

Process optimization for Zn (II) removal by activated carbon prepared from rubber seed coat using chemical activation

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Abstract

The purpose of this research is to obtain optimal activation conditions for activated carbon prepared from rubber seed coat (RSCAC) using response surface methodology (RSM). Central composite design (CCD) was used to determine the properties of the three preparation variables; activation temperature, activation time and $ZnCl_2$: char impregnation ratio (IR) on RSCAC yield and percentage of Zn (II) removal. Based on the CCD, two quadratic models were developed for both responses. The most influential factor on each experimental design response was identified from the analysis of variance (ANOVA). The optimum conditions obtained for preparing the RSCAC were activation temperature of 797°C, activation time 1h and IR of 2.61 resulting in 22.90 % RSCAC yield and 84.12 % Zn (II) removal.

Keywords: Activated carbon, Central composite design, Optimization, Rubber seed coat, Zinc (II) removal

1. Introduction

The revolution towards industrially based economy has changed the lifestyle of human, also the urbanization of cities. However, the industrialization has brought serious effects to the source of life; water. Variety of toxic and harmful matters that brings bad impacts to the water environment is discharged by industrial activities. Many metals are toxic and hazardous to human and environment and most of the metals are drained into the water bodies and natural environment in high level of concentrations that affect human health (Kazemipour et al., 2008). According to Chuah et al. (2005), chromium, cobalt, nickel, and zinc are significantly discovered in the receiving water bodies from textile mill products, mining, tanneries, petrochemical, electroplating and electronics projects. (Kazemipour et al., 2008). Therefore, it is proven that industrial waste is the primary source of heavy metal contamination in natural water bodies. The key heavy metals such as zinc (Zn), copper (Cu), cadmium (Cd) and lead (Pb) pollutes the water stream via wastewater as

reported by Dhakal et al. (2005). Therefore, several method has been used for the elimination and retrieval of heavy metals from wastewaters such as precipitation, ion exchange, and electrolysis (Doan et al., 2008). Activated carbon (AC) adsorbents are widely applied in wastewater treatment due to its large mesopore volumes and surface area. The production expenses of commercially activated carbons are enough high. Therefore, activated carbon adsorption has been established to be advanced contrasted to other methods in the concept of its ease of design, simple operation, and high efficiency .From the literature, certain agrowaste such as tree fern (Ngah et al., 2008), sawdust (Prakash Kumar et al., 2006), rice bran (Ip et al., 2008), wheat bran (Hameed et al., 2008), corncobs (Sun and Webley, 2010) have been broadly studies of the elimination of heavy metals from the aqueous solution. In this work, an attempt was made in preparing activated carbon from rubber seed coat. By utilizing rubber seed coat into activated carbon will reduce the cost of waste disposal and also converted this waste into value-added product. Currently no study has been done on optimization of the RSC preparation conditions for Zn (II) removal using the response surface methodology (RSM) approach. The effects of preparation variables; activation temperature and activation time were studied simultaneously to give a high RSCAC yield and high Zn (II) percentage removal from aqueous solution.

2. Materials and Methods

2.1 Materials

Rubber seed coat (RSC) as precursor was obtained locally in the location of Bukit Mertajam, Penang, Malaysia. Stock solutions of Zn (II) was prepared by

Zn(NO₃)₂.6H₂O (Merck, 99% purity) Deionized water was used to prepare all solutions.

2.2 Preparation of RSCAC

RSC had been cleaned in detail to do away with dirt and inorganic matters on their surfaces. The pre-treated precursors had been then dried in an oven (Model Memmert 600, Germany) at a temperature of 105 °C for 24 h to remove all of the moisture contents. The dried precursors have been ground into compact pieces and were sieved to the size of 1-2 mm. 30 g of RSC was positioned within a vertical tubular reactor that was placed in the furnace. Nitrogen gas was applied as the inert gas purging by means of the furnace. The flow frequency of nitrogen gas and the heating rate was kept constant at 150 cm³/min and 10 °C /min, correspondingly. The temperature was ramped from room temperature to 700 °C and kept for 2 hours. At several impregnation ratios (IR), the chars produced were impregnated using potassium hydroxide (KOH). The impregnation ratio was measured as:

$$IR = \frac{w_{KOH}}{w_{char}} \quad (1)$$

Where w_{KOH} is the dry weight (g) of potassium hydroxide pellets and w_{char} is the dry weight (g) of char. A definite amount of char and KOH pellets (hinge on the IR) were mix up with deionized water by using 250 mL beaker. At stable temperature and speed rate, the mixture was stirred and then the beakers were immersed in water bath shaker. The mixing was performed at 30 °C for 3 h at 100 rpm to make sure that the KOH pellets were completely dissolved. Then, the beaker of was put in the oven (Model Memmert 600, Germany) for overnight at temperature 105 °C for dehydrating purpose, leaving only KOH on the samples.

2.3 Adsorption studies

200 mL of adsorbate options with identified initial concentration of 10-100 mg/L for Zn(II)) were formed a series of 250 mL Erlenmeyer flasks. The quantity of adsorbent that was inserted into every single flask containing Zn (II) remedy was fixed at 0.2 g. The flasks had been concealed with aluminium foil, plus the flasks had been then stored in an isothermal water bath shaker (Model Protech, Malaysia) at continuous temperature (30 °C), with rotation speed of 120 rpm, until an equilibrium point was accomplished. The percentage removal at equilibrium was measured in the Equation 2 as follows.

$$Removal(\%) = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

Where C_o and C_e are the liquid-phase concentrations of adsorbate at the initial condition, and equilibrium (Zn(II)/L) (Ayranci and Hoda, 2004; Pavlovic et al., 2005)

2.4 Activated carbon yield

Activated carbon yield was measured by the equation as follows:

$$Yield(\%) = \frac{w_c}{w_o} \times 100 \quad (3)$$

where w_c is the dry weight of final activated carbons and w_o is the dry weight of precursors.

2.5 Design of experiments

In this work, a standard RSM design called CCD was applied to study the variables for preparing the activated carbon from rubber seed coat (RSCAC).

The CCD consists of three variables, involving 8 factorial points, 6 axial points and 6 replicates at the center points. The variables studied are activation temperature (x_1), activation time (x_2) and impregnation ratio (IR) (x_3) indicating that altogether 20 experiments for this procedure as calculated from equation 4 :

$$N = 2^n + 2n + n_c = 2^3 + 2*3 + 6 = 20 \quad (4)$$

Where N is the total number of experiments involved, and n is the number of factors. These three variables together with their respective ranges were chosen based on literature and preliminary studies are reported in Table 1.

Table 1: Variables together with their respective ranges

Variables (factors)	Coded variable levels				
	$-\alpha$	-1	0	+1	$+\alpha$
Activation temperature	648	700.00	775.00	850.00	901
Activation time	0.32	1.00	2.00	3.00	3.68
Impregnation ratio (IR)	0.15	1.00	2.25	3.50	4.35

The centre points are applied to figuring the experimental mistake, as well as the reproducibility of the information. The axial points are situated at ($\pm a$, 0, 0), (0, $\pm a$, 0) and (0,

0, $\pm a$) where a act as the distance of the axial point in the center and creates rotatable style (Tan et al., 2009).

The experimental order was randomized with the objective to minimize the results from the uncontrolled aspects. Zn(II) removal (Y_1) and activated carbon yield (Y_3) act because the three responses. Every in the response was applied to type an empirical model which correlated the response towards the three activated carbon preparation variables by the use of a second-degree polynomial equation as shown in Equation 5 (Zainudin et al., 2005):

$$Y = b_o + \sum_{i=1}^n b_i x_i + \left(\sum_{i=1}^n b_{ii} x_i \right)^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij} x_i x_j \quad (5)$$

Where Y is the predicted response, b_o the constant coefficient, b_i the linear coefficients, b_{ij} the interaction coefficients, b_{ii} the quadratic coefficients and x_i, x_j are the coded values of the activated carbon preparation variables.

2.6 Model fitting and statistical analysis

The experimental data were examined by statistical software Design Expert Version 7.0.0 (STAT-EASE Inc., Minneapolis, USA) for regression evaluation to fit the second-degree polynomial equation and as well as for the assessment of the statistical significance of the equations generate.

2.7 Characterization of optimized RSC

The surface morphology of the samples was examined using a scanning electron microscope (JEOL, JSM-6460 LV, Japan). Proximate analysis was carried out using thermogravimetric analyser (Perkin Elmer TGA7, USA) and elemental analysis was performed using Elemental Analyzer (Perkin Elmer Series II 2400, USA). The surface area, pore volume and average pore diameter of the RHAC were determined by nitrogen adsorption isotherm at 77 K using Micromeritics ASAP 2020 volumetric adsorption analyzer. The surface area of the sample was determined using Brunauer-Emmett-Teller (BET) equation. The total pore volume was estimated to be the liquid volume of nitrogen at a relative pressure of 0.98. To decide the pore size distribution, Barret-Joyner-Halenda (BJH) model was utilized.

3. Results and Discussion

3.1 Development of regression model equation

Table 2 shows the complete design matrixes together with both the response values obtained from the experimental work. The table also comes together with the three response values gained in the experimental performs. Run 15-20 inside the centre point had been tested for the reproducibility of the results. In line with the sequential model sum of squares, the models had been chosen primarily based on the highest order polynomials exactly where the additional terms were necessary, along with the models were not aliased. For responses to Zn(II) removal (Y_1) and RSCAC yield (Y_3) the software program had proposed the quadratic models. The last empirical formula models for the Zn(II) removal (Y_1) and RSCAC yield (Y_3) in relations of coded aspects are represented by Equation 6 and 7 respectively.

$$Y_1 = 94.8 + 4.74 x_1 + 0.55 x_2 + 7.26 x_3 - 0.25 x_1 x_2 + 0.23 x_1 x_3 - 0.016 x_2 x_3 - 2.54 x_1^2 - 0.035 x_2^2 - 5.51 x_3^2 \quad (6)$$

$$Y_3 = 22.51 - 2.21 x_1 - 1.54 x_2 - 1.38 x_3 - 1.58 x_1 x_2 + 1.15 x_1 x_3 + 0.25 x_2 x_3 - 1.19 x_1^2 + 0.32 x_2^2 - 0.41 x_3^2 \quad (7)$$

Table 2 Experimental design matrix for preparation of RSCAC

R u n	Level			RSCAC Preparation Variable			Zn(II) removal, Y_1 (%)	RSCAC yield, Y_3 (%)
				Activat ion temper ature, X_1 (°C)	Activ ation time, X_2 (h)	IR , X_3		
1	-1	-1	-1	700	1	1	66.7	24.5 4
2	1	-1	-1	850	1	1	75.2	22.2 1
3	-1	+1	-1	700	3	1	68.4 4	25.1 1
4	+1	+1	-1	850	3	1	75.3	14.2 1
5	-1	-1	+1	700	1	3.	78.4	21.0

					5		2
6	+1	-1	+1	850	1	3. 5	21.0 3
7	-1	+1	+1	700	3	3. 5	20.3 6
8	+1	+1	+1	850	3	3. 5	88.9 16.3
9	-1.6 82	0	0	648	2	2. 25	66.3 23.9
10	+1. 682	0	0	901	2	2. 25	84.3 16.2
11	-1.6 82	0	0	775	0.32	2. 25	82.2 26.7 3
12	+1. 682	0	0	775	3.68	2. 25	84.9 21.8 7
13	-1.6 82	0	0	775	2	0. 15	52.2 25.6 7
14	+1. 682	0	0	775	2	4. 35	85.9 18.8 5
15	0	0	0	775	2	2. 25	83.4 22.3 2
16	0	0	0	775	2	2. 25	83.1 22.8 1
17	0	0	0	775	2	2. 25	83.9 21.5
18	0	0	0	775	2	2. 25	84.4 23.0 5
19	0	0	0	775	2	2. 25	82.7 22.8 7
20	0	0	0	775	2	2. 25	85.2 22.2

The value of the models established was assessed based on the correlation coefficient, R^2 . In this experiment, the R^2 values for Equations 6 and 7 were respectively 0.9239 and 0.9166 which indicated that 92.39 and 91.66% of the total variation in the Zn(II) removal and RSCAC yield, respectively were attributed to the experimental variables studied. The R^2 values of equations 6 and 7 were deemed moderately high as the values reveal that the Zn(II) removal and RSCAC yield were closer and precise between model and actual values.

3.2 Analysis of variance

The validity of the models were further justified through analysis of variance (ANOVA), the results are presented in Tables 3 and 4. F-value from the model implied the significance of the model similarly, the value of Prob > F less than 0.05 indicated that the model terms were significant. Table 3 reports the ANOVA values

for RHAC yield. The model F-value of 12.2 and Prob. > F of 0.0003 implied that this model was significant. x_1 , x_2 , x_3 , x_1^2 and x_3^2 were significant model terms whereas x_2^2 , x_1x_2 , x_1x_3 , and x_2x_3 were all insignificant to the response. From the statistical results obtained, it was shown that the above models (Equation 6-7) were adequate to predict the Zn(II) removal and the RSCAC yield within the range of variables studied.

TABLE 3 Analysis of variance (ANOVA) for response surface quadratic model for RSCAC yield

Source	Sum of squares	Degree of freedom (DF)	Mean square	F-Value	Prob. > F	Comment
Model	180.78	9	20.09	12.20	0.0003	significant
x_1	66.91	1	66.91	40.65	0.0001	<
x_2	32.27	1	32.27	19.61	0.0013	
x_3	25.96	1	25.96	15.77	0.0026	
x_1x_2	19.97	1	19.97	12.13	0.0059	
x_1x_3	10.53	1	10.53	6.40	0.0299	
x_2x_3	0.52	1	0.52	0.32	0.5864	
x_1^2	20.31	1	20.31	12.34	0.0056	
x_2^2	1.43	1	1.43	0.87	0.3725	
x_3^2	2.372	1	2.372	1.441	0.2576	

The ANOVA for the quadratic model for Zn(II) removal of RSCAC is listed in Table 4. The model F-value of 22.61 and Prob. > F less than 0.0001 implied that this model was significant. x_1 , x_3 , x_1^2 , x_3^2 were significant model terms whereas x_2 , x_2^2 and interaction terms x_1x_2 , x_1x_3 , x_2x_3 , were insignificant to the response.

TABLE 4 Analysis of variance (ANOVA) for response surface quadratic model for Zn(II) removal of RSCAC

Source	Sum of squares	Degree of freedom (DF)	Mean square	F-Value	Prob. > F	Comment
Model	1560.1	9	173.35	22.61	1 < 0.0001	significant
x_1	345.92	1	345.92	45.12	1 < 0.0001	
x_2	1.44	1	1.44	0.19	1 < 0.674	
x_3	785.70	1	785.70	102.4	1 <	

			9	0.000
			1	0.954
x_1x_2	0.03	1	0.03	0.00
x_1x_3	7.49	1	7.49	0.98
x_2x_3	1.94	1	1.94	0.25
x_1^2	100.09	1	100.09	13.06
x_2^2	1.27	1	1.27	0.17
x_3^2	336.09	3	336.09	43.84
	3	1	3	0
				0.000
				1

3.3 Activated carbon yield and Zn (II) removal

Figure 1 (a) illustrates the effect of activation temperature and activation time around the RSC activated carbon yield, with IR fixed at zero level (IR = 2.25), whereas Figure 1 (b) illustrate the impact of activation temperature and IR on the response, with activation time fixed at zero level ($t = 2$ h). For RSCAC yield, all variables were identified to be significant in the response, with activation temperature imposing the greatest impact followed by activation time and IR. It was discovered in that the yield of RSCAC decreased with increased in activation temperature and activation time as shown in Figure 1 (a) consequence volatiles and moisture released from the sample at such conditions (Guo et al., 2009). Hayashi et al. (2002) has also found that the yield for the preparation of chickpea activated carbon decreases as the activation temperature increase. In addition, Ahmad et al. (2009) also obtained the results showing that the rattan sawdust activated carbon yield decreases with an increase in the activation temperature, activation time, and IR.

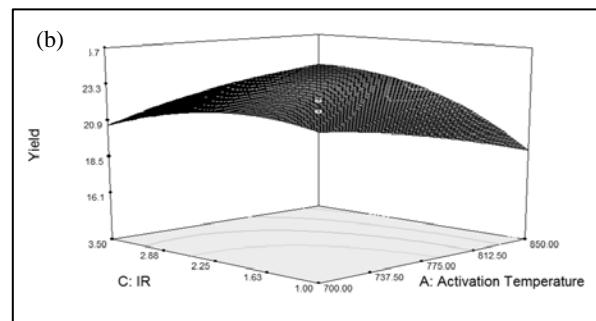
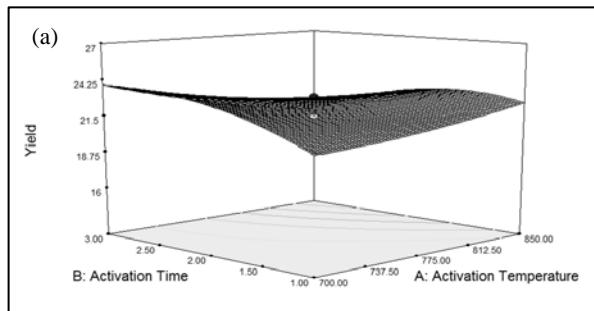


Figure 1 Three-dimensional response surface plot of RSCAC yield (a) Effect of activation temperature and activation time, IR = 2.25; (b) Effect of activation temperature and IR, $t = 2$ h

Based on the F-value shown in Table 4, activation temperature and impregnation ratio (IR) have been identified to have considerable effects on the Zn(II) removal of by RSCAC, whereas activation time show the least significant effect on this response. IR was located to impose the greatest impact on the Zn(II) removal demonstrated by the highest F-value of 785.7. In addition, the quadratic effects of activation temperature and IR were also high. Figure 2 shows the three-dimensional response surfaces that were constructed to show the interaction effects of the activation temperature and IR on the Zn(II) removal by RSCAC. For this plot, the activation time was fixed at zero level ($t = 2$ h). From Figure 4.5, the Zn(II) removal elevated within the two variables studied. The escalating of activation temperature and IR at enough activation time may lead to the improvement of pore structure as well as produced new porosities on the RSCAC's surface suitable to adsorb Zn(II) molecule.

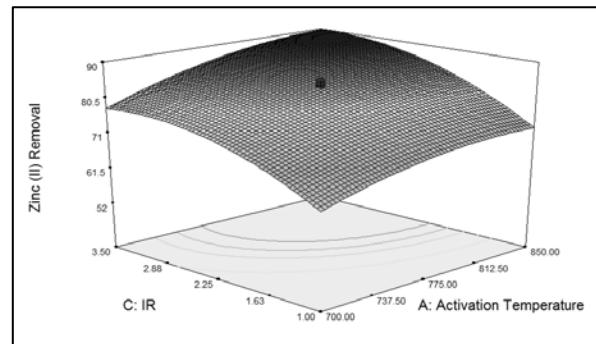


Figure 2 Three-dimensional response surface plot of Zn(II) removal (Effect of activation temperature and IR, $t = 2$ h) of RSCAC

3.4 Process optimization

One of the main aims of this study was to find the optimum process parameters which activated carbons produced should have a high RSC yield and a high Zn(II) removal. However, it is difficult to optimize both these responses under the same condition because the interest region of factors is different. When adsorption performance increases, carbon yield will decrease and vice versa. Therefore, the function of desirability was applied using Design-Expert software in order to compromise between these two responses. In the optimization analysis, the target criteria was set as maximum values for the two responses of RSC yield and Zn(II) removal while the values of the three variables were set in the ranges being studied. The experimental conditions with the highest desirability were selected to be verified. The predicted and experimental results of RSC yield and Zn(II) removal obtained at optimum conditions are listed in Table 5.

Table 5 Model validation for activated carbons prepared for Zn(II) removal

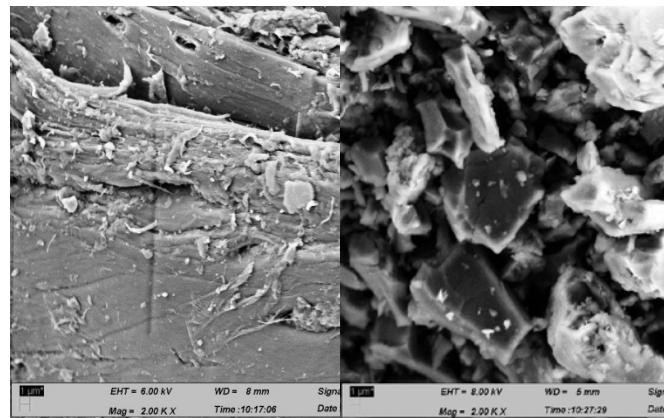
Activ ate d carb on	Mod el desir abilit y	Activ ation tempe ratur e, x_1 (°C)	Acti vati on time , x_2 (h)	Zn(II) removal (%)		Activated yield (%)		carbon		
				I R , x_3	Pre dict ed	Exper iment al	Er ro (%)	Pre dict ed	Exper iment al	Er ro (%)
RSC	0.846	797	1.0	2. 6 1	86.9 5 1	84.12 25	3. 7	23.6 22.90	3. 27	
-ZN										
CC- ZN	0.922	768	3.0	3. 5	71.2 3	69.00 13	3. 4	43.8 41.92	4. 39	

Table 5 listed the experimental conditions used to prepare the optimum RSCAC for Zn(II) removal. It was observed that the experimental values obtained were in good agreement with the values predicted by the models. This indicated that the model was suitable and sufficient to predict the responses of the operating variables fixed.

3.5 Activated carbon characterization

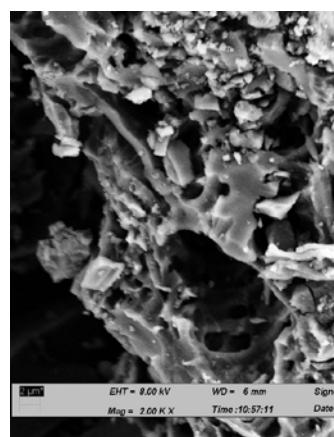
The surface morphology of the RSC precursor together with its respective chars and activated carbons prepared were examined using scanning electron microscopy (SEM). From the SEM micrographs obtained, the surface structure and pore development in the samples could be clearly seen. Plate 1(a)-(c) respectively show the SEM micrographs of the RSC precursor, RSC char, RSC-ZN. The SEM micrographs from Plate 4.1 represent the

morphological changes of the carbon materials during carbonization and activation. The precursor's surface textures were rough, uneven, undulating and very little pores were presence as observed in another biomass (Ahmad et al., 2009). After carbonization process, some irregular holes and pores were developed and found on the surfaces of the chars due to the sudden burst of the thermal expansion from pyrolysis. Pore development in the char during pyrolysis was important as this would enhance the surface area and pore volume of the activated carbon after the activation process used, which involved both chemical and physical activating agents of KOH and CO₂ (Tan et al., 2008).



(a)

(b)



(c)

Plate 1 (a)RSC precursor, (b)RSC char, (c)RSC-ZN

It can be seen that almost homogeneous type pores structure were distributed on the surface of the RSCAC in Plate.1 (c). Large and well-developed pores were clearly found on the surface of activated carbons, compared to the

original precursor and the chars as well. Therefore, the SEM micrographs revealed that the combination activation process of KOH and CO₂ were effective in creating well-developed pores on the surface of RSC. resulting to large surface area activated carbon with good mesoporous structure. This result is consistent with the results obtained earlier for the high mesopores surface area and the average pore diameter which in the mesopore range for both RSCAC. Similar observations were reported by other researchers in their works of preparing activated carbons from oil palm fronds (Salman and Hameed, 2010) , coconut shell (Mohd Din et al., 2009) and oil palm empty fruit bunch (Tan et al., 2009).

4. Conclusions

Rubber seed coat (RSC) selected agrowaste precursors were successfully used for preparation of activated carbons using physiochemical activation method consisting of potassium hydroxide (KOH) impregnation with carbon dioxide (CO₂) gasification. Activated carbons prepared were mesoporous in nature with a relatively high mesopore surface area with average pore diameters larger than 2.2 nm. The particle size of RSCAC was 0.12. Adsorptions of Zn (II) on all the RSC-ZN were best fitted by the. The activated carbons prepared showed adsorption capacities of 46.08–63.69 mg/g for adsorption of Zn (II) at 30 °C. Adsorptions of Zn (II) on all the RSC-ZN were best fitted by the Langmuir isotherm and best represented by the pseudo-second-order kinetic model. The film-diffusion-controlled mechanism mainly governed all the adsorption processes.

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