

Effect of preparation conditions of activated carbon prepared from rubber seed coat by KOH activation for removal of Cu (II) from aqueous solution

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Abstract

Rubber seed coat based activated carbon (RSC) was prepared by physiochemical activation consisting of potassium hydroxide (KOH) impregnation plus carbon dioxide (CO₂) gasification. The effects of adsorbate initial concentration (10-100 mg/L), contact time, solution temperature (30-60 °C), solution pH (2-6), isotherms and kinetics of the adsorption systems were evaluated through batch adsorption test. Adsorptions of Cu(II) on all the activated carbons prepared were best represented by the pseudosecond-order kinetic model. From the analysis of variance (ANOVA), the significant factors on each experimental design response were identified. The Cu(II) adsorption uptakes increased with increasing initial concentration and contact time.

Keywords: Activated Carbon, Cu (II) Removal, Optimization, Rubber Seed Coat

1. Introduction

Due to the large quantities of water used by the industry such as metal finishing, electronics, tanneries, electroplating, petrochemical and textile mill, heavy metals contamination has become one of the largest problem which demands a smart solution. Several methods are 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 broadly applied in wastewater treatment. Its benefits originate mostly from its mesopore volumes and large surface area. The production expenses of commercially activated carbons are high enough. 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 rubber seed coat based activated carbon (RSCAC). By utilizing rubber seed coat into activated carbon will decrease the cost of waste disposal and also converted this waste into a value-added product. Currently, no study has been done on optimization of the RSC preparation conditions for Cu (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 Cu (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 Cu (II) was prepared by $\text{Cu(NO}_3)_2.3\text{H}_2\text{O}$ (Merck, 99% purity) in deionised distilled water. Deionized water was used to prepare all solutions.

2.2 Preparation of activated carbon

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:

$$IR = \frac{w_{KOH}}{w_{char}} \tag{1}$$

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 3h 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 Cu(II) were formed a series of 250 mL Erlenmeyer flasks. The quantity of adsorbent that was inserted into every single flask containing Cu(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 oC), with rotation speed of 120 rpm, until an equilibrium point was accomplished. The percentage removal of adsorbate was then evaluated as:

$$q_e = \frac{(C_o - C_e)V}{W} \tag{2}$$

where $C_{\rm o}$ and $C_{\rm e}$ are the liquid-phase Cu (II) concentrations at the initial state and equilibrium (mg/l), respectively

2.4 Activated carbon yield

Activated carbon yield was measured as the dry weight of final activated carbons, w_c to the dry weight of precursors, w_o as stated by the equation as follows:

$$Yield(\%) = \frac{w_c}{w_0} x 100 \tag{3}$$

2.5 Design of experiment

The RSCAC was prepared using physiochemical activation method. The variables studied are activation temperature (xI), activation time (x2) and impregnation ratio (IR) (x3). 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	Coded variable levels						
(factors)	$-\alpha$	-1	0	+1	$+\alpha$		
Activation	648	700	775	850	901		
temperature							
Activation	0.32	1.00	2.00	3.00	3.68		
time							
Impregnation	0.15	1.00	2.25	3.50	4.35		
ratio (IR)							

Response surface methodology (RSM) design known as a central composite design (CCD) was used up to analyze the parameter of making activated carbons from RSC. 23 complete factorial CCD for the three variables, involving 8 factorial points, 6 axial points and 6 replicates at the center points were utilised, signifying that general 20 experiments for this process for each and every precursor, as measured from Equation 4 (Azargohar and Dalai, 2005):

$$N = 2^{n} + 2n + n_{c} = 2^{3} + 2^{*}3 + 6 = 20$$
 (4)

where N is the total number of experiments involved, and n is the number of factors.

As outlined by (Tan et al., 2009), the center 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 an act as the distance of the axial point in the center and creates rotatable style. From these findings, the value was fixed at 1.682 (rotatable). The experimental order was randomized with the objective to minimize the results from the uncontrolled aspects. Cu(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 preparation



variables by the use of a second-degree polynomial equation as shown by (Zainudin et al., 2005):

$$Y =$$

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

where Y is the expected 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 analyzer (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 RSCAC were determined by nitrogen adsorption isotherm at 77 K using Micromeritics ASAP 2020 volumetric adsorption analyzer. The surface area of the sample was Brunauer-Emmett-Teller determined using (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 center 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 Cu(II) removal (Y_2) and RSCAC yield (Y_3) , the software program had proposed the quadratic models. The last empirical formula models for the Cu(II) removal (Y_1) and RSCAC yield (Y_3) in relations of coded aspects are represented by Equation 6 and 7 respectively.

$$Y_2 = 83.74 + 5.03 x_1 + 0.32 x_2 + 7.58 x_3 + 0.058 x_1 x_2 + 0.97 x_1 x_3 - 0.49 x_2 x_3 - 2.64 x_1^2 + 0.3 x_2^2 - 4.83 x_3^2$$
 (6)

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

Table 2 Experimental design matrix for preparation of RSCAC

					C Preparatio			
					z Preparado 'ariable	11		DOG
					апавіе	1	Cu(II)	RSC
Ru				Activatio	Activati		remov	AC
n		Level		n	on	IR,	al, Y ₁	yield,
			temperat	time,	X ₃	(%)	Y ₃	
			ure, X ₁	X ₂ (h)	21.3	(/0)	(%)	
				(°C)	A ₂ (II)			
1	-1	-1	-1	700	1	1	77.9	24.54
2	1	-1	-1	850	1	1	87.23	22.21
3	-1	+1	-1	700	3	1	79.3	25.11
4	+1	+1	-1	850	3	1	86.3	14.21
5	-1	-1	+1	700	1	3.5	88.2	21.02
6	+1	-1	+1	850	1	3.5	97.1	21.03
7	-1	+1	+1	700	3	3.5	88.2	20.36
8	+1	+1	+1	850	3	3.5	97.44	16.3
9	-							
	1.68					2.2		
	2	0	0	648	2	5	77.23	23.9
10	+1.6					2.2		
	82	0	0	901	2	5	95.2	16.2
11		-						
		1.68				2.2		
	0	2	0	775	0.32	5	91.3	26.73
12		+1.6				2.2		
	0	82	0	775	3.68	5	95.3	21.87
13			-					
			1.68			0.1		
	0	0	2	775	2	5	60.32	25.67
14			+1.6			4.3		
	0	0	82	775	2	5	95.33	18.85
15						2.2		
	0	0	0	775	2	5	94.2	22.32
16						2.2		
	0	0	0	775	2	5	95.63	22.81
17						2.2		
	0	0	0	775	2	5	94.8	21.5
18						2.2		
	0	0	0	775	2	5	95.3	23.05
19						2.2		
	0	0	0	775	2	5	94.02	22.87
20						2.2		
	0	0	0	775	2	5	95.33	22.2

The value of the models established was assessed based on the correlation coefficient, R^2 . The models developed appears to be the best at higher R^2 value (closer to unity) since it will give predicted worth closer towards the actual quality of the responses (Ahmad et al., 2009; Sahu et al., 2009). In this experiment, the R^2 values for Equations 6 and 7 were respectively 0.9239 and 0.9166 which indicated that 92.39and 91.66% of the total variation in the

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Cu(II) removal and RSCAC yield, respectively were attributed to the experimental variables studied. The standard deviations for the two models were 3.55 and 1.28 for Equations 6 and 7 respectively. The R² values of 0.9239 and 0.9166 for Equation 6 and 7 respectively were deemed moderately high. The values reveal that the Cu(II) removal and RSCAC yield were closer and precise between model and actual values.

3.2 Analysis of variance

Inside the ANOVA, the mean squares were obtained by dividing the sum from the squares of each and every of the variation sources, the model along with the error variance, by the respective degrees of freedom. If the value of Prob. > F significantly less than 0.05, the model terms are thought of as significant (Sahu et al., 2010). The ANOVA for the quadratic model for Cu(II) removal of RSCAC is listed in Table 3. The model F-value of 13.5 and Prob > F of 0.0002 implied that this model was substantial. In this case, the activation temperature (x_I) , IR (x_3) , x_I^2 and x_3^2 were significant model terms whereas activation time (x_2) along with the quadratic (x_2^2) and interaction terms (x_Ix_2, x_Ix_3, x_2x_3) were insignificant to the response.

Table 3 Analysis of variance (ANOVA) for response surface quadratic model for Cu(II) removal of RSCAC

Source	Sum of	Degree of	Mean	F-	Prob. >	Comment
	squares	freedom	square	Value	F	
		(DF)				
Model	1532.27	9	170.25	13.50	0.0002	significant
x_I	306.44	1	306.44	24.29	0.0006	
x_2	4.16	1	4.16	0.33	0.5785	
x_3	718.96	1	718.96	56.99	< 0.0001	
x_1x_2	0.50	1	0.50	0.04	0.8469	
x_1x_3	0.41	1	0.41	0.03	0.8606	
x_2x_3	0.00	1	0.00	0.00	0.9899	
x_{I}^{2}	92.98	1	92.98	7.37	0.0218	
x_{2}^{2}	0.02	1	0.02	0.00	0.9708	
x_3^2	436.9493	1	436.949	34.636	0.0002	

From the ANOVA for response surface, the quadratic model for RSCAC yield listed in Table 4, 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 Cu(II) removal and the RSCAC yield within the range of variables studied.

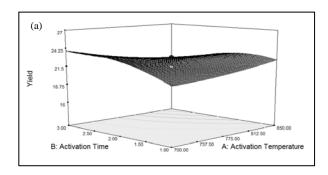
TABLE 4 Analysis of variance (ANOVA) for response surface quadratic model for RSCAC yield

Source	Sum of squares	Degree of	Mean square	F-Value	Prob. > F	Comment
	-	freedom	-			

		(DF)				
Model	180.78	9	20.09	12.20	0.0003	significant
x_I	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	

3.3 RSCAC yield

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. Also, 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.



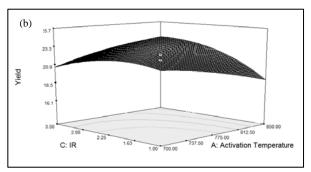




Figure 1 Three-dimensional response surface plot of RSCAC yield (a) Effect of activation temperature and activation time, IR = 2.25; (b) Effect of activation temperature and IR, t = 2h

3.4 Cu (II) removal

Based on the F-value as shown in Table 3, the activation temperature was found to have significant effects on the Cu (II) removal. The quadratic effects of activation temperature and activation time as well as the interaction effects between these two variables were considered moderate. Figure 2 shows the three-dimensional response surfaces that had been constructed to show the effect of two variables (activation temperature and IR) to the Cu (II) removal by RSCAC. For this plot, the activation time was fixed at zero level (t = 2h). It may be observed that Cu (II) removal increased together with an increase in activation temperature and IR. The highest Cu (II) removal was obtained when both variables had been in the maximum point within the studied range.

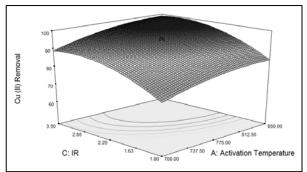


Figure 2 Three-dimensional response surface plot of Cu(II) removal (Effect of activation temperature and IR, t=2h) of RSCAC

3.5 Process optimization

One of the main aims of this study was to find the optimum process parameters that activated carbons produced should have a high RSC yield and a high Cu (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 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 Cu(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 Cu (II) removal obtained at optimum conditions are listed in Table 3.

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

Act ivat ed car bon	Mod el desir abili ty	Activation temperature, x1 (°C)	Activation time, x ₂ (h)	I R , x	Pre dict ed	II) remov (%) Expe rimen tal	E rr or (%	Pre dict ed	vated carb yield (%) Expe rimen tal	E rr or (%
RS C- CU	0.86 7	793	1.0	2. 4 6	96. 34	94.96	1. 4 3	23. 90	22.68	5. 1 1

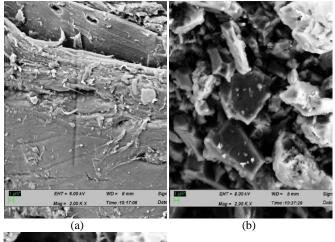
It was observed that the experimental values obtained were in good agreement with the values predicted by the models. Relatively small errors between the predicted and the actual values were observed, especially in the case of Cu(II) removal that had deviation errors of less than 2% for RSCAC. This indicated that the model was suitable and sufficient to predict the responses of the operating variables fixed.

3.6 Characterization of optimized RSCAC

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)-(d) respectively show the SEM micrographs of the RSC precursor, RSC char, RSC-CU. The SEM micrographs from Plate 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).



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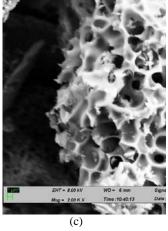


Plate 1 (a)RSC precursor, (b)RSC char, (c)RSC-CU

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 welldeveloped 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).

Table 6 Proximate and elemental analysis

Sample		Proximate Analysis (%)				doi:10.1016/j.biortech.2007.06.011 Elemental Analysis (%)			
	Moisture	Volatile	Fixed	Ash	C	Н	S	$(N+O)^a$	
			Carbon						

RSC raw	8.82	67.52	21.45	2.21	21.72
RSC char	7.52	34.02	56.31	2.15	36.21
RSCAC	6.38	18.36	73.93	1.33	67.10
^a Estimated by	difference				

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The precursor of RSC was found to be rich in volatile matters and high carbon contents as well, proving that these precursors were suitable to be utilized as activated carbons. The proximate analysis, after undergoing carbonization and activation process, showing that the volatile matter content of the precursors decreased significantly whereas the fixed carbon content increased in the produced activated carbons

4. Conclusions

Rubber seed coat (RSC) selected agrowaste precursors were successfully used for the 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 Cu(II) on all the RSC-CU were best fitted by the. The activated carbons prepared showed adsorption capacities of 81.97-82.64 mg/g for adsorption of Cu(II) at 30 °C. Adsorptions of Cu(II) on all the RSC-CU were best fitted by the Langmuir isotherm and best represented by the pseudo-second-order kinetic model. The filmdiffusion-controlled mechanism mainly governed all the adsorption processes.

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