Journal of Environmental Science and Technology

ISSN 1994-7887

science alert

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Journal of Environmental Science and Technology 6 (3): 106-118, 2013
ISSN 1994-7887 / DOI: 10.3923/jest.2013.106.118

Optimization of Parameters Affecting Adsorption of Nickel (II), Zinc (II) and Lead (II) on Dowex 50 W Resin Using a Response Surface Methodology Approach

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ABSTRACT

The effects of resin dosage, pH of the medium and contact time on the adsorption of Ni^{2+}, Zn^{2+} and Pb^{2+} from battery industry wastewater using Dowex 50 W ion exchange resin were investigated. Batch-mode experiments were carried out to determine the adsorption equilibrium. The adsorption parameters were optimized using a Response Surface Methodological (RSM) approach. The Central Composite Rotatable Design (CCRD) in RSM was used to design the experiments and estimate the percentage of metal adsorption. The optimum parameters for achieving the maximum adsorption of Ni^{2+}, Zn^{2+} and Pb^{2+} were as follows: resin dosage, 0.3-0.42 g; pH, 6-8.5; contact time, 90 min. Experimental results showed that the adsorption efficiency of the heavy metals was in the range of 80-100%. The results of this work suggest that RSM is a useful tool for optimizing reaction parameters and predicting response (adsorption capacity) during the ion exchange of heavy metals to achieve the desired adsorption efficiency, while reducing the number of experiments and the overall cost of treatment.

Key words: Heavy metals, adsorption, response surface methodology, battery industry effluent

INTRODUCTION

Heavy metal pollution has become one of the most serious environmental problems which undermine industrial and modern societies (UNEP, 2004; Kalantari *et al.*, 2006; Cavaco *et al.*, 2007). The increasing amount of metals and chemicals in various industrial processes such as metal plating facilities, mining operations, fertilizer industries, tanneries, batteries, paper making has resulted in generation of large quantities of effluents containing high levels of heavy metals. These contaminated effluents pose environmental-disposal problems due to their non-biodegradability and tendency to accumulate in living organisms (Petrus and Warchol, 2003; Rana *et al.*, 2009). Furthermore, the leaking of heavy metals into the soil can lead to groundwater and surface water contamination and consequently, adversely affect human, animal and aquatic life.

Recently, the Malaysian Department of Environment (DOE, 1998) reported that several Malaysian rivers were polluted by heavy metals. This has been caused by the growth of the electronic industry which produces computer chips and semiconductors and generates a substantial amount of wastewater polluted with heavy metals such as Pb, Zn and Ni. According to Malaysian-Environmental Quality Act 1974 (Department of Environment, 1997), the maximum discharge limits under standard B is 0.5 mg L^{-1} for Pb (II) and 1.0 mg L^{-1} for Zn (II) and Ni (II).

Industrial wastewater from the manufacture of batteries contains large amounts of Pb (II), Ni (II) and Zn (II) which must be treated prior to disposal. The ion exchange technique is effective in separating solute particles and capable of extracting positively charged ions (Pehlivan and Altun, 2006). Due to the effective and efficient technique, ion exchange resins have been introduced as major alternatives in wastewater treatment process (Edebali and Pehlivan, 2010). The advantages of ion exchange processes are high treatment capacity, high removal process efficiency, fast kinetics and the variety of specific resins available (Kang *et al.*, 2004; Clifford, 1999). Synthetic resins are widely used due to the effective removal of heavy metals from aqueous solution (Alyuz and Veli, 2009). Dowex 50 W resins are robust, insoluble and compatible with various conditions. The sodium form of this resins imparts both stability and a long shelf life (Pehlivan and Altun, 2006).

According to Pehlivan and Altun (2006), in removing toxic and harmful transition metals from aqueous solutions, a Dowex 50W resin was more efficient than conventional resins and natural adsorbents. In 2006, Pehlivan and Altun attempted to use Dowex 50 W-type ion exchangers for removing heavy metals such as Pb (II), Cu (II), Zn (II), Cd (II) and Ni (II) from different aqueous solutions. In that study, varying concentrations of test metals were prepared and experimental measurements were carried out by batch sorption (pH range 2-10, resin amount from 0.01-0.13 g). The results indicated that the higher removal of Pb (II) (80%; 70 min contact time) and Ni (II) and Zn (II) (97%; at 60 and 70 min contact time, respectively), was achieved in the pH range between 8 and 9.

The cited experimental work utilized a classical approach: the One-Factor-At-Time (OFAT) method. This approach is a lengthy and time consuming technique (Garg *et al.*, 2008). The limitations of OFAT can be eliminated by optimizing the process/reaction parameters using factorial strategies, by means of statistical experimental design with Response Surface Methodology (RSM) (Elibol, 2002; Garg *et al.*, 2008). The objective of using RSM is not only to investigate the response over the entire factor space but also to find the optimum magnitude of the parameter.

The objective of this study was the optimization of the reaction parameters affecting the performance of Dowex 50 W ion exchange resin in adsorption of Pb (II), Ni (II) and Zn (II) from battery manufacturing industrial wastewater using RSM. The Central Composite Rotatable Design (CCRD) was selected to quantitatively identify the significant and interactive factors in this process (Aslan, 2008). In addition, the design facilitates the detection of non-linear behavior of an effect and the determination of the best setting of the experimental factors that produce the maximum outcome. The formulated model was analyzed using analysis of variance (ANOVA) by means of MINITAB™ statistical software. The method is described by Cavaco *et al.* (2007) and Aslan (2008).

MATERIALS AND METHODS

Materials: All chemicals and reagents used were of analytical grade. Solutions of 0.1 M NaOH and 0.1 M HNO_3 were used for pH adjustment while preserving the heavy metal contents of the industrial wastewater. Synthetic Dowex 50W in hydrogen form (16-45 mesh size) was obtained from Fluka Co. The properties of the resin are presented in Table 1.

Table 1: Properties of dowex 50 W

Type	Strong acid cation exchanger gowex 50 W-X8
Active group	Sulfonic acid
Matrix	Gel (microporous)-styrene divinylbenzene
Ionic forms as shipped	H^+
Physical form	Spherical beads
Standard mesh sizes (wet)	20-50
Mean particle size (μm)	560
Effective pH range	0-14
Total exchange capacity	H^+ form
mequiv./g dry resin	4.8

Sample collections: Final effluent from the battery manufacturing industry was collected, preserved and analyzed according to standard methods for the examination of water and wastewater (APHA, AWWA and WEF, 1995).

Batch system: Adsorption studies for all solution samples were investigated on a batch basis. Effluent samples (30 mL) placed in falcon tubes were used in all experiments. A specific amount of resins was weighed and added to the tubes. It is according to the value generated MINITAB factorial design software). The pH of the solution was adjusted to desired value using 0.1N HCl or 0.1N NaOH. The tubes were placed in a water bath shaker and agitated at 200 rpm for the desired contact time. The final metal ion concentration was measured, after filtration through Whatman filter paper, using inductively coupled plasma-mass spectrometry (ICP-MS, ELAN-6000, Perkin Elmer).

Response surface methodology: The experiments were statistically designed using Response Surface Method (RSM). In particular, CCRD was used with the aid of MINITAB™ statistical software. Using CCRD, the experiments were divided into three parts:

- 2^3 Hadamard matrix factorial run (1-8)
- Star point runs (9-14)
- Centre point runs (15-20)

The synergistic effect of the factor(s) were determined based on the Hadamard matrix and center point runs while the non-linear response behavior was analyzed using the star point and center point runs. The center point run was repeated six times to better estimate the experimental error. The runs were carried out in a random order, to reduce the effect of time-dependent factors that are not included in the study. The procedures for experimental design are as follows:

- Three parameters that affect the response parameters, the pH of the feed solution, the resin dosage and the contact time, were selected. The values are listed in Table 2. The appropriate experimental design was chosen (central composite design)
- Experimental materials were randomized in terms of the runs and order of runs

Table 2: Coding system for design of experiments of the batch process on ion exchange system

Factor	Units	Low level (-1)	High level (+1)
pH		4.5[a]	8.5
Resin dosage	Gram	0.1[a]	0.5
Contact time	Minute	30.0	120.0

a: Minimum value, b: Maximum value

Table 3: Experimental results of batch mode application of ion exchange process conducted for removal Ni (II), Zn (II) and Pb (II) on dowex 50 W

				Removal (%)		
Standard order	pH	Resin dosage, RD (g)	Contact time (min)	Ni(II)	Zn (II)	Pb (II)
1	5.5 (-1)	0.2 (-1)	52.5 (-1)	51.0	55.0	47.9
2	7.5 (+1)	0.2 (-1)	52.5 (-1)	65.0	60.6	55.6
3	5.5 (-1)	0.4 (-1)	52.5 (-1)	63.0	53.9	53.0
4	7.5 (+1)	0.4 (-1)	52.5 (-1)	74.0	75.8	68.0
5	5.5 (-1)	0.2 (+1)	97.5 (+1)	91.3	91.0	84.5
6	7.5 (+1)	0.2 (+1)	97.5 (+1)	92.5	80.0	73.7
7	5.5 (-1)	0.4 (+1)	97.5 (+1)	91.4	86.8	88.0
8	7.5 (+1)	0.4 (+1)	97.5 (+1)	97.8	96.4	67.0
9	4.5 (-1.682)	0.3 (-1)	75.0 (0)	97.0	55.0	80.0
10	8.5 (+1.682)	0.3 (-1)	75.0 (0)	96.7	94.8	87.0
11	6.5 (0)	0.1 (-1.682)	75.0 (0)	83.0	55.0	42.7
12	6.5 (0)	0.5 (+1.682)	75.0 (0)	94.3	90.9	61.7
13	6.5 (0)	0.3 (0)	30.0 (-1.682)	38.0	43.0	47.9
14	6.5 (0)	0.3 (0)	120.0 (+1.682)	98.0	96.2	90.8
15	6.5 (0)	0.3 (0)	75.0 (0)	95.0	95.0	90.3
16	6.5 (0)	0.3 (0)	75.0 (0)	95.0	95.6	88.0
17	6.5 (0)	0.3 (0)	75.0 (0)	97.9	95.4	89.0
18	6.5 (0)	0.3 (0)	75.0 (0)	97.0	96.0	91.0
19	6.5 (0)	0.3 (0)	75.0 (0)	98.0	99.6	92.3
20	6.5 (0)	0.3 (0)	75.0 (0)	96.0	95.6	90.0

[a]Based on standard order designed by CCRD, [b]( ): Coded value as assigned by CCRD, (-1) indicates lower part of the Hadamard matrix, (+1) the upper part and (0) the center point. The values (±1.682) denote locations outside the matrix

- The experiments as shown in Table 3 (column 1, 2 and 3) were conducted to obtain the value for the percentage of heavy metals removal
- Regression analysis and modeling were carried out using MINITAB
- ANOVA was determined by using MINITAB
- The contour and surface response graphs were plotted using MINITAB
- A conclusion was drawn from the optimum combination of factors from the surface response graphs to optimize the performance of the resins

Data analysis: In each experiment, the performance of the ion exchange process on a batch basis was expressed in terms of the percentage of adsorbed metal ions, This is defined as:

$$\%\ \text{Adsorbed metal ion} = \frac{C_o - C_f}{C_o} \quad (1)$$

Where:
C_o = Initial metal ion concentrations in the adsorption medium
C_f = Final metal ion concentration

RESULTS AND DISCUSSION

Table 3 shows the experimental results of batch mode application of the ion exchange process conducted for the removal of Ni (II), Zn (II) and Pb (II) from waste water using Dowex 50 W. Columns 5, 6 and 7 represent summaries of the percentage of removal of the heavy metal ions. As shown in Table 3, the experiments were conducted based on the generated value of independent variables such as pH, amount of resin dosage (g) and contact time (minutes) to obtain the value of the dependent variable (percentage of heavy metals removal).

Response surface models and statistical analysis: The main objective of response surface analysis was to develop a statistical model to show the combined effect of the independent parameters investigated on the response, i.e., the percentage of metal removal of Ni (II), Zn (II) and Pb (II). Based on the results in Table 3, further analysis was carried out to achieve the above objective.

Table 4 shows the results of ANOVA for adsorption process of Ni (II), Zn (II) and Pb (II) ions. The significant lack-of-fit (less than 0.005) showed that a quadratic model is invalid for this study. The p-value of the lack-of-fit for these heavy metals (0.000) indicates that they were not significant for this set of experiments. This implies that the predictive knowledge of the model was statistically insufficient.

To improve the model another attempt was carried out based on the p-value and only the significant parameters were considered in the analysis of the quadratic terms. Omitting single and interaction variables having p>0.2 gave a p-value for the remaining terms compared to the first attempt. Table 5 summarizes the p-values for the response surface modeling analysis for the heavy metal removal. The significant value of lack-of-fit (0.000;0.001;0.025) for Ni (II), Zn (II) and Pb (II) indicated that no accurate models could be derived based on the experimental evidence so far.

Table 4: Summary of p-value for the response surface modeling analysis for percentage of heavy metal removal (full quadratic model)

	Full quadratic (p-value[a])		
Term	Ni (II)	Zn (II)	Pb (II)
pH	0.080	0.004	0.032
Resin dosage (RD)	0.020	0.006	0.087
Time (t)	0.000	0.000	0.000
pH×pH	0.399	0.004	0.013
RD×RD	0.007	0.002	0.070
t×t	0.000	0.001	0.008
pH×RD	0.865	0.096	0.910
pH×t	0.198	0.181	0.054
RD×t	0.245	0.927	0.426
R^2, %; LOFT value	96.7%; 0.002	91.2%; 0.001	86.4%; 0.016

[a]0.01-0.04: Highly significant, 0.05-0.1: Significant, 0.1-0.2: Less significant, >0.2: Insignificant, LOFT: Lack of fit test

Table 5: Summary of p-value for the response surface modeling analysis for percentages of heavy metal removal (second attempt)

	p-value[a]		
Term	Nickel (II)	Zinc (II)	Lead (II)
pH	0.074	0.004	0.022
Resin dosage	0.016	0.006	0.067
Time	0.000	0.000	0.000
pH×pH	-	0.004	0.008
RD×RD	0.006	0.002	0.053
t×t	0.000	0.001	0.004
pH×RD	-	0.092	-
pH×t	-	-	0.039
RD×t	-	-	-
R^2, %; LOFT value	95.3%; 0.000	91.2%; 0.001	85.4%; 0.025

[a]0.01-0.04: Highly significant, 0.05-0.1: Significant, 0.1-0.2: Less significant, >0.2: Insignificant

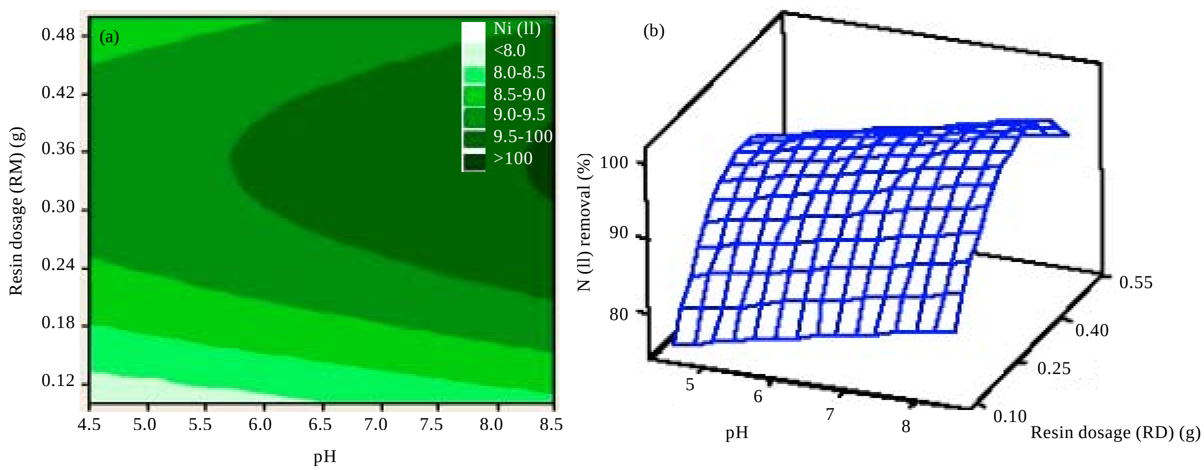

Fig. 1(a-b): Response surface contour and 3 D plot representing relationship between percentage of Ni (II) removal, resin dosage and pH

Effect of pH: The adsorption of metal ions depends on the nature of the adsorbent surface and adsorbate species in the solution (El-Sayed *et al*., 2011). The retention of metals on the resin was studied as a function of pH value. As shown in Table 4, the pH value was the most important factor (p-value = 0.074; 0.004; 0.022) for Ni (II), Zn (II) and Pb (II) removal. This means that the metals adsorption capacity increases with the increase in pH for all metals ions within the pH range 6-8.5. Similar results were obtained by Cagin (2006), who found that the pH value had an obvious impact on Ni (II) removal by Dowex 50W resin.

As shown in Fig. 1 and 2, at the pH range 6-8.5, Ni (II) removal reached 80% at 60 min contact time. The highest removal percentage of Ni (II) (almost 100%) was found at pH above 6, after 90 min contact time. Furthermore, Ni (II) was poorly adsorbed at pH<4.5. The lowest percentage of Ni (II) removal was less than 50% after 40 min and at pH<5. Even after extending the contact time to 60 min the removal was still <80%. Based on the contour and response surface 3D plots, the optimum pH value for this experiment is 8.5 at which the highest removal (almost 100%) was achieved in 90 min contact time.

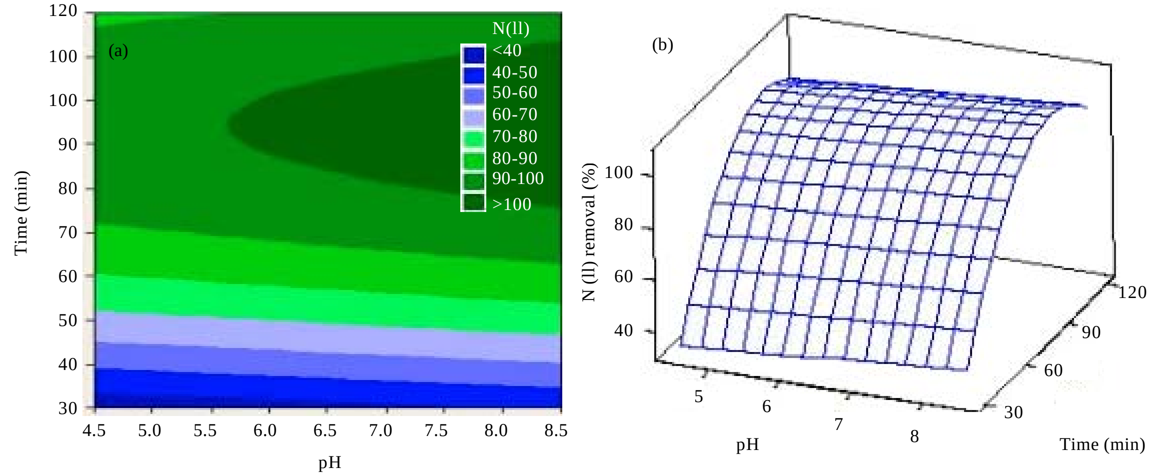

Fig. 2(a-b): Response surface contour and 3 D plot representing relationship between percentage of Ni (II) removal, pH value and contact time

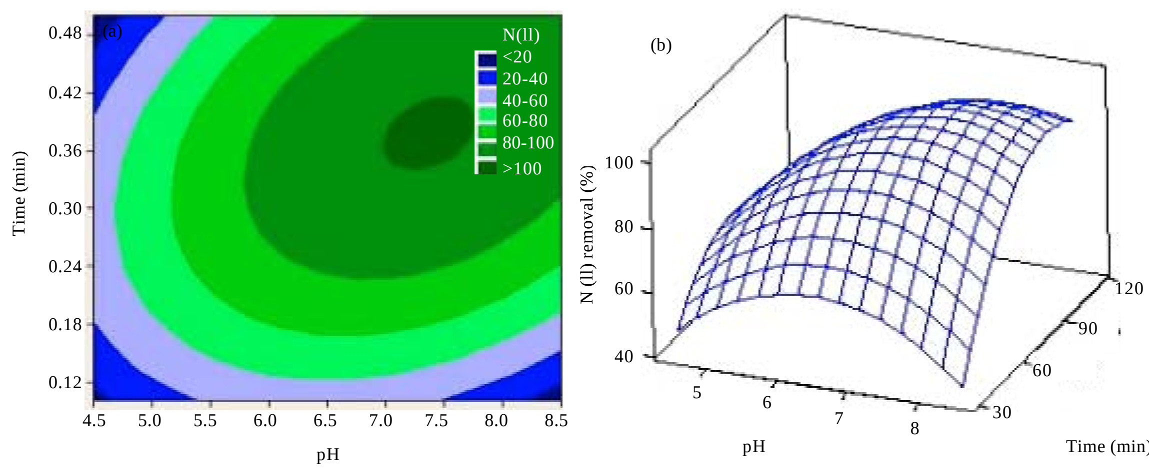

Fig. 3(a-b): Response surface contour and 3 D plot representing relationship between percentage of Zn (II) removal, resin dosage and pH

Response surface contour and 3D plots representing relationship between Zn(II) and Pb (II) removal, pH value, amount of resin dosed and contact time are shown in Fig. 3-6. As shown in all graphs, the removal percentage increases in the sequence of Ni (II)>Zn (II)>Pb (II). The highest removal percentage of Zn (II) and Pb (II) (almost 100% and 80%, respectively) were found in the pH range between 6 and 8 and 7.5, respectively, after 90 min contact time. Furthermore, Zn (II) was poorly adsorbed at pH<4.5. In contrast, the lowest percentage of Zn (II) removal was <40% after 40 min at pH<4.5 which increased to <60% removal at 60 min contact time. Further, the lowest percentage of Pb (II) removal was found at pH above than 8.5, with less than 60% removal. The low adsorption capacity of both metals ions (Ni (II) and Zn (II)) at low pH (<4) can be ascribed

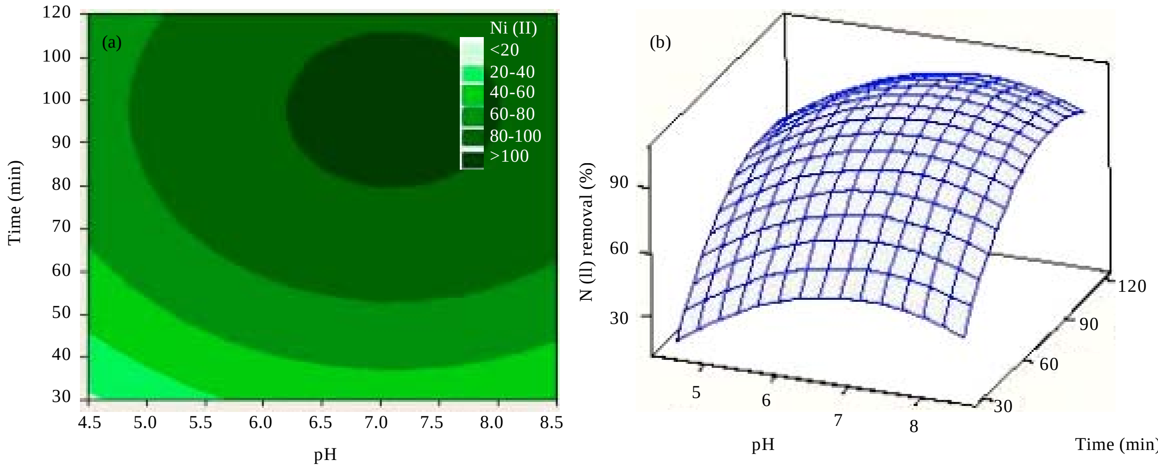

Fig. 4(a-b): Response surface contour and 3 D plot representing relationship between percentage of Zn (II) removal, pH and contact time

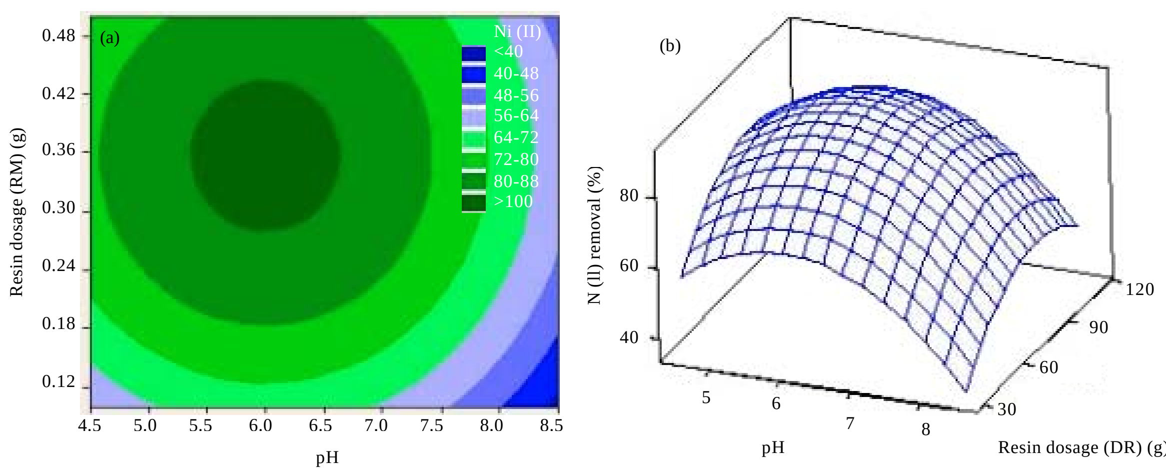

Fig. 5(a-b): Response surface contour and 3 D plot representing relationship between percentage of Pb (II) removal, resin dosage and pH

to competitive adsorption of the hydronium (H_3O^+) ions and metal ions for the same active adsorption sites. As the pH increases, the adsorption zones tend to attain less positive charges, leading to an increase in electrostatic attraction between the metal ions and sulfonic acid groups (Nasef *et al*., 2010).

This behavior may be attributed to the electrostatic effects that cause the adsorption capacity to increase with the decrease in the radius of hydrated metal ions which changes in the sequence of Ni (II)<Zn (II)<Pb (II). These results are in agreement with those reported by Alpatova *et al.* (2004), Demirbas *et al.* (2005) and Pehlivan and Altun (2006).

Effect of resin dosage: As shown in Table 4 and 5, resin dosage is a significant parameter in determining the percentage of metals removal for Ni (II) (0.016), Zn (II) (0.006) and Pb (II) (0.067).

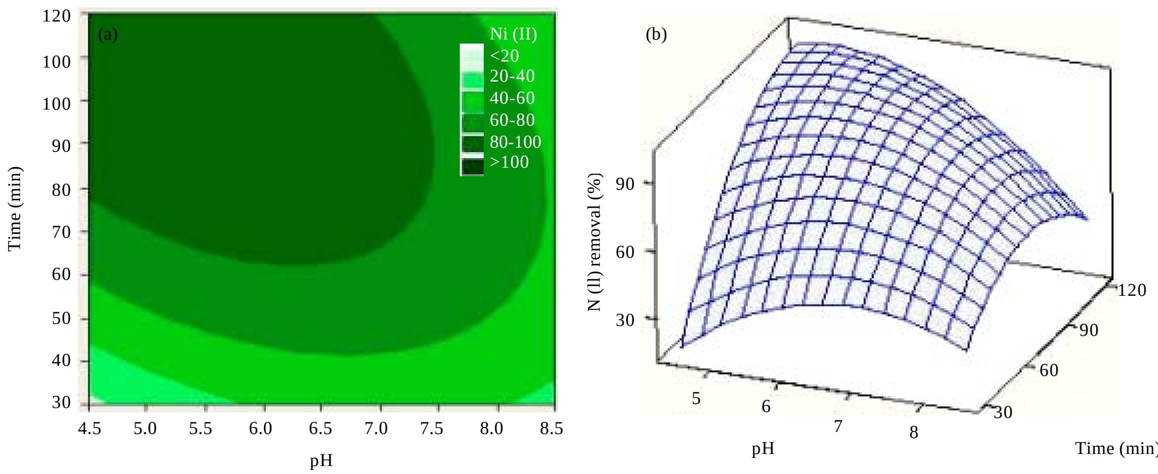

Fig. 6(a-b): Response surface contour and 3 D plot representing relationship between percentage of Zn (II) removal, pH and contact time

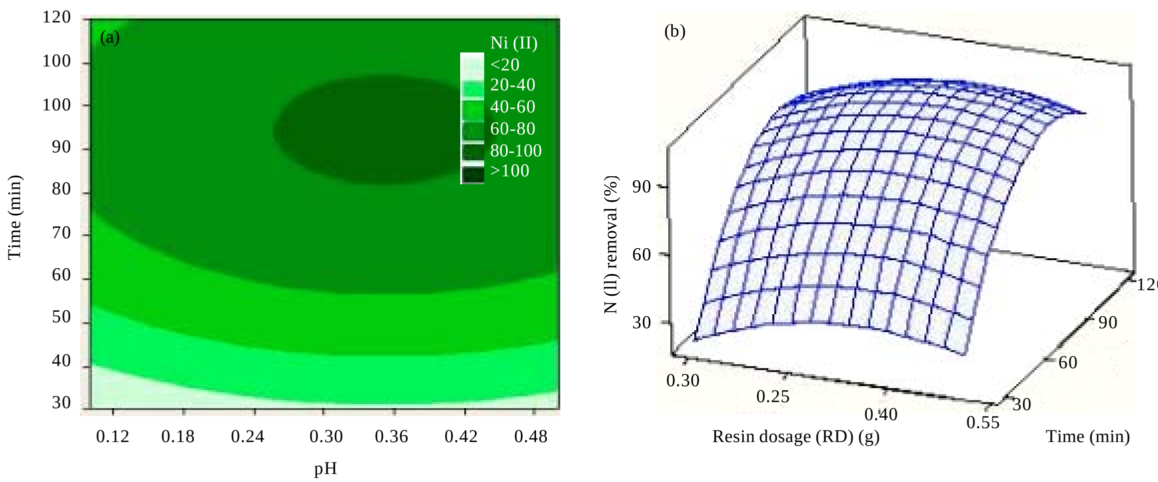

Fig. 7(a-b): Response surface contour and 3 D plot representing relationship between percentage of Ni (II) removal, resin dosage and time

It is evident from these figures that the increase in the resins amount led to an increase in the sorption density and the amount adsorbed metal ion per unit mass. This was due to the increase in the number of available sorption sites with the increase in the resin amount. Thus, by increasing the adsorbent dose, the removal efficiency increased but the ion exchange density decreased. This was in agreement with literature reports (Pehlivan and Altun, 2007). The decrease in the ion-exchange density can be attributed to the fact that some of the ion-exchange sites remain unsaturated during the sorption process, whereas the number of available ion-exchange sites increases with the additional amount of sorbent, thus raising the removal efficiency (Namasivayam and Senthilkumar, 1999).

Effect of contact time: Figure 7-9 illustrate that the percentage of metal ion adsorbed increased with time and it reaching 80% of metal ions removal in 60 min for Ni (II) and Zn (II) and in almost 70 min for Pb (II).

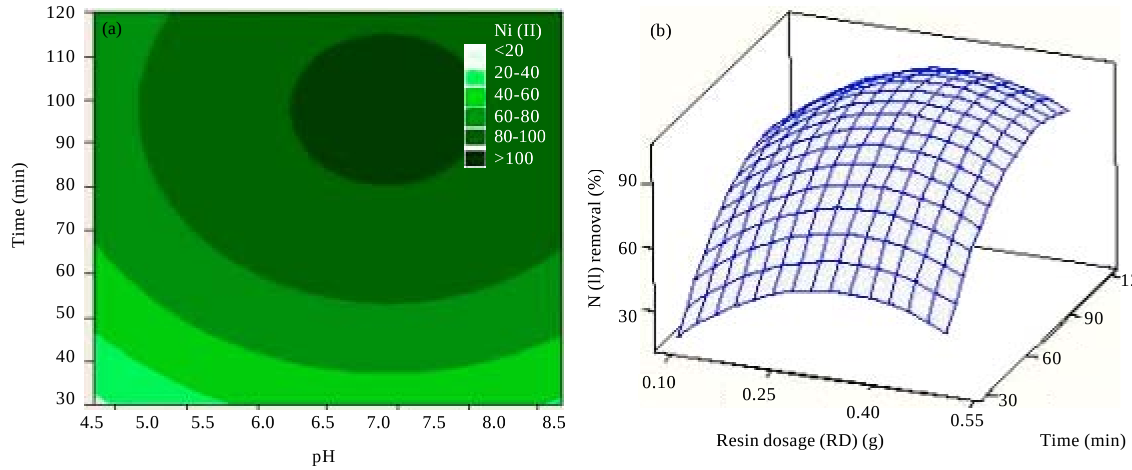

Fig. 8(a-b): Response surface contour and 3 D plot representing relationship between percentage of Zn (II) removal, rasing dosage and time

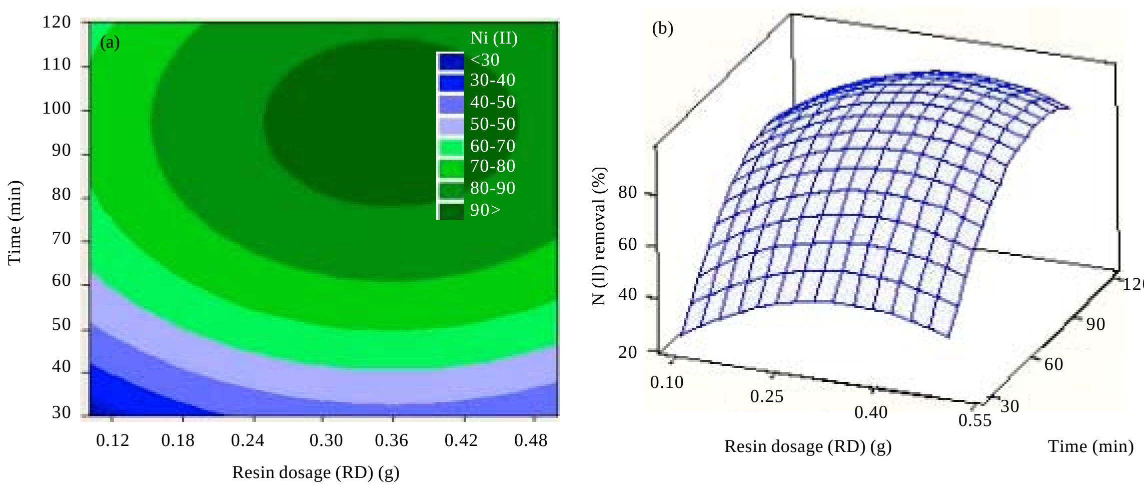

Fig. 9(a-b): Response surface contour and 3 D plot representing relationship between percentage of Pb (II) removal, resin dosage and time

Interpreting the experimental results by the distribution coefficient: The experimental results can also be interpreted by using the distribution coefficient K_d (L g^{-1}) that expresses the affinity of ion exchangers for metal ions. K_d can be defined as the relative absorption of the metal (see Eq. 1) multiplied by the volume of the solution (V) and divided by the mass of absorbent (m):

$$K_d = \frac{(C_0 - C_f) \cdot V}{C_0 \cdot m} \qquad (2)$$

The results of Fig. 10a-c show the K_d values of the metal ions between the ion exchanger (Dowex 50 W) and the liquid solution. The following order of metal ion affinities was found; Ni

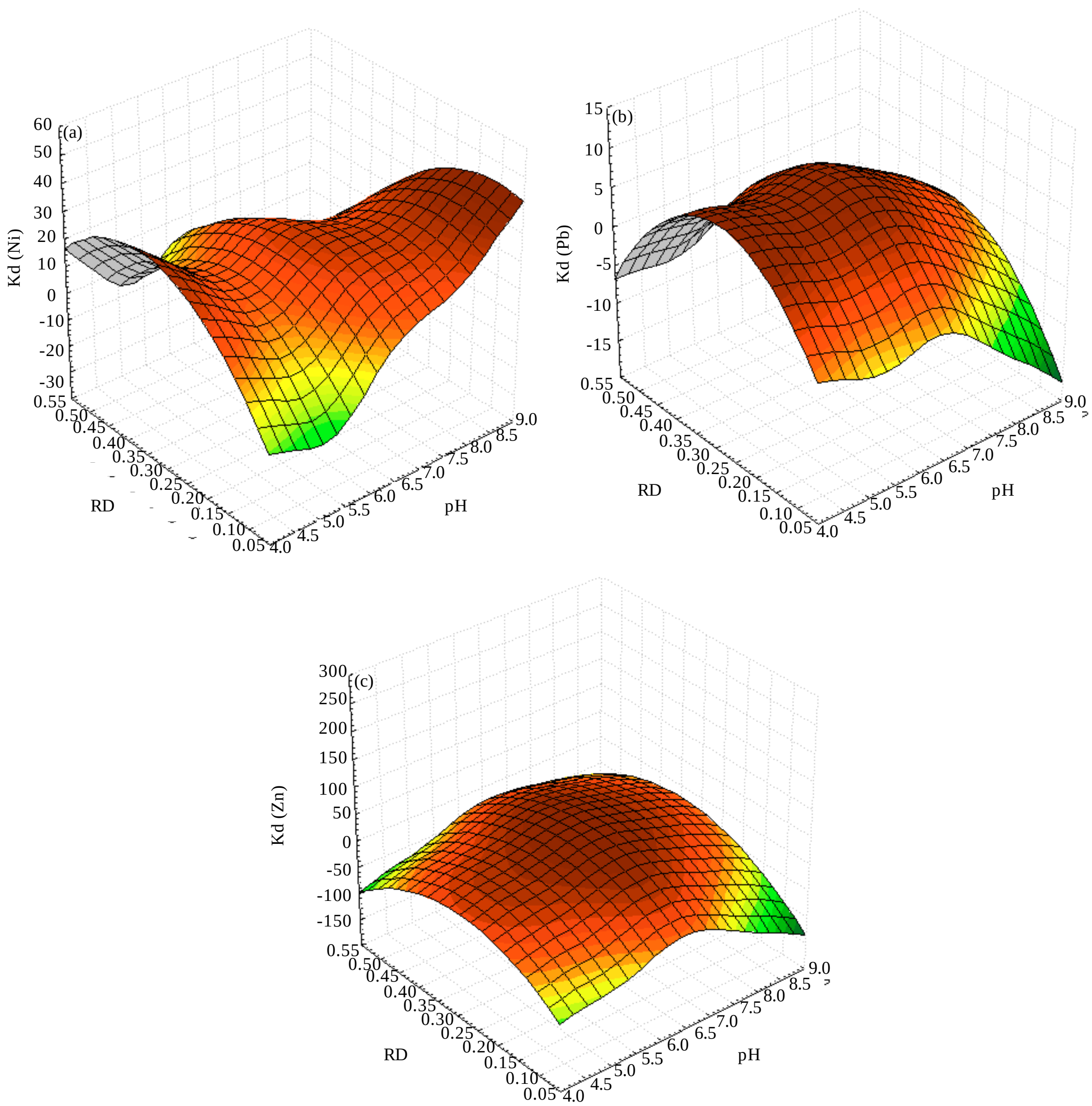

Fig. 10(a-c): Response surface 3 D plot representing the relationship between distribution coefficient (K_d) of the metal, the pH value and amount of resin dosage (a) Ni (II), (b) Pb (II) and (c) Zn (II)

(II)>Zn (II)>Pb (II). The K_d values were very low up to about pH 4.5 for the three metal ions. At higher pH value, K_d increased and reached a maximum value at about pH 9 for Ni (II) and Zn (II) and 7 for Pb (II). However, the K_d value for Pb (II) decreased at pH>7. There are two points that can explain the effect of the pH on the distribution coefficient. First, the pH value could influence the surface structure, charge properties and adsorption of the resins. In general, the oxidation and reduction properties of acidic groups on ion exchange resins are attributed to pH changes. Second, metal speciation also depends on the change in pH. Different ion speciation exhibits different characteristics and can affect the adsorption process (Liu *et al*., 1999).

CONCLUSION

Response surface methodology was used to optimize the process variables for the removal of some heavy metals namely Ni (II), Zn (II) and Pb (II) from solutions mimicking battery-industry wastewater using commercial ion exchange resins (Dowex 50 W). It is found that the pH, amount of resin dosage and contact time had significant effects on the removal of metal ions. The experimental results illustrated in the smooth 3D graphs also give a clear indication that there was a well-defined optimum parameters for the metal ion removal. The optimum pH applied for maximum removal of Ni (II) and Zn (II) is in the range 6-8.5 and pH<7.5 for Pb (II). The optimum contact time was around 90 min and the amount of resin dosage was between 0.30 and 0.42 g. The removal efficiency (%) increased in the sequence of Ni (II)>Zn (II)>Pb (II). The results of this study suggest that factorial strategies are effective proving the applicability of the tested resins for treatment of industrial wastewater from the battery industry. Response Surface Methodology (RSM) was a useful tool for optimizing the reaction parameters and reducing the number of experiments and overall cost of testing.

ACKNOWLEDGMENT

The authors wish to acknowledge the financial support for this research from the Malaysian Ministry of Science, Technology and Innovation (MOSTI), under the E-Science fund program (Vot No. 79283) and the Ministry of Higher Education (MOHE), under the Fundamental Research Grant Scheme (FRGS) (Vot No. 78400).

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