Fixed bed column adsorption of phenol onto locally available soil: linear kinetics modeling

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Abstract

In the present study analysis of fixed column adsorption studies of phenol onto the soil was investigated. The soil minerals were characterized by of X-ray diffraction peaks at diffraction angles of 21.208, 26.985 and 50.509 and adsorption studies were conducted at a pH of 6.00. The effects of process variables on the bed performance were studied, these included flow rate and bed depth. The results showed that a decrease in flow rate at a constant bed depth increased the breakthrough volume ($V_b$) and therefore the breakthrough time ($T_b$), due to an increase in empty bed contact time (EBCT). At the flow rate of 10 mL/min and at varying depths 5, 10 and 15 cm the EBCT was found 2.45, 4.91 and 7.36 min. Similarly for a flow rate of 20 and 30 mL/min, at varying depths 5, 10 and 15 cm, the EBCT was found 1.23, 2.45 and 3.68 min and 0.82, 1.64 and 2.45 min, respectively. For these test columns, the optimum flow rate should not be higher than 10 mL/min to provide a shorter adsorption zone and a longer service time. The adsorption capacity ($N_o$) of soil was calculated to be 713.37, 978.34 and 978.34 mg/L by using the flow rates of 10, 20 and 30 mL/min. The results of the examination of the dynamic adsorption system were also demonstrated to be significantly influenced by the modelling methodology.

Keywords: Fixed column; Empty bed contact time (EBCT); Bed depth service time (BDST); Breakthrough curves; Adsorption; Flow rate

1. Introduction

Growing wastewater has been a result of the global industrial production sector’s quick development. Water quality is decreased when wastewater is discharged, making it impossible to use water without first desalinating it for drinking purposes and other industrial uses [1–3]. Even at low concentrations, wastewater including resins, dyes, pesticides, phenol, and phenolic compounds may endanger the lives of people as well as aquatic life [4]. The production of pharmaceuticals, personal care products, nylon, resins, pulp and paper mills, coal gasification operating units, and petroleum refineries and industries so on so forth use phenols. Due to its carcinogenic characteristics and extreme difficulty in biological degradation, it is categorised as extremely hazardous [5]. Fish have toxic phenol levels between 9 and 25 mg/L, while human toxic phenol levels are between 10 and 24 mg/L. Around 150 mg/100 mL is the fatal quantity in human blood. But there are numerous methods for recovering and getting rid of phenol. The most popular techniques include steam stripping, ion exchange, adsorption, hot gases, and solvent extraction. These techniques have been discovered to be effective for removing phenol [6–10]. These methods remove phenol directly from water and wastewater. Whereas the biological treatment process also removes phenol, phenolic wastewater is passed through activated carbon in moving, fixed, or fluidized beds.

To evaluate the feasibility and economics of adsorbent, a laboratory study is usually conducted. The batch study gives the removal capacity of specific waste contaminants,
but the continuous flow provides practical application of the process. For industrial application fixed bed adsorption column study is an important step [11,12]. The breakthrough curves must be predictable under predetermined operating circumstances for a packed bed adsorption process to be properly designed and operated [13]. It is required to stop the adsorption period in a process before the saturation, a thorough understanding of adsorption characteristics is necessary [14]. At industrial scale, the effects of design parameters, such as bed height and flow rate, have been investigated. The column study for adsorption starts with laboratory testing to establish the breakthrough curve. The amount of adsorbate adsorbed within a bed depends both on space and time. As fluid enters the bed, it meets the first few layers of absorbent. Solute adsorbs, filling up some of the available sites [15]. As the top layers of adsorbent become saturated with adsorbate, the mass transfer zone (MTZ) will move down in the bed until breakthrough occurs. The length of the MTZ is typically a function of the hydraulic loading rate applied to the column and the characteristics of the adsorbent.

There are many different types of techniques by which the main point of contact between adsorbate and adsorbent occurs in the adsorption system, including batch, continuous moving bed, continuous fixed bed (up flow or downflow), continuous fluidize bed, and pulsed bed. A representation of the pollutant-effluent concentration vs. time profile in a fixed-bed column, through which breakthrough curves, are used to analyse the performance of fixed-bed columns. The mechanisms underlying this adsorption include axial dispersion, film diffusion resistance, intraparticle diffusion resistance (both pore and surface diffusion), and sorption equilibrium with the sorbent [11,12,16]. Thomas, bed depth service time (BDST), Yoon–Nelson, and Clark are some of the mathematical models that have been used to forecast the breakthrough curve and model parameters. To create the procedure for the columns, the characteristics of parameters connected to the models were obtained by linear and nonlinear regression. All the models are equivalent in how they describe the adsorption fixed-bed columns based on error analysis and adsorption conditions [17]. Using Ni/MgO metal oxide for microwave exposure and thermal disintegration at 550°C, Ali [18] had developed an affordable adsorbent, called carbon nanotube. Using process variables like initial concentration, flow rate, and bed height, nanotube underwent column experiments for the removal of arsenite and arsenate. Thomas and Adams–Bohart models were used to examine the data, and it was discovered that the maximum removals for arsenite and arsenate, respectively, were 13.5 and 14.0 mg/g. The adsorption of cesium was studied using synthetic adsorbents, specifically polyacrylonitrile-potassium cobalt hexacyanoferrates and polyacrylonitrile-potassium nickel hexacyanoferrates. On the adsorption of cesium, the effects of liquid flow rate, bed height, and the presence of other cations were examined. The experimental data were analysed using the BDST model and the Thomas model, and the model's parameters were assessed [16,19]. Adams–Bohart, Thomas, and BDST were used by López-Cervantes et al. [20] in their investigation of fixed bed isotherm mathematical modelling. The BDST model demonstrated strong correlation coefficient values and good agreement with the experimental data. The maximum capacity for dye removal capacity was found to be 343.59 mg/g. The objective of the current study is to explore the phenol removal capability of locally available soils using a fixed-bed adsorption column study. Investigation is done into how the performance of the column is affected by bed height, concentration, and flow rate. The models Adams–Bohart, Thomas, and BDST are used to study the breakthrough curves.

2. Methodology

2.1. Preparation of adsorbents

The soil used in the study was from the Kalathur soil series at Thanjavur, Tamil Nadu, and India. Kalathur is situated in Thanjavur district, Tamil Nadu, with latitude = 10.34 and longitude = 79.20. The Kalathur soil series consists of very dark grey-brown, very deep, calcareous, fine textured Cauvery River alluvium. The soil is classified as vertisols. Vertisols are weathered soils and are poorly drained, with relatively low native fertility. Vertisols consist mainly of quartz, kaolinite, oxides, and organic matter. They appear structure less and have the feel of a loamy texture. While some are loamy or even coarser textured, many have a fine or very-fine particle-size class, but the clay is aggregated in a strong grade of fine and very fine granular structure [21].

2.2. Selection and characterization of the adsorbent

2.2.1. Soil sample

After being crushed (for 24 h in a ball mill) and sieved (with a 100–635 (0.15–0.02 mm) SIEVE NO. ASTM E11-87) to get particles with an average diameter of 0.05 mm, the soil sample was dried overnight in an electric oven at 105°C. The sample was then retrieved and conserved for later analysis by being dried, desiccated, and kept in airtight containers. To determine the soils’ fundamental features, physical and chemical investigations were performed (IS: 1527 (1969)) [22]. The samples were treated with increments of 5 mL of 30% H₂O₂ in a water bath at 80°C to eliminate organic materials. The absence of effervescence suggested that all organic debris had been completely removed.

2.2.2. X-ray diffraction analysis for the soil sample

X-ray diffraction is an effective tool for qualitative mineralogical analysis. The Kr soil minerals were characterized by of X-ray diffraction peaks at diffraction angles of 21.208, 26.985 and 50.509. The major mineral components found in the two soils are quartz (Q), iron-silicon sulfide (ISS), aluminium oxide (AO), iron-silicon (IS), geothite (G), and iron oxide hydroxide (IOH). Iron oxides (like hematite and goethite) were found in the soil fractions too. The test was performed on Bruker D8 Focus under an alternating voltage of 40 kV and a current of 40 mA. Samples were subjected to CuKα radiation. The 2θ step size for the analysis ranged from 20° to 80°. The Fourier-transform infrared spectroscopy (FTIR) spectrum soil was analyzed and the percentage transmission for various wave numbers, absorption bands
identified in the spectra and their assignment to the corresponding functional group (the author published X-ray fluorescence, FTIR and scanning electron microscopy data published elsewhere).

2.2.3. Preparation of the adsorbate

The analytical reagent grade phenol (C₆H₅OH) was used to make synthetic adsorbate solutions with a range of initial concentrations (Cᵢ) between 10 and 100 mg/L. To prepare one liter of phenol for future research, the required amount of phenol was accurately weighed and combined phase-wise with distilled water. Daily preparation and storage of the new phenol solution in a 5-L dark-colored glass container prevented photo-oxidation. Each experimental research began with a determination of the initial concentration (Cᵢ).

2.3. Column adsorption studies

A soil adsorbent was contained in a glass column with an internal diameter of 2.50 cm and a length of 30 cm as a fixed bed adsorber. Glass wool and glass beads were used to support and seal the bed, ensuring even liquid distribution. To ensure that the particles were tightly packed and free of cavities, channels, or fissures, the bed was rinsed with distilled water and left to dry overnight. To prevent channelling of the effluent, the phenol solution was fed through a bed of soil packed adsorbent in an up-flow mode. At the entrance and outflow, a peristaltic pump was employed to regulate the flow rate. Periodically, effluent samples were taken, and their remaining phenol concentrations were measured (Cₑ). Therefore, 10% of the input feed concentration (100 mg/L), or 0.1 Cᵢ/Cₑ, or 10 mg/L, was chosen as the target breakthrough concentration (Cᵢ). The flow through the tested column was kept going until the exhaustion point, which was indicated by the column effluent’s phenol concentration approaching 0.9 Cᵢ/Cₑ (Cₑ). The breakthrough curve is the relationship between Cᵢ/Cₑ and volume treated between Cᵢ and Cₑ. Adsorption capacity calculations are made based on plots of the concentration vs. time. Curves are integrated analytically to obtain capacities measured in time units. The time required for a bed to become totally saturated is obtained by integrating as time goes to infinity:

\[ t = \int_{0}^{t} \left(1 - \frac{C}{C_{i}}\right) dt \]  

(3)

where \( t \), time for total capacity, in operation, the process is to be stopped the process before solute gets breakthrough, the integration to the breakpoint time gives the “usable” capacity:

\[ t_{Usable} = \int_{0}^{t_{break}} \left(1 - \frac{C}{C_{i}}\right) dt \]  

(4)

2.3.1. BDST model

The effects of process variables on the bed performance were studied, these included flow rate and bed depth. The three different flow rates: 10, 20 and 30 mL/min, were compared at a constant bed depth. The studied bed depths were 5, 10, and 15 cm. After the volume of breakthrough (Vᵢ) and time of breakthrough (Tᵢ) were determined, the BDST curves were plotted between Tᵢ vs. bed depth (D).

According to Adams–Bohart model, the relationship between Tᵢ and D is described as in the following equation [23].

\[ T_{i} = \frac{N_{D} D}{C_{e} V} \cdot \frac{1}{K C_{e}} \cdot \ln \left[ \frac{C_{i}}{C_{e}} - 1 \right] \]  

(5)

where \( T_{i} \) is the empty bed contact time (EBCT) in minutes, \( N_{D} \) is the number of transfer units, \( D \) is the bed depth in m, \( C_{e} \) is the effluent concentration (mg/L), \( C_{i} \) is the inlet concentration (mg/L), and \( V \) is the flow rate in L/min. The numerical value of the slope \( a = N_{D} / V C_{e} \) and the intercept \( b = [-1/KC_{i}] \cdot \ln[C_{i}/C_{e} - 1] \).

The adsorptive capacity of the system, \( N_{D} \), and the rate constant, \( K \), can be evaluated from the slope and intercept of a straight line, respectively. The minimum bed depth \( D_{min} \) can be calculated from the relationship of Eq. (5) by letting \( T_{i} = 0 \) and solving for \( D \) [24].

2.3.2. Empty bed contact time

Empty bed contact time (EBCT) is the time a fluid spends in the column, on the basis that the column is empty (Cooney, 1999). EBCT can be defined as follows:
2.3.3. Adams–Bohart column model

Adams–Bohart established the fundamental relationship between $C/C_0$ and $t$ for the adsorption of chlorine on charcoal in 1920. However, this model was equally good for other systems also and therefore adopted by several researchers. Adams–Bohart A model was used to describe the initial part of the breakthrough curve.

$$\ln \left( \frac{C_i}{C_0} \right) = K_{AB} C_0 - \frac{K_{AB} N_0 Z}{F}$$

(7)

where $C_i$ — initial concentration of phenol, $C_r$ — concentration of effluent at time, $F$ — linear velocity calculated by dividing the flow rate by the column section area (cm/min), $K_{AB}$ — Adams–Bohart rate constant (L/mg·min), $N_0$ — adsorption capacity of the adsorbent (mg/L) and $Z$ — bed depth of the column (cm). A plot is plotted between $\ln(C_i/C_0)$ vs. $t$.

3. Results and discussion

3.1. Design of a column studies

In the present study analysis of fixed column adsorption studies of phenol onto the soils were investigated. Adams–Bohart, BDST, and EBCT are a few of the mathematical models used to forecast the breakthrough curve and model parameters. To create the procedure for the columns, linear regression was used to acquire the properties of the model-related parameters. The results showed that a decrease in flow rate at a constant bed depth increased the breakthrough volume ($V_b$) and therefore the breakthrough time ($T_b$), due to an increase in EBCT. Since the front of adsorption zone reached the top of the column later by using a slower flow rate, more $V_b$ was obtained. The results show that the breakthrough curve was obtained quickly with higher flow rates. The time required to reach saturation increased significantly with an increase in the bed depth. Further increase in bed depth from 10 to 15 cm and the effect of flow rates were studied. The time required to reach saturation increased significantly with a decrease in flow rate (Fig. 3).

The breakthrough curve of the lower flow rate (10 mL/min) (Fig. 4) shows steep rise near the exhaustion point, suggesting that the adsorption zone was shorter, and the adsorbent was more completely saturated with the phenol [26,27]. Fig. 5 represents the breakthrough curve at a flow rate of 10, 20 and 30 mL/min.
rate of 20 mL/min and are very steep compared to the breakthrough curves at a flow rate of 10 mL/min. This is because the flow rate is large and the contact time is very short. The zone of adsorption was very shallow. Fig. 6 also shows that the breakthrough curve of 30 mL/min became less sharp because the adsorption zone was deeper. This result suggests that a lower flow rate or a longer contact time may be required for phenol adsorption by a column of soil adsorbent and the results comply with the results of other researchers [28]. Table 1 shows the details of bed depth (i.e., 5, 10 and 15 cm) and flow rate (i.e., 10, 20 and 30 mL/min), the column studies were conducted by varying flow rate and varying the depth of the column bed. At the flow rate of 10 mL/min and at varying depths 5, 10 and 15 cm the EBCT was found 2.45, 4.91 and 7.36 min. Similarly for a flow rate of 20 and 30 mL/min, at varying depths 5, 10 and 15 cm, the EBCT was found 1.23, 2.45 and 3.68 min and 0.82, 1.64 and 2.45 min, respectively. For these test columns, the optimum flow rate should not be higher than 10 mL/min to provide a shorter adsorption zone and a longer service time. The researchers studied EBCTs at different flow rates for the adsorption of AB1 and AB113. The present findings agree with the results reported by the Samarghandi et al. [29].

### 3.2. Effect of bed depth on breakthrough curve

Figs. 4–6 show the breakthrough curves of different bed depths at constant flow rates of 10, 20 and 30 mL/min, respectively. $V_b$ or $T_b$ increased with increasing bed depth while the shape and gradient of the breakthrough curves were slightly different with the variable bed depths. However, the breakthrough curves of the longer beds tended to be more gradual. This may be because of the reason that the column was difficult to be completely exhausted. This effect was higher by using the higher flow rate of 30 mL/min (Fig. 6). This study indicated that the flow rate at the outlet was unstable when the bed depth was too high due to a higher flow resistance, which resulted from a tighter packing of the longer bed containing more amount of adsorbent. Although an increasing bed depth increased $V_b$ or $T_b$, too high bed depth is not useful for a single column; otherwise, the multiple beds need to be designed. Since the treated effluent was not properly drawn off at the top of the packed bed higher than 15 cm for the diameter

### Table 1

<table>
<thead>
<tr>
<th>$Q$ (mL/min)</th>
<th>Bed depth (cm)</th>
<th>Bed volume (cm$^3$)</th>
<th>$W_c$ (g)</th>
<th>EBCT (min)</th>
<th>$V_b$ (mL)</th>
<th>$T_b$ (min)</th>
<th>Usage (g/mL)</th>
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<td>24.3513</td>
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<td>2.45313</td>
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<td>15</td>
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of 2.5 cm, the optimum depth-to-diameter ratio of column containing agro-based adsorbent should be lower than or equal to 6 (15/2.5). Actually, this ratio can be applied for the scale up of the column adsorber.

### 3.3. BDST with variation in bed depth and flow rate

The data of EBCT, $V_i$ and $T_b$ increased with decreasing flow rate or increasing bed depth. However, the flow rate should not be too low, depending on the detention time and the volume of wastewater for treatment. As mentioned above, the bed depth should also not be too high; therefore, the multiple beds operated in series or parallel should be preferable. From the data in Tables 1 and 2, the BDST plots ($T_b$ vs. $D$) were constructed, as shown in Figs. 7 and 8. The equations of linear relationship were obtained with all $R^2$ values in the range 0.92–0.99, showing that the model has strong applicability. The values of $K_{ab}$ were found to decrease with increase in flow rate and while its value increased with increase in bed height (Table 3) indicating that the overall system kinetics was dominated by external mass transfer while its value increased with increase in bed height (Table 3). Of all the models, the best fittings with $R^2$ values in the range 0.92–0.99 indicating that the model has good applicability. The present findings agree with the results reported by Samarghandi et al. [29].

### 3.5. Dynamic column studies

A glass column of 30 cm length and 2.50 cm diameter was used to contain soil and soil-agro blend adsorbent as a fixed-bed adsorber. The experiments were carried out on different flow rates for different depths. The theoretical adsorption capacity of an adsorbent at any final concentration can be calculated by making use of any one best fitting model. As mentioned earlier Adams–Bohart model is used for the prediction of initial part of the breakthrough curves. The model parameters are given in Table 3. The table exhibits the effect of bed height and flow velocity. It could be seen from Figs. 9–11 that Adams–Bohart plot exhibits multi-linearity. Thus, it is possible to predict the breakthrough curve using Adams–Bohart model parameters evaluated at different service times.

The values of maximum adsorption capacity ($N_o$) and Adams–Bohart rate constant ($K_{ab}$) were determined from the intercept and slope of Adams–Bohart plot at different bed height and flow rates as shown in Table 3. The maximum adsorption capacity ($N_o$) value increased when the flow rate increased (Table 3), but it did the opposite as the bed height increased. It has the best fits out of all the models, with $R^2$ values in the 0.92–0.99 region, showing that the model has strong applicability. The values of $K_{ab}$ were found to decrease with increase in flow rate and while its value increased with increase in bed height (Table 3) indicating that the overall system kinetics was dominated by external mass transfer while its value increased with increase in bed height (Table 3). Of all the models, the best fittings with $R^2$ values in the range 0.92–0.99 indicating that the model has good applicability. The present findings agree with the results reported by Samarghandi et al. [29].

### Table 2

<table>
<thead>
<tr>
<th>$Q$ (mL/min)</th>
<th>$V$ (cm/min)</th>
<th>Slope</th>
<th>Intercept</th>
<th>$D_{min}$ (cm)</th>
<th>$N_o$ (mg/L)</th>
<th>$K$ (L/mg·min)</th>
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Fig. 7. Bed depth service time curves for 10% breakthrough.

Fig. 8. Bed depth service time curves for 50% breakthrough.
isotherm model. The value of the adsorptive capacity thus obtained represents the amount of adsorbate adsorbed per unit weight of the adsorbent when effluent the effluent concentration equals to the for estimation by making use of the breakthrough curve. Breakthrough being defined as the point when a specified amount of the influent is detected in the effluent [33]. A breakthrough of 10% occurs when the concentration of the effluent is 10% of the influent concentration [34–36]. Influent phenol concentration of about 100 mg/L was used for all the attempted influent concentration and 90% breakthrough occurs when the concentration of the effluent is 90% of the influent concentration. The area between the ordinate and the breakthrough curve gives the concentration of the adsorbate removed over the test time. The amount of adsorbate removed per gram of adsorbent is then obtained by dividing the total amount of adsorbate removed by the mass of the adsorbent in the column. A continuous flow study was performed to compare the adsorption capacity obtained from a continuous column experiment to those determined from batch studies.

3.6. X-ray diffraction analysis for the soil sample

X-ray diffraction is an effective tool for qualitative mineralogical analysis. The soil minerals were characterized by X-ray diffraction peaks at diffraction angles of 21.208, 26.985 and 50.509. Hematite, a common iron oxide with the chemical formula Fe2O3, has a distinctive peak in the 2θ scale between 33 and 36 and is what gives soil its red colour. While being the most prevalent of iron oxides, goethite (which has a molecular structure of FeOOH) exhibits a distinctive peak between 20 and 22 and is what gives soil its yellow colour. The soil samples contained these minerals. Gibbsite was another mineral, and its primary peak was at roughly 25.0° 2θ. Quartz was also observed in the X-ray diffraction analysis. This mineral exhibits characteristics peaks around 22 and 27.0 in the 2θ scale.

4. Conclusions

In a column investigation, soil (Kalthur soil) was found to be capable of dephenolation from aqueous phenolated solution to a degree comparable to commercial activated carbon, but at a significantly lower cost. The design parameters bed depth and flow rate for an adsorption bed were

<table>
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<th>Sl. No.</th>
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Fig. 9. Adams–Bohart model (C0 100 ppm, flow rate 10 mL/min).

Fig. 10. Adams–Bohart model (C0 100 ppm, flow rate 20 mL/min).

Fig. 11. Adams–Bohart model (C0 100 ppm, flow rate 30 mL/min).
determined based on column studies. The optimal rate of flow of 10 mL/min, the $D_{\text{max}}$ was determined to be 4 cm for a bed depth of up to 14 cm. According to the Adams–Bohart model, $K_B$ values were observed to rise with rising flow rates while falling with rising bed heights. Although the maximum adsorption capacity ($N_e$) value decreased with an increase in bed height, it increased with an increase in flow rate. Regarding the analysis of soil sample the FTIR record shows that these samples include a wide range of functional groups, such as lactones, phenols, ethers, and carboxyl. The adsorbent's FTIR analysis before and after adsorption revealed distinct spectrum changes, including band disappearance and spectral shifts and expansions. Adsorption alters the peaks of –C–O–C bonds, the –C=C, C–O, and aliphatic (–CH3 and –CH2) functional groups, as well as adsorbent-specific oscillations in the –OH group, among other things.

**References**


