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The effect of surface roughness on fluid-to-particle mass transfer in a packed adsorber bed

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Abstract—Surface roughness is shown to have a significant effect on fluid-to-particle mass transfer in a packed absorber bed. Mass transfer coefficients are determined using three spherical adsorbents in the Reynolds number range 0.9–12 using a mini-column technique. Experimental breakthrough curves were successfully modelled using a model which includes external film transfer and intraparticle surface diffusion. The Schmidt number used is 1190. It is shown that adsorbent particle surface roughness significantly enhances the external film mass transfer coefficient.

INTRODUCTION

FLUID–PARTICLE heat or mass transfer in fixed beds is an important item of the basic information required for the design and development of various heat and mass transfer operations and chemical reactors involving contacting systems of particles and a fluid. This phenomenon has been widely investigated in the past and the literature has been reviewed by Dwivedi and Upadhyay [1]. The results are usually presented in terms of correlations of the three dimensionless groups, the Sherwood number, the Reynolds number and the Schmidt number. In a number of these investigations account has been taken of the particle shape. However, as far as can be established no account has been taken of the roughness of the packing material. Recent results by van Vliet and Young [2] have shown that the surface roughness of the packing can have a profound effect on the film mass transfer coefficient. These results were obtained in a completely mixed batch reactor. The surface roughness of the particles was quantified using a technique called fractal analysis [3] and the fractal dimension of the adsorbent particle was shown to be related to its mass transfer enhancement. It was also shown that minute surface roughness elements have a significant effect on mass transfer, and though the particle may appear macroscopically smooth it may be rough for mass transfer purposes. It is the purpose of this paper to show that the surface roughness of the packing in a packed bed has a profound effect on the mass transfer coefficient.

The fact that surface roughness enhances film mass transfer has been recognized for a long time. However, as far as could be established the only published experimental evidence of this phenomenon is for mass transfer from flat plates. The effect of regularly roughened surfaces on film mass transfer has been reported by Tantirige and Trass [4] and Dawson and Trass [5]; and the effect of randomly roughened surfaces has been reported by Acosta *et al.* [6]. These workers concluded that film mass transfer could be significantly enhanced by surface roughness. Acosta *et al.* [6] also concluded that the mass transfer surfaces had to be optically smooth in order that no mass transfer enhancements are observed.

In the past there have been two main ways of measuring mass transfer coefficients. The first method involves using a solid surface which dissolves in the chosen liquid, and the rate at which the surface dissolves is dependent on the film coefficient [7]. The second method is an electrochemical approach where the surface under investigation forms one of the electrodes. In some of the experiments the electrode is inert [8] and in others plating of metal occurs at the electrode [9]. Discrepancies of up to fourfold in the results obtained for flat plates by these two methods have been reported by Dawson and Trass [5] which they attribute to the formation of minute roughnesses on the surface as it dissolves. It thus appears that the first technique is not satisfactory. In most of the experiments done on packed beds using the electrochemical technique the electrode is one active sphere in a packed bed of inert spheres [8]. Benzina *et al.* [9] performed experiments where the whole bed is active since this represents more exactly what happens in a packed bed. The results they obtained were consistently lower than using a single active sphere. Unfortunately the active bed was not inert and plating of copper occurred on the active bed. This may have caused minute surface roughnesses which could have interfered with the results.

The present work measures mass transfer coefficients of three spherical adsorbents using a mini-column technique proposed by Weber and Liu [10] and also used by Roberts *et al.* [11] and Cornel *et al.* [12]. Their results show a small discrepancy between adsorbents and also differences between their results and existing correlations. However, they offer no satisfactory explanation for these discrepancies. They also do not present enough data for definitive conclusions to be drawn. The Reynolds number range used in the present work is 0.9–12 which is typical of the Reynolds numbers used in packed bed adsorbers

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NOMENCLATURE

A	Freundlich isotherm parameter $[\mathrm{mg\,g^{-1})(l\,mg^{-1})^{\gamma}}]$
C_c	liquid adsorbate concentration $[\mathrm{mg\,l^{-1}}]$
C_{ce}	initial effluent concentration $[\mathrm{mg\,l^{-1}}]$
C_{pi}	liquid concentration at particle boundary $[\mathrm{mg\,l^{-1}}]$
C_s	solid adsorbate concentration $[\mathrm{mg\,g^{-1}}]$
d_p	particle diameter [cm]
D	diffusivity of adsorbate in water $[\mathrm{cm^2\,s^{-1}}]$
D_s	surface diffusion coefficient $[\mathrm{cm^2\,s^{-1}}]$
k_f	film transfer coefficient $[\mathrm{cm\,s^{-1}}]$
L	packing length [cm]
r	radial coordinate [cm]
r_0	particle radius [cm]
Re	Reynolds number, $d_p V\rho/\mu$
Sc	Schmidt number, $\mu/\rho D$
Sh	Sherwood number, $k_f d_p/D$
Q	volume flow rate $[\mathrm{ml\,min^{-1}}]$
t	time [s]
V	superficial velocity $[\mathrm{cm\,s^{-1}}]$
Z	longitudinal coordinate [cm].

Greek symbols

β	mass transfer enhancement factor
γ	Freundlich isotherm parameter
ε	column voidage
μ	liquid viscosity $[\mathrm{g\,cm^{-1}\,s^{-1}}]$
	particle density [g (dry adsorbent) $\mathrm{l^{-1}}$ (particle)]
ρ	liquid density $[\mathrm{g\,cm^{-3}}]$.

for waste water treatment. The mini-column technique is not suited to measuring mass transfer coefficients over a wide range of Reynolds numbers. The three adsorbents were chosen in order to cover the whole range of adsorbent surface types, from very smooth to very rough, see van Vliet and Young [2] for more details. The adsorbents chosen are all macroscopically spherical so that shape considerations do not complicate the analysis.

MODELLING

Model equations

The system to be modelled is a packed bed adsorber. In the derivation of the column material balance plug flow is assumed, the effect of longitudinal dispersion is neglected and the adsorbate concentration is low so that V becomes constant. These assumptions have been shown to be justified by Weber and Liu [10]. The adsorbent particles are assumed to be spherical. The column material balance under these assumptions is [13]

$$\frac{V}{\varepsilon}\frac{\partial C_c}{\partial Z}+\rho_p\left(\frac{1-\varepsilon}{\varepsilon}\right)\frac{\partial \bar{C}_s}{\partial t}+\frac{\partial C_c}{\partial t}=0 \tag{1}$$

where $\bar{C}_s$ is the average solid phase concentration defined by

$$\bar{C}_s=\frac{3}{r_0^3}\int_0^{r_0} C_s(r,t)r^2\,\mathrm{d}r. \tag{2}$$

The boundary conditions for this equation are

$$C_c(0,t)=C_{c0} \tag{3}$$

$$C_c(Z,0)=0. \tag{4}$$

The rate of adsorption is determined using the homogeneous surface diffusion model [13]. This model includes external film transfer and intraparticle surface diffusion. Intraparticle diffusion can take place via surface diffusion along the pore walls of the adsorbent or by pore diffusion in the adsorbent pores. The effect of pore diffusion is neglected since surface diffusion has been shown to be the determining transport mechanism in most cases [13, 16]. The model assumes that the surface diffusion can be modelled by a single effective surface diffusion coefficient. This model has been experimentally verified using a number of different adsorbents and adsorbates [14–16, 18]. The concentration profile inside the adsorbent particles is obtained by solution of the following equation:

$$\frac{\partial C_s}{\partial t}=\frac{D_s}{r^2}\frac{\partial}{\partial r}\left(r^2\frac{\partial C_s}{\partial r}\right). \tag{5}$$

The boundary conditions for this equation are

$$C_s(r,0)=0 \tag{6}$$

$$\frac{\partial C_s}{\partial r}(0,t)=0 \tag{7}$$

$$\frac{\partial C_s}{\partial r}(r_0,t)=\frac{k_f}{D_s}(C_c-C_{pi}) \tag{8}$$

where C_{pi} is the liquid concentration at the adsorbent particle surface.

It is assumed that there is equilibrium between the solid and the liquid at all times. This assumption is well established and is discussed by Crittenden [13]. The equilibrium solid concentration is related to the equilibrium liquid concentration via the non-linear Freundlich isotherm

$$C_s=AC_p^{\gamma}. \tag{9}$$

The liquid concentration at the particle surface may then be expressed as

$$C_{pi}=\left(\frac{C_s(r_0,t)}{A}\right)^{1/\gamma}. \tag{10}$$

The column material balance, equation (1), may be reformulated and expressed in the form

$$\frac{V}{\varepsilon}\frac{\partial C_{\mathrm{c}}}{\partial Z}+3\rho_{\mathrm{p}}\left(\frac{1-\varepsilon}{\varepsilon}\right)\frac{k_{\mathrm{f}}}{r_0}(C_{\mathrm{c}}-C_{\mathrm{pi}})+\frac{\partial C_{\mathrm{c}}}{\partial t}=0. \tag{11}$$

The boundary conditions are equations (3) and (4).

The complete solution of the model is obtained by solving the partial differential equations (5) and (11) coupled by equation (10). The equations were solved numerically. Equation (5) was solved using the NAG FORTRAN software library routine D03PGF. Equation (11) was solved using the QUICK algorithm [17].

Small time solution

The character of the model solutions was investigated by Weber and Liu [10]. They performed model simulations covering the whole range of experimentally observed D_{s} values. They concluded that the initial stage of the breakthrough curve is always dominated by film transfer.

At small times the loading of adsorbate onto the adsorbent particles may be neglected and the concentration of adsorbate at the particle surface is effectively zero. This is particularly true if the isotherm is favourable ($\gamma < 1$). Since film transfer dominates the rate expression, the initial effluent breakthrough concentration is given by

$$C_{\mathrm{ce}}=C_{\mathrm{c0}}\exp\left[\frac{-3(1-\varepsilon)Lk_{\mathrm{f}}}{Vr_0}\right]. \tag{12}$$

Using equation (12) and roughly estimating k_{f} from existing correlations [1, 7–9], adsorbent columns which manifest incipient breakthrough (the initial effluent concentration is significant) may be designed.

From the experimentally measured effluent concentration, equation (12) may be used to estimate the film coefficient. This procedure was checked by solving the full model equations.

EXPERIMENTAL

Three adsorbents were chosen for this study, they are BACM activated carbon, XE-348 and XE-340 carbonaceous adsorbents. These three adsorbents were chosen because they are all macroscopically spherical and represent a wide spread of surface types. The adsorbents were from the same batch of adsorbents used by van Vliet *et al.* [14] in earlier experimental work. The adsorbents were all sieved into narrow size ranges.

The BACM carbon has a very rough surface, the XE-348 is fairly rough and the XE-340 is very smooth. Electron micrographs of the three adsorbents are presented in Fig. 1 and show this difference. In ref. [2] it is shown that these differences produce very significant differences in the film transfer coefficient for adsorbent particles in a completely mixed batch reactor. It was found that the surface roughness enhanced the mass transfer. The surface roughness of a number of adsorbent types was quantitatively investigated [2] using a technique called fractal analysis [3]. From this work the roughest, the smoothest and an intermediate adsorbent were chosen. The adsorbate used in all cases was phenol.

The experimental apparatus is shown in Fig. 2. A solution of known phenol concentration flows from a constant head reservoir through a flow control valve to a small column which contains a packed bed of virgin adsorbent particles. The adsorbent particles were packed between two layers of glass beads of approximately the same size as the adsorbent particles in order to minimize entrance and exit effects. The column diameter was 0.99 cm. The height of the adsorbent layer was not less than 1.5 cm. To avoid air bubbles the column was carefully packed under water. The experiments were conducted in a constant temperature room at 20°C.

Before the experiment commenced the water level was reduced to just above the packing to minimize dilution. The concentration of the phenol solution used in all the experiments was 50 mg l^{-1}. Flow of the phenol solution through the column commenced at time $t = 0$ and samples of the effluent were taken at known times. These samples were analysed by UV spectrophotometry at a wavelength of 268 nm.

The experimental procedure for the column experiments is the same as that used by van Vliet *et al.* [14] where more details of the experimental procedure may be found.

Comparison of model and experiment

The film coefficient was estimated using equation (12). The initial effluent concentration was estimated by extrapolating from the experimental data. Any experimental points taken before about 100 ml of solution has passed through the column were ignored because they were artificially low due to dilution with interstitial water. The equilibrium data were obtained from the earlier experimental work of van Vliet [18]. He performed equilibrium experiments from which the required Freundlich isotherm parameters were determined. The relevant isotherm parameters and other pertinent physical properties of the adsorbents are listed in Table 1.

The only unknown parameter remaining is the surface diffusion coefficient D_{s}. This was estimated by minimizing the sum of squared errors between the measured concentrations and the model-predicted concentrations by choice of the diffusion coefficient. This was done on one representative breakthrough curve and it was found that this surface diffusion coefficient value gave a good prediction of all the other breakthrough curves for a given adsorbent. Additionally this whole procedure was tested on some of the experimental data by minimizing the sum of squared errors between the experimental con-

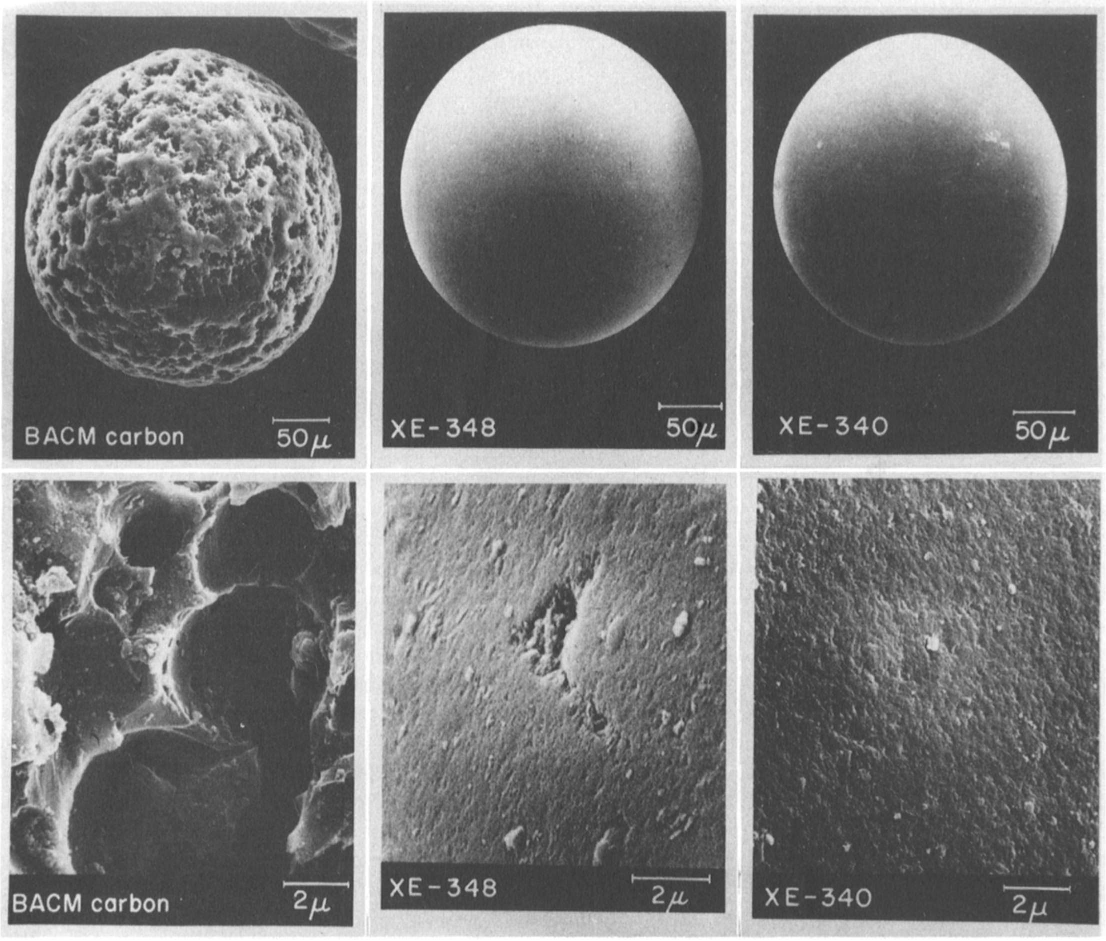

FIG. 1. Electron micrographs of the adsorbents, showing overall shape and texture and fine texture (after van Vliet and Weber [20]).

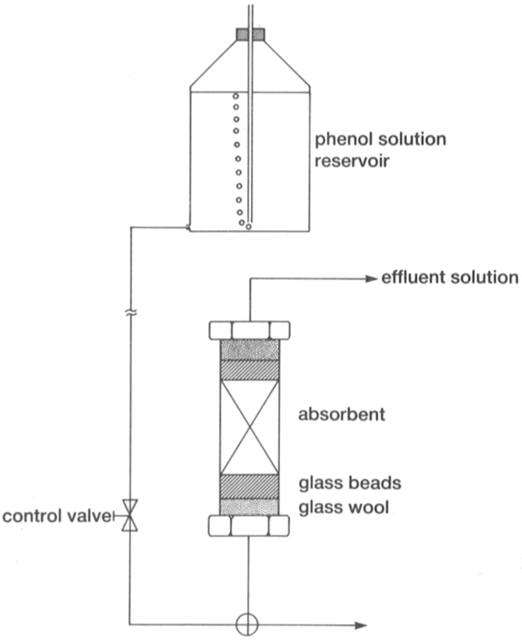

FIG. 2. Diagram of the experimental apparatus.

centrations and the model predicted concentration by choice of both the surface diffusion coefficient and the film transfer coefficient. This procedure which required a large amount of computer time was found to give very similar results.

The two parameter search technique was done on all experimental breakthrough curves for XE-348. The fitted value for D_s ranged from 0.56×10^{-9} to $0.70 \times 10^{-9}\ \mathrm{cm^2\ s^{-1}}$. There was no noticeable trend of D_s with Reynolds number. The two parameters may be estimated independently from the earlier and the later part of the breakthrough curve respectively since the early portion of the breakthrough curve is dominated by external film transfer and the later portion by surface diffusion [10]. In the work of Roberts *et al.* [11] the film coefficient was estimated using the earlier portion of the breakthrough curve only, and no account was taken of surface diffusion.

The fact that the D_s obtained is independent of the Reynolds number is noteworthy and indicates that D_s is indeed a measure of internal diffusion. The surface diffusion coefficients obtained are listed in Table 1. An example of the type of agreement obtained between the model and experiment is presented on

Table 1. Adsorbent properties

Adsorbent	A $[(\mathrm{mg\,g^{-1}})(\mathrm{l\,mg^{-1}})^{\gamma}]$	γ	ρ_p [g (dry adsorbent) $\mathrm{l^{-1}}$ (particle)]	r_0 [μm]	$D_s \times 10^9$ $[\mathrm{cm^2\,s^{-1}}]$
BACM	63.2	0.245	992	230	7.2
XE-348	42.9	0.231	984	180	0.66
XE-340	18.6	0.308	955	180	0.24

Figs. 3–5. On each figure three breakthrough curves are represented covering the whole range of Reynolds numbers used in this study. On the whole the agreement can be seen to be very good. The BACM carbon has a higher D_s value and the effect of film transfer is more marked. At the lowest Reynolds number the breakthrough curve is dominated by film transfer over the entire range of experimental points. This contrasts with XE-340 where the last experimental points are insensitive to the film transfer coefficient. Thus the effect of surface diffusion is more marked for XE-340; the earlier experimental points are nevertheless dominated by film transfer. As the Reynolds number increases the period of film transfer domination decreases. Owing to practical limitations in the experimental equipment it is not possible to measure the concentration at very small times and so the determination of the film coefficient becomes less accurate at higher Reynolds numbers. Thus the present method is not suitable for determining the film coefficient over a wide range of Reynolds numbers. The present results are well within the region of accuracy of the method since all the model curves are still sensitive to the film coefficient.

This point is illustrated on Fig. 6 where the sensitivity of the model to the film coefficient is shown. The model is least sensitive to the film coefficient when the Reynolds number is high. For this reason the highest experimental Reynolds number used for XE-340 and BACM carbon are shown. The centre model curve in each case represents the model with the previously estimated parameters. The lower and the upper model curves represent 25% more and 25% less than the estimated k_f, respectively.

The experimental results for BACM carbon can be seen to be more sensitive to k_f than those for XE-340. This is due to the lower surface diffusion coefficient of XE-340. The experimental results for XE-340 presented on Fig. 6 represent the experiment which is the least sensitive to k_f of all the experiments done. This sensitivity is acceptable and it may be concluded that the model is sensitive enough to the film coefficient, in the Reynolds number range used in this work. It can also be seen from Fig. 6 that the later part of

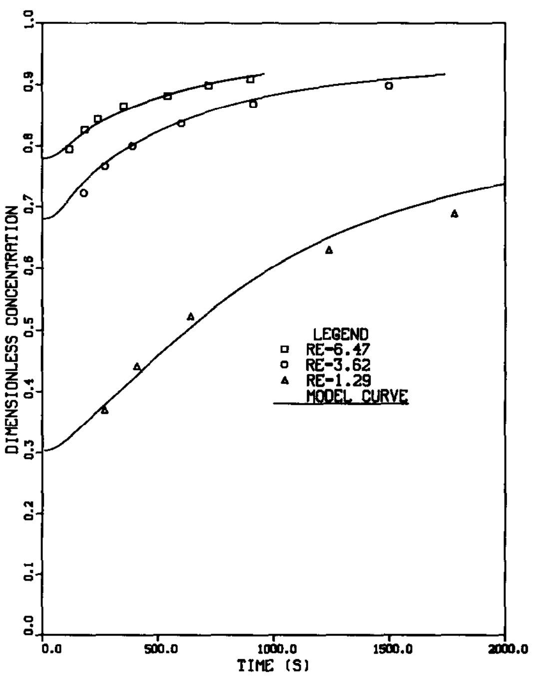

Fig. 3. Breakthrough curves for XE-340 resin.

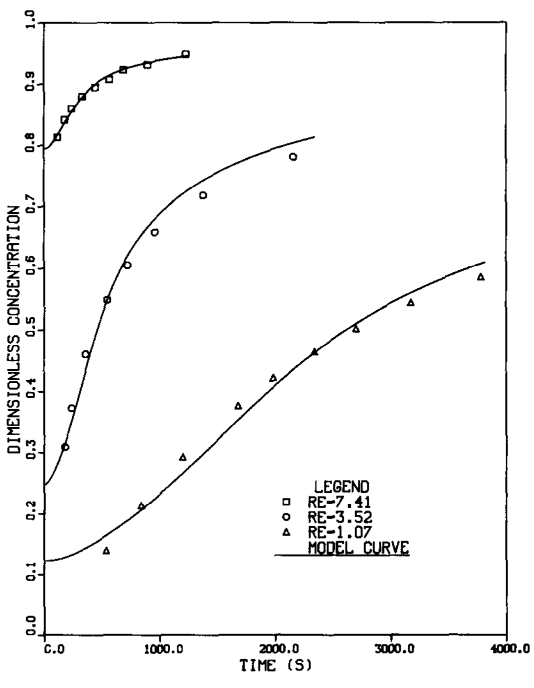

Fig. 4. Breakthrough curves for XE-348 resin.

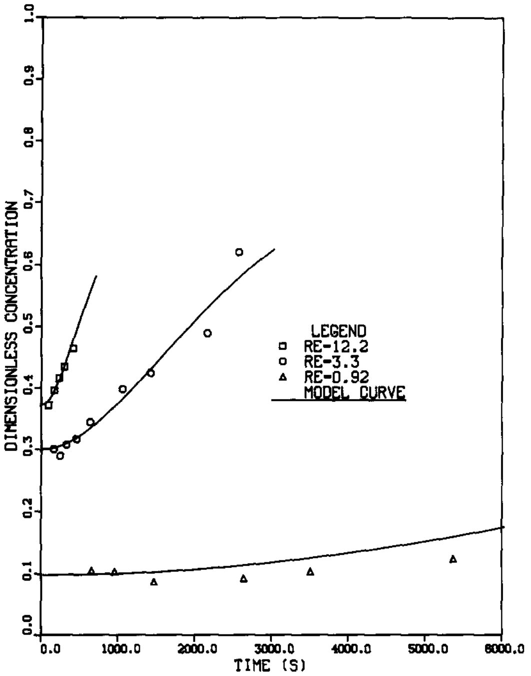

FIG. 5. Breakthrough curves for BACM carbon.

the breakthrough curve is independent of the film coefficient. This means that the surface diffusion coefficient can be determined from the later portion of the curve.

On the basis of the good agreement between the model and experiment it is felt that acceptable estimates of the film transfer coefficient are possible from the experimentally determined breakthrough curves.

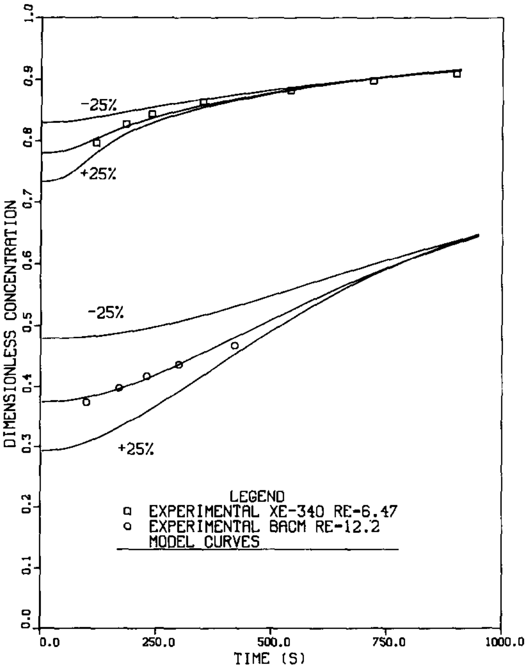

FIG. 6. Sensitivity of the model to the film coefficient.

RESULTS AND DISCUSSION

The experimental conditions and results are presented in Table 2.

The results were compared with existing correlations. Three representative correlations out of the many available, were chosen. These correlations were chosen in order to be representative of the ways in which mass transfer coefficients are measured. The first correlation is that of Williamson *et al.* [7] which was measured using dissolution of benzoic acid spheres and has the form

$$Sh = 2.4\varepsilon^{0.66}\,Re^{0.34}\,Sc^{0.42}. \tag{13}$$

This correlation suffers from the possible weakness that the surface roughness of the benzoic acid spheres changes as the dissolution proceeds. The value of ε used to plot equation (12) is 0.51 which is the voidage obtained for the BACM carbon columns. The work of Kaye *et al.* [19] has shown that the surface roughness may change significantly as dissolution occurs. It is believed that this may interfere with the experimental results.

The second correlation is that of Karabelas *et al.* [8] who did experiments using an electrochemical technique with one inert active sphere in a packed bed. This may not be truly representative of a packed bed where all the spheres are active. This correlation has the form

$$Sh^6 = (4.58Re^{1/3}\,Sc^{1/3})^6 + (2.39Re^{0.56}\,Sc^{1/3})^6. \tag{14}$$

Table 2. Conditions and results of the experiments

	Mass of dry adsorbent [g]	Q [ml min^{-1}]	L [cm]	$k_f \times 10^3$ [cm s^{-1}]
BACM	0.779	9.2	2.00	3.62
	0.643	22	1.65	7.48
	0.701	33	1.80	7.46
	0.935	39	2.40	8.33
	0.623	59	1.60	10.4
	0.934	66	2.40	10.1
	2.493	68	6.40	12.1
	1.168	120	3.00	16.0
	1.226	123	3.15	13.0
XE-348	1.145	13.8	3.15	2.5
	1.099	18.5	2.90	2.3
	1.236	28	3.50	2.6
	2.149	43.5	5.95	4.4
	1.291	50	3.50	4.0
	2.320	35	6.30	3.0
	1.814	58	4.90	4.4
	1.059	75	2.85	3.5
	1.258	76	3.40	3.8
	0.671	95	1.85	3.2
XE-340	1.851	16	4.20	1.04
	1.809	16.5	4.10	0.94
	1.610	42.0	3.65	1.10
	1.543	46.5	3.50	1.11
	2.118	63.0	4.80	1.10
	1.897	83.0	4.30	1.04
	1.476	86.0	3.35	1.10

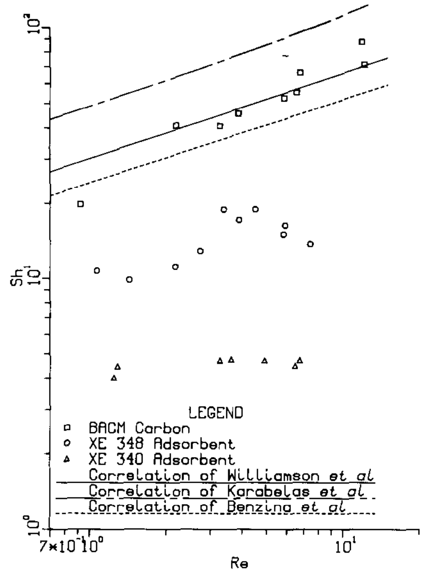

FIG. 7. Comparison of the present experimental work with some existing correlations.

The third correlation is that of Benzina *et al.* [9] who carried out experiments using an electrochemical technique where the whole bed forms the electrode. However, the electrode is not inert and plating of copper occurs on its surface. This may cause minute surface roughnesses on the particle surface which may interfere with the results. This correlation has the form

$$Sh = 2.277 Re^{1/3} Sc^{1/3}. \qquad (15)$$

These three correlations are compared with the experimental results of the present work on Fig. 7. The data for BACM carbon fits the Williamson correlation quite well. This correlation has been widely used in the design of activated carbon adsorbers [13]. It is immediately apparent from Fig. 7 that the experimental Sherwood number values for XE-340 are very much smaller than all existing experimental data. It is considered to be due to the exceptional smoothness of the XE-340 resin, and this is in essential agreement with results obtained in completely mixed batch reactor studies with the same adsorbents [2]. The results for the XE-348 resin lie between those of XE-340 and BACM. This is to be expected since XE-348 is rougher than XE-340 and smoother than BACM. In the earlier work done in a completely mixed batch reactor a mass transfer enhancement factor β was defined. This was defined to be the ratio of the Sherwood number of the adsorbent over the corresponding Sherwood number of XE-340 at the same Reynolds number. The XE-340 was used as the best available reference for a smooth sphere. The average β value obtained for a number of different adsorbates for XE-348 and BACM carbon were 4.3 and 7.5, respectively. Inspection of Fig. 6 shows that the present results are consistent with the earlier work. These results emphasize the very significant impact of surface roughness in fluid-to-particle, mass transfer.

In the earlier work done on flat plates [4–6] the slope of the Sherwood Reynolds number plot on log axes (the exponent on the Reynolds number in the correlations) was shown to be dependent on surface roughness. The same type of phenomenon is manifested here, and the difference in slope is particularly evident between the results for XE-340 and BACM. This means that β values are dependent not only on surface roughness but on Reynolds number as well.

CONCLUSIONS

The present work shows that surface roughness may have a very significant impact on fluid-to-particle mass transfer in packed beds. This aspect of mass transfer has been largely ignored in the past and deserves further attention.

It is apparent that the method used to measure mass transfer coefficients in packed beds has to be very carefully selected, firstly to ensure that it is representative of what happens in the packed bed and secondly to ensure that the particle surface does not change during the course of the experiment. The method used in this paper meets these requirements but is not suited to experiments over a wide range of Reynolds numbers. The method of Benzina *et al.* [9] using an inert active bed would alleviate this problem.

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EFFET DE LA RUGOSITE DE SURFACE SUR LE TRANSFERT DE MASSE FLUIDE–PARTICULE DANS UN LIT FIXE ADSORBANT

Résumé—La rugosité de surface a un effet marqué sur le transfert de masse fluide–particule dans un lit fixe adsorbant. Les coefficients de transfert de masse sont déterminés en utilisant trois absorbants sphériques dans la domaine de nombre de Reynolds 0,9–12, et une technique sur mini-colonne. Des courbes expérimentales sont modélisées de façon satisfaisante avec un modèle qui tient compte du transfert de film externe et de la diffusion de surface intraparticulaire. Le nombre de Schmidt utilisé est 1190. On montre que la rugosité de la surface de la particule d'adsorbant augmente significativement le coefficient de transfert de masse dans le film externe.

EINFLUSS DER OBERFLÄCHENRAUHEIT AUF DEN STOFFÜBERGANG ZWISCHEN FLUID UND PARTIKELN IN EINEM FESTBETTABSORBER

Zusammenfassung—Es wird gezeigt, daß die Oberflächenrauheit einen bedeutenden Einfluß auf den Stoffübergang zwischen Fluid und Partikeln in einem Festbettabsorber hat. Stoffübergangskoeffizienten werden für drei kugelförmige Absorptionsmittel im Reynolds-Zahl-Bereich von 0,9–12 bestimmt. Es werden experimentell ermittelte Kurven unter Benutzung eines Modells dargestellt, welches externe Stoffübertragung durch einen Film und Oberflächendiffusion innerhalb der Partikel beinhaltet. Die verwendete Schmidt-Zahl beträgt 1190. Die Oberflächenrauheit der Absorptionspartikel erhöht den externen Film-Stoffübergangskoeffizienten bedeutend.

ВЛИЯНИЕ ШЕРОХОВАТОСТИ ПОВЕРХНОСТИ НА МАССОПЕРЕНОС ОТ ПОТОКА К ЧАСТИЦЕ В ПЛОТНОМ СЛОЕ АДСОРБЕНТА

Аннотация—Показано, что шероховатость поверхности оказывает существенное влияние на массоперенос от потока к частице в плотном слое адсорбента. С помощью техники мини-колонны определяются коэффициенты массопереноса для трех сферических адсорбентов в диапазоне чисел Рейнольдса 0,9–12. Экспериментальные кривые отклика успешно моделировались с помощью модели, учитывающей перенос через внешнюю пленку и внутреннюю диффузию на поверхности. Используется число Шмидта, равное 1190. Показано, что шероховатость поверхности частицы адсорбента существенно увеличивает коэффициент массопереноса через внешнюю пленку.