Batch and Column Adsorption of BOD and COD in vegetable oil industry effluents Using Activated Carbon from Fluted Pumpkin (*Telfairia, Occidentalis*. Hook. F) Seed Shell

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ABSTRACT

The use of waste to reduce pollution has been advocated by many researchers. In this study five samples of physically prepared fluted pumpkin seed shell activated carbons (PFACs) prepared elsewhere were successfully used in reducing Biochemical oxygen demand (BOD) and chemical oxygen demand (COD) of vegetable oil industry effluents (VOIE). BOD readings of effluent were measured using the Lovibon BOD IR Sensomat while COD was determined by use of PCcheckit COD Vario (Lovibond) consisting of PCcheckit COD Vario photometer and COD reactor ET 108 model. Batch adsorption had better performance (BOD and COD removal of 52 % to 83 %) over column adsorption (BOD_5 and COD removal of 35 % to 86 %). Batch adsorption gave better BOD and COD reduction. Though BOD and COD removal varied widely with carbon dose, pH, temperature and contact time the effects of theses factors investigated in the reduction of BOD and COD were complex and difficult to streamlined.

Keywords: Batch; Column; Removal; Vegetable oil; Effluents; Adsorption

1. INTRODUCTION

When discharged into the environment the deleterious effects of vegetable oil industry effluents are significant due to high Biochemical oxygen demand (BOD) and Chemical oxygen demand (COD) [1-3]. These two parameters reflect the organic content of the effluent and thus its oxygen consumption which is factor militating against aquatic life. In most third world countries including Nigeria industry effluents are discharged directly into water bodies usually without any form of treatment.

Production of refined vegetable oil involves many technological processes such as pre-treatment of oilseeds, refining and modification of oils [4]. These processes require large volumes of water and since the end product does not contain water, waste water commonly called effluent is usually much. The effluent mainly comes from degumming, deacidification and deodorization steps [5] which are part of refining process. Acid splitting is also part of refining process in which sulphoric acid is added to the soap stock causing free fatty acids to be separated from the medium.

The resulting effluent is highly acidic. The composition of VOIE may vary widely from day to day depending on operating conditions, type and source of oil processed. As a result of complexity of wastewater and variations in quantity and characteristic, the choice of wastewater treatment methods depends on many local conditions [6].

Many authors have discussed physical methods like ultra filtration [7] and reverse osmosis [8] in the treatment of waste water. Physicochemical methods which includes precipitation, coagulation, flocculation and flotation [7,9,10] have also been investigated. These have major drawback in their cost and efficiency and so physicochemical methods followed by biological processes [11-13] biological methods [14] and other methods like thermo chemical treatment [15] and photocatalysis [16] for oily wastewater treatment have equally revealed encouraging results. Adsorption technology is considered to be the most effective and proven technology with wide potential applications in both water and wastewater treatment [17,18]. Adsorption hold promise in the treatment of wastewater, as it is convenient, easily operable and simply designed. Sorption is a rapid phenomenon of passive sequestration of sorbet from an aqueous or gaseous phase onto a solid phase [19]. For the removal of pollutants, generally sorbents with high surface area are preferred. Activated carbon has been found to be a versatile sorbent, however its use is being equally limited because of its high cost. As such efforts have been directed towards developing low-cost alternative sorbents form locally sourced materials and vast amount of literature exist [20]. However, studies on the reduction of BOD and COD of vegetable industry effluents are not available to our knowledge. The present paper was therefore to evaluate the possible use of locally produced activated carbon in reducing BOD and COD from vegetable oil industry effluents.

2. MATERIALS AND METHODS

The pH and conductivity were determined according to the method of ASTM D3838-80 with slight modification as follows; 1.0 g of each carbon was weighed and transferred into a 250 ml beaker and 100 ml of distilled water was added and stirred for 1 hour. Samples were allowed to stabilize and then pH measured using an electronic pH/Conductivity meter, Jenway 430 Model. The same samples were further used for electrical conductivity (EC) of the AC_S and results read off in μS [21].

TDS was determined using the electrical conductivity method in which after calibrating the meter with 1000 $\mu S/cm$ standard the TDS mode key was pressed. The probe was again rinsed with some portion of VOIE sample before immersing into sample. The reading displayed was allowed to stabilize before salinity readings were recorded.

All BOD readings of effluent were measured using the Lovibon BOD IR Sensomat. A 10ml sample volume was collected into the 500 ml BOD flask. The IR-pressure sensor was connected to the BOD flask and the start button on the Sensomate depressed. Then the IR sensor was logged into the BOD-Sensomat and reading converted directly to mg/l of BOD

and BOD value read and recorded. COD was determined by use of PCcheckit COD Vario (Lovibond,Germany) consisting of PCcheckit COD Vario photometer and COD reactor ET 108. Briefly, 20 ml effluent sample was put into contact with the acid solution that then held at 148 °C for 2hr. After coning the sample was then placed into sample cell of the PCcheckit COD Vario photometer. The colour of the samples varied from orange to dark green indicating the COD value of the range 15-3800 mg/l.

2. 1. BOD and COD variation with contact time

50 mls of VOIE of BODi and CODi were taken into five conical flasks and 1gs of various PFAC was added and agitated at various time periods of 1, 2, 3, 4, 5 and 6 hrs respective. The solutions were filtered through Whatman no 41, and then centrifuged at 1000 rpm for 5 mins, decanted and their BODs and CODs read off with use of a digital BOD manometer model.

2. 2. BOD and COD variation with carbon dose

This was carried out according to [22,23] with modifications. Briefly, various mass of PFAC in grams from 0.5, 1, 1.5, 2, 2.5 and 3 were weighed and transferred into 250mls conical flask. VOIE of BODi and CODi were placed into the conical flask. The flask were tightly stopped with aluminium foil and agitated for 1hr by centrifugation at 1000 rpm for 5mins, allowed to settle, decanted and filtered with Whatman no. 41. The BOD and COD of filtered samples were measured. This procedure was repeated using FAC_1 to FAC_5.

2. 3. BOD and COD variation with pH in batch Experiment

50 mls of VOIE with a known BOD and COD and respectively were place in five conical flask in which five 1gms of PFAC have been previously weighed and placed. The pHs of the solutions were adjusted with 0.5 m HCl and 0.1 m NaOH solutions to obtain pHs of 2, 4, 6, 8, 10, 12 respectively. The solutions adjusted pHs were then tightly covered with aluminium foil and agitated. At the expiration of 1 hr the solutions were filtered using Whatman No 41 centrifugation at 1000 rpm for 10 mins, decanted and BOD and COD in turns read with a digital BOD manometer.

2. 4. BOD and COD variation with temperature using 1g of PFAC

50 mls of VOIE with BOD_i and COD_i were measured into five 250 mls conical flask in which 1gs of PFAC were previously weighed and placed. The conical flask labelled 10, 20, 30, 40, 50, 60 and 70 degrees centigrade. The flasks were properly covered using aluminium foil, agitated with hand for 2 mins and heated on a thermostatic water bath to their appropriate temperatures. The solutions were centrifuged at 1000 rpm for 5 mins, decanted and then BOD and COD values read using a BOD and COD manometer model.

3. RESULTS AND DISCUSSION

3. 1. Nature of adsorbents

Adsorbents used in this work were prepared elsewhere according to [24]. They were called physically prepared fluted pumpkin activated carbon (PFACs) and were characterized using standard methods. Table1 shows some physico-chemical parameters of vegetable oil industry effluents (VOIE) used. Major characteristics exhibited variations and complexity and

have been fully discussed [20]. Vegetable oil industry effluents are an acidic complex aqueous media composed of widely-distributed organic and inorganic materials dissolved as well as suspended in water. The organic contaminants lead to high BOD and COD.

Table 1. Physico-chemical parameters of palm oil mill effluents.

Parameters	Mean ± SD (mg/l)	WHO	Comment
Temperature	27± 0.01	-	-
pH	4.67 ± 0.015	6-9	Acidic effluent
Cond. (µS/cm)	253 ± 1.15	-	-
Sal (%)	625 ± 4.50	600	Above standard
TDS (mg/l)	127.7 ± 5.77	2000	Above standard
TSS (mg/l)	563.6 ± 3.15	30-50	Above standard
BOD (mg/l)	347I.83 ± 1.70	50 at 20 °C	Above standard
Permanaganate value	16.28 ± 5.50	-	-
TOC (mg/l)	3851.37 ± 4.41	-	-
COD (mg/l)	3959 ± 3.82	250	Above standard
Sulphates (mg/l)	684 ± 5.60	-	-
Phosphates (mg/l)	890 ± 3.50	-	-

Batch and column experiments were conducted and the physicochemical characteristics of effluent measured for each activated carbon sample coded FAC_1, FAC_2, FAC_3, FAC_4 and FAC_5.

3. 2. Resistance factors and removal efficiency of vegetable industry effluents

The resistance factors determined were: change in biochemical oxygen demand (ΔBOD) and chemical oxygen demand ΔCOD, and ratios of BOD/COD for each carbon sample after adsorption for an equilibrium period of six hours [25]. Percent BOD and COD were calculated from equation 1

$$\%BODr = \frac{BODi - BODf}{BODi} \times 100 \dots\dots\dots 1$$

were % BODr is percent BOD reduction, BODi and BODf are initial and final BOD values respectively. A similar expression for COD can be written.

Table 2. Resistance factors of VOIE after adsorption for batch and column systems.

	ΔBOD	ΔCOD	% BOD	% COD	BOD/COD
FAC1 Batch Column	2611	1507	75	79	0.24
	C 2701	4410	78	61	0.28
FAC2 Batch Column	B 1146	1187	61	67	0.29
	C 1180	1270	63	35	0.29
FAC3 Batch Column	B 2002	1577	72	73	0.22
	C 2165	3926	78	65	0.29
FAC4 Batch Column	2002	1613	74	79	0.20
	2661	5210	82	66	0.22
FAC5 Batch Column	3007	1653	83	79	0.21
	3104	5382	86	67	0.19

Table 2 shows resistance factors reported for both batch and column adsorption of BOD and COD of VOIE using 1g of each PFAC. Lowest values for both % BOD (61) and % COD (35) were recorded for FAC_2. Though FAC_5 had similar % COD (79) as FAC_1 and FAC_3, it showed highest values of 83 % and 79 % respectively.

All other values of percent COD removal had no significant difference from each other at $P > 0.05$. The ΔBOD and ΔCOD were significantly different, though the trend was the similar; the ΔCOD was higher than ΔBOD as expected except for FAC_5. The BOD/COD ratio ranged from FAC_4 (0.20) to FAC_2 (0.29), was basically the same and lower than unity. This means that the VOIE treated with PFAC in batch was more biogradable.

When PFACs were used for reducing BOD and COD of VOIE in column process results (table 2) showed that ΔBOD ranged from FAC_2 (1180) to FAC_5 (3104) where as ΔCOD ranged from FAC_5 (5382) to FAC_2 (1270). Percent reduction in BOD ranged from FAC_1 (59) to FAC_5 (86).

The increasing order for preferring to reduce BOD in a column using PFACs was as follows: $FAC_2 > FAC_1 > FAC_3 > FAC_4 > FAC_5$. Except for FAC_5 (0.19), values for BOD/COD for column adsorption using PFACs showed a narrow range. These values are highly recommended for effluent by EHSG standards prior to discharge.

Table 3. Physicochemical properties of effluent after adsorption PFAC in a batch system.

	FAC 1	EAC 2	FAC 3	FAC 4	FAC 5
pH	8.17 ± 0.25	8.10 ± 0.30	6.70 ± 0.26	6.80 ± 0.10	7.07 ± 0.25
Temp °C	26.33 ± 1.15	26.00 ± 1.00	26.00 ± 1.00	24.33 ± 1.52	25.67 ± 0.57
Cond. μS/cm	936.67±21.50	866.33±8.50	867.67±19.50	862.67± 26.00	864.00±15.10
TDS mg/l	310.00 ± 9.00	305.67±6.03	297 ± 9.02	319 ± 15.95	238.67 ± 6.51
D.O mg/l	6.43 ± 0.12	6.70 ± 0.36	5.80 ± 0.20	6.63 ± 0.32	7.23 ± 0.35
BOD mg/l	8.60 ± 0.30	7.33 ± 0.15	7.78 ± 0.42	8.13 ± 0.87	6.03 ± 0.21
COD mg/l	15.07 ± 0.70	11.87 ± 0.65	15.77 ± 0.75	16.13 ± 0.61	16.53 ± 0.65
BOD/COD	0.51 ± 0.03	0.62 ± 0.07	0.44 ± 0.05	0.48 ± 0.55	0.34 ± 0.03

Results of pH ranged from FAC_5 (6.5 ±0.2) in a batch experiment to FAC_4 (9.7 ± -0.5) in a batch It was observed that pH of effluence varied in batch and column experiment for the same AC sample. On the other hand temperature was generally uniform for most samples in batch and column experiments. The fluctuation in pH was reflected in conductivity measurements. This may be explained by proposing that the washing of the ACs may not have been proper and so ionisable substances as impurities may cause the changes in pH and conductivity. These values are however within acceptable limits for effluents from vegetable oil industry [26]. TDS in mg/l ranged from FAC_1 (216 ±12) to FAC_2 and FAC_3 (320 ±10) dissolved oxygen raged from FAC_2 (30 ±5) to FAC1 (40 ±4.0) while the BOD values ranged from FAC4 (508 ±3.0) to FAC_1 (840 ±20) while COD ranged from FAC_2 (2100 ±10) to FAC_1 (3423 ± 33). The biodegradability factor BOD/COD ratio was generally low and ranged from FAC_4 (0.19) to FAC_2 (0.29).

Results reveal that batch experiments enhance the biodegradability of the effluents batch experiment values for biodegradability were in increasing order:

$FAC_4 < FAC_5 < FAC_3 < FAC_1 < FAC_2$,

while column experiment showed the order order:

$FAC_5 < FAC_4 < FAC_3 < FAC_2 < FAC_1$.

From results presented here batch adsorption showed better reduction in organic load of effluents using PFAC.

This could be associated with good development of internal surface usually observed in chemically prepared activated carbon [27]. The batch system therefore could be a better technique for reducing organic pollutants of vegetable oil industry effluents.

3. 3. Factors affecting adsorption of BOD and COD from vegetable oil industry effluents

Three samples of the industrial effluent were collected once a week for five weeks and preserved at 4 °C and then analysed the next day. For contact time, carbon dose, pH and temperature.

3. 4. Contact time variation with BOD and COD

Figure 1 shows that there was a general reduction of BOD effluents with time for all five PFAC while FAC_1 FAC_2 and FAC_4 showed comparable reduction in BOD, FAC_3 and FAC_5 were different with a higher reduction. The order of reduction of BOD as a function of contact time is a follows: $FAC_5 > FAC_3 > FAC_4 > FAC_2 > FAC_1$.
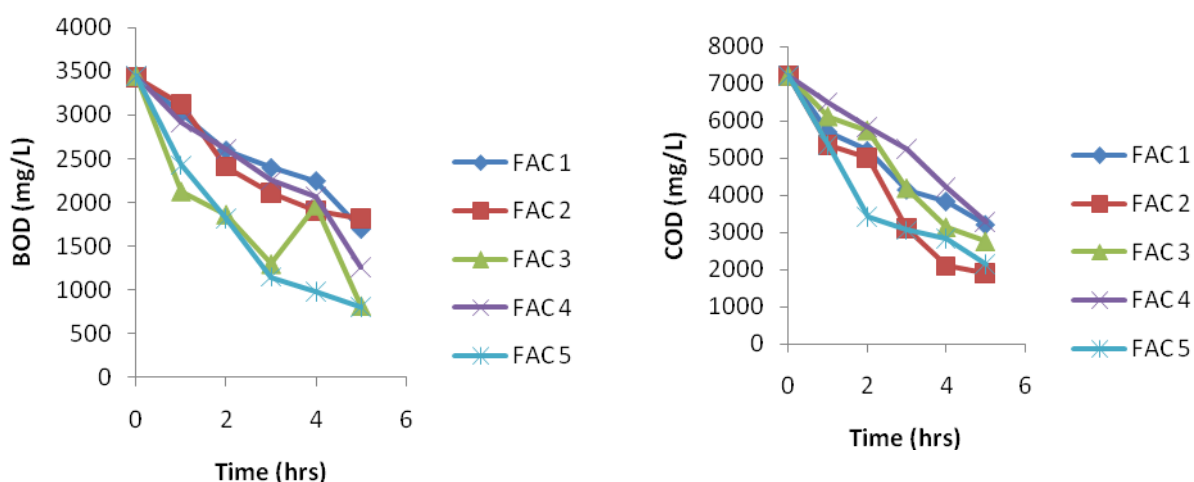
Figure 1. Effect of contact time on BOD of VOIE in a batch system treated with 1 g of PFAC.

There was an anomalous behavior observed for FAC_3 at contact time of 4hours the BOD seem to increase before dropping to a value close to that for FAC_5. There was fast reduction at initial stage i.e. over 1 to 2 hours but the reduction in BOD slowed drastically by the end of the 5th hours. The fifth hours may correspond to equilibrium time. [28] explains that this may be due to the fact that initially a large number of vacant sites were available for adsorption which progressively became smaller with time. Figure 2 shows the effect of contact time on COD for 1gs of FFAC in a batch system in terms of COD. FAC_1 FAC_3 and FAC_4 showed similar trend while FAC_2 and FAC_5 were closer both at initial stage and at the end of five hours as expected occurred at the initial stages most of PFACs. Less reduction from initial stage to the last stage with FAC_4, while FAC_2 showed a higher reduction at the end of the fifth hour. The decreasing order of COD removal using 2 gs of PFAC in a batch with respect to contact time as follows: $FAC_2 > FAC_5 > FAC_3 > F AC_1 > FAC_4$.

The effect of BOD variation with contact time using 1 gs of PFAC is shown graphically in Figure 4.13. FAC_3 and FAC_5 showed higher reduction in BOD both at initial stage and final stage. While FAC_1 and FAC_2 showed anomalies; but it should be noted that all five PFACs showed similar reduction in BOD at the initial stage and again, [28] suggested an explanation to this that more active surfaces available for adsorption at the initial stage and towards the end of the experiment- longer contact time, the active sites are consumed and are less available COD reduced steadily with time for FAC_3 and FAC_5 which shows higher values of cod. FAC_1 and FAC_2 showed less reduction of COD even higher contact time. At five hours

FAC_1 and FAC_2 had very close COD reduction which are lower than for FAC_3, FAC_4 and FAC_5. A lower curve shows better reduction of COD with higher gradient representing good adsorption. The decreasing order of COD with contact time when 1g of PFAC is used for adsorption is as follows: $FAC_3 > FAC_5 > FAC_4 > FAC_2$ and FAC_1.
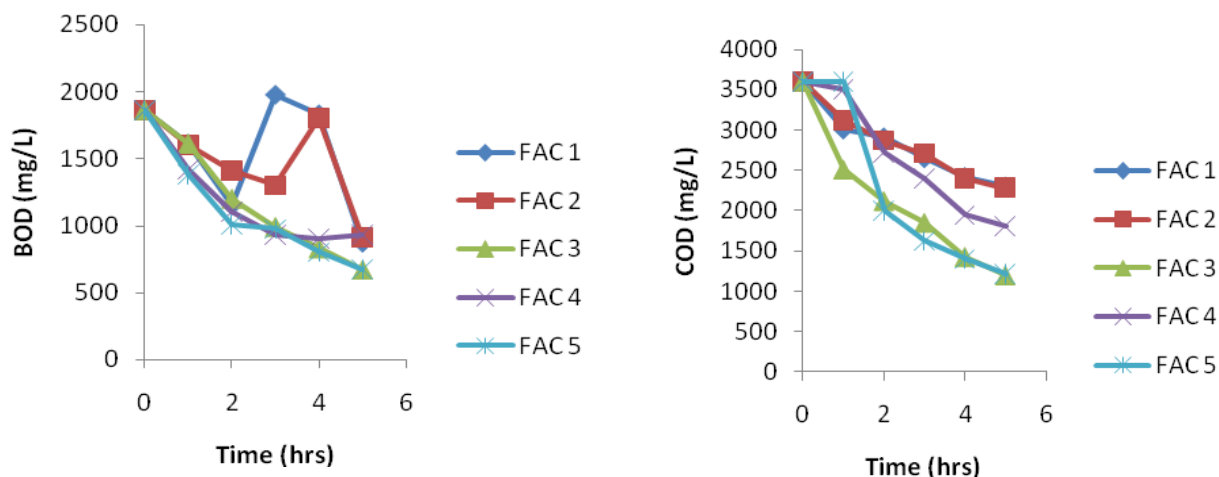

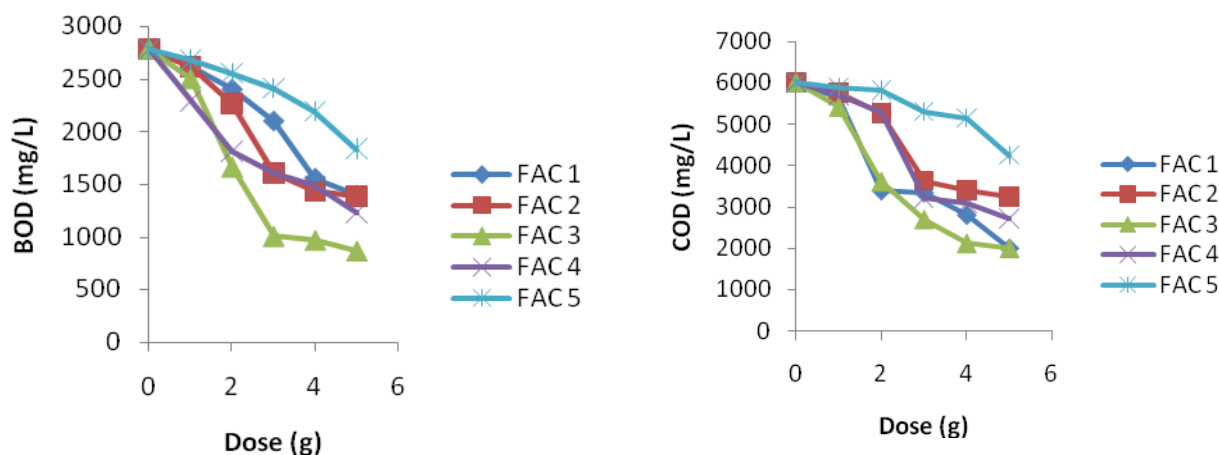**Figure 3.** Effect of contact time on BOD and COD of effluent treated in a column.

Figure 4. Variation of BOD and COD with Carbon does using PFAC in a batch system.

Figure 4 shows the effect of varying carbon dose on BOD in a batch process. Four PFAC showed poor adsorption while FAC_3 showed better adsorption; the adsorption was similar for all PFACs. Higher Carbon dose showed good adsorption. This can be attributed to increase in adsorption surfaces provided by increasing amount of AC. The order of preferred adsorption as Carbon dose of FAC_5 being increased was $FAC_3 > FAC_4 > FAC_2 > FAC_1 > FAC_5$. Increasing Carbon dose from 3g to 5g did not have any effect on FAC_5 even though FAC_5 showed the best adsorption whereas increase carbon dose from 3 g to 5 g for FAC_1 showed good reduction in BOD. After carbon dose of 4g there was no much change in BOD of effluents for most PFAC.

Figure 5 represent the effects of Carbon dose variation on COD of effluent in a batch. FAC_3 and FAC_1 showed better adsorption leading to reduction of COD than all other AC sample preferred by physical activation. The order of preferred AC performance: $FAC_3 > FAC_1 > FAC_4 > FAC_2 > FAC_5$. At all carbon doses, except at 2 g, FAC_3 showed excellent reduction of COD while FAC_5 was poorest at all carbon dose.
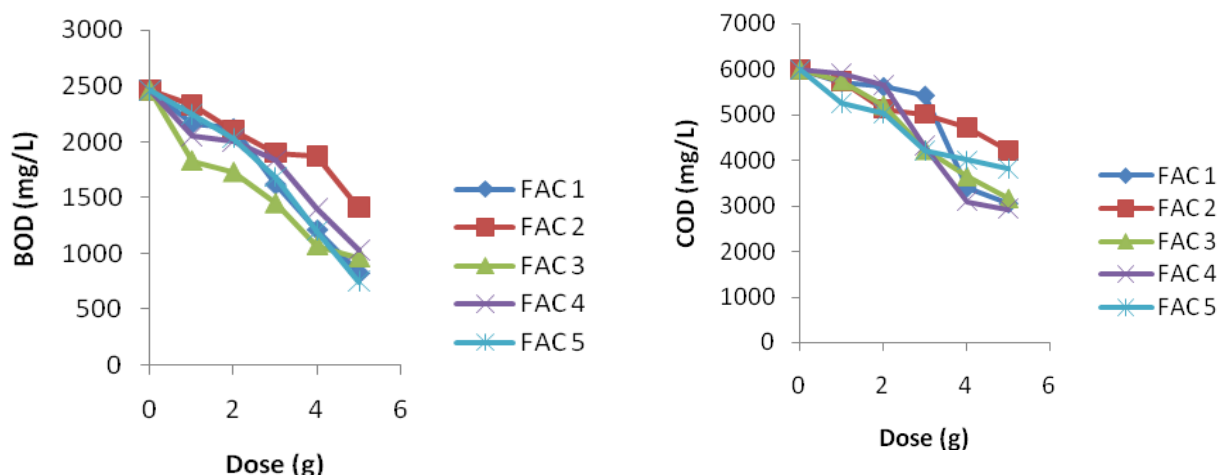

Figure 5. Carbon dose experiment of BOD and COD using PFAC in a column process.

Figure 5 shows the effect of varying Carbon dose on BOD of effluents using PFAC in a column. FAC_5 exhibited highest removal of BOD than all others while FAC_1 was lowest. The reduction on BOD was essentially similar for all PFAC. However the order of decreasing preference of PFAC towards reducing BOD in a column with respect to carbon doses was as follows:

$FAC_5 > FAC_1 > FAC_3 > FAC_4 > FAC_2.$

Generally, as carbon dose increases the number of sorption sites available for organic pollutants increases due to increase in surface area. Several researches have reported this principle [29-31].

Figure 6 shows that at lower carbon dose (1-2 g), the BOD showed little variation for most PFAC but as from Carbon dose of 2 to 4 g there is a sharp decrease in BOD for FAC1 and FAC4 while there is steady decrease of BOD for FAC3 and FAC2 whereas FAC5 showed good BOD reduction at lower values of 1-2 g Carbon dose.

3. 5. pH variation with BOD and COD using 1 g of pfac in batch experiment

Figure 6 represents plots of BOD versus pH for various CFAC in a batch system. The curves shows that reduction of BOD was steady as pH increased from 2 to 9 but starts to level off for FAC_3, FAC_1, and FAC_5, while for FAC_2 and FAC_4 the decrease in pH continued steadily till pH 11. The order of decreasing BOD reduction with respect to pH varying for all PFAC using in batch process was: $FAC_3 > FAC_1 > FAC_2 > FAC_5 > FAC_4$. It should be noted steeper gradients for BOD against pH curves were observed at pH range of 5 to 8 implying that better reduction of BOD was favored within this pH range. Figure 8 shows the variation of COD with pH for 1g of PFAC in a batch system the curves showed that FAC_3 and FAC_4 had the best reduction in COD of VOIE at pH= 11 while FAC_1 was lowest in reducing COD

of VOIE in a batch at some pH = 11 using 2 g of PFAC. At lower pH (2-6), FAC_1 was the best adsorbent followed by FAC_3. At lower pH values the preference for 2 g PFAC in a batch was: $FAC_1> FAC_3> FAC_4> FAC_5> FAC_2,$ whereas at higher pH values (8-11) the order was observed to be:
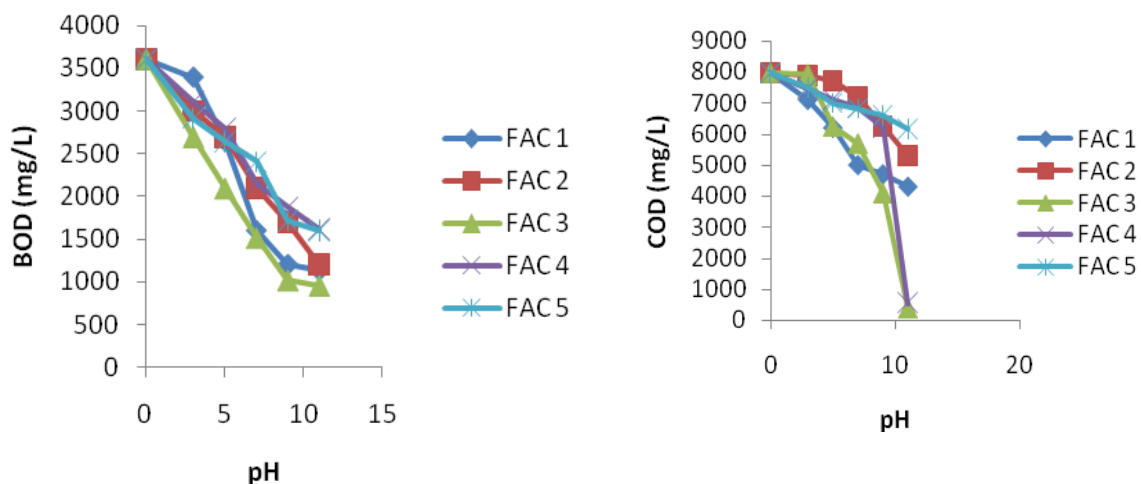
$FAC_3>FAC_4>FAC_1>FAC_2>FAC_5.$

Figure 6. Variation of BOD with pH with 1g of PFAC in a batch process.

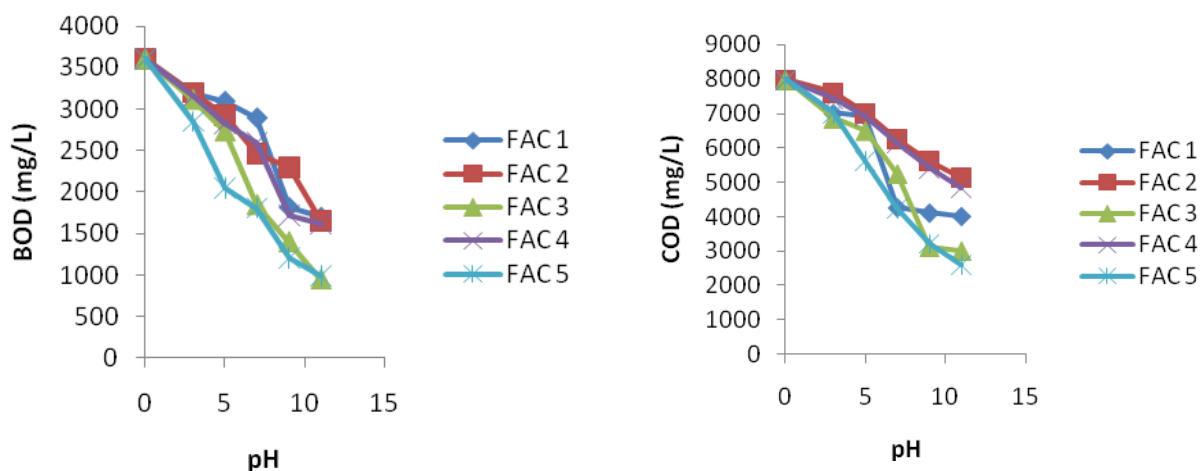### 3. 6. pH variation with bod using 1 g of pfac in column experiment

Figure 9. Variation of BODand COD with pH for 1g of PFAC in a column process.

Figure 9 represent the variation of BOD of VOIE as a function of pH using 1 g PFAC in a column process. At lower pH values (1-4), FAC_1, FAC_2 and FAC_3 show similar behavior, which is reduction of BOD, was almost same but at high pH values (5-11) the reduction in BOD was same for FAC_1 FAC_2 and FAC_4 and same for FAC_3 and FAC_5 with FAC_3 and

FAC_5 showing better reduction than the other three. There was preference for reduction of BOD at higher temperatures while at lower temperature the order of preference was: $FAC_5>$ $FAC_3> FAC_4> FAC_2> FAC_1$. Higher temperatures favored BOD reduction of VOIE using physically prepared activated carbon.

The variation of COD with pH for 1 g of PFAC in a column is shown in figure 4.25. The reduction of COD at lower pH is smaller than for the reduction at higher pH values. There is however a division of reduction potentials of PFAC into three groups. At higher pH values, FAC_4 and FAC_2 showing lower reducing tendencies while FAC_3 and FAC_5 were higher, FAC1 was alone as on intermediate. The general preference for reduction of COD for varying pH values in a column experiment using PFAC was: $FAC_5> FAC_3> FAC_1> FAC_4>$ FAC_2.
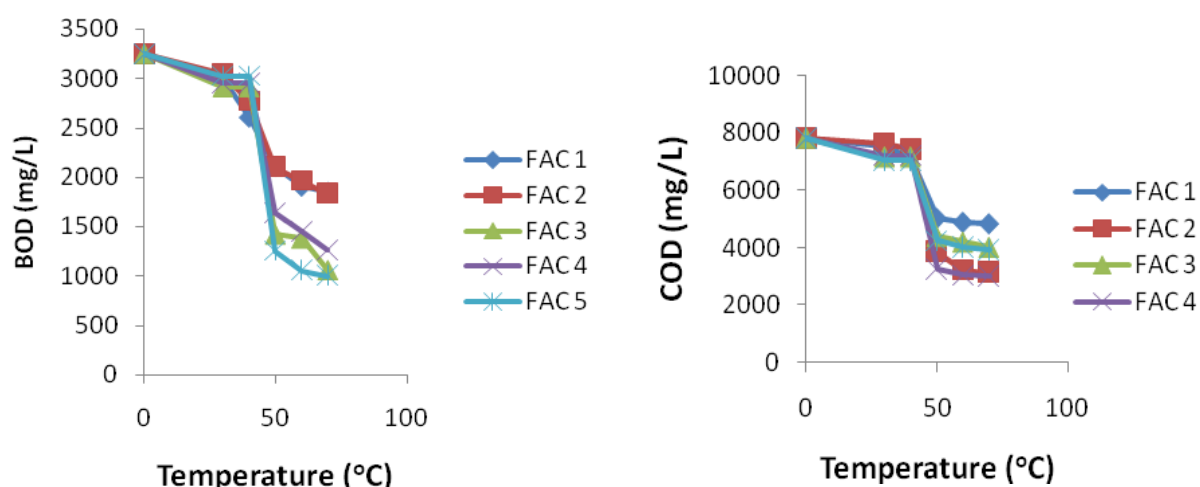

Figure 10. Variation of BOD with temperature using 1 g of PFAC in a batch process.

Figure 10 shows BOD reduction of VOIE as a function of temperature for 1 g of PFAC in a batch system. At lower temperatures 10-30 °C, there was very little reduction in BOD. All values of BOD reduction were very similar at this range. However at above 40 °C, there was a drastic reduction in BOD which again slowed down for all five PFAC above 50 °C at higher temperatures, FAC_2 and FAC_1 showed similar behavior. FAC_5 and FAC_3 behaved similar as well while FAC_4 was intermediate. The order of preference of reduction of BOD using 1 g of PFAC and varying pH showed the following orde:

$FAC_5>FAC_3>FAC_4>FAC_2>FAC_1$.

Figure 11 shows the effect of varying temperature on COD of VOIE using 1 g of PFAC in a batch. The curves behaved similar to those in Figure 4.30 at lower temperature, the reduction in COD was minimal whereas at higher temperature, a huge reduction in COD was observed. Above 50 °C, the PFAC were divided into three groups with FAC_1 in the first group showing lowest reduction in COD; FAC_3 and FAC_5 in the second group showing intermediate reduction and lastly, group three with FAC_4 and FAC_2 showing highest reduction in COD.

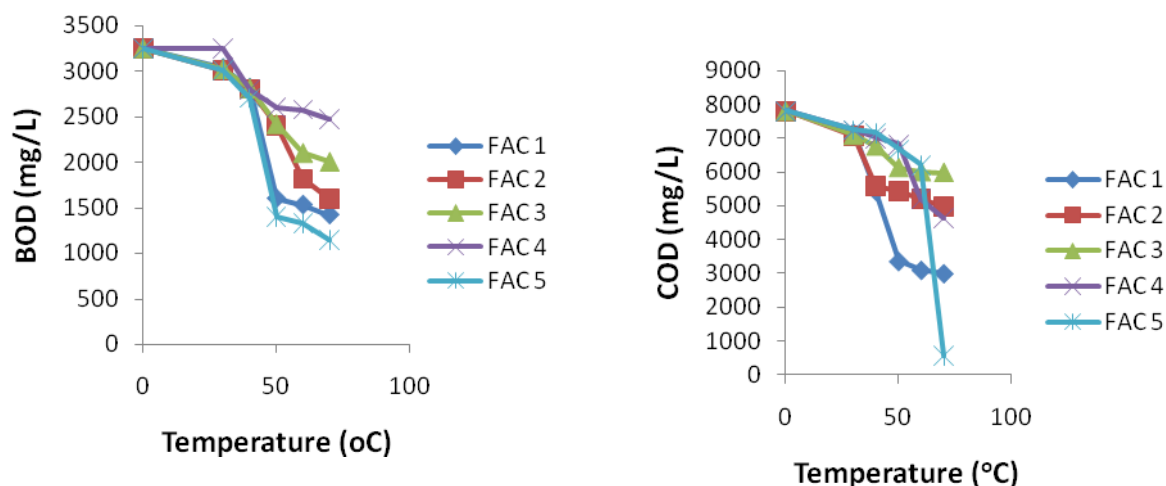## 3. 7. Temperature variation with BOD using 1g of pfac in column experiment

Figure 12. Variation of BOD and COD with temperature using 1g of PFAC in a column process.

Figure 12 represents plots of BOD of VOIE against temperature using 1 g of PFAC in a column experiment. the graphs showed that FAC_3, FAC_2, FAC_5 and FAC_1 were better in BOD reduction than FAC_4 at lower temperature (10-30 °C). Above 40 °C there was an appreciable reduction by all PFAC. The order from highest to least reduction of BOD at higher temperatures is:

$FAC_5 > FAC_1 > FAC_2 > FAC_3 > FAC_4$.

FAC_4 showed lower reduction at both lower and higher temperatures. Figure 13 shows a drastic reduction of COD between 60 °C to 70 °C for FAC_5. This is anomalous as most carbons have shown drastic reduction of COD at temperatures between 40 °C to 50 °C. Figure 13 equally shows that at lower temperatures 10 °C to 30 °C, all PFAC had insignificant reduction in COD. The order of reduction of COD at the temperatures above 40 °C was:

$FAC_5 > FAC_1 > FAC_4 > FAC_3 > FAC_2$.

There was however poor performance by FAC_1 to FAC_4 in reducing COD of VOIE using 1 g of PFAC in a column process.

4. CONCLUSION

All adsorbents showed good BOD and COD reduction though carbons prepared at lower temperatures had poor adsorption capacities. Values of resistance factors were higher for BOD than COD indicating that PFACs could have a positive effect on biodegradability of the effluents. Batch adsorption gave better BOD and COD reduction than column absorption. The maximum BOD reductions were obtained for carbons prepared at higher temperatures in the case of higher carbon doses employed in longer adsorption times. However factors

influencing adsorption of BOD and COD were generally difficult to streamline thus requiring more in depth studies.

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