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ADSORPTION PERFORMANCE OF FIXED-BED COLUMNS FOR THE REMOVAL OF PHENOL USING BAOBAB FRUIT SHELL BASED ACTIVATED CARBON

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ABSTRACT: A continuous adsorption study in a fixed-bed column using baobab fruit shell activated carbon (BF-AC) was investigated for phenol removal from an aqueous solution. Baobab fruit shell (BFS) was chemically activated using potassium hydroxide (KOH) at 700 °C in a nitrogen (N_2) atmosphere. Scanning electron microscope (SEM), X-ray diffraction (XRD), and BET surface area analyses were performed for the characterization of BF-AC. Fixed-bed experiments were carried out and the effect of feed flowrate (10, 15, 20 mL/min) and bed height (5, 10, 15 cm) on the adsorption were investigated by evaluating the breakthrough curves. BET surface area of BF-AC was 1263 m^2/g, indicating its well-developed pores and its good quality as an adsorbent. The findings showed that the exhaustion time (t_δ) and breakthrough time (t_b) reduced as the flowrate augmented, while they increased as the bed height augmented. With the increase in the bed height and the flowrate, phenol solution volume treated was augmented. Also, BF-AC with bed height of 15 cm provided better elimination of phenol with carbon usage rate (CUR) of 1.74 g/L and empty bed contact time (EBCT) of 9.9 minutes. According to the findings, BF-AC is an effective adsorbent for removing phenol from aqueous solutions.

ABSTRAK: Kajian penjerapan berterusan menggunakan kulit buah baobab diaktifkan karbon (BF-AC) telah dikaji mengguna pakai kolum lapisan tetap bagi penyingkiran fenol daripada larutan cecair. Kulit buah Baobab (BFS) diaktifkan secara kimia menggunakan kalium hidroksida (KOH) pada suhu 700 °C dalam atmosfera nitrogen (N_2). Imbasan mikroskop elektron (SEM), pembelahan sinar-X (XRD, dan analisis permukaan BET dijalankan bagi pencirian BF-AC. Eksperimen kolum lapisan tetap bagi mengkaji kesan penjerapan pada aliran suapan (10, 15, 20 mL/min) dengan ketinggian (5, 10, 15 cm) dinilai melalui lengkung bulus. Kawasan permukaan BET BF-AC adalah 1263 m^2/g, menunjukkan liang yang elok terbentuk dan berkualiti baik sebagai penyerap. Penemuan ini menunjukkan bahawa puncak masa maksima (t_δ) dan masa terbaik (t_b) berkurangan pada kadar aliran bertambah, sebaliknya ianya meningkat pada ketinggian bertambah. Dengan penambahan ketinggian katil dan kadar aliran, jumlah larutan fenol yang dirawat telah bertambah. Selain itu, BF-AC pada ketinggian 15 cm menunjukkan penghapusan fenol terbaik pada kadar penggunaan karbon (CUR) 1.74 g/L dan masa sentuhan kolum kosong (EBCT) 9.9 minit. Ini menunjukkan, BF-AC adalah penyerap yang berkesan bagi menyingkirkan fenol daripada larutan cecair.

KEYWORDS: *adsorption; Baobab fruit shell; breakthrough curves; fixed-bed column; phenol*

1. INTRODUCTION

Phenol is regarded as an environmental problem owing to its discharge in wastewater from a variety of industries, including petroleum refineries, petrochemicals, pharmaceuticals, pesticide manufacturing, resins, steel mills, coke manufacturing, paints, dye production, and mine discharge [1]. Currently, about 7.8 million tons of phenol are produced annually around the world, with production rates on the rise [2]. Phenol has been classified as a hazardous substance due to the potential danger it poses when it comes into contact with living organisms. United States Environmental Protection Agency (USEPA) has designated phenol as a priority pollutant due to its toxicity, with an allowed limit of 0.001 mg/mL and 0.1 mg/L in water supplies and wastewater, respectively [3].

Several methods have been used to remove phenol from aqueous solutions, involving ion-exchange [4], distillation [5], adsorption [6], chemical oxidation [7], extraction [8], coagulation [9], bio-degradation [10], and electro-chemical oxidation [11]. Adsorption has been shown in numerous studies to be a highly effective method for removing phenol from wastewater, and activated carbon (AC) has been frequently used as an adsorbent [3]. This method attracted a lot of attention because of its benefits, including ease of use, superior design flexibility, selectivity, high performance, no need for secondary purification, and the absence of undesirable by-product production [12,13]. Commercially available AC is an established and widespread adsorbent for the removal of various pollutants [14]. Despite its extensive use, AC remains an expensive material [1]. Moreover, saturated-AC regeneration is very tedious, difficult, and expensive [14]. This necessitates finding and developing inexpensive adsorbents from inexpensive and abundant raw materials.

Using lignocellulosic biomass as a raw material for the production of AC provides an alternative to conventional sources and is considered an effective way to discard massive waste quantities and reduce the cost of AC production as well. Lignocellulosic biomasses are cheap, plentiful, renewable, non-toxic, easily accessible, and eco-friendly [1]. Many lignocellulosic biomasses have been utilized as precursors to produce AC for phenol removal from wastewater. However, these studies were limited to fixed bed columns. Only a few studies have been carried out on fixed-bed investigations. This could be due to the restriction caused by a significant lack of an adsorbent material in substantial quantity [15]. Lignocellulosic biomasses that have been utilized to remove the phenol from wastewater in fixed-bed are *Lantana camara* [16], sugarcane bagasse [17], date palm [18], fox nutshell [19], corn cob [20], neem leaves [21], and rice husk [22]. These biomasses offer an alternative to traditional sources, which may serve as potential precursors to produce AC. As a result, to find a new renewable alternative source for AC, efforts have been made to produce AC from baobab fruit shell (BFS) by chemical activation [1,23-25].

Baobab fruit shell is a lignocellulosic residue of the baobab fruit, which is one of the most abundant agriculture wastes with an annual average amount of 30,285 tons [26]. BF-AC was utilized as an adsorbent for the removal of environmental pollutants, including heavy-metals [27,28], dyes [24,29], and phenolic compounds [1,23,25]. In order to remove the phenol from aqueous solutions, batch studies of the potential BF-AC have been conducted [1]. Phenol adsorption on BF-ACs adsorbents produced by chemical activation utilizing $ZnCl_2$, KOH, and H_3PO_4 has also been reported [1]. The findings showed that the phenol adsorption rate by BF-ACs was rapid, and that AC treated with KOH could absorb more than 95% of phenol. The optimization of phenol removal on BF-AC has also been studied [23]. The results demonstrated that BF-AC has high efficiency in the removal of phenol from aqueous solutions with an adsorption capacity of 196.86 mg/g [3,23]. The earlier studies were confined to batch studies only [1,3,23,25]. However, understanding the column mode adsorption pattern is necessary to suggest potential applications in the field.

However, the goal of the current research is to examine the ability of the BF-AC for the removal of phenol in a continuous fixed-bed adsorption column. Continuous flow conditions are regarded as necessary and effective in large-scale industrial wastewater treatment owing to their simplicity, convenient operation, handling, and regeneration capacity [30]. The current study investigated the effects of some operating factors on adsorption, including flowrate and bed height. Prior to using the BF-AC as an adsorbent, SEM, BET surface area, and XRD analyses were performed to characterize the material.

2. METHODOLOGY

2.1 Materials

In the current work, baobab fruit has been obtained from Sudan, at the end of the rainy season. Potassium hydroxide (KOH), hydrochloric acid (HCl), and phenol (C_6H_5OH) were acquired from Sigma-Aldrich (Malaysia). Nitrogen gas (99.95 %) was purchased from Fuelink Marketing Sdn. Bhd. (Selangor, Malaysia).

2.2 Methods

2.2.1 Adsorbent Preparation

In the current work, baobab fruit was obtained from Sudan, at the end of the rainy season. Baobab fruit shells (BFS) were washed several times with distilled water. Wet BFS were dried in the oven before being milled and sieved into fine particles (< 1 mm). An amount of KOH was added to crushed BFS using a 1:2 impregnation ratio (IR) (BFS:KOH). Distilled water was added to the mixture in an amount equal to four times the total quantity of BFS and KOH mixture. The sample was stirred at 50 °C for 1 hr and then inserted in an oven at 100 °C and left overnight. Nitrogen gas (N_2) was utilized for purging a quartz tube a few minutes before carbonization began. N_2 was kept in place throughout the activation and cooling processes. Sample carbonization was carried out for 1 hr at 700 °C. The product was placed in a desiccator to cool, then neutralized with a HCl solution (0.5 M, 50 mL), followed by a wash with warm distilled water (70 °C) until a constant pH of the washing solution was reached. BF-AC produced was dried in the oven at 110 °C.

2.2.2 Characterization of BF-AC

The morphological structure of powdered BF-AC and its structural alterations that resulted from the chemical activation process were both seen using SEM (JEOL-IT 100). For X-ray Diffraction (XRD) analysis, a Bruker D2 phaser (Bruker AXS, Germany) was utilized. The porosity of the BF-AC was assessed using Quanta-chrome, Autosorb-1C, and a surface area analyzer in accordance with Brunauer-Emmett-Teller (BET) model through physical adsorption of N_2 (at 77 k).

2.2.3 Adsorbate Preparation

An appreciable amount of phenol was dissolved in distilled water to prepare the stock solution of adsorbate (phenol) with a concentration of 150 mg/L.

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2.2.4 Fixed-Bed Column Adsorption Studies

Column experiments were conducted utilizing a stainless-steel tube of 29 mm in diameter and 300 mm in height. The phenol solution was stored in a storage tank, where the tank had retained a steady depth of pollutant by recharging the tank to retain the pressure of the solution in the tank. The adsorbent was immersed in distilled water, then rinsed and poured several times for the removal of the fine particles. BF-AC was dried overnight at 110 °C and known quantities of activated carbon were placed into the column at various heights (5, 10, and 15 cm), which were measured before the tests. The columns were filled with distilled water, then preserved and immersed before filling the column with the phenol. Afterward, phenol solutions of 150 mg/L were pumped through the column bed, which would lower bed utilization as explicated using a peristaltic pump at various flowrates of 15, 10, and 20 mL/min [16]. Activated carbon weight was determined after it was filled into the column to the required height. Finally, the bed exhaustion time was determined by collecting treated phenol samples from the outlet at time intervals and measuring the remaining phenol. The flowrate was kept constant with a control valve at constant rates. Samples were collected from the collection port in the column at different time intervals. Residual concentrations of phenol were measured at 270 nm wavelength using a spectrophotometer. The experimental setup from this study of the column is shown in the schematic diagram (Fig. 1). Table 1 illustrates the range of each parameter that was utilized for this study. Each parameter used was specified depending on the information obtained in the literature review [16].

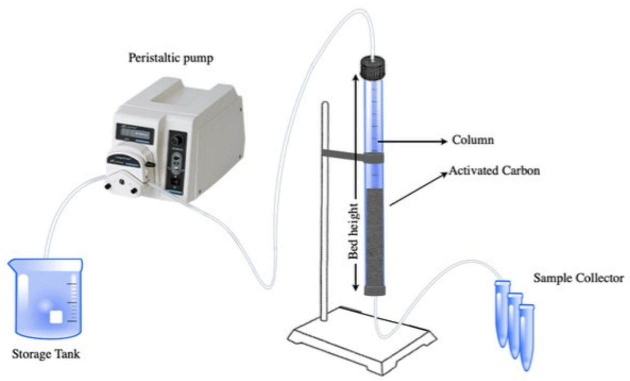


Fig. 1: Bed column experimental setup.

Table 1: Column operating parameters and conditions

Parameters	Variations
Column bed depth (cm)	30
Column diameter (cm)	2.9
Flowrate (mL/min)	10, 15, 20
Bed height (cm)	5, 10, 15
BF-AC mass (g)	5.5, 11, 16.5
Initial concentration of phenol (mg/L)	150

2.2.3 Fixed-Bed Adsorption Column Analysis

A: Columns Effluent Analysis

The concentration of residual phenol was utilized to determine the breakthrough curve graph by plotting the fractional ratio (Eq. 1) versus time.

$$f = \frac{C}{C_0} \tag{1}$$

where C_0 is the initial concentration of phenol and C is the residual concentration of phenol.

B: Performance Evaluation of Adsorbent

The breakthrough curve was plotted to determine the time needed to reach the breakthrough point (t_b) and the time needed to reach the exhaust point (t_x) using Eq. 2 and Eq. 3, respectively. The time for phenol to exhaust the mass transfer zone/active adsorption zone (t_δ) was determined using Eq. 4.

$$t_b = \frac{V_b}{Q} \tag{2}$$

$$t_x = \frac{V_x}{Q} \tag{3}$$

$$t_\delta = \frac{(V_x - V_b)}{Q} \tag{4}$$

Also, the specific throughput was determined with Eq. 5 to evaluate the BF-AC performance in the column. A water quantity that might be processed by BF-AC in liters/g was determined from a specified throughput and compared at a breakthrough time (t_b) and at exhaustion (t_x).

A specific throughput and the empty bed contact time (EBCT) were determined using the following equations:

$$Specific\ throughput = \frac{Q t_b}{M} \tag{5}$$

$$EBCT = \frac{V_f}{Q} = \frac{L.A_F}{v.A_F} = \frac{L}{\mathrm{v}} \tag{6}$$

where EBCT is the contact time in the empty bed (h), V_f is the volume occupied by BF-AC media including porosity volume (m^3), Q is the flowrate to adsorber (L/hr), A_F is the BF-AC area available for flow (m^2), L is the BF-AC depth (m), and v is the superficial flow velocity (Q/A_F) in (m/h).

Equation 7 was used to get the carbon use rate (CUR), which is the amount of BF-AC consumed throughout the treatment process (g/L).

$$CUR = \frac{M}{Q\ t_b} = \frac{1}{specific\ throughtput} \tag{7}$$

The length of the mass transfer zone (MTZ) is typically a function of the hydraulic loading rate applied to the column and the characteristics of the adsorbent. If the loading rate is too great, the height of the MTZ will be larger than the AC bed depth, which results in pollutants not being completely removed by the column. Equation 8 was used to determine a MTZ (H_{MTZ}) height [31].

$$\mathrm{H}_{MTZ} = \mathrm{Z}\left[\frac{(V_E - V_B)}{V_E - f(V_E - V_B)}\right] \tag{8}$$

The bed column parameters, as well as its unit and formula, are presented in Table 2.

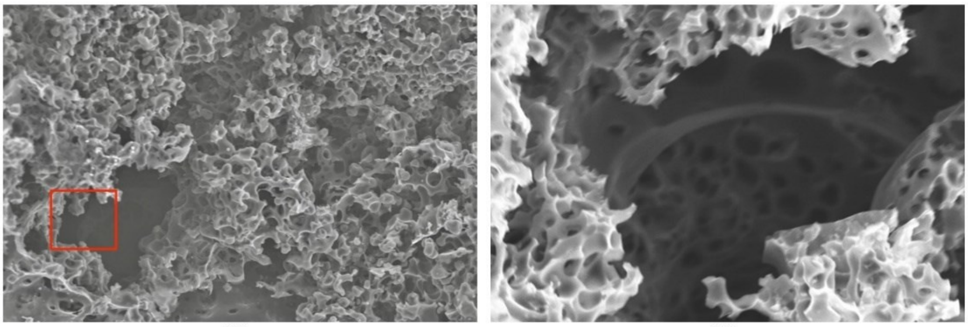
Table 2: Details fixed-bed column parameters

Packed bed column parameters	Formula	Unit
Volume at breakthrough *(V_b)*	$V_b = \frac{t_b}{Q}$	mL
Volume at exhaustion point *(V_x)*	$V_x = \frac{t_x}{Q}$	mL
Breakthrough time (t_b)	$t_b = \frac{V_b}{Q}$	min
Exhaustion time (t_x)	$t_x = \frac{V_x}{Q}$	min
Time taken to exhaust the mass transfer zone (t_δ)	$t_\delta = \frac{(V_x - V_b)}{Q}$	min
Height of Mass Transfer Zone (H_{MTZ})	$H_{MTZ} = Z\left[\frac{(V_E - V_B)}{V_E - f(V_E - V_B)}\right]$	cm
Carbon Usage Rate (CUR)	$CUR = \frac{M}{Q\,t_b} = \frac{1}{specific\ throughtput}$	g/L
Specific throughput	$Specific\ throughput = \frac{Qt_b}{M}$	mL/g
Empty bed contact time (EBCT)	$EBCT = \frac{V_f}{Q} = \frac{L.A_F}{v.A_F} = \frac{L}{\mathrm{v}}$	min

3. RESULTS AND DISCUSSION

3.1 BF-AC Characterization

For observing the physical surface morphology of BF-AC, the SEM technique was used. Figure 2 displays SEM micrographs of BF-AC produced at 1000 and 2500 magnifications. SEM micrographs show that the AC surface is highly porous with many irregular-shaped uneven pores. Higher porosity results in more surface area, which facilitates greater adsorbate removal from its aqueous solution [1]. The details are also described in our previous studies [3,25].



(a) (b)

Fig. 2: SEM images of produced BF-AC magnification of (a) x1000, (b) x2500.

XRD analysis was performed in order to determine the crystalline composition of the prepared BF-AC. Figure 3 presents the XRD profile of BF-AC. The lack of a clearly defined peak for any aspect of the diffraction profile, as seen by the XRD spectrum, suggests that the BF-AC were not subjected to any mineral peaks. Similar findings and conclusions have been reached by several investigations [27,29].

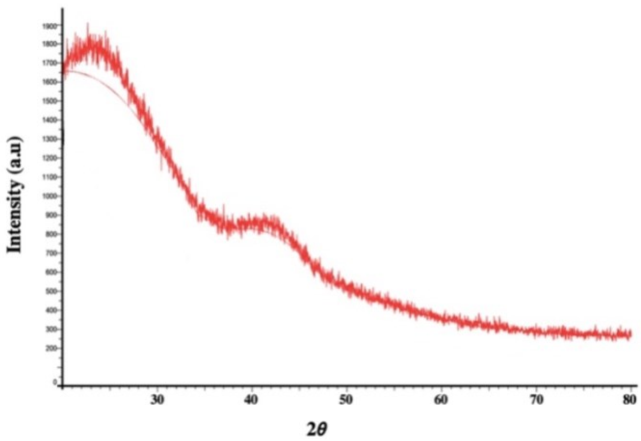


Fig. 3: X-Ray Diffraction pattern of BF-AC.

N_2 adsorption−desorption isotherms of produced BF-AC are displayed in Fig. 4. The adsorption isotherm is classified into Type I isotherm, which is microporous adsorbents (containing pores < 2 nm), according to IUPAC classification approach [32]. The findings confirmed the microporosity of BF-AC as indicated in Table 3 and are consistent with the pore size distribution curve.

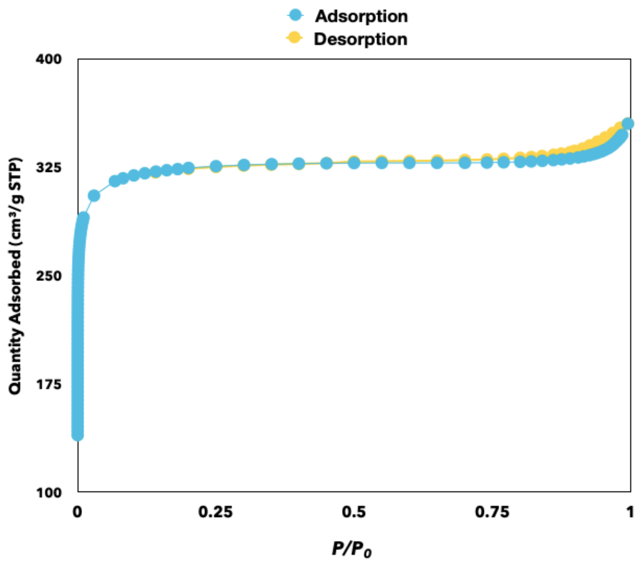


Fig. 4: N_2 adsorption−desorption isotherms of produced BF-AC.

Table 3 shows the pore volume, pore diameter, and BET surface area of BF-AC. The findings showed that BF-AC has a high surface area (1263 m^2/g). With a particular micropore area of 1152 m^2/g, a micropore volume of 0.453 cm^3/g, an exterior surface area of 111.24 m^2/g, and pore diameter of 1.74 nm, the BF-AC is clearly highly developed.

Table 3: The surface area and pore distribution of BF-AC.

Sample	BET specific surface area (m^2/g)	Micropores area (m^2/g)	External surface area (m^2/g)	Average pore diameter (nm)	Total pore volume (cm^3/g)	Volume of micropores (cm^3/g)
BF-AC	1263. 127	1152.886	111.24	1.739	0.549	0.4531

3.2 Fixed-Bed Adsorption Column Analysis

The experiments were carried out with varied bed heights (5, 10, 15 cm), varied flow rates (10, 15, 20 mL/min), and phenol concentration of 150 mg/L (evaluated by batch mode in our earlier study [3]. According to the experimental results, the corresponding breakthrough curves (C_t/C_0) vs *t* were plotted for each observation.

3.2.1 Effect of Flowrate

The flowrate is a significant factor in the packed bed column adsorption process, which helps to predict the efficiency of the BF-AC. The breakthrough curves were investigated at various flowrates. The effects of flowrate on the phenol removal by BF-AC were investigated at different flowrates (10, 15 and 20 mL/min), whereas the phenol initial concentration and bed depth were kept constant at 150 mg/L and 5 cm, respectively. To achieve a bed height of 5 cm, 5.5 g BF-AC were poured into the column. The results obtained from the experiments were utilized for constructing the breakthrough curves (C/C_0 vs time) for 5, 10, and 20 mL/min flowrates (Fig. 5).

From the breakthrough graphs, it is evident that as the flowrate increased, the breakthrough curves became steeper. The adsorbate residence time in the column decreased as the flowrate increased, which had an impact on how quickly the phenol aqueous solution left the column. This phenomenon could explain the pattern observed in the graph. Consequently, the BF-AC column effective adsorption capacity decreased, because the adsorbate solvents in the column did not have enough time to disseminate the solute into the pores of the adsorbent [33]. According to the results presented in Table 4, it can be noted that the exhaustion time (t_δ) and breakthrough time (t_b) declined as the flowrate augmented [15,16,34]. The mass transfer zone length increased with the rise in flowrate, leading to quicker column saturation [16]. The best results at a low flowrate of 10 mL/min can be inferred. Other researchers reported similar findings [16,34].

By comparing the various flowrates, it becomes evident that the flowrate of 20 mL/min treated the highest volume, which was 6745 mL followed by the flowrate of 15 mL/min which was 5590 mL, followed by the flowrate of 10 mL/min with 4200 mL. So, the treated volume increased as the flow rate increased. During the column adsorption process, MTZ normally transfers from the influent end to the effluent end of the adsorbed bed. It means that the region of active adsorbent shifts to the effluent-end of the bed where the adsorbate has yet to be saturated, and this occurs after the adsorbent near the influent becomes saturated [35]. The low elimination of contaminants is done when the MTZ height is greater than the bed height [35,36]. From the results, the MTZ of the three different columns was higher than the bed height of 5 cm. It was also observed that MTZ decreased with increased flowrate. Similar remarks have been reported recently by Patel [37].

The rate of use of the adsorbent is often reported as a carbon use rate (CUR), which is an essential factor when column studies are scaled. Another significant parameter when the column activity is performed is the EBCT, which is the volume of the bed occupied by the adsorbent divided by the flowrate. According to the findings, the EBCT decreased as the flowrate rose. Similar observations were recently reported by Muthamilselvi et al. [14].

3.2.2 Effect of Bed Height

The effects of bed height on the phenol removal by BF-AC were also investigated in the range of 5, 10 and 15 cm, while the inlet of the phenol concentration and flowrate were kept constant at 150 mg/L and 20 mL/min, respectively. Figure 6 displays the graphs of phenol concentration vs time at various bed heights. The length of the bed (in which the phenol aqueous solution flows) increases as the bed height increases. Comparing the various bed heights, the column bed height of 15 cm recorded 12296 mL of the treated volume, which was the highest volume treated. This obviously presented a higher capacity with 15 cm bed height. This was because, with a rising bed height, further binding sites were available for the adsorbate to diffuse through the pores of the adsorbent and as a consequence, the area of adsorption rose [16,30]. It was clearly observed that the breakthrough time (t_b) and exhaustion time (t_x) as the bed height augmented (Table 5). Other researchers have reported similar results [16,38].

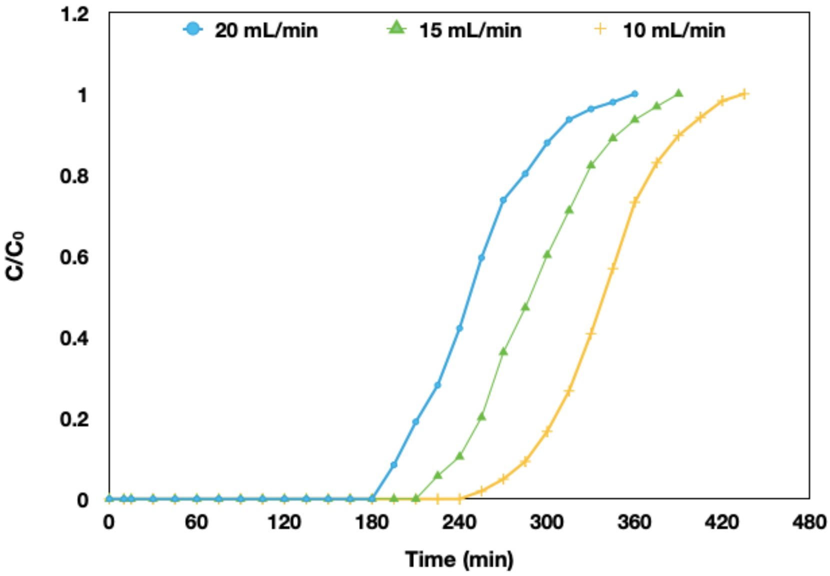


Fig. 5: Phenol adsorption breakthrough curves at various flowrates (10,15 and 20 mL/min), 5 cm bed height, and 150 mg/L initial phenol concentration.

Also, the bed height of 5 cm provided a low effluent due to its MTZ, which was greater than the bed height. Moreover, MTZ declined with the rise in bed height. Comparable observations were reported by Kapur & Mondal [39]. From Table 5, the BF-AC with a bed height of 15 cm was chosen owing to its greater phenol removal and the amount treated.

Table 4: Column adsorption parameters for phenol at different flowrates

Packed Bed Column Parameters	Flowrate (mL/min)		
	10	15	20
Initial phenol concentration (mg/L)	150	150	150
Bed depth (cm)	5	5	5
Volume treated (mL)	4200.31	5590.24	6745.08
Volume at breakthrough (mL)	2715.94	3328.33	3524.74
Volume at exhaustion point (mL)	4122.19	5471.19	6575.59
Breakthrough time, t_b (min)	271.59	221.89	176.23
Exhaustion time, t_x (min)	412.22	364.75	328.78
Time taken to exhaust the mass transfer zone, t_δ (min)	140.62	142.86	152.54
Height of Mass Transfer Zone, H_{MTZ} (cm)	10.23	11.75	13.92
Carbon Usage Rate, CUR (g/L)	2.025	1.65	1.56
Specific throughput (mL/g)	493.80	605.15	640.86
Empty bed contact time, EBCT (min)	19.80	13.20	9.90

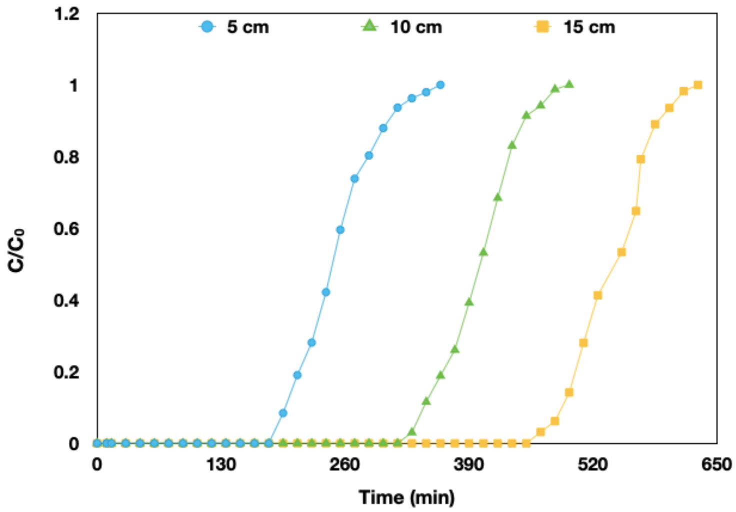


Fig. 6: Phenol adsorption breakthrough curves at various bed heights (5, 10 and 15 cm), 20 mL/min flowrate, and 150 mg/L initial phenol concentration.

Table 5: Bed column parameters for phenol at different bed heights

Packed Bed Column Parameters	Bed Height (cm)		
	5	10	15
Initial phenol concentration (mg/L)	150	150	150
Flowrate (mL/min)	20	20	20
Volume Treated (mL)	6745.08	9606.06	12296.12
Volume at breakthrough (mL)	3524.74	6727.27	9460.3
Volume at exhaustion point (mL)	6575.59	9454.55	12146.87
Breakthrough time, t_b (min)	176.24	336.36	473.01
Exhaustion time, t_x (min)	328.78	472.73	607.34
Time taken to exhaust the mass transfer zone, t_δ (min)	152.54	136.36	134.33
Height of Mass Transfer Zone, H_{MTZ} (cm)	13.92	10.99	9.33
Carbon Usage Rate, CUR (g/L)	1.56	1.63	1.74
Specific throughput (mL/g)	640.86	611.57	573.35
Empty bed contact time, EBCT (min)	9.90	9.90	9.90

4. CONCLUSION

Activated carbon from baobab fruit shell was used to remove phenol from the aqueous solutions. The characterization studies confirm the adsorption behavior of prepared BF-AC with the BET surface area of 1263.127 m^2/g. BF-AC has a large BET surface area and more pore volume, making it a practical and efficient way to remove phenol in a packed bed system. This study revealed that bed height and flowrate have a significant impact on breakthrough times and removal capacities. The removal of phenol was found to be favored by an increase in flowrate and bed height. The breakthrough time increased with an increase in bed height but decreased with an increase in flow rate. BF-AC with a bed height of 15 cm provided better elimination of phenol with CUR of 1.74 g/L and EBCT of 9.9 minutes. The AC prepared from the baobab fruit shell was promising for removing the phenol from the aqueous solution in a fixed bed column.

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