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Research paper

Tea fiber waste as an adsorbent to remove phenol from wastewater

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Abstract

The current work investigates the phenol adsorption from wastewater using tea fibre waste as an adsorbent. The agricultural waste was treated with phosphoric acid and various properties like surface are, pore volume and average particle size of the adsorbent were determined. The optimization of the parameters like temperature, pH, dosage and concentration were done with response surface methodology (RSM). The quadratic model developed from the optimization was used to relate the variation of percent removal with respect to experimental parameters. The equilibrium data obeyed Freundlich model while kinetic data followed first order model. From the thermodynamic studies, the feasibility of the process was found. The adsorption capacity for the tea fibre was obtained as 12.59 mg/g.

Keywords: *Tea Fiber Waste; Phenol, Kinetics; Optimization; Adsorption Capacity*

1. Introduction

The usage of phenol and phenolic compounds are of great importance in different chemical process industries [1]. They generally exists in wastewater released from industries like petroleum refining, distillery, coal gasification, pulp and paper and coke oven industries [2], [3]. Therefore, from the wastewater, phenol enters the aquatic bodies and the water sources in the environment. Phenol is considered as toxic at very low concentration. It reduces the quality of drinking water. It also leads to various health problems such as damage of skin, gastrointestinal and cardiovascular illness [1], [2], [3]. Thus removing phenol from the wastewater is required and is a major challenge for the environmentalist from the pollution point of view.A large number of treatment methods like chemical oxidation, biodegradation, ion exchange, adsorption and membrane separation are used for treating phenol [4]. Adsorption using adsorbent is considered as an effective method, since it treats the pollutant to a very low level.

Tea waste fibre which was easily available in tea plantations in Sivasagar, Assam was taken as the adsorbent [5].The tea fibre waste is used to the minimal extent, since it cannot be used as firewood or a source as manure. It is rarely used for feeding some mountain animals in the Assam region. Therefore the present study focusses on the phenol removal from wastewater using tea fibre waste as an adsorbent. The batch studies were carried to determine the influence of the operating conditions on the adsorption process. The optimization of the parameters were done using response surface methodology.

2. Materials and methods

2.1. Preparation of adsorbent

The tea fibre waste was washed with water and then dried in sunlight. Then it was dried in an oven at 100ºC for 1 hour so that all the moisture was removed from the sample. The sample was impregnated with 2M ortho-phosphoric acid in 1:4 ratio for about 20 minutes. Then the slurry was heated inside muffle surface at 450°C for 1.5 h to allow the formation of pores and also the properties of the adsorbent are improved. The sample was cooled and washed with water to remove excess acid [6].The process of washing was continued so that the pH reaches seven. [7] Finally the material was subjected to filtration, drying and used for the later experiments.

2.2. The characterization of the adsorbent

Using the standard procedure, the proximate analysis of the chemically treated adsorbent was performed [8] to calculate the fixed carbon, volatile matter, moisture and ash content. The average particle size was found using particle size analyzer (CILAS 1064, France).The measurement of surface area and pore volume were done using BET apparatus (Smart Instruments, India). The various functional groups which exists on the surface of the adsorbent were determined using Fourier transform infrared spectroscopy (FTIR) instrument, Shimadzu, Japan.

2.3. Adsorption experiments

The studies were conducted in 250 mL conical flasks having 200mL of phenol solution (100 mg/L) at 125 rpm. The equilibrium time of the adsorption was calculated as 8 hours. The phenol concentration was measured using U-V spectrophotometer (Shimadzu, Japan) at 270 nm wavelength. The effect of operating parameters like pH, temperature, dosage and concentration on the phenol removal was investigated. The values of phenol concentration were considered in the range 40 to 100 mg/L based on the concentration of pollutants in the industrial wastewater [9]. The optimization of the conditions were done using RSM. The values of dosage, pH and temperature used in the experiments were taken from the literature available

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with respect to similar agricultural materials [10], [11].It is a statistical tool used for determining the optimal conditions and the model equation from the given experimental data [12]. A general RSM technique like central composite design was employed for finding the effect of experimental parameters on percent removal. The present work studies the influence of four independent parameters pH, temperature, dosage and concentration at five coded levels (Table1).The isotherm experiments were conducted at six different concentration values of 40 mg/L to 140 mg/L. The kinetic studies were performed at the optimal values. The number of experiments were obtained from central composite design for four variables, its higher and lower range and by considering the star points, factorial points and center points [13]. The total design matrix with the experiments conducted and the percent removal results are given in Table 2.

Table 1: Independent Parameters and its level

Independent parameters	Symbol	Range and level				
		-α	-1	0	+1	+α
pH	X_1	2	4	6	8	10
Temperature (ºC)	X_2	20	30	40	50	60
Dosage (g)	X_3	0.2	0.8	1.4	2.0	2.6
Concentration (mg/L)	X_4	10	40	70	100	130

Table 2: Design Matrix for the Experimental Parameters and Results Obtained

Expt No:	Coded parameters				Actual parameters				
	x_1	x_2	x_3	x_4	pH	Temperature (ºC)	Dosage (g)	Concentration (mg/L)	%rem
1	-1	-1	-1	-1	4	30	0.8	40	46.25
2	1	-1	-1	-1	8	30	0.8	40	53.75
3	-1	1	-1	-1	4	50	0.8	40	33.75
4	1	1	-1	-1	8	50	0.8	40	42.5
5	-1	-1	1	-1	4	30	2	40	70
6	1	-1	1	-1	8	30	2	40	68.75
7	-1	1	1	-1	4	50	2	40	76.25
8	1	1	1	-1	8	50	2	40	77.5
9	-1	-1	-1	1	4	30	0.8	100	39
10	1	-1	-1	1	8	30	0.8	100	37.5
11	-1	1	-1	1	4	50	0.8	100	33
12	1	1	-1	1	8	50	0.8	100	37.5
13	-1	-1	1	1	4	30	2	100	61
14	1	-1	1	1	8	30	2	100	64
15	-1	1	1	1	4	50	2	100	67
16	1	1	1	1	8	50	2	100	67.5
17	-2	0	0	0	2	40	1.4	70	63.5
18	2	0	0	0	10	40	1.4	70	64.3
19	0	-2	0	0	6	20	1.4	70	77.1
20	0	2	0	0	6	60	1.4	70	62.8
21	0	0	-2	0	6	40	0.2	70	11.4
22	0	0	2	0	6	40	2.6	70	88.6
23	0	0	0	-2	6	40	1.4	10	95
24	0	0	0	2	6	40	1.4	130	60.8
25	0	0	0	0	6	40	1.4	70	70
26	0	0	0	0	6	40	1.4	70	70
27	0	0	0	0	6	40	1.4	70	69.3
28	0	0	0	0	6	40	1.4	70	70
29	0	0	0	0	6	40	1.4	70	71.4
30	0	0	0	0	6	40	1.4	70	70

3. Results and discussions

3.1. Characterization of the adsorbent

The proximate analysis of the chemically treated sample is given in table 3. It represents that the material possesses higher fixed carbon content and it shows that the material has good adsorption capacity for the pollutant [14]. The pore volume and surface area of the material were determined as 1.3021 cm^3/g and 1235.91 m^2/g respectively showing the potential of the adsorbent to adsorb phenol [15]. The average particle size was 54.80 µm which improves the surface area and thus the efficiency of the adsorbent.

The FTIR spectrum of the chemically treated sample shows broad adsorption peak at 3490.92 cm^{-1} that corresponds to overlapping of –OH and –NH groups [16]. The peak found at 2356.85 cm^{-1} and 1623.95 cm^{-1} corresponding to the $C \equiv N$ group and the $C = C$ group [17]. The peak formed at 1569.95 cm^{-1} and 1434.94 cm^{-1} depicts the $–NO_2$ group and the C-H group with alkyl halides respectively. From these results, it can be inferred that these bonds are responsible for adsorption of phenol onto the chemically treated carbon and are shown in figure 1.

Table 3: Proximate Analysis of the Treated Adsorbent

Sl. No	Parameter	Percentage	Sl.No	Parameter	Percentage
1	Moisture	8.5	2	Ash content	5
3	Volatile matter	28.5	4	Fixed carbon content	58

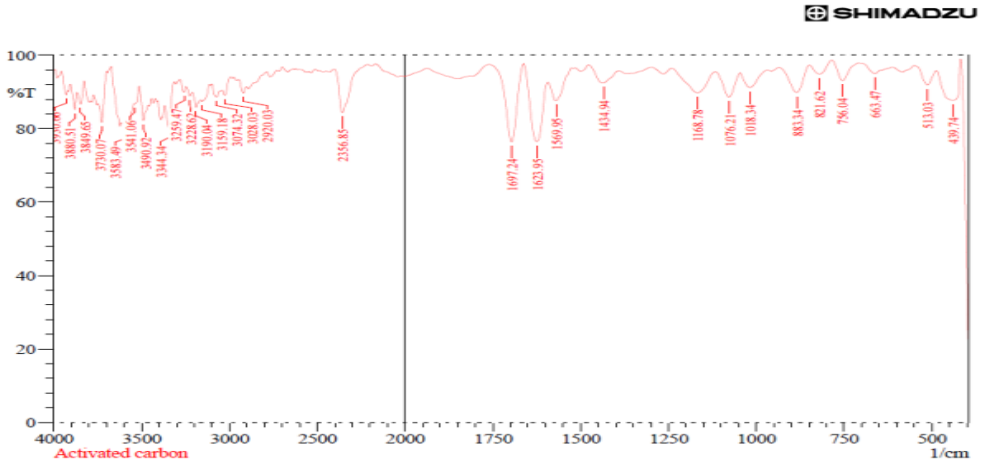


Fig. 1: The FTIR Spectra of the Treated Adsorbent.

3.2. Development of model equation and optimization of experimental parameters

The design matrix having response values i.e. percentage removal calculated from the experiments are given in table 1. The percentage removal values obtained from the range of experiments varied from 11.4% to 95%. A quadratic polynomial equation was obtained which shows the interaction between the variables and is also helpful to optimize the parameters [18], [19]. The model equation for percentage removal in terms of coded factors by neglecting all the insignificant factors is given as

$$\%\, rem = 68.44 + 15.96\, x_3 - 5.44x_4 - 3.13x_1^2 - 6.61x_3^2 \quad (1)$$

The value of correlation coefficient (R^2) and standard deviation was employed for checking the validity of the model [20]. The R^2value for the equation was obtained as 0.8566 and this implies that 85.66% of the result obtained was because of the variation in the experimental conditions [13], [21], [22]. Also it shows that 14.34% of the variation is not discussed by the model and is due to residual errors [21]. The model can be validated if the value of R^2 is approaching unity and value of standard deviation is lower [23]. The standard deviation was obtained as 7.43, showing good fit with the model. The value of coefficient of variance 12.25 shows better precision of the experimental data [22]. The optimal experimental conditions were obtained as 40 mg/L of concentration, 2 g of dosage, temperature of 43.4°C, pH of 6.2 and percent removal of 85.54%. The verification experiments were conducted at these optimum conditions and the error was obtained as 0.63%.

3.3. Isotherm studies

Several models have been reported in literature to explain the equilibrium relationships between adsorbent and the adsorbate. The most common models like Langmuir, Freundlich and Temkin models are discussed below.

The Langmuir isotherm is explained assuming that adsorption is occurring uniformly on the surface. The simplified form of the model is represented as [24]

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{k_L q_{mx}} \quad (2)$$

q_e and C_e the adsorption capacity (mg/g) and solute concentration (mg/L) at equilibrium respectively, $k_L$the model constant (L/mg) and q_{mx} the monolayer adsorption capacity (mg/g). The parameters k_L and q_{mx} are calculated through the linear plot of C_e/q_e v/s C_e.

Freundlich model is obtained from the concept that adsorption is happening non-uniformly in multilayers.[25, 26] The energy distribution of the process is non-uniform on the solid surface. It is given in the linear form as

$$logq_e = logk_f + \frac{1}{n}\, logC_e \quad (3)$$

n In addition, k_f $\left(mg^{1-\frac{1}{n}}\, L^{1/n}\, g^{-1}\right)$ are the model constants. The model parameters k_f and n are obtained through the linear plot of $logq_e$ v/s$logC_e$.

The Temkin isotherm describes indirectly how the adsorbent and pollutant molecules interact with each other. It is represented as [11]

$$q_e = BlnA + BlnC_e \quad (4)$$

A In addition, B are the model parameters? The values of constants are calculated by plotting q_e v/slnC_e.

The isotherm parameters obtained from the models are represented in Table 4. The model was validated based on the value of regression coefficient and it was observed that the experimental data was fitting best with the Freundlich isotherm. Therefore it describes that the process is physical adsorption and pollutant gets adsorbed on the solid surface in multilayers. The monolayer adsorption capacity was calculated to be 12.59 mg/g.

Table 4: The Model Parameters from Different Isotherm Models.

Model	Isotherm constants		
Langmuir	q_{mx}=12.59 mg/g	k_L =0.064 L/mg	R^2=0.9442
Freundlich	k_f=1.459 $\left(mg^{1-\frac{1}{n}}\, L^{1/n}\, g^{-1}\right)$	n=1.953	R^2=0.9838
Temkin	A=0.376 L/g	B=8.199 J/mol	R^2=0.9383

3.4. Thermodynamic studies

The feasibility of the process was found by performing thermodynamic studies and determining the values of ΔG, ΔH and ΔS from the equations.

$$\Delta G = \Delta H - T\Delta S \quad (5)$$
$$\Delta G = -RT\, \ln K_d \quad (6)$$

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$$K_d = \frac{q_e}{C_e} \quad (7)$$

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (8)$$

where ΔG, ΔH and ΔS are changes in Gibbs free energy, enthalpy, and entropy respectively. The parameters ΔH and ΔS are determined through the plot of $\ln K_d$ v/s $1/T$ and are shown in Table 5.

Table 5: The Values of Calculated Thermodynamic Parameters at Different Temperatures.

Parameter	ΔG (kJ/mol) 293 K	ΔG(kJ/mol) 313 K	ΔG(kJ/mol) 333 K	ΔS (J/mol K)	ΔH (kJ/mol)
	-2.6762	-2.2049	-1.2384	-35.4184	-13.1261

The ΔG values shows that the reaction is feasible and spontaneous at the temperatures. The negative value of ΔS and ΔHimplies that randomness reduces at solid liquid interface and process is exothermic in nature [27], [28].

3.5. Kinetic studies

Kinetic studies monitors the experimental parameters that affect the speed of the reaction. The different aspects like the mechanism, how the adsorbent- pollutant complex formed and how the interaction takes place can be understood from kinetic studies [29]. It helps in evaluating the residence time required for the solute in the adsorbent solution medium [30].

Pseudo-first-order kinetic model

The model is derived by assuming reversible physical adsorption. The model is represented in the form [31], [32]

$$\log(q_e - q_t) = \log q_e - \frac{k_{fs}}{2.303} t \quad (9)$$

Where q_t is the capacity at any time t (mg/g), k_{fs} the model rate constant (min^{-1}). The values of constants k_{fs} and q_e are evaluated through the plot of $\log(q_e - q_t)$ v/s t.

Pseudo-second-order kinetic model

The model is generally applicable to the systems where chemisorption is the slowest step. The model is written in the simplified form as [31], [33]

$$\frac{t}{q_t} = \frac{1}{k_s q_e^2} + \frac{1}{q_e} t \quad (10)$$

where k_s the second-order rate constant (g/mg min). The model constants are obtained through the plot oft/q_t v/s t .

Intra-particle diffusion model

In the case of porous particles, particle diffusion may be taken as the slowest step. The rate equation can be given as [34]

$$q_t = k_p t^{0.5} + I \quad (11)$$

k_p is the model rate constant ($\frac{mg}{g min^{0.5}}$) , Ithe boundary layer thickness. The model is generally applied if the plot of q_t v/s $t^{0.5}$ is passing through the origin.

The kinetic constants evaluated from the models are given in Table 6. The value of regression coefficient was used to validate the model. It was found that higher R^2value was obtained for pseudo first order model in comparison with other models. The experimental adsorption capacity of 4.25 mg/g was matching closely with the calculated value of first order model. Therefore it explains that physisorption is happening on the surface of the adsorbent.

Table 6: The Constants Calculated from the Kinetic Models

Pseudo-first order model			Pseudo-second order model			Intraparticle diffusion		
k_{fs}, min^{-1}	$(q_e)_{cal}$ mg/g	R^2	k_s, g/mg min	$(q_e)_{cal}$, mg/g	R^2	k_p, $(\frac{mg}{g} min^{0.5})$	I	R^2
0.01197	5.631	0.9863	0.00239	5.909	0.9783	0.2056	0.2911	0.9082

4. Conclusion

The current study proved the capability of the tea fibre waste as an effective adsorbent to remove phenol from wastewater. The adsorbent treated with phosphoric acid showed improvement in property which was beneficial for the adsorption process. The optimum conditions from the RSM were obtained as concentration of 40 mg/L, 2 g of dosage, temperature of 43.4°C, pH of 6.2 and percent removal of 85.54%.The isotherm data obeyed Freundlich model and the kinetic data followed pseudo first order model showing that physical adsorption is the possible process. The thermodynamic studies implied that the process is feasible and spontaneous. The monolayer adsorption capacity for the tea fibre waste was calculated as 12.59 mg/g.

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