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Response surface methodology for optimising the operating conditions of nickel(II) adsorption

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In this paper, activated carbon has been produced by physical activation for sawdust, the produced activated carbon has been characterised and used as adsorbent for removal of nickel (Ni) (II) from aqueous solution. The present work also represents two models used to optimise the operating conditions that affect the nickel(II) removal from aqueous solution using activated carbon prepared by physical activation of sawdust as adsorbent. These operating conditions include the initial concentration of nickel ion, pH and dosage of adsorbent. One of these models is developed using linear regression and the other using response surface methodology (RSM). The two models were validated with experimental data and showed good agreement, with R^2 ranges from 0·92 to 0·98.

Notation

A	pH of the solution
B	weight of adsorbent (g/L)
b	Langmuir constant (L/mg)
C	nickel(II) concentration (mg/L)
C_e	concentration of heavy metal at equilibrium (mg/L)
C_f	final concentration of the solute in the bulk phase at time t (mg/L)
C_0	initial heavy metal concentration (mg/L)
X_0	uncoded value of the ith test variable at centre point
X_i	uncoded value of the ith test variable
x_i	coded value of the ith variable
Y	the predicted response
β_i, β_j, β_{ij}	coefficients estimated from regression

Introduction

Heavy metals have a great tendency to bio-accumulate and end up as permanent additions to the environment. The removal of heavy metals from wastewater has recently become the subject of considerable interest owing to strict legislations introduced to control water pollution. Industrial wastewaters contain many toxic heavy metals such as lead (Pb), mercury (Hg), chromium (Cr), nickel (Ni), cadmium (Cd), copper (Cu) and zinc (Zn), and discharging them into the environment leads to serious soil and water pollution (Motsi *et al*., 2011). Some metals have high levels of toxicity, especially lead(II), mercury(II), cadmium(II), nickel(II) and chromium(VI). Toxicity levels of heavy metals depend on the type of metal, its valence states, its biological role and volume or concentration (Qiu and Qiu, 2009; Shi *et al*., 2009). The concentrations of these metals must be reduced to acceptable levels before discharging them into the environment (Hassan *et al*., 2004). According to the World Health Organization (WHO), the metals of most immediate concern are chromium, copper, zinc, iron, nickel, mercury and lead (WHO, 2010). Nickel is a well-known heavy metal pollutant, which is present in effluents of electroplating industries in concentrations of 20–200 ppm. It is a toxic heavy metal found in the environment as a result of various natural and industrial activities. Nickel has been implicated as an embryotoxin and teratogen (Chen and Lin, 1998). The higher concentration of Ni causes poisoning effects like headache, dizziness, nausea, tightness of the chest, dry cough, vomiting, chest pain, shortness of breath, rapid respiration, cyanosis and extreme weakness (Revathi, 2005). These harmful effects of nickel(II) necessitate its removal from wastewater before release into streams. Studies of human cell cultures have indicated that nickel is a possible carcinogen, so it is essential to clean up nickel pollution by removing it from soil, industrial wastewater and effluents.

Various methods have been reported in the literature to remove nickel such as electrocoagulation (Aji *et al*., 2012; Ferreira *et al*., 2013), ion-exchange separation (Kumar, 2010; Shaidan and Eldemerdash Awad, 2012), photo catalysis (Shaomin *et al*., 2010), ultrafiltration (Kadioglu *et al*., 2010; Trivunac and Stevanovic, 2006), biosorption (Farooq *et al*., 2010), membrane-filtration (Borbély and Nagy, 2009) and chemical precipitation (Blais *et al*., 2008; Giannopoulou and Panias, 2008). Among the possible techniques for water treatments, the adsorption process by solid adsorbents shows potential as one of the most efficient methods for the treatment and removal of heavy metals in wastewater treatment (Argun, 2008). Adsorption has advantages over the other methods because of simple design and can involve low investment in terms of both initial cost and land required (Chen *et al*., 2009). The adsorption processes are widely used for treatment of industrial wastewater from organic and inorganic pollutants and have received much attention from researchers (Krishnan *et al*., 2011). In recent years, the search for low-cost adsorbents that have pollutant-binding capacities has intensified. Materials locally available such as natural materials, agricultural wastes, and industrial wastes can be utilised as low-cost adsorbents. Activated carbon produced from these materials can be used as adsorbent for water and wastewater treatment (Crini, 2005). Traditional techniques using commercially available activated carbon for removal of heavy metals of nickel have been the preferred choice as the adsorption efficiency of activated carbon depends on surface area and pore volume and these principal characteristics make activated carbon ideal for adsorption (Gottipati and Mishra, 2010). Activated carbon widely used in the adsorption process can be made from several materials among which coal (Yang *et al*., 2002), wood (Wang *et al*., 2009) and coconut shells (Su *et al*., 2006) are popular because of high porosity and large surface area. However, high cost and dependence on importation for most developing countries would mean that alternatives with similar surface characteristics and efficiency have to be sought. Nowadays, low cost and environmentally benign and abundant resources are getting widespread attention for nickel effluent treatment, where agricultural waste and biomass products are converted into a value-added system as effective adsorbents.

The present work deals with the application of response surface methodology (RSM) tools for modelling and optimisation of the operating conditions of nickel removal from aqueous solution using activated carbon prepared by physical activation for sawdust as adsorbent. The aim of the experiments was to optimise the operating conditions such as initial concentration of nickel, pH and dosage of adsorbent to ensure the maximum removal efficiency.

Materials and methods

Materials

The sawdust was collected, screened to 300-μm particle size, then washed with distilled water and oven-dried at 60°C for 4 h. It was then stored for use in order to achieve the same conditions for the sawdust used in the experiments as for the raw material sawdust. All chemicals were analytical grade reagent and the glassware was Pyrex washed with soap, rinsed thoroughly and then washed with deionised water.

Preparation of activated carbon from sawdust

The preparation of activated carbon by activation process takes place by physical activation. The carbonisation of the dried sawdust was carried out using a muffle furnace which allows limited supply of air. Carbonisation was carried out at 700°C until the sawdust was transformed completely into black powder (98% of sawdust turned to activated carbon).

Characterisation of activated carbon

Morphology analysis

In order to know the structure of the activated carbon, scanning electron microscopy (SEM) was employed to visualise sample morphology. In the present work, the activated carbon was analyzed by this technique using SEM 'JEOL JSM 6360LA'.

Fourier transform infrared spectroscopy analysis

The surface functional groups and structure were studied by Fourier transform infrared (FTIR) spectroscopy. The FTIR spectra of the resulting activated carbon were recorded between 500 and 4000 cm^{-1} in FTIR-8400 S Shimadzu.

Batch studies

A stock solution of nickel(II) (1000 ppm) was prepared by dissolving 4·953 g of nickel nitrate, $Ni(NO_3)_2.6H_2O$ (analytical reagent grade) in 10 ml of concentrated sulfuric acid and diluted to 1 L with de-ionised distilled water. Samples with various nickel(II) concentrations ranged from 25 to 75 ppm were prepared from the stock solution. Single-stage batch adsorption tests were conducted in 100 ml solution. Nickel solutions contained in flasks were stirred using IKA RT5 model magnetic stirrer operated at 200 rpm. The percentage removal of nickel(II) was evaluated from the following equation

$$\% \text{ Removal} = \frac{(C_0 - C_\text{f})}{C_0} * 100 \quad (1)$$

where C_0 is the initial concentration of the adsorbate solution, and C_f is the final concentration of the solute in the bulk phase at time t.

Modelling by full factorial design

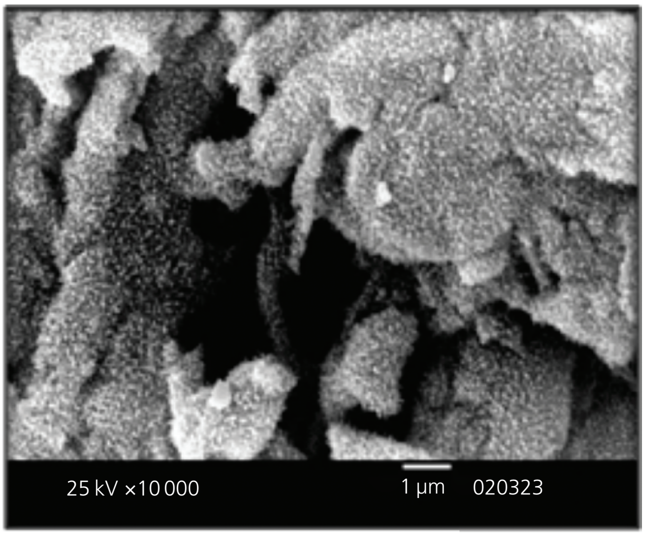
A number of factors influencing the adsorption process such as pH, nickel(II) concentration and adsorbent dose have been studied. But examining each and every factor is quite tedious and time consuming. Thus, factorial design is employed to reduce the total number of experiments in order to achieve the best overall optimisation of the process (Box *et al*., 1978; Brasil *et al*., 2005; Montgomery, 1997). The design determines the effect of each factor on response as well as how the effect of each factor varies with the change in level of the other factors (Arenas *et al*., 2006). Interaction effects of different factors could be attained using design of experiments only (Brasil *et al*., 2005; Montgomery, 1997). Greater precision in estimating the overall main factor effects and interactions of different factors can be achieved using factorial design.

Results and analysis of experiments

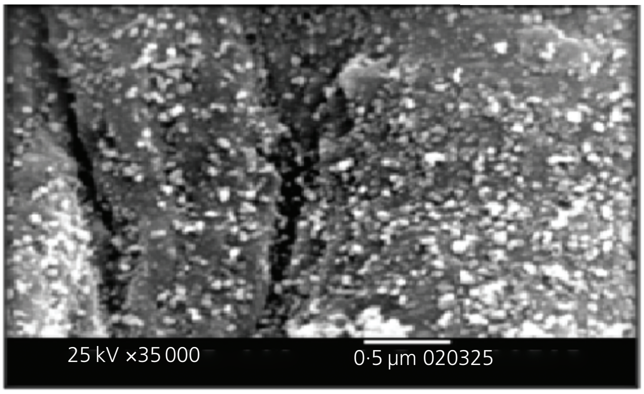
Characterisation of the adsorbent

Surface morphology

SEM images with different levels of magnification for activated carbon produced from sawdust were presented in order to show the major features of the powder morphology after treatment. Figure 1 shows the SEM for activated carbon with magnification factor 10 000 and 35 000, respectively, which investigates the total surface of treated powder as flocculated particles. By increasing the magnification factor (from 10000 to 35000), this figure shows that the adsorbent has irregular cavities, pores and rough surfaces on the



(a)



(b)

Figure 1. (a) SEM for physical activation at 700°C with magnification factor 10 000 and (b) with magnification factor 35 000

prepared carbon samples, which indicate high surface area and pore volume (Gottipati and Mishra, 2010). The particle size distribution for the activated carbon prepared by physical activation was between 8·4 and 29·2 nm.

FTIR for collected figure

The interpretation of the FTIR results on the activated carbon particle is important on account of the functional group contributions and their effect on metal sorption. The FTIR analysis permits spectrophotometric observation of activated carbon produced from sawdust in the range 500–4000 cm^{-1} and serves as a direct means for the identification of the organic function on the surface. An examination of the treated sawdust provides information regarding the surface groups that might have participated in the adsorption reaction and also indicates the surface site(s) on which adsorption can take place. In Figure 2, the FTIR spectrum of the treated sawdust (Figure 2) showed that the most prominent peaks in the spectrum originate from OH vibrations (3400–3850 cm^{-1}), and C–H asymmetric and symmetric stretching vibrations at 2935–2300 cm^{-1}). Apart from the −OH vibration and C–H stretching, the N–H group stretching as well as the C–C group and C–O groups are important aspects of the adsorbent functional groups. Very intense peaks in the 1450–1575 cm^{-1} region originate from the stretching mode of the N–H group, in the 700–1300 cm^{-1} region they may be stretching of the C–C group, while at 500–800 cm^{-1} the aromatic region may be related to the C–O group (Gottipati and Mishra, 2010).

Effect of operating condition on removal of nickel(II)

In order to establish the equilibration time for the maximum uptake of the adsorption process, the adsorption of nickel(II) using activated carbon was studied as a function of contact time.

The results showed that the removal was rapid in the initial stage and significant removal of nickel(II) ions occurred after 30 min. Figures 3 and 4 show the effect of pH on the percentage removal of

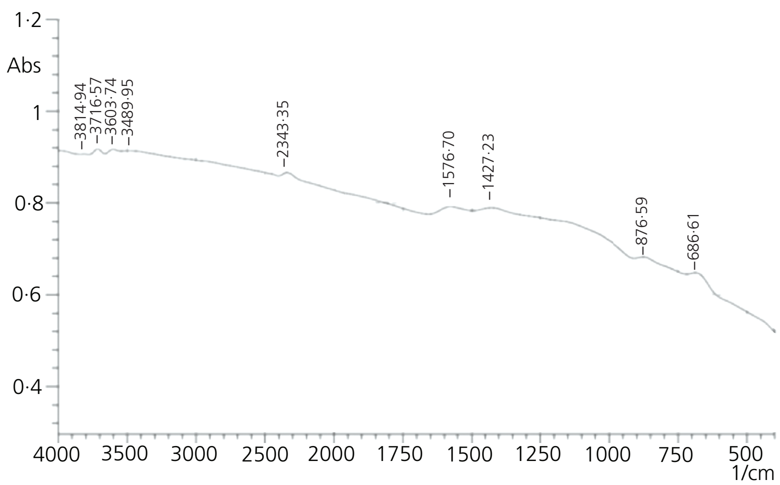


Figure 2. FTIR for activated carbon produced from sawdust

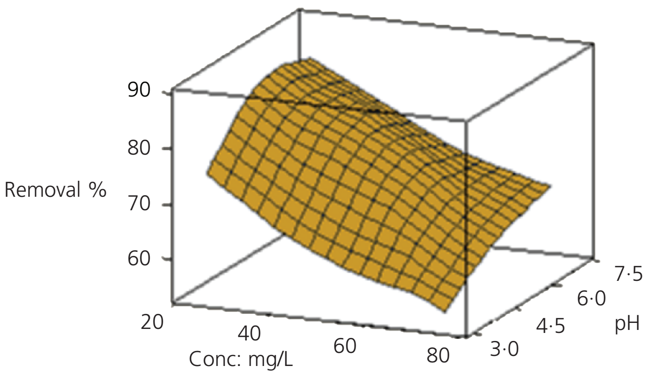


Figure 3. Effect of pH and nickel(II) concentration on the removal of nickel(II) (time 30 min, 200 rpm)

nickel(II). It can be observed that the removal efficiency increases with increasing initial pH and it almost reaches a peak value around 5 and then there is no appreciable change above the pH of 5. The removal of nickel(II) at lower pH is due to the higher H^+ ion concentration which decreases by increasing the pH and competes with the positively charged nickel ion for the adsorbent active sites resulting in reduced uptake of nickel(II). The optimal nickel(II) removal efficiency occurs at pH 5. This result is considerably consistent with the literature (Ciesielczyk *et al*., 2013; Karthika and Sekar, 2013; Thamilarasu *et al*., 2011).

Figures 4 and 5 show the effect of adsorbent dosage on the percentage removal of nickel(II). It was found that with an increase in the adsorbent dosage from 1·0 to 5·0 g/L, the removal of nickel increases from 60% to 90%. This can be explained by the fact that the greater the mass available, the greater the contact surface offered to the adsorption. These results are qualitatively in good agreement with those found in the literature (Aregawi and Mengistie, 2013).

Figures 3 and 5 show the effect of initial nickel(II) concentration on the percentage removal of nickel(II). The percentage removal of nickel ion by the adsorbents decreases with increasing nickel ion concentration and a maximum value is reached at concentrations of 25 mg/L. However, further increase in initial concentration of adsorbates cause the percentage removal to decrease. This can be explained based on the fact that all the adsorbents had a limited number of active sites, which would have become saturated above a certain metal ion concentration.

Optimisation models

Multiple linear regression modelling

A multiple linear regression (MLR) model correlates the response with the factors which have a strong effect on the performance of a process. The concentration, adsorbent dosage and pH of the solution were considered in the development of mathematical models for the nickel(II) removal rate. The correlation between factors (concentration, adsorbent dosage and pH of the solution) and nickel(II) removal rate was obtained by multiple linear regressions. The standard commercial statistical software package MINITAB was used to derive the models of the form

$$\text{Metal removal} = 75{\cdot}04 + 2{\cdot}61 * A + 1{\cdot}087 * B + 0{\cdot}935 * C \; R^2 = 0{\cdot}92 \quad (2)$$

where A is the pH of the solution, B is the weight of adsorbent (g/L) and C is nickel(II) concentration (mg/L).

Experimental design by RSM

A full factorial design, which includes all possible factor combinations in each of the factors, is a powerful tool for understanding complex processes that describe factor interactions in multifactor systems. RSM is an empirical statistical technique employed for multiple regression analysis by using quantitative data obtained from properly designed experiments to solve multivariate equations simultaneously. The experiments with different pH, adsorbent dosage and initial metal concentration were employed simultaneously covering the spectrum of variables for the removal of nickel(II) in the central composite design. In order to describe the effects of pH, adsorbent dosage and initial nickel ion concentration on percentage removal of nickel(II), batch experiments were conducted. The coded values of the process parameters were determined by the following equation.

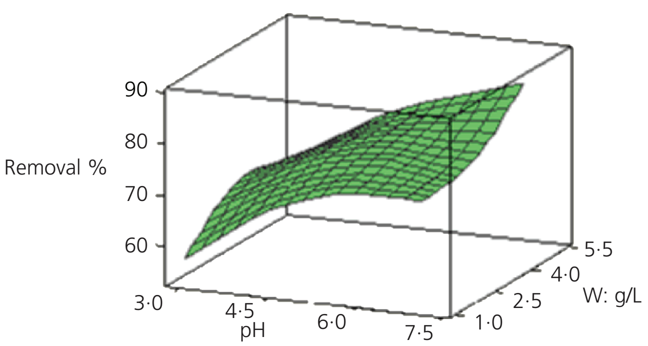


Figure 4. Effect of pH and weight of adsorbent on the removal of nickel(II) (time 30 min, 200 rpm)

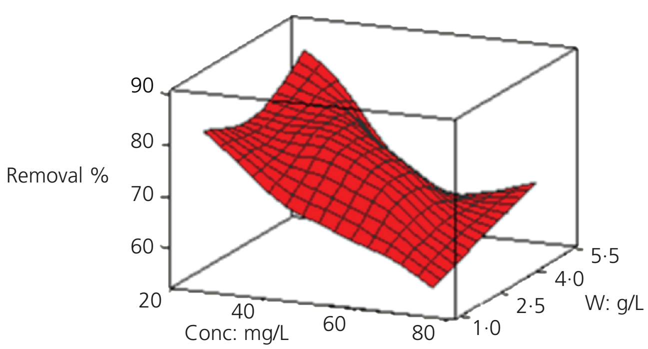


Figure 5. Effect of nickel(II) concentration and weight of adsorbent on the removal of nickel(II) (time 30 min, 200 rpm)

Independent variable	Range and level		
	–1	0	1
pH (*A*)	3	5	7
Sorbent dosage (*B*): g/L	1	3	5
Ni concentration (*C*): mg/L	25	50	75

Table 1. The experimental range and levels of independent variables

$$x_i = \frac{X_i - X_0}{\Delta X} \quad (3)$$

where x_i is the coded value of the *i*th variable, X_i is the uncoded value of the *i*th test variable and X_0 is the uncoded value of the *i*th test variable at the centre point. The experimental range and levels of independent variables are given in Table 1. The regression analysis was performed to estimate the response function as a second-order polynomial.

$$\text{Metal removal} = \beta_0 + \sum_{i=1}^{k} X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1}\sum_{j=1}^{k} \beta_{ij} X_i X_j \quad (4)$$

where *Y* is the predicted response, β_i, β_j, β_{ij} are coefficients estimated from the regression; they represent the linear, quadratic and cross products of X_1, X_2 and X_3 on response. A statistical program package, Minitab 14, was used for regression analysis of the data obtained and to estimate the coefficient of the regression equation. The equations were validated using the analysis of variance (Anova). Anova is a collection of statistical models used to analyse the differences between group means and their associated procedures (such as 'variation' among and between groups) (Gottipati and Mishra, 2010). The significance of each term in the equation is to estimate the goodness of fit in each case. The goodness of fit is examined by using the determination coefficient (R^2). The coefficient of determination (R^2) was calculated to be 0·983.

The experimental results are analysed through RSM to obtain an empirical model for the best response. The mathematical expression of the relationship to the response with variables is shown below.

$$\text{Metal removal} = 45{\cdot}18 + 15{\cdot}7366 * A + 4{\cdot}1257 * B - 0{\cdot}5164 * C - 0{\cdot}1187 * A * B - 0{\cdot}0004 * A * C - 0{\cdot}0053 * B * C - 1{\cdot}2957 * A^2 - 0{\cdot}3975 * B^2 + 0{\cdot}0013 * C^2 R^2 = 0{\cdot}983 \quad (5)$$

The results of multiple linear regressions conducted for the second-order response surface model are given in Table 2. The significance of each coefficient was determined by Student *t* test and *p* values, which are listed in Table 2. The larger the magnitude of the *t* value and smaller the *p* value, the more significant is the corresponding coefficient. In this case, *A*, *B*, *C*, A^2 and B^2 are significant model terms for the sorption of nickel. Values greater than 0·10 indicate that the model terms are not significant. This implies that the linear effects of pH, sorbent dosage and nickel ion concentration are more significant factors.

Predictor	Coefficient	*t* value	*p* value
Constant	45·18	3·289	0·022
A	15·736	4·132	0·009
B	4·1257	1·361	0·023
C	–0·5164	1·955	0·011
*A***B*	–0·1187	0·367	0·729
*A***C*	–0·0004	0·017	0·987
*B***C*	–0·0053	0·240	0·820
A^2	–1·2957	3·951	0·011
B^2	–0·3975	1·212	0·028
C^2	0·0013	0·589	0·582

Table 2. Anova for response surface quadratic model

From the statistical results of RSM and MLR predictions shown in Figure 6, it can be concluded that the RSM model is more accurate in predicting the percentage removal of nickel(II) than MLR. This might be due to the large amount of data required for developing a sustainable regression model, while the response surface could recognise the relationships with less data for distributed and parallel computing natures. A second reason is the effect of the predictors on the dependent variable, which may not be linear in nature. In other words, the RSM model could probably predict the percentage removal of nickel(II) with a better performance owing to their

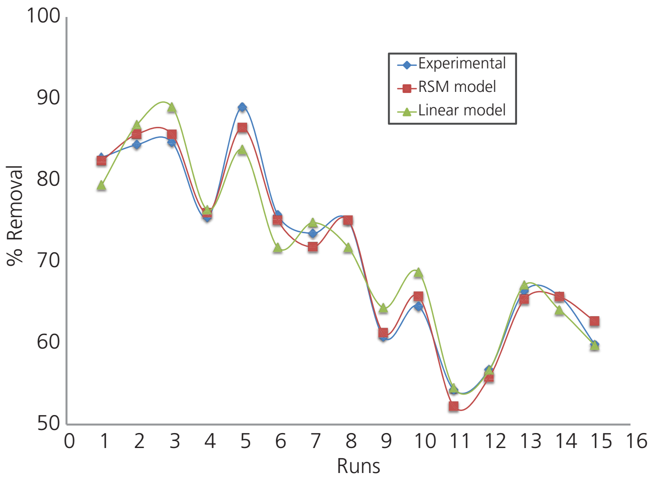


Figure 6. Comparison between experimental, regression, and RSM results

greater flexibility and capability to model nonlinear relationships. Therefore, in the case of data sets with a limited number of observations in which regression models fail to perform reliably, advanced soft computing approaches like RSM may be preferred (Patel and Brahmbhatt, 2015).

Conclusion

Activated carbon has been produced by physical activation of sawdust, the produced activated carbon has been characterised and used as adsorbent for removal of nickel(II) from aqueous solution. Two models used to optimise the operating conditions that affect the nickel(II) removal from aqueous solution using activated carbon prepared by physical activation of sawdust as adsorbent have been developed. The nickel(II) removal by adsorption using activated carbon produced from sawdust can be simulated using RSM and linear regression and the RSM model predicted the experimental results with relatively higher accuracy (R^2 = 0·985). The results showed that the RSM model is more accurate in predicting the percentage removal of nickel(II) than MLR.

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