path length for the liquid flow within the packed bed (14,29), especially the upper layer of the bed, as shown in Figure 5. The reduced path length for the bed and liquid hold up reduced the contact time between the activated carbon and nickel solution (25). At low rotational speed of 200 rpm to 875 rpm, the effect given by liquid flow path length decrement had outperformed the increase in mass transfer coefficient by centrifugal force and thus, the removal efficiency was decreased. Nevertheless, it was worth noting that the removal efficiency was found to increase again when the rotational speed was increased from 875 rpm to 1200 rpm. It was suggested that the decrement in liquid flow path length had reached a stable condition at 875 rpm. Further increase in the rotation speed provided higher centrifugal force which enhanced the effective collision rate for adsorption. In this case, the improve in mass transfer had outperformed the effect of liquid flow path length decrement (14,29). Thus, higher removal efficiency was achieved.

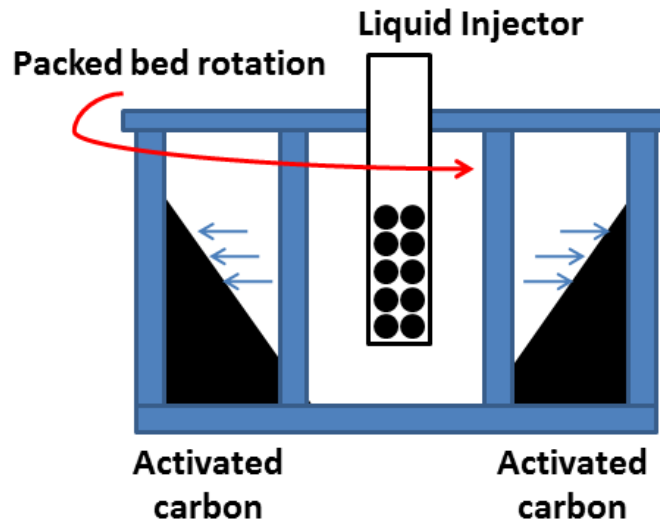


Figure 5: Displacement of activated carbon bed under centrifugal force

The increase in activated carbon dosage from 3 g to 13g is found to improve the removal efficiency from 4.84 % to 24.64 %, as shown in Figure 4d. The increase in activated carbon dosage in the RPB provided higher number of adsorption sites and contact surface area for higher adsorption rate. Nevertheless, it was worth noting that the improvement given by activated carbon dosage was dependent on the rotational speed of the RPB. This is well observed in the interaction effect between the rotational speed and activated carbon dosage on the removal efficiency, as shown in Figure 6. The removal efficiency is improved from 14.09% to 44.45% when the activated carbon dosage is increased from 3g to 13g at 200rpm. On the other hand, the improvement was found to be increased from 12.85% to 22.07% only at similar increment in activated carbon dosage when the system was operated at 1200 rpm. Higher removal enhancement at low rotational speed could be due to the fact that the increase in activated carbon dosage not only provided more adsorption sites but also provided higher packing density of the carbon bed which reduced the effect of the liquid flow path length and contact time reduction in the RPB as a result of centrifugal force (29). In contrast, the enhancement given by high packing density was relatively lower at higher rotational speed. This suggested that the interaction effect between the activated carbon dosage

and rotational speed was the crucial factors that must be considered in designing a rotating packed bed contactor.

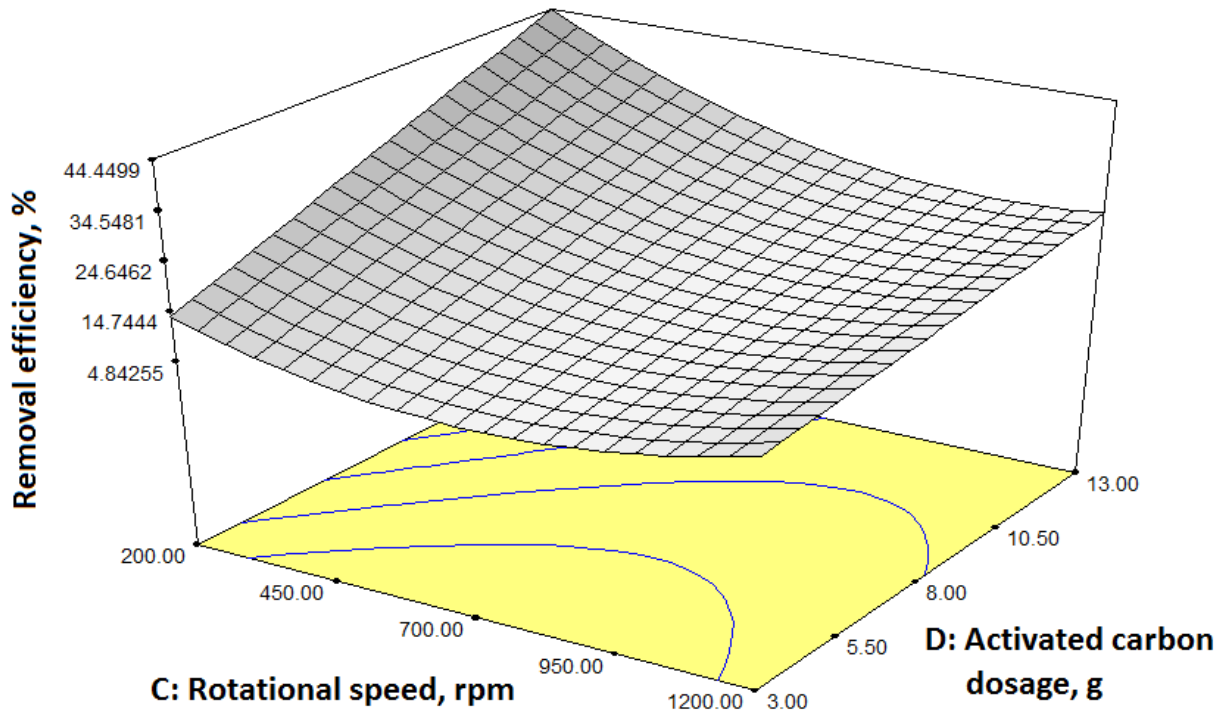


Figure 6: Interaction effect between rotating speed and activated carbon dosage on the removal efficiency

3.2.3 Validation of the model

Table 4: Optimum experimental conditions suggested by Design Expert

Parameters	Conditions
Volume of solution, L	1
Initial concentration, mg/L	61
Activated carbon dosage, g	13
Flow rate, L/h	50
Rotational speed, rpm	1200

An experiment was conducted to validate the accuracy of the model based on the optimum operating conditions suggested by the software. The operating conditions are as shown in Table 4. The experimental result showed that a removal efficiency of 36.30% was achieved, whereby the predicted

removal efficiency was at 38.12%. This concluded that the mathematical model generated is validated, as the difference between the predicted result and experimental result is close.

3.3 Effect of modes of operations

The effect of different modes of operation on removal of nickel from water using RPB was investigated under three configurations, namely: i) batch mode, ii) continuous mode, and iii) multiple cycle mode, using the operating conditions that are as shown in Table 4.

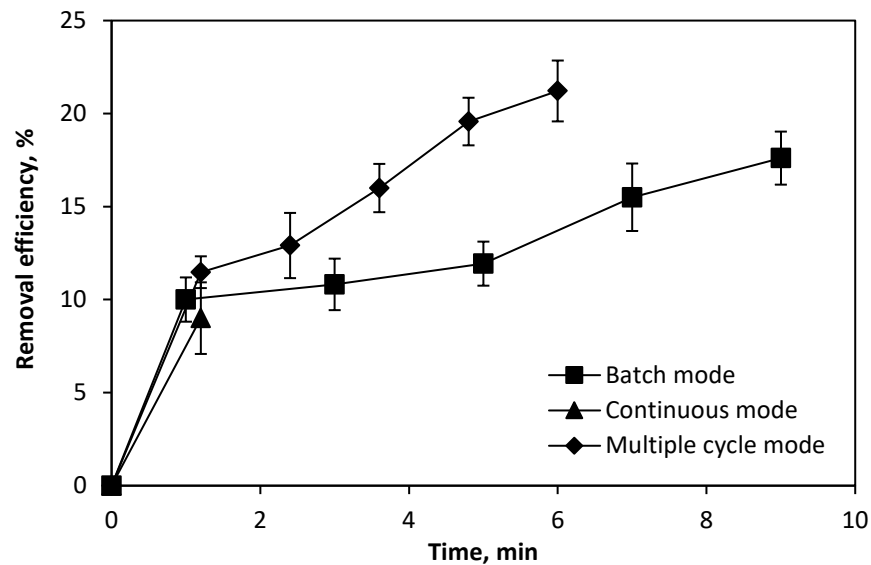


Figure 7: Removal efficiency of nickel using rotating packed bed contactor under different mode of operations

The experimental results were tabulated based on the contact time between the solution and the RPB, where the time required for a fully single pass of the nickel solution through the RPB was 1.2 min. Figure 7 suggests that multiple cycle mode has the highest removal efficiency (21.22%), followed by batch mode (17.61%) and continuous mode (9.00%). The difference in removal efficiency may be attributed to the effective contact time between the nickel solution and the RPB. Under similar operating conditions, continuous mode operation provided relatively lower contact time of 1.2 min in comparison to batch mode and multiple cycle mode, which were operated for 9 min and 6 min, respectively. The contact time is the crucial factor for the adsorption process, as it governs the effective collision rate between nickel

and activated carbon. This is well observed in the result whereby the removal efficiency is in range of 9-11 % for all the three modes when their contact times are similar at 1.2 min, as shown in Figure 7. The prolonged contact time in batch mode and multiple cycle mode improved nickel adsorption onto the activated carbon bed in the RPB. This is in line with Chang and Lee(28) whom reported that the liquid hold up time (contact time) plays crucial role in the performance of RPB.

Nevertheless, it was also found that the use of multiple cycle mode showed higher removal efficiency at 21.22% in comparison to batch mode at 17.61% at shorter contact time. As shown in Figure 7, higher increment in removal efficiency is observed in multiple cycle mode in comparison to batch mode, regardless of contact time. This was plausibly to be caused by the effective contact between the solution and the activated carbon. Unlike batch mode operation, the use of multiple cycle ensured all nickel solution had passed through the RPB and contacted with the activated carbon for nickel adsorption. In contrast, the use of batch mode in this study may create isolated area in the reservoir which is not contacted with RPB throughout the experiment, as shown in Figure 8. This may create concentration polarization at the respective area, and therefore, lower removal efficiency was obtained.

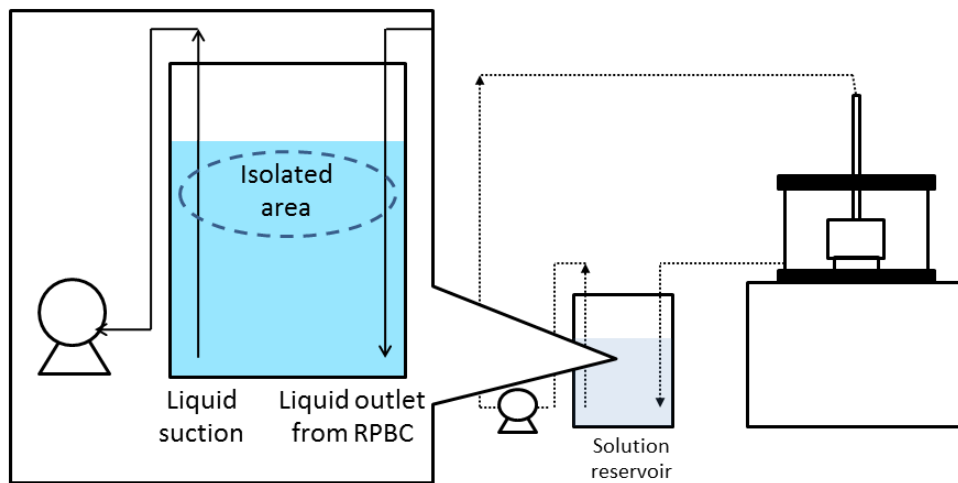


Figure 9: Isolated area in the solution reservoir in batch mode operation

Conclusions

The removal of nickel from water has been successfully carried out using a RPB contactor. The study revealed that the rotational speed and activated carbon dosage played the most important roles in affecting nickel adsorption in an RPB system. At low rotational speed (200 rpm - 875 rpm), the centrifugal force did not provide improvement to adsorption. Instead, it decreased the thickness and liquid flow path length of the carbon bed, which further reduced the contact time between the bed and nickel solution, eventually the removal efficiency. In contrast, higher rotational speed (875 rpm - 1200 rpm) was found to improve nickel adsorption as the reduction in bed thickness and liquid flow path length had reached a stable condition and further increase in centrifugal force improved the effective collision rate between nickel and the activated carbon bed. This condition can also be improved using higher activated carbon dosage as it not only provided higher number of active sites for adsorption but also improved the packing density which minimized the bed thickness reduction under centrifugal force. From the view of mode operation, multiple cycle mode operation showed better performance (21.22% in 6 min) due to its advantages of more consistent contact between the solution and the packed bed and higher contact time than batch mode operation (17.61% in 9 min) and continuous mode operation (9% in 1.2 min), respectively.

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