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PHOSPHORUS REMOVAL FROM PHOSPHATE CONTAINING EFFLUENT USING ANIMAL BONE BASED ACTIVATED CARBON

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ABSTRACT

Phosphate removal capacity of Animal bone based activated carbon (BNE) was investigated through adsorption process. BNE was characterized using Fourier Transform Infrared (FT-IR), Energy dispersive X-ray (EDX) and Scanning Electron Microscopy (SEM). Parameters such as adsorbent dosage and pH of the effluent solution were varied. Results obtained show that FT-IR spectrum of the activated carbon displays a number of absorption peaks, reflecting the complex bio-mass structure and a variety of functional groups such as –OH, -NH, C=O, C-H, C-N, CH3 and CH2 which explains its improved adsorption behaviour on the colloidal particles. SEM shows the morphological changes with respect to shape and size of the activated carbon and it is evident that the carbon particles are in the form of spheres with a wide range of sizes. EDX patterns for BNE adsorbent indicated that C and O are the main constituents though other elements such as Al, Ca, Sn ,P, S and K are also present in low proportion. Removal efficiency of more than 96% was obtained for BNE at the optimum pH of 6, dosage of 50g/l and particle size of 0.2mm.

KEYWORDS: Phosphate, Activated carbon, Effluent, Adsorbent, Bio-mass.

INTRODUCTION

Phosphorus pollution is a major problem resulting from improper disposal of wastes generated especially from indigenous chemical industries such as fertilizers, soap and detergent companies. The excess content of phosphorus in receiving waters leads to extensive algae growth (eutrophication), coloured, murky, odourous and unwholesome surface waters, fish kills and loss of many other aquatic animals [1]. Failure to halt further deterioration of environmental quality might jeopardize the health of large segment of the population with serious political and socio-economic implications. The removal of phosphate from wastewater can be achieved through several conventional methods such as chemical precipitation, coagulation, reverse osmosis, ion-exchange and other biological methods but the cost, inefficiency and the detrimental effects of the chemicals used in those methods have necessitated the needs for alternative methods. To this end, this work focuses on the use of animal bone based activated carbon through batch adsorption process for the removal of phosphorus from wastewater. Activated carbons are amorphous solid adsorbents that can be produced from almost all carbon- rich materials, including wood, fruit stones, peat, lignite, anthracite, shells, animal bones and other raw materials [2,3]. Their unique adsorption properties result from their high surface area, adequate pore size distribution, broad range of surface functional groups and relatively high mechanical strength. Carbon-Oxygen surface groups are by far the surface characteristics such as wet ability, polarity, acidity and chemical reactivity of these materials. In fact, the combined oxygen is often found to be the source of the property by which a carbon becomes useful or effective in certain respects [4]. The most important and common groups influencing the surface and adsorption properties of activated carbon are functional groups containing oxygen, and their presence enhances the adsorption of polar species [2]. Consequently, activated carbons are used extensively for the removal of undesirable odour, colour, taste and other organic and inorganic impurities from domestic and industrial wastewater and in a variety of gas-phase applications [5,6,7].

MATERIALS AND METHODS

Animal bone used as base material was obtained from open market in Umuoma,Uli near Anambra State University Campus. The bone was scrapped and washed with clean water to remove any accompanying dirt and impurities. Size reduction was done manually

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with local grinding stone before drying in an oven at 1100C for 36hours. The dried bone was weighed and then introduced into the hot zone of the muffle furnace for carbonization. The temperature of the muffle furnace was increased by 100C per minute from 2000C to 8000 C and kept constant for 2hours for charring . The charred material was removed from the furnace and allowed to cool to room temperature. The carbonized material was ground and sieved using 0.2mm mesh. The sieved 0.2mm particle size material was weighed and purified by washing with 0.5M HCl solution and rinsed three times with distilled water to remove excess acid solution. The purified material after drying, was subjected to activation with 1M Al2 (SO4)3. The activated BNE was packed in an air tight sample bag with labels. Proximate analysis was carried out on BNE to determine the weight loss, bulk density,% moisture content,% ash content, iodine number, % volatile matter and fixed carbon using Standard methods [8,9].Surface area was estimated using Sear's method [10]. BNE was characterized using Fourier Transform Infrared (FT-IR) to identify the presence of their functional groups, Energy dispersive X-ray (EDX) was used for the identification of the constituent elements and Scanning Electron Microscopy (SEM) for the surface morphologies and pore diameter distribution.

Preparation of Phosphate Solution from Phosphate Rock

The phosphate solution used as an effluent in this study was prepared by first dissolving 500g of phosphate rock sample in 1000cm3 of distilled water. The solution was thoroughly stirred and left for about 30minutes to allow the particles enough time to dissolve. After that the solution was again stirred and filtered off the silts, organic matters and insoluble phosphate rock particles. The homogeneous solution was further diluted with 3000cm3 distilled water before it was tested for pH level with digital pH meter and its phosphorus content concentration was measured in UV-spectrophotometer set at a wavelength of 650nm. The initial pH was 2.8 and phosphorus concentration was 373mg/l.

Batch Adsorption Experiment

The adsorption experiment was carried out by batch method. The pH level of the phosphate rock solution was adjusted to 2 with the aid of 0.5M HCl. 0.5g of BNE was mixed with 10ml of prepared solution and placed in a centrifuge. A constant rotational speed of 200rpm was maintained throughout and at time intervals of 30minutes. At the end of 30minutes agitation, the solution was removed from the centrifuge and filtered. The filtrate was then tested for residual concentration of phosphate in a UV-spectrophotometer which was set at 650 nm . The same procedure w as repeated for pH levels of 4,6, 8 and 10 respectively.

% Removal of phosphate can be calculated using [1].

$E\ \% = [(C_0 - C_1)/C_0] \times 100$, where C_0 and C_1 are the initial and final phosphate concentrations respectively.

RESULTS AND DISCUSSION

The characteristic results of the BNE activated with Al2(SO4)3 are presented in Table1. The surface morphology of the activated carbons before and after sorption of phosphorus was measured with the help of SEM-EDX and is presented in Plate.1 and Plate.2 respectively. The morphological aspects of the solids show that both materials, before and after sorption, are in flake-like form. Plate.1 clearly reveals the surface texture of the materials. It is evident that the carbon particles are in the form of spheres with a wide range of sizes. Plate.2 shows the morphological changes with respect to shape and size of the activated carbon after adsorption of phosphorus. It can be clearly observed that the surface of the activated carbon has been changed into a new shinning bulky particles and whitish patches structure after phosphorus adsorption. The qualitative elements composition of the activated carbon is analyzed and presented in Table2. The dispersive X-ray patterns for the BNE indicated that C and O are the main constituents, though other elements such as Al, Ca, Si, P, S, Na, Mg, K and Fe are also present in low proportion.

FT-IR spectrum of the BNE as presented in Fig.1 displays a number of absorption peaks, reflecting the complex biomass structure. The broad absorption peak at 3415.08cm-1 represents –NH and bounded –OH groups. The bands at 1635.69 cm-1 correspond to C=O stretching vibration of carboxyl groups. Other significant band positions of BNE are noted at 1442.8 cm-1 (indicative of scissoring and bending vibrations of CH3 and CH2 groups). 1039.67 cm-1 (indicative of C-N stretching of C-O group), 874.75 cm-1(indicative of bending vibration in C-H group) and 587.34cm-1 is assigned to the alkanes group.

Effects of Dosage on Adsorption Process

Fig.2 shows the removal efficiency of BNE activated with $Al_2(SO_4)_3$ as a function of time for various adsorbent dosages. It can be seen from the figure that the removal efficiency increases very fast with dosage within the first 60minutes after which the rate of removal of phosphorus begins to decrease. For example, at constant pH of 6, particle size of 0.2mm,

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the removal efficiencies of BNE at 10g/l and 50g/l dosages at 60minutes were 94.4% and 96.24% respectively. The sharp increase in removal efficiency at the early stages of adsorption may be due to the availability of the active sites of BNE which are yet to be saturated by the adsorbates. However, as the process proceeds, the rate of removal begins to decline due to the fact that most of the available sites get occupied by the adsorbates thereby reducing the area of contact between the solute and the adsorbent [11].

Effects of pH on Adsorption Process

Fig.3 shows the removal efficiency profile of BNE activated with $Al_2(SO_4)_3$ as a function of time at various pH levels. It can be seen from the figure that the profile of the percent removal increases sharply for the first 60minutes after which the rate of removal efficiency begins to fall. The reduction in removal efficiency was more noticeable as from pH8 to pH10. This may be as a result of phosphate competing with hydroxide ions for positively charged surface adsorption sites at high pH values. Further more, the pH corresponding to the point of zero charge (pH_{pzc}) also plays an important role in the adsorption phenomenon. The pH_{pzc} of an adsorbent is the pH at which the bulk surface charge is equal to zero [12]. At the pH below pH_{pzc} , the adsorbent surface is positively charged, consequently at low pH, phosphate adsorption is facilitated by electrostatic and chemical attraction onto the abundant positively charged adsorbent. However, as pH rises above the pH_{pzc} , the surface becomes predominantly negatively charged and phosphate adsorption decreases [12].

CONCLUSIONS

The present study shows that the animal bone, an abundant, non-toxic environment friendly agricultural waste can be used as sorbent for the removal of phosphorus from aqueous solutions. The amount of phosphates removed was found to vary with solution pH and adsorbent dosage. More than 96% removal efficiency of BNE was obtained at the optimum pH6, particle size of 0.2mm and dosage of 50g/l.

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Table 1: Characterization results of adsorbents activated with Al2(SO4)3

Parameter	BNE
Weight loss	42.65
Bulk density (g/cm^3)	0.49
% Ash content	6.95
Iodine number (mg/g)	417.12
Volatile matter (%)	18.65
Moisture content (%)	8.21
Fixed carbon	63.18
Surface area (m^2/g)	592.79

Table2: Characterization results of BNE using EDX

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	41.32	0.4645	33.99	0.49	46.90
O K	43.67	0.4204	39.69	0.48	41.10
Al K	0.76	0.8377	0.35	0.03	0.21
P K	4.31	1.3482	1.22	0.06	0.65
S K	25.94	0.9734	10.18	0.14	5.26
Ca K	35.69	0.9728	14.02	0.17	5.79
Sn L	1.11	0.7656	0.55	0.11	0.08
Totals			100.00		

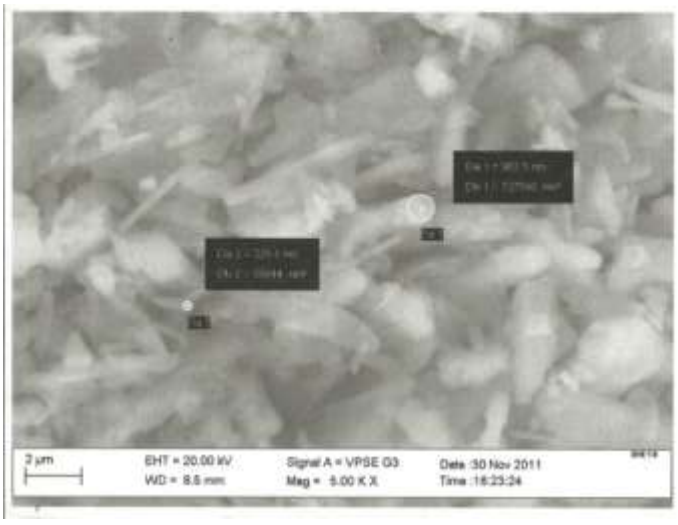

Plate1: SEM-EDX image of BNE

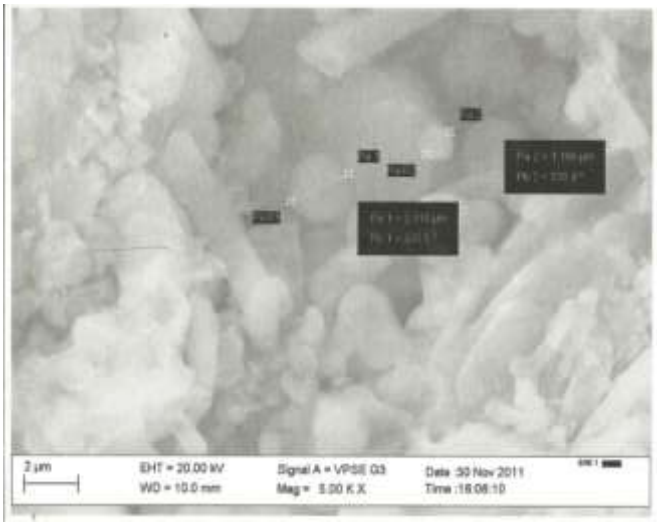

Plate2: SEM-EDX image of BNE after sorption before sorption

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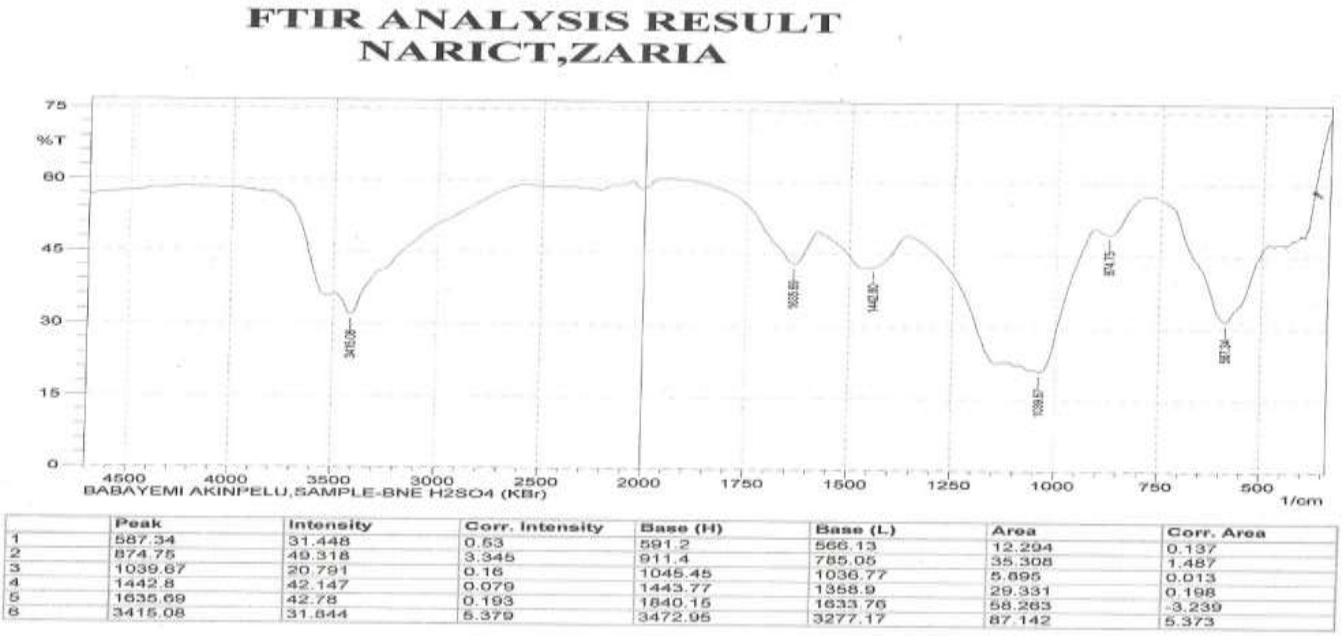

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	587.34	31.448	0.63	591.2	566.13	12.294	0.137
2	874.75	49.318	3.345	911.4	785.05	35.308	1.487
3	1039.67	20.791	0.16	1045.45	1036.77	5.895	0.013
4	1442.8	42.147	0.079	1443.77	1358.9	29.331	0.198
5	1635.69	42.78	0.193	1640.15	1633.76	58.263	-3.239
6	3415.08	31.844	5.379	3472.95	3277.17	87.142	5.373

Fig.1: FT-IR Spectrum of the activated carbon (BNE)

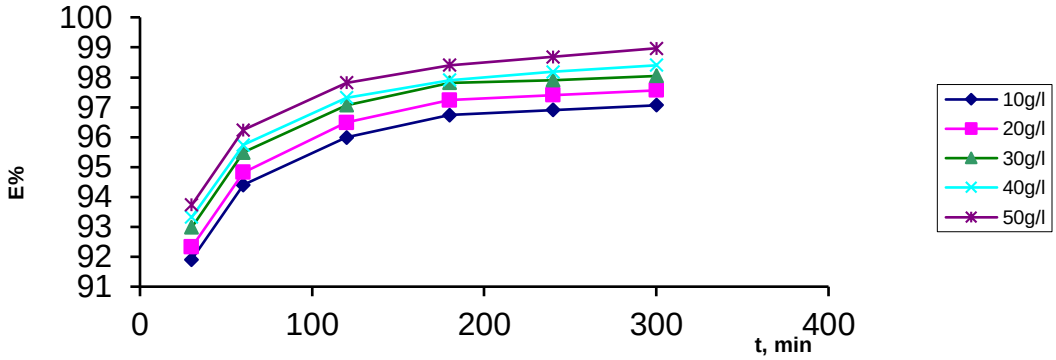

Fig.2.: Removal Efficiency of BNE activated with $Al_2(SO_4)_3$ at different adsorbent dosage

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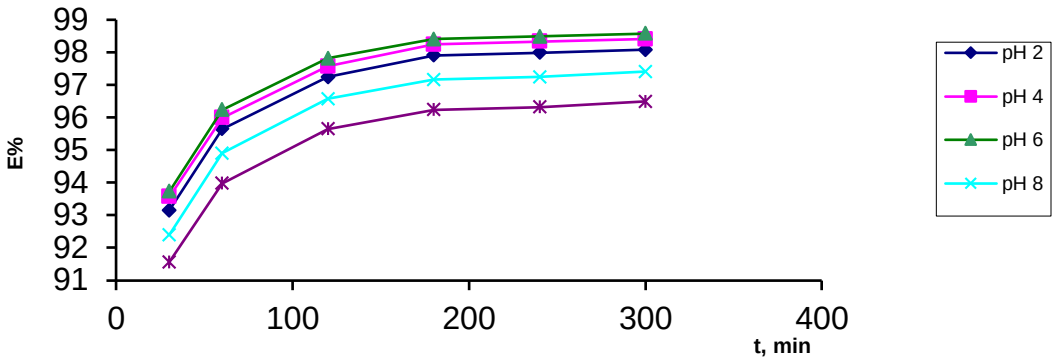

Fig. 3: Removal Efficiency of BNE activated with $Al_2(SO_4)_3$ at different pH levels and adsorbent dosage.=50g/l