

Performance Evaluation of Column Dynamics for Phenol Adsorption by Coal Fly Ash

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ABSTRACT

In this study coal fly ash (CFA) which is the waste product of coal fired power plant is used as an adsorbent for adsorption of phenol from aqueous solution in packed bed column. The effect of bed height (7.5, 13.5, 27.5 cm), effect of flow rate (0.375, 0.75 and 1.0 ml/min) and initial phenol concentration (70.0, 292.7, 651.2, 1039.9 mgL⁻¹) on the adsorption were studied by assessing the breakthrough curve. Thomas and Yoon-Nelson models were used to evaluate the column performance. The result shows that with increase in flow rate, break point time decreases while the values of maximum adsorption capacity (q_0) decreased and the Thomas constant (K_{Th}) increased. As the bed height and initial concentration increases, break point time increases with bed height but decreases with initial concentration but q_0 increased while K_{Th} decreased. The maximum adsorption capacity was found to be 5.90 mg/g at 1.0 ml/min flow rate and 27.5 cm bed height with phenol concentration of 1039.9 mg/L.

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1. Introduction

Phenolic wastewater is a serious environmental problem and this water cannot release into the environment without treatment [1]. The nature of phenol and their derivatives for hazards and toxicity have been well documented [2, 3, 4] and can cause several health problems. Phenolic compounds have been classified as high-priority pollutants by the USA EPA, 1984 [5]. Phenolic waste water are generated from the plastic, pharmaceutical, petrochemical, paint, paper & pulp, solvent, coal conversion industries. They are known one of the priority pollutants in wastewater, because they are harmful to organisms even at low concentrations [6, 7, 8].

CFA is considered as a waste for power generation plants, but from coal utilization point, it is a resource yet to be fully utilised and exploited. Currently, the cement industry might use them as a raw material to produce concrete. The coal fly ash generated in power plants are designated as a by-product and it is used as a recycling materials in the various fields like agriculture and engineering is widely attempted [9, 10]. As one of the effective use of coal fly ash, its conversion into zeolites has also been widely attempted [11].

Many researchers are studied the batch adsorption of phenol on to coal fly ash, but limited of them worked on the column dynamics of phenol with CFA. Packed bed columns are the continuous contacting apparatus which includes the continuous flow of the influent from the top of the column on the adsorbent bed and continuous withdrawal of the effluent from bottom resulting in the removal of adsorbate on the surface of the adsorbent. Column adsorption is preferred over batch adsorption because of its ease of operation, high yields, and high liquid residence times and can be scaled up from a laboratory step [12].

2. Materials and Methods

2.1. Materials

The material used for this research work includes:

(i) Adsorbate: Phenol

(ii) Adsorbent: 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_0). Fresh solutions were prepared on a time of experiment for phenol concentration.

2.1.2. Adsorbent

Fly ash is the fine powdery residue obtained from the coal fired power station after combustion of the coal. The fly ash utilized in this study was a low grade Coal Fly Ash (CFA) obtained from the Paras Thermal Power Station at Paras village in Akola district of Maharashtra State, India. It is washed several times with distilled water to remove any contaminants like dirt, soil etc. and dried at 100°C for an hour before using for experimentations.

2.2 Experimental Methods

2.2.1. Dynamic column study

CFA was packed in the glass column having 45 cm in length and 10 mm internal diameter with the support of glass bed and cotton at the bottom of the column. 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 fly ash and the influent. The experiments are conducted for the effect of bed height (7.5, 13.5, 27.5 cm), the effect of flow rate (0.375, 0.750, 1.0 ml min⁻¹) and initial phenol concentration (70.0, 292.7, 651.2, 1039.9 mgL⁻¹) on the adsorption by assessing the breakthrough curve. The pH of the influent was kept at 6.0 for all the experiments. Effluent from the bottom of the column was collected with fixed interval of time and phenol concentration was determined by UV spectrophotometer.

Adsorption parameters are calculated by using the following equations 1 to 5 [13]. Thomas and Yoon-Nelson models have been studied for getting the maximum adsorption capacity and rate constant as well as the time required for 50 % adsorbate breakthrough.

The effluent volume was calculated by using the following equation [15].

$$V_{\text{eff}} = Q \cdot t_{\text{total}} \quad \dots\dots\dots(1)$$

Where, V_{eff} is volume of effluent collected in ml, Q is volumetric flow rate in ml/min, and t_{total} is total flow time in minutes.

Maximum bed capacity for given flow rate and influent phenol concentration was calculated by using the following equation.

$$q_{\text{total}} = Q \int C_{\text{ad}} dt / 1000 \quad \dots\dots\dots(2)$$

Where, q_{total} is maximum bed capacity in mg and C_{ad} is adsorbed phenol concentration in mg/liter. The value of integral is obtained from the area under the curve of adsorbed phenol concentration versus time.

The total amount of phenol fed to the column was calculated by using the following equation.

$$M_{\text{total}} = C_0 Q t_{\text{total}} / 1000 \quad \dots\dots\dots(3)$$

Where, M_{total} is the total amount of phenol fed to the column in gm and C_0 is the initial phenol concentration in mg/lit.

The percentage of phenol removal was calculated by using the following equation.

$$\% \text{ Removal} = (q_{\text{total}} / M_{\text{total}}) \times 100 \quad \dots\dots\dots(4)$$

Equilibrium phenol uptake was calculated by using the following equation.

$$q_{\text{eq(exp)}} = q_{\text{total}} / m \quad \dots\dots\dots(5)$$

Where $q_{\text{eq(exp)}}$ is the equilibrium phenol uptake in mg/gm and m is the amount of fly ash present in the column.

2.2.2. Effect of Bed Height

The column was packed with glass bed and the cotton wool at the bottom as the support for the fly ash bed. Then the different weight of the fly ash has been poured into the column to form the bed of different height. In this study fly ash having 5, 10 and 20 grams are taken which results in the 7.5, 13.5 and 27.5 cm bed height of the CFA respectively. Cotton was used as the supporting layer to keep the glass bed and cotton wool in position and to support the fly ash. The process was performed at the constant flow rate of 0.0375 ml/min and phenol concentration was 1039.9 mg/liter. Effluent concentrations were measured as a function of time for each column and analyzed by UV spectrophotometer. The breakthrough curves and breakpoint has been drawn for each column. Thomas and Yoon Nelson model have been used to explain the dynamic adsorption process.

2.2.3. Effect of Flow rate

The same column as described above is used to study the effect of flow rate on adsorption process. Different flow rate of 0.375, 0.750 and 1.0 ml/min are used for different bed height of 7.5, 13.5 and 27.5 cm. separately. Effluent concentrations were measured as a function of time for each column and analyzed by UV spectrophotometer. The breakthrough curves and breakpoint has been drawn for each column. Thomas and Yoon-Nelson model have been used to explain the dynamic adsorption process.

2.2.4. Effect of initial concentration

The same column as described above is used to study the effect of initial phenol concentration on adsorption process. Different initial concentration of 70, 292.7, 651.2 and 1039.9

mg/L are used for different bed height of 7.5, 13.5 and 27.5 cm. with a constant flow rate of 1.0 ml/min separately. Effluent concentrations were measured as a function of time for each column and analyzed by UV spectrophotometer. The breakthrough curves and breakpoint has been drawn for each column. Thomas and Yoon-Nelson model have been used to explain the dynamic adsorption process.

2.2.5. Modeling of Fixed bed column

Experimental data can be used as the basis for the design of fixed bed column operations. Many models have been proposed for the evaluation of efficiency and applicability of the models for industrial operations. To design a dynamic column adsorption operation, break through curve and adsorbent capacity prediction for the required adsorbate under given set of operating conditions is necessary. In the present study, adsorption data from fixed bed dynamic column were analyzed using Thomas model and Yoon-Nelson model.

2.2.6. Thomas Model

The Thomas model is frequently applied to calculate the adsorption capacity of adsorbent and predict break through curve assuming the second order reversible reaction kinetics and the Langmuir isotherm [14, 15]. Linear form of Thomas model is given as under

$$\ln [(C_0 / C_t) - 1] = K_{\text{TH}} q_0 M / Q - K_{\text{TH}} C_0 t \quad \dots\dots\dots(6)$$

Where, C_0 is initial phenol concentration, mg/lit; C_t is effluent phenol concentration at time t , ppm; K_{TH} is Thomas model constant, L/min.mg; q_0 is prediction adsorption capacity, mg/gm. M is mass of adsorbent, gm; Q is inlet flow concentration, ml/min. The value of K_{TH} and q_0 are determined from slope and intercept of a plot of $\ln (C_0/C_t - 1)$ versus t .

2.2.7. Yoon - Nelson Model

The main aim of Yoon-Nelson model is to predict the time of column run before regeneration of column becomes necessary. The model is a very simple way to represent the break through curve. According to this model, the amount of phenol adsorbed in a fixed bed is half of the total phenol entering the adsorbent bed within time period 2τ , where τ is the time required for 50 % break through. Linear form of Yoon-Nelson model is given below [16].

$$\ln [C_t / (C_0 - C_t)] = K_{\text{YN}} t - \tau K_{\text{YN}} \quad \dots\dots\dots(7)$$

Where, C_0 is initial phenol concentration, mg/lit; C_t is phenol concentration at time t , mg/lit; t is flow time, min.; τ is time required for 50 % breakthrough, min; K_{YN} is Yoon-Nelson rate constant, 1/min. The values of K_{YN} and τ are determined from the slope and intercept of $\ln [C_t / (C_0 - C_t)]$ versus t .

The adsorption capacity in mg/gm can be calculated by the following equation [17].

$$q_0 = C_0 Q \tau / m \quad \dots\dots\dots(8)$$

Where, C_0 is initial phenol concentration, mg/lit; Q is inlet flow concentration, ml/min; τ is time required for 50 % breakthrough, min; and m is the mass of adsorbents, gm.

3. Results and Discussions

3.1 Effect of operating Conditions

3.1.1 Effect of bed height

The effect of bed height on fixed bed adsorption at different bed height of 7.5, 13.5 and 27.5 cm is shown in following figure

It is observed that as the bed height increases, breakpoint time also increases. This shows that at the smaller bed height the effluent phenol concentration ratio increases more rapidly than for a higher bed height.

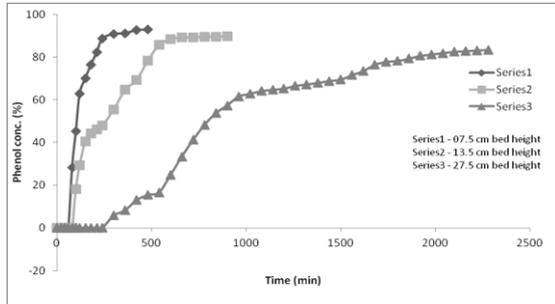


Fig 1. Effect of bed height on breakthrough curve for phenol adsorption. (Amount of CFA= 5, 10 and 20 grams, Bed Height = 7.5, 13.5 and 27.5 cm, Influent flow rate = 0.375 ml/ min, Initial concentration (C₀) = 1039.9 mg/lit).

Additionally the bed is saturated in less time for smaller bed height than the bigger one. Smaller bed height corresponds to less amount of adsorbent..As a result, a smaller capacity of the bed to adsorb phenol from solution and a faster increase in the rate of adsorption is expected [18]. For the bed height of 7.5, 13.5 and 27.5 cm, the break point time was observed as 68, 124 and 246 minutes respectively. This shows that as the bed height increases the velocity variation effect on breakthrough curve increases. The column parameters obtained from effect of bed height are listed in following table 1.

Table 1. Effect of bed height on adsorption

Bed Height (cm)	q _{total} (mg)	M _{total} (gm)	% Phenol Removal	q _{eq(exp)} (mg/gm)
7.5	12.43	187.18	6.64	2.48
13.5	30.91	397.76	7.77	3.09
27.5	82.58	889.11	9.04	4.02

As shown in table 1, the total adsorption capacity of the bed increased with increase in bed height. At higher bed depth, more sites were available for adsorption and this resulted in higher phenol removal.

3.1.2 Effect of Flow rate

The effect of varying flow rate as 0.375, 0.750 and 1.0 ml/min on adsorption column for different bed height of 7.5, 13.5 and 27.5 cm are shown in following figures.

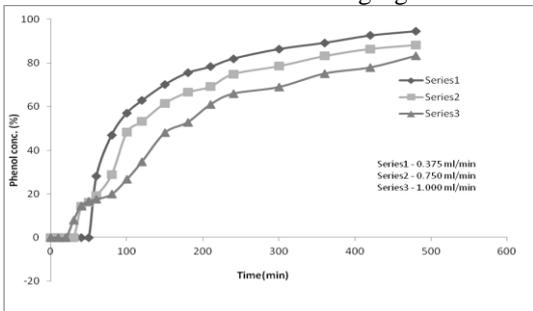


Fig 2. Effect of flow rate on breakthrough curve for phenol adsorption. (Bed Height = 7.5 cm, Amount of CFA= 5 gm, Initial concentration (C₀) = 1039.9 mg/lit).

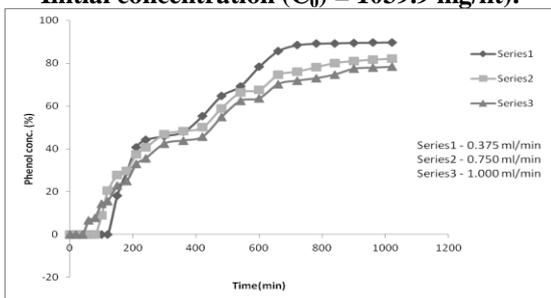


Fig.3. Effect of flow rate on breakthrough curve for phenol adsorption. (Bed Height = 13.5 cm, Amount of CFA= 10 gm, Initial concentration (C₀) = 1039.9 mg/lit).

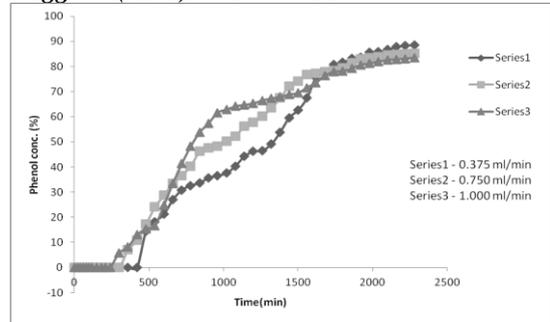


Fig 4. Effect of flow rate on breakthrough curve for phenol adsorption. (Bed Height = 27.5 cm, Amount of CFA= 20 gm, Initial concentration (C₀) = 1039.9 mg/lit).

It was observed from the figures 2, 3 and 4 that as the flow rate increases, the break point time decreases and the breakthrough curves become steeper. This occurs due to short residence time of phenol in the column for equilibrium adsorption is to be reached at high flow rate. So at high flow rate the phenol solution leaves the column before equilibrium occurs. Phenol adsorption on CFA at lower flow rate is more. At higher flow rate column gets saturated soon.

Table 2. Effect of flow rate on adsorption

Flow rate (ml/min)	Bed Height (cm)	q _{total} (mg)	M _{total} (gm)	% Phenol Removal	q _{eq(exp)} (mg/gm)
0.375	7.5	12.5241	187.18	6.69090	2.50483
0.75		22.7211	374.36	6.06925	4.54422
1.00		26.0803	499.15	5.22493	5.21607
0.375	13.5	22.8370	397.76	5.7414	2.28370
0.75		42.2909	795.52	5.31611	4.22909
1.00		52.7767	1060.6	4.97566	5.27767
0.375	27.5	38.6897	889.11	4.35149	1.93448
0.75		86.0946	1778.2	4.84159	4.30473
1.00		118.007	2370.9	4.97718	5.90003

As shown in table 2, as the flow rate increases, percent removal of phenol decreases for bed height 7.5 and 13.5 cm but increases for bed height 27.5 cm. The equilibrium phenol uptake increases for all the bed heights. This may happen because of more volume of phenol solution is in contact with the more adsorbents sites.

3.1.3 Effect of initial concentration

The effect of varying initial concentration 1039.9, 651.2, 292.7 and 70.0 mg/l on adsorption column for different bed height of 7.5, 13.5 and 27.5 cm are shown in following figures 5, 6 and 7.

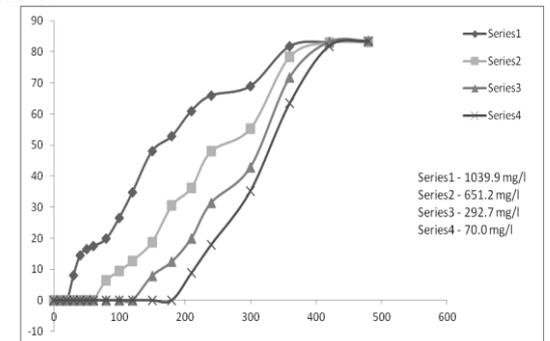


Fig 5. Combined breakthrough curve for phenol adsorption. (Bed Height = 7.5 cm, amount of CFA= 5 grams, Influent flow rate = 60.0 ml/ hr, Initial concentration (C₀) = 1039.9, 651.2, 292.7 & 70.0 mg/l).

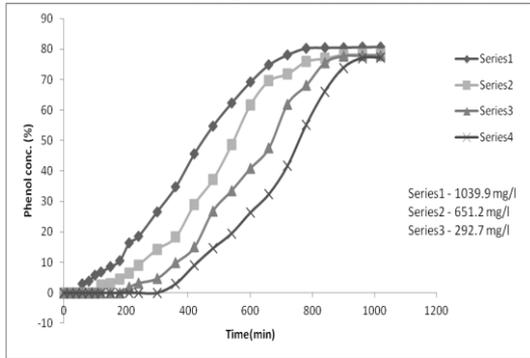


Fig 6. Combined breakthrough curve for phenol adsorption. (Bed Height = 13.5 cm, Amount of CFA= 10 grams, Influent flow rate = 60.0 ml/ hr, Initial concentration (C_0) = 1039.9, 651.2, 292.7 & 70.0 mg/l).

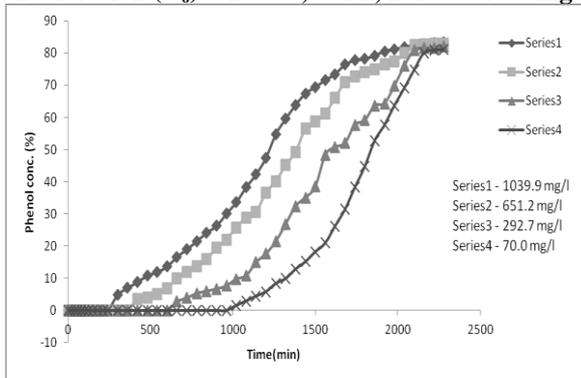


Fig 7. Combined breakthrough curve for phenol adsorption. (Bed Height = 27.5 cm, Amount of CFA= 20 grams, Influent flow rate = 60.0 ml/ hr, Initial concentration (C_0) = 1039.9, 651.2, 292.7 & 70.0 mg/l).

It was observed from the figures 5, 6 and 7 that as the initial phenol concentration increases, the break point time decreases. For large phenol concentration, steeper breakthrough curves are found, because of the lower mass-transfer difference from the phenol solution to the particle surface due to the weaker driving force. In addition at higher concentration, the isotherm gradient is lower, resulting in higher driving force along the adsorbent pores.

Table 3. Effect of initial phenol concentration on adsorption

Phenol Conc. (ml/lit)	Bed Height (cm)	q_{total} (mg)	M_{total} (gm)	% Phenol Removal	$q_{eq}(exp)$ (mg/gm)
70.0	7.5	14.3918	33.6	42.8329	2.87837
292.7		16.9936	140.496	12.0954	3.39872
651.2		20.8607	312.576	6.6738	4.17213
1039.9		26.7897	499.152	5.36704	5.35793
70.0	13.5	27.4135	71.4	38.3943	2.74135
292.7		34.8933	298.554	11.6874	3.48933
651.2		42.7972	664.224	6.44319	4.27972
1039.9		50.8495	1060.69	4.79396	5.08495
70.0	27.5	45.6222	159.6	28.5853	2.28111
292.7		64.0108	667.356	9.59170	3.20054
651.2		85.6434	1484.73	5.76825	4.28217
1039.9		99.7392	2370.972	4.206682	4.986963

The above table 3 indicates that with increase in initial phenol concentration, the adsorption capacity of the bed increased for all bed heights, but percentage phenol removal decreased with increase in concentration. This may happen because of the required driving force for adsorption increases with increase in concentration.

3.2 Modelling of the Breakthrough Curves

In this study, Thomas and Yoon-Nelson models were used for the prediction of dynamic behavior of the column.

3.2.1 Thomas and Yoon-Nelson Model Parameters for Effect of Bed Height

Table 4. Column kinetic parameters for Thomas model and Yoon-Nelson model obtained at different experimental conditions

Model	Parameters	Bed Height		
		7.5 cm	13.5 cm	27.5 cm
Thomas	K_{th} (ml/min.mg)	6.92374E-06	3.94269E-06	1.8271E-06
	q_0 (mg/gm)	3652.649	13692.44	21132.89
	R^2	0.8395	0.9256	0.8338
Yoon-Nelson	K_{YN} (min^{-1})	0.0072	0.0041	0.0019
	τ (min)	46.75	351.12	1083.84
	q_0 (mg/gm)	3.652648	13.69244	21.13289
	R^2	0.8398	0.9256	0.8338

As shown in table 4, for Thomas model, the value of q_0 increased and the value of K_{th} decreased with the increase in bed height. Similarly for Yoon-Nelson model, it was found that by increasing the bed height the value of K_{YN} decrease and the τ and q_0 increases. The value of q_0 for Yoon-Nelson model is nearer to experimental q_0 as compared to Thomas Model. These results are in good agreement with [13].

3.2.2 Thomas and Yoon-Nelson Model Parameters for Effect of Flow Rate

Table 5. Column kinetic parameters for Thomas model and Yoon-Nelson model obtained at different experimental conditions

MODEL	BED Height - 7.5 cm			
	Flow Rate (ml/min)	0.375	0.750	1.000
Thomas	K_{th} (ml/min.mg)	0.020	0.0112	0.0084
	q_0 (mg/gm)	2.100	18.36	44.58
	R^2	0.915	0.843	0.892
Yoon-Nelson	K_{YN} (min^{-1})	0.0077	0.0084	0.0084
	τ (min)	74.68	163.28	222.92
	q_0 (mg/gm)	5.82	25.47	46.36
	R^2	0.91	0.84	0.89
BED Height - 13.5 cm				
Thomas	K_{th} (ml/min.mg)	0.0109	0.0045	0.0037
	q_0 (mg/gm)	4.937	24.67	51.67
	R^2	0.925	0.890	0.885
Yoon-Nelson	K_{YN} (min^{-1})	0.0041	0.0034	0.0037
	τ (min)	351.12	438.67	516.78
	q_0 (mg/gm)	13.69	34.21	53.74
	R^2	0.92	0.89	0.88
BED Height - 27.5 cm				
Thomas	K_{th} (ml/min.mg)	0.0058	0.0025	0.0019
	q_0 (mg/gm)	8.304	31.75	54.19
	R^2	0.979	0.920	0.833
Yoon-Nelson	K_{YN} (min^{-1})	0.0022	0.0019	0.0019
	τ (min)	1181.1	1128.89	1083.8
	q_0 (mg/gm)	23.02	44.02	56.35
	R^2	0.97	0.92	0.83

As shown in table 5, with increase in flow rate for Thomas model, the value of q_0 increased and the value of K_{th} decreased. Similarly for Yoon-Nelson model, it was found that by increasing the flow rate the value of K_{YN} increases for the bed height 7.5 cm while it decreases for 13.5 and 27.5 cm bed height. The τ and q_0 values are increases with increase in flow rate.

3.2.3 Thomas and Yoon-Nelson Model Parameters for Effect of Initial Phenol Concentration

As shown in table 6, with increase in initial phenol concentration for Thomas model, the value of q_0 increased and

Table 6. Column kinetic parameters for Thomas model and Yoon-Nelson model obtained at different experimental conditions

MODEL	BED Height - 7.5 cm				
	Phenol conc. (mg/lit)	70.0	292.7	651.2	1039.9
Thomas	K_{th} (ml/min.mg)	0.000218571	4.50974E-05	1.7199E-05	8.46235E-06
	q_0 (mg/gm)	4804.28	18600.2	36523.0	45316.01
	R^2	0.9609	0.9653	0.9531	0.906
Yoon-Nelson	K_{YN} (min^{-1})	0.0153	0.0132	0.0112	0.008
	τ (min)	343.163	317.734	280.42	217.8
	q_0 (mg/gm)	4.804288	18.60020	36.5202	45.31601
	R^2	0.9609	0.9653	0.9531	0.9066
BED Height - 13.5 cm					
Thomas	K_{th} (ml/min.mg)	9.71429E-05	2.28903E-05	9.0602E-06	5.09664E-06
	q_0 (mg/gm)	5444.456	20558.46	41430.67	58204.970
	R^2	0.9647	0.9457	0.9244	0.9122
Yoon-Nelson	K_{YN} (min^{-1})	0.0068	0.0067	0.0059	0.0053
	τ (min)	777.77	702.37	636.22	559.717
	q_0 (mg/gm)	5.444456	20.55846	41.43067	58.204970
	R^2	0.9647	0.9457	0.9244	0.9122
BED Height - 27.5 cm					
Thomas	K_{th} (ml/min.mg)	6.14286E-05	1.09327E-05	4.29975E-06	2.30791E-06
	q_0 (mg/gm)	6530.267	24782.09	47214.33	67866.47
	R^2	0.9910	0.9891	0.9749	0.9595
Yoon-Nelson	K_{YN} (min^{-1})	0.0043	0.0032	0.0028	0.0024
	τ (min)	1865.791	1693.344	1450.071	1305.2500
	q_0 (mg/gm)	6.530269	24.78209	47.21431	67.866470
	R^2	0.9910	0.9891	0.9749	0.9595

the value of K_{th} decreased. Similarly for Yoon-Nelson model, it was found that by increasing the initial phenol concentration the value of K_{YN} and τ decreases while the value of q_0 increases.

4. Conclusion

The disposal problem of coal fly ash (CFA) has been resolved by using it as an adsorbent for removal of phenol from aqueous solution to some extent. The CFA as an adsorbent showed maximum adsorption capacity of 5.90 mg/gm. The effect of operating parameters such as bed height, flow rate and initial phenol concentration were investigated. The adsorption capacity increases with increase in bed height, flow rate and initial phenol concentration. The Thomas and Yoon-Nelson model was used for experimental data to predict the breakthrough curve and to determine the adsorption capacity of the column. The Yoon-Nelson model found the good agreement with the experimental data rather than Thomas model.

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