Design of Laboratory Scale Packed bed column for adsorption of phenol on to modified coal fly ash (MCFA)

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Abstract— **Designing a packed bed adsorption column from laboratory scale experimental data is of great importance for the commercialisation of the continuous adsorption process. In this study, the experimental data from laboratory scale packed bed adsorption column was used for designing the adsorption column. The laboratory scale column study was carried out using modified coal fly ash (MCFA) as adsorbent for removal of phenol from aqueous solution. The breakthrough curve parameters obtained from laboratory scale experimental data were used to design the packed bed column. The designing has been done by scale-up approach method. It was observed that the flow rate affects the breakthrough curve and adsorption efficiency decreases with increase in flow rate. The empty bed contact time (τ) of the laboratory scale packed column was found 10.5975 minute while the breakthrough time was 113.98 minute. Fraction of capacity left unused was found 45.71% for the laboratory scale packed column. The design parameters for packed column was found as; area (A) 117.75 cm^2, diameter (d) 12.2435 cm and the mass of MCFA required 3338.1703 grams for the flow rate of 150 cm^3/min. The purpose of this study is to use laboratory experimental data for design of the packed bed column.**

Index Terms— ***Adsorption, .Breakthrough curve, Design, Modified coal fly ash, Phenol, , Packed bed column.***

1) INTRODUCTION

Packed bed column adsorption study can be operated in series or in parallel. Influent can be fed in the packed bed column by two different modes such as down flow and up flow. In case of down flow mode, the adsorption of toxicants can occur in a single step. Sometimes up flow packed bed columns have been used but down flow columns are used more commonly to avoid the accumulation of particulate material on the bottom of the bed. Laboratory scale column experimental data can be used to replicate the potential performance of the adsorbent and the results obtained are used in the design of full-scale columns. In case of continuous influent flow, the mass transfer zone moves downward through the bed. As the mass transfer zone moves the bottom of the column bed, the concentration of adsorbate in the effluent increases and at last equals the influent adsorbate concentration [1].

Based on this, the concept of breakthrough curve and breakthrough point comes in the fixed bed column study. The breakthrough curve is a plot of the adsorbate concentration at the column outlet versus time (C_t/C_0 vs. t) and the breakthrough time is the time at which the column exit concentration of adsorbate attains a maximum permissible concentration. Similarly the column exhaustion time is the time at which the column exit concentration of adsorbate equals the influent adsorbate concentration. Hence the breakthrough and exhaustion are defined as the phenomena when the ratios of effluent-to-influent concentrations are 5% and 95% respectively. Values of column breakthrough and exhaustion are often used to evaluate adsorption parameters in column adsorption systems.

Packed bed column can be designed using two approaches; scale-up and kinetic approach. In both approaches a breakthrough curve from the test column, either laboratory or pilot scale is required and the column should be as large as possible to minimize side-wall effects. The procedure for scale-up approach for the packed column design is as follows [1]:

1) Use a laboratory test column filled with the MCFA (adsorbent) to be used in full scale application.
2) Apply a filtration rate and contact time (EBCT) which will be the same for full-scale application (to obtain similar mass transfer characteristics).
3) Obtain the breakthrough curve.
4) Work on the breakthrough curve for scale-up.

One advantage of packed bed column adsorption study is that the fresh adsorbates are come in contact with the adsorbent as time proceeds. This condition does not exist in the batch study.

This work is associated with the adsorption of phenol on MCFA form aqueous solution in a laboratory scale packed bed adsorption column and design a packed bed column from the breakthrough curve parameters generated. The scale-up approach was used to design the packed bed adsorption column.

2. MATERIALS AND METHODS

2.1. Materials

The material used for this study includes:
(i) Adsorbate: Phenol
(iii) Adsorbent: Modified coal Fly Ash.

2.1.1. Adsorbate

The phenol composition of the aqueous solutions prepared for experimentation was similar to that generated by the various industries which produces the phenol containing wastewater. The stock solution was prepared by diluting the required quantities of phenol in the distilled water to obtain adsorbate solutions of various initial concentrations (C_o). Fresh solutions were prepared on a time of experiment for phenol concentration.

2.1.2. Adsorbent

Modified coal fly ash was prepared as given in our earlier research paper [2]. It was dried at 100^0C for one hour before using for experimentations.

2.2. Experimental Methods

MCFA was packed in the glass column having 45 cm in length and 1cm internal diameter with the support of glass bed and cotton wool at the bottom and top of the MCFA bed respectively. The column was operated under down flow condition which allows the influent to be gravity fed and also ensure that the bed remains packed and steady during the entire operation, which results in the maximum contact between the MCFA and the influent. The experiments were conducted by varying the flow rate (0.375, 0.750, 1.0 ml/ min) with initial phenol concentration of 1039.9 mg/L and assessing the breakthrough curve. Effluent from the bottom of the column was collected with fixed interval of time for phenol concentration which was determined by UV spectrophotometer. The block diagram of the experimental set-up for this study is represented in Fig.1.

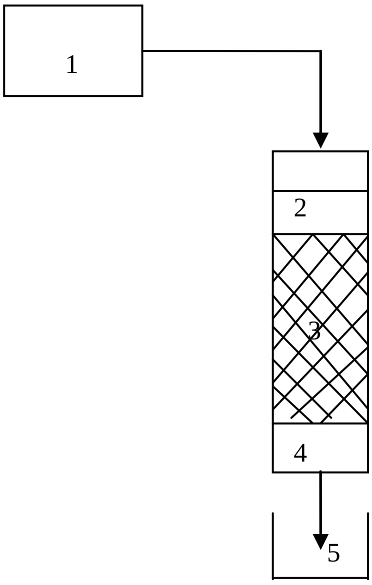

Fig.1. Block diagram of the experimental set-up for packed bed adsorption study. (1. Influent phenol solution; 2. Cotton wool; 3. MCFA bed; 4. Glass bed; 5. Sample collection)

3. RESULTS AND DISCUSSIONS

3.1. Effect of flow rate

The effect of flow rate on phenol removal by MCFA was studied by varying the flow rate in the range of 0.375, 0.750 and 1.000 ml/min, while the bed height and initial phenol concentration were held constant at 13.5 cm and 1039.9 mg/L respectively. The breakthrough curves obtained by plotting effluent phenol concentration versus time at different flow rates and its effect on phenol uptake and column exhaust time are represented in Figure 2 and 3 respectively.

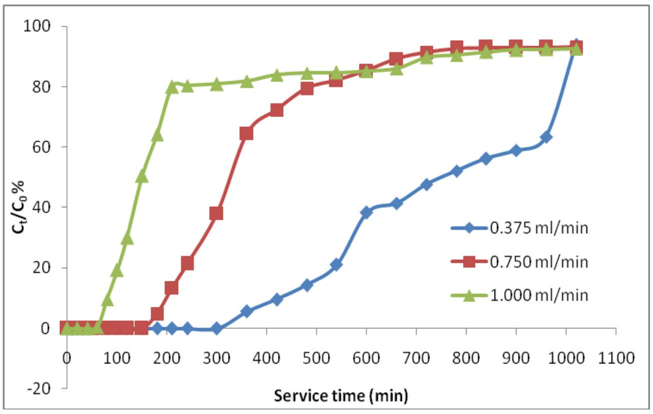

Fig. 2. Breakthrough curve.

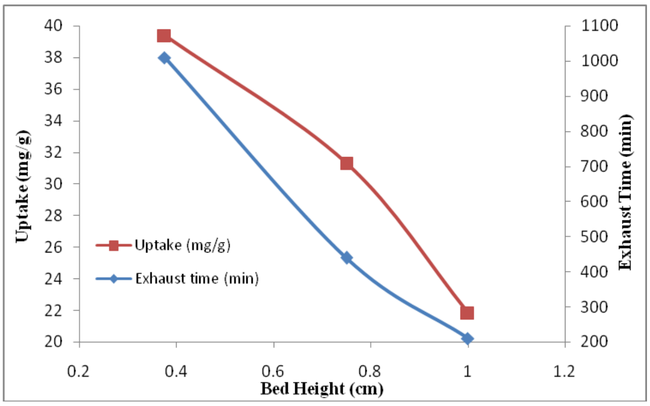

Fig. 3. Phenol uptake and column exhaust time.

It was observed that (Fig. 3) the adsorption efficiency was higher at lower flow rate. This might be due to the fact that at lower flow rate the diffusion of phenol in the pores of MCFA becomes slow and hence the MCFA needs more time to bind the phenol molecule. However if residence time of the phenol in the column is not large enough for adsorption equilibrium to be reached at that flow rate the phenol solution leaves the column before equilibrium occurs. It was also observed that the breakthrough time decreases as flow rate increases. The faster breakthrough occurred at higher flow rates and thus the shortened bed service time was (Fig. 2). The breakthrough curve became steeper when the flow rate was increased which implicates the mass transfer zone was shortened indicating more effective intraparticle diffusion effects (Pablo D. R. *et al.* 2015). Goyal M. *et al.* (2009) reported that as flow rate increases beyond the certain value it results in decrease in adsorption rate because of the decrease in the residence time of the adsorbate in the column.

3.2. Scale-up of packed bed column

The laboratory scale test column as described above was used for full scale design of packed bed column. The laboratory scale packed bed column experimental data are tabulated as under.

<u>Laboratory Scale Data</u>

Flow rate (Q) = 1 ml/min (1 cm^3/min)

- Column diameter = 10 mm (1 cm)
- Column depth (packed bed) = 135 mm (13.5 cm)
- Density of MCFA = 2.1 g/ml (2.1 g/cm^3)
- Breakthrough volume ($V_{breakthrough}$) = 114 ml (114 cm^3) at 20% breakthrough
- Exhaustion volume ($V_{exhaustion}$) = 210 ml (210 cm^3) at 80% breakthrough

a) Filtration rate of the Laboratory scale test

Filtration rate (FR) = Q (Flow rate)/A (cross-sectional area)

Area = $\pi\ d^2/4$ = $3.142 * (1^2)/4$

$= 0.785\ cm^2$

FR $= 1/0.785$

$= 1.27388$ cm/min

This same FR is applied to packed column.

b) Area of the Packed Column

Area = Q (Flow rate)/FR (Filtration rate)

If the flow rate of the packed column design is to be 150 cm^3 /min.

Area = 150/1.27388

$= 117.75\ cm^2$

Since, Area = $\pi d^2/4 = 117.75$, the column diameter can be calculated as

d = √ [(4*117.75)/ π]

d = √ [(471)/ 3.142

d = √ 149.9045

d = 12.2435 cm

Hence area 117.75 cm^2 and the diameter 12.2435 cm was found for packed column.

c) Empty Bed Contact Time of the Laboratory scale Packed column

τ (Empty bed contact time) = Volume of bed / Q (Flow rate)

Volume of bed = A (Cross sectional area) * Height

$= 0.785\ cm^2 * 13.5$ cm

$= 10.5975\ cm^3$

τ (Empty bed contact time) = 10.5975/1

= 10.5975 min.

10.5975 minutes is the EBCT of the Packed Column.

d) Height of the Packed Column

(EBCT) τ * Filtration rate (FR) = 10.5975 * 1.27388

= 13.49994 cm ≈ 13.5 cm

This is the same as the height of the laboratory scale test column because height of a column is set by the EBCT(τ) and FR and these are the same for laboratory scale test column and the packed column.

e) Mass of MCFA required in the scale-up Packed Column

Volume of the packed column = cross sectional area * Height

Volume of the packed column = ($\pi d^2/4$) * Height

Volume of the packed column = $[(3.142*12.2435^2)/4]$ * 13.49994

Volume of the packed column = [(3.142*149.9032)/4] * 13.49994

Volume of the packed column = [(470.9961)/4] * 13.49994

Volume of the packed column = [117.7490] * 13.49994

Volume of the packed column = 1589.6049 cm^3

= (1589.6049 ml)

Packed bed MCFA density is = 2.1g/ml

Mass of the MCFA = 2.1 * 1589.6049

Mass of the MCFA = 3338.1703 g

= (3.3381703 kg)

f) Determination of q_e (Phenol removed)

Mass of MCFA in the laboratory scale test column= Volumef lab. scale column * Density of MCFA

= 10.5974 ml *2.1 g/ml

= 22.2547 g k

Total capacity (t) = Volume at exhaustion * Time to reach exhaustion

= 210 * 210

= 44100 ml (44100 cm^3)

Phenol removed by 22.2547 g of the MCFA =Total capacity / Mass of the MCFA in the Laboratory scale test column

= 44100 (ml)/ 22.2547(g)

= 1981 ml/g

= (1981*1.07) g/g

= 2119.67 g/g

= 21.1967 mg/g

The laboratory scale test column found the 21.84 mg/g phenol removed

g) Fraction of Capacity Left Unused (Laboratory scale column)

Total capacity = 210 * 210 = 44100 ml

Phenol removed before breakthrough = 114 * 210

= 23940 ml

Fraction of capacity left unused (f) = (44100-23940)/44100

= 0.4571

= 45.71%.

This fraction of capacity left unused will apply to the packed column also.

h) Breakthrough time of the Packed Column

Phenol loading rate = 210 * 150 = 31500 g/min

MCFA consumption rate = 31500/1981 = 15.90 g/min

Amount of MCFA consumed = 3338.1703 (1- 0.4571)

= 1812.29 g.

Breakthrough time = Amount consumed/MCFA consumption rate

= 1815.29/15.90

= 113.98 min ≈ 114 min.

This is the same as the packed bed column: 114 min.

i) Volume Treated Before Breakthrough

Volume treated = Flow rate × breakthrough time

= 150 * 113.98

= 17097 cm^3

= 17097 ml

= 17.097 Litre

4. CONCLUSION

The effect of flow rate on breakthrough curve showed that the breakthrough curves became steeper and the slope of the breakthrough curve increased with increasing flow rate. The faster breakthrough observed at higher flow rates which results in the faster exhaustion of the bed. Higher phenol uptake was observed at lower flow rate. The obtained parameters from laboratory scale column were used to design a packed bed column using scale-up approach. The design parameters for packed column was found to be area 117.75 cm^2, diameter(d) 12.2435 cm, the mass of MCFA required 3338.1703 grams and the volume treated before breakthrough 17097 cm^3 for the flow rate of 150 cm^3/min.

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