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Chemical Engineering Communications

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713454788

EFFECTIVE DIFFUSIVITY OF PHENOL IN ACTIVATED CARBON

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To cite this Article Bhatia†, S. K. , Kalam, A. , Joglekar, H. S. and Joshi, J. B.(1990) 'EFFECTIVE DIFFUSIVITY OF PHENOL IN ACTIVATED CARBON', Chemical Engineering Communications, 98: 1, 139 — 154

To link to this Article: DOI: 10.1080/00986449008911566

URL: http://dx.doi.org/10.1080/00986449008911566

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Chem. Eng. Comm. 1990, Vol. 98, pp. 139–154
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Printed in the United States of America

EFFECTIVE DIFFUSIVITY OF PHENOL IN ACTIVATED CARBON

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(Received January 4, 1990; in final form July 18, 1990)

The desorption of phenol from activated carbon in aqueous systems is studied here at various temperatures and adsorbed concentration levels in a stirred batch system. It is found that at sufficiently rapid stirring rates the desorption is controlled by intraparticle mass transfer. The effective diffusivity value is estimated to be $2.1 \times 10^{-10}\,m^2/s$ at 30°C and increases to $6.7 \times 10^{-10}\,m^2/s$ at 80°C, yielding tortuosity values that are essentially temperature independent.

KEYWORDS Effective diffusivity Activated carbon Desorption Phenol
Regeneration of activated carbon.

INTRODUCTION

Adsorption of toxic organic compounds on activated carbon is one of the best proven method for treatment of wastestreams (Robertaccio, 1973). However, irrespective of the carbon used, economic considerations provide a strong incentive for its regeneration and reuse. Desorption forms the principle objective of the regeneration, be it induced thermally, chemically, or by means of solvent.

The present study concerns the regeneration of activated carbon loaded with phenol, an established toxin. Regeneration can be conveniently carried out in aqueous phase by Wet Air Oxidation (Gitchel *et al.*, 1975), or by chemical conversion of phenol into the less adsorbable sodium phenolate by means of sodium hydroxide (Himmelstein *et al.*, 1973). However, the kinetics of the desorption, so crucial to process design, have only been given peripheral attention in the literature (Friendrich *et al.*, 1985). These authors have pointed out the role of intraparticle and external mass transfer on the aqueous phase adsorption and desorption rates for phenol on activated carbon.

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In addition to the above studies of aqueous systems, organic solvents have also been used. Hamilton (1963) reported the use of ethylene dichloride, 1,2-propylene dichlorides, methanol and isopropanol as solvents for desorption. Sutlkno and Himmelstein (1983) studied the desorption with acetone as a solvent in a packed column and stirred reactor, and reported external mass transfer control. Balice (1979) used a variety of solvents for the desorption of phenol on activated carbon. The desorption efficiencies of solvents were related to their physico-chemical properties namely, the surface tension, viscosity, dipole moment and dielectric constant. It was observed that, by increasing the solvent surface tension or viscosity, the desorption efficiency decreases. However, an increase in the solvent dipole moment and dielectric constant enhances the desorption efficiency. Allen *et al.* (1971) have reported the extraction of phenol from activated carbon using chloroform and methanol, and reported that the presence of moisture on activated carbon affects the desorption efficiency. Pahl *et al.* (1973) studied ten different solvents for the desorption of phenol from wet activated carbon, and found that desorption efficiencies were related with the ability of the solvent to form hydrogen bonds with phenol.

Desorption of phenols in boiling water has also been reported (Pawlowski, 1984). Dryden and Kay (1954) studied the kinetics of batch adsorption and desorption of acetic acid on activated carbon. The effective diffusivity was found to be rather low, of the order of 1.66×10^{-11} m^2/s. The low value was explained on the basis of long tortuous path and restrictivity effects associated (Anderson and Quinn, 1974) with small pores.

In this paper, effective diffusivity of phenol has been measured in powdered activated carbon by measuring the rates of desorption. Initially, the mathematical model has been briefly described, the details have been given by Bhatia (1988). The effect of temperature on effective diffusivity and tortuosity has been measured in this paper.

MATHEMATICAL MODEL

The process of the desorption of phenol from the activated carbon particles is accomplished by diffusion through the porous structure of the solid. In order to develop a mathematical model for the process, it is therefore, necessary to assume a suitable model for the porous structure. Following the results of Bhatia (1987), we assume that the solid is comprised of non-overlapping microporous spherical grains of uniform size, r_g. Diffusion through the porous carbon particles is then accomplished by the diffusion through the macropores between the grains and through the micropores within the grains. An estimate of the ratio of the time scales for micropore and macropore diffusion may now be obtained as:

$$\frac{t_{\text{micro}}}{t_{\text{macro}}} = \frac{r_g^2/D_{e\mu}}{R_0^2/D_{eM}} \tag{1}$$

where R_0 is the particle radius, $D_{e\mu}$ is the effective diffusivity in the grains and D_{eM} is the effective macropore diffusivity. For the activated carbon used in this work, R_0 was 487 ± 30 microns. Further, $D_{e\mu}$ and D_{eM} are expected to be the same order of magnitude while r_g is of the order of a fraction of a micron. It is therefore, clear from Eq. (1) that the time scale for micropore diffusion is far less than that for macropore diffusion and consequently the diffusional gradients in the grains may be neglected. Under these conditions, the effective diffusivity may be defined as (Leung and Haynes, 1984):

$$D_e = D_{eM} + (1 - \varepsilon_M)D_{e\mu} \tag{2}$$

where ϵ_M is the macroporosity and in which surface diffusion has been neglected. Assuming spherical particles, the overall transport is represented by the following Fickian relation:

$$\frac{\partial q}{\partial t} = \frac{D_e}{R^2} \cdot \frac{\partial}{\partial R}\left(R^2 \frac{\partial C}{\partial R}\right) \tag{3}$$

Here $C(R, t)$ is the local liquid phase phenol concentration at radial position R, and $q(R, t)$ is the total local phenol content per unit volume given by:

$$q = [\varepsilon_M + \varepsilon_\mu(1 - \varepsilon_M)]C + [S_M + S_\mu(1 - \varepsilon_M)]C_S \tag{4}$$

in which C_s is the internal surface phenol concentration, ε_μ is the microporosity of the grains, s_μ is the micropore surface area per unit volume of the grains and S_M is the macropore surface area per unit particle volume. Assuming local equilibrium between the adsorbed and bulk phenol and linearizing the desorption isotherm over the narrow concentration span of a given experiment

$$C_S = A + KC \tag{5}$$

Substitution of Eq. (5) in (3) and (4) gives:

$$\frac{\partial C^*}{\partial \tau} = \frac{1}{\eta^2} \frac{\partial}{\partial \eta}\left(\eta^2 \frac{\partial C^*}{\partial \eta}\right) \tag{6}$$

where,

$$\eta = \frac{R}{R_0} \tag{7}$$

$$\tau = \frac{D_e t}{R_0^2[(\varepsilon_M + KS_M) + (1 - \varepsilon_M)(\varepsilon_\mu + KS_\mu)]} \tag{8}$$

$$C^* = \frac{C}{C_0} \tag{9}$$

Here C_0 is the initial liquid phase concentration of phenol in the pores. Equation (6) has the following initial condition:

$$C^* = 1 \quad \text{at} \quad \tau = 0 \tag{10}$$

and the following boundary conditions;

$$\frac{\partial C^*}{\partial \eta} = 0 \quad \text{at} \quad \eta = 0 \tag{11}$$

$$C^* = C_L^* \quad \text{at} \quad \eta = 1 \tag{12}$$

in which external mass transfer is assumed rapid and where the dimensionless external phenol concentration satisfies the following mass balance:

$$VC_0 \frac{dC_L^*}{dt} = -D_e \frac{\partial C}{\partial R}\bigg|_{R_0} \cdot \frac{3W}{\rho R_0} \tag{13}$$

in which V is the volume of external liquid and remains practically constant, W is the mass of activated carbon and ρ its bulk density. In dimensionless form, Eq. (13) becomes,

$$\alpha \frac{dC_L^*}{d\tau} = -\left(\frac{\partial C^*}{\partial \eta}\right)_{\eta=1} \tag{14}$$

where,

$$\alpha = \frac{V\rho}{3W[\varepsilon_M + KS_M + (1-\varepsilon_M)(\varepsilon_\mu + KS_\mu)} \tag{15}$$

Equations (6), (10)–(12) and (14) form the basic mathematical model to be solved in order to obtain the temporal change in the external liquid phase concentration (C_L^*) which is measured variable. To simplify the analysis, we introduce the local fractional desorption, given by

$$Q(\eta, \tau) = \frac{1 - C^*(\eta, \tau)}{1 - C_\infty^*} \tag{16}$$

where C_∞^* is the final equilibrium value of C^*. With this definition Eqs. (6), (10) and (11) assume the forms

$$\frac{\partial Q}{\partial \tau} = \frac{1}{\eta^2} \frac{\partial}{\partial \eta}\left(\eta^2 \frac{\partial Q}{\partial \eta}\right) \tag{17}$$

$$Q = 0 \quad \text{at} \quad \tau = 0 \tag{18}$$

$$\frac{\partial Q}{\partial \eta} = 0 \quad \text{at} \quad \eta = 0 \tag{19}$$

while Eq. (14) combines with Eqs. (6), (11) and (12) to yield the following additional boundary condition

$$1 + 3\alpha - 3\alpha Q(1, \tau) = 3\int_0^1 \eta^2 Q(\eta, \tau)\, d\eta \tag{20}$$

which holds at $\eta = 1$. The total measured fractional desorption defined by

$$Q_T = \frac{C_L}{C_\infty} \tag{21}$$

is now given as

$$Q_T = 3\int_0^1 \eta^2 Q(\eta, \tau)\, d\eta \tag{22}$$

Equations (17) to (20) form an attractive formulation of the problem which has an established analytical solution (Crank, 1979). However, this solution is not very convenient to use as it involves the summation of an infinite series, a procedure requiring careful numerical treatment for the series to be truncated after a finite number of terms. Alternately the system of equations may also be solved numerically by the orthogonal collocation technique. The latter route was chosen here since the necessary computer program, previously tested against the analytical solution, had been developed in prior work (Bhatia, 1987) and was readily available.

EXPERIMENTAL

Materials

Granular activated carbon (GAC) obtained from M/s Narbada Valley Chemical Industries Pvt. Ltd., Bombay, was used for the experimental studies. The GAC was crushed and sieved to the desired particle size (diameter of 975 μm) and washed with hot deionised water (prepared by condensing boiler steam) to remove finer particles. Washed GAC was dried at 120°C to remove moisture. This GAC was used for determination of adsorption/desorption isotherms and batch desorption studies. Deionised water was used for the preparation of solutions. Phenol was of analytical reagent grade. The bulk density of GAC was estimated to be 0.607 kg/m^3 by mercury pycnometry, its BET surface area was 1000 m^2/g. The average pore diameter was 46 Å measured by mercury porocimetry.

Adsorption/Desorption Isotherm

The initial concentration of phenol for adsorption isotherm determination was 10,000 mg/L. 100 ml of phenol solution with varied quantity of GAC was taken in a 250 ml Erlenmeyer flask and sealed with stopcock. A blank solution was also kept to check any change in the initial concentration during the adsorption process. The flasks were kept in a temperature controlled orbital shaker. The temperature was maintained within ±1°C of the desired temperature (30–80°C). The time required to reach the equilibrium value was in the range of 8–10 days. After the equilibrium was attained, the contents were filtered and the liquid phase was analysed for phenol content.

The desorption isotherm data was generated by desorption of phenol loaded on GAC into the aqueous phase. The phenol on GAC was loaded by adsorption from phenol solution. After reaching the equilibrium, GAC was separated and the aqueous phase was analyzed. The amount of phenol on GAC was calculated by material balance. Various quantities of the known amount of phenol loaded on GAC were desorbed in 100 ml of deionized water in 250 ml Erlenmeyer flasks, sealed with stopcock. The flasks were kept in the temperature controlled orbital shaker and the desired temperature was maintained. About 8–10 days were required to reach its equilibrium value. After the equilibrium was attained, small samples (1–5 ml) were filtered and liquid phase was analysed for the phenol content.

Batch Desorption Rates

Desorption of phenol was carried out in a 1000 ml glass reactor immersed in a temperature controlled water bath. The reactor was fully baffled and equipped with a mechanical agitator. Experiments were conducted with the reactor charged with 900 ml of deionized water and 18 grams of GAC loaded with the desired amount of phenol. The speed of agitation was adjusted to a predetermined value and the desired temperature maintained during the course of an experiment. Intermediate samples (1–5 ml) of the aqueous phase (GAC free) were withdrawn and analysed for phenol content.

Separate controlled experiments indicated negligible phenol loss by adsorption on the glassware or to the atmosphere during the adsorption/desorption studies.

Analytical Procedure

A Perkin–Elmer–Lambda 3B UV-spectrophotometer was used for analysis. Samples were analyzed in 10 mm silica cell at the maximum absorption wavelength of 270 nm.

RESULTS AND DISCUSSION

Adsorption–Desorption Isotherm

The adsorption isotherm of phenol at various temperatures is shown in Figure 1. It was observed that the initial adsorption was much faster and about 88 per cent of phenol was adsorbed in the first 24 hours, whereas the time taken for the utilization of complete adsorption capacity was 10 days. The desorption isotherms were investigated for finding the extent of reversibility of the adsorption/desorption process. The adsorption–desorption isotherms are shown in Figure 2. It is well known that the irreversible adsorption is due to the formation of stronger bonds between surfaces of adsorbate and the adsorbent (chemisorption). The extent of the irreversibility is a function of the strength of the adsorbate/adsorbent bond. Cookson (1975) reported the formation of acidic and basic surface oxides during the high temperature processing of activated carbon. It is likely that, these oxides form stronger bonds with phenol and cause irreversible adsorption. Mattson *et al.* (1964) also showed the possibility of formation of charge transfer complexes between adsorbate and adsorbent, which results in irreversible adsorption. The effect of temperature on the desorption isotherm is shown in Figure 3. As expected, higher temperatures, are favourable for desorption.

RATE OF DESORPTION

Effect of Speed of Agitation

The speed of agitation was varied in the range of 0 to 1350 rpm (Figure 4) and it was found that the external mass transfer resistance for desorption was eliminated

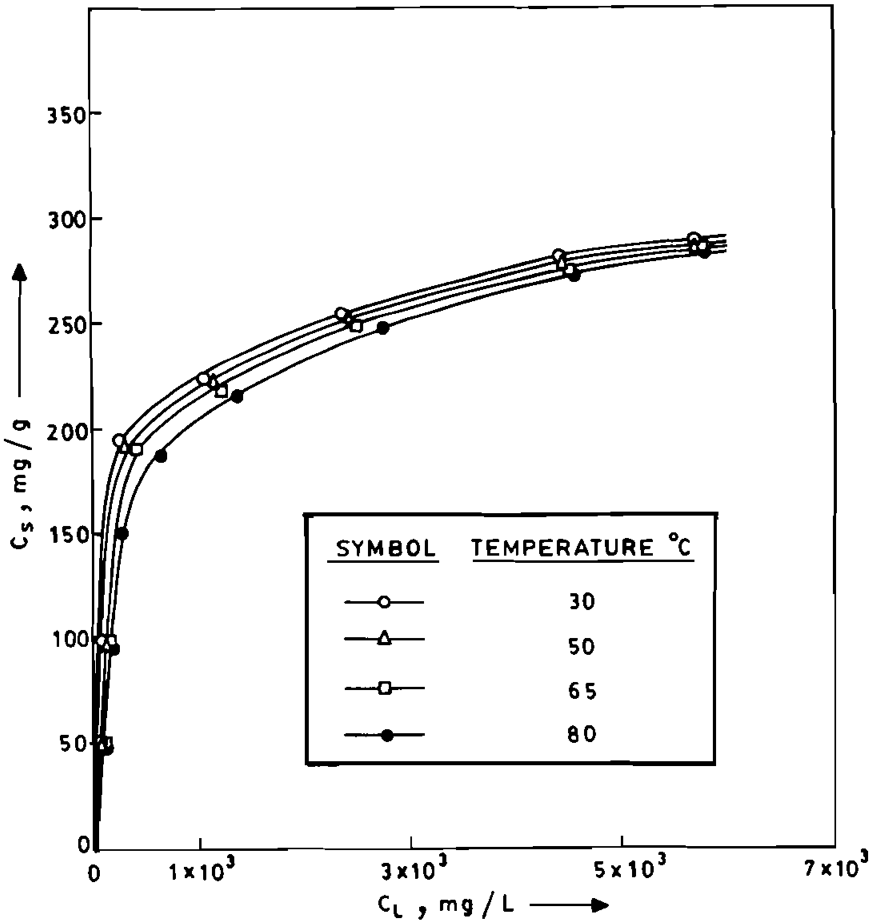

FIGURE 1 Adsorption isotherm of phenol on activated carbon.

at speeds of agitation more than 900 rpm. Consequently, all the further experiments were performed above the agitation speed of 900 rpm. The prior work with phenol on activated carbon, Freidrich *et al.* (1985) have also successfully eliminated the external mass transfer resistance by judicious choice of agitation speed.

Effect of temperature and phenol concentration

The desorption of phenol from GAC with an initial loading of 165 mg/g at different temperatures, in the range of 30°C to 80°C, is shown in Figures 5 to 8. Figures 9 and 10 show the desorption dynamics at 30°C with different initial loadings. The initial desorption is much faster, which is followed by the slower desorption and the rate of desorption was found to increase with an increase in temperature.

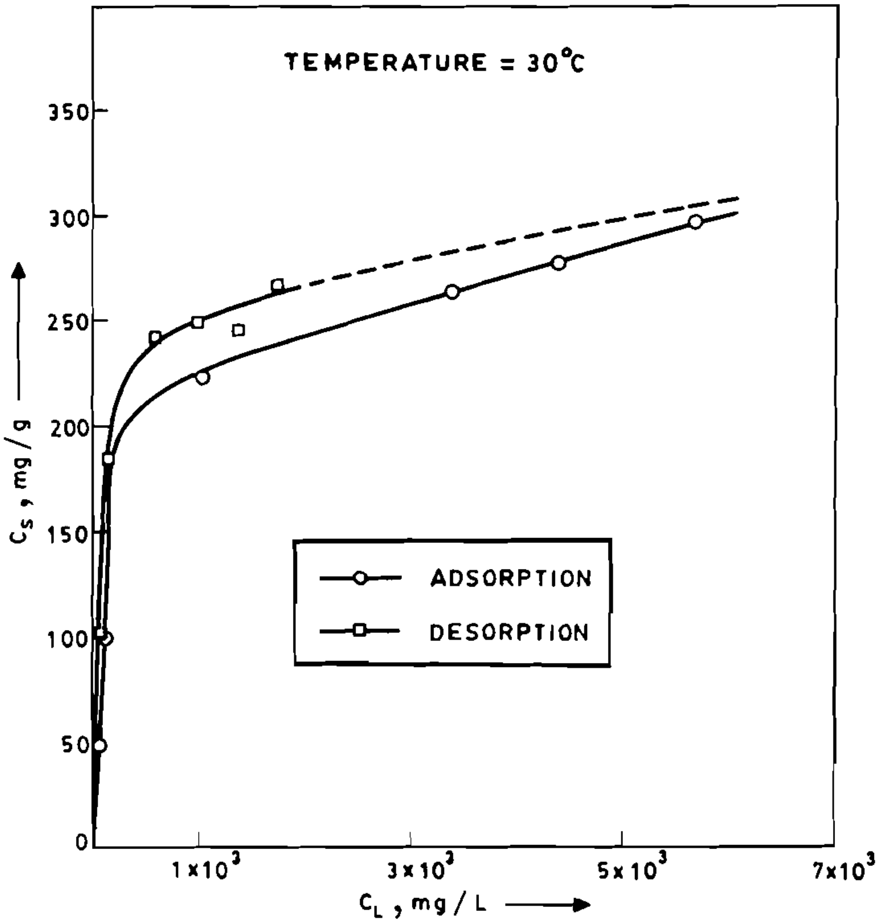

FIGURE 2 Adsorption/desorption isotherm at 30°C.

Evaluation of Effective Diffusivity

Equations (17) to (20) may be solved as previously indicated, given the parameter α, and upon suitably matching experimental and predicted fractional desorptions given by Eqs (21) and (22) respectively, the effective diffusivity can be estimated by virtue of Eq. (8). For this purpose the least squares method may be used over all the data points gathered during the course of an entire experiment. However, in this case it was considered desirable to know if the effective diffusivity varied during the course of an experiment as that would be an indication of the importance of surface diffusion which has been ignored in the present model. As is well known (Suzuki *et al.* 1984) the surface diffusion coefficient varies with concentration, and hence it may be expected to change with increased fractional desorption. Keeping this aspect in view it was decided to match the experimental and predicted times for 50 per cent desorption and use the effective diffusivity

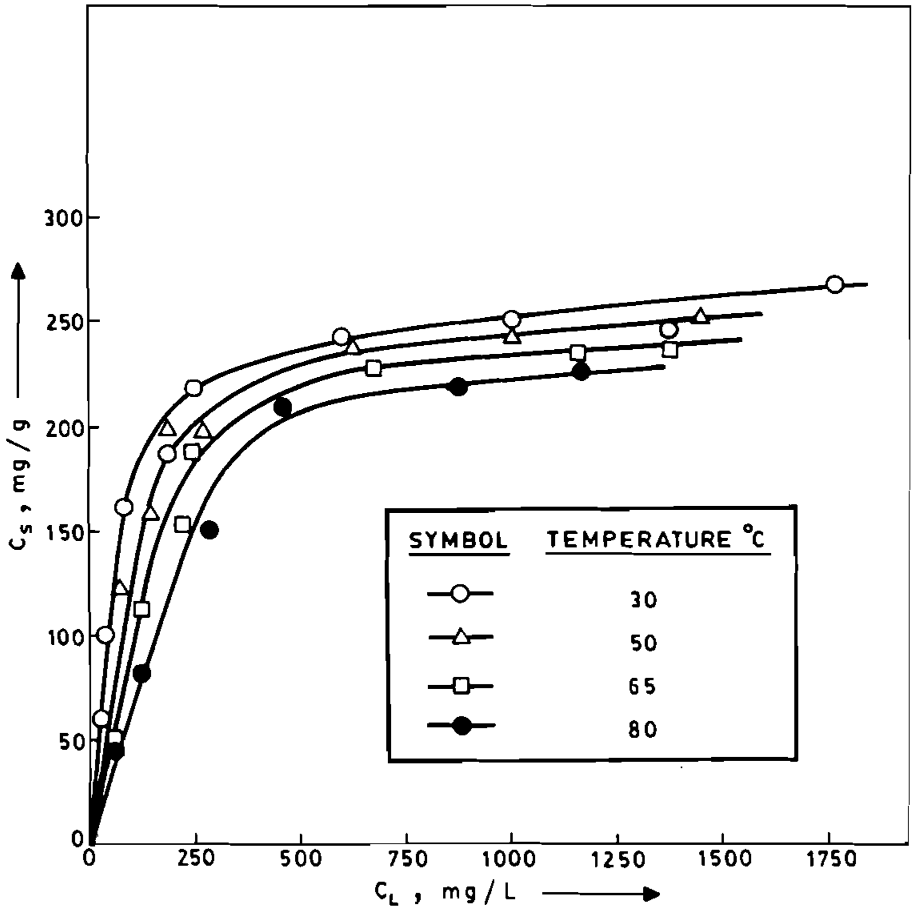

FIGURE 3 Desorption isotherm of phenol on activated carbon.

thus calculated to compare the experimental and predicted fractional desorption versus-time curves. If surface diffusion effects are indeed unimportant then the two curves should match satisfactorily.

To obtain the value of α for an experiment it is necessary to estimate the local slope of the desorption isotherm, for Eqs. (4) and (5) yield.

$$\frac{dq}{dc} = \varepsilon_M + KS_M + (1 - \varepsilon_M)(\varepsilon_\mu + KS_\mu) \tag{23}$$

and the quantity on the right hand side appears in the definition of α and dimensionless time τ. For the values of α thus calculated the experimental (by interpolation) and predicted times for 50 per cent desorption were matched and the value of effective diffusivity estimated by virtue of Eq. (8). The solid curves in Figures 5 to 10 show the theoretical predictions for the temporal variation of Q_T and are in good agreement with the data notwithstanding the fact that only

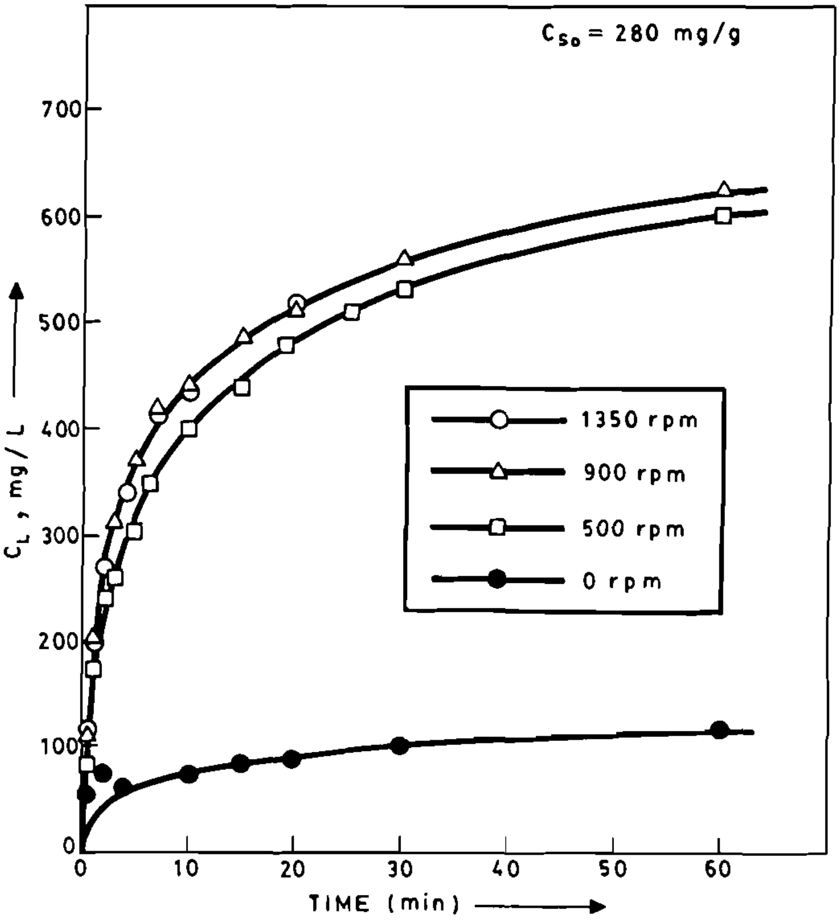

FIGURE 4 Effect of speed of agitation on the rate of desorption.

the 50 per cent point rather than the whole curve was used in the fits. A further feature is that the effect of variation in phenol loading over the range of 99.5 to 280 mg/g at 30°C is explained by a single value of the effective diffusivity (obtained from the data at 280 mg/g) at this temperature confirming the insignificance of surface diffusion effects. Table I lists the values of the estimated effective diffusivities at the four temperatures used. The average error in these values was ±18%. It is seen in the table that the ratio of tortuosity to porosity estimated from

$$\frac{\gamma}{\varepsilon}=\frac{D}{D_e} \tag{24}$$

is independent of temperatures (within the above error estimate). The values of activation energy for the diffusive process for D and D_e were found to be 18 and 20 kJ/gmol, respectively. The estimate of molecular diffusivity in the above

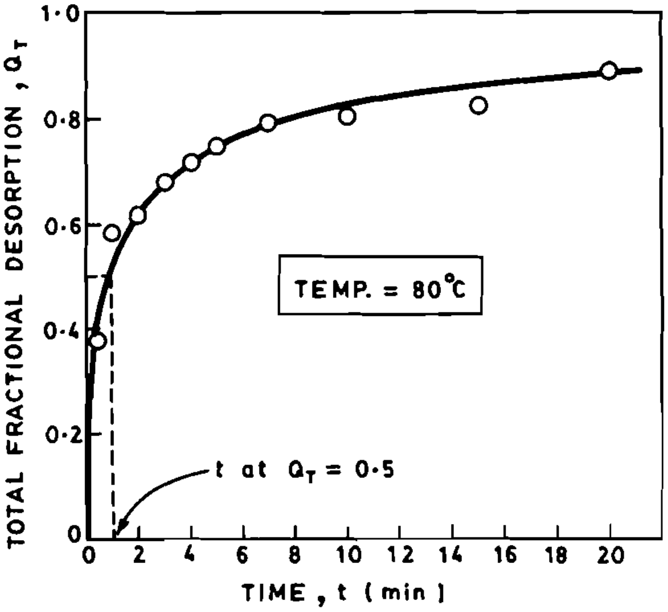

FIGURE 5 Desorption kinetics at 80°C.

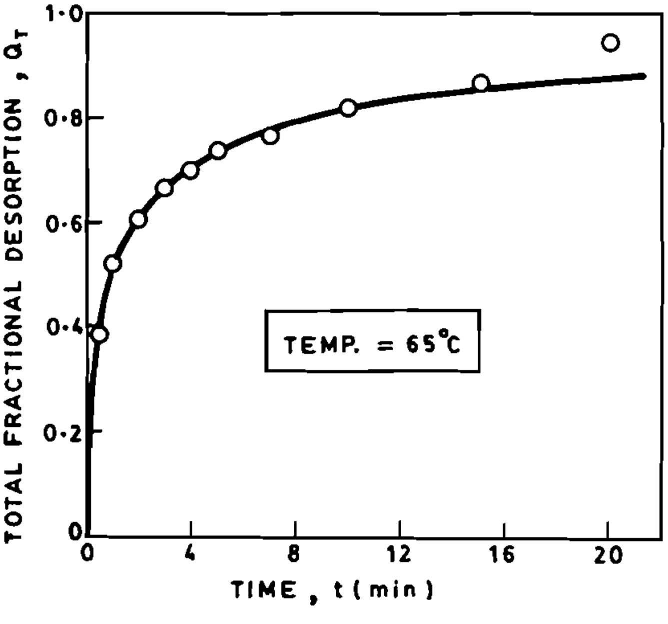

FIGURE 6 Desorption kinetics at 65°C.

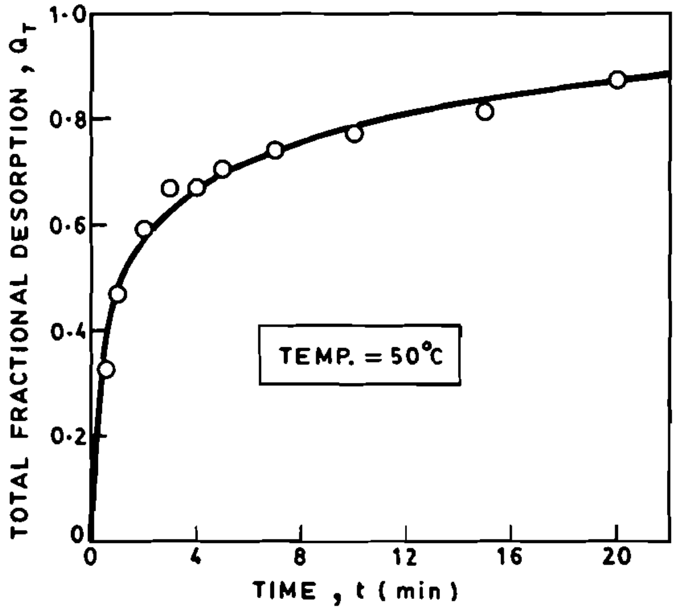

FIGURE 7 Desorption kinetics at 50°C.

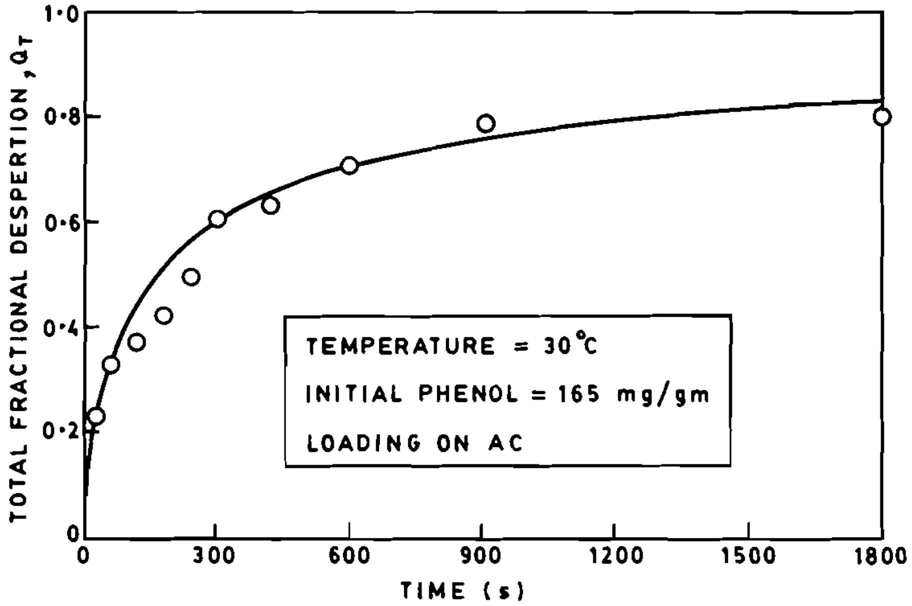

FIGURE 8 Desorption kinetics at 30°C.

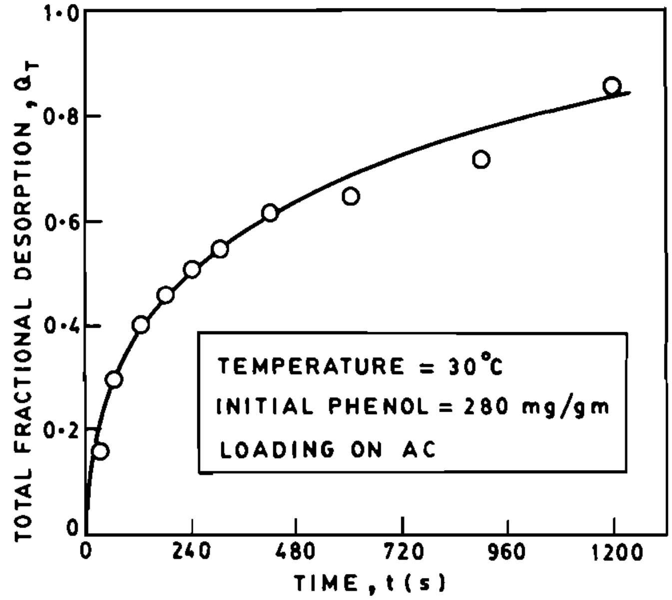

FIGURE 9 Fractional desorption versus time at 30°C with initial phenol loading on $AC = 280$ mg/gm.

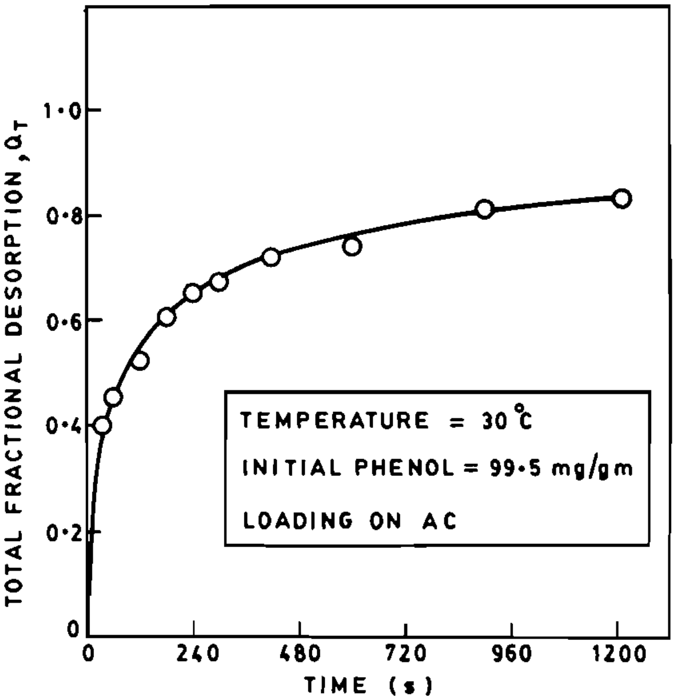

FIGURE 10 Fractional desorption versus time at 30°C with initial phenol loading on $AC = 99.5$ mg/gm.

TABLE I

Effect of temperature on true and effective diffusivity of phenol

T, °c	$D \times 10^9$ m^2/s	$D_e \times 10^{10}$ m^2/s	Tortuosity/ε
30	1.26	2.1	6.0
50	1.94	2.9	6.7
65	2.63	5.3	5.0
80	3.44	6.7	5.1

expression is obtained using the Wilke–Chang equation (Bird *et al.* 1962). Further interpretation of the above tortuosity to porosity ratio is difficult as it is known to be a complex function of pore structure (Bhatia, 1986, 1988), and in this case may also be affected by the restriction factor associated with small pores (Anderson and Quinn, 1971).

CONCLUSIONS

It is seen from the present work that measurement of desorption rates forms a convenient means of estimating effective diffusivity values in activated carbons. In the case of phenol, the adsorbed species used in the present work the effective diffusivity is found to be dominated by molecular diffusion effects, with surface diffusion contributing insignificantly. The tortuosity to porosity ratio determined by this method is essentially temperature independent consistent with the expectation that it is a function only of the pore structure.

NOMENCLATURE

A	Constant in Eq. (5)
C	Local liquid phase phenol concentration with the particle
C_L	Liquid phase phenol concentration in the bulk
C_0	Initial concentration of phenol in the bulk
C_S	Surface phenol concentration
C^*	Dimensionless concentration [Eq. (6)]
C^*_∞	Dimensionless equilibrium concentration
C^*_L	Dimensionless liquid phase concentration in the bulk
D	True diffusivity
D_e	Effective diffusivity
D_{eM}	Diffusivity through macropores
$D_{e\mu}$	Diffusivity through micropores

k	Equilibrium partition coefficient
q	Total local phenol content per unit volume [Eq. (4)]
Q	Local fractional desorption [Eq. (16)]
Q_T	Total fractional desorption
R	Radial distance from centre
R_0	Radius of carbon particle
r_g	Radius of microporous spherical grains
S_M	Macropore surface area
S_μ	Micropore surface area
t	Time
V	Volume of liquid
W	Weight of activated carbon

Greek Letters

α	Constant defined by Eq. (15)
ε	Porosity
ε_M	Macroporosity
ε_μ	Microporosity
τ	Dimensionless time [Eq. (8)]
η	Dimensionless radius [Eq. (7)]
γ	Tortuosity
ρ	Bulk density

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ACKNOWLEDGEMENT

The present work was supported by a grant from Hindustan Lever Research Foundation, Bombay.