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Removal kinetics and mechanism for phenol uptake by surfactant-modified alumina

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ABSTRACT

The present work has been devoted to study the kinetics and the mechanism of the phenol removal from water by surfactant-modified alumina (SMA). Sodium dodecyl sulfate (SDS), an anionic surfactant (AS) was used for the surface modification of neutral alumina. Micelle-like structures are formed on the surface of alumina, which was used for the removal of phenol from aquatic environment through the process, called adsolubilization. The SMA was found to be very efficient showing >90% phenol removal from a 50 mg/L phenol bearing solution with only 12 g/L of adsorbent dose. The kinetic study was conducted and the experimental data were analysed by different kinetic models viz., first order, second order, pseudo-first and pseudo-second order models. The details of rate-limiting step were studied. Isotherm study was conducted to find the maximum adsorption capacity and different isotherm models were analysed. The experiments were conducted with both phenol-spiked distilled water and synthetically prepared wastewater.

Keywords: Alumina; Anionic surfactant; Sodium dodecyl sulfate; Adsorption; Surfactant-modified alumina; Phenol; Removal; Adsolubilization

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1. Introduction

Many industrial wastes contain refractory organics, whose removals are rather difficult or impossible by conventional biological treatment processes. Many of the common herbicides and insecticides contain the phenolic moiety as a structural unit and are resistant to biodegradation [1]. Phenolic compounds are popularly used in the preparation of antiseptics, dyes, antirust products, synthetic resins, biocides, photographic chemicals, ink, varnishes, etc. They are present in the wastewater of many industries such as oil refineries, phenol-producing industries, explosive manufacturing industries, pesticide industries, fertilizer industries, pharmaceutical industries, coal conversion process industries, cooking plants, dye manufacturing industries etc. Phenols may also come to the environment through the agricultural runoff and domestic waste [2]. Phenolic compounds are water soluble and highly mobile and hence are likely to reach drinking water sources downstream from discharges, where, even at low concentrations, they can cause severe odour and taste problems and pose risks to populations. Various treatment technologies such as adsorption [1–6], photodegradation [7,8], coagulation flocculation [9], chemical oxidation [10,11], biological process [12,13] etc. are available for the removal of phenol from the wastewater. Biological process is

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particularly suitable to wastewater containing small amount of phenol. Oxidation is another possible alternative when phenol concentration in wastewater is very high. In coagulation and flocculation process large amount of sludge is generated which may cause disposal problems. Among various physicochemical processes, adsorption is widely used for the removal of phenol from wastewater [1,14]. Recently removal of organic contaminants using surfactant-modified solid surface has drawn much attention. This new separation technology has been used during the last two decades [15–17] for the removal of different organic compounds such as dye, nitrophenol, perchloroethylene, aromatic hydrocarbons etc. by different researchers. The main emphasis of those work were to study the physicochemical aspects of the separation process. The engineering aspects of these techniques, however, still remain unexplored. Very recently we have discussed [18] how the surfactant-modified alumina (SMA) could be used efficiently for removing phenol from wastewater while it was present at a very high concentration. To gain further insight into the processes the present work has been devoted to study the kinetics and the mechanism of the phenol removal from wastewater onto SMA. The kinetic study helps in designing engineering related problems and the reaction rate constant value is very important for designing any field reactor, such as completely mixed batch reactor, completely mixed flow reactor, plug-flow reactor etc.

2. Materials and methods

2.1. Reagents

Acridine orange (ACO), phenol, sodium dodecyl sulfate (SDS), glacial acetic acid, toluene was from BDH (AR grade) and was used as received. All other chemicals used in this study were of high purity and used without further purification.

2.2. Instrumentation

A high precision electrical balance (Sartorious GMBH) was used for weighing. Digital pH meter (DHP-500, SICO, India) was used for pH measurements. A spectrophotometer (Thermo Spectronic UV1, UK) was used for absorbance measurement.

2.3. Analytical method

A rapid and reliable solvent extraction spectrophotometric method has been developed for the determination of anionic surfactant (AS) [19]. ACO chemically known as 3,6-bis (dimethylamino) acridine having a colour ($\lambda_{\max} = 467$ nm) has the potential for being used as an ion-pairing agent with AS. Sample solution (10 mL) containing SDS in the range of 0.1–6 mg/L was transferred into a 25 mL separating funnel. ACO (5×10^{-3} M) and glacial acetic acid 100 μL each was added. Then 5 mL of toluene was added to it and shaken for 1 min. The aqueous layer was then discarded and the toluene layer was used for absorbance measurement at 467 nm.

Phenol was determined using spectrophotometric method. Phenol at alkaline condition reacts with potassium ferricyanide and 4-aminoantipyrine to form a red coloured complex. The absorbance of the complex having $\lambda_{\max}$ at 500 nm is a direct measure of phenol concentration.

2.4. Preparation of SMA

Alumina was supplied by SRL, India and used as such without further grinding and sieving. The granulation of neutral alumina is 70–290 mesh ASTM, molecular weight is 101.96 and zero point charge (Z_{pc}) is 9.15.

Preparation of SMA under optimized condition was described earlier [20]. Alumina (200 g) was shaken for 24 h with 2 L of SDS solution having 20,000 mg/L concentration in the presence of NaCl at a dose of 2500 mg/L at pH 4.4 ± 0.1. After shaking, the supernatant was discarded and the alumina was washed thoroughly initially with tap water and finally with distilled water. Then the material (SMA) was dried at 60°C for 24 h. The loading of SDS on alumina was 111.6 mg/g. This SMA was used for the removal of phenol from aquatic environment.

2.5. Experimental studies

The batch experiments were carried out at 25 ± 2°C using synthetic samples of phenol prepared in distilled water and these were shaken in a mechanical shaker at an agitation speed of 150 rpm. The pH of solutions was 6.7 ± 0.1. In all the experiments, the initial phenol concentration selected was 50 mg/L, which is very high but often found in industrial wastewaters [3,4,6]. Experiments were carried out to see the effects of adsorbent dose. The adsorbent dose was varied from 0 to 24 g/L. The shaking time was 1.5 h. In kinetic study, solutions (20 mL) of phenol were shaken for 0–3 h. The adsorbent dose was 12 g/L. Kinetic study was also carried out for initial phenol concentration of 25 and 100 mg/L with same adsorbent dose.

Experiments were carried out to find out the effectiveness of SMA for removing phenol from synthetic wastewater samples under the experimental conditions used for our studies. The wastewater was prepared by dissolving phenol in tap water. The pH of wastewater was 7.0 ± 0.1, turbidity 19 NTU, total dissolved solids 330 mg/L and hardness was 120 mg/L as $CaCO_3$. The phenol concentration in wastewater was 50 mg/L. The effect of adsorbent dose was studied in the range of 0–24 g/L. The shaking time was 1.5 h and the temperature was 25 ± 2°C. Kinetic studies were carried out with adsorbent dose of 12 g/L. The shaking time was varied from 0 to 3 h.

2.6. Analysis of reaction kinetics data

In order to investigate the mechanism of solute adsorption onto the adsorbent, four kinetic models viz., first order reaction model [21] based on the solution concentration, pseudo-first order equation of Lagergren [22] based on the solid capacity, second order reaction model based on the solution concentration [21] and pseudo-second order reaction model of Ho and Mckay [23] based on the solid phase sorption were analysed and a comparison of the best fit sorption mechanism was made.

The linearized forms of different reaction models are shown below.

First order: $\ln C_t = \ln C_o - K_1 t$

Pseudo-first order: $\frac{dq_t}{dt} = K_{S1}(q_e - q_t)$

Second order: $\frac{1}{C_t} - \frac{1}{C_o} = K_2 t$

Pseudo-second order: $\frac{t}{q_t} = \frac{1}{K_{S2} q_e^2} + \frac{1}{q_e} t$

Where

C_t = solute concentration at any time t

C_o = solute concentration at time $t = 0$

q_t = amount of solute adsorbed per unit weight of adsorbent at any time t

q_e = amount of solute adsorbed per unit weight of adsorbent at equilibrium

K_1 = first order reaction rate constant

K_{S1} = pseudo-first order reaction rate constant

K_2 = second order reaction rate constant

K_{S2} = pseudo-second order reaction rate constant

The experimental reaction kinetics data were analysed using the above four kinetic models.

2.7. Determining the pore and film diffusion coefficients

The rate-limiting step in adsorption process is of prime importance and it could be done by using the values of pore and film diffusion coefficients. The procedure to find the pore and film diffusion coefficients has been discussed here.

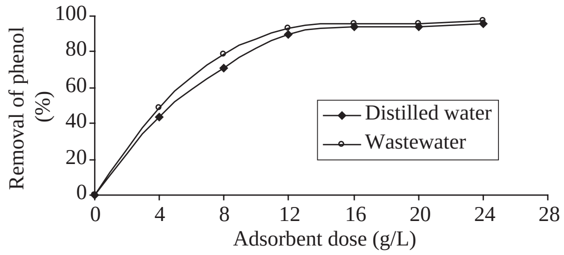

Fig. 1. Effect of adsorbent dose for removal of phenol from distilled water and wastewater.

Assuming spherical geometry of the sorbents and using the first order rate constant, K_1 obtained from first order kinetics profiles, the pore diffusion and film diffusion coefficients were calculated as shown below [24].

$$t_{1/2} = 0.030 \frac{r_0^2}{D_p}$$

$$t_{1/2} = 0.23 \frac{r_0 \delta}{D_f} \frac{\overline{C}}{C}$$

Where

$t_{1/2}$ = half time

r_0 = radius of adsorbent particle (= 0.013 cm for present case)

D_p = pore diffusion coefficient (cm^2/s)

D_f = film diffusion coefficient (cm^2/s)

$\overline{C}$ = concentration of adsorbate on the adsorbent (= 45 mg/L for present case)

C = concentration of adsorbate in solution (= 5 mg/L for present case)

δ = film thickness (cm)

$t_{1/2}$ can be calculated using the following relation [25].

$$t_{1/2} = -\frac{[\ln(0.5)]}{K_1}$$

3. Results and discussions

3.1. Optimum adsorbent dose

Since the adsorbent dose has significant effect on the removal of phenol, the effect of this parameter was studied. Under experimental conditions, the optimum adsorbent dose was found to be 12 g/L. The removal

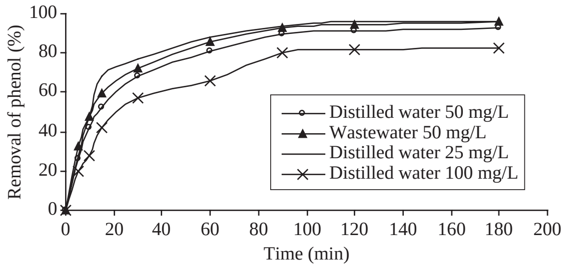

Fig. 2. Removal of phenol with respect to time.

efficiency up to 90% could be achieved under suitable conditions (Fig. 1). The untreated alumina could remove phenol from aquatic environment only up to 6% using an adsorbent dose of 12 g/L, initial phenol concentration of 50 mg/L and shaking time of 1.5 h.

In case of wastewater the initial phenol concentration was kept at 50 mg/L. The effect of adsorbent dose was studied using 0–24 g/L of adsorbent. The shaking time was 1.5 h and the temperature was 25 ± 2°C. It was very interesting to observe that the removal of phenol was more in wastewater in comparison to that in distilled water (Fig. 1). The reason might be that, the weak intermolecular forces (e.g., hydrogen bonds) between phenol and water in the wastewater were easily disrupted by the different ions present in the wastewater, and as a consequence the phenol molecules were forced to be solubilized within the surfactant bilayers on alumina. This is analogous to the 'salting-out' of proteins from water containing salts at a high concentration. Thus the removal efficiency of phenol was higher in wastewater than that in phenol-spiked distilled water.

3.2. Sorption kinetic

In batch experiments, kinetic study is very important to find out the contact time of the adsorbent with adsorbate, and to evaluate the reaction coefficients. The kinetic study was conducted with optimum adsorbent dose of 12 g/L for both phenol-spiked distilled water with varying concentrations (25, 50, 100 mg/L of phenol) and wastewater (50 mg/L of phenol). The shaking time was varied from 0 to 3 h. Fig. 2 showed the removal of phenol with respect to time. The rate of adsorption was very rapid initially and equilibrium time was found to be 1.5 h for phenol-spiked distilled water for all concentrations. The equilibrium time for wastewater was found to be slightly shorter than that obtained for phenol-spiked distilled water.

Reaction kinetics data for the removal of phenol by SMA were analysed using four reaction models as described in Section 2.6. The equations and values of determining coefficients (r^2) of the linear fit lines for first order, pseudo-first order, second order and pseudo-second order reaction models were shown in Table 1. From the r^2 values of the best fit linear lines, it was clear that the reaction followed the pseudo-second order kinetics best among the other models. The basic assumption of pseudo-second order reaction model is that the process following this model is based on chemical adsorption or chemisorption [23]. Our observations suggest that rate-limiting step for the phenol removal by SMA was chemisorption. The value of reaction constant was found to be 0.01 and 0.013 g/mg min for phenol-spiked distilled water and wastewater, respectively. The reaction constant for wastewater was higher than that for distilled water, which indicated that the reaction was faster for wastewater than distilled water. The reaction constant, so found, could be used for the design of different types of batch reactors generally used in the field. The pseudo-second order kinetic model could be used for the rough estimation of the adsorption capacity of phenol uptake by SMA. The adsorption capacity was found to be 5.5 mg/g, which was slightly lower than the adsorption capacity calculated from the isotherm study as discussed in Section 3.4.

3.3. Rate-limiting step

In adsorption process, the rate of reaction is of prime importance and an understanding of the rate-limiting step greatly aids in the selection of

Table 1
Equations and r^2 values of the linear fit lines of kinetics models for phenol removal by SMA

Model	Sample	Equation of linear fit line	r^2
First order kinetic model	Distilled water	$\ln C_t = 3.593 - 0.0201t$	0.9449
	Wastewater	$\ln C_t = 3.534 - 0.0232t$	0.9536
Pseudo-first order kinetic model	Distilled water	$\ln(q_1 - q_t) = 1.133 - 0.0371t$	0.9751
	Wastewater	$\ln(q_1 - q_t) = 1.112 - 0.0401t$	0.9715
Second order kinetic model	Distilled water	$\frac{1}{C_t} - 0.014 = 0.0018t$	0.9806
	Wastewater	$\frac{1}{C_t} - 0.005 = 0.0029t$	0.9780
Pseudo-second order kinetic model	Distilled water	$\frac{t}{q_t} = 3.434 + 0.185t$	0.9995
	Wastewater	$\frac{t}{q_t} = 2.705 + 0.18t$	0.9996

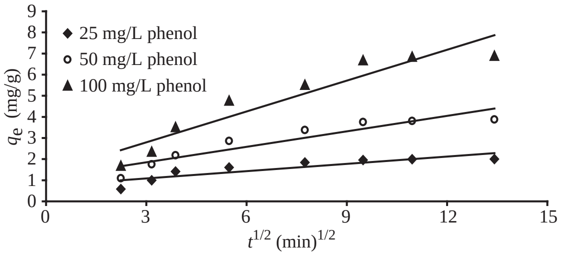

Fig. 3. Plots of phenol adsorption vs. square root of time for various initial phenol concentrations.

adsorber configuration [24]. The rate-limiting step was determined here in two different ways – using first order kinetics data and using data from the effect of initial concentration.

The first order kinetics data could be used for the determination of pore and film diffusion coefficients as discussed in Section 2.7. The value of K_1 for phenol-spiked distilled water was found to be 1.206 h^{-1}. Using the value of K_1, the value of $t_{1/2}$ was calculated and it was found to be 0.5747 h. Assuming spherical geometry of the sorbents, $\delta = 0.001$ cm [24] and using calculated values of $t_{1/2}$, the film diffusion coefficients (D_f) and pore diffusion coefficients (D_p) were calculated and these were 4.68×10^{-5} cm^2/s and 8.82×10^{-6} cm^2/s respectively. According to Michelson [26] in sorption process, the film diffusion to be the rate-limiting step, the value of D_f should be in the range of 10^{-6}–10^{-8} cm^2/s, whereas for pore diffusion to be rate limiting, the value of D_p should be in the range of 10^{-11}–10^{-13} cm^2/s. In the present study, the calculated values of diffusion constants do not lie in the above specified ranges.

The data of kinetic study conducted for different initial concentrations could be used for the determination of rate-limiting step. Kinetic study was conducted with initial phenol concentration of 25, 50 and 100 mg/L using an adsorbent dose of 12 g/L. Weber and Morris [27] reported that if film diffusion is involved in the process then a plot of adsorbate uptake vs. the square root of time would result in a linear relationship and the film diffusion would be the rate-limiting step if this line passes through the origin. As indicated in Fig. 3, the results could be represented by such a linear relationship but the line did not pass through the origin. This indicated that film diffusion was involved in the process, but it was not the rate-limiting step.

Moreover, the agitation speed had no effect on phenol removal by SMA. The effect of agitation speed was studied in the range of 90–210 rpm. The initial phenol concentration was 50 mg/L, pH 6.7 ± 0.1 and adsorbent dose 12 g/L. It was found that the agitation speed in the range of 90–210 rpm had no effect on the removal process. This also indicated that the film diffusion was not rate limiting. Otherwise, increase/decrease in agitation speed would also increase/decrease the removal efficiency.

Hence from the above observations, it could be concluded that the film and pore diffusions had no effect on the removal of phenol by SMA. Thus the film and pore diffusion were not rate-limiting steps in the present case. Actually the removal of phenol occurred in the surfactant bilayer through sorption. The kinetic study showed that it followed pseudo-second order model indicating chemisorption as the rate-limiting step. From the observations it can be suggested that the removal of phenol from wastewater could effectively be done by both batch and continuous mode. Hence, any configuration of reactors containing SMA can be used in the actual field for the removal of phenol by SMA.

3.4. Isotherm study

The adsorption isotherm defines the equilibrium state of the process. Several models have been developed to define the adsorption isotherm [28]. Among

Table 2
of Langmuir and Freundlich isotherm model for the phenol removal by SMA from distilled water spiked samples

Model	Parameters	Values
Langmuir isotherm model	Equation	$\frac{1}{q_e} = 0.731\,(1/C_e) + 0.1505$
	Maximum adsorption capacity, $q_{\max}$ (mg/g)	6.64
	Constant related to energy of sorption system, b	0.2060
	r^2	0.9476
Freundlich isotherm model	Equation	$\ln q_e = 0.3595 \ln C_e + 0.5532$
	Freundlich constant related to adsorption capacity, k_f [(mg/g)(L/mg)$^{1/n}$]	1.7388
	Adsorption intensity, $1/n$	0.3595
	r^2	0.8925

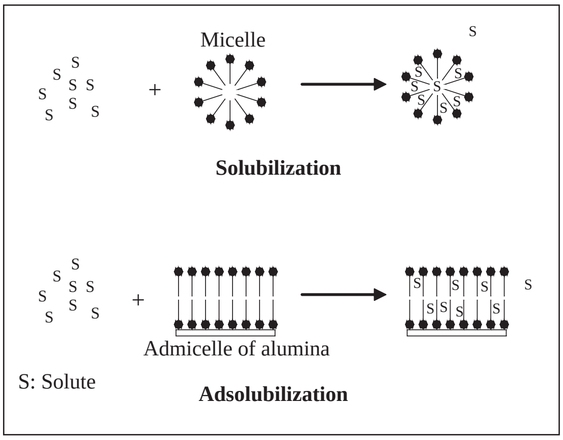

Fig. 4. Schematic diagram showing solubilization and phenol adsolubilization in SMA.

them Langmuir isotherm model and Freundlich isotherm model were used in the present study.

The sorption isotherm was studied at 25 ± 2°C with initial phenol concentration of 50 mg/L. The dose of SMA was varied from 0 to 24 g/L. The shaking time was 1.5 h. The experimental data were fitted with Langmuir and Freundlich isotherm models. Both the isotherm plots showed a linear pattern, with Langmuir isotherm fitting more accurately compared to Freundlich isotherm as compared from their r^2 values. The values of the constants for both the models were calculated and shown in Table 2. The adsorption capacity of SMA for the removal of phenol was found to be 6.64 mg/g. The adsorption capacities of palm seed coat carbon [6], wood charcoal [3], activated carbon [5] and bentonite [4] were 7, 3.2, 58, 0.7 mg/g respectively. The adsorption capacity of SMA is comparable with other adsorbents except activated carbon.

3.5. Mechanism of phenol removal by SMA

Adsorption of AS on positively charged alumina surface occurs due to electrical attraction between positive surface and anionic head groups of AS and also due to interaction between the long hydrocarbon chains of surfactant molecules (tails) to form bilayer structure [29]. This bilayer structure is analogues to micelle and termed as admicelle. These admicelles can solubilize organic molecules within its structure in the same manner that micelles can do. In the present study, the phenol molecules are extracted from water environment and solubilized in the admicelles of AS formed on the surface of alumina. Thus the removal of phenol by SMA from water becomes possible. This phenomenon is called adsolubilization. The solubilization of organic solutes from water environment into micelle and adsolubilization of phenol from wastewater into admicelle had been illustrated in Fig. 4. It was observed that the removal was very much dependent on the surfactant coverage on the alumina surface. As the coverage increases, more number of phenol molecules could be accommodated in the admicelle, which resulted in increased phenol-removal efficiency.

4. Conclusion

From the present study it is found that the SMA can be used very efficiently for the removal of phenol from the water environment even when present at a very high concentration. Also a short equilibrium time is required for the removal of phenol in this case. It was found that the removal of phenol followed the pseudo-second order reaction model. It was also found that neither film diffusion nor pore diffusion was rate-limiting for this process. It suggests that the removal of phenol from wastewater could effectively be done by both batch and continuous mode and any configuration of reactors containing SMA can be used in the actual field for the removal of phenol. The kinetic analysis showed that the phenol removal by SMA takes place via chemisorption as the rate-limiting step. Isotherm studies showed that Langmuir isotherm fitted more accurately compared to Freundlich isotherm. The maximum adsorption capacity ($q_{\max}$) was found to be 6.64 mg/g. The removal of phenol from wastewater is better than that from distilled water.

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