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Study of 1-Chloro-4-Nitrobenzene adsorption on Carbon nanofibers by experimental design

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ABSTRACT: In this study, the adsorption of 1-chloro-4-nitrobenzene (1C4NB) on carbon nanofibers (CNFs), was investigated in a batch system. The combined effects of operating parameters such as contact time, pH, initial 1C4NB concentration, and CNFs dosage on the adsorption of 1C4NB by CNFs were analyzed using response surface methodology (RSM). The analysis of variance results confirmed that there was significant agreement between the model and experimental data. In addition, it was indicated that the residuals followed a normal distribution. The screening experiments showed that significant factors in 1C4NB removal were CNFs dosage, interaction between initial 1C4NB concentration-CNFs dosage and CNFs dosage-contact time. High efficiency removal (>90%) was obtained under optimal value of process parameters in the first 6 min of the removal process. The results indicate that RSM is a suitable method for modeling and optimizing the process, so that experimental design by RSM leads to time and cost saving. Non-linear form of Langmuir, Freundlich and Temkin models were fitted to adsorption equilibrium data. The results showed that the isotherm data can be well described by Freundlich isotherm equation.

Keywords: *Adsorption; Carbon nanofibers; Experimental design; Response surface methodology; 1-Chloro-4-nitrobenzene.*

INTRODUCTION

Benzene derivatives, such as a number of chloro- and nitro- benzenes are toxic and carcinogenic [1, 2]. Therefore, benzene compounds removal from industrial effluents is considered as a major concern and responsibility. A broad range of physicochemical methods has been proposed for treatment of these effluents including biodegradation [3-6], photocatalytic degradation [7-10], catalytic oxidation [11-14], adsorption [15-18], and so on. Adsorption method seems to be a good choice in terms of cost and operation for removal of benzene and its derivatives. It is important and interesting to develop a novel adsorbent with a large surface area, small diffusion resistance and high capacity for adsorption. The relative large specific surface area of nanomaterials enables them to become candidate for adsorption of adsorbates. However, high cost of adsorption method by nanoadsorbents limits extensive application of them in organic and inorganic pollution elimination. This constrain could be overcome via optimization of removal reaction conditions using statistical approaches. The response surface methodology (RSM) which is utilized broadly in treatment processes, involves a collection of useful statistical and mathematical techniques for analyzing the causal relationship between independent variables, responses, and their interactions through the construction of polynomial mathematical models which leads to time and cost saving . Second-order models are widely used in RSM to approximate the true response surface. The central composite design (CCD) is the most popular RSM and is used to build second-order models [19-22]. Several studies have been recently published on the potential applications of RSM in the adsorption of aromatic compounds such as benzene, phenol and 2,4-dichlorophenol [23-25].

The aim of this study was to conduct a CCD analysis for the significant factors that influenced the

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adsorption of 1-chloro-4-nitrobenzen (1C4NB) from aqueous solution by carbon nanofibers (CNFs) and understand their impact on the process. The effect of some operating variables, such as contact time, initial pH, initial 1C4NB concentration and CNFs dosage, and also their interactions on adsorption was studied using CCD, which gives a mathematical model that shows the influence of each variable and their interactions.

EXPERIMENTAL

Materials

Carbon nanofibers and 1-chloro-4-nitrobenzen ($C_6H_4ClNO_2$, Mw= 157.55 g mol^{-1}) were supplied by Sigma-Alderich. Average diameter, pore size and specific surface area of nanofibers was about 130 nm, 0.075 $cm^3 g^{-1}$ and 24 $m^2 g^{-1}$, respectively.

Adsorption Experiments

Batch adsorption experiments were down in an Erlenmeyer flask on a stirrer (300 rpm) by adding a 50 mL of 1C4NB solution and desired amount of CNFs. At predetermined time intervals, samples were drawn and were centrifuged. The concentration of 1C4NB was measured by UV-Vis spectrophotometer (UV mini-1240, λ_{max} = 280 nm). The percentage 1C4NB removal, *R (%)*, and amounts of adsorbed 1C4NB at equilibrium, q_e (mg g^{-1}), were calculated from the following equations (1–2), respectively:

$$R\ (\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e)}{m} \times V \quad (2)$$

where C_0, C_t and C_e are the initial, at any time *t* and equilibrium 1C4NB concentration (mg L^{-1}), respectively. *V* is the solution volume (L) and *m* is the adsorbent mass (g).

Experimental design

As previously mentioned, RSM is an important tool to optimize the conditions for wastewater treatment. Such statistical approach reduces the number of runs and provides valuable information on possible interactions between the variables and response. RSM based CCD requires an experimental number, given by the formula $N = 2^k + 2k + c_p$, where *k* is the factor number and c_p is the replicate number of the central point. All factors are studied in five levels ($-\alpha$, -1, 0, +1, $+\alpha$). α-Values can be calculated by $\alpha = 2^{k/4}$. For two, three, and four variables, they are, respectively, 1.41, 1.68, and 2.00 [26]. In the present study, CCD was used to propose and estimate a mathematical model of the adsorption process behavior. In order to evaluate the influence of operating parameters on the removal efficiency of 1C4NB, four independent factors were chosen: initial 1C4NB concentration (mg L^{-1}), CNFs dosage (g L^{-1}), initial pH of the solution and contact time (min). As the *k* value is equal four in this research, so $\alpha = 2^{4/4} = 2$. The experimental ranges and the levels of the operational variables are presented in Table 1. A total of 31 experiments were done, including $2^4 = 16$ cubic points, $2 \times 4 = 8$ axial points and seven replications at the center point. Experimental data were analyzed using the response surface regression procedure of a statistical analysis system (Minitab software version 17).

The mathematical relationship between the response and variables can be approximated by the following second-order polynomial equation (3):

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

where *y* is a predicted response of removal efficiency; β_0, the constant; β_i, the regression coefficients for linear effects; β_{ii}, the regression coefficients for quadratic effects; β_{ij}, the regression coefficients for interaction effects; x_i and x_j are coded experimental levels of the operational variables [26].

RESULTS AND DISCUSSION

Second-order polynomial model

The details of the designed experiments along with experimental results and predicted values for removal efficiencies of 1C4NB are presented in Table 2.

A semi-empirical expression in equation (4) consisting of 13 statistically significant coefficients was obtained from the data analysis using the statistical graphics software Minitab.

$$y = 90.9 - 1.803x_1 + 156.8x_2 - 1.06x_3 + 1.45x_4 + 0.0333x_1^2 - 441x_2^2 - 0.0339x_3^2 - 0.0428x_4^2 - 6.66x_1x_2 + 0.0014x_1x_3 + 0.33x_2x_3 - 10.66x_2x_4 + 0.130x_3x_4 \quad (4)$$

where *y*: percentage of 1C4NB removal, x_1: initial value of 1C4NB concentration, x_2: CNFs dosage, x_3: pH and x_4: contact time.

By comparing the measured values against the predicted responses by the model for the removal

efficiency (Table 2), this can be seen that this model explains perfectly the results in the experimental range studied. In equation the sign (+) indicates that removal efficiency increases in the presence of high levels of the respective variables, while the sign (“) indicates that removal efficiency decreases in the presence of high levels. Positive quadratic or second order polynomial coefficients indicate a synergistic effect, while negative coefficients, an antagonistic effect between or among the variables. The importance of each individual factor as well as interactions depends on the coefficient in equation (4). For example it can be seen that CNFs dosage plays an important role in percentage of 1C4NB removal.

Table 1: Experimental ranges and levels of the operational variables.

Variable	Symbol	Range and level				
		-α (-2)	-1	0	+1	+α (+2)
initial 1C4NB concentration (mg L^{-1})	x_1	4	8	12	16	20
CNFs dosage (g L^{-1})	x_2	0.04	0.08	0.12	0.16	0.20
initial pH of the solution	x_3	1.5	3.5	5.5	7.5	9.5
contact time (min)	x_4	2	4	6	8	10

Table 2: The 4-factor CCD matrix with the experimental and predicted responses.

Run	x_1	x_2	x_3	x_4	R (%)	
					Experimental	Predicted
1	8	0.08	3.5	4	93.41	92.36
2	16	0.08	3.5	4	90.90	88.51
3	8	0.16	3.5	4	97.10	96.76
4	16	0.16	3.5	4	96.33	97.93
5	8	0.08	7.5	4	89.60	90.73
6	16	0.08	7.5	4	83.11	85.18
7	8	0.16	7.5	4	94.76	94.49
8	16	0.16	7.5	4	95.12	93.95
9	8	0.08	3.5	8	94.81	93.06
10	16	0.08	3.5	8	89.34	89.96
11	8	0.16	3.5	8	97.53	95.80
12	16	0.16	3.5	8	96.77	97.72
13	8	0.08	7.5	8	94.02	92.77
14	16	0.08	7.5	8	90.55	87.97
15	8	0.16	7.5	8	93.40	94.87
16	16	0.16	7.5	8	93.68	95.08
17	4	0.12	5.5	6	94.90	98.00
18	20	0.12	5.5	6	94.90	94.36
19	12	0.04	5.5	6	83.16	82.97
20	12	0.20	5.5	6	96.72	94.48
21	12	0.12	1.5	6	93.89	95.64
22	12	0.12	9.5	6	90.56	91.38
23	12	0.12	5.5	2	90.03	92.45
24	12	0.12	5.5	10	94.13	94.28
25	12	0.12	5.5	6	93.43	93.34
26	12	0.12	5.5	6	93.07	93.34
27	12	0.12	5.5	6	94.83	93.34
28	12	0.12	5.5	6	94.34	93.34
29	12	0.12	5.5	6	93.51	93.34
30	12	0.12	5.5	6	92.51	93.34
31	12	0.12	5.5	6	91.71	93.34

Second-order polynomial model validation

The measurement of the residuals is a method to evaluate the adequacy of the model where the residuals represent the differences between the observed and the predicted response values. Normal probability plot is a suitable graphical method to know about the normality of the residuals. As shown in Fig. 1a, the inclination of the residuals is towards normal distribution. Fig. 1b shows that the residuals in the plot rise and fall randomly around the central line. These two plots revealed that the model is adequate for explaining the process under study.

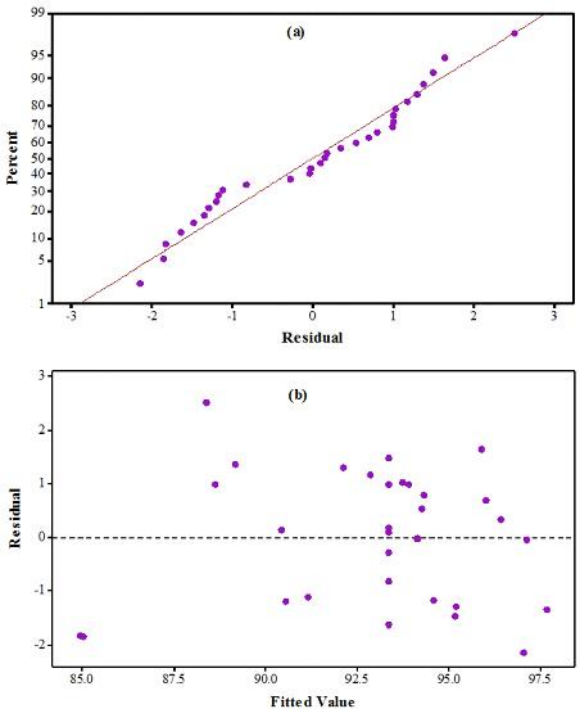

Fig. 1: (a) Normplot of Residuals and (b) Residuals versus Fits.

Analysis of variance and fitting the quadratic model

The analysis of variance (ANOVA) suggests that the equation and the actual relationship between the response and the significant variables represented by the equation were adequate. The larger the value of *F* and the smaller the value of *P*, the more significant is the corresponding coefficient term [27, 28]. The *F* value for the model is 7.50 and the corresponding *P*-value is <0.0001 (Table 3). These results indicated that the model was statistically significant. In addition, Table 3 shows the most significant factors and interactions that affected the response. As shown in Table 3, x_1, x_2, x_3, x_4, x_2^2, x_1x_2 and x_2x_4 are significant factors. The other model terms that *P*-values are greater than 0.1000 are not significant factors.

Table 3: Analysis of variance.

Source	DF	Sum of squares	Mean square	*F*-value	*P*-value
Model	14	304.332	21.738	7.50	0.000
x_1	1	14.784	14.784	5.10	0.038
x_2	1	181.894	181.894	62.78	0.000
x_3	1	34.068	34.068	11.76	0.003
x_4	1	13.456	13.456	4.64	0.047
x_1^2	1	8.123	8.123	2.80	0.113
x_2^2	1	14.238	14.238	4.91	0.041
x_3^2	1	0.525	0.525	0.18	0.676
x_4^2	1	0.840	0.840	0.29	0.598
x_1x_2	1	18.184	18.184	6.28	0.023
x_1x_3	1	0.002	0.002	0.00	0.980
x_1x_4	1	0.00	0.00	0.00	1.000
x_2x_3	1	0.011	0.011	0.00	0.951
x_2x_4	1	11.637	11.637	4.02	0.062
x_3x_4	1	4.338	4.338	1.50	0.239
Error	16	46.355	2.897		
Lack-of-Fit	10	39.667	3.967	3.56	0.067
Pure Error	6	6.687	1.115		
Total	30	350.687			

Effect of operational variables

Single factors:

Fig. 2 shows the influence of single factors while maintaining all other factors constant at midway value codified as value 0 (x_1= 12 mg L^{-1}, x_2= 0.12 g L^{-1}, x_3= 5.5 and x_4= 6 min), between their low and high values. The curve slope is proportional to the effect size whereas the line direction specifies a positive or negative influence of the effect. This diagram indicated that the percentage of removal decreased with an increase in the initial concentration of 1C4NB; it is apparent that the adsorbent has a limited number of active sites and these sites were saturated in a certain concentration, so the saturated adsorbent would not be able to uptake more 1C4NB molecules [29]. Increasing the removal efficiency by increasing adsorbent dosage was due to the availability of greater surface area and adsorption sites for the constant number of 1C4NB molecules [30]. Also, as shown in Fig. 2, removal of 1C4NB dropped for pH >3, that can be explained by the following reasons. The surface charge is neutral at isoelectric

point (IEP), where the pH_{IEP} value is obtained 3-4 for CNFs (not shown here). At pH values lower than the IEP, the net surface charge is positive whereas at pH values higher than the IEP the net charge is negative. Since 1C4NB is a kind of unionizable compound, the high adsorption is occurred at pH_{IEP}, where the charge of CNFs surface is neutral and can support the adsorption of 1C4NB. Similar result was reported for the adsorption of 1C4NB onto single-walled carbon nanotubes [31].

Interaction factors

Based on the ANOVA results (Table 3), high values of F and low values of P indicate a significant interaction between x_1 x_2, (initial 1C4NB concentration-CNFs dosage) and x_2 x_4 (CNFs dosage- contact time). Fig. 3 shows the contour plots of the interaction between two factors by holding all other factors at fixed levels. Fig. 3a represents the contour plot for x_1 (initial 1C4NB concentration) versus x_2 (CNFs dosage). To achieve removal efficiency higher than 95%, as the initial concentration of 1C4NB is increased form 4 to 12 mg L^{-1}, the CNFs dosage should be increased from 0.07 to 0.15 g L^{-1} accordingly. Hence, increasing adsorbate initial concentration results in increasing the amount of adsorbent. Fig. 3b exhibits the contour plot for x_2 (CNFs dosage) with respect to x_4 (contact time). The saddle nature of the contour plot confirms that a significant interaction exists between these two factors. The most suitable conditions to have a removal efficiency of more than 95% are high CNFs dosages (0.16–0.20 g L^{-1}) and contact time from 2-8 min. As it is clear from Fig. 3b, improvement on removal efficiency is not obvious above 8 min, because higher contact time cause reduces in the available surface area for adsorbing of 1C4NB molecules.

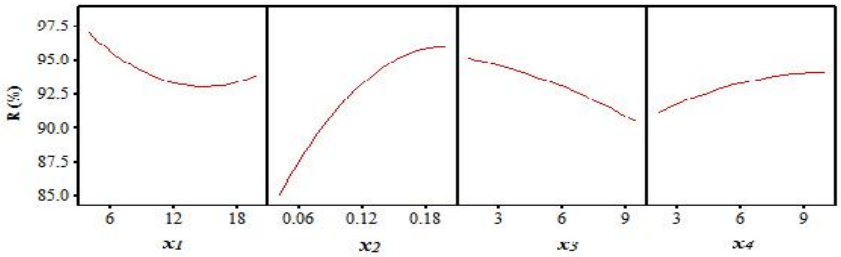

Fig. 2: Graphical presentation of the statistical evaluation of the individual factors on 1C4NB removal (x_1: initial 1C4NB concentration (mg L^{-1}), x_2: CNFs dosage (g L^{-1}), x_3: pH and x_4: contact time (min)).

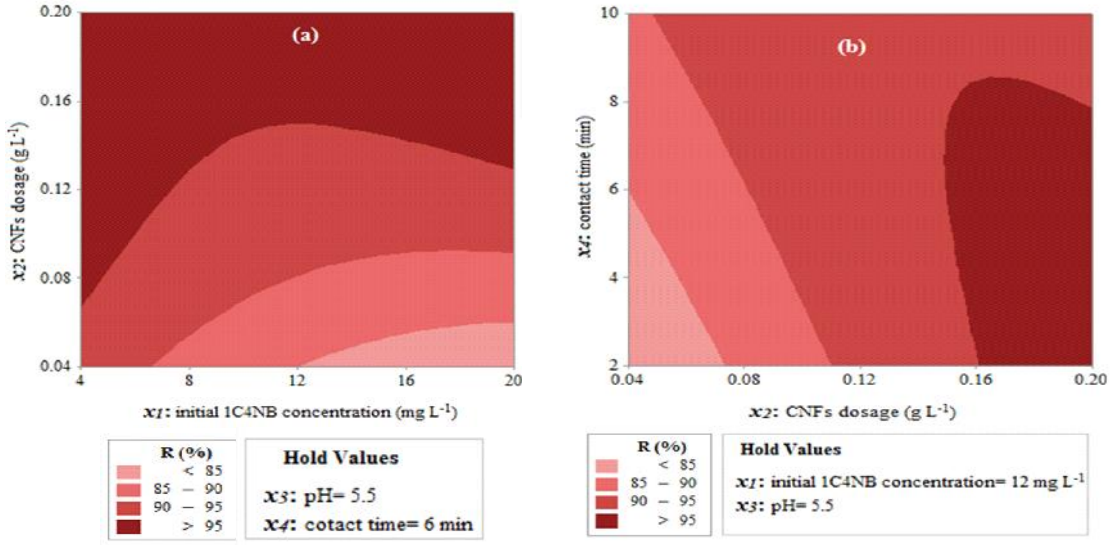

Fig. 3: Contour plots for 1C4NB removal as function of (a) initial 1C4NB concentration and CNFs dosage; (b) CNFs dosage and contact time interactions.

Adsorption isotherm studies

Isotherm studies were conducted with various CNFs dosage (0.04–0.20 g L^{-1}), while maintaining all other factors constant at midway value (initial 1C4NB concentration= 12 mg L^{-1}, pH= 5.5 and contact time= 6 min), between their low and high values. Non-linear form of Langmuir, Freundlich and Temkin models were fitted to adsorption equilibrium data (equations 5-7, respectively):

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \quad (5)$$

$$q_e = K_F C_e^{\frac{1}{n}} \quad (6)$$

$$q_e = B_1 \ln(K_L C_e) \quad (7)$$

where q_m is the maximum amount of adsorption and K_L is the affinity constant. K_F and n are the Freundlich constants indicating adsorption capacity and adsorption strength, respectively. K_T and B_1 are the Temkin model constants [32-34]. The curves of experimental data and results obtained from different models are shown in Fig. 4. Polymath software (version 6.10) was used for determining the isotherm constants. We have used correlation coefficient (R^2) and adjusted correlation coefficient (R^2 adjusted) parameters to predict which represents the data correctly. If R^2 value was close to one and also close to R^2 adjusted, it could be concluded that the regression model is correct [35]. The isotherm parameters data along with R^2 and R^2 adjusted values are listed in Table 4.

By comparing the values of R^2 and R^2 adjusted, it is found that the Freundlich model is the most suitable model to satisfactorily describe the studied sorption phenomenon. In this isotherm, if the value of n is below unity, then the adsorption is chemical; otherwise, the adsorption is physical [36]. Thus, in this research the value of 0.87 for n indicates that the adsorption of 1C4NB onto CNFs is a favorable chemical process.

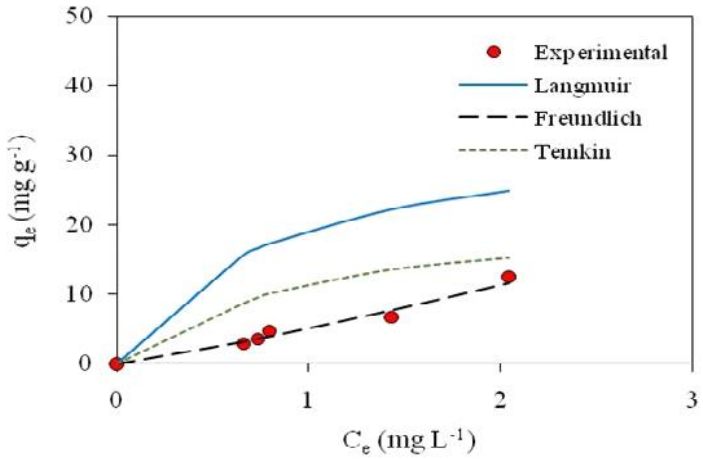

Fig. 4: Plots of non-linear adsorption isotherm models (initial 1C4NB concentration= 12 mg L^{-1}, CNFs dosage= 0.04-0.20 g L^{-1}, pH= 5.5 and contact time= 6 min).

Table 4: Isotherm parameters data.

Model	R^2	R^2 adjusted	parameters	values
Langmuir	0.745	0.732	q_m (mg g^{-1})	34.60
			K_L (L mg^{-1})	1.26
Freundlich	0.969	0.961	K_F [(mg g^{-1})(L mg^{-1})$^{1/n}$]	5.12
			n	0.87
Temkin	0.874	0.885	B_1	7.56
			K_T (L g^{-1})	2.17

CONCLUSION

Response surface methodology was successfully employed in this study to optimize the individual and interaction effects of the operational parameters. The screening experiments showed that significant factors in 1C4NB removal were CNFs dosage, interaction between initial 1C4NB concentration-CNFs dosage and CNFs dosage-contact time. ANOVA results confirmed that there was significant agreement between the model and experimental data. The results clearly demonstrated that RSM with a central composite design was one of the reliable methods to model and optimize the operational variables. Adsorption isotherm studies showed that the Freundlich isotherm equation provided the best correlation of the adsorption data.

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