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# Synthesis of *Borassus flabellifer* fruit husk activated carbon filter for phenol removal from wastewater

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## Abstract

The present study investigated the adsorption of phenol from aqueous solution by adsorption onto *Borassus flabellifer* fruit husk activated carbon (BFAC). The activated carbon was produced by three activation processes like pyrolysis (BFAC), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>-BFAC), and zinc chloride (ZnCl<sub>2</sub>-BFAC) method. BET, SEM, EDAX, XRD, and FTIR studies were examined to test the adsorption behavior of synthesized carbons. Characterization studies and batch studies reveal that among the three carbons, ZnCl<sub>2</sub>-BFAC was found to be the best one for the removal of phenol with high surface area (1389 m<sup>2</sup>/g) and maximum adsorption efficiency (95%). Isotherm and kinetics studies were explained that adsorption of phenol by all the three carbons follow multilayer adsorption process. Desorption studies indicated that spent carbon could be regenerated by using 0.1 M NaOH. 87.6% of phenol removal was achieved by ZnCl<sub>2</sub>-BFAC in real textile industry wastewater. Cost analysis suggested that the prepared activated carbons are economically cheaper than the commercial activated carbons.

**Keywords** Activated carbon · *Borassus flabellifer* fruit husk · Phenol · Pyrolysis · Sulfuric acid · Zinc chloride

## Introduction

Water pollution by phenols and its compounds is a major environmental problem. They are considered as chief contaminants in water since they pose serious threats to human beings and aquatic organisms (Jun Shen et al. 2017). The wastewater from various industries like oil, coal conversion, pulp, petrochemicals, pharmaceuticals, pesticides, and dye manufacturing industries are the main sources of phenol and its derivatives (Kujawski et al. 2004; Entezari and Petrier 2005). Phenolic compounds make significant change in the characteristics of drinking water even at very low concentration (5 µg/L) and water becomes unfit for living organisms.

High concentration of phenol (10 to 240 mg/L) in drinking water causes cardiovascular disease, gastrointestinal damage, skin damage, muscle tremors, etc. Therefore, phenol is considered as a high-priority pollutant in water, and removal of phenol from water is considered important.

A number of techniques have been discussed in the literature for the removal of phenol from water and wastewater. Some of the methods are biodegradation, biosorption, photocatalytic degradation, evaporation, solvent extraction, distillation, membrane technology, advanced oxidation process, wet air oxidation, reverse osmosis, ion exchange, and adsorption (Somnath Mukherjee et al. 2007). Among these, adsorption by activated carbon is the most prominent and efficient technology for the wastewater treatment because of its high surface area and porosity. Coal, lignite, wood, and peat are the carbonaceous raw materials generally employed in the commercial production of activated carbon. However, activated carbon prepared from agricultural solid waste materials is abundant and of low cost and shows good adsorption efficiency than the commercial activated carbon. In previous research, many materials have been investigated to prepare low-cost activated carbon like coir pith (Kavitha and Namasivayam 2006), date stone (Alhamed 2009), tamarindus indica (Kumar et al. 2010), banana peel (Achak et al. 2009), sugarcane bagasse (Karunarathne and Amarasinghe

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2013), and lantana camara (Girish and Ramachandra Murty 2014). Although many agriculture products and by-products are reported in the literature, so-called *B. flabellifer* fruit husk has not been used as a source for the preparation of activated carbon.

*Borassus flabellifer* is a tall tree well known as Palmyra in India. Tamil Nadu, a southern state of India, has the highest number of palmyra trees. According to Tamil Nadu agricultural university, out of the estimated 85.9 million of palmyra trees in India, about 51.0 million are in Tamil Nadu state alone. The white kernel of the ripe palm fruit is edible. After removing the sprout, the fruit husk is thrown as solid waste. This fruit husk is a lignocellulosic polymer which consists of 53.4%  $\alpha$ -cellulose, 29.6% hemicellulose, and 17% lignin (Sudhakara et al. 2013). These wastes take long time for decomposition and makes environmental pollution when burnt in open space. Hence, it is important to convert this into useful products. Earlier research has shown *B. flabellifer* fruit husk in the preparation of reinforced polyester composite (Arumuga prabu et al. 2015), thermal insulating fired clay bricks (Bullibabu et al. 2018), and cellulose microfibers (Obi Reddy et al. 2016). Therefore, the present study deals with the preparation of activated carbon from *B. flabellifer* fruit husk and its application for the removal of phenol from water and wastewater.

Adsorption tendency of activated carbon depends on the factors like pH, temperature, surface functional group, particle size, surface morphology of sorbent, and associated contaminants in wastewater. Dursun and Kalayci (2005) reported that the removal of phenol increased with temperature while it adsorbed onto chitin. The study of adsorption of phenol on sediments concluded that the presence of surfactant along with phenol in wastewater suppress the adsorption, whereas the presence of heavy metals enhance the adsorption (Gao et al. 2012). According to Yousefa and El-Eswed (2009), adsorption of phenol increases with pH of the solution and the adsorption capacity is determined by the size of the phenol. Particle size of the sorbent may also affect the rate of adsorption. Mohammad et al. (2016) stated that adsorption of phenol increases with decrease in particles size. Hence, to prepare high-efficiency activated carbon from *B. flabellifer* fruit husk, three different preparation methods (pyrolysis,  $H_2SO_4$  activation, and  $ZnCl_2$  activation) were investigated in this study. The synthesized carbons were analyzed by the determination of physico-chemical properties and characterization analysis such as BET, SEM, EDAX, FTIR, and XRD. Batch-mode adsorption studies were conducted by varying the experimental factors like initial phenol concentration, adsorbent dosage, pH, and equilibrium contact time. Isotherm and kinetic studies were made to compare the efficiency of the prepared carbons. Finally, the possible use of prepared carbons in the treatment of phenol was studied with a textile industry

wastewater. The improvement in the quality of the treated water was determined by the decrease in amount of phenol, BOD, COD, and TDS values. The experiments were performed in triplicate to ensure consistency of the obtained results. The raw material was collected in Coimbatore, Tamil Nadu, India, and the studies were carried out between June 2015 and October 2018.

## Materials and methods

### Collections of *Borassus flabellifer* fruit husk

*Borassus flabellifer* fruits were collected from the district of Coimbatore located in the state of Tamil Nadu, India, and its husk was removed manually and washed thoroughly with distilled water to remove water-soluble matters and dried at 110 °C in hot-air oven. Then the dried husk was stored in an airtight container for the preparation of activated carbon.

### Preparation of raw *B. flabellifer* fruit husk activated carbon (BFAC)

The dried fruit husk was first filled in a copper vessel with 36 cm length and 3 cm width. This vessel was tightly closed with its lid to avoid the contact with atmospheric air. This vessel was placed in muffle furnace and heated to about 700 °C with the heating rate of 10 °C/min (Kavitha and Namasivayam 2006). After reaching 700 °C, temperature of the furnace was maintained for 60 min. Then the material was allowed to cool in the same furnace for 24 h. The carbonized material was removed from the tube and ground in a mortar. The carbon particles were sieved with the mesh size of 150 to 100  $\mu m$ . The sieved carbon was kept inside a vacuum desiccator for 24 h to remove the moisture content. Then the carbon is weighed and stored in an airtight container for adsorption studies.

### Preparation of $H_2SO_4$ activated carbon ( $H_2SO_4$ -BFAC)

The dried fruit husk was charred with concentrated  $H_2SO_4$  solution in 1:1 weight ratio (Srihari and Das 2009) and allowed to immerse for 24 h at room temperature. Then the excess  $H_2SO_4$  was washed by distilled water until the drained solution becomes neutral. The washed char was dried in hot-air oven at 110 °C to remove the moisture and subjected to pyrolysis in a muffle furnace at 600 °C for 60 min with a heating rate of 10 °C/min. The pyrolyzed material was allowed to cool to the room temperature in the furnace for 24 h, and then it is ground and sieved to a particle size of 150 to 100  $\mu m$ . The carbon was cooled in vacuum desiccator for 24 h, weighed, and stored in an airtight container for further process.



## Preparation of ZnCl<sub>2</sub> activated carbon (ZnCl<sub>2</sub>-BFAC)

The dried *B. flabellifer* fruit husk was mixed with ZnCl<sub>2</sub> solution of 1:2 impregnation ratio (50 g of dried fruit husk was mixed with 100 g of solid ZnCl<sub>2</sub>) in 500 ml of distilled water (Muthanna J. Ahmed 2011). Then the solution was agitated at 100 rpm for 1 h at 80 °C in thermostated mechanical shaker machine. The slurry then was filtered and dried in a hot-air oven for 24 h at 110 °C. The dried sample was carbonized in a muffle furnace at about 500 °C. The carbonization temperature was reached with a heating rate of 10 °C/min and held for 60 min in the final temperature for carbonization. The obtained activated carbon was allowed to cool in the furnace for 24 h, and then carbon was taken out from the tube and soaked with 0.1 N HCl solution to remove excess ZnCl<sub>2</sub>. This mixture was left overnight at room temperature and then filtered. The filtered carbon was neutralized with distilled water followed by drying in hot-air oven at 110 °C for 24 h to remove the moisture. Finally, the obtained carbon was sieved to a particle size of 150 to 100 μm, cooled, and stored in an airtight container for further process.

## Characterization studies

BET surface area, SEM, elemental compositions, FTIR, and XRD analysis were made to find the sorption efficiency of prepared carbons for the removal of phenol from aqueous solution. The physical and chemical characteristics of the activated carbons were found out by ASTM methods.

## Adsorption studies

Batch adsorption studies were studied to estimate the optimum pH, adsorbent dosage, the initial phenol concentration, and contact time for the effective uptake of phenol from 50 ml test solution in a 250-ml stoppered conical flask. 0.1 N HCl and 0.1 N NaOH solutions were used to adjust the pH value of the solution. The concentration of residual phenol was determined by 4-aminoantipyrine method (Yuan Cun-Guang 1998).

## Desorption studies

Desorption studies were conducted by treating phenol-loaded activated carbon with 50 ml of desorbing solution (NaOH). The solution along with the spent carbon was agitated for equilibrium time in an orbital mechanical shaker machine. After the agitation, the amount of phenol left in the solution was determined by 4-aminoantipyrine method.

## Textile wastewater examination

Wastewater obtained from the textile industry located in the district of Tirupur, India, was used for this investigation. Removal of phenol has been examined by using optimum dosage, pH, and contact time determined from the batch-mode studies of various synthesized carbons. Main parameters like BOD, COD, TDS, and phenol content of wastewater before and after adsorption were determined by standard methods in laboratory.

## Results and discussion

### Characterization studies

#### BET surface area

The results on BET surface area (m<sup>2</sup>/g) of various activated carbons are represented in Table 1. The BET surface area of adsorbents increased from 248.90 to 1388.92 m<sup>2</sup>/g. This was due to the increase in number of pores in the surface. Among the three carbons, maximum surface area was reported by ZnCl<sub>2</sub>-BFAC (1388.9 m<sup>2</sup>/g). Hence, it shows maximum phenol removal (95%) than BFAC and H<sub>2</sub>SO<sub>4</sub>-BFAC (78% and 86%, respectively). BET surface area of ZnCl<sub>2</sub>-BFAC reported in this study was found to be better than previously reported activated carbons (Table 2). Because of increase in the interspaces between carbon layers of ZnCl<sub>2</sub>-BFAC due to the action of ZnCl<sub>2</sub>, micropores appeared in the adsorbent (Namasivayam and Kavitha 2003). The increase in the microporous nature of ZnCl<sub>2</sub>-BFAC is also attributed to the spaces left by ZnCl<sub>2</sub> in the activated carbon after the repeated washing to remove the excess ZnCl<sub>2</sub> attached to it. ZnCl<sub>2</sub> also causes swelling, an electrolytic action in the molecular structure of cellulose which is the major component in *B. flabellifer* fruit husk. The lateral bonds in the cellulose molecules are broken by the swelling action of ZnCl<sub>2</sub> and leads to increase in inter- and intramicelle voids of ZnCl<sub>2</sub>-BFAC (Sabio and Reinoso 2004; Smisek and Cerny 1970). Therefore, more surface area was observed for ZnCl<sub>2</sub>-BFAC when compared to the other two activated carbons prepared in the absence of ZnCl<sub>2</sub>.

### Physicochemical characterization

The physicochemical properties of the BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC were determined using ASTM standard methods, and the results are presented in Table 1. Prepared carbons showed very low ash content like 3.2, 4.6, and 8.2%, respectively, for BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC. Abdullah et al. (2000) suggested that the ash content within a range of 1 to 20% makes a carbon suitable for absorption.



**Table 1** Mean and standard deviation (SD) of physicochemical properties of activated carbons prepared from *B. flabellifer*

Property	BFAC	H <sub>2</sub> SO <sub>4</sub> -BFAC	ZnCl <sub>2</sub> -BFAC
	Mean ± SD	Mean ± SD	Mean ± SD
BET surface area (m <sup>2</sup> /g)	248.90 ± 3.8	381.50 ± 2.5	1388.92 ± 4.3
pH	9.92 ± 0.06	5.62 ± 0.03	6.71 ± 0.11
pH <sub>ZPC</sub>	9.1 ± 0.13	5.2 ± 0.24	7.0 ± 0.05
Electrical conductivity (mS/cm)	0.248 ± 0.02	0.456 ± 0.03	0.252 ± 0.07
Bulk density (g/mL)	0.12 ± 0.12	0.14 ± 0.08	0.16 ± 0.24
Moisture content (%)	2.3 ± 0.29	3.5 ± 0.75	3.3 ± 0.38
Volatile content (%)	27.7 ± 0.56	56.0 ± 0.34	42.3 ± 0.27
Ash content (%)	3.2 ± 0.01	4.6 ± 0.05	8.2 ± 0.1
Fixed carbon (%)	66.8 ± 1.2	35.9 ± 0.77	46.2 ± 0.06
Decolorizing power (mg/g)	15.00 ± 0.05	39.00 ± 0.13	129.00 ± 2.7
Iodine number (mg/g)	456.10 ± 3.8	550.87 ± 1.6	958.99 ± 2.1
Ion exchange capacity (meq/g)	0.100 ± 0.07	0.110 ± 0.14	0.130 ± 0.34
<i>Elemental composition by EDAX analysis</i>			
Carbon	87.78	63.93	91.01
Oxygen	11.45	34.02	7.62
Magnesium (%)	0.22	–	–
Potassium (%)	0.33	–	–
Calcium (%)	0.22	–	–
Sodium (%)	–	2.05	–
Chlorine (%)	–	–	1.13
Zinc (%)	–	–	0.24

**Table 2** Comparison of surface area of different adsorbents

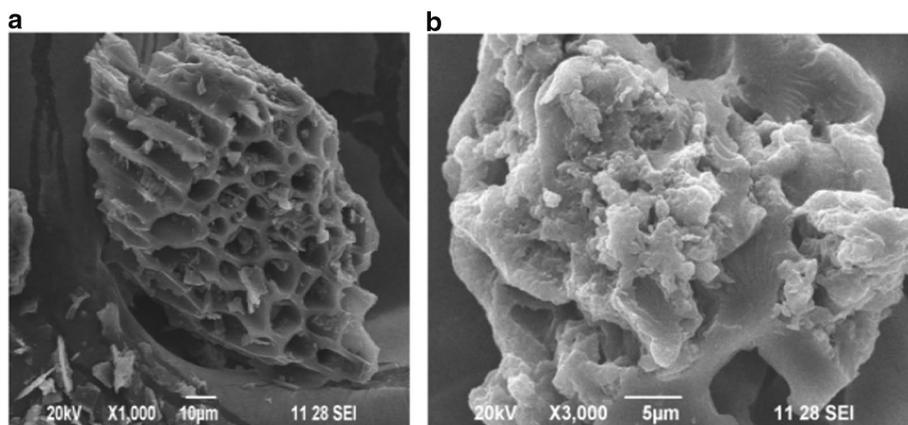
Adsorbents	Surface area (m <sup>2</sup> /g)	References
Mesoporous carbon from peach stones	1521	Silvia Alvarez-Torrellas et al. (2015)
Activated carbon prepared from <i>N. microphyllum</i>	1940	Mousavi et al. (2017)
Activated carbon of rattan hydrochar	1135	Islam et al. (2017)
Sugarcane bagasse-based activated carbon	697.37	Magda AA Akl et al. (2014)
Nickel hydroxide-nanoplated modified activated carbon	960.3	Shilpi Agarwal et al. (2016)
Activated carbon of porous clay heterostructure	305.5	Sofia Arellano-Cardenas et al. (2005)
ZnCl <sub>2</sub> activated coir pith carbon	910	Subha and Namasivayam (2010)
Palm seed coat activated carbon	577	Rengaraj et al. (2002)
Activated carbon from <i>Tectona grandis</i> saw dust	585	Kaustubha Mohanty et al. (2005)
BFAC	248.90	Present study
H <sub>2</sub> SO <sub>4</sub> -BFAC	381.50	Present study
ZnCl <sub>2</sub> -BFAC	1388.92	Present study

The moisture content of all the carbons was lower (2.3, 3.5 and 3.3%) than the carbons reported in the previous literature (Ekpete et al. 2017). Among the three carbons, BFAC had low moisture content and ash content. The prepared activated carbons also showed high decolorizing power, iodine number, and ion exchange capacity. The results validate that ZnCl<sub>2</sub>-BFAC showed better surface characteristics with high surface area, decolorizing power (129.00 mg/g), iodine number (958.99 mg/g), and ion exchange capacity (0.130 meq/g) than other two carbons.

### Scanning electron microscope analysis and EDAX analysis

The SEM enables the direct observation of the changes in the surface microstructure of the adsorbents due to the modifications (Jain et al. 2010). SEM pictures of synthesized carbons confirmed the presence of isolated pores of varying dimensions from micropores to macropores. The SEM images of ZnCl<sub>2</sub>-BFAC before and after adsorption of phenol are shown in Fig. 1. The SEM image of ZnCl<sub>2</sub>-BFAC before adsorption of phenol shows regular pores of large

**Fig. 1** SEM image of **a** ZnCl<sub>2</sub>-BFAC and **b** ZnCl<sub>2</sub>-BFAC after adsorption with phenol

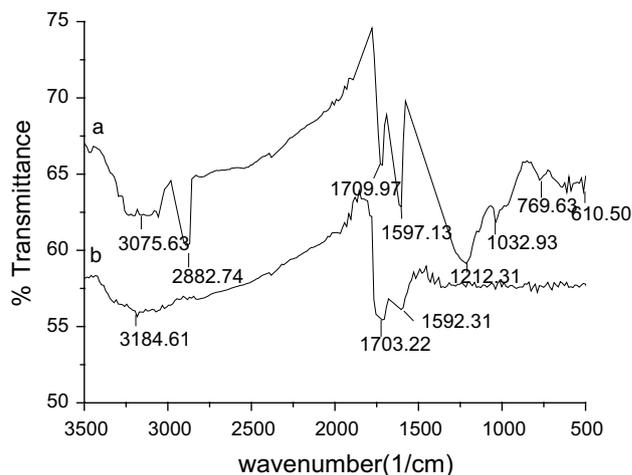


dimensions with honeycomb-like structure. The inorganic irregular deposits were also clearly seen on the carbon surface. The SEM image of ZnCl<sub>2</sub>-BFAC after adsorption reveals that active centers of adsorbent were covered by the adsorbate and concluded that adsorption has occurred on the micropores to macropores of the activated carbons. The phenol deposits appeared more on ZnCl<sub>2</sub>-BFAC than H<sub>2</sub>SO<sub>4</sub>-BFAC and BFAC. Thus, ZnCl<sub>2</sub>-BFAC is superior to H<sub>2</sub>SO<sub>4</sub>-BFAC and BFAC. The fact of SEM analysis confirmed the potential of ZnCl<sub>2</sub> in creating highly porous activated carbon.

Elemental composition of the prepared carbon has been identified by EDAX analysis. EDAX analysis shows that ZnCl<sub>2</sub>-BFAC has the highest percentage of carbon (91.01%) among the three carbons. The carbon percentage of BFAC and H<sub>2</sub>SO<sub>4</sub>-BFAC is 87.78% and 63.93%, respectively. This result suggests that ZnCl<sub>2</sub> activation would be a better choice to prepare activated carbon with maximum percentage of carbon than simple pyrolysis and acid activation process.

### FTIR analysis

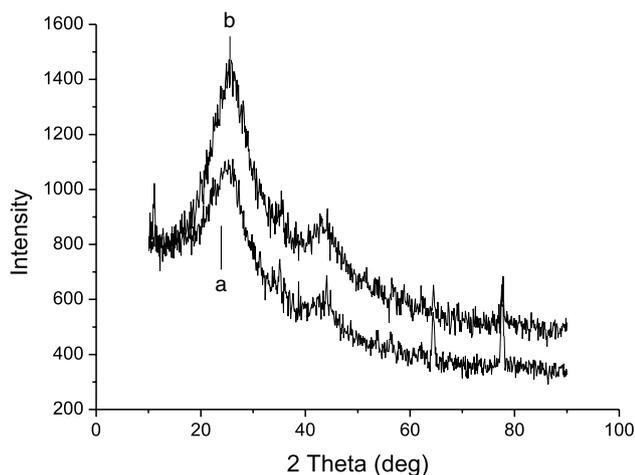
FTIR studies are used to detect the functional groups responsible for adsorption. The adsorption properties of activated carbon are determined not only by its porous structure but also by the surface functional groups present on the activated carbons (Muthukumaran and Sophie Beulah 2010). Activated carbon contains acidic and basic groups. These groups are derived from the starting material, and they stay behind as a result of imperfect carbonization, or they can turn into chemically bonded to surface during activation. The FTIR spectra of ZnCl<sub>2</sub>-BFAC (before and after adsorption of phenol) are shown in Fig. 2. Figure illustrates that some peaks are shifted or disappeared after adsorption which indicates that the functional groups present on the adsorbent are involved in the adsorption of phenol. A broad absorption peak observed



**Fig. 2** FTIR spectra of ZnCl<sub>2</sub>-BFAC **a** before adsorption and **b** after adsorption

at 3073.63 cm<sup>-1</sup> in FTIR spectra of before adsorption is attributed to the –OH stretching vibration of alcohol and phenol. After the adsorption, this peak was shifted to the higher wave number, i.e., 3184 cm<sup>-1</sup>. This shift confirmed –OH group was responsible for the adsorption of phenol on ZnCl<sub>2</sub>-BFAC. The peaks at 2882 cm<sup>-1</sup> (stretching bands of CH, CH<sub>2</sub> and CH<sub>3</sub>), 1212 cm<sup>-1</sup> (C–O stretching of COOH and –OH bending modes of alcoholic, phenolic and carboxylic groups), 1032 cm<sup>-1</sup> (C–O stretching of COOH), 769 cm<sup>-1</sup> (stretching vibrations in the aromatic ring), and 610 cm<sup>-1</sup> (stretching vibrations in the aromatic ring) disappeared in FTIR spectra of after adsorption. This reveals that the methylene group and C–O groups are involved in the adsorption process. Hence, the presence of methylene group and carbonyl group on the surface of ZnCl<sub>2</sub>-BFAC could be responsible for its high adsorption ability than other two carbons.





**Fig. 3** XRD spectra of  $\text{ZnCl}_2$ -BFAC **a** before adsorption and **b** after adsorption

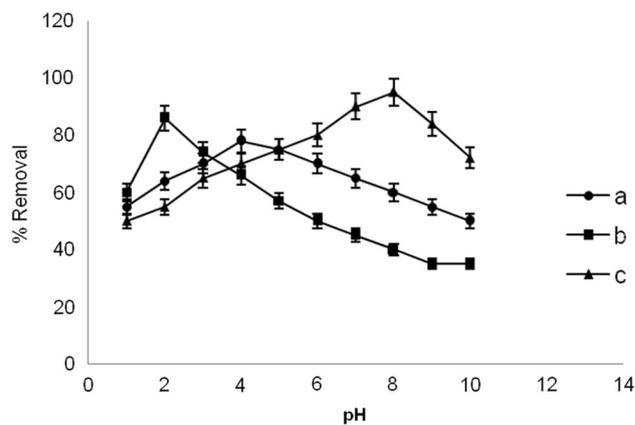
### XRD analysis

Powder X-ray diffraction spectrums of  $\text{ZnCl}_2$ -BFAC before and after adsorption of phenol are shown in Fig. 3. The spectrums showed three strong peaks ( $2\theta$ ) at  $25^\circ$ ,  $43^\circ$ , and  $77^\circ$  which are associated with 002, 100, and 110 planes of graphite (Saleh 2011). The broad peak at  $25^\circ$  in the XRD spectrum shows the amorphous nature of activated carbon which may be responsible for highest percentage removal of phenol. There was no change in position of peaks in the spectrum of carbons after adsorption. However, there was a slight increase in the intensities of the peaks, and this may be due to the morphological changes in carbon surface by adsorption process.

### Batch studies

#### Effect of pH

The adsorption process is mainly influenced by the pH of the adsorbate. pH of the solution is responsible for the surface charge of adsorbent (Mambo Moyo et al. 2012). The effect of pH on the adsorption of phenol of various carbons is shown in Fig. 4. The maximum adsorption of phenol by BFAC,  $\text{H}_2\text{SO}_4$ -BFAC, and  $\text{ZnCl}_2$ -BFAC was obtained at pH 2, pH 4, and pH 8, respectively. The effect of pH reveals that among these carbons,  $\text{ZnCl}_2$ -BFAC filter could be employed in neutral pH to achieve maximum removal of phenol (95%). This phenomenon shows the amphoteric nature of  $\text{ZnCl}_2$ -BFAC. Hence, there would be a chance to treat wastewater at neutral pH with the help of  $\text{ZnCl}_2$ -BFAC in real-time applications.



**Fig. 4** Effect of pH on removal of phenol by **a** BFAC, **b**  $\text{H}_2\text{SO}_4$ -BFAC, and **c**  $\text{ZnCl}_2$ -BFAC with  $\pm 5\%$  error bars in percentage removal

#### Effect of initial phenol concentration

This process was investigated by using various phenol concentrations (10, 20, 30, 40, and 50 mg/L). It can be observed that the uptake of phenol increased with time and reached a maximum value at some point of time and then remained more or less constant, and the system was in a state of dynamic equilibrium. From the results, it was observed that all the three carbons achieved maximum percentage of adsorption only at low initial concentration (10 mg/L). Adsorption slowed down in the later stage because initially more number of active sites were available for adsorption and, after some time, the remaining active sites may be difficult to occupy due to repulsive forces between adsorbate molecules on the adsorbent and bulk phase (Bishnoi et al. 2004). Hence, all the batch studies were performed at low initial concentration of phenol (10 mg/L).

#### Effect of adsorbent dosage

This study helps to determine the optimum amount of adsorbent required for the effective removal of adsorbate from the solution. To find the optimum quantity of adsorbent, various quantities of carbon from 10 to 300 mg were treated with phenol solution (10 mg/L). The percentage removal of phenol was increased from lower to higher adsorbent dosages. This may be due to the increase in active sites by the increase in adsorbent dosage. The maximum amount of adsorbents required for the removal of phenol by BFAC,  $\text{H}_2\text{SO}_4$ -BFAC, and  $\text{ZnCl}_2$ -BFAC are 100, 100, and 80 mg, respectively. After this dosage, there was no appreciable change in the adsorption process. Very gradual increase in the removal of phenol beyond an optimum dose of activated carbons may be attributed to the accomplishment of equilibrium between the phenol and carbon surface at the operating conditions. This

effect is called “solid concentration effect” (Bishnoi et al. 2004). Among the three carbons, dosage required to achieve maximum adsorption (95%) was less for ZnCl<sub>2</sub>-BFAC due to its high surface area and carbon percentage.

**Effect of contact time**

The optimum time at which equilibrium obtained was determined by treating the prepared activated carbons with 10 mg/L initial concentration of phenol at its optimum pH and optimum carbon dosage. It shows that there was a rapid adsorption of phenol within 30 min, and then adsorption equilibrium was attained at 80, 60, 50 min for BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC, respectively. After this period, adsorption almost remains constant. About 50% of adsorption has occurred within 20 min in all carbons which may be due to the presence of more number of active sites on the adsorbent in the starting stage. The accumulation of adsorbate in the active sites might be responsible for the decrease in the adsorption of phenol after the equilibrium time (Ektepe and Horsfall 2011). Compared with the equilibrium time reported in the adsorption of phenol on activated carbon made from fluted pumpkin stem waste (Ektepe and Horsfall 2011), activated carbon from wood fiber board waste (Jin and Zhu 2014), and tea industry waste (Ali Gundogdu et al. 2012), shorter equilibrium time was observed in the present study.

**Adsorption isotherms**

Equilibrium data obtained from the adsorption studies were used to obtain the adsorption isotherms. In the present study, four different isotherms (Freundlich, Langmuir, Temkin, and Dubinin–Radushekevich (D-R) models) were studied for predicting the behavior of adsorption process. All the above isotherm parameters were determined by nonlinear regression analysis using solver, an add-in available in Microsoft Excel 2007 (Microsoft corporation, USA) (Ho et al. 2005, Amtul Qayoom et al. 2017). In order to evaluate the goodness of fit with isotherm model, correlation coefficient (R<sup>2</sup>), nonlinear Chi-square (X<sup>2</sup>), residual root-mean-square error (RMSE), average relative error (ARE), and normalized standard deviation (NSD) were also determined. Mathematical expression of used error functions are as follows (Ghaffari et al. 2017):

(i) Correlation coefficient (R<sup>2</sup>)

$$= \frac{(q_{e,exp} - \overline{q_{e,cal}})^2}{\sum (q_{e,exp} - \overline{q_{e,cal}})^2 + (q_{e,exp} - q_{e,cal})^2} \quad (1)$$

(ii) Nonlinear Chi - square (X<sup>2</sup>)

$$= \sum_{i=1}^n \frac{(q_{e,cal} - q_{e,exp})^2}{q_{e,exp}} \quad (2)$$

(iii) Residual Root - Mean - Square - Error (RMSE)

$$= \sqrt{\frac{1}{n-2} \sum_{i=1}^n (q_{e,exp} - q_{e,cal})^2} \quad (3)$$

(iv) Average Relative Error (ARE)

$$= \frac{100}{n} \sum_{i=1}^n \left| \frac{q_{e,exp} - q_{e,cal}}{q_{e,exp}} \right| \quad (4)$$

(v) Normalized Standard Deviation (NSD)

$$= \left[ \sqrt{\frac{\sum (q_{e,exp} - q_{e,cal}/q_{e,exp})^2}{N}} \right] \times 100 \quad (5)$$

where q<sub>e,cal</sub> is calculated quantity of phenol adsorbed at equilibrium (mg/g), q<sub>e,exp</sub> is the quantity of phenol adsorbed at equilibrium obtained from experimental (mg/g), and n is the number of data.

**Freundlich isotherm**

The Freundlich isotherm was determined to find the chance of multilayer adsorption. The Freundlich can be given by the equation (Ghaffari et al. 2017)

$$q_e = K_F C_e^{1/n}, \quad (6)$$

where q<sub>e</sub> = amount of phenol adsorbed at equilibrium (mg/g), C<sub>e</sub> = amount of phenol adsorbed at equilibrium (mg/L), K<sub>F</sub> = adsorption capacity of the adsorbent (mg/g), 1/n = adsorption intensity of the adsorbent.

The values of K<sub>F</sub> and 1/n were obtained from the nonlinear regression analysis graph and are presented in Table 3. High correlation coefficient (R<sup>2</sup>) and comparatively low error functions values of Freundlich isotherm equation for all the three carbons showed better fit than other isotherm models. This indicated that adsorption of phenol on to BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC was a multilayer adsorption. The adsorption intensity (1/n) of BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC was found to be 0.4249, 0.3591, and 0.2809, respectively. These values were less than unity. As shown in Foo and Hameed 2010, this 1/n value indicated the heterogeneous nature of the system. Thus, among the three carbons, BFAC showed high 1/n value which indicates that BFAC has greater heterogeneity than other two carbons.

**Table 3** Isotherm parameters and error deviation data for the adsorption of phenol on BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC by nonlinear regression method

Isotherms		Parameters			Different error functions				SD
Freundlich isotherm	Carbon	$K_F$ (mg/g)	$1/n$		$R^2$	$X^2$	RMSE	ARE	
	BFAC	2.8279	0.4249		0.9951	0.002	0.056	0.04	0.005
	H <sub>2</sub> SO <sub>4</sub> -BFAC	3.946	0.3591		0.9901	0.002	0.058	0.142	0.025
	ZnCl <sub>2</sub> -BFAC	7.2904	0.2809		0.9958	0.001	0.050	0.027	0.08
Langmuir isotherm	Carbon	$q_m$ (mg/g)	$b$ (L/mg)	$R_L$	$R^2$	$X^2$	RMSE	ARE	SD
	BFAC	13.97	0.1234	0.4476	0.9781	0.067	0.952	0.816	0.395
	H <sub>2</sub> SO <sub>4</sub> -BFAC	13.42	0.2488	0.2866	0.9873	0.036	0.742	0.575	0.409
	ZnCl <sub>2</sub> -BFAC	17.02	0.5188	0.1616	0.9852	0.231	2.195	1.213	2.527
Temkin isotherm	Carbon	$A$ (L/g)	$B$ (kJ/mole)		$R^2$	$X^2$	RMSE	ARE	SD
	BFAC	1.3	0.8301		0.9511	0.088	1.091	0.935	0.363
	H <sub>2</sub> SO <sub>4</sub> -BFAC	2.78	0.8904		0.9694	0.036	0.739	0.572	0.219
	ZnCl <sub>2</sub> -BFAC	11.5607	0.8373		0.9792	0.032	0.823	0.455	0.475
D-R isotherm	Carbon	$q_D$ (mg/g)	$K_D$ (mol <sup>2</sup> /J)	$E$ (kJ/mol)	$R^2$	$X^2$	RMSE	ARE	SD
	BFAC	9.28	$-2 \times 10^{-06}$	-0.5	0.9244	23.9	17.96	15.40	2.601
	H <sub>2</sub> SO <sub>4</sub> -BFAC	9.81	$-6 \times 10^{-07}$	-0.9174	0.8479	27.2	20.14	15.61	2.799
	ZnCl <sub>2</sub> -BFAC	14.51	$-1.30 \times 10^{-06}$	-0.1616	0.9376	37.0	27.81	15.3	3.734

**Langmuir isotherm**

Langmuir isotherm denotes the monolayer adsorption process. The nonlinear Langmuir isotherm can be represented as (Ho et al. 2005):

$$q_e = \frac{q_m b C_e}{1 + b C_e} \tag{7}$$

where  $q_m$  (maximum adsorption capacity (mg/g)) and  $b$  (energy of adsorption (L/mg)) are Langmuir isotherm constants. Langmuir parameters and its corresponding error functions are given in Table 3. It was observed that ZnCl<sub>2</sub>-BFAC has the maximum sorption capacity 17.02 mg/g when compared to other two activated carbons (BFAC = 13.97 and H<sub>2</sub>SO<sub>4</sub>-BFAC = 13.42). Even though high sorption capacity was observed for all the carbons, Langmuir model does not fit with experimental data due to its high  $X^2$ , RMSE, ARE, and NSD value and low  $R^2$  value than Freundlich isotherm. Hence, there could be some heterogeneity in the system (Khalid et al. 2015). Moreover, similar  $q_m$  values were reported in the literature by Sarkar and Acharya 2006 (17.1 mg/g) and Giraldo and Moreno-Pirajan 2008(13.7 mg/g). The favorable characteristics of the adsorption process can be identified by separation factor  $R_L$ . It can be expressed as:

$$R_L = \frac{1}{1 + b C_0} \tag{8}$$

where  $C_0$  is initial concentration of solute and  $b$  is Langmuir constant. The  $R_L$  value indicates the nature of adsorption process to be favorable ( $0 < R_L < 1$ ), linear ( $R_L = 1$ ), unfavorable ( $R_L > 1$ ), or irreversible ( $R_L = 0$ ) (Dorra Tabassi et al. 2017).  $R_L$  values of all the carbons were found as 0.4476, 0.2866, and 0.1616. Thus, from  $R_L$  values, it was clear that the adsorption of phenol onto BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC is a feasible process.

**Temkin isotherm**

The Temkin model assumes that, because of indirect adsorbate–adsorbent interactions, the heat of adsorption of all the molecules in the layer diminishes linearly with adsorption (Khalid et al. 2015). The nonlinear form of Temkin isotherm can be expressed as:

$$q_e = B \ln A C_e, \tag{9}$$

where  $B = RT/b$  which is related to heat of adsorption.  $A$  (L/g) and  $B$  (kJ/mol) are Temkin constants. The values of  $A$  and  $B$  are given in Table 3. Correlation coefficient value was approximately 0.95 for all the carbon. Even though  $R^2$  values were high, Temkin isotherm shows moderate fit with experimental data due to its high error function values than other isotherms. This suggested that adsorption of phenol onto BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC shows more attraction toward nonlinear energy distribution than linear. The potential of adsorbent ( $B$ ) for BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC was found to be 0.8301, 0.8904, and 0.8373 kJ/mole, respectively. Since these values are in the range of

physisorption (less than 8 kJ/mole), it indicates that the adsorption of phenol onto the prepared carbons is physical adsorption (Boldizar Nagy et al. 2014).

### Dubinin–Radushkevich isotherm

This isotherm suggests adsorption mechanism with Gaussian energy distribution onto heterogeneous surface with energy distribution (Dabrowski 2001). The D-R equation is given as (Foo and Hameed 2010):

$$q_e = q_D \exp(-K_D \varepsilon^2) \quad (10)$$

where  $q_D$  is the D-R isotherm constant related to the degree of adsorption and  $\beta$  is the free energy of adsorption. The parameter  $\varepsilon$  is expressed as:

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right). \quad (11)$$

The adsorption energy,  $E$ , per mole of adsorbate can be obtained by the following equation:

$$E = \frac{1}{\sqrt{2K_D}}. \quad (12)$$

The D-R constant  $K_D$  suggests the difference of physisorption and chemisorption of adsorbate with its free energy (Redlich and Peterson 1959). D-R parameters are given in Table 3. The mean energy values ( $E$ ) for the adsorption of phenol by all the three adsorbents were less ( $-0.5$  to  $-0.91$  kJ/mol), and these low values revealed that the adsorption process was physisorption, since physisorption process had adsorption energies less than  $-40$  kJ/mole. Likewise, the negative values of  $E$  represent the adsorption process could be an exothermic in nature (Srihari and Das 2008). The amount of phenol adsorbed ( $q_D$ ) by all the carbons (BFAC = 9.28 mg/g, H<sub>2</sub>SO<sub>4</sub>-BFAC = 9.81 mg/g, and ZnCl<sub>2</sub>-BFAC = 14.51 mg/g) are lower than the Langmuir  $q_m$  (BFAC = 13.97 mg/g, H<sub>2</sub>SO<sub>4</sub>-BFAC = 13.42 mg/g, and ZnCl<sub>2</sub>-BFAC = 17.02 mg/g) which showed the lower effectiveness of adsorption process. Similar style was observed in the biosorption of Cu (II) onto dead immobilized *Rhizopus Arrhizus* (Khalid et al. 2015). Error analysis also confirmed the poor fit of D-R model with equilibrium data.

### Kinetics studies

Kinetics studies explain the rate of phenol uptake by prepared adsorbents. Four different kinetics models like pseudo-first-order, pseudo-second-order, Elovich, and intraparticle diffusion models were used to study the adsorption of phenol by prepared activated carbons. Best fit of experimental data with various kinetics models was analyzed by nonlinear regression method as mentioned in adsorption studies.

### Pseudo-first-order kinetics and pseudo-second-order kinetics model

The nonlinear form of pseudo-first-order kinetics model can be given as (Vieira et al. 2014):

$$q_t = q_e(1 - \exp(-K_1 t)) \quad (13)$$

where  $K_1$  (min<sup>-1</sup>) is the rate constant of the pseudo-first-order adsorption and  $q_e$  is the amount of phenol adsorbed onto adsorbent (mg/g).

Pseudo-second-order kinetics can be expressed as (Vieira et al. 2014):

$$q_t = \frac{K_2 q_e^2 t}{1 + K_2 q_e^2 t} \quad (14)$$

where  $K_2$  (g/mg/min) is the rate constant of pseudo-second-order kinetics model. The factors of pseudo-first-order and pseudo-second-order kinetics are given in Table 4.  $R^2$  values of pseudo-first-order kinetics of BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC were found to be 0.9817, 0.9680, and 0.9302 respectively, which are relatively lower than  $R^2$  values of pseudo-second-order kinetics (BFAC = 0.996, H<sub>2</sub>SO<sub>4</sub>-BFAC = 0.9919, and ZnCl<sub>2</sub>-BFAC = 0.9942). Furthermore, experimental  $q_e$  values of all the three carbons (3.90, 4.30, and 5.94 mg/g) did not match with calculated  $q_e$  values of pseudo-first-order kinetics (4.06, 4.38, and 5.70), whereas calculated  $q_e$  values of pseudo-second-order kinetics were close to the experimental values. In addition to that, Chi-square, RMSE, ARE, and NSD values of pseudo-second-order kinetics were less when compared to the other models. Hence, adsorption of phenol by BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC showed best fit with pseudo-second-order kinetics which indicates that adsorption mechanism depends on both adsorbent and adsorbate (Jia Feng et al. 2015).

### Elovich model

Elovich equation is related to chemisorption. This Elovich equation is suitable for the heterogeneous adsorbent surface. The Elovich equation (Vieira et al. 2014) is written as:

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) \frac{1}{\beta} \ln(t) \quad (15)$$

where  $\alpha$  (mg/g/min) is the initial adsorption rate and  $\beta$  (g/mg) is related to the degree of surface coverage and the activation energy of chemisorption. Elovich coefficients can be determined by the graph plotted between  $q_t$  versus  $t$ . Values of  $\alpha$  and  $\beta$  are given in Table 4. The results implied that the experimental data were not in correlation with Elovich model since the  $R^2$  values of all the carbons are found to be lower than other models. But the error analysis confirms the



**Table 4** Kinetic parameters and error deviation data for adsorption of phenol on BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC by nonlinear regression method

Kinetics	Parameters	Different error functions							SD
		$R^2$	$X^2$	RMSE	ARE	SD	$q_e$ , cal (mg/g)	$q_e$ , exp (mg/g)	
Pseudo-first-order	Carbon	$K_1$ (1/min)	$q_e$ , cal (mg/g)	$q_e$ , exp (mg/g)	$R^2$	$X^2$	RMSE	ARE	0.317
	BFAC	0.0797	3.1635	3.9	0.9817	0.937	3.187	3.395	
	H <sub>2</sub> SO <sub>4</sub> -BFAC	0.051	4.1913	4.3	0.968	0.359	2.092	1.985	
	ZnCl <sub>2</sub> -BFAC	0.0612	5.5931	5.94	0.9302	0.655	3.342	2.266	
Pseudo-second-order	Carbon	$K_2$ (g/mg/min)	$q_e$ (mg/g)	$q_e$ , exp (mg/g)	$R^2$	$X^2$	RMSE	ARE	0.065
	BFAC	0.0216	4.059	3.9	0.996	0.017	0.434	0.463	
	H <sub>2</sub> SO <sub>4</sub> -BFAC	0.0245	4.3824	4.3	0.9919	0.231	1.676	1.591	
	ZnCl <sub>2</sub> -BFAC	0.0342	5.7015	5.94	0.9942	0.221	1.944	1.318	
Elovich model	Carbon	$\alpha$	$\beta$		$R^2$	$X^2$	RMSE	ARE	0.149
	BFAC	3.4107	1.9045		0.9150	0.340	1.92	2.045	
	H <sub>2</sub> SO <sub>4</sub> -BFAC	4.0277	1.7198		0.8694	0.397	2.2	2.087	
	ZnCl <sub>2</sub> -BFAC	10.7831	1.4854		0.9280	0.150	1.6	1.085	
Intraparticle diffusion model	Carbon	$K_{diff}$ (mg/g/min)	$C$ (mg/g)		$R^2$	$X^2$	RMSE	ARE	0.263
	BFAC	0.2118	1.6629		0.9430	0.020	0.468	0.499	
	H <sub>2</sub> SO <sub>4</sub> -BFAC	0.2498	1.8441		0.9191	0.002	0.166	0.158	
	ZnCl <sub>2</sub> -BFAC	0.3177	2.8998		0.9105	0.019	0.572	0.388	

moderate fit of Elovich model since the error function values were relatively lower than the pseudo-first-order kinetics.

**Intraparticle diffusion model**

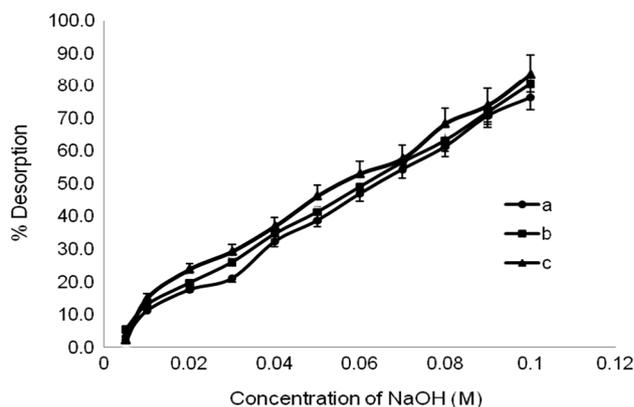
The linear form of intraparticle diffusion is given by (Shilpi Agarwal et al. 2016):

$$q_t = K_{diff}t^{(1/2)} + c \tag{16}$$

where  $K_{diff}$  is rate constant of intraparticle diffusion (mg/g/min) and  $C$  is the thickness of boundary layer (mg/g). The values are given in Table 4. The low  $R^2$  values indicated that intraparticle diffusion was not a rate-determining step for the adsorption of phenol, but the corresponding error values are lower (approximately 0.002) than pseudo-second-order model (approximately 0.2). The intercept value or thickness of the boundary layer ( $C$ ) of all the carbons was higher than zero (1.66, 1.84 and 2.89 mg/g). Therefore, the result confirmed that the regression of all the carbons was not linear, and thus the sorption process was controlled by intraparticle diffusion in addition with pseudo-second-order kinetics. The same trend was observed in the investigation of adsorptive removal of phenol over commercial GAC by Dorra Tabassi et al. 2017.

**Desorption studies**

To regenerate the spent activated carbon, phenol-loaded carbon was treated with various concentration of NaOH from 0.005 to 0.1 M. The results are shown in Fig. 5. It



**Fig. 5** Desorption of phenol by **a** BFAC, **b** H<sub>2</sub>SO<sub>4</sub>-BFAC, and **c** ZnCl<sub>2</sub>-BFAC with  $\pm 5\%$  error bars in percentage desorption

concludes that maximum desorption occurred at 0.1 M NaOH solution for all the carbons (BFAC = 76.3%, H<sub>2</sub>SO<sub>4</sub>-BFAC = 80.4%, and ZnCl<sub>2</sub>-BFAC = 83.7%). This could be due to the formation of sodium phenates (C<sub>6</sub>H<sub>5</sub>ONa) by the reaction between NaOH and adsorbed phenol molecules (Dorra Tabassi et al. 2017). The activated carbons attain negative charge in the alkaline medium and hence rate of desorption increases due to the electrostatic repulsion between negatively charged carbon and sodium phenates. The results showed that the uptake of phenol onto BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC was reversible. Hence spent carbon could be easily regenerated and reused.

**Table 5** Treatment of textile industry wastewater

Parameters	Before adsorption			After adsorption Amount (ppm)			
	Amount (ppm)	BFAC (Mean)	SD	H <sub>2</sub> SO <sub>4</sub> - BFAC (Mean)	SD	ZnCl <sub>2</sub> -BFAC (Mean)	SD
BOD	2840	32	0.07	28	0.25	25	0.34
COD	12,240	162	1.2	148	2.05	134	1.9
TDS	2561	420	1.82	380	1.31	332	2.2
Phenol	5.64	1.5	0.53	1.1	0.12	0.7	0.94

### Treatment of textile industry wastewater

The amount of BOD, COD, TDS, and phenol of textile wastewater before and after adsorption is given in Table 5 with standard deviation. The results confirmed the potential of prepared carbons. Maximum phenol removal (87.6%) was recorded by ZnCl<sub>2</sub>-BFAC (SD = 0.94), whereas BFAC and H<sub>2</sub>SO<sub>4</sub>-BFAC showed 73.4% (SD = 0.53) and 79.8% (SD = 0.12) removal, respectively. Along with phenol, the other parameters like BOD, COD, and TDS were also reduced to considerable amount during adsorption. As a result, this study substantiates the prominent potential of ZnCl<sub>2</sub>-BFAC in the treatment of phenol from wastewater.

### Cost estimation of *B. flabellifer* fruit husk activated carbons

The cost estimation was made for the prepared carbons. Agriculture waste product was readily available at no cost. Hence, the cost of raw material was not included in the cost estimation, whereas power consumption and chemical used in the preparation were considered to determine the cost for 1 kg of carbon. The results suggested that the estimated price of BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC was found to be 0.19, 0.23, and 0.34 USD, respectively. This cost is very less than the cheapest commercial activated carbon existing in the market at 5.16 USD per kg. In previous research work, the cost of 1 kg of activated carbon prepared from tamarind seeds, neem leaves, saw dust, and fly ash was reported as 0.52 USD, 1.15 USD, 0.06 USD, and 0.51 USD, respectively (Gupta and Babu 2008), and also the cost of activated carbon prepared from Artocarpus integer fruit-processing waste was found to be 1.67 USD per kg (Geetha Selvaraju and Bakar 2017). Chopra et al. (2011) reported that total cost required for the water treatment by chemical precipitation and electrocoagulation method was 15.21 US\$/m<sup>3</sup> and 8.62 US\$/m<sup>3</sup>, respectively. Thus, we can conclude that the activated carbons prepared from biomass would be cost-effective and a promising alternative method to other technologies available for water treatment.

### Conclusion

In this work, *B. flabellifer* fruit husk, a waste biomass, was used as raw material for the preparation of activated carbon by pyrolysis, sulfuric acid, and zinc chloride activation methods. The efficacy of the prepared carbons as an adsorbent were analyzed and compared with the adsorption of phenol from wastewater. Characterization and batch studies revealed that ZnCl<sub>2</sub>-BFAC was found to be the best adsorbent among three carbons. The maximum adsorption percentage of phenol by ZnCl<sub>2</sub>-BFAC was observed as 95% with minimum carbon dosage of 80 mg at optimum pH of 8. Isotherm analysis for the removal of phenol by BFAC, H<sub>2</sub>SO<sub>4</sub>-BFAC, and ZnCl<sub>2</sub>-BFAC showed best fit with Freundlich isotherm which confirms multilayer adsorption. The equilibrium study indicated that removal of phenol by all the three prepared carbons follows the mechanism of pseudo-second-order kinetics which has high  $r^2$  value and low error analysis value than other model. Intraparticle diffusion study also confirmed the multilayer adsorption. High percentage desorption of phenol from spent carbon concluded that all the carbons can be regenerated and reused. Testing with real wastewater implied that ZnCl<sub>2</sub>-BFAC was able to remove phenol up to 87.6% among the three carbons. Finally, cost analysis confirmed that the prepared activated carbons would be an economically promising material. Thus *B. flabellifer* fruit husk would be effectively used to prepare activated carbons for the water treatment at low cost.

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### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.



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