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# RESIDENCE TIME DISTRIBUTION STUDIES AND MODELLING OF PHENOL ADSORPTION ONTO LANTANA CAMARA IN PACKED BED

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## ABSTRACT

*Adsorption is considered as the effective method for removing phenol from wastewater. The phenol adsorption can be carried out in batch and continuous mode. The residence time distribution (RTD) studies are necessary to study mixing, flow behavior inside the column and ideality of the system. Therefore, in the current work, RTD studies were carried out in a column packed with the adsorbent viz Lantana camra (forest waste), which causes threat to the ecosystem. The experiments were conducted for different flow rates of phenol by using sodium chloride as tracer. The performance equation of the column was obtained by making mass balance of solute in a bed volume element. The dispersion coefficient calculated from the RTD was used for calculating mass transfer coefficient. The calculated mass transfer coefficient was used for developing the correlation in terms of dimensionless groups Reynolds number and Sherwood number.*

**Key words**: Phenol, Packed column, Residence time distribution, Dispersion coefficient, Mass transfer coefficient.

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# 1. INTRODUCION

The wastewater released from various industries contains pollutants which is a major concern for environmental conservation [1]. Phenol is one of the pollutant released and is recognised as priority pollutant by the environmental regulations [2]. It causes adverse effects on environment and public health. Its allowable concentration has to be lower than 0.1 mg/L before it is discharged into the aquatic environment [3]. Therefore, there is an essential requirement to treat wastewater before it is released into water bodies. Different processes such as biodegradation, biosorption, pervaporation, membrane separation, extraction, advance oxidation process and adsorption were employed for treating phenolic wastewater [4]. Adsorption using activated carbon is one of the effective wastewater treatment method. A variety of materials are investigated as adsorbents to treat phenol from wastewater. The prominent of these materials comprises of silica gel [5, 6], red mud [7, 8], zeolites [9, 10], activated alumina [11, 12], and activated carbon. But considering the cost of these materials, there is a requirement for adsorbent preparation from low cost agricultural materials. Therefore attempts are made to produce adsorbent by treating dry bark of *Lantana camara* with different chemicals. The packed bed experiments were conducted to obtain breakthrough curves and adsorption capacity [13].

But the literature on the RTD studies in the packed column for non-ideal flow pattern are limited. It is necessary to explore mixing and flow behavior inside the column [14]. It was established from the theory that the fluid elements in ideal reactor system possess the same residence time. But in real reactor system, the time the fluid elements spends are different [15]. Therefore, RTD examination is also helpful to find whether the system is approaching any of the ideal behavior or not. Conventional techniques are not available in the literature for the evaluation of the mixing performance of continuous operated systems [15].

Conventional methods are not available in the literature to evaluate the mixing performance of continuous systems [15]. Therefore to evaluate RTD, stimulus–response techniques are employed [16]. The stimulus used can be a tracer pulse input injected suddenly or a step input introduced continuously at a constant rate. The calculated RTD parameters are helpful to study the flow and mixing parameters in continuous operated systems [17].

The concentration v/s time data from the stimulus-response experiments are used for calculating the RTD parameters. Starting from mixing-cup measurement, the closed-vessel boundary condition was employed for pulse tracer experiments. This is different from the open-vessel boundary condition where through-the wall measurement are employed at the outlet for the boundary condition [15, 16].

Dispersion model is used to investigate the axial dispersion, the mixing state and the segregated flow [18]. The calculated dispersion coefficient is used for modelling purposes. From the RTD studies [16], has introduced the dimensionless group called dispersion number used for measuring the axial dispersion. The lower value of dispersion number shows lesser dispersion and plug flow in the column can be attained. The higher value shows larger dispersion and mixed flow can be assumed [18]. Various mathematical models are used to describe the behaviour of packed bed column. The model used for describing the column performance is obtained by making the mass balance of solute in a bed volume element. The partial differential equation is developed for showing the column performance.

Therefore in the current work, to find the non-ideal flow behaviour, the RTD studies were carried out. The experiments were conducted in a column packed with lantana camara adsorbent, at different flow rates of phenol by using sodium chloride as tracer. By using lantana camara, the menace of disposing the forest waste can be solved and it serves as low cost agricultural material for adsorbent preparation. The RTD parameters obtained from the experiment were to find the dispersion coefficient. From the performance equation of the packed column, mass transfer coefficient was calculated by numerical method. Finally the obtained mass transfer coefficient was used for developing a new empirical correlation in terms of dimensionless groups.

## 2. MATHEMATICAL ANALYSIS

### 2.1. Mathematical modelling of fixed bed adsorption column

Various mathematical models are developed for discussing the behaviour of packed bed column. The model is derived by making the mass balance of solute in an element of bed volume. The flow pattern is explained by the axially dispersed plug flow and the rate of mass transfer by linear driving force model (LDF).

The following assumptions are employed for developing the performance equation:

1. The system is maintained under the conditions of constant temperature.
2. The adsorption equilibrium is given by Langmuir isotherm.
3. The adsorbent particles are sphere shaped, have same size and density.
4. The flow rate and porosity is maintained constant with position in the column and no chemical reactions occurs.
5. The mass transfer by convection plays a significant role [19].
6. Mass transfer between the bulk liquid phase and the solid particle is given by external-film mass-transfer coefficient$(k_f)$.
7. Various mechanisms contributing to axial mixing are combined into a single dispersion coefficient [19, 20, 21, 22, 23]

By using the above assumptions, the packed bed model is given by the Eq (1):

$$\frac{\partial c}{\partial t} + v\frac{\partial c}{\partial z} = D_z\frac{\partial^2 c}{\partial z^2} - \rho\frac{\partial q}{\partial t} \tag{1}$$

where $v$ the interstitial velocity of fluid (cm/min), $c$ the adsorbate concentration (mg/L), $q$ the amount adsorbed (mg/g), $z$ the distance from the column inlet (cm), $D_z$ the dispersion coefficient (cm$^2$/min), $\rho$ the bulk density (g/cm$^3$) and $t$ the operating time (min).

### 2.2. Mass transfer

The external film mass transfer is considered as the rate controlling mechanism. The LDF model can be used for describing the rate of mass transfer.

The mass transfer rate is given by the following expression

$$\frac{\partial q}{\partial t} = k_{fa}(c - c_s) \tag{2}$$

where $k_{fa}$ the overall mass transfer coefficient (min$^{-1}$), $a$ surface area of adsorbent to the volume of particle slurry (m$^2$/m$^3$), $k_f$ the external film mass transfer coefficient (m/s) and $c_s$ solute concentration at the interface (mg/L) [23, 24].

The adsorbent surface area to the volume of particle free slurry ($a$) is

$$a = \frac{6m}{V d_p \rho (1-\varepsilon)} \tag{3}$$

Where $V$ the solution volume (L), $m$ the mass of adsorbent (g) and ε the bed porosity [25]. The following Danckwerts boundary conditions are considered.

$$\text{At } z = 0, t > 0, D_z \frac{dc}{dz} = v(c - c_o) \tag{4}$$

$$At\ z = L, t \geq 0, \frac{dc}{dz} = 0 \tag{5}$$

where $c_o$ the solute concentration in bulk liquid-phase (mg/L).

## 2.3. Theoretical investigation

The mean residence time ($t_m$) defined by the average transient time the material spends in the system is calculated by the equation [26, 27].

$$t_m = \frac{\int_0^\infty tcdt}{\int_0^\infty cdt} \tag{6}$$

The variance ($\sigma^2$) that indicates the spread of the tracer distribution is represented by the expression

$$\sigma^2 = \frac{\int_0^\infty (t-\bar{t})^2 cdt}{\int_0^\infty cdt} \tag{7}$$

The dimensionless variance function ($\sigma_D^2$) showing the measure of dispersion, is evaluated by the equation [28].

$$\sigma_D^2 = \frac{\sigma^2}{t_m^2} \tag{8}$$

The tracer experiments were conducted under closed vessel conditions and the dispersion coefficient was calculated from the following expression [28].

$$\sigma_D^2 = 2\left(\frac{D_z}{vL}\right) - 2\left(\frac{D_z}{vL}\right)^2 \left[1 - e^{-vL/D_z}\right] \tag{9}$$

where $D_z$ ($cm^2/min$) the dispersion coefficient, $L$ the length of the vessel (cm) and $v$ the interstitial velocity ($cm/min$). The calculated dispersion coefficient from RTD studies was used in the performance equation for the determination of mass transfer coefficient. The dimensionless group, Dispersion number ($D_z/vL$) which characterizes the extent of axial dispersion was also evaluated [18].

## 2.4. Numerical solution

The differential equation was solved by applying upwind difference scheme to convection term and central difference scheme to dispersion term.

$$D_z\left(\frac{c_{i+1}^{n+1} + c_{i-1}^{n+1} - 2c_i^{n+1}}{\Delta z^2}\right) - v\left(\frac{c_i^{n+1} - c_{i-1}^{n+1}}{\Delta z}\right) - k_{fa}\left(c_i^{n+1} - c_s\right) - \left(\frac{c_i^{n+1} - c_i^n}{\Delta t}\right) = 0 \tag{10}$$

The length of the column was divided into 10 parts (nodes). $\Delta z$ the length of each node (cm). The discretization of the differential equation was done at 10 parts to get 11 different equations. The boundary conditions are applied at node 1 and node 11. The equations are represented in the tridiagonal form and solved using Thomas Algorithm [29]. The value of $k_{fa}$ is assumed by trial and error method. Then for the assumed $k_{fa}$ value, the values of the concentration at different nodes were determined. If the evaluated value of concentration at the exit of the column ($z = L$) closely matches with the experimental value of concentration at the interface($c_s$), then the assumed $k_{fa}$ value is correct.

## 3. MATERIALS AND METHODS

### 3.1. Adsorbent

The *Lantana camara* bark was washed with distilled water to remove all the earthy matter. The dry bark was later dried in sunlight for 48 h, cut into pieces, grinded to fine powder, screened to obtain particles lesser than 0.075mm. The materials were then treated with different chemical agents $H_3PO_4$, $KNO_3$, $H_2SO_4$, $ZnCl_2$, HCl and KOH to improve the properties of the carbon and batch experiments were carried out. From the preliminary results of batch studies, HCl treated adsorbent was taken for packed bed experiments [13].

### 3.2. Adsorbate

Phenol with chemical formula $C_6H_5OH$ has molecular weight of 94 g/mol. Phenol of analytical grade was used for the stock solution preparation. The phenol solution of 150 mg/L concentration was used for RTD studies. The other chemicals potassium hydroxide, potassium nitrate, zinc chloride, hydrochloric acid, sulphuric acid, and orthophosphoric acid were used for the chemical treatment in the batch studies. Sodium chloride was used as tracer.

### 3.3. Tracer experiments

The experimental arrangement of the RTD studies is as shown in Fig 1. The studies were carried out in the packed column having 2 cm inside dia and 50 cm height. The column was packed with 5.93 g of HCl treated adsorbent to make a bed height of 10 cm. The phenol solution of initial concentration 150 mg/L was pumped from the bottom of the column through a peristaltic pump at different flow rates 5ml/min, 10 ml/min, 15 ml/min and 20 ml/min. Sodium chloride which is an inorganic salt, electrolyte and does not interfere with the solution is taken as tracer. Before starting the experiments, the column was completely saturated with phenol solution to prevent the sodium chloride getting adsorbed in the column. After saturating the column with tracer, 10 ml of 1M sodium chloride was introduced as pulse input at the bottom near the column inlet as shown in the Fig 1. Conductivity measurement was done at the bed exit, with a probe inserted into a specially designed mixing cup of volume 100ml. The tracer Concentration was measured with the help of mixing cup and an electrode.

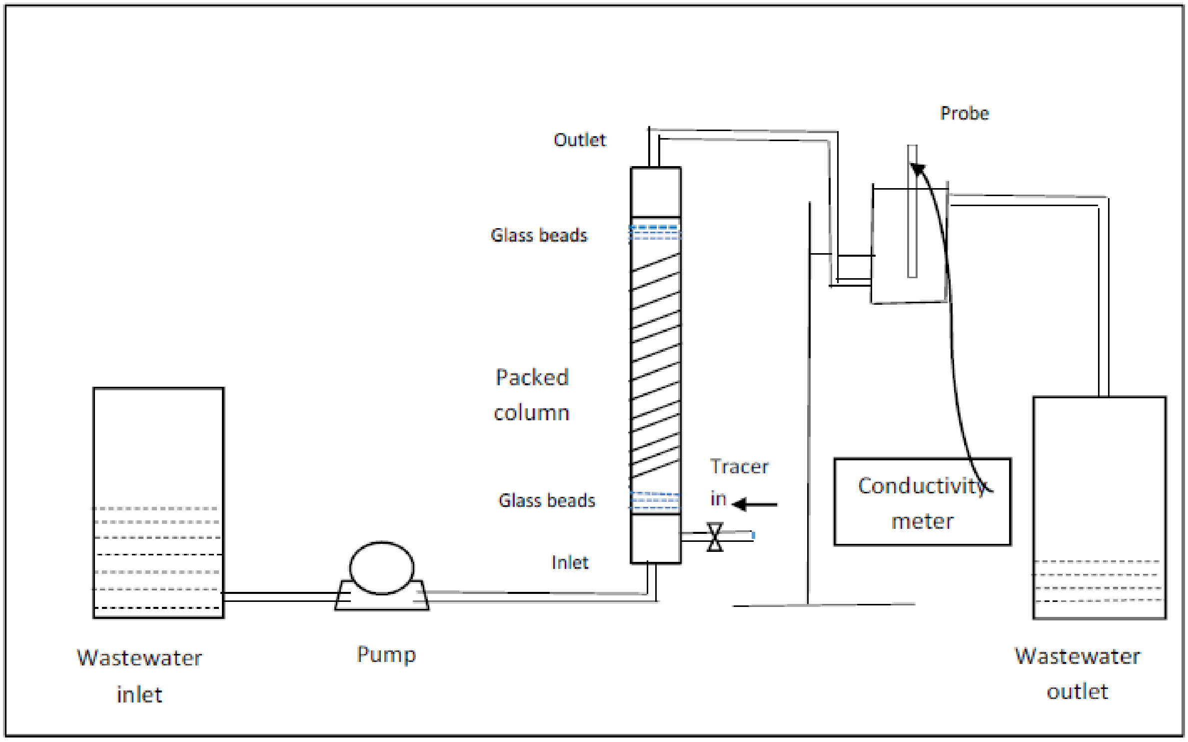


**Figure 1** The diagrammatic representation of RTD studies experimental arrangement

The conductivity measurements were done for different flow rates of phenol using conductivity meter. The experiments were done till the conductivity at the end of the column attains the value as that of the feed. Using the linear relation between the conductivity and concentration, the conductivity values were taken directly for RTD calculations [30].

# 4. RESULTS AND DISCUSSION

## 4.1. Determination of RTD parameters

The parameters like mean residence time, variance, dimensionless variance, dispersion coefficient and dispersion number at various flow rates were found and are given in Table 1. It was found that the mean residence time $(t_m)$ decreases as the flow rate increases. This is due to the reason that by increasing the flow rate, the time spent by each fluid element inside the column decreases. The dimensionless variance $\sigma_D^2$ value increases by increasing the flow rate. The probable reason is that by increasing the flow rate, dispersion also increases [14]. The two statistical moments $t_m$ and $\sigma^2$ shows the temporal and spatial distribution of the tracer inside the packed column.

By using the values of dimensionless variance and mean residence time, the dispersion coefficient $(D_z)$ was evaluated under the closed vessel conditions and are given in Table 1. It was observed that by increasing the flow rate, the dispersion number increases. From this, it can be concluded that a lower dispersion number signifies less dispersion and the flow approaches towards plug flow. The higher dispersion number shows large dispersion and the flow tend towards mixed flow. The results are similar to those reported by the authors [18].

The dispersion coefficients were obtained in the range of 0.05002 to 0.210176 $cm^2$/min for interstitial velocities varying from 0.2725 to 1.092 cm/min. The values were in agreement with those reported in the work. The eq. (2) has been developed for showing the relationship between interstitial velocity and dispersion coefficient and the dependence between the variables is shown in Fig 2. A similar expression has obtained in the work reported by authors [31].

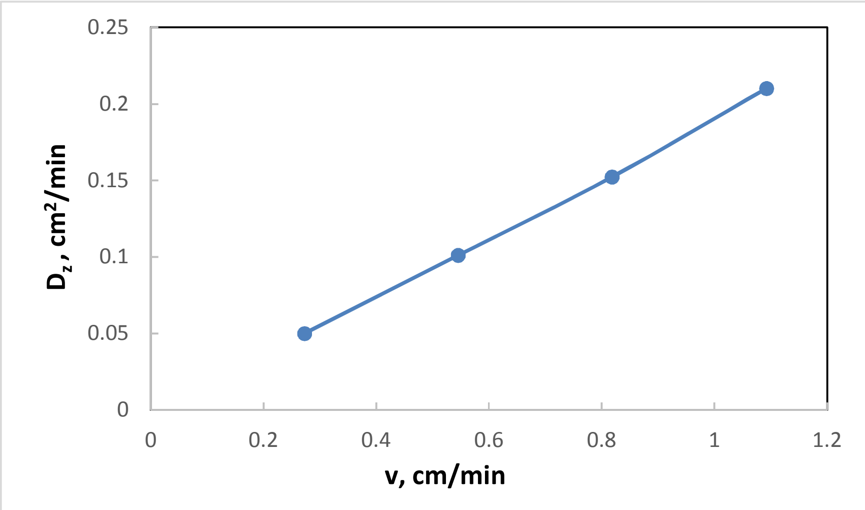


**Figure 2** The influence of interstitial velocity on the dispersion coefficient.

$$D_z = 0.1893v \qquad (11)$$

**Table 1** The values of RTD parameters and the dispersion number

| Flow rate, ml/min | $t_m$, min | $\sigma^2$,$min^2$ | $\sigma_D^2$ | $D_z$,$cm^2$/min | Dispersion number |
|---|---|---|---|---|---|
| 5 | 142.82±0.1367 | 735.54±25.72 | 0.03604±0.0012 | 0.05002 | 0.01835 |
| 10 | 98.625±0.1377 | 354.16±12.45 | 0.0364±0.0012 | 0.101 | 0.01853 |
| 15 | 70.42± 0.0168 | 184.67±6.003 | 0.0372±0.0012 | 0.1522 | 0.01860 |
| 20 | 44.84±0.04641 | 77.387±7.019 | 0.0384± 0.0018 | 0.21017 | 0.01924 |

## 4.2. Relationship between velocity and mass transfer coefficient

The overall mass transfer coefficient was calculated assuming that external film resistance controls the process [24]. The values were obtained in the range of 0.41 to 0.81 $min^{-1}$ for the interstitial velocity varying from 0.2725 to 1.092 cm/min and are represented in Table 2. It was observed that by increasing flow rate, the mass transport resistance across the liquid film decreases and thus results in larger mass transfer coefficient values.

**Table 2** The values of evaluated mass transfer coefficients and the dimensionless groups.

| Flow rate, ml/min | $v$,cm/min | $k_{fa}$, $min^{-1}$ | $k_f \times 10^4$, m/sec | $Re$ | $Sh$ |
|---|---|---|---|---|---|
| 5 | 0.272 | 0.41 | 5.33 | 0.5908 | 1.9074 |
| 10 | 0.545 | 0.51 | 6.21 | 1.1816 | 2.2251 |
| 15 | 0.818 | 0.65 | 7.59 | 1.7736 | 2.7184 |
| 20 | 1.092 | 0.81 | 9.36 | 2.3677 | 3.3495 |

It was studied from the literature that the overall mass-transfer coefficient $k_{fa}$ depends on the flow conditions in the system. Therefore a relationship between $k_f$ and v was developed and it can be correlated to dimensionless groups like the Sherwood number $(Sh)$ and Reynolds number $(Re)$ [32].

$$Sh = k_f d_p / D_m \tag{12}$$

where $k_f$ is liquid film mass transfer coefficient (m/s)

$$Re = d_p v \rho / \mu \tag{13}$$

The $D_m$ was calculated from the Wilke-Chang equation [32]. As we know, the Sherwood and Reynolds numbers are related to the mass transfer coefficient and interstitial velocity respectively. Then a relationship between $v$ and $k_f$ in terms of dimensionless numbers was obtained. The influence of interstitial velocity on the overall mass transfer coefficient and in terms of dimensionless groups is shown in Fig 3 and 4 respectively [33].

$$k_f = 8.48626 v^{0.3912} \tag{14}$$

$$Sh = 2.2439 Re^{0.3919} \tag{15}$$

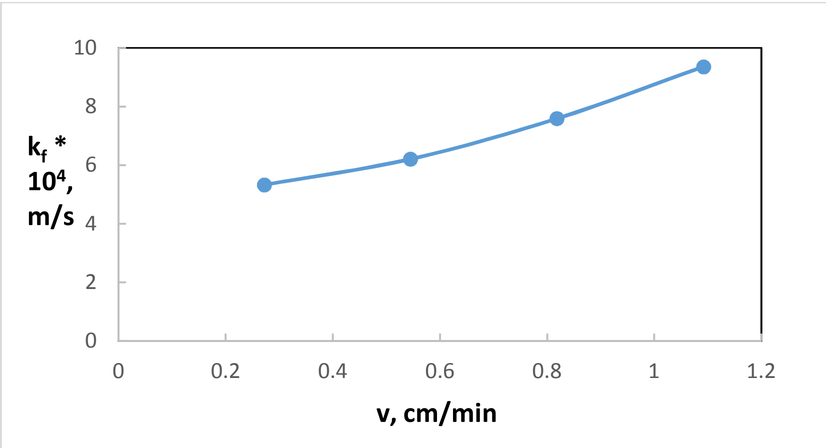


**Figure 3** The influence of interficial velocity (v) on the mass transfer coefficient ($k_f$).

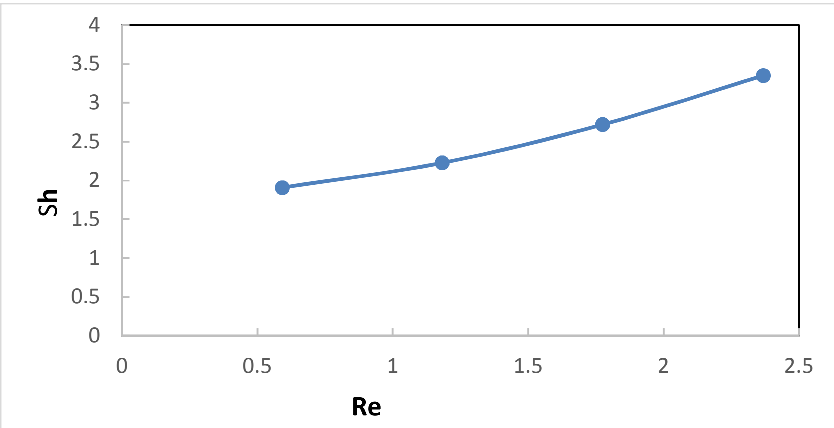


**Figure 4** The variation of Reynolds number with the Sherwood number

## 5. CONCLUSIONS

The RTD studies were carried out in a column packed with low cost agricultural waste lantana camara as adsorbent. It was observed from the tracer experiments that the mean residence time, variance decreases as the flow rate increases. The two statistical moments $t_m$ and $\sigma^2$ shows the temporal and spatial distribution of the tracer in the column. It was also found from the results that the dispersion number increases by increasing flow rate signifying that the rapid spreading of the curve takes place and is deviating more from the ideal behaviour at higher flow rates. Thus, it can be summarized that lesser the dispersion number the flow tend towards plug flow, the larger dispersion number shows flow approaches to mixed flow. The overall mass transfer coefficient obtained in the range of 0.41 to 0.81 $min^{-1}$ for the interstitial velocity varying from 0.2725 to 1.092 cm/min respectively. A new empirical relation was developed between dimensionless groups to show the influence of interstitial velocity on mass transfer coefficient which is beneficial for the scale up of the process.

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