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Fixed-Bed Column Studies on Adsorption of 4-Nitroaniline from Aqueous Solution by Bagasse Fly Ash

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Abstract: *Sorptive removal of 4-nitroaniline from aqueous solutions by bagasse fly ash (BFA) was investigated at 303 K under dynamic conditions in a packed bed. The effects of sorbent bed height (Z = 5 - 15 cm), flow rate (Q = 10 - 30 mL/min), and initial concentration (C_0 = 50 -150 mg/L) for a constant bed diameter (D =2.54 cm) on the sorption characteristics of 4-nitroaniline were investigated at an influent pH of 6.5. The column performance improved with increasing Z and decreasing Q. The Thomas and Yoon-Nelson models were applied to the experimental data to represent the breakthrough curves and determine the characteristic design parameters of the column. The sorption performance of the BFA columns could be well described by the Thomas and Yoon - Nelson models at effluent-to-influent concentration ratios (C/C_0) higher than 0.08 and lower than 0.99. Both the models can be applied to describe the dynamic behavior of the column sorption with respect to bed height, concentrations and flow rates. The sorption capacity of the adsorbent was close to the value predicted from the models.*

Keywords– *Fixed bed adsorption, 4-nitroaniline, Break through curve, Adsorption models*

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I. Introduction

Among the aromatic compounds, 4-nitroaniline is the most important chemical that has been widely used as precursor in chemical synthesis of various azo dyes, antioxidants, pesticides, antiseptic agents, poultry medicines, fuel additives, and important corrosion inhibitors [1]. However, the presence of 4-nitroaniline in water, even at very low concentrations, is extremely harmful to aquatic life and human health in terms of its hematoxicity, splenotoxicity and nephrotoxicity [2, 3]. The chemical stability and toxicity of 4-nitroaniline make it hazardous [4]. The treatment and disposal of wastewater containing 4-nitroaniline have emerged as an important environmental concern. Furthermore, it shows toxicity, mutagenicity and carcinogenicity towards different experimental model organisms [5, 6]. Consequently, many developed and developing countries have enlisted 4-nitroaniline as priority pollutant and imposed restriction on its production, usage and disposal [1]. 4-nitroaniline metabolites are considered to be non biodegradable or only slowly degradable [7].

Many developed and developing countries have enlisted 4-nitroaniline as priority pollutant and imposed restrictions on its production, usage and disposal etc [1]. In consideration of the huge threat of 4-nitroaniline to the environment, this compound has been officially included in the black list of water environmental preferred controlled pollutants by Ministry of Environmental Protection of China in 1989 [8]. The discharge limit for 4-nitroaniline containing water is also very stringent in China, which is less than 1.0 mg/L in Chinese National Standard GB 8978–1996 [9]. The presence of a nitro group in the aromatic ring of 4-nitroaniline makes it resistant to chemical and biological oxidative degradation, while its anaerobic degradation produces nitroso and hydroxyl amine compounds that are carcinogenic[10]. Consequently, the effective removal of 4-nitroaniline in surface and drinking water is quite important for water quality improvement, and it is also a big challenge to environmental engineers [11]. Currently, the 4-nitroaniline containing wastewater is usually treated by photo-catalysis [12, 13], biodegradation [4, 14], advanced oxidation [1, 15] and adsorption [16, 17, 18 and 19]. Among these technologies, adsorption has been proven to be effective and convenient in separating 4-niroaniline from various water bodies. Different types of adsorbents have been investigated and utilized to remove 4-nitroaniline, such as activated carbon fiber [16], polymeric adsorbents [17, 18], and carbon nanotube [19]. Therefore, new cost effective technologies for the removal of

4-nitroaniline are crucial. Adsorption has been chosen as one of the most widely acceptable effective techniques to remove 4-nitroaniline and other organic pollutants at higher concentration due to its relatively simple design, cost effectiveness, ease of operation and simple adsorbent regeneration [18].

Bagasse fly ash (BFA) entails no cost, excepting its collection and transportation to the utility point. BFA is a waste material obtained from the particulate collection equipment attached downstream to the boilers/furnaces using bagasse as the fuel. Because of its excellent sorption characteristics, BFA has been used by several investigators [20, 21, 22] as an adsorbent for the removal of organics, dyes, phenols, etc.

Batch experiments are usually carried out to measure the effectiveness of adsorption for removing specific adsorbates as well as to determine the maximum adsorption capacity. The continuous adsorption in fixed-bed column is often desired from industrial point of view [23]. It is simple to operate and can be scaled-up from a laboratory process [24]. A continuous packed bed adsorber does not run under equilibrium conditions and the effect of flow condition (hydrodynamics) at any cross-section in the column affects the flow behavior downstream. The flow behavior and mass transfer aspects become peculiar beyond a particular length to diameter ratio of the column [25]. In order to design and operate fixed-bed adsorption process successfully, the breakthrough curves under specified operating conditions must be predictable. The shape of this curve is influenced by the individual transport process in the column and in the adsorbent [26]. Breakthrough determines bed height and the operating life span of the bed and regeneration times [27]. Adsorption in fixed-bed columns using activated carbon has been widely used in industrial processes for the removal of contaminants from aqueous industry effluents, since it does not require the addition of chemical compounds in the separation process [28].

The aim of the present work is to explore the possibility of utilizing BFA for the adsorptive removal of 4-nitroaniline in continuous mode using a packed bed column. In order to analyze the column dynamics in the adsorption process, the influence of the bed height (Z), inlet 4-nitroaniline concentration (C_o), and flow rate (Q) on breakthrough curves have been investigated. Breakthrough curve has been modeled using various equations to test the adequacy and accuracy of the model equations.

II. Materials And Methods

2.1 Adsorbent and its Characterization

Bagasse fly ash was obtained from Davangere Sugar Company Limited, Kukkawada, Karnataka, India. It was washed with hot water, dried and sieved using IS sieves (IS 437-1979). The mass fraction between −1000 and +180 was used for the sorption of 4-nitroaniline from the aqueous solution. The physico-chemical characteristics of BFA were determined using standard methods. Proximate analysis of BFA was carried out using the standard procedure (IS: 1350-1984, part-I). Bulk density was determined using a MAC bulk density meter. The specific surface area, total pore volume and mean pore diameter of the BFA particles were measured by nitrogen adsorption isotherm using Bellsorp II, Japan instrument by Brunaer–Emmett–Teller (BET) method, with Belsorp adsorption/desorption data analysis software-ver.6.4.0.0. Nitrogen was used as cold bath (77.15 K).

2.2 Adsorbate

4-nitroaniline procured from S.D. Fine chemical Limited, Mumbai, India, was used as adsorbate. Stock solution of 1000 mg/L was prepared by dissolving accurate quantity of 4-Nitroaniline. Solution of required concentration was prepared by diluting the previously prepared stock solution with distilled water whenever required.

2.3 Analytical Measurement

The concentration of 4-nitroaniline was determined by finding out the characteristic maximum absorbance wavelength using UV/VIS spectrophotometer (Schimadzu, UV-1800, Japan). A standard solution of known concentration was taken and the absorbance was determined at different wavelengths to obtain a plot of absorbance versus wavelength. The wavelength corresponding to maximum absorbance (λ_{max}) was determined. The λ_{max} for 4-nitroaniline is 381 nm. Calibration curve was plotted between the absorbance and the concentration of 4-nitroaniline. Linear portion of this curve was used for determining the unknown concentration of 4-nitroaniline solution.

2.4 Fixed Column Studies

A glass column of 2.54 cm ID, and 100 cm length was used for the column study. The column was packed with BFA followed by a layer of glass beads on both the sides to provide uniform flow of solution. To avoid entrapping of air bubbles inside the BFA the particles were soaked in appropriate amount of water and agitated until no air bubbles were observed in the solution. 4-nitroaniline of different concentrations (50, 100 and 150 mg/L) was percolated downward through the varying bed height (5, 10 and 15 cm) at a desired flow rate of 10, 20 and 30 mL/min. Initial pH of all the solutions was kept constant at its natural pH, ($pH_0 = 6.5$).

Samples were withdrawn from the bottom of the column at different time intervals. These samples were filtered and analyzed for the residual 4-nitroaniline concentration.

2.5 Analysis of Column Data

The time for breakthrough and the shape of the breakthrough curve are very important characteristics for determining the operation and the dynamic response of an adsorption fixed-bed column. The effluent volume was calculated by using equation (1).

$$V_{eff} = Qt_{total} \tag{1}$$

Where, V_{eff} is the effluent volume collected in mL, Q is the volumetric flow rate in mL/min and t_{total} is the total flow time in min. The maximum column bed capacity q_{total} in mg for a given inlet 4-nitroaniline concentration and flow rate was calculated by using equation (2).

$$q_{total} = \frac{QA_c}{1000} = \frac{Q}{1000}\int_{t=0}^{t=t_{total}} C_{ads}\,dt \tag{2}$$

Where, q_{total} is the maximum bed capacity in mg and C_{ads} is the adsorbed 4-nitroaniline concentration in mg/L. The plot of adsorbed 4-nitroaniline concentration (C_{ads}, mg/L) versus time (t, min) gives area under the breakthrough curve (A_c). The maximum adsorption capacity i.e. $q_{o,exp}$ at the exhaustion time was calculated by using equation (3)[29].

$$q_{o,exp} = \frac{q_{total}}{m} \tag{3}$$

Where, m is the amount of the adsorbent in grams packed in column.

2.6 Modeling of Column Data

In the present study, the Thomas model [30] and Yoon–Nelson model [31] were fitted to the dynamic flow experimental data performed in the fixed-bed column to predict the breakthrough curves and to determine the characteristic parameters of the column.

2.6.1The Thomas Model

This kinetic model was developed by Thomas. The Thomas model is one of the most general and widely used methods in column performance theory [32]. The expression by Thomas for an adsorption column is given by equation (4).

$$\ln\left(\frac{C_o}{C} - 1\right) = \frac{k_T q_o m}{Q} - k_T C_o t \tag{4}$$

Where C is effluent concentration (mg/L), C_0 is influent concentration (mg/L), k_T is Thomas rate constant (L/min mg), q_o is the maximum adsorption capacity (mg/g), m is mass of adsorbent (g), and Q is flow rate (mL/min). From the plot of ln (C/C_0-1) verses t, constants K_T and q can be determined.

2.6.2 Yoon-Nelson Model

This model is mathematically equivalent to the Thomas model and it has also been applied to a range of concentrations in the effluent between the breakthrough and saturation time of the column. This model is based on the assumption that the rate of decrease in the probability of adsorption for each adsorbate molecule is proportional to the probability of adsorbate adsorption and probability of adsorbate breakthrough on the adsorbent [33]. The linear form of Yoon - Nelson equation regarding a single-component system is given by equation (5).

$$\ln\left(\frac{C}{C_o - C}\right) = K_{YN} t - t_{0.5} K_{YN} \tag{5}$$

Where K_{YN} is the Yoon - Nelson rate constant, $t_{0.5}$ is the time required for 50 % sorbate breakthrough and t is the breakthrough (sampling) time. The parameters K_{YN} and $t_{0.5}$ may be determined from a plot of ln[$C/(C_0$-C)] versus sampling time (t). The maximum adsorption capacity i.e. $q_{o,exp}$ from Yoon - Nelson model was calculated by the equation (6).

$$q_{o\ YN} = \frac{C_o Q t_{0.5}}{m} \tag{6}$$

Where $t_{0.5}$ is the time required for 50 % sorbate breakthrough in minutes and Q is the volumetric flow rate through the column in mL/min, m is the mass of the adsorbent and C_o is the initial concentration of the the 4-nitroaniline in mg/L.

III. RESULTS AND DISCUSSION

3.1 Characterization of Adsorbent

The physico-chemical properties of BFA like, bulk density (mass per unit bed volume), BET surface area, total pore volume, mean pore diameter, moisture content, volatile matter, ash content, and fixed carbon were found to be 145.23 kg/m^3, 215.82 m^2/ g, 0.121 cm^2/g, 20.54 Å, 1.29%, 14.16%, 26.23%, and 58.32%, respectively.

3.2 Effect of Bed Height

In order to determine the effect of bed height on the breakthrough curve, 4-nitroaniline having influent concentration 50 mg/L and flow rate 20 mL/min was passed through the column by varying the bed height. The breakthrough curves obtained for 4-nitroaniline sorption onto BFA at different bed heights are shown in Fig. 1. From figure 1, it was observed that the breakthrough time varied greatly with bed height. The breakthrough time increased from 30 min to 210 min for BFA with increasing bed height from 5 to 15 cm. The mass of the sorbent forming the homogeneous fixed bed is proportional to the bed height and as a result the number of sorption sites increases with the increase in bed height leading to a larger sorption capacity [34]. The slope of the breakthrough curve decreased with increasing bed height which resulted in a broadened mass transfer zone. At lower adsorbent bed height, axial dispersion phenomenon predominated and reduced the diffusion of adsorbate [35]. Higher uptake was observed at the highest bed height due to an increase in the surface area of the sorbent, which provided more binding sites for the sorption [36]. As the bed height was increased, the adsorbate molecules had more time to contact with the adsorbent that resulted in higher 4-nitoaniline removal efficiency.

3.3 Effect of Flow Rate

Flow rate is an important parameter in evaluating the performance of a sorption process, particularly for continuous treatment of wastewater on industrial scale [37]. Therefore, the effect of flow rate on sorption of 4-nitoaniline by BFA was studied by varying the flow rates from 10, 20 and 30 mL /min and keeping the initial 4-nitroaniline concentration 50 mg/L and bed height (5 cm) constant. The effect of flow rate on breakthrough performance at the above operating condition is shown in Fig. 2. From figure 2, it was observed that, the breakthrough time as well as the sorption efficiency of BFA was lower at higher flow rate. As the flow rate increased from 10 to 30 mL/min, the breakthrough time decreased from 90 to 30 minutes. This is due to the fact that at higher flow rate the contact time of the 4-nitroaniline with the sorbent surface was very short and hence the 4-nitroaniline molecules did not have enough time to capture the binding sites on the sorbent surface or diffuse into the pores of the sorbent, leaving the column before equilibrium occurs [23]. However, the breakthrough curve became steeper when the flow rate was increased with which the breakthrough time and the 4-nitroaniline removal efficiency decreased. But, when the flow rates were low, the external mass transfer controlled the process and it was also ideal for intra particle diffusion system. Thus, at lower flow rates, the more effective was the diffusion process and higher was the residence time of the sorbate, which ultimately resulted in high sorption capacity [36].

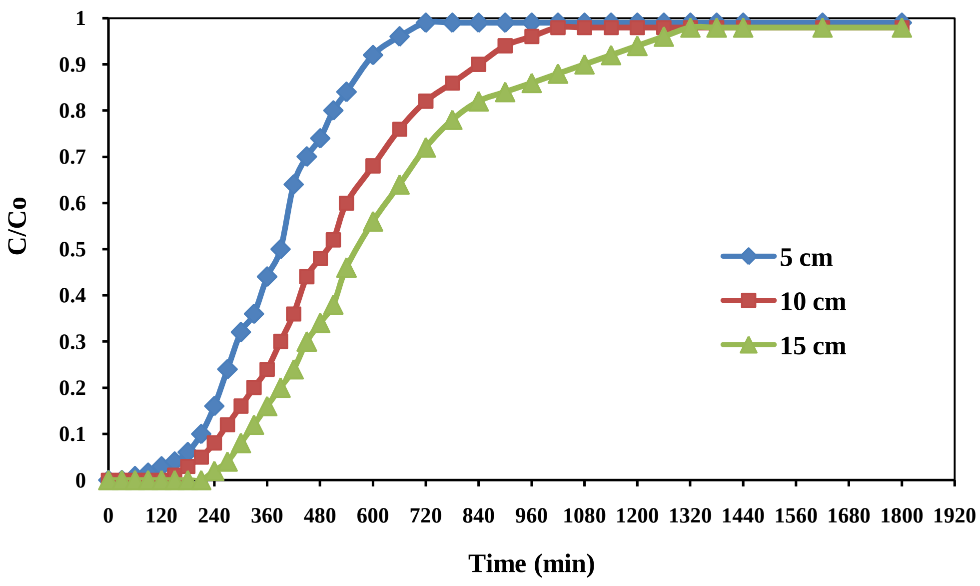

Fig. 1 Breakthrough Curves for 4-nitroaniline Sorption onto BFA at Different Bed Heights. (Flow Rate = 20 mL/ min, Concentration = 50 mg/L).

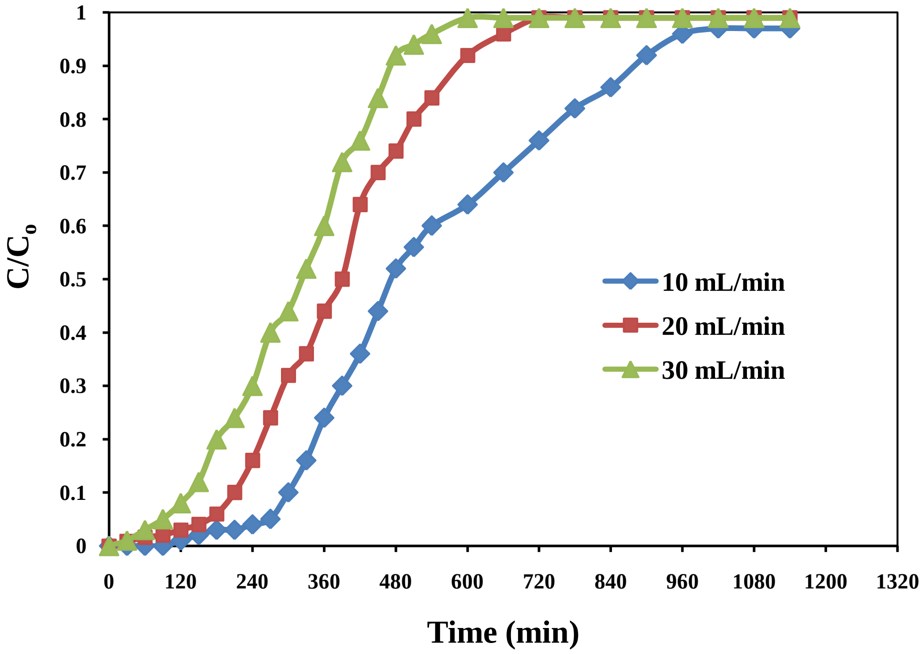

Fig. 2 Breakthrough Curves for 4-nitroaniline Sorption onto BFA at Different Flow Rates. (Bed Height = 5 cm, Concentration = 50 mg/L).

3.4 Effect of Initial Concentration

The effect of initial concentration of 4-nitroaniline (50, 100 and 150 mg/L) on the sorption by BFA at a constant bed height 5 cm and flow rate 20 mL/min is shown in Fig. 3.

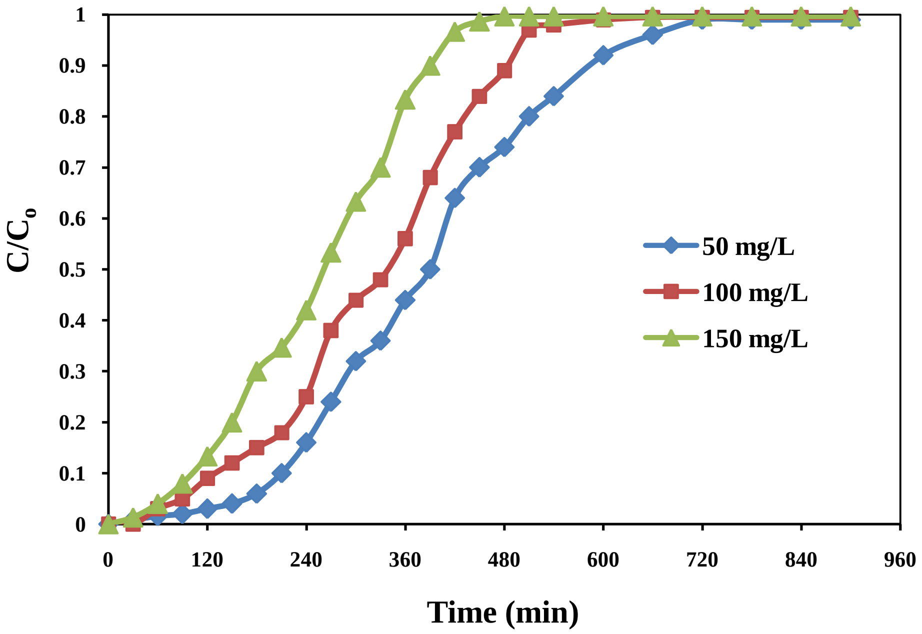

Fig. 3 Breakthrough Curves for 4-nitroaniline Sorption onto BFA at Different Initial Concentration. (Bed Height = 5 cm, Flow Rate = 20 mL/min).

From figure 3, it was observed that, as the initial 4-nitroaniline concentration increased from 50 to 150 mg/L, the breakthrough and exhaustion time decreased considerably. In the sorption of 4-nitroaniline by BFA, the breakthrough time decreased from 60 to 30 minutes and exhaustion time decreased from 720 to 480 minutes, This is due to the fact that higher concentration of the solute led to quick saturation of the sorbent, which in turn leads to the earlier breakthrough and exhaustion time [38].

The various parameters as described above viz. total amount of 4-nitroaniline adsorbed (q_{total}) together with the exhaustion times, q_o experimental and $t_{0.5}$ (the time required for 50% sorbate breakthrough) are presented in Table 1.

Table 1. Column Data Parameters obtained at Different Inlet 4-Nitroaniline Concentrations, Bed Heights and Flow Rates (T=303 K).

C_o (mg/L)	Q (mL/min)	D (cm)	Z (cm)	q_{total} (mg)	$q_{o\ exp}$ (mg/g)	$t_{0.5\ (exp)}$ (min)
50	20	2.54	5	53.17	35.44	390
50	20	2.54	10	115.77	38.59	510
50	20	2.54	15	185.82	41.29	600
50	10	2.54	5	59.74	38.54	480
50	30	2.54	5	52.16	33.65	330
100	20	2.54	5	62.70	40.45	350
150	20	2.54	5	63.53	40.99	270

3.5 Application of the Thomas Model

The Thomas model was applied to the data for $0.008 < C/C_0 < 0.99$ with respect to different bed height, flow rate and concentration. A linear plot of $\ln[(C_0/C) - 1]$ versus time t (min) enabled the determination of the kinetic coefficient k_{TH} and the sorption capacity of the bed q_0 according to equation (4). The slope and intercepts obtained from the linear regression performed on each set of transformed data provides the different coefficients. The k_{TH} and q_o values are reported in Table 2. From Table 2, it was observed that as the bed height was increased the value of q_o increased from 34.12 to 40.93 mg/g, but the value of k_{TH} decreased. As the inlet concentration increased the values of q_o increase and the values of k_{TH} decreased. The reason was that the driving force for adsorption was the concentration difference between the adsorbate on the adsorbent and the adsorbate in the solution [39, 40]. With flow rate increasing, the value of q_o decreased from 36.29 to 32.00 mg/g, but the value of k_{TH} increased. So lower flow rate, lower initial concentration and higher bed heights favored increase in the adsorption of 4-nitroaniline on the BFA loaded column. The Thomas model was suitable for adsorption processes where the external and internal diffusions will not be the limiting steps [39]. The column adsorption capacity calculated based on Thomas model and experimental q_o value are in close agreement. The values of coefficient of determination R^2 ranging from 0.970 to 0.988 indicate and that the experimental data fitted well with this model.

Table 2. Thomas Model Parameters at Different Conditions for 4-Nitroaniline Sorption on BFA using Linear Regression Analysis.

C_o (mg/L)	Q (mL/min)	D (cm)	Z (cm)	k_{TH} (L/mg min)	q_o (mg/g)	R^2
50	20	2.54	5	0.00026	34.12	0.986
50	20	2.54	10	0.00022	37.53	0.984
50	20	2.54	15	0.00016	40.93	0.986
50	10	2.54	5	0.00016	36.29	0.981
50	30	2.54	5	0.00028	32.00	0.986
100	20	2.54	5	0.00015	36.89	0.970
150	20	2.54	5	0.00012	37.78	0.988

3.6 Application of the Yoon - Nelson Model

The values of k_{YN} (a rate constant) and $t_{0.5}$ (the time required for 50% sorbate breakthrough) were determined from plots of $\ln[C/(C_0- C)]$ versus $t_{0.5}$ at different bed height, flow rate and concentrations. These values are listed in Table 3. From Table 3, the rate constant k_{YN} increased and the 50% breakthrough time $t_{0.5}$ decreased with increasing both inlet concentration and flow rate. As the bed height increased from 5 to 15 cm, the values of $t_{0.5}$ increased while the values of k_{YN} decreased drastically. The column adsorption capacity calculated based on Yoon - Nelson model and experimental q_o values are comparable. The Yoon - Nelson model gave values of the time for the 50% breakthrough point that were comparable to the experimental time. The values of coefficient of determination R^2 ranging from 0.970 to 0.992 indicate and that the experimental data fitted well with this model. In a comparison of values of R^2 and breakthrough curves, both the Thomas and Yoon - Nelson models can be used to describe the behavior of the adsorption of 4-nitroaniline in a fixed-bed column loaded with BFA.

Table 3. Yoon - Nelson Model Parameters at Different Conditions for 4-Nitroaniline Sorption on BFA using Linear Regression Analysis.

C_o (mg/L)	Q (mL/min)	D (cm)	Z (cm)	$t_{0.5\ (cal)}$ (min)	k_{YN} (min^{-1})	$q_{0\ YN}$ (mg/g)	R^2
50	20	2.54	5	371.38	0.00026	33.76	0.992
50	20	2.54	10	517.80	0.00020	34.52	0.983
50	20	2.54	15	598.25	0.00016	39.88	0.961
50	10	2.54	5	475.20	0.00020	31.68	0.981
50	30	2.54	5	325.57	0.00028	29.60	0.979
100	20	2.54	5	361.67	0.00015	38.07	0.970
150	20	2.54	5	276.28	0.00012	39.47	0.975

IV. CONCLUSIONS

Sorption of 4-nitroaniline from aqueous solution was investigated at 303 K in a continuous packed column of BFA. The effects of sorbent bed height (Z), flow rate (Q), and initial concentration of 4-nitroaniline (C_0) on the sorption capacity and breakthrough characteristics were investigated. It was found that an increase in bed height and a decrease in flow rate improved the sorption performance. Thomas and Yoon - Nelson models reported in the literature were tested for their validity to explain the sorption data. Model constants were determined by linear regression for use in the column design. The bed sorption capacity q_o determined from both the model is comparable with the experimental q_o values. The Yoon - Nelson model gave values of the time for the 50% breakthrough point that were comparable to the experimental time. Both Thomas and Yoon - Nelson models were found to be suitable for describing the dynamic behavior of the whole or a definite part of the column with respect to all Z, Q and C_o values.

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