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Modeling of the Temperature Variation Effects on the Polymerization Reactions of Phenolics on Granular Activated Carbon

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ABSTRACT

The effect of temperature variation on the kinetics of adsorption and adsorption–reaction combination of phenol and *o*-cresol on granular activated carbon (GAC) was evaluated throughout this study. Batch experiments were performed under oxic and anoxic conditions at temperatures of 8, 21, and 35°C. The results showed that the equilibration time for physical adsorption increased with the decrease in temperature and occurred in the time range of 7.5–11 days for the adsorption–reaction combination (oxic cases). The polymerization reactions lagged by about 10 hours under all of the temperature values. Diffusivity coefficients in the anoxic cases increased proportionally with temperature, with the highest difference between oxic and anoxic diffusivities at a temperature 35°C. The homogeneous surface diffusion model (HSDM) accurately predicted the anoxic batches and deviated from the oxic data. Temperature increases the adsorption and polymerization reaction rates, and thus the predictivity of the HSDM for both oxic and anoxic batches improves. Under controlled dissolved oxygen (DO) and temperature conditions, the isotherm and batch equilibrium capacities matched with a maximum deviation of 4%. The effect of temperature on the rate of the DO-induced enhancement was found to follow a self-retardant reaction model. The developed model showed strong predictive capability for the temporal formation of polymers.

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INTRODUCTION

Adsorption on activated carbon (AC) is a useful and effective process for the treatment of industrial and hazardous wastewater and for advanced treatment of secondary effluents. However, adsorption on AC is a relatively costly process. This has motivated research on the operational parameters that affect its adsorptive capability.

Dissolved oxygen (DO) was found to enhance the adsorptive capacity of AC for aromatics in general and phenolics in particular (1–8). This enhancement phenomenon was attributed to polymerization reactions taking place on the granular activated carbon (GAC) surface in the presence of DO (3, 6). Abuzaid et al. (8) identified two dimers of phenol, 2-dihydroxy-1,1-biphenyl and 4-phenoxyphenol, as products of such reactions.

Temperature variation is an important factor affecting AC adsorption. Since adsorption is an exothermic process, adsorption capacities should increase with the decrease in temperature. This fact was supported by Maqsood and Benedek (9) and Alben et al. (10). The effect of temperature on the DO-induced polymerization reactions of phenolics on GAC was studied by Grant and King (1). They found that higher temperature enhances the polymerization reaction and hence increases the adsorption capacity. In their experimental scheme, pH and temperatures effects were not separated from the effect of DO. Extreme pH values of 12 and 1.8 at a solution temperature of 80°C were studied. However, those conditions are unrealistic in environmental engineering applications. Recently, Nakhla et al. (6) studied the effect of temperature on the enhancement of GAC adsorption capacity. They conducted isotherm studies on phenol and *o*-cresol at neutral pH and temperature values of 8, 21, and 35°C under oxic and anoxic conditions. In the aforementioned study, it was found that physical adsorption, measured by the anoxic capacity, decreases with the increase in temperature. Uptake enhancement due to polymerization was highest at a temperature of 35°C. Oxic isotherms capacities were found to be relatively independent of temperature, thus suggesting that the positive and adverse impacts of temperature on chemical reactions and adsorption, respectively, tend to balance.

The studies discussed earlier were related to adsorption and reaction equilibrium. However, the effect of DO on the adsorption kinetics of phenolics was investigated by Vidic and Suidan (3). They found that DO affects the kinetics of *o*-cresol adsorption on GAC in batch experiments, resulting in two different uptake curves and diffusivity coefficients. Abuzaid and Nakhla (7) studied the kinetics of phenol adsorption on GAC under four different DO levels. Equilibrium was attained only after 48 hours in the anoxic batches while taking about 14 days in the oxic batches, thus indicating that polymerization is the limiting step in the overall uptake rate while uptake progresses more slowly in the presence of DO, attested by an equilibration time of 14 days.

The homogeneous surface diffusion model (HSDM) has been used for predicting kinetics in batches and columns. The mathematical formulation of the HSDM is available in the literature (11) and will be presented later in this paper. Discrepancies between isotherm capacities and those attained in the kinetics experiments have been reported (12–16). They were attributed to adsorption irreversibility and to a continuous decrease in the adsorbate concentration during an isotherm experiment (12). As a result of the aforementioned discrepancy, serious problems were experienced in attempting to use diffusion models to predict GAC adsorbate–adsorbent systems. Crittenden and Weber (13) had to adjust the adsorptive capacity of AC as given by the adsorption isotherm in order to fit column breakthrough data. Furthermore, they assumed the ratio of the capacities given by that new pseudoisotherm and the isotherm obtained using the standard bottle-point technique to be constant. Later, Liu and Weber (14) concluded that only column studies can be used to determine single-solute adsorption isotherms that would permit accurate prediction of breakthrough curves.

The main objective of this research was to study the effects of temperature on the kinetics of phenol and *o*-cresol uptake under oxic and anoxic conditions. Batch kinetic experiments were conducted at neutral pH and temperature values of 8, 21, and 35°C. The predictive capability of the HSDM for the kinetics of physical adsorption and polymerization reactions was examined. The effect of temperature on the equilibrium capacities attained in isotherm and batch experiments under controlled environment was also investigated. Furthermore, the effect of temperature on the kinetics of the polymerization reactions was simulated using self-retardant reaction model.

EXPERIMENTAL

The 10 × 16 fraction (geometric mean diameter of 0.156 cm) of F-400 GAC supplied by Fisher Scientific, USA, in 10 × 40 US mesh size, was used for this study. The physical properties of F-400 GAC are presented elsewhere (17). The selected carbon size was washed several times with deionized water to remove all fines, then dried at 110°C for 1 day, and stored in a desiccator until use.

The experimental procedures for the study of the effects of temperature on the adsorption isotherms were reported by the authors (6) and, therefore, are not repeated here. Single-solute stock solutions (1000 mg/L each) of phenol and *o*-cresol were prepared and subsequently buffered with KH_2PO_4 to maintain neutral pH. The rate experiments were conducted

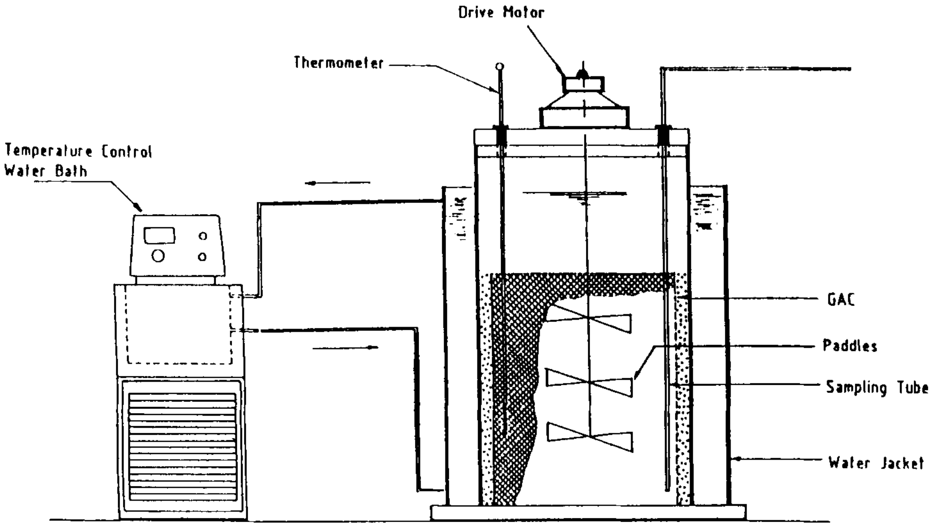

FIG. 1 Schematic of batch reactor.

in 5-L volume completely mixed finite-bath reactors of the type shown in Fig. 1 (7) with a liquid volume of 4.5 L. GAC particles were trapped in a basket inside the reactor to increase external mass transfer by maximizing the fluid velocity relative to that of GAC. One anoxic (zero level of DO) and one oxic (DO around 30 mg/L) batches were simultaneously run at neutral pH and temperature values of 8, 21, and 35°C at identical mixing conditions, initial sorbate, and GAC concentrations. The aforementioned temperature values were controlled with water circulating from temperature-controlled water baths surrounding water jackets.

The sorbate uptake was monitored with time until equilibrium was attained. After being filtered, samples were analyzed using a Spectronic 21 spectrophotometer (Bausch and Lomb Model UV-D) at a wavelength of 270 nm for phenol and *o*-cresol. A computer search for the values of k_f (the external mass transfer coefficient) and D_S (the surface diffusivity) that best fit the HSDM model to the experimental data accounted for the mass of adsorbate removed by sampling.

RESULTS AND DISCUSSION

Freundlich Isotherm

The effect of temperature on the adsorption equilibria of phenol and *o*-cresol on GAC was investigated and reported by the authors (6). Therefore, it is not going to be discussed here. However, the isotherm parameters are presented in this paper because they are going to be used later in the analysis. The parameters (k and $1/n$) of the Freundlich isotherm ($q_e = kc_e^{1/n}$) at different temperature and DO conditions for phenol and *o*-cresol are presented in Table 1.

The Homogeneous Surface Diffusion Model (HSDM)

The homogeneous surface diffusion model (HSDM) takes the form (11)

$$\frac{\partial q}{\partial t} = D_S \left[\frac{\partial^2 q}{\partial r^2} + \frac{2}{r} \frac{\partial q}{\partial r} \right] \tag{1}$$

with the initial condition (Eq. 2) stipulating no presence of adsorbate in the particle, while the boundary conditions (Eqs. 3 and 4) respectively stating that the flux at the center of particle is always equal to zero because of symmetry, and that the total mass of adsorbate in the system comprising the adsorbent and the liquid is constant.

$$q(r, 0) = 0 \tag{2}$$

$$\frac{\partial q(0, t)}{\partial r} = 0 \tag{3}$$

$$4\pi r_S^2 \rho_p n \int_0^t \left[-D_S \frac{\partial q}{\partial r} \right] dt = V_l(C_0 - C) \tag{4}$$

TABLE 1
Freundlich Constants for Phenol and *o*-Cresol Isotherms (6)

Adsorbate	DO designation	Temperature (°C)	k [(mg/g) (L/mg)$^{1/n}$]	95% Confidence interval (k)	$1/n$	95% Confidence interval ($1/n$)	R^2 [a]
Phenol	Anoxic	8	41.8	39.9–43.7	0.21	0.200–0.219	0.96
	Oxic	8	96.7	93.6–99.8	0.16	0.143–0.177	0.95
	Anoxic	21	31.7	30.4–33.0	0.24	0.236–0.244	0.99
	Oxic	21	83.5	80.6–84.8	0.18	0.167–0.193	0.97
	Anoxic	35	19.0	17.8–20.2	0.25	0.237–0.263	0.94
	Oxic	35	57.3	54.2–60.4	0.2	0.192–0.208	0.96
o-Cresol	Anoxic	8	104.0	101.2–106.8	0.18	0.166–0.208	0.96
	Oxic	8	197.5	193.4–201.6	0.13	0.121–0.139	0.95
	Anoxic	21	88.6	86.9–90.3	0.19	0.182–0.198	0.96
	Oxic	21	190.4	186.2–194.6	0.13	0.114–0.146	0.99
	Anoxic	35	76.1	74.2–78.0	0.20	0.188–0.212	0.97
	Oxic	35	175.0	171.4–178.6	0.14	0.126–0.154	0.94

[a] R^2 is the coefficient of determination.

where q is the carbon loading at any time (mg adsorbate/mg adsorbent), D_S is the surface diffusion coefficient (L^2/T), r is the distance from the center of the particle (L), r_S is the radius of the particle (L), t is the time (T), ρ_p is the particle density (mg/cm), n is the number of particles, V_l is the volume of liquid (L^3), and C_0 and C are, respectively, the sorbate concentration initially and at any time ($mg/L^3/T$).

The influence of temperature variation on the kinetics of adsorption and adsorption–reaction combination was evaluated by simultaneously running six batches for each adsorbate, one anoxic batch, and one oxic (30 mg/L DO) at temperature values of 8, 21, and 35°C. The anoxic and oxic batch kinetic data for phenol and *o*-cresol at 8, 21, and 35°C are shown in Figs. 2(a–c) and 3(a–c), respectively. The variation of the apparent surface diffusivity, estimated from the HSDM fit with temperature, is presented in Table 2.

Temperature variation affects the kinetics of adsorption (anoxic case) as well as adsorption–reaction combination (oxic case). The data of Figs. 2 and 3 combined with those in Table 2 show that equilibration time for physical adsorption increases with decreases in temperature, as attested by the drop in D_S (Table 2). In general, the kinetics of the adsorption–surface reaction combination were fastest at 21°C and uptake was highest at 8°C. Although the net anoxic uptake increased with temperature drop, emphasizing the exothermic nature of adsorption, the rate of adsorption increased significantly with temperature, particularly in the 8–21°C range. The HSDM model described the anoxic data very well, with the fit improving with temperature. On the other hand, the HSDM underpredicted the initial data and overpredicted the later data. It must be emphasized that the actual phenomena involved are diffusion of sorbate followed by limited reaction. The use of the HSDM model to represent this combined mechanism is a pseudoapproach using an apparent diffusivity mechanism. Thus, the apparent surface diffusivity can be perceived as the net combined effect of physical diffusion and surface reaction. Since the HSDM is forced to fit the equilibrium data in an adsorption–reaction system, and uptake due to surface reactions is slow as evidenced by the prolonged equilibration time for oxic batches, the apparent surface diffusivity is lower than in an adsorption system with comparable final uptake but higher than the rate constant for a surface reaction dominated system. Initially, sorbate must diffuse and adsorb on GAC before reaction can take place. This is corroborated by the concurrence of the kinetic data in various batches for the initial 10 hours (Figs. 2 and 3). Thus, sorbate adsorption limits the overall uptake initially and therefore proceeds at a faster rate than predicted by the HSDM. Consequently, in the latter part of the kinetic experiment, the surface reaction rate limits the uptake and thus the HSDM underpredicts the data.

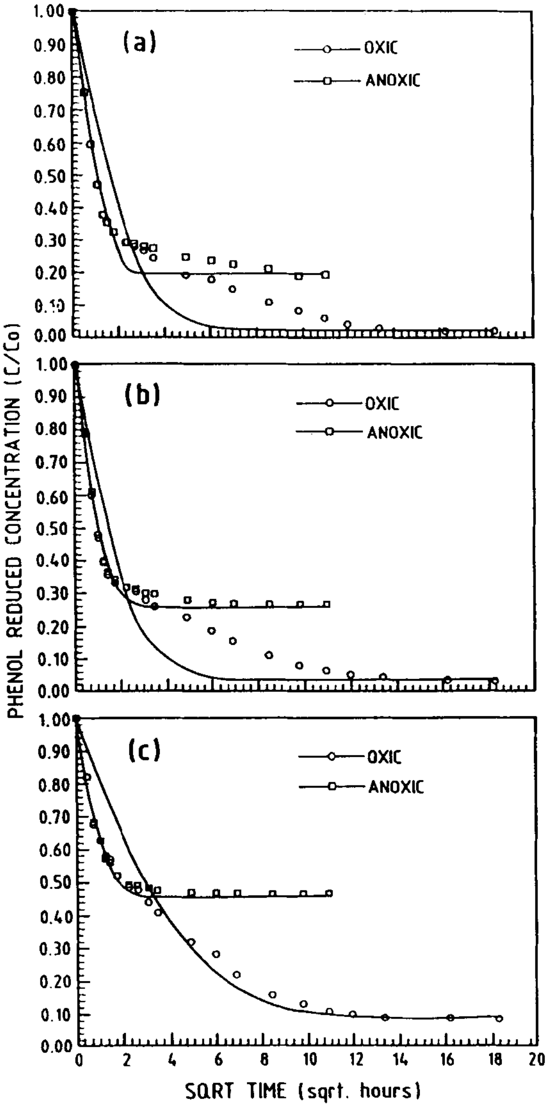

FIG. 2 Closed batch kinetic data with HSDM fit for phenol at different temperatures: 8°C (a), 21°C (b), 35°C (c).

The fit of the surface reaction-controlled part of the kinetic curve improved substantially with temperature as is apparent in Figs. 2(c) and 3(c). It is recognized that temperature enhances both surface and diffusion reaction rates. The improved conformance of the uptake curve under oxic

TABLE 2
Apparent HSDM Surface Diffusivities of Phenol and *o*-Cresol

Adsorbate	DO designation	T (°C)	D_S (cm^2/s)
Phenol	Anoxic	8	4.9×10^{-8}
	Oxic	8	6.7×10^{-9}
	Anoxic	21	6.3×10^{-8}
	Oxic	21	7.6×10^{-9}
	Anoxic	35	8.8×10^{-8}
	Oxic	35	2.9×10^{-9}
o-Cresol	Anoxic	8	4.8×10^{-8}
	Oxic	8	1.1×10^{-8}
	Anoxic	21	0.3×10^{-8}
	Oxic	21	1.4×10^{-8}
	Anoxic	35	8.7×10^{-8}
	Oxic	35	9.9×10^{-9}

conditions in the HSDM model at high temperatures merely suggests that the surface reaction rate is more strongly dependent on temperature than is the diffusion rate.

In order to emphasize the findings discussed earlier about the effect of temperature variation on oxic and anoxic uptakes, D_S values were plotted versus temperature for phenol and *o*-cresol (Fig. 4). It is clear from the figure that for anoxic uptake (physical adsorption), D_S for phenol and *o*-cresol increases with temperature. For the oxic condition, D_S was highest at 21°C. This is thought to be due to the fact that temperature has two opposite effects on DO. While the increase in temperature reduces oxygen solubility, it increases its diffusivity. Furthermore, under the oxic condition, D_S values were always lower than in the anoxic case, which caused longer equilibration times. However, the difference between D_S values in the oxic and anoxic conditions increased with temperature, implying that the rate of the reaction increases with temperature.

The activation energies were estimated by the Arrhenius equation for phenol and *o*-cresol, respectively, using the anoxic surface diffusivities at the temperatures studied. The equation is

$$D_S = D_{SO} \exp - \frac{-E_a}{2.3R_gT} \tag{5}$$

which can be linearized as:

$$\log(D_S) = \log D_{SO} - \frac{-E_a}{2.3R_gT} \tag{6}$$

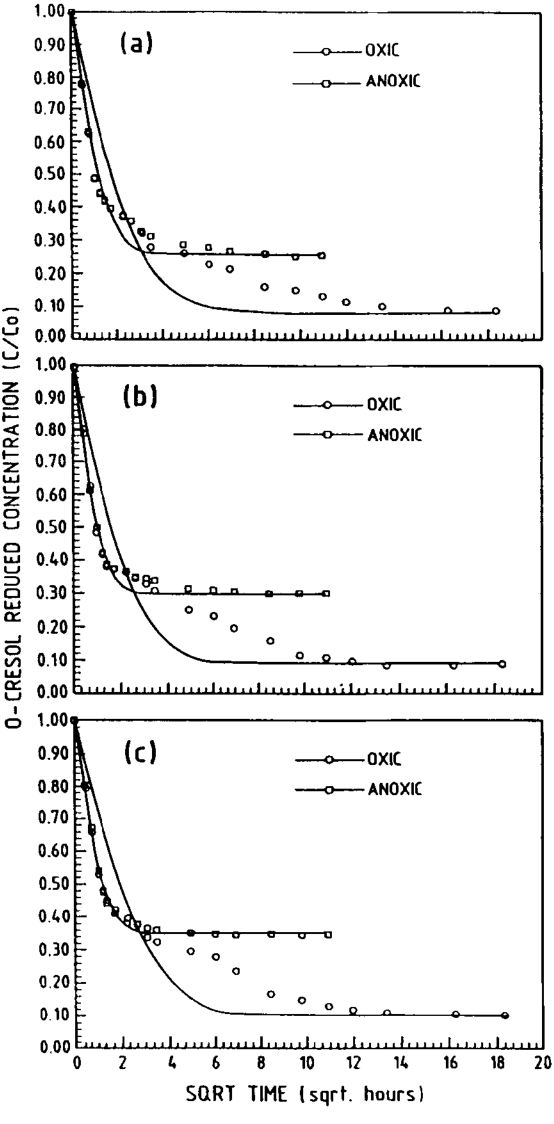

FIG. 3 Closed batch kinetic data with HSDM fit for *o*-cresol at different temperatures: 8°C (a), 21°C (b), 35°C (c).

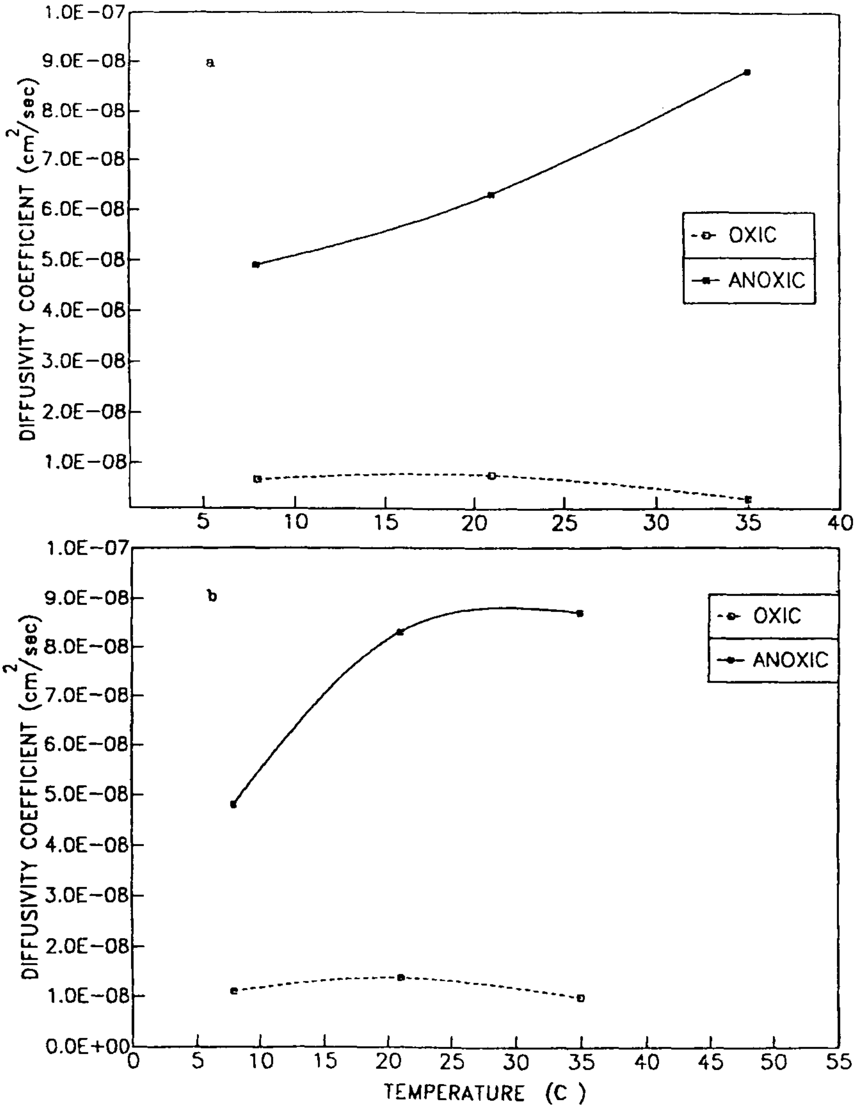

FIG. 4 Relationship between HSDM apparent diffusivities and temperature for phenol (a) and *o*-cresol (b).

where, D_S is the diffusivity coefficient, D_{S0} is the intercept, R_g is the universal gas constant = 8.31 J/(mole·k), E_a is the activation energy, and T is the temperature in Kelvin. Figure 5 presents the relation in Eq. (6) for phenol and *o*-cresol, from which the intercepts were −4.48 and −4.05 for phenol and *o*-cresol, respectively. The activation energies were calculated from the slopes and found to be 15238.7 and 15355.4 J/mole for phenol and *o*-cresol, respectively. Actually, for the anoxic condition, Fig. 4 shows some difference in the trends of D_S versus temperature for phenol and *o*-cresol which is not easy to explain. However, the close values of the aforementioned activation energies for phenol and *o*-cresol reflect similar responses for the kinetics to temperature variation.

The isotherm capacities and those attained in the kinetic batches were calculated at equilibrium for the different temperature and DO conditions; they are presented in Table 3. The isotherm capacities were calculated using the Freundlich model incorporating parameters pertinent to the environmental conditions (DO, temperature) which were presented in Table 1. From Table 3 it is clear that at neutral pH and at each of the temperature and DO conditions, the isotherm and batch capacities are similar, with a

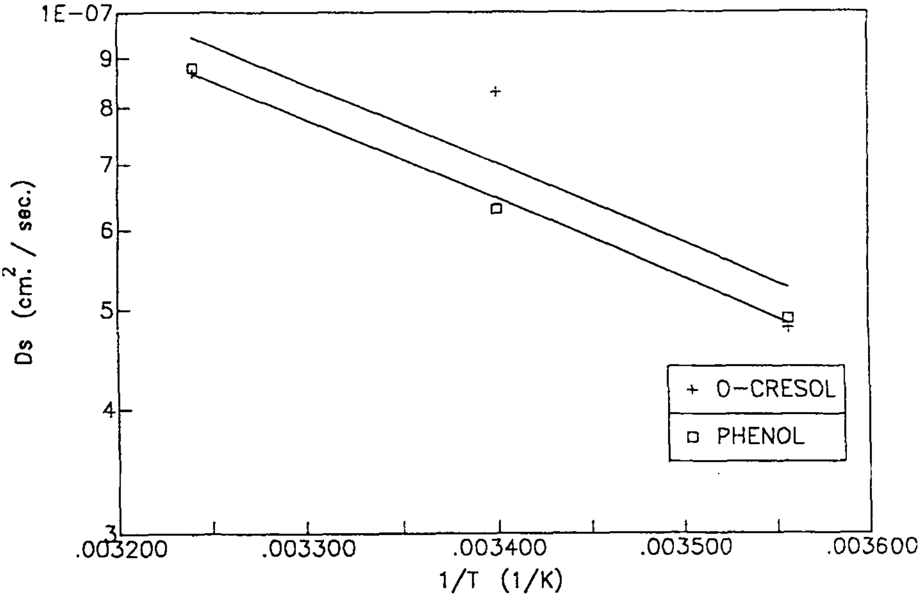

FIG. 5 Relationship between diffusivities and temperature for phenol and *o*-cresol along with lines of best fit.

maximum difference of 5%. This finding suggests that the discrepancy problem reported in the literature (12–16) can be eliminated by running both the isotherm and the continuous adsorption systems under exactly the same operational conditions.

Self-Retardant Reaction Model

Based on the fact that the DO-induced enhancement phenomenon in the adsorption capacity of phenolics on GAC is due to the formation of polymers of the sorbate on the carbon surface (3, 6–8), it can be postulated that such a conversion would allow more solute to migrate to the adsorbent. Therefore, at any time, the formation of such polymers is related to the difference between the oxic and anoxic uptakes. In order to investigate the effect of temperature variation on those polymerization reactions, the differences between the oxic and anoxic uptakes (*P*) were calculated for the curves of Figs. 2 and 3 and presented in Fig. 6 for phenol and *o*-cresol.

The polymerization reactions are controlled by either the limited and nonrenewable amounts of DO in the adsorption batches and/or by the limited GAC surface. Due to the complexity of the problem, in this study a simplified zero-order self-retardant reaction model (18) is proposed:

$$\frac{dp}{dt} = \frac{K}{t} \tag{7}$$

which can be solved as

$$P = K*\log t \tag{8}$$

where *P* is the amount of polymers produced determined as the difference between the oxic and anoxic uptakes, *t* is time, greater than 10 hours (lag time), and *K* is the rate constant. Accordingly, *P* was plotted against log *t* and is presented in Fig. 7 for the data shown in Figs. 2 and 3. From Fig. 7 the reaction lag time (about 10 hours) which was discussed earlier is obvious. After the lag time the reaction started and the curves mostly demonstrated a linear relation between *P* and log *t*. The slopes of the lines were calculated for the different temperature values for phenol and *o*-cresol.

Actually, Fig. 7 shows that there were two stages with respect to time for phenol and *o*-cresol. The first stage was the lag time in which the reaction was not occurring (around 10 hours). In the second stage, which continued up to the end of the equilibration time, there was a distinct rate for each of the temperature values and hence the slopes had different values. The slopes of the lines were calculated for the different DO levels

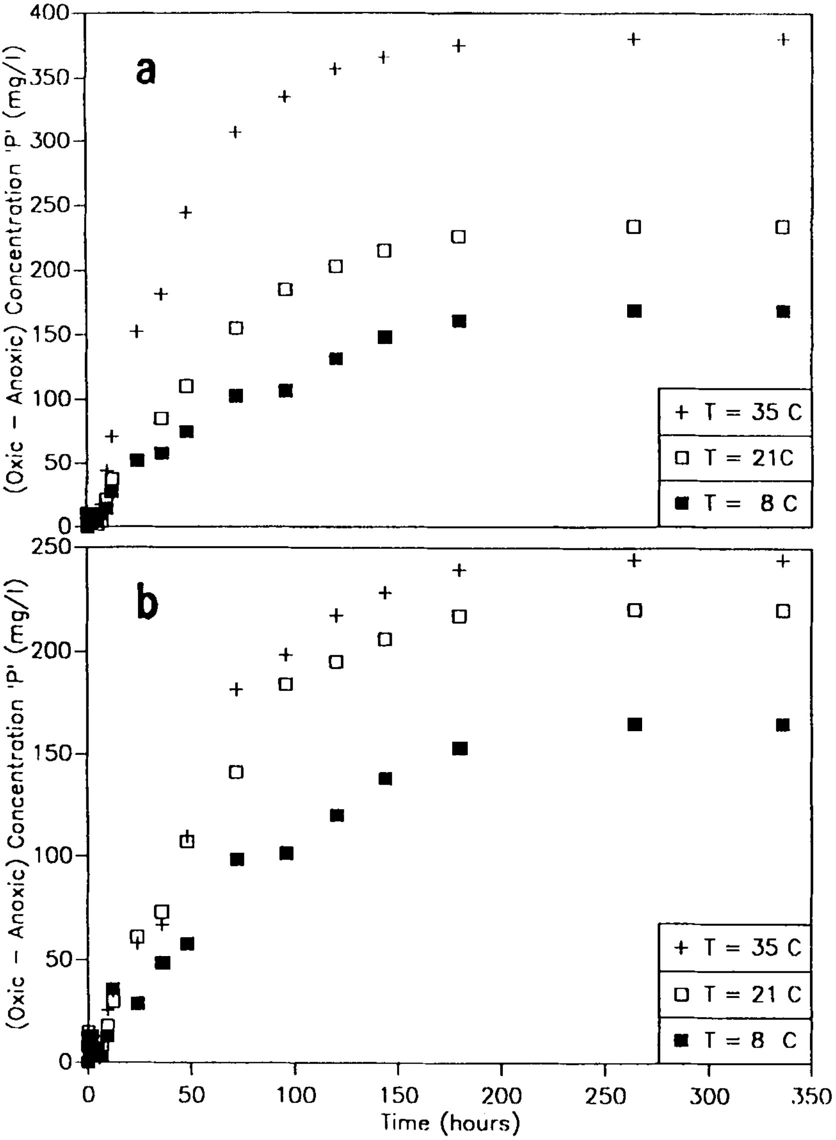

FIG. 6 Relationship between polymerization reactions product *P* and time at different temperature values for phenol (a) and *o*-cresol (b).

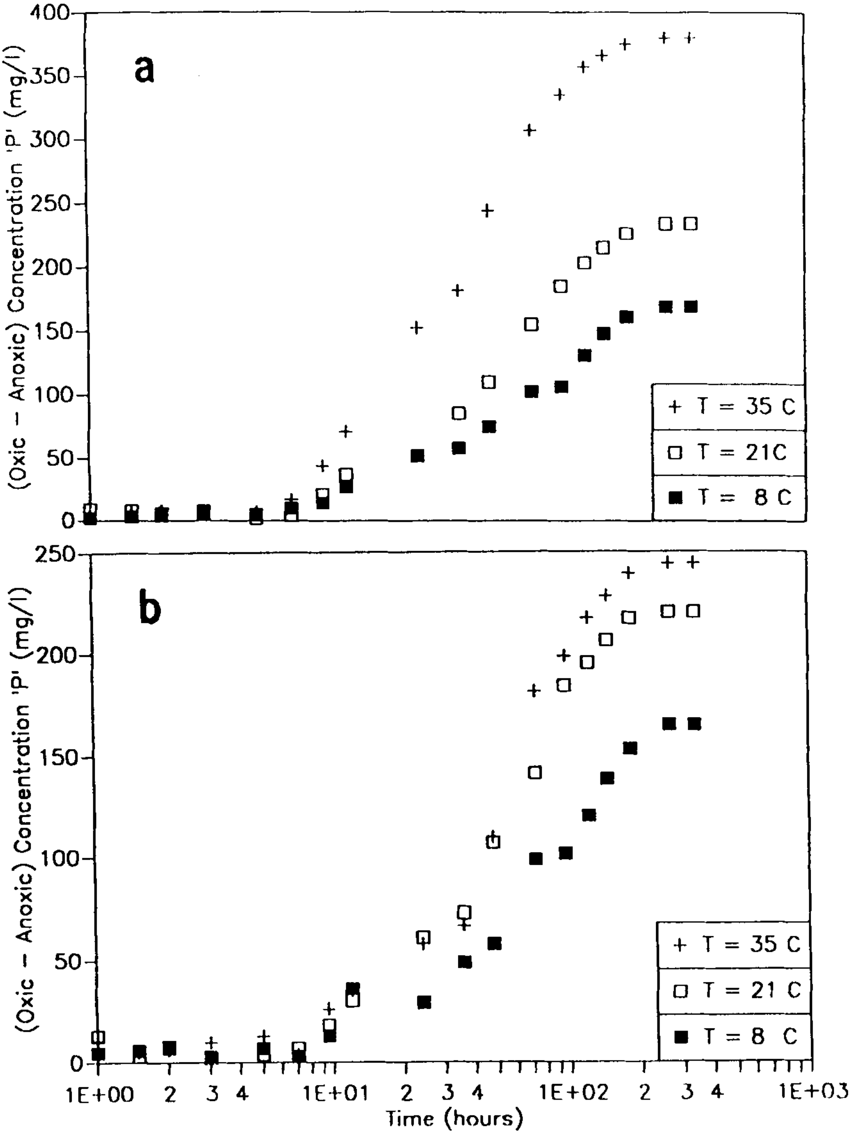

FIG. 7 Relationship between polymerization reactions product *P* and log time at different temperature values for phenol (a) and *o*-cresol (b).

TABLE 3
Equilibrium Isotherm and Batch Capacities under Different Temperature and DO Conditions for Phenol and *o*-Cresol

Adsorbate	DO designation	*T* (°C)	Isotherm capacity (mg/g)	Batch capacity (mg/g)
Phenol	Anoxic	8	125.9	127.8
	Oxic	8	157.2	153.1
	Anoxic	21	120.6	116.8
	Oxic	21	153.2	152.9
	Anoxic	35	88.1	84.3
	Oxic	35	138.2	144.0
o-Cresol	Anoxic	8	283.4	276.8
	Oxic	8	352.4	342.8
	Anoxic	21	262.0	262.2
	Oxic	21	337.1	344.6
	Anoxic	35	245.2	244.9
	Oxic	35	334.4	336.8

for both phenol and *o*-cresol. Table 4 presents the rate values, *K*, in the second stage of the polymerization reactions for phenol and *o*-cresol.

In order to test its validity, the proposed rate model (Eq. 8) along with the pertinent constants (Table 4) were used to predict the value of *P* with time under each of the temperature conditions (Fig. 6). Figure 8 depicts the predictability of the model for the data. From Fig. 8 it is clear that the prediction capability of the model for the polymerization of phenol is better than that of *o*-cresol. This might be due to the distinct trend of phenol polymerization under each of the temperature conditions unlike

TABLE 4
Rate Constant Values of Phenol and *o*-Cresol

Adsorbate	*T* (°C)	*K* (mg/L·h)
Phenol	8	62.6
	21	91.1
	35	161.1
o-Cresol	8	58.5
	21	86.8
	35	95.8

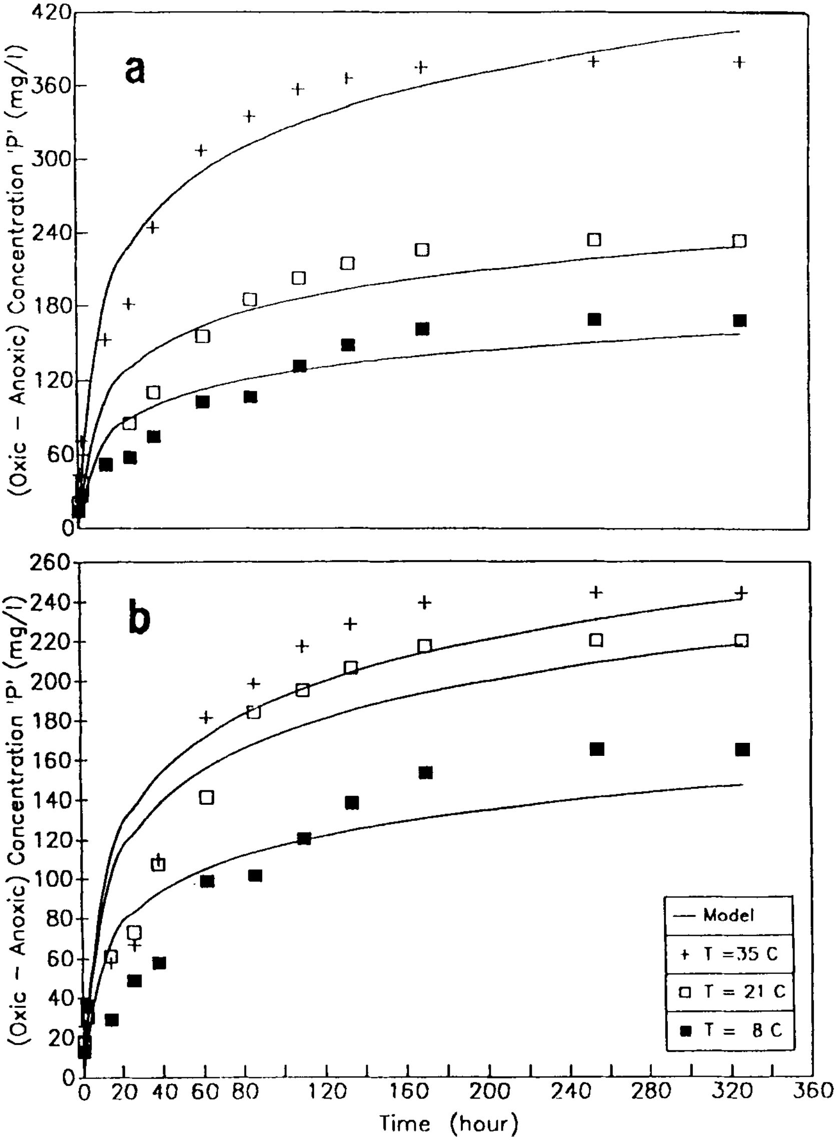

FIG. 8 Closed batch kinetic data for phenol (a) and *o*-cresol (b) along with the prediction lines of Eq. (8).

the case of *o*-cresol in which the data points were close in some stages of the experiment. In fact, the aforementioned data could be fitted much more accurately by a curve-fitting equation. However, Eq. (8) has the physical concept of self-retarding reactions which simulates the polymerization reactions of phenolics on GAC.

We tried to get a relationship between the slopes related to temperature variations and temperature using the nonlinear regression analysis program available in the SAS package at KFUPM (19). The rate constant in Eq. (8) was related to temperature as

$$K = k(T^c) \tag{9}$$

and hence, Eq. (8) can be shown as

$$P = k(T^c)*\log t \tag{10}$$

were T is temperature in °C, k is a constant equals to 16.8 and 29.1 for phenol and *o*-cresol, respectively, and c is the exponent equal to 0.607 and 0.343 for phenol and *o*-cresol, respectively.

Equation (10) can be used to evaluate the extent of polymer formation (or additional capacity) with time at different temperature values. This is very important from a practical standpoint. Since economy is the main factor affecting design and operation of such treatment systems and the initial GAC cost is relatively high, a trade-off between temperature adjustment and the other operational variables can be performed to come up with optimum and cost-effective designs.

CONCLUSIONS

The influence of temperature variation on the kinetics of adsorption and adsorption–reaction combination was evaluated by simultaneously running six batches for each adsorbate, one anoxic batch, and one oxic batch (30 mg/L DO) at temperature values of 8, 21, and 35°C.

Equilibration time for physical adsorption increases with the decrease in temperature. However, for the oxic conditions in which polymerization reactions take place, the equilibration time occurs in the time range of 7.5–11 days. Diffusivity coefficients in the anoxic cases increase proportionally with temperature while the highest difference between oxic and anoxic diffusivities was at 35°C.

Anoxic uptakes were predicted accurately by HSDM under all of the temperature values, but this was not the case for the oxic data. Due to the fact that temperature increases the adsorption and polymerization reaction rates, the predictivity of the HSDM for both oxic and anoxic batches improves. The polymerization reactions lagged by about 10 hours under all of the temperature values.

The effect of temperature variation on the rate of DO-induced enhancement was modeled with a simplified form of the self-retardant reaction model. The aforementioned model demonstrated good predictive capability for the temporal formation of polymers, particularly for phenol.

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