



Agricultural Wastewater Treatment Using Activated Carbon Produced from Groundnut Husk and Abizia Saman Pod

¹DANIEL, Enebojojo Sunday*; ²EGBUNU, Moses Majiyebo;
³OCHIMANA, Barnabas Akor

¹⁻²Department of Agricultural & Bio-environmental Engineering Technology
Kogi State polytechnic, Lokoja Kogi State Nigeria.

³Joseph Sarwuan Tarka University, Makurdi, Benue State Nigeria.

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*Corresponding Author

Daniel Enebojojo Sunday

E-mail: danielenebojojo@gmail.com

Phone: +2348066034022

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ABSTRACT

Population explosion, haphazard rapid urbanization, agricultural, industrial and technological expansion, energy utilization has rendered many waters unwholesome and hazardous to man and other living resources. Fertilizer application is one of the major activities carried out on farms to improve crop yield, and one of the major fertilizers used is the NPK fertilizer but such activity also causes pollution to the water bodies through runoff or discharge from such agricultural lands. Recently, the removal of N and P from aqueous solutions via adsorption has attracted much attention. Column study was carried out on groundnut husk and albisia saman pods as activated carbon in the removal of N, P and K from agricultural wastewater considering factors such as flow rate (20, 30 and 40 ml/min) and bed height of the column (6, 9 and 12 cm). The physico-chemical characteristics of the adsorbent such as; specific surface area, pH, ash content, organic content, organic matter, bulk density, nitrogen and moisture content were determined. Result from the adsorption experiment showed that the higher the bed height the better the performance of the column. Bed height of 12cm at constant flow rate of 20 ml/min. was observed to give the best result for the two adsorbents. At the bed height of 12 cm, the initial concentration of N, P and K (2.58, 1.47 and 3.28 mg/L) were reduced to 0.02, 0.01 and 0.03 mg/L respectively for the first 10 minutes using groundnut husk activated carbon (GHAC). Also, at the bed height of 12 cm, the concentration of N, P and K were reduced to 0.01, 0.01 and 0.02 mg/L respectively at the first 10 minutes using albisia saman pod activated carbon (ASPAC). However, as the treatment time increases, the performance of the adsorbent was observed to decrease for both adsorbents. The result from the experiment also showed that the flow rate of 20 ml/min at constant bed height of 9 cm gave the best performance. At the flow rate of 20 ml/min, the concentration of N, P and K were reduced to 0.06, 0.05 and 0.11 mg/L respectively using ASPAC while the concentrations of N, P and K at the same flow rate were reduced to 0.07, 0.05 and 0.10 respectively using GHAC. This study showed that activated carbon prepared from both ASPAC and GHAC are suitable for the adsorption of N, P and K and as such could be used as a cost effective adsorbent in the treatment of agricultural wastewater.

INTRODUCTION

Population explosion, haphazard rapid urbanization, agricultural, industrial and technological expansion, energy utilization and waste generation from domestic, agricultural and industrial source have rendered many waters unwholesome and hazardous to man and other living resources. Despite the laws and legislation on environmental pollution in Nigeria, many industries and farms discharge untreated or inadequately treated wastewater into water ways.

Fertilizer application is one of the major activities carried out on farms to improve crop yield, and one of the major fertilizers used is the NPK fertilizer. Without doubt, this bring boost to crop production, but such activity also causes pollution to the water bodies through runoff or discharge from such agricultural lands into water bodies.

Both surface and groundwater are known to contain levels of Nitrogen (N) and Phosphorus (P) in various compounds, which is important for living organisms. Although the levels of concentrations of these compounds is balanced in natural conditions, when their input to waters is greater than living organisms can assimilate, the problem of pollution occurs (Rybicki, 1997). Eutrophication of water bodies

is known to promote aquatic plant growth and may lead to the proliferation of undesirable algae blooms and toxic cyano-bacteria that can pose serious health hazard to humans and livestock (Henderson *et al.*, 2007). To safeguard and protect surface water bodies against the impacts of eutrophication and to maximize the health and environmental benefits associated with the use and discharge of wastewater, several legislations and guidelines, both at national and international levels have been developed (WHO, 2006). In order to meet effluent discharge standards and guidelines, wastewater treatment facilities are obliged to meet discharge consents of nutrients into the environment (Karostynska *et al.*, 2012).

N, P and K removal from wastewater has been widely investigated and several techniques have been developed including adsorption methods, physical processes (settling, filtration), chemical precipitation (with aluminium, iron and calcium salts), and biological processes that rely on biomass growth (bacteria, algae, plants) or intracellular bacterial polyphosphates accumulation (De-Bashan *et al.*, 2004). Recently, the removal of N, P and K from aqueous solutions via adsorption has attracted much attention. The key problem for many N, P and K adsorption methods, however, is finding an efficient adsorbent from several

low-cost or easily available clays, waste materials and by-products.

Low-cost but efficient adsorbents are becoming the focus of many researchers. These adsorbents could be produced from many raw materials such as agricultural and industrial waste. Throughout the world, much research is being conducted on the use of waste materials in order to either prevent an increasing toxic threat to the environment or to simplify present waste disposal techniques by making them more affordable. Among several agricultural wastes studied as adsorbents for the removal of pollutants, is groundnut husk (GH) and albizia saman pods (ASP) which can be of great importance as adsorbents for the removal of different types of pollutants.

Groundnut Shell as Activated Carbon

Groundnut shell, apart from its availability in abundance, has proved to be an excellent raw material for the production of activated carbon. Abdul and Aberuagba (2005) prepared an activated carbon from groundnut shell using steam activation method. It was used excellently to adsorb phosphate from aqueous solution and they recommended the adsorbent for use in waste water treatment. Removal of up to 94.5% of Malachite green on adsorbent from groundnut shell with $ZnCl_2$ activating agent was also recorded. The activated carbon was found to have higher adsorption efficiency compared to commercially available carbon (Malik *et al.*, 2006).

Aminu *et al.* (2010) prepared an activated carbon from groundnut shell for dichlorvos uptake. The highlights of the study show that adsorbent generated from groundnut shell compete well with those reported in literatures. Sorbents generated using the one step

activation method gave less % burn off (high yield), less moisture content and high bulk density than those generated using the two step method. Groundnut shell based biosorbents used in the study are critical for pesticide uptake in solutions. Removal of dichlorvos ranging from 98-100% was achieved by all the Activated carbons prepared.

Albizia Saman Pod

Albizia saman pod is a species of flowering tree in the pea family, Fabaceae that is native to the Neotropics. Its range extends from Mexican south, Africa and south-east Asia, as well as the Pacific Islands, including Hawaii. Common names include saman, rain tree and monkey pod. Saman is a Wide-Canopied tree with large symmetrical crown. It usually reaches a height of 25m and a diameter of 40 cm. The leaves fold in rainy weather and in the evening, hence the name "rain tree" and "five o'clock tree" in Malay. Several lineages of this tree are available, e.g., with reddish pink and creamish golden coloured flowers.

Albizia saman plant grows in the lowland from sea level to 300m with annual rainfall of 600-3000mm. Naturally occurs on savannah and in deciduous forest and riparian corridor.

Mature pods are black-brown, oblong lumpy, 10-30cm long, 15-30mm wide, 6mm thick, straight or slightly curved, not dehiscing but eventually cracking irregularly and filled with a sticky brown pulp.

Although not much research has been done on the use of Albizia saman pod as activated carbon, it is believed that it may be a good precursor for making activated carbon because of its carbonaceous nature. Figure 1a and 1b show the picture of Albizia saman tree and Albizia saman fruit respectively.



Figure 1a: Albizia saman plant



Figure 1b: Albizia saman pod

MATERIALS AND METHOD

Study Area

Olam rice farm Rukubi is the study area which is located in Rukubi town, Doma Local Government Area

of Nasarawa State. It is located at about 30 km South-west of Lafia and 22 km North-west of Makurdi town. The farm is located between Latitudes $7^{\circ}19'28''$ and $7^{\circ}55'45''N$, Longitudes $8^{\circ}18'20''E$ and $8^{\circ}30'56''E$ and has an altitude of 452m above sea level (Google earth, 2016).

Rukubi is one of the communities in Doma Council area of Nasarawa State that is undergoing a silent agricultural revolution. This is courtesy of rice project initiated by Integrated Agro-Firm, Olam Nigeria Limited. This project, which has transformed previously unused lands to huge rice fields, is also helping to curb the problem of unemployment in the state.

The Olam commercial production of rice on 4,000 hectares farm, practices surface irrigation system which supplement rainfall during dry season farming, mills some 200,000 metric tons of paddy annually. Figure 2 presents the map of Nasarawa state showing the study area.

Preparation of Adsorbent

Precursor

The precursors used for the preparation of activated carbon in this study are groundnut husk (GH) and Albizia saman pod (ASP). The fresh GH was obtained from NorthBank Market in Makurdi, Benue State while the ASP was collected from Federal Housing Estate, Northbank, Makurdi, Benue State, Nigeria. The samples were washed with tap water and then distilled water to remove impurities, dried at 110°C for 12 hours and crushed with mortar and pestle. The crushed particles were then sieved to obtain the particle sizes of 1- 3 mm.

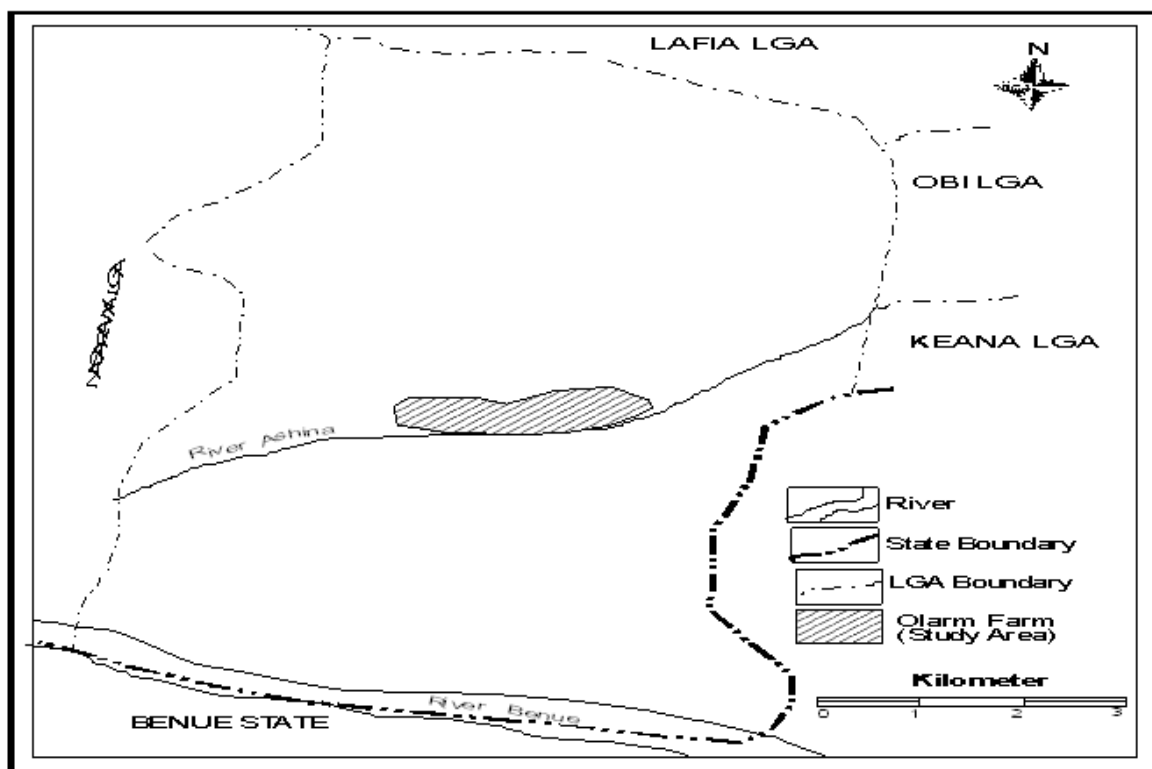


Figure 2: Map of Nasarawa State showing the Study Area

Source: Extracted from Google earth, 2016.

Activation/ Carbonization

The GH and ASP particles were then mixed with ZnCl_2 and heated in a burner for 30 min to activate the carbon and allowed to settle. The activated materials were then left soaked in the ZnCl_2 overnight (about 12 hours) to enable full activation after which they were washed several times with distilled water and then sun-dried.

The carbonization was done in the advance physic Laboratory, University of Agriculture, Makurdi, Benue State using Digital Furnace. The activated pods granular were taken to the furnace where they were heated at temperature of 600°C for 2 hours for carbonization.

Characteristics of Absorbents

i) Particle size

The particle size was determined by passing the crashed precursor through a set of sieves. Particles retained on sieve 1-3 mm were used as the adsorbents.

ii) Bulk density

The particle bulk density of ASPAC and GHAC were determined using Ahmedna (1997). Procedure as follows: an empty measuring cylinder was weighed and noted. The cylinder was filled with the samples of the activated carbon and gently tamped until no more change in the level of the sample in the measuring cylinder was noted. The volume occupied by the packed sample was recorded and noted. The bulk density was then calculated using equation 1

$$\text{Bulk Density} = \frac{W_2 - W_1}{v} \quad (1)$$

Where:

W_1 = Weight of empty measuring cylinder

W_2 = Weight of cylinder filled with sample

V = Volume of cylinder

iii) Moisture content

Small amount of activated carbon sample (GH and ASP) weight was measured and then taken in a petri dish. It was spread nicely on the dish. It was then heated in an oven at a temperature of (105-110°C) for 1.5 hours. The petri dish was left open or not covered during heating process. After heating petri dish, it was then removed and cooled in a desiccator to obtain the weight. The moisture content was calculated using equation 2.

$$\text{Moisture content} = \frac{100(B-F)}{B-G} \quad (2)$$

Where:

B = weight of petri dish + original sample

F = weight of petri dish + dried sample

G = weight of petri dish

iv) Ash content

This was calculated using equation 3

The ash content A_c is given in % by:

$$A_c = \frac{100(F-G)}{B-G} \quad (3)$$

Where:

G = mass of empty crucible in g

B = mass of crucible plus dried sample in g

F = mass of crucible plus ash sample in g

v) pH

The determination of pH of the samples were determined by weighing 1 g each of GH and ASP activated carbon, boiled in a beaker containing 100 cm³ of distilled water for 5 min, the solution was then diluted to 200 cm³ with distilled water and cooled at room temperature, the pH of each was measured using a pH meter (model ATPH-6) and the readings were recorded (Abdel-Halim *et al.*, 2006).

vi) Specific surface area

The specific surface area measurements (m²/g) of the activated carbon samples were made by low temperature nitrogen adsorption, by BET equation (4, 5) (Brunauer *et al.*, 1938) at advance physics laboratory of University of Agriculture Makurdi using Micromeritics (ASAP, 2010) operated at 77 K.

$$S_{\text{total}} = \frac{v_m N s}{V} \quad (4)$$

$$S_{\text{BET}} = \frac{S_{\text{total}}}{a} \quad (5)$$

Where:

S_{total} = total surface area;

S_{BET} = specific surface area;

V_m is the unit of the molar volume of the adsorbate gas;

N = Avogadro's number;

s = the adsorption cross section of the adsorbing species;

V = the molar volume of the adsorbate gas;

a = the mass of the solid sample or adsorbent.

vii) Organic carbon

A representative sample was grinded and passed through 2mm sieve. The activated carbon samples were weighed out in duplicate for each pod and transferred to 250 ml Erlenmeyer flask. 10 ml of 1 NK₂Cr₂O₇ solution was pipetted accurately into each flask for each pod and swirled gently to disperse the activated carbon. 20 ml of concentrated H₂SO₄ was then added rapidly using an automatic pipette directing the stream into the suspension. Immediately, the flask was swirled gently until activated carbon and reagents were mixed, and swirled vigorously for one minute. The beaker was rotated again and the flask was allowed to stand on a sheet of asbestos for about 30min. 100 ml of distilled water was then added after standing for 30 mins. Three drops of indicator was added and titrated with 0.5 N ferrous sulphate solutions. The %organic Carbon in activated was calculated according to the formula:

$$\% \text{Organic C} = \frac{(\text{meK}_2\text{Cr}_2\text{O}_7 - \text{meFeSO}_4) \times 0.003 \times 100 \times (f)}{\text{g of air-dry soil}} \quad (6)$$

f = Correction factor = 1.33

me = Normality of solution x ml of solution used

ix) Organic matter

Organic matter in activated carbon was evaluated as follows:

$$\% \text{Organic Matter in activated carbon} = \% \text{Organic C} \times 1.729 \quad (7)$$

Wastewater Sampling and Analysis

Wastewater samples were collected from a discharge canal at Olam Rice Farm, in Rukubi, Nassarawa State, Nigeria. Collection of wastewater samples were done for three weeks, once in a week at the discharge points of the canal. Collection of samples was done using a clean plastic container which was dipped inside the wastewater flow channel to draw water. The samples were then taken to the Advanced laboratory of Makurdi mega water works (Water Board) Makurdi, Benue State, where it was analysed for N,P and K. Water parameters such as Total Solid, Total Dissolved Solid,

pH, Temperature, and turbidity were also tested for in the wastewater.

Adsorption Test

Continuous flow adsorption experiments were conducted; the reactor setup (Figure 4) used in this study was constructed of pyrex plastic tube of 30 cm height, and 2 cm internal diameter. The column was made in a methacrylate cylinder, thus allowing for visual examination of the progress of the wetting front and detection of preferential flow channels along the column walls. At the bottom of the column, a 0.5mm stainless steel sieve was attached followed by glass wool. Known quantities of adsorbent were placed into the column to obtain the bed heights of 6 cm, 9 cm and 12 cm at different occasions. Wastewaters of known concentrations were introduced downward into the column bed by gravity using flow valve/tape to regulate the flow rate.

The flow was set at constant flow rate of 20 ml/min for the bed height of 6 cm, 9 cm and 12 cm of ASPAC adsorbent to determine the effect of column bed depth on the treated effluents. The experiment was repeated for GHAC adsorbent following the same procedure. The flow valve was then adjusted to flow rate of 30 ml/min and then 40 ml/min at a constant bed height of 9 cm of ASPAC adsorbent to determine the effect of flow rate on the treated effluent. The same procedure was also repeated for GHAC adsorbent.

Samples were collected at the column outlet at 10 minute intervals and were analysed for N, P and K and some water parameters (turbidity, total suspended solid, total dissolved solids and total solids) concentration by a UV-Vis spectrophotometer (Hach DR/2000).

Kinetic models

Two kinetic models were used to predict the performance of the adsorbents for the adsorption of NPK in the column.

i) Thomas – BDST Model

The expression by Thomas for an adsorption column is given as follows (Baek *et al*, 2007).

$$\frac{C_e}{C_o} = \frac{1}{1 + \exp\left[\frac{K_T}{Q}(qM - C_o V)\right]} \quad (1)$$

The linearized form of the Thomas model is given in equation (19) (Kavak and Öztürk, 2004):

$$\ln\left(\frac{C_o}{C_e} - 1\right) = \frac{K_T q_o M}{Q} - K_T C_o T \quad (2)$$

Where

C_o , C_e = the effluent and inlet solute concentrations (mg/l) respectively,

q_o = the maximum adsorption capacity (mg/g),

M = the total mass of the adsorbent (g),

Q = volumetric flow rate (ml/min),

T = breakthrough time and

K_T = the Thomas rate constant (ml/min/mg).

ii) Yoon and Nelson model

The Yoon and Nelson equation regarding to a single component system is expressed as (Aksu and Gönen, 2004):

$$\frac{C_e}{C_o} = \frac{1}{1 + \exp[K(\tau - t)]} \quad (3)$$

Where

k is the rate constant (l/min),

τ is the time required for 50% adsorbate breakthrough (min) and

t is the breakthrough (sampling) time (min).

The linearized form of the Yoon and Nelson model is as follows:

$$\ln\left(\frac{C_e}{C_o - C_e}\right) = Kt - \tau K \quad (4)$$

Design of Full Scale Adsorption Column

The design of the full scale adsorption column for removal of N, P and k was done using scale up method.

Data Analysis

Data collected from the experiment was subjected to the analysis of variance (ANOVA) using Genstat Discovery edition 4 Statistical Software at 5% level of probability. Where significant differences exist among or between means, means were separated using Fischer's least significant difference (F-LSD).

Graph showing breakthrough curves of the treated constituents were also generated using Microsoft Excel.

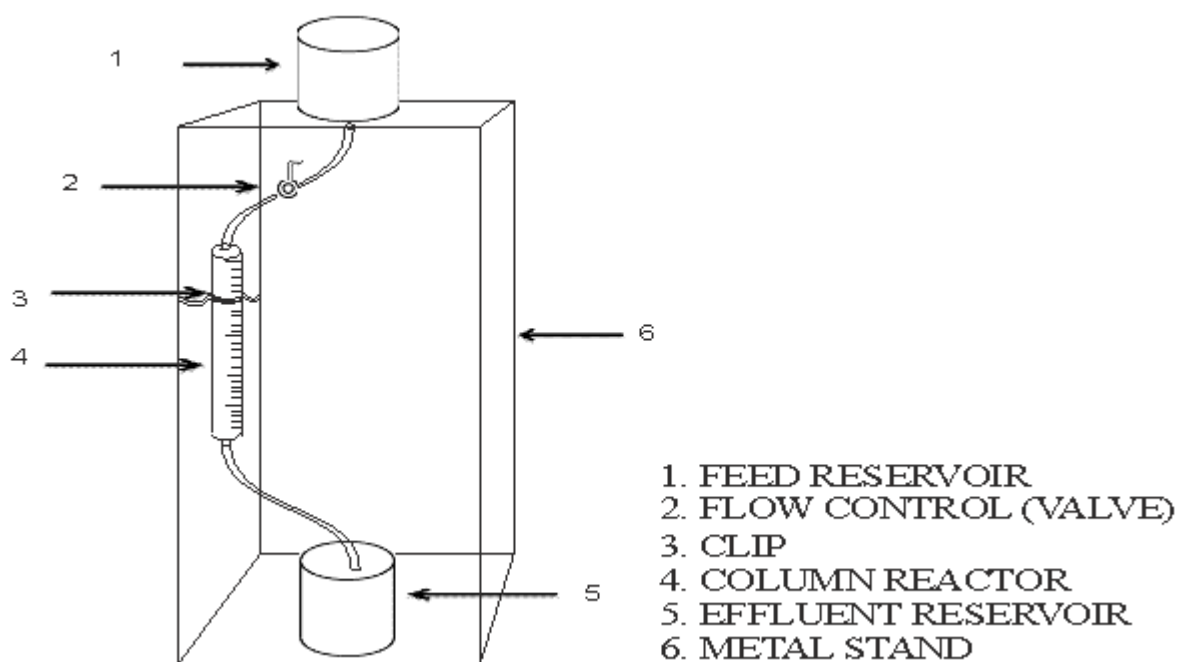


Figure 3: Schematic diagram of fixed bed column experimental set up



Figure 4: fixed bed column experimental set up

RESULTS

Characterization of the Adsorbents

The physico-chemical properties of GHAC (Groundnut Husk Activated Carbon) and ASPAC (Albizia Saman Pods Activated Carbon) adsorbents are presented in Table 1.

Wastewater Analysis.

The analysis of the wastewater showing the concentration of N, P, K, turbidity, total dissolved solids (TDS), total suspended solids (TSS) and total solids

(TS) in the influent (before treatment) and in the effluent (after treatment) with ASPAC and GHAC in the column were presented. The analysis using different bed heights of 6 cm, 9 cm and 12 cm of ASPAC and GHAC in the column respectively. Also, the analysis using different influent flow rate of 20 ml/min, 30ml/min and 40ml/min on a constant column bed height of 9 cm of ASPAC and GHAC respectively. From the tables, the initial concentration of water quality parameter, that is; turbidity, TDS, TSS and SS were 575 NTU, 72.4, 535 and 604 respectively, while the concentration of the pollutants; N, P and K were 2.58, 1.47 and 3.28mg/L respectively.

Column Adsorption Experiments

Continuous flow adsorption experiments were conducted to study the adsorption behaviour of fixed beds on ASPAC and GHAC. All the experiments were conducted at constant conditions. The behaviour of the column was investigated through the concentration of the effluent taken at every ten minute intervals of treatment and also through the analysis of the breakthrough curves of the experiments in terms of C_e/C_o versus time in minutes for N, P and K.

Table 1: Physico-chemical Properties of GH and ASP Adsorbents

Parameters	GH	ASP
Specific Surface Area (m ² /g)	789	712
Ph	6.13	6.60
Ash Content (%)	0.48	0.51
Organic Content (%)	4.28	4.31
Organic Matter (%)	7.40	7.45
Bulk Density (g/cm ³)	0.39	0.38
Nitrogen (%)	2.04	1.96
Moisture Content (%)	2.32	2.34

Effect of bed height

The effect of column bed height on the concentration of the effluent was investigated using bed height of 6 cm, 9 cm and 12 cm at constant flow rate of 20 ml/min. Table 2 and 3 shows the effect of various bed heights on the treatment of the wastewater (effluent) using ASPAC and GHAC respectively. The tables show

clearly that significant difference exist along the different bed heights.

The effect of bed height was also investigated for Nitrogen (N), Phosphorus (P) and Potassium (K) onto ASPAC and GHAC using breakthrough curves. The experimental breakthrough curves for the effect of bed heights on the adsorption of N, P and K onto ASPAC are presented on figure 5, 6 and 7 respectively while figure 8, 9 and 10 shows the breakthrough curves for the adsorption of N, P and K respectively at different bed heights of 6cm, 9cm and 12cm onto GHAC at a constant flow rate of 20ml/min.

Effect of Flow Rate

The effect of influent flow rate on the concentration of the effluent was investigated using flow rate of 20 ml/min, 30 ml/min and 40 ml/min at constant bed height of 9 cm. Table 4 and 5 shows the effect of various flow rate on the treatment of the wastewater (effluent) using ASPAC and GHAC respectively. The tables show clearly that significant difference exist along the various flow rates.

Also breakthrough curves were used to investigate the effect of flow rate on the adsorption of Nitrogen (N), Phosphorus (P) and Potassium (K) onto ASPAC and GHAC. The experimental breakthrough curves for the effect of flow rate on the adsorption of N, P and K onto ASPAC are presented on figure 11, 12 and 13 respectively while figure 14, 15 and 16 shows the breakthrough curves for the adsorption of N, P and K respectively at different flow rate of 20 ml/min, 30 ml/min and 40 ml/min onto GHAC at a constant bed height of 9 cm.

Table 2: Effect of ASPAC Bed Height on Concentration of Effluent Constituents

Bed Height (cm)	N	P	K	TURBIDITY	TDS	TSS	TS
6	1.914 ^a	1.1200 ^a	2.417 ^a	361.8 ^a	50.62 ^a	328.8 ^a	392.7 ^a
9	1.546 ^b	0.9617 ^b	2.036 ^b	268.5 ^b	42.45 ^b	229.6 ^b	264.8 ^b
12	1.051 ^c	0.6864 ^c	1.338 ^c	187.9 ^c	31.33 ^c	183.6 ^c	215.8 ^c
LSD(P≤0.05)	0.0778	0.03095	0.0979	32.92	2.374	25.18	30.79

Table 3: Effect of GHAC Bed Height on Concentration of Effluent Constituents

Bed Height (cm)	N	P	K	TURBIDITY	TDS	TSS	TS
6	1.972 ^a	1.1572 ^a	2.544 ^a	361.6 ^a	52.5 ^a	329.7 ^a	382.2 ^a
9	1.654 ^b	0.9817 ^b	2.113 ^b	289.8 ^b	44.7 ^b	250.2 ^b	294.9 ^b
12	1.211 ^c	0.7147 ^c	1.542 ^c	222.9 ^c	36.7 ^c	197.3 ^c	231.6 ^c
LSD(P≤0.05)	0.0830	0.03397	0.1009	31.20	7.04	26.00	25.05

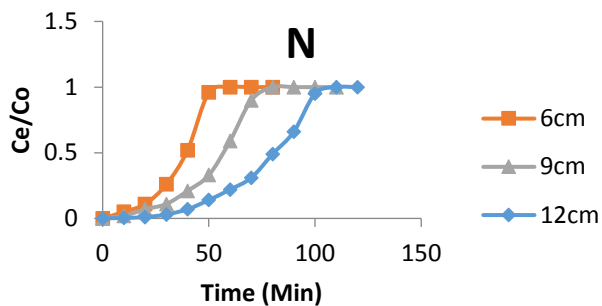


Figure 5: Breakthrough Curve for N Adsorption at Different Bed Heights of ASPAC (Flow Rate at 20ml/min)

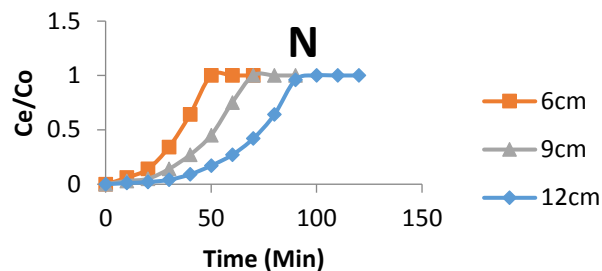


Figure 8: Breakthrough Curve for N Adsorption at Different Bed Heights of GHAC (Flow Rate at 20ml/min)

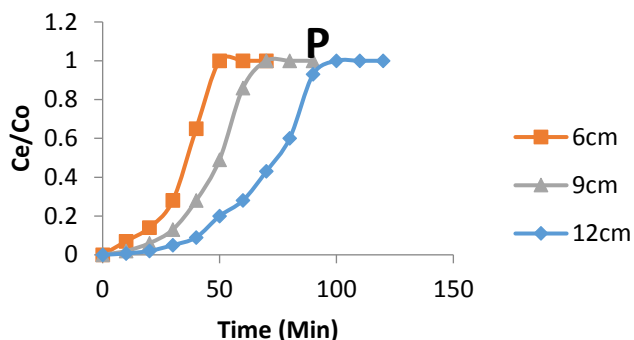


Figure 6: Breakthrough Curve for P Adsorption at Different Bed Heights of ASPAC (Flow Rate at 20ml/min)

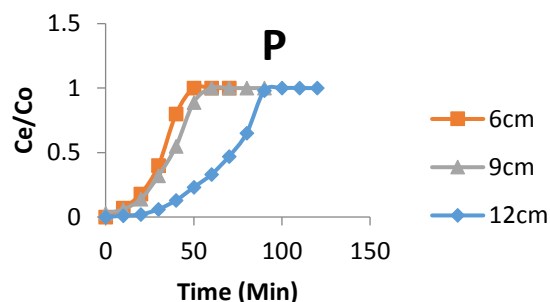


Figure 9: Breakthrough Curve for P Adsorption at Different Bed Heights of GHAC (Flow Rate at 20ml/min)

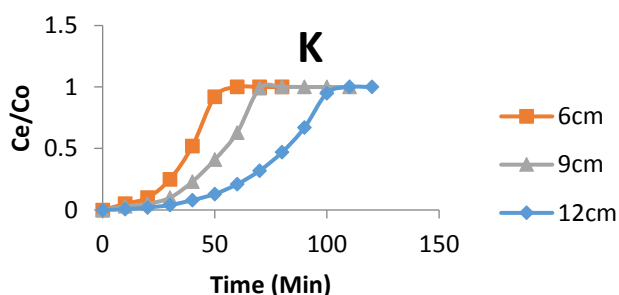


Figure 7: Breakthrough Curve for K Adsorption at Different Bed Heights of ASPAC (Flow Rate at 20ml/min)

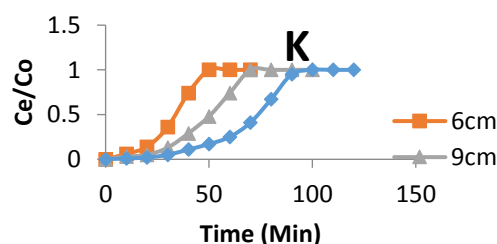


Figure 10: Breakthrough Curve for K Adsorption at Different Bed Heights of GHAC (Flow Rate at 20ml/min)

Table 4: Effect of Flow rate on Effluent Concentration using ASPAC.

FLOW RATE (ml/min)	N	P	K	TURBIDITY	TDS	TSS	TS
20	1.546 ^c	0.9617 ^c	2.036 ^c	268.5 ^c	42.45 ^c	229.6 ^c	264.8 ^c
30	1.919 ^b	1.1117 ^b	2.438 ^b	357.9 ^b	51.59 ^b	340.0 ^b	391.6 ^b
40	2.157 ^a	1.2692 ^a	2.769 ^a	408.2 ^a	59.56 ^a	377.3 ^a	436.9 ^a
LSD(P≤0.05)	0.0935	0.03434	0.1159	38.23	1.956	30.67	34.47

Table 5: Effect of Flow rate on Effluent Concentration using GHAC.

FLOW RATE (ml/min)	N	P	K	TURBIDITY	TDS	TSS	TS
20	1.654 ^c	0.9817 ^c	2.113 ^c	289.8 ^c	44.68 ^c	250.2 ^c	294.9 ^c
30	1.992 ^b	1.1789 ^b	2.561 ^b	373.2 ^b	52.82 ^b	347.4 ^b	400.2 ^b
40	2.132 ^a	1.2800 ^a	2.820 ^a	414.7 ^a	60.44 ^a	379.1 ^a	439.5 ^a
LSD(P≤0.05)	0.0809	0.03493	0.1157	39.69	1.985	30.58	29.40

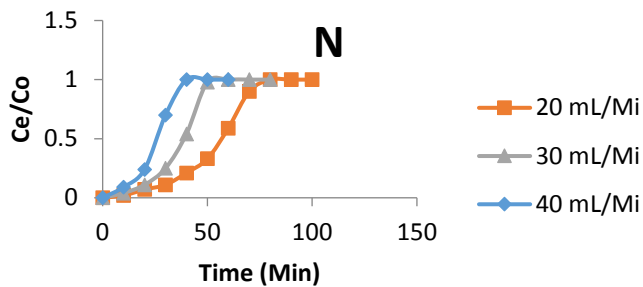


Figure 11: Breakthrough Curve for N Adsorption at Different Flow Rate of ASPAC (Bed Height of 9cm)

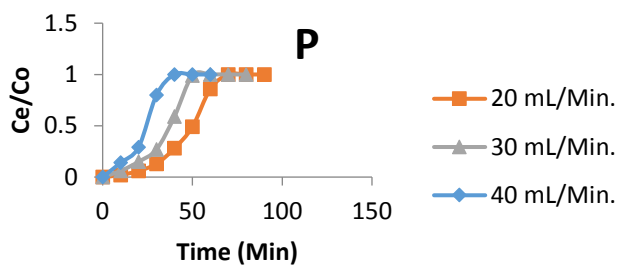


Figure 12: Breakthrough Curve for P Adsorption at Different Flow Rate of ASPAC (Bed Height of 9cm)

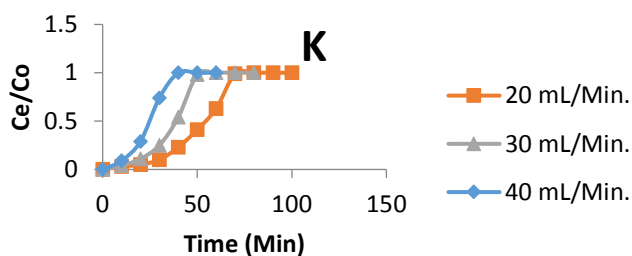


Figure 13: Breakthrough Curve for K Adsorption at Different Flow Rate of ASPAC (Bed Height of 9cm)

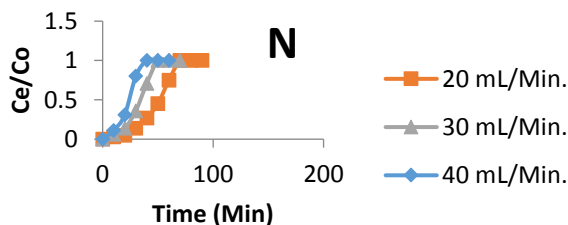


Figure 14: Breakthrough Curve for N Adsorption at Different Flow Rate of GHAC (Bed Height of 9cm)

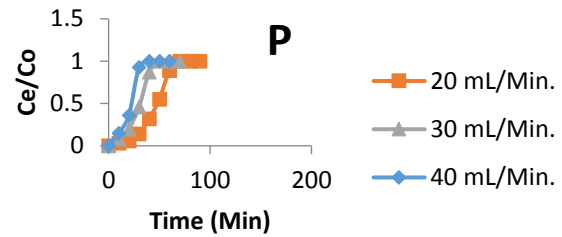


Figure 15: Breakthrough Curve for P Adsorption at Different Flow Rate of GHAC (Bed Height of 9cm)

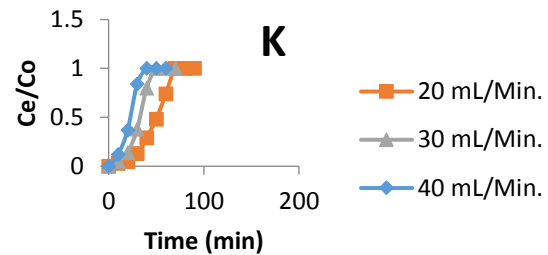


Figure 16: Breakthrough Curve for K Adsorption at Different Flow Rate of GHAC (Bed Height of 9 cm)

Design of Full Scale Adsorption Column

This design of the full scale adsorption column was carryout using N adsorption on ASPAC of 12 cm bed height and 20 ml/min rate. The same procedures can be repeated to obtain the design for the other pollutants (P and K). The packed column parameters are the assumed design parameters used for the design.

The design of packed column using scale-up procedure

Experimental Data (laboratory column test experiment)

- Column Diameter (d) = 2 cm (0.02 m),
- Bed Height (H) = 12 cm (0.12 m),
- Flowrate (Q) = 20ml/min (1.2 l/h),
- Packed carbon density = 0.39 g/cm³ (390 kg/m³),
- Breakthrough time = 25 minute,
- Exhaustion time = 72 minute,
- mass of carbon = 14.97 g (0.015 kg),
- Initial concentration of N = 2.58 mg/l

Packed column parameters

- Wastewater flowrate = 80 m³/d
- Unit flowrate of 1.6 l/s.m²

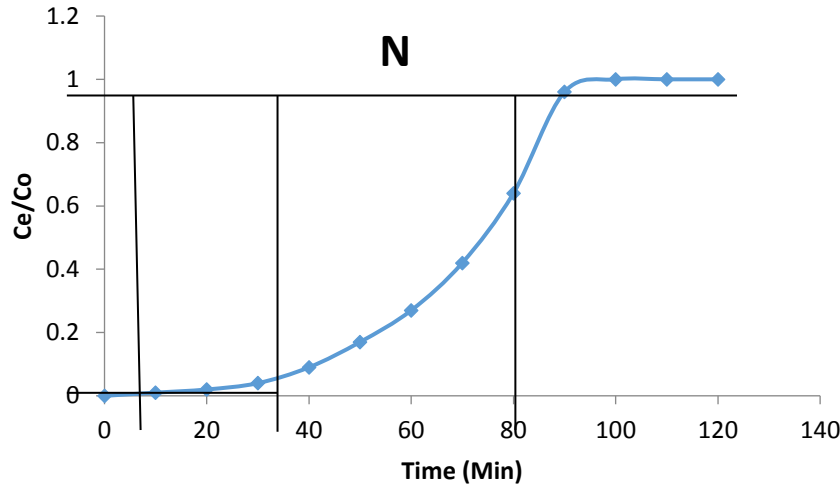


Figure 17: Design Breakthrough Curve for N Adsorption at 12 cm bed height and 20 ml/min flowrate of GHAC for the laboratory column

a) Filtration rate of the laboratory column (FR)

$$FR = \frac{Q}{A}$$

$$= 1.2L/hr \times \frac{1}{3.142 \times \left[\frac{2cm}{2}\right]^2} \times \frac{1000cm^3}{L}$$

$$= 382 \frac{cm^3}{hr.cm^2}$$

The same FR applies to packed column

b) Area of the packed column

$$FR = \frac{Q}{A}, \quad A = \frac{Q}{FR}$$

$$A = \left[80 \frac{m^3}{d}\right] \times \left[\frac{1}{382} \cdot \frac{hr.cm^2}{cm^3}\right] \times \left[\frac{1d}{24hr}\right] \times \frac{10^6 cm^3}{1m^3}$$

$$= 8726 cm^2$$

$$d = \sqrt{\frac{4 \times 8726}{\pi}} \quad A = \pi \left[\frac{d}{2}\right]^2$$

$$= 105 cm$$

c) Empty bed contact time of the laboratory column (τ)

$$\tau = \frac{\text{volume (V)}}{\text{flowrate (Q)}}, \quad V = A \times H = \pi \left[\frac{d}{2}\right]^2 \times H$$

$$= 3.142 \times \left[\frac{2}{2}\right]^2 \times 12$$

$$= 37.7 cm^3 = 0.038L$$

$$\text{Therefore, } \tau = \frac{0.038L}{1.2L/hr}$$

$$= 0.03 hr = 2 min$$

2 minutes is the EBCT of the packed column (the same as laboratory column)

d) Height of the packed column

$$H = \frac{\tau Q}{A}, \quad FR = \frac{Q}{A}$$

$$H = \tau \times FR$$

$$= 2 min \times 382 \frac{cm^3}{hr.cm^2} \times \frac{1hr}{60 min}$$

$$= 12.7 cm = 0.13 m$$

The same as the height of the laboratory column because height is set by τ and $\frac{Q}{A}$, and these are the same for both laboratory column and packed column.

e) Mass of carbon required the packed column

$$\text{Volume of packed column (V)} = H \times A$$

$$= 8726 cm^2 \times 13 cm = 113438 cm^3 = 0.113 m^3$$

$$\text{Packed bed carbon density} = 0.39 g/cm^3 = 390 kg/m^3$$

$$M = \text{Volume} \times \text{density}$$

$$= 0.113 m^3 \times 390 kg/m^3$$

$$= 44 kg$$

f) Determination of q_e

Volume of N treated at breakthrough for laboratory column using the breakthrough curve

$$V = Q \times \text{Time}$$

$$= 0.02 \frac{L}{min} \times 44 \text{ min} = 0.88 L$$

Volume of N treated at breakthrough for laboratory column using the breakthrough curve

$$= 0.02 \frac{L}{min} \times 91 \text{ min} = 1.82 L$$

N removed by 0.015kg C.

$$= C_o \times \text{volume treated at exhaustion}$$

$$= 2.58 \frac{mg}{L} \times 1.82 L = 4.7 \text{ mg}$$

$$q_e = \frac{4.7 \text{ mg}}{0.015 \text{ kg}} = 0.313 \frac{mg}{g} C$$

g) Fraction of capacity left unused (laboratory column)

Total capacity = 4.7 mg

N removed before breakthrough

$$= 0.88 L \times 2.58 \text{ mg/L} = 2.3 \text{ mg}$$

Fraction of capacity left unused

$$= \frac{4.7 \text{ mg} - 2.3 \text{ mg}}{4.7 \text{ mg}} \times 100\% = 51\%$$

This fraction of capacity left used will be applied to the packed column also.

h) Breakthrough time of the packed design column

$$\text{Organic loading} = \left[2.58 \frac{mg}{L} \right] \times \left[80 \frac{m^3}{d} \right] \times \left[\frac{1000 L}{1 m^3} \right]$$

$$= 2.1 \times 10^5 \text{ mg/d}$$

Carbon consumption rate

$$= 2.1 \times 10^5 \text{ mg/d} \times \frac{1}{0.313 \frac{mg}{g} C}$$

$$= 671 \text{ kg/d}$$

$$\text{Amount of carbon consumed} = 44 \text{ kg} \times (1 - 0.51)$$

$$= 21.6 \text{ kg}$$

$$\text{Breakthrough time} = \frac{21.6 \text{ kg}}{671 \frac{kg}{d}} = 0.032 \text{ day} = 46.4 \text{ min}$$

i) Volume treated before breakthrough

$$V = 80 \frac{m^3}{d} \times 0.032 \text{ days} = 2.56 \text{ m}^3$$

IV DISCUSSION OF RESULTS

Characteristics of Adsorbents

Specific surface area

The surface area as a fundamental parameter used in the characterization of activated carbon, is the measure of the microspores content of the activated carbon. The microspores are key factor for the large surface area for activated carbon particles and are mostly created during the activation process. It was noted that ASPAC (789 m²/g) produced higher value of surface area than GHAC (712 m²/g) which is responsible for higher performance in the treatment of wastewater as showed in Table 1. AWWA (1991) specified that activated carbons with surface area ranging from 600-1100 m²/g are recommended for use in the treatment of water or wastewater.

pH

The Potential of Hydrogen (pH) is an important parameter for adsorption of ions from aqueous solution because it affects the solubility of the ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbate during reaction. The pH of ASPAC and GHAC were 6.60 and 6.13 respectively; the GHAC showed to be slightly more acidic than ASPAC. It was observed that both ASPAC and GHAC could be acceptable in the applications involving adsorption since they showed values of pH within the range of 6 – 8. Carbons with pH 6-8 are useful for most applications involving adsorption from aqueous solutions (Okimemmen *et al*, 2007).

Bulk Density

According to AWWA (1991), lower limit of bulk density is 0.25 g/cm³ for activated carbons to be put into practical use. The bulk density for ASPAC and GHAC were 0.39 g/cm³ and 0.38 g/cm³ respectively. The values of the bulk density for ASPAC and GHAC are above the lower limit as recommended by AWWA (1991); this makes both ASPAC and GHAC suitable for adsorption of pollutants in the wastewater treatment.

Moisture Content

The moisture content in this work was found to be 2.32% and 2.34% for ASPAC and GHAC respectively. Moisture content, according to Aziza *et al.*, (2008) has a relationship with porosity (α) of a given carbon. Adsorbent with high moisture content is expected to swell less, thus retarding pore size expansion for adsorbate uptake.

Ash Content

Ash is a measure of inorganic impurities in the carbons (Bansode *et al.*, 2003). The ash content in this research was found to be 0.48% and 0.51% for ASPAC and GHAC respectively which is in range of most ash content of agricultural waste.

Analysis of the wastewater

Turbidity

This is the cloudiness or haziness of water caused by large numbers of individual particles that are generally invisible to the naked eye. The turbidity was reduced to 10 NTU for the first 10 minutes using ASPAC and gradually increased as the time progressed until it reached constant value 575 NTU at 110 minute (initial concentration) as showed in table 3. Also using GHAC, the turbidity was reduced to 23 NTU for the first 10 minute and gradually increases to a constant value of 575 NTU at 90 minute. This is due to the fact that the adsorbents became saturated at this time and could no longer reduce the cloudiness in the wastewater.

Total suspended solids (TSS)

The TSS of the wastewater was drastically reduced to 9 mg/l and 12 mg/l (ASPAC and GHAC respectively) for the first 10 minute. As the treatment continued and the time increases, the value increases to a point when it became constant and the same with initial value (110 minute and 100 minute for ASPAC and GHAC) as showed in table 3 and 4 respectively. This is because as treatment process (treatment time increases) continued more volume of the wastewater passes through the column, more particles escape into the effluent as a result of the adsorbent gradually getting saturated until it became fully exhausted and could no holds the particles.

Total dissolved solid (TDS)

The total dissolved solid was remarkably reduced from 72.4 mg/l to 1.9 and 2.0 mg/l for the first 10 minute for both ASPAC and GHAC respectively; although the initial concentration of the wastewater TDS falls within the national and international discharge standard of 500mg/l according to Enugu State Water Corporation as reported by Emeka (2015).

Nitrogen

Nitrogen is essential for plant growth, but the presence of excessive amount in water body presents a major pollution problem. Nitrogen compound may enter water from agricultural fertilizer (as in the case of the wastewater in this study), human sewage, farm manure and the host of others. The two adsorbents used in this study were able to reduce the nitrogen content from the initial concentration of 2.58 mg/l to 0.06 using ASPAC and 0.07 mg/l using GHAC respectively at bed height of 9 cm and flow rate of 20 ml/min for the first 10 minutes.

Although these values are high when compared with acceptable standard for $\text{NH}_3\text{-N}$ by Australia (0.03 mg/l) but when the bed height was increased to 12 cm as showed in the appendix, the N content was reduced to 0.01 mg/l using ASPAC and 0.02 mg/l for GHAC at 10-minute effluent discharge, these falls within the standard.

Phosphorus

Phosphorus accelerates the growth of algae and aquatic plants. Total P > 0.03 mg/l will increase plant growth and cause eutrophication. The two adsorbents were able to reduce the phosphorus level in the wastewater although the initial concentration of the wastewater falls within the acceptable limit (3.5 mg/l) by international and national standard according to Enugu State Water Corporation as reported by Emeka (2015). The two adsorbents used in this study were able to reduce the phosphorus content from the initial concentration of 1.47 mg/l to 0.05mg/l for both ASPAC and GHAC respectively at bed height of 9 cm and flow rate of 20 ml/min for the first 10 minutes. The concentration of the effluent increases gradually until the adsorbent became saturated at 70 minutes for both ASPAC and GHAC respectively.

Effect of Adsorbent Bed Height

There was significant effect of bed height on the effluent concentration of N, P and K as well as other effluent attributes such as turbidity, TDS, TSS and TS, as the bed height increases, the NPK concentrations and other attributes of the effluents decreased at various flow time (service time) and vice-versa. Similar results were earlier reported by Unuabonal *et al.* (2010), Mulgunmath *et al.* (2012) and Noreen *et al.* (2013). The lower concentration at higher bed height could be due to the large amount of the binding sites that are available than that obtained with lower bed heights. Furthermore, the smaller bed height is saturated in less time than higher bed heights, hence they correspond to less amount of adsorbent and subsequently, a smaller capacity for the smaller bed to adsorb adsorbate from solution.

The effect of bed height was also investigated for nitrogen, phosphorus and potassium adsorption onto ASPAC and GHAC using through curves. The breakthrough curves for the adsorption of N, P and K onto ASPAC and GHAC at various bed heights (6, 9 and 12 cm) and constant flow rate of 20 ml/min clearly show that increase in bed depth increases the breakthrough time and the residence time of the solute in the column.

Both the breakthrough and exhaustion time increased with increasing the bed height. Higher N, P and K uptake was also expected at a higher bed height due to the increase in available fixation binding sites for the N, P and K to adsorb on ASPAC and GHAC. The increase in the adsorbent mass in a higher bed provided a greater service area which would lead to an increase in the volume of the solution treated. (Gupta *et al.*, 2004) reported in their work that when the bed height is reduced, axial dispersion phenomena predominates in

the mass transfer and reduces the diffusion of the solute, and therefore, the solute has not enough time to diffuse into the whole of the adsorbent mass.

Sivakumar and Palanisamy (2009) also reported that the throughput volume of an aqueous solution increased with increase in bed height, due to the availability of more number of sorption sites. This shows that at smaller bed height the effluent adsorbate concentration ratio increased more rapidly than for a higher bed height. Furthermore, the bed is saturated in less time for smaller bed heights. Small bed height corresponds to fewer amounts of adsorbent and binding sites.

Effect of flow rate

The effect of flow rate on the adsorption of N, P and K onto ASPAC and GHAC was investigated by varying the feed flow rate (20, 30 and 40 ml/min.) at a constant adsorbent bed height of 9 cm as shown by the breakthrough curves in figure 15 to 20. The trend of the curves showed that at higher flow rate, the front of the adsorption zone quickly reached the top of the column. This implies that the column was saturated early. Lower flow rates resulted in longer contact time, as well as a shallow adsorption zone. Higher flow rate is seen by the steeper curve with relatively early breakthrough and exhaustion time; they resulted in less adsorption uptake (Sarin *et al.* 2006).

Kananpanah *et al.*, (2009), reported that decrease in the volumetric flow rate favour more ion exchange conditions. As flow rate increased, the breakthrough curves become steeper and reached the breakthrough quickly. This is because of the residence time of the adsorbate in the column, which is long enough for adsorption equilibrium to be reached at high flow rate. This means that the contact time between the adsorbate and the adsorbent is minimized, leading to early breakthrough (Sivakumar and Palanisamy, 2009). Increasing the flow rate gave rise to a shorter time for saturation.

V CONCLUSION

This research showed that activated carbon produced from ASP and GH is good for the production of activated carbon for the removal of N, P and K from agricultural wastewater.

The physico-chemical properties of the ASP and GH activated carbon produced in these experiments such as surface area, carbon content, pH and their ability to remove N, P and K ions reveals that they are good adsorbents for treatment of wastewater as significant reduction (adsorption) in the concentration of the ion were recorded. Moreover, ASP and GH (waste) are inexpensive and readily available.

This study also revealed that employing adsorbent in a column is not only beneficial in term of adsorption alone but can also serve as filtration medium.

The turbidity (575 NTU) and total suspended solids (532 mg/l) were reduced to 2 NTU and 4.7 mg/l respectively using ASPAC at bed height of 12 cm and flow rate of 20 ml/min

Comparing treatment performance of the column operated under different experimental conditions, it can be concluded that higher adsorbent bed depths and lower feed flow rate can contribute to highly efficient treatment system with a relatively long adsorbent expectancy prior to exhaustion. In this study, 12 cm adsorbent bed depths and 20 ml/min hydraulic loadings gave the best performance for wastewater treatment; as the flow rate increased, the breakthrough point is reached earlier and the time required reaching saturation decreases more rapidly. For smaller bed height, (C_e/C_0) ratio increases more rapidly than for a higher bed height, steeper breakthrough curves are obtained and break point is achieved sooner due to the lesser contact time of the wastewater with adsorbent.

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