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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM BOVINE HORNS

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ABSTRACT

Activated carbon has long been known to be a good adsorbent which has a very wide application. This study investigated the use of bovine horns as an alternative and effective adsorbent to other known adsorbents. Activated carbon was prepared from bovine horns and was chemically activated using sodium hydroxide. The structural properties of the adsorbent were studied using SEM and FTIR. The SEM results showed irregular surfaces, with roughness and high porosity which indicated that the prepared carbon has ability to carry out adsorption. FTIR wasused to obtain information about the chemical structure and functional groups of the prepared activated carbon and the results showed absorption band of the activated bovine carbon. This showed that the activation process was successful.

Keywords: Activated carbon, adsorbent, bovine horn, carbonization

INTRODUCTION

Several techniques have been used to manage water pollution. Some of these processes include filtration, biological methods, adsorption, electrolysis, and activated sludge. Among these processes, adsorption has been known to be a very effective method for the removal of pollutants such as heavy metals, dyes, phenols, suspended solids, dissolved organic compounds, and so many others from so many industrial wastewater and other effluentsusing suitable adsorbents.

Presently, activated carbon is the most widely employed adsorbent worldwide, it is efficient in the removal of heavy metals as well as other contaminants in water [1].

Activated carbon also known as activated charcoal or decolourizing carbon is a solid, porous, carbonaceous material prepared by carbonizing and activating organic substances. The activation is usually with the use of chemical activating agent and the carbonization is done at an elevated temperature, followed by removal of the chemical activating agent by water washing [2].

Nowadays, many environmental and adsorption researchers are searching for analternative and environmentally friendly way to produce the most cost-effective and cheapest adsorbent for economical wastewater treatment by testing variety of raw materials ranging from industrial waste product to agricultural product[3].

The aim of this study therefore, is to prepare and characterize activated carbon from bovine horns since bovine horns are sometimes readily available.Thus activated carbon prepared from bovine horns is being proposed as a probable adsorbent for removing pollutants from wastewater before their eventual discharge into water bodies.

MATERIALS AND METHODS

Sample Collection and Preparation

The Bovine horns (cow horns) used for this study were collected from an abbatoir in Zango, Zaria, Kaduna State, Nigeria. The bovine horns were washed thoroughly with detergent and rinsed with tap water to remove impurities, rinsed with distilled water and sun-dried for 21 consecutive days . They were cut to sizes of about 5 mm to l0 mm. They were then grounded to powder form with mortar and pestle. The grounded powder was sieved using 300 and 350 µm mesh size. The ground horn powder was then soaked in chloroform for 24 hours to remove the fat, after which it was sun dried and washed with de-ionized water to remove the color. This was stored in an air tight container for subsequent work [4].

Plate 1: Collected bovine horns

Carbonization Process

The prepared bovine horns were carbonized using the method described by Yahya et al [5]. About 100 g of the prepared bovine horns was weighed into large ceramic crucibles and

introduced into the tubular furnace in an inert atmosphere of nitrogen and was carbonized at a temperature of 400 ^{o}C for 2 hours. This was allowed to cool at room temperature. This process was repeated until a substantial amount of carbonized sample was obtained. The resulting sample was stored in sealed containers for activation.

Plate 2: Carbonized bovine horns

Chemical Activation of the Prepared Carbon

The carbonized sample was chemically activated using Sodium hydroxide (NaOH).The charred and carbonized bovine horns was impregnated with NaOH (Sigma-Aldrich) at the ratio of 1:2, while stirring. The impregnated material was introduced into a tubular furnace that was heated at a temperature of 400˚C for 2 hours and was then cooled down to room temperature. The nitrogen was streamed through furnace at rate of 150 cm^3/min from the start of heating to the time the tubular furnace cooled down. The resulting activated carbon was then washed with 1M HCl followed by deionized water to remove any residual organic and mineral matter and to maintain a pH of 7.0. The bovine horn activated carbon was then dried at 110°C overnight, before being cooled and stored for further studies [5].

Plate 3: Carbonized bovine horns impregnated with NaOH while stirring

Plate 4:Washing of the activated carbon with deionized water

Proximate Analysis of the Granulated Bovine Horns

Moisture content determination

An empty dish was wash, dried in an oven at 105 ^{o}C for 3 hours and allowed to cool in a desiccator. Exactly 3 g of the sample was introduced into the dried dish and placed into the oven at 105 ^{o}C for 3 hours. The resulting weight of the sample was noted after drying (6).

The percentage moisture content (MC %) was calculated using equation 1

$$Moisture\ content(\%) = \frac{(W_1 - W_2)}{W_1} \times 100 \qquad \text{Equation 1}$$

Where: W_1= weight of sample (g) before drying

W_2= weight of sample (g) after drying

Ash content determination

The ash content was determined by the method described by Ilaboya et al[6]. Exactly 1 g of the bovine horn was taken, dried in an oven at a temperature of 105°C for 1 hour. The final weight after drying was measured and recorded as (Ag = Oven dry weight). Thereafter the dried bovine horn was placed in a cold Muffle furnace and the temperature was allowed to rise until it reached 500 °C. After 1 hour, it was removed and allowed to cool in a desiccator to room temperature and reweighed again and its weight was again recorded as Bg (Ash weight). The percentage ash content was then calculated from equation 2.

$$\text{Ash}\ (\%) = \frac{\text{Ash Weight (g)}}{\text{Oven Dry Wt.(g) of sample}} \times 100 \qquad \text{Equation 2}$$

Bulk density (Apparent density) determination

The sample was introduced gently without compacting into a pre-weighed 100 cm^3 graduated cylinder till it filled up to mark. The weight of the sample and the cylinder was noted and the bulk density of the sample was calculated using the expression below:

$$Bulk\ density\ (\frac{g}{cm^3}) = \frac{W_2 - W_1}{V} \quad \text{Equation 3}$$

Where: W_2= weight of cylinder and sample

W_1= weight of empty cylinder

V = volume of cylinder

Carbon content determination

The dried weight, W_0, of each sample was determined. The carbon yield, y_{ch}, was determined by:

$$Y_{ch} = (\frac{W_{ch}}{W_o}) \times 100 \quad \text{Equation 4}$$

Where: W_{ch} = weight of Carbon retrieved from the furnace.

W_o = Dried weight of the sample.

Characterization of the Prepared Activated Carbon

FTIR-spectroscopy

Fourier transform infrared (FTIR) spectroscopic analysis was used to study the surface chemistry of the activated carbon prepared. FTIR spectra was recorded between the spectral range of 4000 and 400 cm^{-1} with a resolution of 4 $cm^{-1.}$ The discs were prepared by mixing 1mg of dried sample with 500 mg of KBr in an agate mortar to give a homogenous blend and then pressing the resulting mixture at 10 tonnes cm^{-2} for 15 minutes under vacuum. The disc containing the sample was then placed in the FTIR spectrometer (Agilent Technologies Cary 630 USA)and the spectrum of the sample was collected. The FTIR spectra gave information about the characteristic functional groups on the surface of the bovine horns activated carbon [7].

Scanning Electron Microscopy (SEM)

The morphology of the activated carbon sample was analyzed by SEM analysis.This was done usingQuanta Scanning Electron Microscope (FEI Quanta 250 USA). This was used to observe the surface pore structure of the prepared activated carbon. The moisture-free activated carbon was mounted onto a substrate with a conductive adhesive, conducting material coating on sample

was done with gold metal by vacuum evaporation to get uniform thickness of sample during analysis [8].

RESULTS AND DISCUSSION

Proximate Analysis

Table 1: Proximate analysis

Parameter	Value
Moisture content (%)	2.22±0.12
Ash content (%)	5.3±0.13
Carbon content (%)	62.1±1.07
Bulk density (g/cm^3)	0.57±0.00

Proximate analysis of activated carbon provides a good idea about the physical properties of the sample. The result of proximate analyses as presented on Table 1 showed the moisture, ash, carbon and bulk density contents to be 2.22±0.12, 5.3±0.13, 62.1±1.07 and 0.57±0.00, respectively. The low moisture content values obtained for the activated bovine horns make them better adsorbent for this study and this corroborates with report that the rate of adsorption of contaminant increases when adsorbents with low moisture contents are employed [9].

The low ash content of the bovine horns gives a clue that the precursor is a good starting material for activated carbon production. This concurs with the existing literature that lower ash content in activated carbon increases the carbon yield [10].

The high fixed carbon makes this material a good precursor for production of activated carbon [11]. The bulk density of activated carbon usually suggests that if the activated carbon is added to water it will sink and this will give better contact with the adsorbate and thereby leading to effective adsorption process. The bulk density of the carbon was also determined to be 0.57±0.001 g/cm^3. This low density of the AC could be as the result of its hollow and fibrous nature, which usually give rise to an important pore volume [12]. The bulk densities obtained in this study revealed that the bovine horns could be of great potential for wastewater treatment as the values are higher than 0.25 g/cm^3, the minimum required for commercial adsorbents as reported by Denver [13].

Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy was used to study the functional group and obtained information on the chemical structure of the prepared AC. The surface chemistry of an AC is determined by the type, quality, and bonding of functional group.

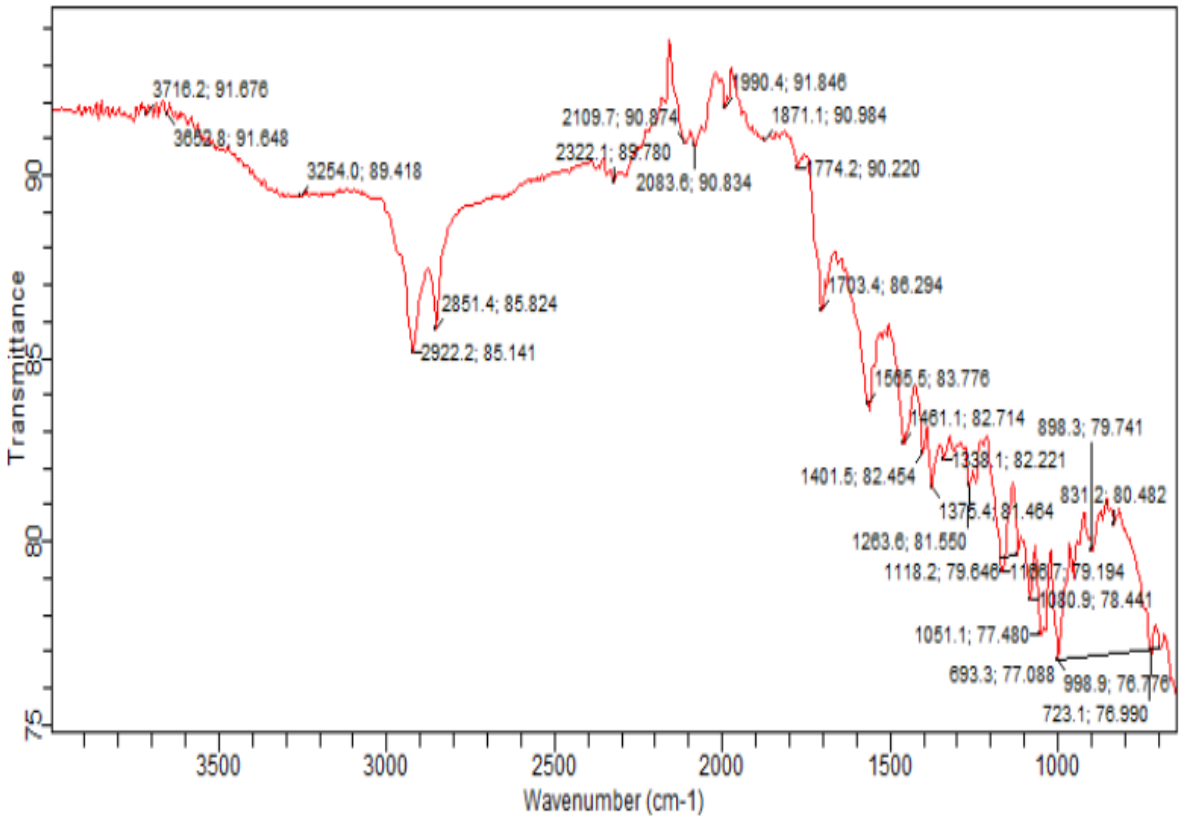

Figure 1: Infrared spectra of the prepared AC

Table 2: FTIR result of activated carbons

S/n	Wave number (cm^{-1})	Functional groups
1	3652	O-H stretch for alcohol
2	3254	=C-H stretch for alkene
3	2922	C-H Stretch for alkane
4	2322	C==c stretch
5	2080	N-H Bend
6	1990	C-H weak

7	1871	C-H weak
8	1774	C=O stretch
9	1556	N-H Bend

As shown in the IR spectra of the activated carbon presented in Figure 1, the adsorptions peaks at 3652 cm^{-1} can be typically attributed to the presence of OH- hydroxyl group (either aliphatic alcohol or phenol), and this could be free or intermolecular bonded hydroxyl group. The peaks at 2923 is ascribed to C-H aliphatic stretching while absorption bands at 1871 cm^{1} portray the presence of C=C groups of aliphatics and aromatics [14].

Also, the peak observed at 2922cm^{-1} is due to the carbon-hydrogen single bond stretch (C-H) within the carboxylic chain. Adsorption is aided by the various functional groups present on the surface of the AC. Functional groups of adsorbents not only affect the adsorption behavior but also dominate the adsorption mechanism [15].

Similar functional groups were also reported on the surface of activated carbon made from palm kernel shells by Akinpelu[16].

Scanning Electron Microscope

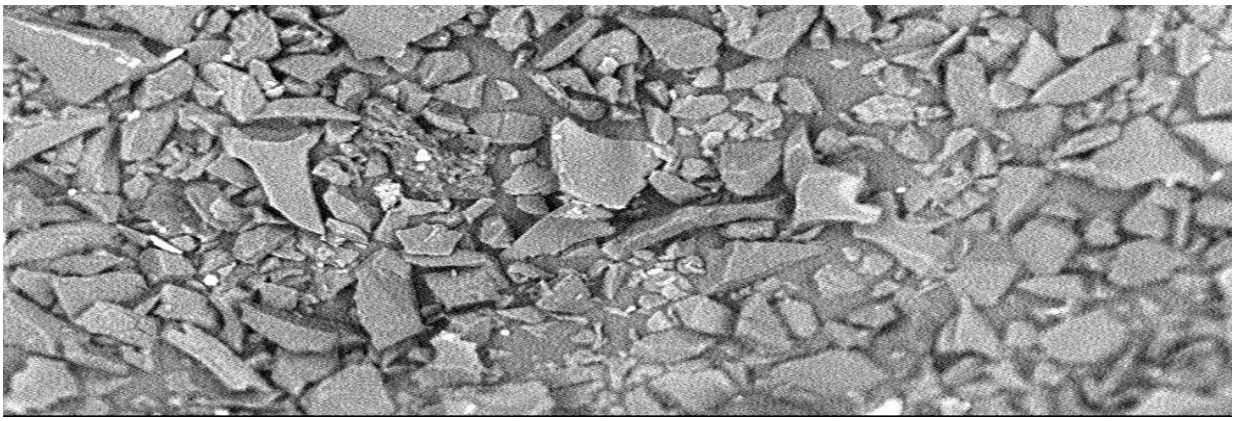

Plate 5: SEM image for bovine horn before activation

Plate 6: SEM image after activation

It can be observed from the micrograph (plate 6), that the activated carbon has irregular surfaces, with roughness and high porosity which is as a result of the surface pyrolysis and NaOH etching which occurred during the activation process, unlike the unactivated carbon (plate 5) which has no porosity. Chemical activation accompanied with further introduction to high temperature induced the formation of thelarge pores in a honeycomb shape showed clearlyon the surface of the bovine horn activated carbon. This indicates the possibility of good textural properties. [17].

CONCLUSION

From the findings of this study, it was seen that bovine horns may constitute a suitable precursor for the manufacture of effective activated carbon through chemical activation with not just acid but with base (sodium hydroxide). The preparedactivated carbon had an acceptable range of moisture and ash contents for producing good adsorbent. It also showed favourable adsorption characteristics. The FTIR showed the severalfunctional groups of the activated carbon and the morphology studies indicated that the prepared activated carbon had clear pores that aid adsorption.

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