

Desorption of Phenol from *Lantana camara*, Forest waste: Optimization using Response Surface Methodology and Kinetic Studies

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Abstract

In the present work, desorption studies to recover the adsorbed phenol from HCl treated adsorbent were carried out with ethanol solution. The experimental conditions like ethanol concentration, desorption time and temperature were optimized using response surface methodology. From the central composite design, a quadratic model was developed to relate the process variables to the percentage desorption as the response. From the analysis of variance (ANOVA), the most significant factor on the response was determined. The predicted percentage desorption values from the model was in good agreement with the experimental values. Therefore to understand the mechanism involved in desorption of phenol, desorption kinetics were performed at optimum conditions. The modelling of desorption data were done using pore-diffusion and first-order kinetic model. The data followed first-order kinetic model better than the pore-diffusion model. The percentage desorption was found to be 82.1% showing that ethanol is effective in desorbing phenol from the adsorbent.

Keywords: Phenol, desorption, central composite design, ANOVA, desorption kinetics

INTRODUCTION

The industrial wastewater contains different pollutants such as organic matter, phenol, dyes and heavy metals. Phenol one of the pollutant present in wastewater is harmful even at very low concentrations because of its toxic and carcinogenic properties. Therefore, it is important to remove phenol from wastewater before it is released into the water bodies. The different methods like biodegradation, biosorption, pervaporation, solvent extraction, membrane separation and adsorption are available to treat phenol from wastewater. Adsorption using chemically treated carbon is one of the effective method for treating phenolic wastewaters. But due to cost of activated carbon, there is a need to produce the adsorbent from locally available agricultural materials. Therefore, efforts are made to produce adsorbent using dry bark of lantana [1]. The phenol removal from wastewater were studied in batch and continuous mode [2]. Both the mode of studies were found to be effective in treating wastewater.

But the important drawback of the adsorption is secondary pollution created by the disposal of the used adsorbent. To overcome the above problem, different methods like thermal regeneration [3], chemical regeneration [4], bio-regeneration [5], ultrasound [6], microwave [7], wet peroxide oxidation [8], and electrochemical methods [9] are used for the regeneration of the adsorbent. Among these thermal regeneration and chemical regeneration are the prominent methods.

Thermal regeneration employs high temperature oxidation for desorption of pollutant which results in loss of adsorbent properties [10]. Hence a better non-destructive method such as chemical regeneration of carbon using solvents is found to be feasible in recovering the property of carbon. The chemical regeneration is favoured over thermal treatment because of the reasons that there is no loss of adsorbent and solvents can be reused [11]. Hence, the regeneration of spent carbon solves the difficulty of the disposal of used carbon and makes the process more efficient [12, 13]. The literature survey showed that solvents such as ethanol, hydrochloric acid, sodium hydroxide, acetone, methanol and acetic acid were employed for desorbing phenol from the agricultural based adsorbent. The important solvents are ethanol used for desorption of phenol from date pit [14], chitosan [15], barley husk [16], oil palm empty fruit bunch [17], Polygonum orientale [18], stems of loosestrife [19]. The other solvents are hydrochloric acid taken for desorption from barley husk [16], sodium hydroxide used for desorbing phenol from coconut coir pith [20], coconut shell [21], barley husk [16], oil palm empty fruit bunch [17], water hyacinth [22]. Also the other solvents such as acetone [23], acetic acid [24] and methanol [23] are used for desorption of phenol. Thus the present work investigates desorption of phenol from the spent adsorbent (HCl treated) lantana camara using ethanol as solvent. The influence of experimental conditions such as ethanol concentration, time of desorption and temperature on the desorption were studied. The optimization of the experimental conditions were done using response surface methodology to enhance the percentage desorption [25]. Finally the mechanism of desorption was investigated at the optimum conditions using pore-diffusion model and first order kinetic model.

MATERIALS AND METHODS

Preparation of the adsorbent

The material Lantana camara was washed with distilled water to remove the earthy matter. Then it is dried in sunlight for 48 h, crushed to pieces, ground to powder, sieved to particles of size lower than 0.075mm. To enhance the properties, powder was treated with different chemicals [1]. From the initial results, the two adsorbents treated with HCl and KOH were taken for batch experiments as discussed in the work [1]. The reuse capacity of the adsorbent treated with HCl and KOH were carried out. Based on the reuse potential, the HCl treated adsorbent was taken for desorption studies. The desorption was done with five different solvents. Based on the desorption results, the desorbing agent ethanol was taken for optimization studies.

Chemicals

Phenol (Merck India Ltd) of analytical grade was used for preparing stock solution of concentration 1000 mg/l. The experimental solution of concentration 150 mg/l was prepared from the stock solution by diluting to accurate proportion. The other chemicals Hydrochloric acid (SD Fine Chemicals, AR grade) were used for treating the adsorbent. Ethanol (Hayman Speciality products, England, AR grade), acetone (Finar Limited, India, AR grade), acetic acid (Finar Limited, India, AR grade) and sodium hydroxide (Fisher Scientific limited, Excelsa R) are used for desorption of phenol.

Desorption experiments

The batch experiments were carried out with HCl treated adsorbent. 0.75 g of adsorbent was added into 250 mL conical flask filled with 200 mL of 150 mg/l concentrated phenol solution. The conical flask was placed in a shaker at a temperature of 30°C, rotation speed of 120 rpm, agitated for 510 min till the equilibrium was reached. The concentration of phenol was determined using UV-vis spectrophotometer. The mass of phenol adsorbed was found by calculating the difference between initial and final concentration. The used adsorbent was then separated out of the solution, washed with water and dried at 108°C in an oven (Servewell Instruments Limited, Bangalore, India) for 4 hours. Then the adsorbent was treated with 200 mL of 1M ethanol, 1M HCl, 1M acetone, 1M sodium hydroxide and 1M acetic acid for phenol desorption. The flask was placed in the isothermal shaker at the temperature of 30°C and for the time duration of 510 min similar to the adsorption experiments. After desorption, the mass of pollutant desorbed was measured in the liquid phase by UV-vis spectrophotometer. The phenol percentage desorption from the adsorbent for all the solvents were determined. Based on the preliminary investigation, ethanol was taken for further studies. The percent desorption (% des) was calculated using Eq. (1).

$$\% \text{ des} = \frac{\text{Mass of pollutant desorbed}}{\text{Mass of pollutant adsorbed}} \times 100 \quad (1)$$

Optimization of experimental parameters

The present work, investigates the influence of three independent parameters, concentration of ethanol, time of desorption and temperature at five coded levels (as shown in Table 1) using central composite design. Then the desorption kinetics was studied at the optimum values.

Table 1: Independent numerical variables and its levels.

Independent variables	Symbol	Coded level of variables				
		$-\alpha$	-1	0	+1	$+\alpha$
Concentration (M)	X_1	0.32	1	2	3	3.68
Time (min)	X_2	331.82	400	500	600	668.18
Temperature (°C)	X_3	23.18	30	40	50	56.81

Modelling the desorption data

From the desorption studies, it is possible to investigate the mechanism and the adsorbent is utilized efficiently [26]. The rate of the process can also be found from the studies. Thus two models, first -order kinetic and pore diffusion models are used for checking the experimental data.

Pore diffusion model [26]

The model is developed based on the principle that the isothermal diffusion of the pollutant takes place in a spherical adsorbent particle. It is also from on the assumption that the pollutant concentration inside the spherical adsorbent is constant. It is expressed by the Eq.(2),

$$\frac{q_t}{q_{tot}} = 6 \sqrt{\frac{D_1 t}{r^2 \pi}} \quad (2)$$

where D_1 is diffusivity of pollutant within the adsorbent ($m^2 s^{-1}$), r the radius of the adsorbent, q_t the quantity of pollutant desorbing from the adsorbent at any time t (mg of pollutant/g carbon) and q_{tot} is total quantity of pollutant attached to the adsorbent (mg of pollutant/g carbon). The model constant D_1/r^2 can be determined from the plot of q_t/q_{tot} versus \sqrt{t} .

First-order kinetic model [26]

It is derived on the assumption that surface bonds binding the adsorbate molecule to the sorbent surface breaks and the process is limited. It is described by the Eq.(3)

$$\ln \left(\frac{C_t}{C_o} \right) = -k_d t \quad (3)$$

where C_t is the phenol concentration inside the carbon at any time t , and k_d the desorption rate constant (s^{-1}). The model constant k_d is calculated from the linear plot of $\ln(\frac{C_t}{C_o})$ versus time t .

RESULTS AND DISCUSSION

Desorption of phenol with different solvents

Initially the reuse potential of adsorbent treated with HCl and KOH over five cycles were obtained as 16.61mg/g and 10.76 mg/g respectively. Based on this adsorbent treated with HCl was taken for desorption studies. The various solvents ethanol, sodium hydroxide, acetone, acetic acid and hydrochloric acid were taken for desorption of phenol. The percentage desorption were found to be 48.3%, 24.96%, 38.8%, 35.66% and 22.36% for ethanol, acetone, sodium hydroxide, hydrochloric acid and acetic acid respectively. Ethanol showed the highest percentage desorption while values with sodium hydroxide were satisfactory. But results with other solvents acetone, acetic acid and hydrochloric acid were not promising.

The results can be discussed from the following possible reasons. It is understood that ethanol is having more solubility with phenol, therefore it increases the interaction between the two molecules. Thus more of phenol can be desorbed by using ethanol [12, 16]. Also the solvent weakens the interaction between adsorbent and solid surface. Phenol interacts with sodium hydroxide forming sodium phenates which helps in the desorption process [13, 20]. The solubility with other solvents was relatively less which may not form the complex phenolic compounds, thus the percentage desorption values were less. From these obtained results, ethanol was considered for optimization studies.

Development of model equation

Central composite design is an effective tool which develops the correlation between the operating variables to the percentage desorption [27, 28]. Table 2 shows the complete design matrix with response values Y (i.e. percentage desorption) obtained from the experiments. By applying multiple regression analysis to design matrix and the desorption values, a quadratic polynomial equation was obtained [29]. The values of percentage desorption varied from 28.93 to 81.8%. The model equation in terms of coded factors after neglecting the insignificant terms for percentage desorption is given in Eq. (4).

$$\% \text{ des } (Y) = 67.93 + 11.80 \times x_1 + 3.59 \times x_2 + 5.98 \times x_3 - 4.68 \times x_1^2 \quad (4)$$

The model equation helps in optimizing the desorption conditions [30]. The fitness of the model was checked by values of correlation coefficient (R^2) and standard deviation [25]. The

R^2 value for the equation was obtained as 0.9509 signifying that 95.09% of the variation in the percentage desorption was due to the experimental variables. It also implies that only 4.91% of the variations are not described by the model and the unexplained part is given as residual error in the ANOVA table [30]. The standard deviation for the model for Eq. (8) was obtained as 3.15. The closer the R^2 value to unity and the lesser the standard deviation, the more accurately the response can be predicted [31]. From the values obtained it also showed that experimental values were agreeing with the predicted values [25]. The Coefficient of variation (CV) was determined as 4.87 that shows that shows a good precision and reliability with the experimental data.

Table 2: Design matrix for the parameters and results obtained.

Experiment No:	Coded values of variables			Actual values of variables			% des
	x_1	x_2	x_3	Concentration (M)	Time (min)	Temp (°C)	
1	-1	-1	-1	1	400	30	45.55
2	1	-1	-1	3	400	30	65.69
3	-1	1	-1	1	600	30	53.47
4	1	1	-1	3	600	30	73.61
5	-1	-1	1	1	400	50	56.11
6	1	-1	1	3	400	50	75.14
7	-1	1	1	1	600	50	62.08
8	1	1	1	3	600	50	81.8
9	-1.68	0	0	0.32	500	40	28.93
10	1.68	0	0	3.68	500	40	77.77
11	0	-1.68	0	2.00	331.82	40	60.55
12	0	1.68	0	2	668.18	40	72.77
13	0	0	-1.68	2	500	23.18	51.25
14	0	0	1.68	2	500	56.81	77.94
15	0	0	0	2	500	40	68.47
16	0	0	0	2	500	40	68.75
17	0	0	0	2	500	40	68.61
18	0	0	0	2	500	40	68.55
19	0	0	0	2	500	40	68.66
20	0	0	0	2	500	40	68.86

Analysis of Variance

The larger value of F signifies that maximum variation in the response is given by the model. The p value is helpful in determining whether F is sufficient to show statistical significance. Any factor with $p < 0.05$ and larger F value is considered as significant. From the ANOVA analysis given in Table 3, the model F -value of 23.23 and $\text{Prob} > F$ of 0.0001 shows that the model was significant. For this case, X_1 , X_2 , X_3 and X_1^2 were observed as significant terms while X_1X_2 , X_2X_3 , X_1X_3 , X_2^2 and X_3^2 were all negligible terms. It can also be observed that the optimum values for phenol desorption

were lying within the range of experimental values since the linear terms (X_1, X_2, X_3) are more significant than the squared terms (X_1^2, X_2^2 and X_3^2) and the interaction terms (X_1X_2, X_1X_3 and X_2X_3) [32]. The highest coefficient ($X_1 = 137.18$) in the fitted model obtained signified that the desorption was highly sensitive to the solvent concentration. Therefore, it can be summarized that the experimental parameters have a significant influence on the percentage desorption.

Table 3: ANOVA for response surface quadratic model for the desorption of phenol

Source	Sum of squares	Degree of freedom	Mean square	F value	p
Model	2899.1	9	322.13	23.23	0.0001*
Concentration	1902.00	1	1902.00	137.18	0.0001*
Time	175.9	1	175.96	12.69	0.0052*
Temperature	488.7	1	488.72	35.25	0.0001*
Concentration*Time	0.060	1	0.060	0.004292	0.9491
Concentration*Temperature	0.29	1	0.29	0.021	0.8874
Time*Temperature	1.29	1	1.29	0.093	0.7668
Concentration ²	326.5	1	326.51	23.55	0.0007*
Time ²	0.042	1	0.042	0.003039	0.9571
Temperature ²	8.86	1	8.86	0.64	0.4426
Residual	138.6	10	13.86		

* The factors are significant

Influence of experimental parameters

From the results of ANOVA, it was found that factor concentration has the highest F value of 137.18 indicating that it is having more influence on phenol desorption. The desorption temperature and time were also considerable influence on the final response. The desorption time was having less influence compared to concentration and temperature. The percentage desorption increases by a large quantity with the increase in the concentration of solvent. The effect of desorption temperature and desorption time was found to a lesser extent. The possible reason for this can be at high temperature the surface bonds that binds the adsorbate with the adsorbent surface breaks and desorption proceeds [26]. As the desorbing solvent comes in contact with adsorbed phenol, the phenol molecules becomes soluble with the ethanol solvent and the desorption takes place [12, 15, 16]. These results were similar with those referred by the authors [12, 20, 22, 26]. It was observed that even with 3 M ethanol complete desorption could not occur, phenol molecules becoming trapped inside the pores were difficult to get removed [12]. With respect to the

time, it was noticed that desorption occurs faster initially, because the phenol molecules which are attached on the outside surface are desorbed faster. In the later stages, the phenol molecules which are present deep inside the pores are removed slowly over a period of time. Therefore, it influences the characteristics of adsorbent with time, leading to decrease in regeneration efficiency [14]. These results are in good agreement with those reported by authors [26, 32].

Optimization of experimental variables

The important objective of the present study is to find the optimum experimental conditions at which highest percentage desorption can be achieved [33]. To optimize the experimental variables, the targeted criteria was set as maximum for phenol desorption (Y) while the other variables (desorption temperature, desorption time and concentration of ethanol) were set in the study range. The optimum experimental conditions were obtained as 2.94 M of desorbing solvent concentration, desorption time of 558 min and temperature of 49.5°C. The predicted desorption percentage at the optimum conditions was found to be 82.18%. The verification kinetics experiments were conducted at the optimum conditions to confirm the validity of the proposed model (Sin et al. 2011). The experimental value obtained was 81.49% with relatively small error (0.84%) existing between the predicted and the experimental values. This also signified that the models developed were effective in predicting the responses from the operating parameters [25].

Desorption kinetics

Two models pore diffusion model and first-order kinetic model were used for studying the desorption mechanism. The experimental data and the predicted values from both the models are compared and are shown in Figure 1. The values of regression coefficient showed that experimental data were fitting with first-order kinetic model. The R^2 values and constants are shown in Table 4. The lower value of desorption kinetic constant implies that the pollutant stays stably on the adsorbent and desorbed from the adsorbent by treating with ethanol [26]. The regenerated adsorbent may be reused once again.

Table 4: The desorption kinetic constants obtained from the models.

Model	Constant	Value (s^{-1})	R^2
First-order	k_d	$1.237 \times 10^{-5} \pm 4.84 \times 10^{-7}$	0.9869
Pore diffusion	D_1/r^2	$2.13 \times 10^{-6} \pm 5.07 \times 10^{-8}$	0.9747

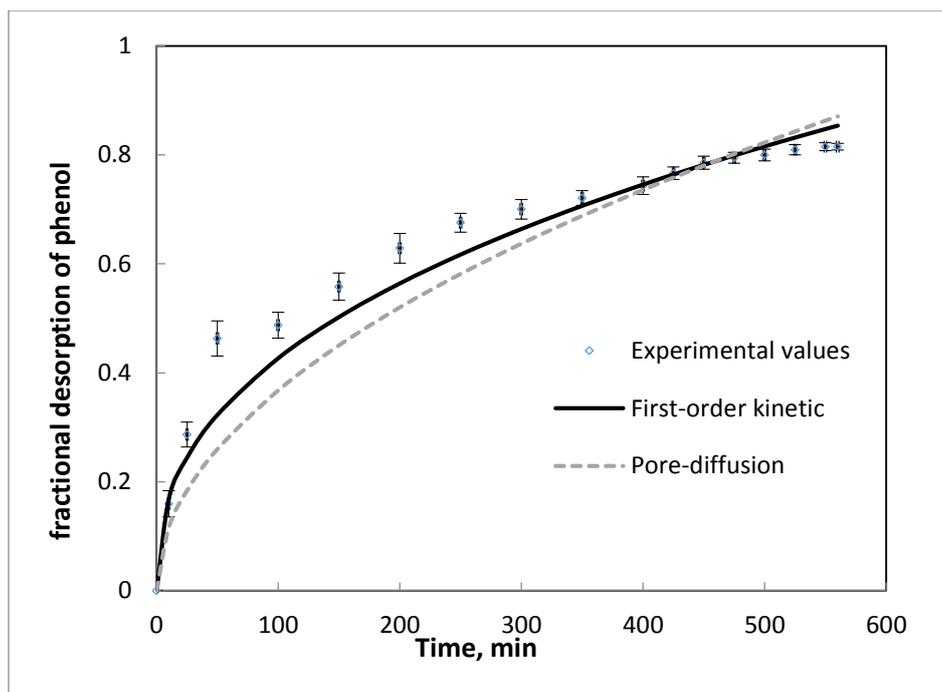


Figure 1: The fractional desorption of phenol from HCl treated adsorbent (Optimum conditions: Concentration 2.94 M, Time 558 min, Temperature 49.5°C)

CONCLUSIONS

The desorption studies were carried out using different solvents ethanol, acetone, sodium hydroxide, hydrochloric acid and acetic acid. It was found that the percentage desorption was maximum with ethanol, because ethanol was more soluble with phenol. The optimization of the process were done with respect to the parameters ethanol concentration, temperature and time of desorption using response surface methodology. It was observed that the percentage desorption increases with increase in ethanol concentration, temperature and desorption time. The desorption data followed first-order kinetic model better than the pore-diffusion model. Thus showing that breaking of the surface bonds that binds the adsorbate with the adsorbent surface becomes the rate limiting step.

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