

Phenol adsorption from water solutions over microporous and mesoporous carbon surfaces: a real time kinetic study

Eva Castillejos · Inmaculada Rodríguez-Ramos ·
Maria Soria Sánchez · Vicenta Muñoz ·
Antonio Guerrero-Ruiz

Received: 1 August 2010 / Accepted: 22 November 2010 / Published online: 2 December 2010
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Abstract This study illustrates the effect of the adsorbent porosity (activated carbon and high surface area graphite) on the phenol adsorption kinetics. We have developed an experimental system where on line analysis of the solution is carried out by an optic fiber probe introduced in the water solution and directly connected with the UV spectrometer. This experimental setup permits to be more precise in determining kinetic parameters, considering that measurements are taken each 20 seconds. Our results show that the choice of the particle diameter of the adsorbent is critical in the control of the adsorption process kinetic, while the porosity of the carbon materials appears to be less relevant.

Keywords Activated carbon · Graphite · Kinetic · Adsorption · Phenol

Abbreviations

r Adsorption rate
 A Area
 Bi Biot number
 C_h Capacity
 X Concentration position
 ρ Density

$\mathcal{D}$ Diffusion coefficient
 λ Dimensionless parameter
 τ Dimensionless time
 q Equilibrium phase concentration
 η Front adsorption
 a Front ring adsorption
 C_L Liquid phase concentration
 k_L Liquid-mass-transfer coefficient
 W Mass
 d_p Particle diameter
 b Pore diffusion parameter
 R Radius
 T Time
 V Volume

1 Introduction

The presence of organic pollutants in the continental waters continuously increases due to the human activity. Phenols and related substances contaminate the environment not only through emissions from numerous chemical industries (textile, mining, paper, petroleum refining, pharmaceutical) but also because they are intermediary products of the biodegradation of many other organic products. Currently, a large panel of methods is at the disposal of engineers to remove organic compounds from water (Cookson et al. 1978; Suffet and McGuire 1980; Cheremishinoff and Cheremishinoff 1993; Slejko 1985, Faust and Aly 1987, Perrich 1981). Adsorption is the most widely used method for pollution treatments and the activated carbons are the most frequently employed adsorbents. Among other the reasons for applying activated carbons as adsorbents are: (i) extended surface area and microporous structure, (ii) high adsorption capacity, (iii) relatively rapid retention kinetics

E. Castillejos · I. Rodríguez-Ramos
Instituto de Catálisis y Petrol, C/Marie Curie 2, Cantoblanco,
28049 Madrid, Spain

M.S. Sánchez · V. Muñoz · A. Guerrero-Ruiz (✉)
Dpto. Química Inorgánica y Técnica, UNED, Senda del Rey
No. 9, 28040 Madrid, Spain
e-mail: aguerrero@ccia.uned.es

M.S. Sánchez · V. Muñoz · A. Guerrero-Ruiz
Unidad Asociada Grupo de Diseño y Aplicación de Catalizadores
Heterogéneos UNED-ICP (CSIC), Madrid, Spain

(Bansal et al. 1988; Mattson and Mark 1971; Jankowska et al. 1991).

At present the kinetic studies of adsorption are critical to allow an efficient development and adequate design of the adsorbent systems on which the organic contaminants are removed from the water. Presently and provably due to experimental difficulties, most of the studies are conducted point by point, that is analyzing different substrates contacted with similar solutions at discrete periodic times (Roostaei and Tezel 2004; Tseng et al. 2003; Nevskaja et al. 1999; Vinod and Anirudhan 2002; Brasquet and Le Cloirec 1997). However results obtained under continuous analysis of the contaminant concentration are scarcely reported. The adsorption kinetic is a function of several parameters: adsorbate and adsorbent nature, characteristics of adsorbent porosity and solid-liquid interface contact. In general, the carbon adsorption processes can be described as three consecutive steps that begin with external mass transfer of adsorbate from the solution bulk towards the adsorbent surface, followed by its diffusion within the adsorbent pores and ending with the adsorption on the active sites of the adsorbent.

In a kinetic study, comparison of the experimental adsorbate concentration decay curves obtained for a given system with a model, which may have an empirical or theoretical basis, is performed. Several models have been proposed to describe the adsorption kinetic. Thus, the homogeneous surface diffusion model (HSDM) used by many authors (Al-Duri and McKay 1991; Crittenden and Weber 1978; McKay 1998; Chatzopoulos et al. 1993) assumes that the adsorbent is a homogeneous solid, with adsorption on the external surface of the pellet, followed by diffusion of the adsorbate into the particles. While the pore diffusion model based on the unreacted shrinking core model (Levenspiel 1972; Lindman and Simonsson 1979; Nerernieks 1974, McKay 1984a, 1984b) considers that there is a diffusion of the adsorbate into the pores with a simultaneous adsorption all along the pore walls. To simplify the mathematical apparatus, usually it is assumed that only one diffusion mechanism is significant. Therefore some researchers have presented mathematical solutions for the rate of mass transfer controlled by: external diffusion (Hougen and Marshall 1947), internal pore diffusion (Rosen 1954), surface adsorption (Masamune and Smith 1965), either external diffusion or surface adsorption (Goldstein 1953).

In this communication we report a simple and efficient method to monitor continuously the phenol adsorption phenomena in order to obtain more precise information concerning the kinetic processes, especially at low solid-solution contact times. Additionally we present a comparative kinetic study of phenol adsorption on two different carbon materials: a microporous activated carbon and a mesoporous high surface area graphite.

2 Experimental

2.1 Adsorbents and adsorbates

Phenol was obtained from Sigma. A commercial activated carbon (RS 0.8) with selected sieve fraction, 0.5–0.75 mm, was supplied by Norit (Amersfoort, the Netherlands). Also the high surface area graphite (HSAG-300, particle size 150 μm) was commercially available, provided by Lonza (Switzerland). Both adsorbents have been extensively characterized and the results presented in a previous paper (Nevskaia et al. 2004). Briefly the microporous activated carbon has an apparent surface area of $982 \text{ m}^2 \text{ g}^{-1}$ and an ash content of 4.6%, while mesoporous graphite has an apparent surface area of $310 \text{ m}^2 \text{ g}^{-1}$ with no inorganic impurities.

2.2 Kinetic adsorption measurements

Different concentrations of phenol solutions (from 2×10^{-4} to $8 \times 10^{-4} \text{ mol l}^{-1}$) were prepared and transferred to a 1.5 l glass reactor of 14 cm of diameter with a known weight of adsorbent (from 0.06 to 1 g). A glass-anchor impeller of 7.5 cm of internal diameter, 9.3 cm of outward diameter and 50 cm of length provided good mixing in the vessel. An Eurostar Power type b variable-speed (50–2000 rev/min) motor was used to drive the impeller.

The concentration of organic samples was determined by UV absorbance measurements using a Varian-Cary Spectrophotometer. A probe immersed in the water solution and connected by optical fiber to the spectrophotometer was used. This analytical probe was introduced in the reactor's mouth and was protected by a glass-shirt, which allows passing only the solution. To facilitate the renovation of the solution in contact with the sensor, a peristaltic pump was continuously operated, with a flow of circulating solution of $120 \text{ cm}^3 \text{ min}^{-1}$. All measurements were made at the wavelength corresponding to maximum absorbance (269.5 nm) of the phenol and at room temperature.

A set of preliminary experiments showed that kinetic experiments were accurately reproduced, adsorbents did not change the particle size distribution during the experiment and a stirring rate of 300 r.p.m. was enough to avoid the external diffusion problems.

3 Results and discussion

3.1 Activated carbon

The adsorption rate depends on the adsorbate concentration and the adsorbent amount introduced into the reactor. Then the Fig. 1 shows the effects of initial concentrations in the

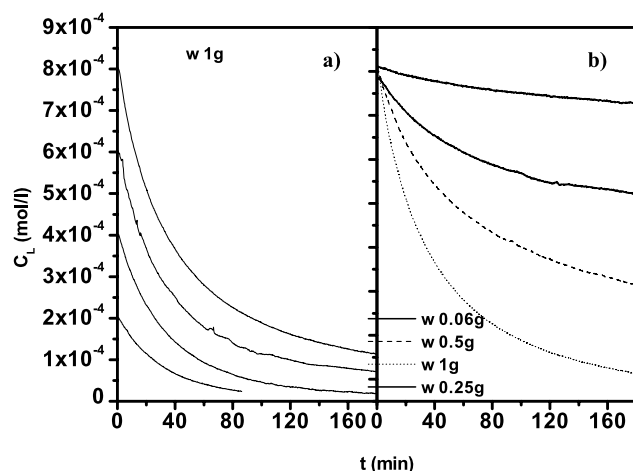


Fig. 1 Adsorption kinetics of Phenol on activated carbon: (a) different concentrations of adsorbate, (b) different concentration of adsorbent

liquid phase (C_{L0}) and of carbon mass (W) on the adsorption rate of phenol over the activated carbon. According to the shape of the decay curves, the following empirical expression has been applied:

$$\frac{(C_L - C_{L\infty})}{(C_{L0} - C_{L\infty})} = \exp(-P_1 t) \quad (1)$$

where C_L is the concentration at time t and $C_{L\infty}$ is the concentration at quasi equilibrium time. The square correlation coefficients R^2 obtained using as equilibrium time 180 and 60 min were of at least 0.96 and 0.999 respectively. This expression is typical of the first order kinetics and might make to think that the external mass transfer could be the controlling-step of the process.

The derivative of C_L at initial conditions corresponds to the mass flow from the solution bulk towards the solid surface, when still there is no adsorbate on the adsorbent surface, being independent of the controlling-step of the process (Goldstein 1953; Calleja et al. 1993; Zhou and Martin 1993; Vinci and McKay 2004). And thus,

$$\left[\frac{d(C_L)}{dt} \right]_{t \rightarrow 0} = (-r_0) = k_L A (C_{L0} - C_{Ls}) = k_L A C_{L0} \quad (2)$$

To obtain the initial rate of the process and from this the liquid-side mass-transfer coefficient (k_L), the derivatives of the empirical expression (accepting these are spherical particles) can be written (McKay et al. 1996) as:

$$-\left[\frac{dC_L}{dt} \right]_{t \rightarrow 0} = k_L A C_{L0} = k_L \frac{W}{V \rho} \frac{6}{d_p} [C_{L0}] \cdot 10^{-3} \quad (3)$$

Table 1 shows the k_L values obtained from (3). The greater deviation was observed for 0.06 g of adsorbent probably due to a higher experimental error committed in this determina-

Table 1 Liquid-side mass-transfer coefficients (k_L) for phenol adsorption on activated carbon surface

Adsorbate	W (g)	C_{L0} (mol l ⁻¹)	d_p (cm)	k_L (cm ² s ⁻¹)
Phenol	1	8×10^{-4}	0.08	8.49×10^{-3}
	0.50	8×10^{-4}	0.08	8.58×10^{-3}
	0.25	8×10^{-4}	0.08	8.25×10^{-3}
	0.06	8×10^{-4}	0.08	7.0×10^{-3}
	1	8×10^{-4}	0.04	1.34×10^{-2}

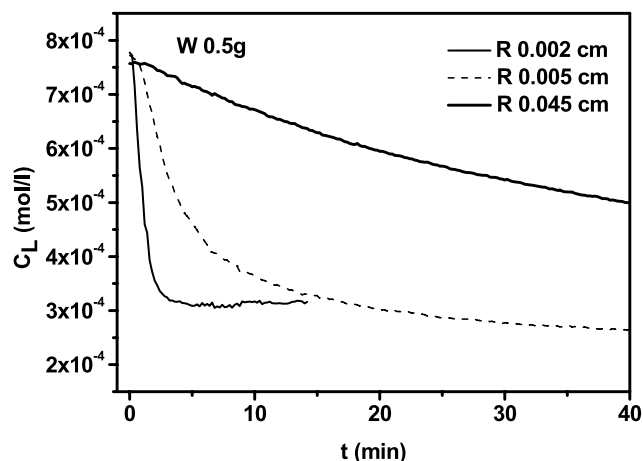


Fig. 2 Adsorption kinetic of Phenol on activated carbon with different particle diameter

tion. However we detected for 0.004 cm of average particle diameter that can only be explained by an increase in the adsorption rate (Fig. 2). As it has been mentioned above, once the barrier of the external surface is surpassed, the diffusion of adsorbate molecules within the adsorbent pores plays a major role, so the transport inside the solid is frequently accepted as the controlling-stage of the process. A simple model is proposed in the texts of Rodrigues (Rodrigues et al. 1989) and Ruthven (1984). It is generally accepted that the solid behaves according to the shrinking core model, in such a way that the slowest stage is the diffusion in the ring saturated by the pollutant, and the adsorption is produced instantaneously. Therefore, the slow stage is the transport within the pores. Considering that the solid is essentially microporous and only one diffusional resistance is significant, and that the system is isothermal and the particles are spheres, the transport in the ring may be described by the following diffusion equation:

$$\frac{\partial q_C}{\partial t} = \frac{1}{R^2} \frac{\partial}{\partial R} \left(R^2 D \frac{\partial q_C}{\partial R} \right) \quad (4)$$

The solution for the uptake curves was previously reported (Boyd et al. 1947):

$$\frac{q_t}{q_{t\infty}} = 1 - 6 \sum_{n=1}^{\infty} \frac{1}{n^2 \pi^2} \exp\left[-\frac{n^2 \pi^2 \mathcal{D} t}{R^2}\right] \quad (5)$$

where q_t is the average concentration through the particle

$$q_t = \int_{R=0}^{R=R} q_{CR} 4\pi R^2 dR$$

If the amount of adsorbed phenol, as is in our case, is not negligible compared with the quantity introduced in the system the concentration in the fluid will not remain constant. Therefore, the boundary condition at the surface of the adsorbent particle will be now time-dependent. And the solution of the uptake curve is (Crank 1954):

$$\frac{q_t}{q_{t\infty}} = 1 - 6 \sum_{n=1}^{\infty} \frac{\exp\left[-\frac{p_n^2 \mathcal{D} t}{R^2}\right]}{(9\lambda/(1-\lambda)) + (1-\lambda)p_n^2} \quad (6)$$

where

$$\lambda = \frac{C_{L0} - C_{L\infty}}{C_{L0}}$$

and p_n is given by the nonzero roots of

$$\tan p_n = \frac{3p_n}{3 + ((1/\lambda) - 1)p_n^2}$$

To confirm the experimental results with this model, the value of q/q_{∞} vs $t^{1/2}$ and the shape of the curves has been compared with the offered ones by the theoretical equations of the model representing q/q_{∞} vs $(t\mathcal{D}^2/R)^{1/2}$ for the corresponding values of the parameter λ . It can be observed that strong analogies appear between both, the biggest difference being at times lower than 2 minutes of experiment, which could be explained by a major influence of mass transfer between phases when the possibility of flow towards the fresh solid surface is major. Comparing for each trial, the theoretical and experimental abscissa values at the same ordinate value, an effective coefficient of diffusion $\mathcal{D}$ values of $2.4 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$ has been estimated.

The Biot mass number ($\frac{k_L R}{\mathcal{D}}$) indicates the relative importance of both phenomena of transport, external and internal diffusion, in the global kinetics. Using the obtained values for k_L and $\mathcal{D}$, a Bi of the order of 10^4 has been calculated. This confirms the hypothesis that diffusion within particles has an important influence on the kinetic process. However, when the diameter particle decreases the external mass transference is a major factor, then a remarkable increase of the rate of adsorption is observed (Fig. 2).

3.2 Graphite

The high surface area graphite has many differences with respect to activated carbon. It is a mesoporous solid with a particle size smaller than activated carbon. Thus, for the same W/V ratio, the area for the external transfer is 40 times higher for graphite than for activated carbon. Thus we have to expect a considerable increase in the adsorption rate. Experimentally it can be checked that C_L is practically constant before the 8 first minutes in the adsorption process.

$$\frac{(\text{area/mass})_{\text{carbon}(1)}}{(\text{area/mass})_{\text{graphite}(2)}} = \frac{(\rho d_P)_2}{(\rho d_P)_1} = \frac{(1.31)(800)}{(1.75)(15)} = 40$$

Considering the mass transport inside the particle, if the transfer mechanism is pore diffusion as in the case of activated carbon, the necessary time to reach any percentage of the total saturation will be proportional to the square radius of the particle. Therefore,

$$\frac{t_{\text{carbon}}}{t_{\text{graphite}}} = \frac{R_{\text{carbon}}^2}{R_{\text{graphite}}^2} = \frac{400^2}{7.5^2} = 2844$$

In accordance with the graphs of the concentration decay in the liquid phase (Fig. 3), the diminution of the phenol concentration is not fast. This signifies that in case of the graphite, the external mass transference is the critical step in the adsorption process.

The kinetic curves have been treated in the same way as for activated carbon, accepting an exponential decay of the concentration in liquid phase (1). Table 2 shows the k_L values obtained from (3). And as it was expected, the k_L values are lower than the obtained for activated carbon probably due to the smaller diameter of graphite particle. Although the value for the square of correlation coefficient (R^2) obtained in the entire range of time was of 0.99. Now just to verify the grade of influence of the internal diffusion on the

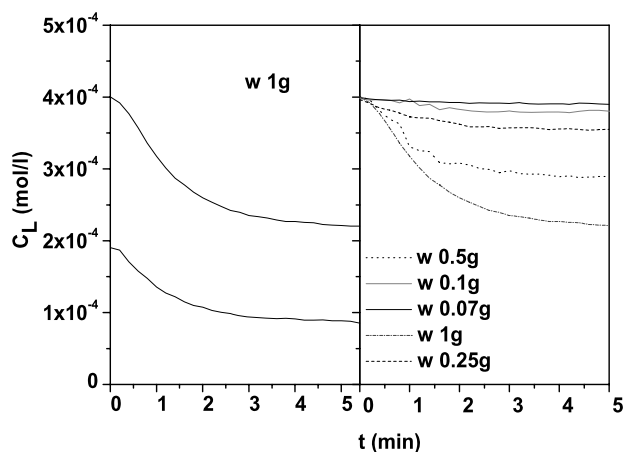


Fig. 3 Adsorption kinetic of Phenol on Graphite: (a) different concentrations of adsorbate, (b) different concentration of adsorbent

Table 2 Liquid-side mass-transfer coefficients (k_L) for phenol adsorption on graphite surfaces

Adsorbate	W (g)	C_{L0} (mol l ⁻¹)	d_p (cm)	k_L (cm ² s ⁻¹)
Phenol	1	4×10^{-4}	1.5×10^{-3}	2.58×10^{-3}
	1	2×10^{-4}	1.5×10^{-3}	3.17×10^{-3}
	0.50	4×10^{-4}	1.5×10^{-3}	3.11×10^{-3}
	0.25	4×10^{-4}	1.5×10^{-3}	1.29×10^{-3}
	0.1	4×10^{-4}	1.5×10^{-3}	2.43×10^{-3}

adsorption process over the graphite surfaces, we can applied the McKay (McKay 1984a, 1984b, 1985; McKay et al. 1996) model, which was developed as a pore diffusion model, based on the unreacted shrinking core mass model, in which both transports, external to the particle and diffusion in the pores, influence the kinetics.

Mass transfer from external phase is given by:

$$r = k_L 4\pi R^2 (C_L - C_{LS})$$

And pore diffusion according to the Fick law by:

$$r = \frac{4\pi d_p C_{LS}}{\frac{1}{R_C} - \frac{1}{R}}$$

Being the rate of the concentration given by

$$r = -4\pi R_C^2 q_{eh} \rho_t \frac{dR_C}{dt}$$

If we accept that $q_{eh} \approx q_\infty$ and the relation between q_t and R_c is

$$\eta = \frac{q_t}{q_\infty} \left[1 - \left(\frac{R_c}{R} \right)^3 \right]$$

The global adsorption rate (in dimensionless mode) will be represented by the equation

$$\frac{d\eta}{d\tau} = \frac{3(1 - C_h \eta)(1 - \eta)^{0.33}}{[1 - [1 - (\frac{\mathcal{D}}{k_L R})](1 - \eta)^{0.33}]} \quad (7)$$

This equation is function of the capacity factor $C_h = \frac{q_\infty W}{C_0 V}$ and of the concentration gradient front $(1 - \eta)^{0.33} = \frac{R_c}{R} = X$ and $\tau = \frac{C_0 t}{\rho_t q_{eh} R} \frac{\mathcal{D}}{R k_L}$ where the time is dimensionless. Using the following boundary conditions limits:

$$\begin{aligned} \tau = 0, \quad \eta = 0, \quad X = 1 \\ \tau = \tau, \quad \eta = \eta, \quad X = X \end{aligned}$$

(McKay et al. 1996) solve (7) as:

$$\begin{aligned} \tau = \frac{1}{6Ch} \left[\ln \left[\left[\frac{X^3 + a^3}{1 + a^3} \right]^{(2b-1/a)} \right] \right. \\ \left. + \ln \left[\left[\frac{X^3 + a^3}{1 + a^3} \right]^{(3/a)} \right] \right. \\ \left. + \left[\frac{1}{a^{30.5} Ch} \right] \arctan \left[\frac{2-a}{a^{30.5}} \right] - \arctan \left[\frac{2X-a}{a^{30.5}} \right] \right] \quad (8) \end{aligned}$$

where $a = ((1 - C_h)/C_h)^{0.33}$ is a simplifying term in the pore diffusion model and $b = 1 - \frac{1}{Bi} = \frac{\mathcal{D} - k_L R}{\mathcal{D}}$ is the parameter of the model. The expression (8) allows to compare with experimental values of C_L and t . It should be notice the coefficient that permits convert (X, τ) into $(q/q_F, t)$ gives place to the $\mathcal{D}$ values. From these calculations the $\mathcal{D}$ values of 2.0×10^{-6} cm s⁻² has been accepted. Therefore, the Biot number takes the value of the unit, though it reinforces the idea that external transference mechanism has an important influence on the kinetics of the adsorption in case of the graphite. Finally it is interesting to remark that features of the two carbon adsorbents similar to the case of the phenol adsorption kinetics were determined in comparable experiments performed with aniline solutions. Hence, the proposed kinetic mechanisms seem to be independent of the adsorbates chemical basicity. This fact can be likely due to the absence of surface functionalities as above indicated on the two studied adsorbents. Therefore, specific interactions of the adsorbates (phenol or aniline) with the surface of the two adsorbents are not expected (Radovic et al. 2001).

4 Conclusions

In conclusion we report a kinetic study of the adsorption of phenol compounds on two carbon materials differing in porosity, an activated carbon and a high surface area graphite. This determination has been carried out in an experimental setup which allows continuous analysis of the adsorbate in the solution. The area for the external transfer is higher for graphite than for activated carbon, which increases considerably its adsorption rate. Our results show that external mass transference has a significant influence on the adsorption rate of the graphite. In the case of the activated carbon the diffusion coefficient within particles is the key parameter, which has a major influence on the global kinetic process. However, when the particle diameter decreases, the external transference and the rate of adsorption increase. Thus the choice of the adsorbent particle diameter is critical to control the kinetic parameters and, in general, the adsorption process. This study also illustrates the effect of two adsorbent surfaces on the phenol adsorption kinetic. Finally, the design of adsorption setups with high resolution for short contact times opens new possibilities for kinetic determination in numerous adsorption processes.

Acknowledgements Authors acknowledge the MICINN Spain (Projects CTQ-2008-06839-C03-01 and 03-PPQ and EUI2008-00185 and -00205) for the financial support.

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