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INCREASING THE ADSORPTION SURFACE AREA OF ACTIVATED CARBON

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ABSTRACT

Experiments were carried out to study the effect of various activated carbon-glass beads weight ratios, influent phenol concentrations, flow rate and bed depth on the performance of fixed bed adsorption column. The equilibrium data and interparticle diffusion coefficients obtained from separate experiments in a batch adsorber were fitted with theoretical model.

The effect of adding different weight ratios of an inert solid material to the adsorbent bed in the adsorption process of phenol onto activated carbon was carried out. Adding 5 wt % glass beads to the activated carbon bed reduces the amount of activated carbon by 5% and increases the operating time by 80%.. Increasing the glass beads from 10 wt % and above makes the adsorption process inefficient compared with 0 wt % glass beads.

A mathematical model was achieved to describe the mass transfer kinetics in a fixed bed column. The results show that the mathematical model includes external mass transfer and pore diffusion using nonlinear isotherms, provides a good description of the adsorption process for phenol in fixed bed adsorber.

KEY WORDS

Fixed bed adsorption, activated carbon, phenol removal, mathematical model of adsorption process.

INTRODUCTION

Phenolic compounds are very poisonous organic compounds that enter the aquatic environment through direct discharge from such industrial sources as cook ovens in steel plants, oil refineries, phenolic resin plants, and pharmaceutical and pesticide manufacturers. The content of the phenolic compounds in the industrial wastewater (about 200-2000 mg/l) is usually higher than the standard limit (0.1 mg/l) established for their release into the environment. Therefore, attention has been paid to the removal of phenolic compounds from industrial wastewater by many researchers around the world using activated carbon (1, 2).

Granular activated carbon (GAC) is efficient in adsorption of numerous bio-resistance organic pollutants from aqueous system, activated carbon adsorption has been recommended by the U.S Environmental Protection Agency (USEPA) as one of the best available technologies for synthetic organic chemical removal (3).

Due to their high porosity, activated carbon is the most widely used material for adsorption of chemicals (4). The adsorption process of a solute on an activated carbon takes place at the liquid-solid boundary; it is thus clearly a “heterogeneous” reaction, and the interface of the two phases represents a special environment under dimensional or topological constraints (5).

To design and operate a fixed bed adsorption process successfully, the column dynamics must be understood; that is the breakthrough curves under specific operating conditions must be predictable. A mathematical model of fixed bed adsorber is very useful for the proper design and the determination of the optimal operating conditions for this equipment. Different models for the adsorption process have been proposed in the literature (6). The differences in the mathematical models of adsorption processes arise from the different representation of the equilibrium behavior, the different expressions of the mass transfer inside and outside the adsorbent particle and from how the axial dispersion is taken into account in the material balance for the adsorbate in the fixed bed column. Also various numerical techniques have been developed for the solution of the fixed bed adsorber models.

Several rate models have been developed that take into account an external film transfer rate step, unsteady state transport in the solid phase and nonlinear equilibrium isotherm to predict adsorption rates in batch reactor and fixed bed (7).

Under a wide range of operating conditions, the key process parameters in adsorption such as isotherm constants and mass transfer coefficients are established by conducting batch studies of adsorption. Langmuir, Freundlich, Radke-Prausnitz, Reddlich-Peterson and Combination of Langmuir-Freundlich isotherms have been applied to describe the equilibrium between liquid-solid phases. The parameters that are responsible for mass transfer operation are the external mass transfer coefficient and interparticle diffusivity or surface diffusion coefficient.

The liquid hold-up is an important characteristic of packing owing to its relation to the wetted area. There are two types of hold-up; static and dynamic (operating) hold-up. The static hold-up was measured as the weight of liquid retained when the column has drained to a constant weight; this was deducted from the total hold-up to obtain the operating hold-up (8). Investigators measured the hold-up for different aqueous solutions and they found that the static hold-up for carbon is greater than that for porcelain, which may be due to porosity of carbon material (8). Experimental work showed that for water flowing over vertical column of spheres, the total hold-up was equal $(0.4+0.596l^{1/3})$ gm per sphere, where l is the flow rate cm^3/sec (9). The first term is the static hold-up due to the meniscus at the junction of the spheres and the second term is the dynamic hold-up in the liquid film. Other investigators found that the dynamic hold-up on random packing varied as a power of the liquid rate between 0.54 and 0.74 (10, 11);

this difference between this power and the theoretical value of 0.33 can be attributed to changes in the wetted area of the packing.

The objective of this study is the removal of phenol by adsorption using granular activated carbon. Although, activated carbon is a preferred adsorbent, its widespread use is restricted due to its high cost. To prove that the system can made more efficient and /or economical by increasing the adsorption surface area of the adsorbent through the reduction of dead zones between the particles in a fixed bed system by adding a non adsorbent materials, such as glass beads. To achieve this objective an experiment was designed and performed using activated carbon as adsorbent and phenol as adsorbate and to compare the experimental results with that simulated by the numerical solution of the general rate model which includes axial dispersion, film mass transfer, pore diffusion resistance and nonlinear isotherm.

MATHEMATICAL MODEL

Fixed bed

Fixed bed dynamics are described by the convection-diffusion equations, coupled with source term due to adsorption and diffusion inside adsorbent particles. The solution of these equations will give rise to the prediction of the needed breakthrough curves (12).

A general rate model refers to models containing a rate expression, or rate equation, which describes the interfacial mass transfer between the mobile phase and the stationary phase. A general rate model usually consists of two sets of differential equations, one for the bulk phase, and the other for the particle phase (13).

The rate model considers the following; axial dispersion, external mass transfer, interparticle diffusion and nonlinear isotherm (14, 15).

For modeling of fixed bed adsorption the column is divided into bulk-fluid phase and particle phase. The model equations are based on the hypothesis of an interparticle mass transfer controlled by diffusion into macropores (pore diffusion model), and this approach considers three phases; they are: the mobile phase in the space between particles, the stagnant film of the mobile phase immobilized in the macropores and the stationary phase where adsorption occurs (16, 17).

The following basic assumptions are made in order to formulate the model (12):

-The adsorption process is isothermal.
-The packing material is porous, spherical, particles of uniform size.
-The concentration gradient in the radial direction of the bed is negligible.
-Local equilibrium exists for the component between the pore surface and the stagnant fluid phase in the pores.
-The film mass transfer mechanism can be used to describe the interfacial mass transfer between the bulk-fluid and particle phase.
-The dispersion coefficient is constant.

-Surface diffusion can be ignored.

Continuity equation of the mobile phase is:

$$-D_b \frac{\partial^2 C_b}{\partial Z^2} + n \frac{\partial C_b}{\partial Z} + \frac{\partial C_b}{\partial t} + r_p \frac{1-e_b}{e_b} \frac{\partial q}{\partial t} = 0 \qquad \text{....1}$$

Using C_p, the concentration in the stagnant mobile phase and writing the expression of the interfacial flux leads to (13):

$$r_p \frac{\partial q}{\partial t} = \frac{3k_f}{R_p}\left(C_b - C_{p,R=R_p}\right) \qquad \text{....2}$$

Substituting equation (2) in equation (1) gives:

$$-D_b \frac{\partial^2 C_b}{\partial Z^2} + n \frac{\partial C_b}{\partial Z} + \frac{\partial C_b}{\partial t} + \frac{3k_f (1-e_b)}{e_b R_p}\left[C_b - C_{p,R=R_p}\right] = 0 \qquad \text{....3}$$

The particle phase continuity equation in spherical coordinates is:

$$(1-e_p)\frac{\partial C_p^*}{\partial t} + e_p \frac{\partial C_p}{\partial t} - e_p D_p \left[\frac{1}{R^2}\frac{\partial}{\partial R}\left(R^2 \frac{\partial C_p}{\partial R}\right)\right] = 0 \qquad \text{....4}$$

Subjected to the initial and boundary conditions stated in reference (18).

Defining the following dimensionless formula (13):

$$c_b = \frac{C_b}{C_o},\ c_p = \frac{C_p}{C_o},\ c_p^* = \frac{C_p^*}{C_o},\ t = \frac{nt}{L},\ r = \frac{R}{R_p},\ z = \frac{Z}{L}$$

$$Pe_L = \frac{nL}{D_b},\ Bi = \frac{k_f R_p}{e_p D_p},\ h = \frac{e_p D_p L}{R_p^2 n},\ z = \frac{3Bih(1-e_b)}{e_b}$$

The model equations can be transformed into the following dimensionless equations:

$$-\frac{1}{Pe_L}\frac{\partial^2 c_b}{\partial z^2} + \frac{\partial c_b}{\partial z} + \frac{\partial c_b}{\partial t} + z\left(c_b - c_{p,r=1}\right) = 0 \qquad \text{....5}$$

$$\frac{\partial}{\partial t}\left[(1-e_p)c_p^* + e_p c_p\right] - h\left[\frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2 \frac{\partial c_p}{\partial r}\right)\right] = 0 \qquad \text{....6}$$

The concentration c_p* in equation (6) is the dimensionless concentration in the solid phase of the particles. It is directly linked to the isotherm, which is the extended Langmuir model:

$$C_p^* = \frac{q_m b r_p C_p}{1 + bC_p} \qquad \text{….7}$$

Subjected to the initial and boundary conditions stated in reference (18).

The concentration c_p* in dimensionless form:

$$c_p^* = \frac{r_p q_m b c_p}{1 + bC_o c_p} \qquad \text{….8}$$

A finite element method is used for the discretization of the bulk-fluid phase partial differential equation and the orthogonal collocation method for the particle phase equation, an ordinary differential equation system is produced. The ordinary differential equation system with initial values can be readily solved using an ordinary differential equation solver such as the subroutine “ODE15S” of MATLAP which is a variable order solver based on the numerical differentiation formulas (NDFs). Optionally it uses the backward differentiation formulas (BDFs), which is also known as Gear’s method.

Batch adsorber

The batch model is described by the following equation:

-Mass balance in the bulk-fluid phase

$$V_L \frac{dC_b}{dt} + \frac{3W_A}{r_p R_p} k_f \left(C_b - C_{p,r=R_p}\right) = 0 \qquad \text{….9}$$

Where V_L = volume of fluid in the batch adsorber.

W_A = mass of activated carbon in the batch adsorber.

$C_b = C_t$

-Mass balance inside the particle

The solute diffusion inside a spherical particle is described by the following equation:

$$e_p \frac{\partial C_p}{\partial t} + r_p \frac{dq}{dt} = e_p D_p \frac{1}{r^2} \frac{\partial}{\partial r}\left(r^2 \frac{\partial C_p}{\partial r}\right) \qquad \text{….10}$$

The solute concentration in the pores is in local equilibrium with the concentration of solute adsorbed on the pore walls.

Subjected to the Initial and boundary conditions stated in reference (18).

For Langmuir isotherm model:

$$q = \frac{q_m b C_p}{1 + b C_p} \qquad \text{....11}$$

The external mass transfer coefficient for the solute adsorbed at certain particle size and optimum speed, can be obtained by the analytical solution of equation (9).

$$k_f = -\frac{R_p\, r_p V_L}{3 W_A t} \ln\left(\frac{C_t}{C_o}\right) \qquad \text{....12}$$

Where R_p and ρ_p are the particle radius and density respectively and C_o and C_t are the reactor concentration at time zero and time t, respectively (19, 20).

Experimental concentration-time data are compared to predicted concentration-time profile for the above batch absorber model and the best statistical fit was used to determine the interparticle pore diffusion coefficient.

Experimental work

The granular activated carbon (GAC) used in the experiments was supplied by Netherland Company.

The GAC was sieved into 30/35 mesh with geometric mean diameter of 0.55mm. The GAC was boiled, washed three times in distilled water and dried at 100°C for 24 hours, before being used as adsorbent. The aqueous solution of phenol was prepared using reagent grade. The glass beads were supplied from the local market. They were sieved into 30/35 mesh with geometric mean diameter of 0.55mm. The glass beads were washed and dried at 100°C for 24 hours before being mixed with the adsorbent.

The experiments were adjusted at an initial pH of 5 for phenol (21). The pH value was adjusted with 0.01 mol/l HCl.

The fixed bed experiments were carried out in Q.V.F. glass column of 50 mm (I.D) and 50 cm in height. The GAC was confined in the column by fine stainless steel screen at the bottom and glass packing at the top of the bed to ensure a uniform distribution of the influent through the carbon bed. The influent solution was introduced to the column through a perforated plate fixed at the top of the column. The feed solution was prepared in Q.V.F. vessel supplied with immersed heater and a thermocouple to adjust the temperature of the solution at 30°C through temperature controller as shown in figure (1).

For the determination of adsorption isotherms, 250 ml flasks were filled with known concentration of solute and a known weight of GAC. The flasks were then placed on a shaker and agitated continuously for 30 hours at 30°C. The concentration of phenol in the solution was determined by a UV-160A spectrophotometer at 270nm.

The adsorbed amount was calculated by the following equation:

$$q_e = \frac{V_L\left(C_o - C_e\right)}{W_A} \qquad \text{….13}$$

The determination of interparticle diffusion coefficient needs the following steps:

- Estimating the optimum agitating speed for batch adsorber to reach the needed equilibrium concentration of phenol.
- Estimating the mass transfer coefficient in batch process at optimum agitation speed.
- Numerical solution of batch adsorber model to obtain the interparticle diffusion coefficient. This was done by using the pore diffusion model to best fit the experimental results.

The interparticle diffusion coefficient for phenol was obtained by 2 liter Pyrex beaker fitted with a variable speed mixer. The beaker was filled with 1 liter of known concentration solution and agitation started before adding the GAC. At time zero, the accurate weight of GAC was added. Samples were taken every 5 minutes.

The necessary dosage of GAC, to reach equilibrium related concentration of C_e/C_o equal 0.05, were calculated from isotherms model and mass balance equation as follows(18):

$$W_A = \frac{V\left(C_o - C_e\right)}{q_e} \qquad \text{….14}$$

Results and discussions

Adsorption isotherm

The adsorption isotherms display a nonlinear dependence on the equilibrium concentration. The adsorption data for the system fitted by Langmuir model (22), Freundlich model (23), Radke-Prausnitz model (24), Reddlich-Peterson (25) and Combination of Langmuir-Freundlich isotherm model (26). The correlation coefficient was employed to ascertain the fit of each isotherm with experimental data. From table (1), the correlation coefficient value was higher for Langmuir than other correlations. The Langmuir adsorption model was selected to be introduced in the fixed bed model where:

$$q_e = \frac{q_m b C_e}{1 + b C_e} \qquad \text{….15}$$

The equilibrium isotherms for phenol onto GAC is presented in figure (2). The parameters q_m, b are the equilibrium isotherm coefficients and they are evaluated to be:

$q_{m,\,phenol}$= 0.142 kg/kg b_{phenol}=159.4 m^3/kg

Interparticle diffusion coefficient

The amount of activated carbon used for phenol was calculated for final equilibrium related concentration of C_e/C_o=0.05. Using the Langmuir equation with mass balance in one liter of solution. The initial concentrations were 0.2 Kg/m^3 with the doses of activated carbon of 2.2×10^{-3}Kg.

The agitation speeds (600, 700, 800, 900, 1000 and 1100 rev/min) were used as shown in figure (3). The optimum agitation speed needed to achieve C_e/C_o=0.05 was found to be 1000 rpm. It is clear that, if the speed is above 1000 rpm, the equilibrium relative concentration is less than 0.05; this is due to attrition of activated carbon at high speed agitation.

There were a good matching between batch experimental results and predicted data using pore diffusion model for batch operation as shown in figure (4).

The pore diffusion coefficient for the system is evaluated from batch experiments and found to be $D_{p,phenol}$ =5.25×10^{-8} m^2/sec.

The external mass transfer coefficients in packed bed model for the system was evaluated by the correlation of Wilson and Geankoplis (21).

$$Sh = \frac{1.09}{e_b} Sc^{1/3} \mathrm{Re}^{1/3} \qquad \text{For } Re=0.0015\text{-}55 \qquad \text{....16}$$

Where: $Sh = \frac{K_f d_p}{D_m}$, $Sc = \frac{m_w}{r_w D_m}$ and $\mathrm{Re} = \frac{r_w u d_p}{m_w}$.

In which the molecular diffusion coefficient D_m of phenol in aqueous solution is 9.6×10^{-9} m^2/sec. These values are substituted in equation (16) to evaluate K_f at different interstitial velocity in the mathematical model.

The axial dispersion coefficient was calculated from Chung and Wen equation (27):

$$\frac{D_b r_w}{m_w} = \frac{\mathrm{Re}}{0.2 + 0.011\mathrm{Re}^{0.48}} \qquad \text{....17}$$

Breakthrough curves

Figures (5) to (9) show the experimental and predicted breakthrough curves for phenol system onto activated carbon at different adsorbent-glass beads weight ratios, flow rate, bed depth and initial concentration of adsorbate at constant temperature of 30^{o}C. It is clear from these figures that:

1. Adding 5% glass beads weight ratio to the activated carbon bed reduces its weight by 5% and increases the operating time by 80%. Increasing the glass beads ratios from 10 wt% and above make the adsorption process inefficient compared with 0 wt%. This may be attributed to the nature of activated carbon particles, where it is entirely different from the glass beads regarding the roughness of the surface and the porosity of the material. The glass beads have more or less smooth surface, while the activated carbon particles have nearly rough surface.

The liquid hold-up in a packed column is important for many reasons. It reduces the porosity of the packed bed (28). From the above and from the principle of liquid hold-up, it can be concluded that:

Static hold-up for activated carbon-activated carbon particles > activated carbon -glass beads > glass-glass beads,

This leads to the losses in surface area for adsorption as follows:-

Area losses in adsorbent-adsorbent particles > adsorbent-glass beads,

The dynamic liquid hold-up will have the same behavior as mentioned above; this is related to the difference in the nature of activated carbon and the glass beads. Due to the roughness of the activated carbon surface, the operating liquid film thickness will be higher for the adsorbent compared with glass beads for a given particle diameter, flow rate, pollutant concentration and temperature. Therefore the phenomena will lead to high resident time for the liquid on the adsorbents particles compared with glass particles, and consequently increases the breakthrough time for the adsorption process. Table (2) shows that the bed porosity increases for different adsorbents-glass beads weight ratios.

It is very important to find the optimum weight ratio of glass particles to adsorbent particles, knowing that the matching between the two mechanisms; static hold-up and operating hold-up will save weight of both adsorbents and minimize the losses in surface area for adsorption.

2. An increase in the initial concentration of phenol makes the breakthrough curves much steeper, which would be anticipated with the basis of increases driving force for mass transfer with the increase of concentration of adsorbate in the solution (29).

3. An increase in the bed depth of activated carbon will increases the breakthrough time and the residence time of the adsorbate in the column. An increase in the bed depth will increase Pe number with constant Bi number, where Pe number is the ratio of axial convection rate to axial dispersion rate. When Pe number is small (the effect of axial dispersion is not negligible) the breakpoint appears early and the breakpoint increases with increasing Pe number as shown in table (3).

4. An increase in the adsorbate flow rate decreases the breakthrough time due to the decrease in the contact time between the adsorbate and the adsorbent along the adsorption bed. Increasing the flow rate may be expected to make reduction of the surface film. Therefore, this will decrease the resistance to mass transfer and increase the mass transfer rate. Also, because the reduction in

the surface film is due to the disturbance created when the flow of the feed increased resulting of easy passage of the adsorbate molecules through the particles and entering easily to the pores. This will decrease contact time between the adsorbate and the adsorbent. An increase in the flow rate will increase Bi number and with slight increase Pe number. Bi number is the ratio of external mass transfer rate to the interparticle mass transfer rate (that is, the interparticle mass transfer is the control step) the breakpoint will appear early as shown in table (4).

5. The breakpoint was related to the flow rate, bed depth and initial concentration, i.e. the time required to reach breakpoint decreases with the increase of flow rate, decrease of bed depth, and increase of solute initial concentration.

CONCLUSIONS

The adsorption process can be made more efficient and/or economical by increasing the adsorption surface area of the adsorbent via adding an inert material (glass beads) to activated carbon bed in different weight ratios.

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NOTATION

SYMBOLS

A	Bed area, m^2
b	Langmuir constant, m3/kg
Bi	Biot number $\left(\dfrac{k_f R_p}{e_p D_p}\right)$
C	Concentration in fluid, kg/m3
C_o	Initial concentration, kg/m3
C*	Micropore stationary phase concentration , kg/m3
D_b	Axial dispersion coefficient, m2/s
D_m	Molecular diffusion coefficient, m2/s
D_p	Pore diffusion coefficient, m2/s
d_p	Particle diameter, m

k_f	Fluid to particle mass transfer coefficient, m/s
L	Length of column, m
Pe	Peclet number $\left(\frac{vL}{D_b}\right)$
Q	Fluid flow rate, m3/s
Q	The amount of adsorbate per unit weight of carbon (Kg/Kg)
q_e	Internal concentration of solute in particle at equilibrium, kg/kg
q_m	Adsorption equilibrium constant defined by Langmuir equation, kg/kg
Re	Reynolds number $\left(\frac{r_w v d_p}{m_w}\right)$
R_p	Radius of particle, m
Sc	Schmidt number $\left(\frac{m_w}{r_w D_m}\right)$
Sh	Sherwood number $\left(\frac{k_f d_p}{D_m}\right)$
T	Time, s
V_L	Volume of solution, m3
W_A	Mass of activated carbon, kg
Z	Axial distance, m

Greek symbols

ε_b	Bed porosity
ε_p	Porosity of adsorbent
V	Interstitial velocity $\left(\frac{Q}{Ae_b}\right)$, m/s
v_s	Superficial velocity $\left(\frac{Q}{A}\right)$, m/s
μ_w	Viscosity of water
ρ_w	Density of water, kg/m3

Subscript

b	Bulk fluid phase
e	Equilibrium
l	Liquid phase
o	Initial
p	Particle phase

Greek symbols

Table 1: Parameters of isotherm for phenol and correlation coefficient for various models

Model	Parameters	Phenol
Langmuir	q_m, b, m^3/kg Correlation coefficient	0.142 159.4 0.9992
Freundlich	K, n, Correlation coefficient	0.1915 6.5697 0.9986
Radk-Prausnitz	K_{RP}, F_{RP}, - N_{RP}, - Correlation coefficient	3.0035 0.1420 0.8673 0.9936
Reddlich-Peterson	A_{R}, B_R, m_{R}, Correlation coefficient	22116.42 966905.8 0.7904 0.9879
Combination of Langmuir-Freundlich	q_m, b, n, Correlation coefficient	0.3934 0.8658 0.2193 0.9979

Table 2: Bed porosity for different activated carbon-glass beads ratios

Glass beads ratios (%)	ε_b

weight	
0	0.4
5	0.43
10	0.47
15	0.5
20	0.54

Table 3: Values of Pe and Bi numbers for different flow rates

Flow rate (m3/sec)	Pe	Bi
1.39×10-6	19.3	147.6
2.78×10-6	19.7	185.9
4.17×10-6	20.0	212.8
8.33×10-6	20.7	268.0

Table 4: Values of Pe and Bi numbers for different bed depth

Bed depth (m)	Pe	Bi
0.05	19.7	185.9
0.08	31.5	185.9
0.1	39.4	185.9
0.15	59.4	185.9

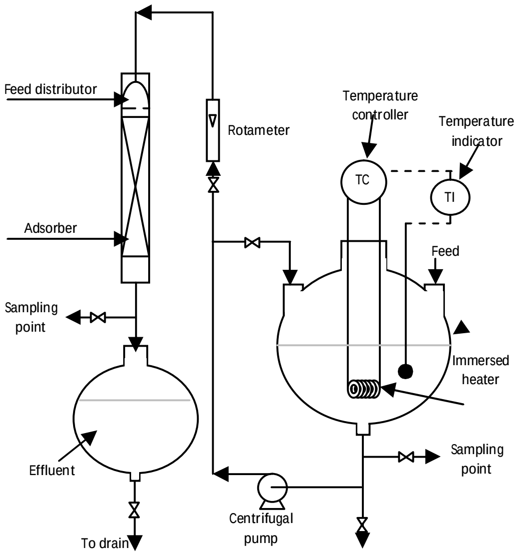

Fig 1: Schematic representation of experimental equipment

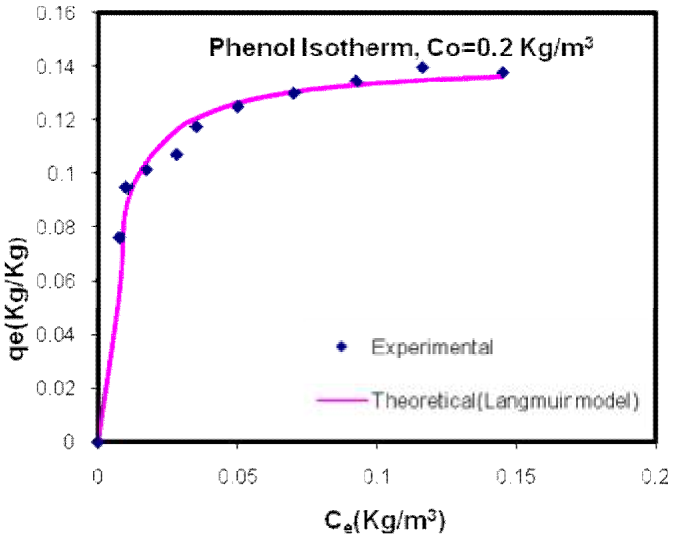

Fig2: Adsorption isotherm for phenol onto activated carbon at 303K

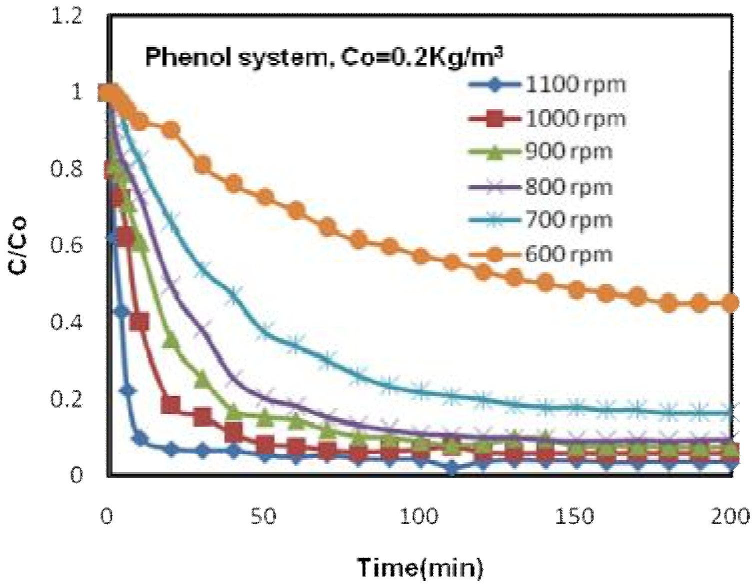

Fig3: Concentration-time decay curves for phenol adsorption onto activated carbon at different agitation speed

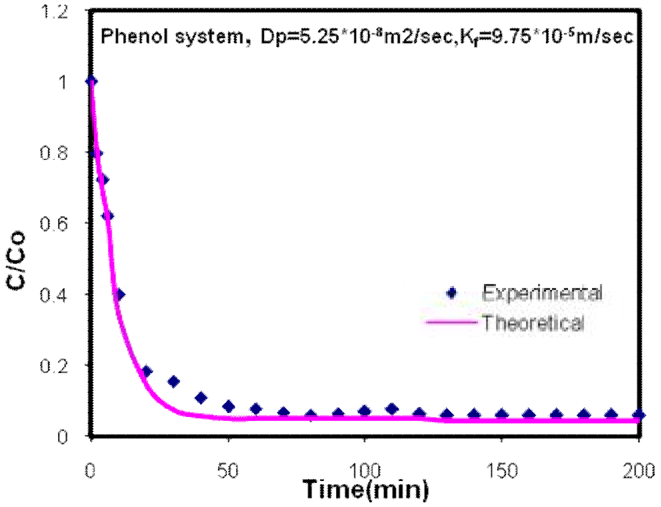

Fig 4: Comparison of the measured concentration-time data with that predicted by pore diffusion model in batch adsorber for phenol system

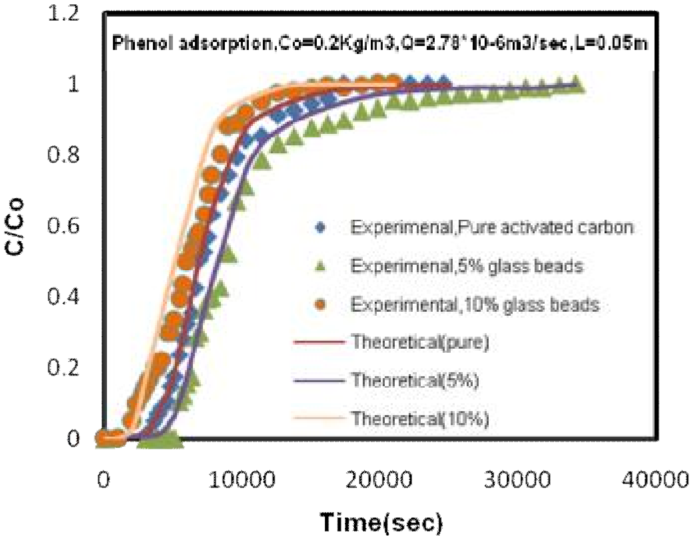

Fig5: Experimental and predicted breakthrough curves for phenol adsorption onto activated carbon at different glass beads weight ratios

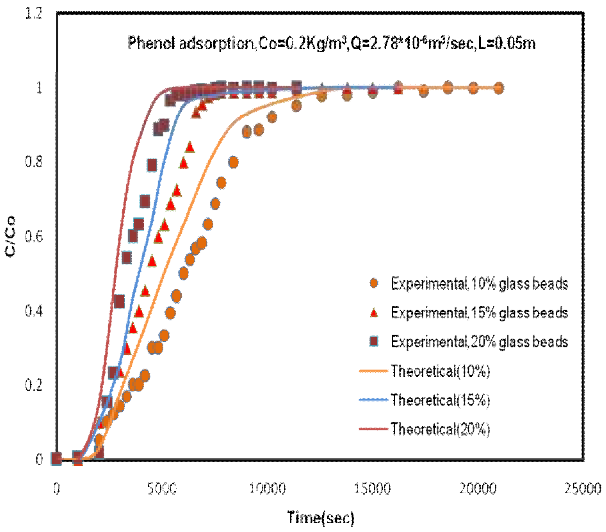

Fig 6: Experimental and predicted breakthrough curves for phenol adsorption onto activated carbon at different glass beads weight ratios

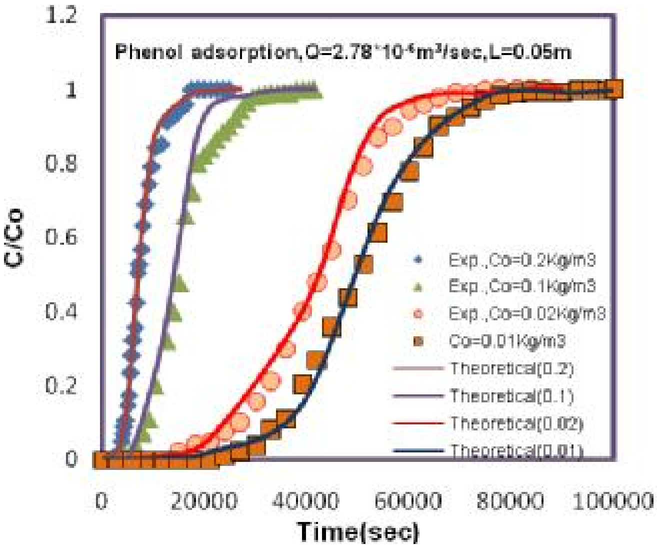

Fig 7: Experimental and predicted breakthrough curves for phenol adsorption onto activated carbon at different initial concentration

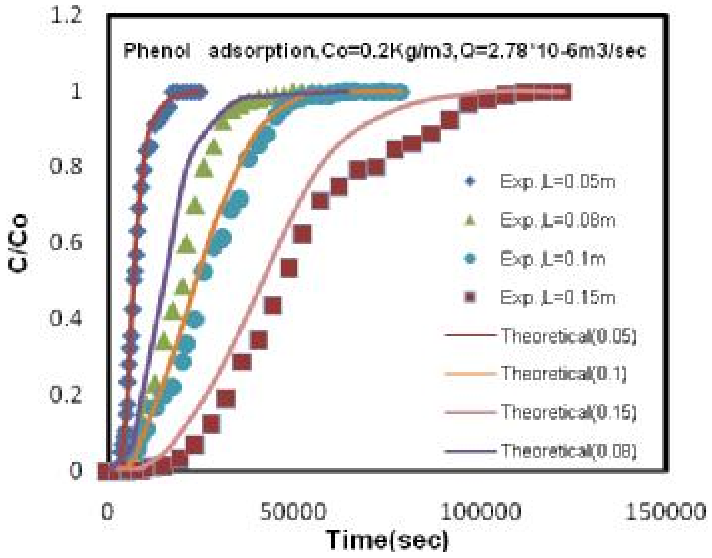

Fig 8: Experimental and predicted breakthrough curves for phenol adsorption onto activated carbon at different bed depths

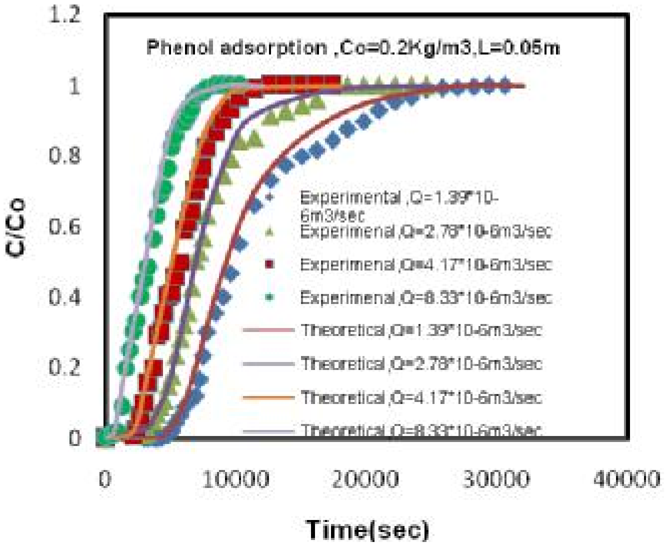

Fig9: Experimental and predicted breakthrough curves for phenol adsorption onto activated carbon at different flow rates