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Mass transfer studies on the biodegradation of phenols in up-flow packed bed reactors

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Abstract

A quantitative analysis of the effect of mass transfer coefficients on the operating parameters namely, flow rate, bed height, particle and column diameter, voidage and initial concentration of phenols, etc. is carried out for the biodegradation of phenols in an up-flow packed bed reactor. A mass transfer correlation of the type, $j_D = K\,(Re')^{n-1}$ is developed to represent the present experimental data obtained using activated carbon–*Pseudomonas pictorum*–alginate, celite–*P. pictorum*–alginate and *P. pictorum*–alginate beads for the continuous biodegradation of phenol. The overall reaction rate was found to be of the first order with the mass transfer as the rate-limiting step. For mass transfer with biochemical reaction, a correlation of experiment with theory is made. A realistic estimate of the effects of external mass transfer coefficients is also made in this present work.

Keywords: Biodegradation; Phenol; Mass transfer coefficients; Packed bed reactors; *P. pictorum*

1. Introduction

Recently, considerable attention has been focused towards using immobilized beads for the degradation of stable organic compounds present in the industrial effluents in continuous reactors, due to high cell density in the reactor even beyond wash out conditions [1]. Immobilization binds the microorganism to a solid support and is a superior alternative to the conventional adsorption methods. The immobilized cell reactors with various reactor configurations [2–4] namely, packed bed reactors, fluidized bed, inversed fluidized beds and tapered beds have been reported and have gained wide importance due to the advantages offered by continuous process over batch operation. The

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Nomenclature

a_m	external surface area for mass transfer ($cm^2\ cm^{-3}$)
A	parameter given by Eq. (11)
C_s	substrate concentration at the surface of immobilized cell ($g\ cm^{-3}$)
C_t	substrate concentration ($g\ cm^{-3}$)
C_0	initial substrate concentration ($g\ cm^{-3}$)
d_c	column diameter (cm)
d_p	particle diameter (cm)
D_f	diffusivity of the substrate ($cm^2\ s^{-1}$)
G	mass flux based on superficial velocity ($g\ cm^{-2}\ s^{-1}$)
H	bed height (cm)
j_D	dimensionless group given by Eq. (9)
k	intrinsic first order rate constant ($cm\ s^{-1}$)
k_f	mass transfer coefficient ($cm\ s^{-1}$)
k_{ps}	pseudo first order rate constant (s^{-1})
K	constant in Eq. (9)
K_m	Michaelis–Menten constant ($g\ cm^{-3}$)
n	exponent in Eq. (10)
N_{ave}	mass transfer rate ($g\ s^{-1}$)
N_{Da}	Damkohler number
Q	volumetric flow rate ($cm^3\ s^{-1}$)
r_r	reaction rate ($g\ cm^{-3}\ s^{-1}$)
Re	Reynolds number (d_pG/μ)
Re'	modified Reynolds number [$d_pG/\mu(1-\varepsilon)$]
Sc	Schmidt number ($\mu/\rho D_f$)
Sh	Sherwood number ($k_f d_p/D_f$)
t	time (s)
X	fractional substrate conversion

Greek letters

ε	bed voidage
μ	fluid viscocity ($g\ cm^{-1}\ s^{-1}$)
ρ	fluid density ($g\ cm^{-3}$)
τ	space time (s)
ν_{max}	maximum specific growth rate ($g\ cm^{-3}\ s^{-1}$)
ν	specific growth rate ($g\ cm^{-3}\ s^{-1}$)

main advantage of a continuous process for the degradation of toxic materials over a batch process is the ease of automation and control, which can lead to a reduction in operational cost and a high increase in throughput with a more consistent rate of treatment. Even though, many stable organic pollutants/toxic compounds are present in the effluent liquors, phenol is considered to be an important toxic as well as a stable com-

pound, which is mainly present in the effluents of petroleum and coal based industries apart from plastics and dye industries. The available literature on the continuous degradation of phenols using immobilized beads, as packing/fluidizing medium are very scanty although some effects on growth kinetics of phenol degrading cultures exist in the literature [5]. Fan and coworkers [6–9] have reported an extensive study on the modeling and simulation of phenol degradation using immobilized culture on activated carbon in a fluidized bed reactor. Cho et al. [10] have used a reactor configuration of packed bed for ethanol production using immobilized yeast. In this present work, an attempt is made to develop a reactor module in the form of packed bed reactor, for the continuous degradation of phenols, using *Pseudomonas pictorum*–alginate beads. Theoretical work on the packed bed bioreactors, using immobilized beads is very scarce and no generalized correlation for the accurate estimation of mass transfer coefficients is available. This present investigation is an attempt to develop a unified correlation for the estimation of mass transfer coefficients as a function of the operating variables, viz. diameter of the beads, bed height, column diameter, flow rates, initial concentration of the substrate and carrier material, etc. for the continuous degradation of phenols in a packed bed reactor using *P. pictorum*–alginate beads taking into account the biochemical reaction also.

2. Experimental

2.1. Microorganism

P. pictorum (NCIM 2077) was obtained from the National Chemical Laboratory, Pune, India and the stock cultures were maintained on nutrient agar medium.

2.2. Minimal medium

The minimal medium consisting of (per liter) KH_2PO_4: 1.5 g; K_2HPO_4: 0.5 g; NaCl: 0.5 g; $MgSO_4{\cdot}7H_2O$: 0.5 g; NH_4NO_3: 3.0 g; $FeSO_4{\cdot}7H_2O$: 0.02 g; $CaCl_2{\cdot}2H_2O$: 0.02 g; glucose: 0.5 g; yeast extract: 2.0 g was prepared and the final pH of the medium was maintained at 7. Phenol, calcium and iron salts were filter sterilized and added separately to the medium to avoid the precipitation of calcium and iron salts. Even though the phenol oxidizing bacteria grow well in mineral medium, glucose and yeast extract were added to the medium to enhance the rate of degradation of phenol [11]. Phenol was determined quantitatively by the photometric method using 4-aminoantipyrine as the coloring agent [12]. The optical density of the microorganism was measured using an UV spectrophotometer at $\lambda_{max} = 600$ nm. The microorganism suspension containing cells of *P. pictorum* (NCIM 2077) was immobilized on to a known amount of activated carbon and celite in sodium alginate solution. Beads of approximately uniform sizes (0.8, 0.5 and 0.2 cm) were prepared and hardened in $CaCl_2$ solution.

Glass columns of two different diameters (3.8 and 6.7 cm) were used as reactors. Initially, the column was filled with *P. pictorum*–alginate beads on to the required bed height. A known concentration of synthetic aqueous phenolic solution was pumped into the bottom of the reactor, using a peristaltic pump (Miclins PP 20) at a desired flow rate (Table 1) and the

Table 1
Range of variables used in the present study

S. no.	Variables	Range
1	Diameter of the column (cm)	3.8–6.7
2	Particle diameter (cm)	0.2–0.8
3	Flow rate (cm/s)	0.005–0.176
4	Height of the packing (H/d_c)	1.34–11.25
5	Initial phenol concentration ($\times 10^3$ g cm^{-3})	0.501–2.504

experiments were carried out at room temperature with an aeration rate of 0.14 l min^{-1}. Samples were collected from the outlet connected at the top of the reactor, at fixed intervals of time, for desired experimental conditions. The experiments were repeated for different conditions by changing the liquid flow rates, bed heights, diameter of the beads and the diameter of the column, etc. The range of the variables used for this present analysis are given in Table 1.

3. Results and discussion

In a specific continuous reactor with a packed bed configuration, with up-flow mode of operation, the mass transfer consideration indicates that particle size, particle shape, pore size, enzyme loading per particle, and substrate flow rate can all affect the rate of degradation. The present experimental data were obtained using immobilized beads with relatively uniform sizes. Three different types of beads were used in the present study, viz. *P. pictorum*–alginate, activated carbon–*P. pictorum*–alginate [13] and celite–*P. pictorum*–alginate. In the latter two cases, activated carbon and celite were used to improve the strength of the bead matrix.

The fluid velocities at regions near the surface of the particles are very low when fluid flows through a bed of particles. A near stagnant film of fluid is present around the exterior of the particles through which the substrate has to be transported. The transport takes place primarily by molecular diffusion and since the rate of molecular diffusion may be quite slow, the observed reaction rate decreases significantly due to increased film thickness [14]. The intrinsic adsorption rate was very rapid until an external surface coverage of phenol or substrate had occurred on the *P. pictorum*–alginate beads or activated carbon–*P. pictorum*–alginate beads or celite–*P. pictorum*–alginate beads.

The present data on biodegradation of phenol are interpreted by assuming a three step model, viz. (i) mass transfer of phenol from bulk liquid to the surface of the alginate beads; (ii) intra particle diffusion; and (iii) the biochemical reaction at the interior surface of the beads. For the case of biodegradation of phenol using alginate beads immobilized with *P. pictorum*, it is assumed that the biochemical reaction is rapid with respect to the first two steps.

The biodegradation of phenol using *P. pictorum* was found to obey the Michaelis–Menten kinetics and the estimated ν_{max} and K_m are 0.1124×10^{-4} g cm^{-3} s^{-1} and 2.4×10^{-3} g cm^{-3}, respectively. Fig. 1 shows the sample Linewaever Burk plot for a particle diameter of 0.2 cm.

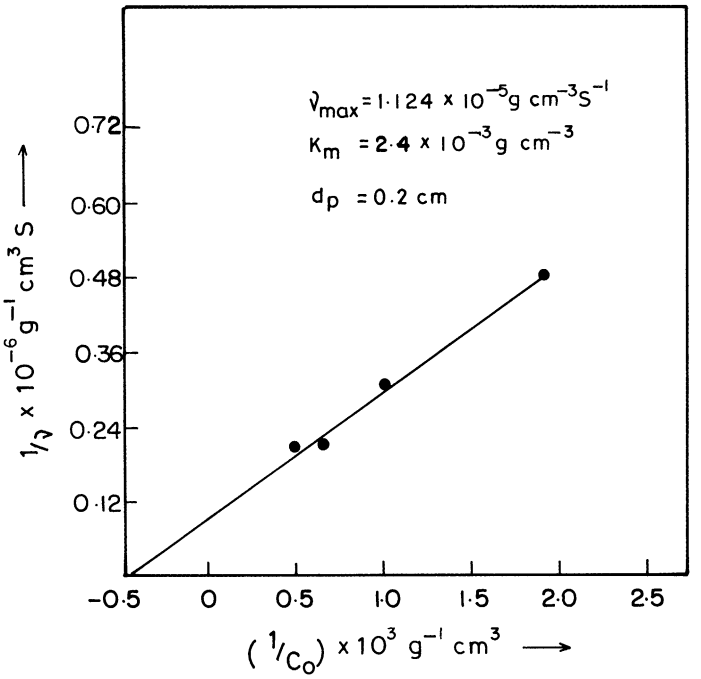

Fig. 1. Linewaever Burk plot for *P. pictorum*–alginate beads.

Material balance coupled with flux, represented in terms of mass transfer coefficients, external surface area for mass transfer and the concentration gradient have been used for the estimation of mass transfer coefficients, i.e.

$$k_f a_m = \frac{N_{ave}}{V_{eff}\,\Delta C} \tag{1}$$

where N_{ave} is the mass transfer rate (g s^{-1}) given as $N_{ave} = (C_1 - C_2)Q$, V_{eff} the effective volume of the reactor and ΔC the overall concentration difference $(C_0 - C_s)$. The surface area for mass transfer was determined from the knowledge of the bed voidage (ε) and the equivalent particle diameter d_p, i.e.

$$a_m = \left[\frac{(1-\varepsilon)\pi d_p^2}{\pi d_p^3/6}\right] = \frac{6(1-\varepsilon)}{d_p} \tag{2}$$

Under steady-state conditions, the rate of supply of phenol by mass transfer is equal to the phenol consumed at the interface of the catalyst. Applying Michaelis–Menten kinetics at the surface [15], the reaction rate is given by

$$k_f a_m (C_0 - C_s) = \frac{\nu_{max} C_s}{K_m + C_s} \tag{3}$$

Damkohler number, N_{Da}, which is defined as the ratio of the maximum reaction rate to the maximum mass transfer rate, i.e.

$$N_{Da} = \frac{\nu_{max}}{k_f a_m C_0} \tag{4}$$

was calculated using the estimated values of ν_{max} and k_f. The Damkohler number thus estimated for three different sizes ($d_p = 0.8$, 0.5 and 0.2 cm) of immobilized beads were found to be far greater than 1. Since $N_{Da} \gg 1$, the mass transfer resistance is large or in other words, mass transfer rate is much less than the reaction rate with the overall reaction rate being a first order with the mass transfer as the limiting process.

3.1. Mass transfer with biochemical reaction

At steady state, the rate of mass transfer of phenol by diffusion is equal to the rate of the first order reaction (r_r)

$$r_r = k a_m C_s \tag{5}$$

where k is the intrinsic first-order rate constant. At steady-state conditions, the unknown phenol concentration at the surface C_s is given as [14]

$$C_s = \frac{k_f C_0}{k + k_f} \tag{6}$$

Substituting Eq. (6) into Eq. (5) and integrating, the mass balance for a plug flow reactor is expressed as

$$\ln\left(\frac{C_t}{C_0}\right) = \ln(1 - X) = -k_{ps}\tau \tag{7}$$

where X is the fractional substrate conversion ($1 - (C_t/C_0)$) and τ the space time (V/Q). The pseudo first order rate constant (k_{ps}), used to elucidate the film diffusion model [14], is given by

$$k_{ps} = \frac{k k_f a_m}{k + k_f} \tag{8}$$

The factor, j_D, a dimensionless term [16], which could be defined in terms of Schmidt Number and Reynolds Number, representing the parameters such as mass transfer coefficients, mass flow rate, density, viscosity and the diffusion coefficients, etc. is used to correlate the present mass transfer data, i.e.

$$j_D = \left(\frac{k_f \rho}{G}\right)\left(\frac{\mu}{\rho D_f}\right)^{2/3} = K\, Re^{n-1} \tag{9}$$

For different conditions of mass transfer, the value of n in Eq. (9) varies from 0.1 to 1.0.

Rearranging Eq. (9) and solving for the mass transfer coefficients, the equation becomes

$$k_f = AG^n \tag{10}$$

where the parameter A is defined as

$$A = \left(\frac{K}{\rho}\right)\left(\frac{\mu}{\rho D_f}\right)^{-2/3}\left(\frac{d_p}{\mu}\right)^{n-1} \tag{11}$$

Substituting Eq. (10) into Eq. (8) and rearranging to yield

$$\left(\frac{1}{k_{ps}}\right) = \left(\frac{1}{Aa_m}\right)\left(\frac{1}{G^n}\right) + \left(\frac{1}{ka_m}\right) \tag{12}$$

A plot of the estimated values of $(1/k_{ps})$ versus $(1/G^n)$, yields a straight line of slope $(1/Aa_m)$ and an intercept $(1/ka_m)$. This analysis was made for all values of n between 0.1 and 1.0. However, not all values of n provide a satisfactory mass transfer correlation for this present study on immobilized packed bed reactor using *P. pictorum*–alginate beads. The value of n that yields a value of a_m matching with the experimental a_m (obtained using Eq. (2)) provides the correct external mass transfer correlation. The experimental data consisting of three different particle sizes, viz. 0.8, 0.5 and 0.2 cm with an a_m of 4.28, 7.08 and 20.1 $cm^2\ cm^{-3}$, respectively, are analyzed separately to study the dependency of mass transfer coefficients on the operating variables.

The sample experimental data of Q and k_{ps}, and the calculated values of $1/k_{ps}$, Re, G, and $1/G^n$ $(0 < n < 1)$ are compared in Tables 2–4 for the packed bed reactor using immobilized *P. pictorum*–alginate beads for $d_p = 0.2$, 0.5 and 0.8 cm. Fig. 2 shows the plot of $(1/k_{ps})$ versus $(1/G^n)$ for an assumed value of $n = 0.72$ for $d_p = 0.2$, 0.5 and 0.8 cm. The value of the intrinsic first order rate constant, k, was calculated from the intercept $(1/ka_m)$ as 0.11×10^{-3} cm s^{-1} and the estimated value of K in Eq. (9) for $d_p = 0.8$ cm was found to be 1.34 which predicts an a_m value of 4.32 $cm^2\ cm^{-3}$ which is in good agreement with the calculated value of a_m according to Eq. (2) as 4.28 $cm^2\ cm^{-3}$. The same for $d_p = 0.5$ cm with K value of 1.43, predicts an a_m value of 6.94 $cm^2\ cm^{-3}$ which is in good agreement with the estimated value of a_m according to Eq. (2), i.e. 7.08 $cm^2\ cm^{-3}$. For $d_p = 0.2$ cm, the calculated and the actual values of a_m are 20.46 and 20.1 $cm^2\ cm^{-3}$, respectively, for an estimated value of $K = 1.51$ (Eq. (9)) showing a good agreement. With these estimated values of K and n, the mass transfer correlation could be represented as

$$j_D = K\,Re^{-0.28} \tag{13}$$

where the values of K are 1.34, 1.43 and 1.51 for $d_p = 0.8$, 0.5 and 0.2 cm, respectively which accurately predicts this present experimental data on mass transfer coefficients for the biodegradation of phenols in a packed bed reactor with immobilized *P. pictorum*–alginate beads. Similarly, activated carbon–*P. pictorum*–alginate beads show an a_m value of 7.63 and 19.00 $cm^2\ cm^{-3}$ for $d_p = 0.5$ and 0.2 cm with a K value of 2.15 and 2.20, respectively.

Fig. 3 shows the plot of mass transfer coefficient versus mass flow rate for three different particle diameters using *P. pictorum*–alginate, activated carbon–*P. pictorum*–alginate, and celite–*P. pictorum*–alginate beads in logarithmic coordinates. The values of A and n thus obtained from Fig. 3, are compared with that calculated according to Eq. (11) and are given in Table 5. The good agreement of these values confirm the applicability of this present proposed mass transfer model for this study on packed bed reactors for the continuous degradation of phenols. The results indicate that phenol degradation is

Table 2
Values of measured parameters used in Eq. (12)[a]

S. no.	Q ($cm^3\,s^{-1}$)	G ($g\,cm^{-2}\,s^{-1}$)	k_f ($\times 10^3\,cm\,s^{-1}$)	Re	k_{ps} ($\times 10^3\,s^{-1}$)	$1/k_{ps}$ (s) (experimental)	$1/G^{0.72}$	$1/k_{ps}$ (s) (calculated)
1	0.16	0.014	0.00753	0.3268	0.14116	7084.1	21.61	7429.1
2	0.5	0.043	0.01723	1.0212	0.2971	3365.8	9.636	3563.3
3	1.0	0.087	0.02788	2.0425	0.4426	2259.4	5.801	2325.1
4	1.5	0.131	0.03755	3.0638	0.5546	1803.1	4.320	1846.9
5	2	0.175	0.04590	4.0851	0.6403	1561.7	3.507	1584.5

[a] $C_0 = 0.501 \times 10^{-3}\,g\,cm^{-3}$, $d_p = 0.2\,cm$, $d_c = 3.8\,cm$.

Table 3
Values of measured parameters used in Eq. (12)[a]

S. no.	Q ($cm^3\ s^{-1}$)	G ($g\ cm^{-2}\ s^{-1}$)	k_f ($\times 10^3\ cm\ s^{-1}$)	Re	k_{ps} ($\times 10^3\ s^{-1}$)	$1/k_{ps}$ (s) (experimental)	$1/G^{0.72}$	$1/k_{ps}$ (s) (calculated)
1	0.16	0.014	0.00545	0.8170	0.0366	27322.4	21.61	28316.2
2	0.5	0.043	0.01198	2.5532	0.0760	13157.8	9.636	13337.8
3	1.0	0.087	0.01995	5.2698	0.1185	8438.8	5.801	8540.57
4	1.5	0.131	0.02671	7.6596	0.1505	6644.5	4.320	6687.97
5	2	0.175	0.03106	10.212	0.1693	5906.6	3.507	5670.98

[a] $C_0 = 0.501 \times 10^{-3}\ g\ cm^3$, $d_p = 0.5\ cm$, $d_c = 3.8\ cm$.

Table 4
Values of measured parameters used in Eq. (12)[a]

S. no.	Q ($cm^3\,s^{-1}$)	G ($g\,cm^{-2}\,s^{-1}$)	k_f ($\times 10^3\,cm\,s^{-1}$)	Re	k_{ps} ($\times 10^3\,s^{-1}$)	$1/k_{ps}$ (s) (experimental)	$1/G^{0.72}$	$1/k_{ps}$ (s) (calculated)
1	0.5	0.0141	0.00450	0.8217	0.0183	54644.8	21.506	56294.4
2	1.0	0.0282	0.00748	1.6434	0.0295	33898.3	13.056	35010.1
3	1.5	0.0423	0.00985	2.4651	0.0380	26315.7	9.7374	26651.1
4	2	0.0565	0.01205	3.2868	0.0456	21929.8	7.9163	22063.9

[a] $C_0 = 0.501 \times 10^{-3}\,g\,cm^{-3}$, $d_p = 0.8\,cm$, $d_c = 6.7\,cm$.

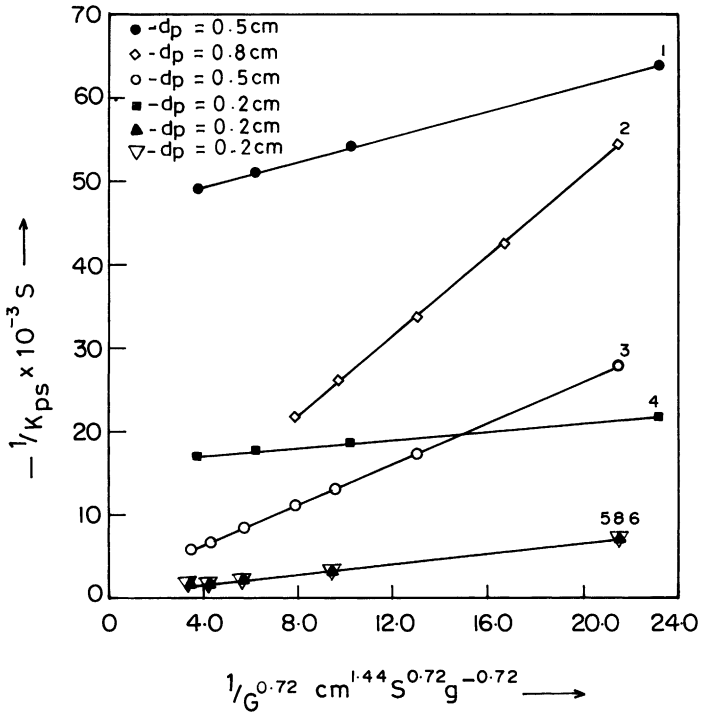

Fig. 2. Plot of $(1/k_{ps})$ vs. $(1/G^n)$ for $n = 0.72$: (1 and 4) activated carbon–*P. pictorum*–alginate beads; (2, 3 and 5) *P. pictorum*–alginate beads; (6) celite–*P. pictorum*–alginate beads.

controlled by the influent flow rate. When the input flow rate increases, the total phenol input per minute increases, resulting in reduction of the surface film thereby increasing the rate of mass transfer [17,18]. The estimated mass transfer coefficients are found to increase with a decrease in the particle diameter. The higher mass transfer coefficient in case of smaller particles is due to the increased surface area [19]. It has also been observed that there is not much change in the mass transfer coefficient due to the increase in bed height.

Table 5
Experimental and calculated values of the parameter A used in Eq. (10)

S. no	d_p (cm)	n	A ($\times 10^4$ g$^{0.72}$ cm$^{-2.44}$ s$^{0.28}$) (experimental using Eq. (11))	A ($\times 10^4$ g$^{0.72}$ cm$^{-2.44}$ s$^{0.28}$) (calculated from Fig. 3)
1	0.2	0.72	1.60 2.38[a]	1.62 2.37[a]
2	0.5	0.72	1.17 1.75[a]	1.17 1.78[a]
3	0.8	0.72	0.963	0.969

[a] Activated carbon–*P. pictorum*–alginate.

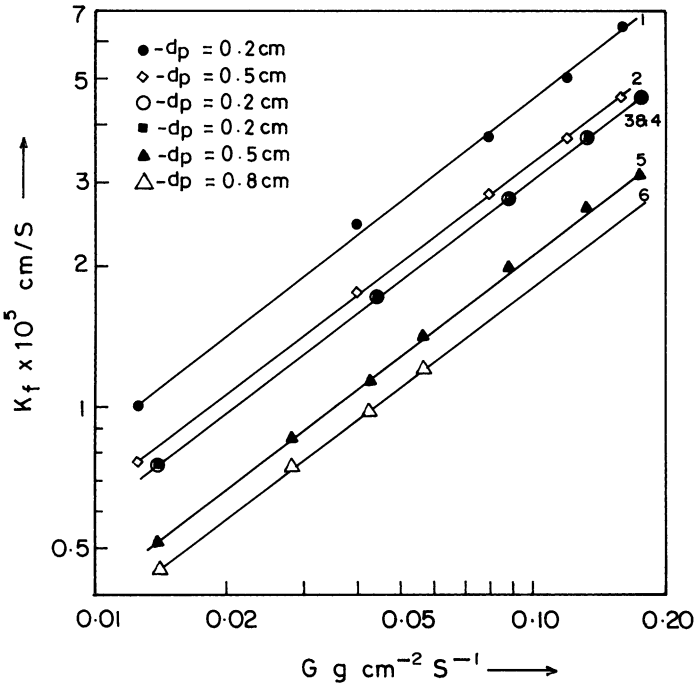

Fig. 3. Effect of mass flow rate on external mass transfer coefficient using different particle diameters: (1 and 2) activated carbon–*P. pictorum*–alginate beads; (3, 5 and 6) *P. pictorum*–alginate beads; (4) celite–*P. pictorum*–alginate beads.

It is also observed from the analysis of the present data, that the value of the constant K in Eq. (9) varies with bed voidage. Since the observed mass transfer rates are directly proportional to the particle size, assuming the particles to be spherical in shape, the effect of bed voidage in the form of ε or $(1-\varepsilon)$ have to be considered instead of the particle diameter d_p. The general form of the correlation in terms of modified Reynolds number is represented as

$$j_D = K(Re')^{n-1} \tag{14}$$

where $Re' = Re/(1-\varepsilon)$. The dependency of j_D on Re' for the biodegradation of phenol using immobilized *P. pictorum* on alginate can be given as follows:

$$j_D = 1.56(Re')^{-0.28} \tag{15}$$

The RMS error for the above correlation was found to be 6.73%. The present experimental results consisting of 472 measurements on mass transfer coefficients obtained using *P. pictorum*–alginate (288 data points), activated carbon–*P. pictorum*–alginate (120 data points) [13] and celite–*P. pictorum*–alginate (64 data points) are used for the regression analysis. The estimated values of constants K, and the indices $n-1$, in Eq. (14), are given in Table 6.

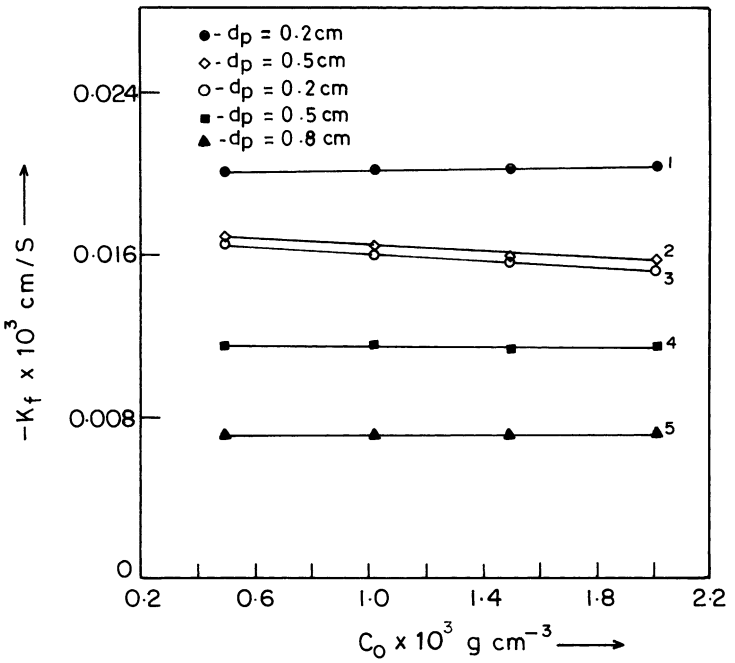

Fig. 4. Effect of initial phenol concentration on the external mass transfer coefficient for different particle diameters at $Q = 1.0\,\mathrm{cm^3\,s^{-1}}$ for *P. pictorum*–alginate beads: (1 and 2) activated carbon–*P. pictorum*–alginate beads; (3–5) *P. pictorum*–alginate beads.

Since celite is an inert material, it acts as a supporting material for the immobilized beads and hence, the constants and the indices are same for the cases 1 and 3. In activated carbon–*P. pictorum*–alginate [13] system, it is expected that apart from the diffusion with biochemical reaction, a small amount of phenol gets adsorbed on the active sites of activated carbon showing a higher value of mass transfer coefficient and hence, a higher correlation constant when compared with other values [20,21].

The effect of initial phenol concentration on the external mass transfer coefficient using three different particle diameters for particular flow rates are shown in Fig. 4. The variation in the mass transfer values with respect to the change in the initial concentration of phenol is negligible, which may be due to the fact that the concentrations used in this study are very low, i.e. very dilute solutions. The basis for choosing these ranges of variation in

Table 6
Constants and indices for Eq. (14)

S. no.	System	K	$n-1$	RMS (%)
1	*P. pictorum*–alginate	1.56	−0.28	6.73
2	Activated carbon–*P. pictorum*–alginate [13]	2.26	−0.28	8.44
3	Celite–*P. pictorum*–alginate	1.56	−0.28	7.2

concentration was, on one hand, that they are approximately equal to the actual effluent concentration that is in the order of ppm level and on the other hand, they could be easily prepared and handled in the laboratory.

4. Conclusion

The present data on external mass transfer coefficients obtained in packed bed reactors using *P. pictorum*–alginate beads, with and without supporting materials (activated carbon and celite), for the degradation of phenols are correlated with the operating variables, viz. substrate flow rate, particle diameter and bed voidage. For steady-state mass transfer combined with biochemical reaction in a packed bed reactor, a mass transfer correlation (Eq. (14)) is developed, which represents the present experimental data accurately. This proposed correlation would be useful for the design and development of up-flow packed bed reactors for the continuous degradation of phenol using activated carbon–*P. pictorum*–alginate, celite–*P. pictorum*–alginate and *P. pictorum*–alginate beads. This proposed model would be useful to quantify the external film diffusion effects for the continuous degradation of phenols in packed bed reactors.

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