



## **Preparation and characterization of an activated carbon from the bones of *Gudali (Bos Indicus)* cattle from Adamawa**

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### **Abstract**

The goal of this work was the production and the characterization of activated carbon from the Gudali species bones. The calcination have been done in the presence of phosphoric acid for the production of animal activated carbon as new and eco-friendly adsorbent. A textural and structural characterization was performed during this work. With regard to the textural analysis, results reveal 0.2-5.0 mm, 1.50 %, 17.63 %, of 6.84 and the of 666.251 mg/g for sieve, residual moisture, residual moisture,  $\text{pH}_{\text{zpc}}$ , iodine value respectively. A large exchange surface of our animal activated carbon which can reach 1045  $\text{m}^2/\text{g}$ . Thus, the adsorbent has a microporous surface. Concerning structural analysis, the elemental analysis and the different crystallized phases of the product adsorbent have been determined by semiquantitative X- ray and X-ray diffraction. It appears that the abundant chemical elements constituting the adsorbent are Ca (49.73%) and P (43.21%). From these analyses, it appears

that the chemical formula of the product adsorbent is  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ . FTIR spectra of the adsorbent showed strong bands at 1023, 872, 724, 599 and 558  $\text{cm}^{-1}$  due to the vibrations of  $\text{PO}_4^{3-}$  groups from the natural constituent hydroxyapatite of bone. Bands ranging from 1454 to 1943  $\text{cm}^{-1}$  indicate the presence of C=O, C=C and C=N groups contained in the bone matrix of the organic phase responsible. The SEM images show that the surface of the adsorbent has inhomogeneous shapes, irregular structure, different sizes and pores, with a very developed porosity. Laser particle size distribution gives information about the particle size distribution of the animal activated carbon of the Gudali species. The adsorbent has a volume average diameter of Herdan  $D [4.3] = 219.613 \mu\text{m}$ , corresponding to the center of gravity of the volume distribution of our particles.

**Keywords:** Activated Carbon, Characterization, Gudali species Bones

## Introduction

Despite the progress made in recent decades, many parts of the world still face serious drinking water supply problems. It is estimated that over 800 million people [1] do not yet have access, under acceptable conditions, to safe water allowing them to meet their basic needs (consumption, cooking, hygiene, etc). These deficiencies have serious health consequences, 502.000 annual deaths attributable to cases of diarrhea caused by the consumption of unsafe drinking water [1, 2, 3]. The existing options for generalizing and securing access to drinking water in the countries concerned are diverse namely technically, financially, institutional and socio-political.

None are completely free from constraints. Hence, it is necessary to identify the solutions best suited to the given problematic and to the contexts in which they are deployed. The decontamination of contaminated water can be carried out by different processes. Among the most important water treatment techniques, the use of adsorbent materials is most affordable and appropriate for less developed country such as Cameroon. The activated carbon is widely used as an adsorbent material in industry to remove unwanted compounds. Its large specific surface area, surface functions, and pore size distribution allow it to absorb wide ranges of toxic pollutants.

In Cameroon, there is enormous potential in hydraulic resources (rainfall, groundwater and surface water) which are, however, unevenly distributed over all of the five main hydrographic basins of the territory. In terms of rainfall and surface water, the northern part of the country is poor relation to the southern part. In the northern region, for many years, village communities have faced serious problems in accessing drinking water. Populations

have always used surface water, to meet up with their basic needs, it is essential to put in place appropriate treatment solutions in order to avoid any health risk.

This is why the scientific community set out to find effective techniques and methods to find solutions. The choice was made on adsorption. Adsorption is an efficient elimination technique, easy to implement with a low cost. Adsorption is the process where molecules of a species called adsorbate (gas or liquid) come to fix themselves on the surface of a solid, called adsorbent. Adsorption can be physical or chemical according to the nature of the interactions which occur between the adsorbate and the surface of the adsorbent. Physical adsorption or physisorption involves very weak interactions between molecular entities such as molecular entities such as the van der Waals forces of attraction and forces due to electrostatic interactions of polarization. It is reversible and not very specific. The physical adsorption is fast and generally limited by the phenomena of diffusion. The strength of the interactions put can be estimated by the energy of adsorption which is between 5 and 40  $\text{kJ}\cdot\text{mol}^{-1}$  and considered as weak: the desorption can thus be total. Chemical adsorption or chemisorption is essentially irreversible and slow. Very specific, it is accompanied by a strong variation of activation energy. Chemical adsorption results from a profound modification of the electronic charge distribution of the adsorbed molecule: the binding forces are of the same type as those involved in the formation of chemical bonds. The energy of adsorption is higher than 80  $\text{kJ}\cdot\text{mol}^{-1}$ : desorption is difficult.

Physical adsorption can be done in monolayer or multilayer, whereas chemical adsorption is only mono-molecular because the presence of valence bonds between the adsorbate and the adsorbent excludes the possibility of multi-molecular layers. In a general way, adsorption is an exothermic phenomenon which occurs with a release of heat which can lead to a heating of the solid [4, 5, 6, 7, 8, 9, 10, 11].

Generally all solids are adsorbents. In the industry, the most used solids are activated carbons, zeolites, silica gels and activated aluminas. The particularly high adsorption capacities of these materials are partly due to their highly developed porous structures. The particularly high adsorption capacities of these materials are partly related to their highly developed porous structures and their large specific surface areas. In this work we have used beef bones of the Gudali species calcined in the presence of phosphoric acid.

Animal activated carbon is obtained by calcining animal bones in an inert atmosphere. This adsorbent has a large specific surface area, 80-90% calcium phosphate and 10% carbon. Animal activated carbon has been used for sugar decolourization, fish oil in the refining industry, and for water purification of metal ions and fluorides [12, 13, 14, 15]. Research

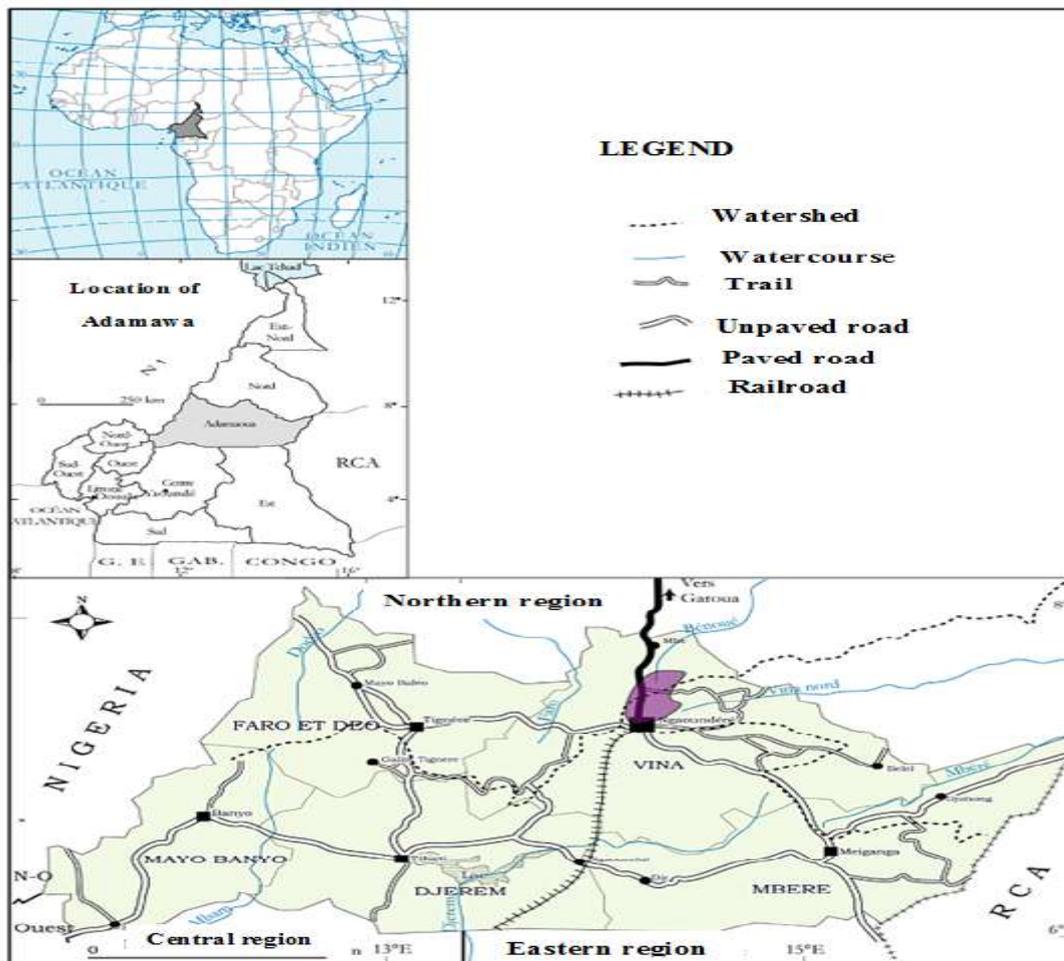
carried out on animal waste has shown satisfactory results during the elimination of Direct Red 75 and Direct Red 80, Basic Red 12, methylene blue, methyl orange, chromium (VI)[16, 17, 18, 19]. However, the efficiency of activated carbon depends on their properties. Hence it is important to characterize activated carbon before use. In fact literature reveals that the bone properties and adsorption efficiency varie from one author to another. This can be justified by the varieties and the nutritional behavior of beef. In our knowledge, Adamawa region of Cameroon is an important source of bone production and no study on the active carbon from this has not been done.

The present study is part of this perspective by using materials from particular animal waste, the bones of beef of the Gudali species (*Bos indicus*) in the Adamawa region of Cameroon. The phosphoric acid is used as an activating agent to improve adsorbent properties. Characterizations of pyrolysis bone Prepared from *Bos Indicus* Gudali are investigated.

## **2. Experimental**

### **2.1. Location of the beef bone sampling site**

The sampling site is located approximately 750 meters from the Commune d'arrondissement of Ngaoundere 3<sup>ieme</sup>. It is located at 13.5961° East longitude and 7.3551° North latitude, in the VINA Department, Adamawa Region.



**Figure 1:** Location of the beef bone sampling site.

## 2.2. Material

### 2.2.1. Preparation of the animal activated carbon

The beef bones were washed, degreased, dried at 50°C until constant mass and then ground in a Retsch SK 100 type mill (picture 1). The resulting grind was sieved through Retsch sieves with a diameter of 0.2 to 5 mm. The resulting grind was impregnated in an 85% orthophosphoric acid solution for 24 hours followed by oven drying at 105°C for 24 hours. Once dried, the materials were pyrolyzed at 500°C in a NABATHERM 30-3000°C muffle furnace for 3 hours respectively. After calcination, the obtained carbons were cooled and rinsed several times with distilled water until a rinsing water of pH between 6 and 7 was reached, then dried again in the oven at 105°C for 24h (picture 2).



**Picture 1:** Beef bones.



**Picture 2:** Animal activated carbon .

### **2.3 Characterization of animal activated carbon**

In this study, moisture content, ash content, porosity, specific surface area, and pH at zero charge point ( $\text{pH}_{\text{zcp}}$ ) were determined.

#### **2.3.1. Moisture content**

Porcelain crucibles were baked at 1000 °C for 3 h and then cooled in a desiccator. The mass of these crucibles was weighed using a 1/10000 precision balance. A mass of 2 g of animal activated carbon was introduced and the mass  $m_1$  was noted. Then they were placed (crucibles + animal activated carbon) in the oven at 105 °C for 24 h. After cooling, the mass  $m_2$  was weighed. The moisture content is given by the following equation 1:

$$\text{MC (\%)} = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad (\text{Eq1})$$

With  $m_0$ : mass of the empty capsule,  $m_1$ : mass of the capsule containing the wet sample and  $m_2$ : mass of the capsule containing the sample after steaming at 105 °C.

### 2.3.2. Ash content

The ash content was determined by the method described by Ahmed and Dhedan [41], and the method of AFNOR [39]. A 0.5g sample of coal from moisture content analysis is placed in a crucible. This crucible is introduced into a muffle furnace set at 650°C for 2h. At the end of the oven, the crucible is cooled to room temperature. The crucible is weighed again. The ash content is determined as follows:

$$AC (\%) = \frac{m_3 - m_2}{m_1} \times 100 \quad (\text{Eq 2})$$

Where  $m_1$  : the mass of coal used in (g),  $m_2$  : the mass of the empty crucible before carbonization in (g),  $m_3$  : the mass of the filled crucible after carbonization in (g).

### 2.3.3. Iodine index

The iodine index test aims to determine the capacity of the coal to adsorb small molecules. It characterizes the micropores accessible to small particles. This test was performed following AWWA B 600-78 from the work of Maazou et al [42]. and Ibrahim et al [35]. In a 100 mL beaker, 0.05 g of charcoal was introduced. 20 mL of the 0.1N iodine solution is added by pipette and the mixture is stirred for 5 min before filtering. A volume of 10 ml of the filtrate is taken and put into an Erlenmeyer flask. From the burette, a 0.1N sodium thiosulfate solution is added little by little to the Erlenmeyer flask containing the filtrate until the solution is completely discolored. Starch is used as a color indicator. The iodine value is given by the following formula :

$$\text{Iodine index} = \frac{(C_0 - \frac{C_{thio} V_{thio}}{2V_{I_2}}) M_{I_2} V_{ads}}{m_c} \quad (\text{Eq 3})$$

With  $C_0$ : Initial concentration of the iodine solution (mol/l),  $C_{thio}$  : Concentration of the sodium thiosulfate solution (mol/l),  $V_{thio}$  : Volume of thiosulfate poured at the equivalence (ml),  $V_{I_2}$  : Volume of iodine dosed (ml),  $M_{I_2}$  : Molar mass of iodine (g/mol) ,  $V_{ads}$  : Volume of adsorption (ml),  $m_c$  : Mass of carbon (g).

### 2.3.4. Specific surface (area)

The specific surface area was determined by the methylene blue adsorption method described by Kra et al [43]. 100mL of  $6.25 \cdot 10^{-6}$ M methylene blue solution is brought into contact with 0.1g of charcoal. The suspension was stirred for 10, 20, 30, 40, 50, and 60 minutes. The residual concentrations were determined by spectrophotometric. The determination of the maximum adsorption capacity is done by applying the Langmuir model

to the adsorption isotherms of methylene blue on charcoal. From the maximum adsorption capacity  $Q_m$ , the specific surface is determined by the following equation:

$$S_L = Q_m N_A S_{MB} \quad (\text{Eq 4})$$

With  $S_L$ : Specific surface of Langmuir ( $\text{m}^2/\text{g}$ ),  $Q_m$ : Maximum adsorption capacity ( $\text{mg}/\text{g}$ ),  $N_A$ : Avogadro number ( $6,022.10^{23} \text{mol}^{-1}$ ),  $S_{BM}$ : Surface occupied by a methylene blue molecule ( $175 \text{Å}$ ).

### 2.3.5 pH of the point of zero charges ( $\text{pH}_{\text{PCN}}$ )

There is a pH for which activated carbon is electrically neutral in solution. That is, the sum of the charges on the surface is zero. This pH is called pH of the point of zero charges ( $\text{pH}_{\text{PCN}}$ ).

To determine the  $\text{pH}_{\text{PCN}}$ , the method of Ibrahim et al [35] was used, that of the first bisector. This method consists of preparing 0.1 M sodium chloride (NaCl) solutions at different pH; 2, 4, 6, 8, 10, 12. The pH values were adjusted with a model pH meter HI 991001 using sodium hydroxide and hydrochloric acid solutions. Thus, 0.1 g of animal activated carbon was contacted with 20 mL of each solution per sample. The mixture was put under magnetic stirring for 24 h. The suspension was filtered through a filter paper and the pH of the filtrate measured with a pH meter.

Thus, the curve  $\text{pH}_f = f(\text{pH}_i)$  is drawn. The point of intersection between this curve and the first bisector gives the pH at the point of zero charge of the activated carbon considered.

## 2.4. Structural characterization of Adsorbent Materials

**FTIR Analysis**: IR spectra of the sample were obtained using a FT/IR-4100 Spectrophotometer (JASCO Benelux BV) in KBr pellets. **SEM**: Scanning electron microscopy (SEM) images were obtained with TESCAN, VEGA II model equipment for animal activated carbon material. A semiquantitative X-ray powder diffraction method was used to determine mineral composition.

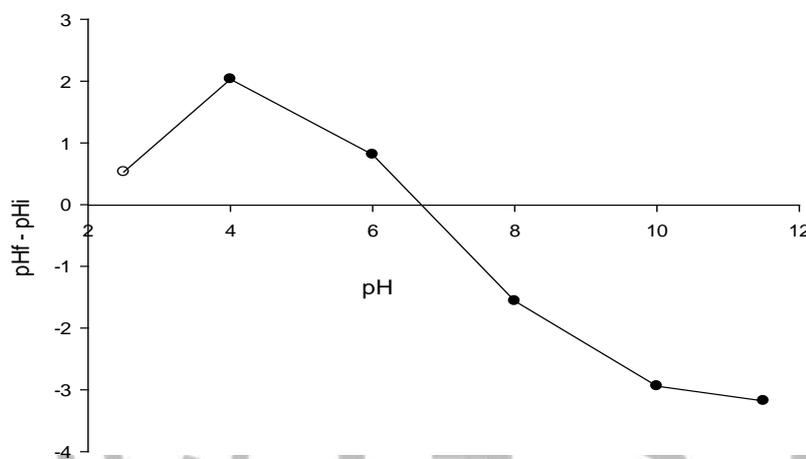
**X ray diffraction**: X-ray diffraction analyses on powders were performed using a Siemens Bruker D8 Advance unit with Co- $K\alpha$  anticathode ( $\lambda = 1.7903 \text{Å}$ ). The diffractograms were recorded from  $5^\circ$  to  $64^\circ$  degrees with analytic conditions of 40 mA, 20 kV,  $0.02^\circ$  steps. d-spacing, dhkl, corresponding to different diffraction pics have been calculated using Bragg's equation and JCPDS (Joint Committee on Powder Diffraction Standards) files were utilized in order to identify the crystalline compounds. **Particle size**: Laser particle size distribution: this technique allows the size distribution of particles to be measured. The granulometer available in the laboratory is of the MALVERN MASTERSIZER 2000 type using Scirocco as a dry

dispersion accessory. The measurements carried out are based on the principle of simple scattering and laser diffraction.

### 3. Results and Discussion

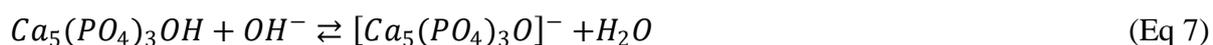
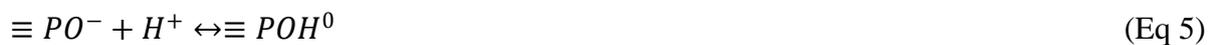
#### 3.1. Textural Characteristics

In the adsorption process the pH plays a crucial role. It gives information about the behavior of the adsorbate, the structure of the adsorbent and influences the adsorption mechanisms. pH plays an important role on the charges on the surface of the adsorbent as it influences the behavior of the adsorbent in acidic and basic environment. [20, 21, 38].



**Figure 2:** Effect of  $pH_{ZPC}$ .

The  $pH_{ZPC}$  of animal activated carbon obtained was 6.84 (Fig.4). From figure 4, it can be seen that the  $pH_{ZPC}$  value of our animal activated carbon is 6.84, this study gives the information about the behavior of the adsorbent mainly on the surface charges in acidic and basic medium. The animal activated carbon is mainly made of calcium hydroxyapatite, its buffering capacity can be influenced by the latter. The chemical reactions on the CaHa surface sites are the following:



According to equation 5 when the initial pH will be lower than  $pH_{ZPC}$  the supply of  $H^+$  ions in the medium would lead to the protonation of the surface of  $\equiv PO^-$  and  $\equiv CaOH^0$  groups of our adsorbent. In acidic medium, the surface charges of CaHa adsorbent will be positively charged, with a dominance of the cations  $\equiv CaOH_2^+$  and  $\equiv POH^0$  groups of neutral charge.

When the initial pH will be higher than  $pH_{zpc}$ , the contribution of  $OH^-$  ions in the medium would lead to the deprotonation of the surface sites  $\equiv CaOH_2^+$  and  $\equiv POH^0$  due to the presence of hydroxyl radicals [21, 37].

From Table 1, it can be seen that the iodine value of the animal activated carbon is 666.251 mg/g. In view of this result, it can be said that the animal activated carbon has micropores on its surface. The micropores alone practically determine the adsorption capacity of an activated carbon: they represent almost the totality of the surface and the volume offered to the adsorption [18]. The specific surface or mass area is the total surface area per unit mass of adsorbent available to the molecules. All the surface of the adsorbent particles is considered, including open porosity considered, open porosity included, for the calculation of the specific surface which thus cumulates the inner surface of all the pores constituting the adsorbent grain. The specific surface includes the external surface and the internal surface of an adsorbent. The animal activated carbon produced has a specific surface of  $1045m^2/g$ , this represents the surface effectively available for adsorption [19].

**Table 1:** Properties of animal activated carbon

Parameters	Values
Sieved (mm)	0.2-5.0
$pH_{zpc}$	6.84
Residual Moisture (%)	1.50
Ash Content (%)	17.63
Iodine Index (mg/g)	666.251
$S_{BM}$ ( $m^2/g$ )	1045

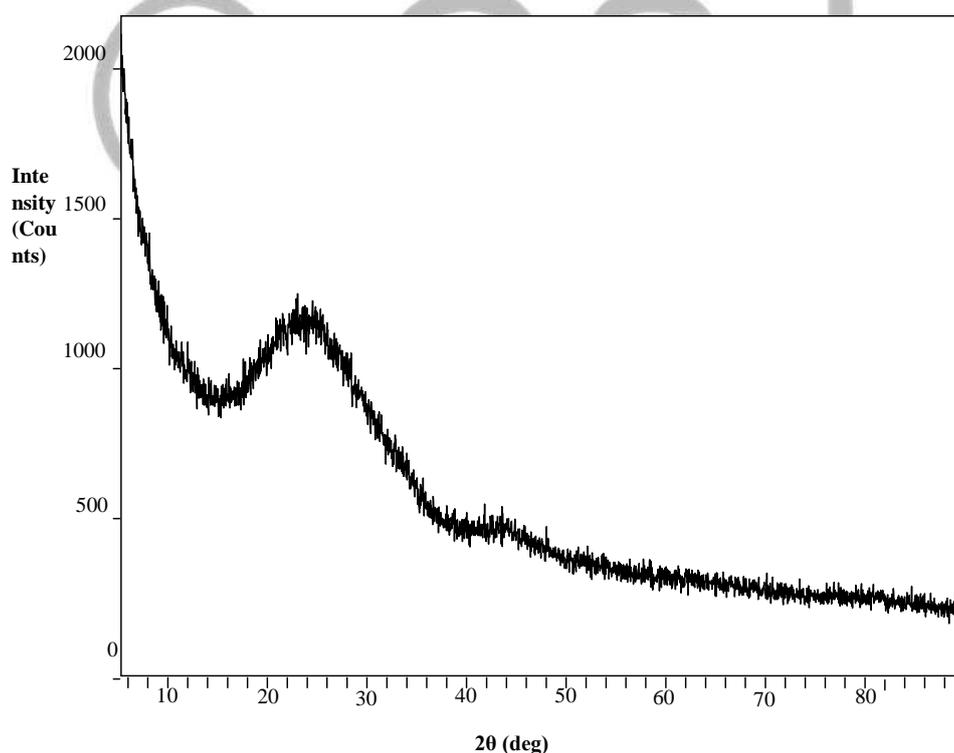
### 3.2. Structural characterization of adsorbent

The analysis of the animal activated carbon by semi-quantitative X-ray diffraction was used to have information on the elemental composition of the adsorbent. It emerges from this analysis that the adsorbent is constituted of major and minor elements. As major elements, we have: Ca (49.73 %) and P (43.21 %) with a ratio (Ca/P) equal to 1.15. As minor elements, we have: Si (3.82%), Mg (1.31%), Na (0.77%), Al (0.32%), Fe (0.26%), Cl

(0.26%), S (0.15%), K (0.07%), Sr (0.03%), Cu (0.05%) and Zn (0.02%). In the bone mineral composition, carbonate is the majority element and this was confirmed in the crystalline phase. Apatite carbonate is the essential element constituting bone and hydroxyapatite that of natural bone [22, 23, 24, 25].

### 3.2.1. XRD Analysis

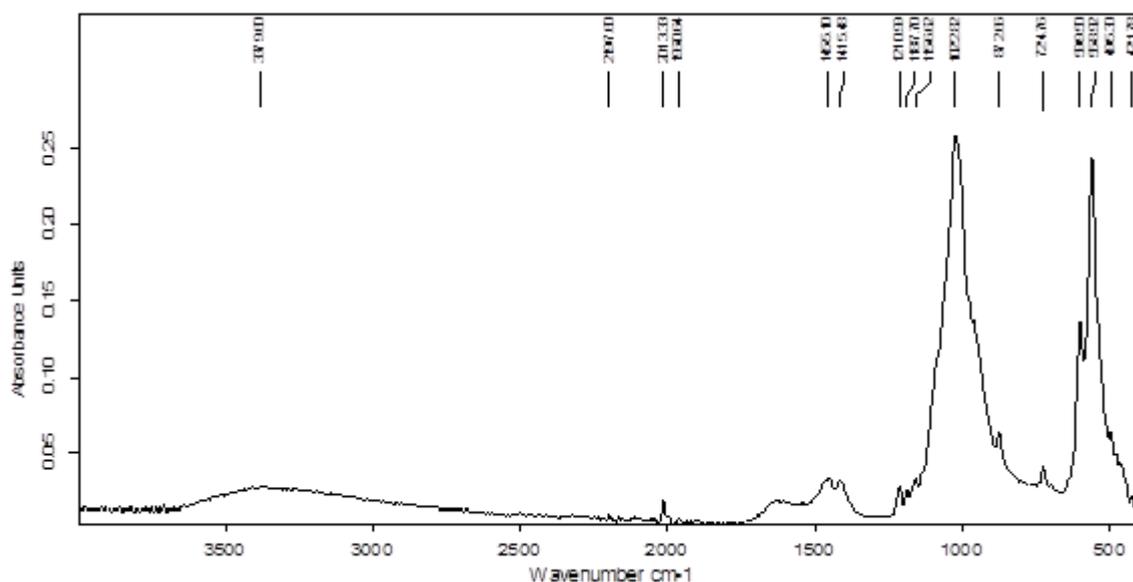
The XRD pattern of Animal activated carbon showed  $2\theta$  diffraction peaks at  $25^\circ$  and  $43^\circ$  which are attributed to the in-plane reflection of the graphitic structure (002) and (100) formed after carbonization respectively. The calculation of the inter-lattice spacing  $d$  for the (002) plane is 0.358 nm which is higher compared to graphite (0.337 nm) due to the O-H functional group present on the surface of our material which comes from its activation with phosphoric acid. The (002) plane indicates a lower degree of graphitization and at a higher degree of disorder which is a valuable property for a highly porous material. This is attributed to the modification of the pore structure upon activation. The same observations have been made by other researchers [40].



**Figure 3:** XRD of the synthesized ACB.

### 3.2.2. FTIR Analysis

The FTIR spectra obtained in Figure 6 of animal activated carbon showed strong bands at 1023, 872, 724, 599 and 558  $\text{cm}^{-1}$  due to the vibrations of  $\text{PO}_4^{3-}$  groups from the natural constituent hydroxyapatite of bone [29, 30, 31, 32, 33]. Bands ranging from 1454 to 1943  $\text{cm}^{-1}$  indicate the presence of C=O, C=C and C=N groups contained in the bone matrix of the organic phase responsible. The band obtained between 2011-3359  $\text{cm}^{-1}$  is due to the vibration of C-H and O-H groups, respectively [27]. The band from 800 to 500  $\text{cm}^{-1}$  is due to the presence of C-H and  $\text{CH}=\text{CH}_2$  groups on aromatic moieties [34], at 3359  $\text{cm}^{-1}$  we notice a diminution of the intensity of -OH bands. The peak at 599  $\text{cm}^{-1}$  corresponds to alkyl halides.

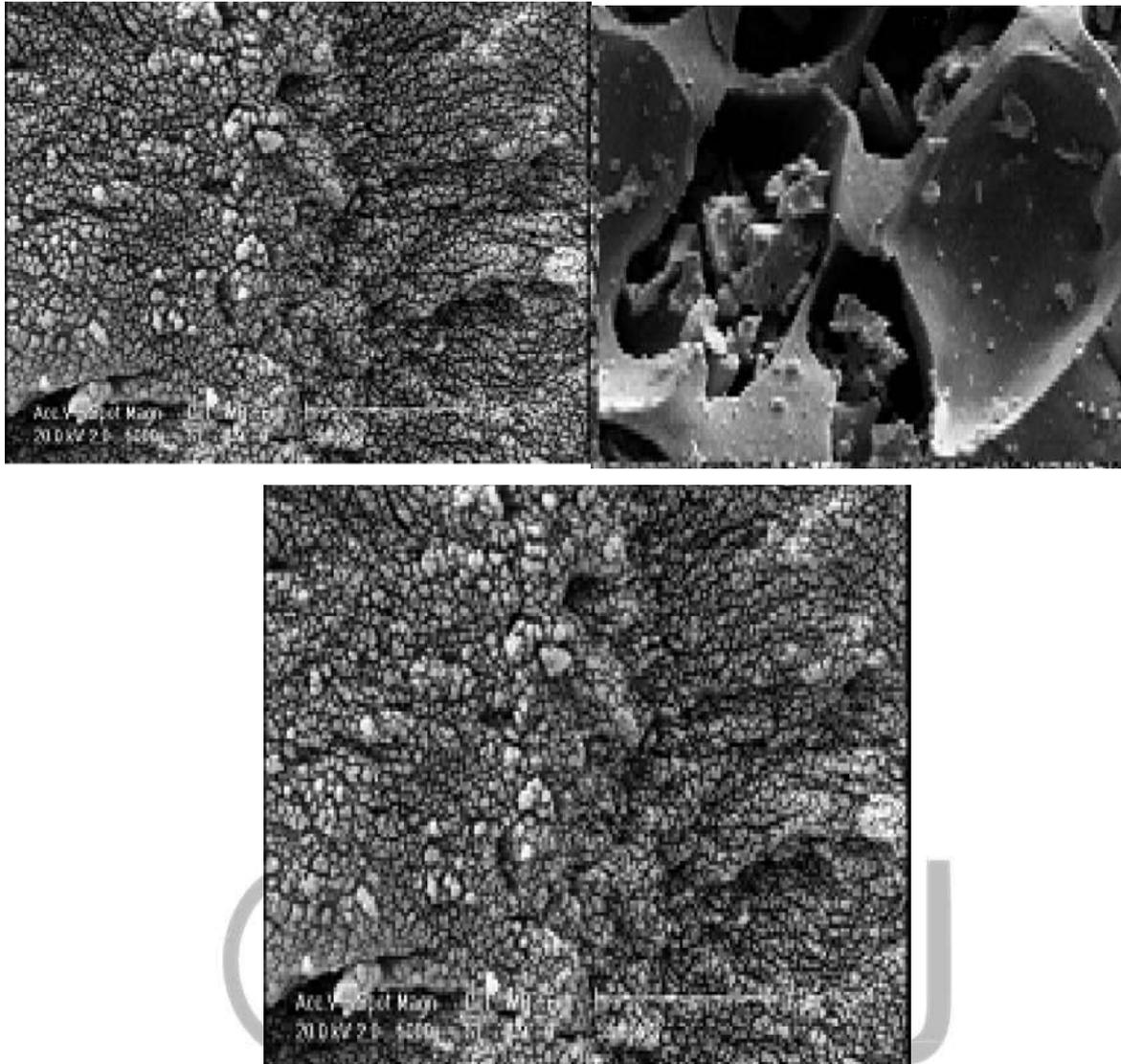


**Figure 4:** FTIR spectra of animal activated carbon.

### 3.2.3. Scanning Electron Micros

The impregnation of phosphoric acid on beef bones is beneficial. This effect will lead to the development of high surface areas and pore volumes in the resulting activated carbons.

The SEM images are indicated in Fig. 7. As it can be seen, the surface of animal activated carbon has porous structure with deep holes in different sizes as the arrow shows in Fig. 7.

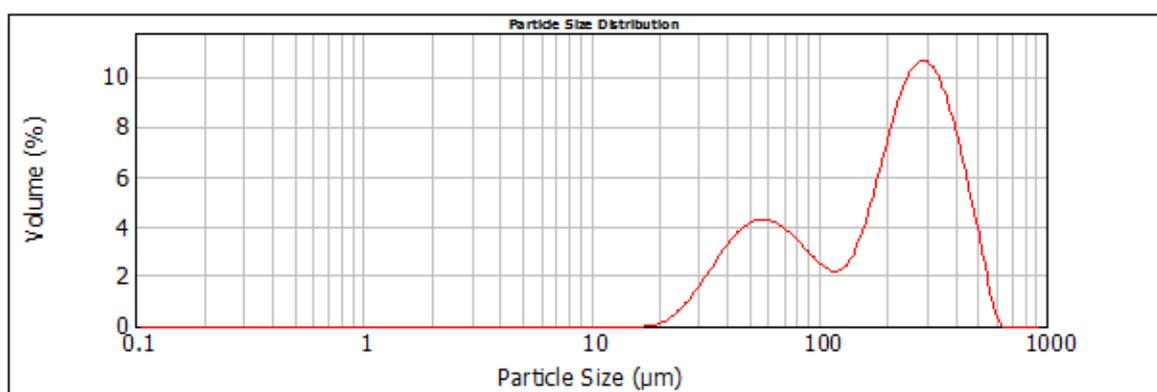


**Figure 5:** SEM images of animal activated carbon.

It can be observed from the micrograph image (SEM) of Figure 7 of animal activated carbon gives an overall view of the shape of the calcined beef bones, it indicates that the surface contains cells of not very homogeneous shapes and an irregular structure. The prepared animal activated carbon contains different sizes and shapes of pores, with a highly developed porosity compared to the raw state. The outer surface of our activated material shows many cavities, and the structure has become perfectly porous. This new structure shows the impact of activating animal activated carbon with phosphoric acid.

#### **3.2.4. Laser granulometry**

The figure below shows the particle size distribution of the animal activated carbon



**Figure 6:** Size distribution of sieved animal activated carbon.

The quality of the results obtained depends very strongly on the operating conditions set for the analysis. For conventional dry operation, the parameters that most influence the measurement are the amplitude of vibration of the hopper which varies between 0 and 100% and the compressed air pressure which also varies between 0.1 and 4 bar. Sample of animal activated carbon has a unique behavior towards pressure. The choice of operating conditions therefore resides in perfect knowledge of the particle system to be analyzed. The analysis conditions applied for the animal activated carbon are: 50% vibration, pressure 4 bar. In this figure, we mainly used a characteristic of this distribution: the mean diameter in volume of Herdan  $D[4.3] = 219.613 \mu\text{m}$ , corresponds to the center of gravity of the volume distribution of our particles of animal activated carbon.

#### 4. Conclusions

Carbonaceous materials constitute an inexhaustible source for both fundamental research and technological developments. This enthusiasm is, in particular, due to the fact that their field of application is extremely wide, ranging from aeronautics to aerospace, including energy storage and water treatment. Carbonaceous materials are thus commonly used in many domestic and industrial applications, particularly in the adsorption of pollutants.

Among the carbonaceous materials, activated carbon, although used for a long time, continues to be developed, particularly due to the increasing demand for good quality water. They are used in water purification channels, in the treatment of urban or industrial wastewater, and also at home. However, the challenge to be met consists in adapting the physicochemical properties of these adsorbents to the resolution of new problems, while

lowering their cost of manufacture and / or of regeneration. This challenge stimulates active research, as shown by numerous scientific publications that appear each year.

This article shows that animal activated carbons of the Gudali species (*Bos Indicus*) are complex porous materials but effective in complexing organic and / or inorganic pollutants. The most important parameter is their high absorption capacity. Animal activated carbons have excellent textural and physicochemical properties which explain their wide use as adsorbents, catalysts and catalyst support. However, it should be noted that the performance of animal activated carbon is intimately linked to the textural properties of the particles and to their surface chemistry. The control of the texture (in particular of the microporosity) is fundamental. It is also clear that it is essential to know the exact nature of the active sites (surface functional groups) and of the molecular surface interactions. All this requires the implementation of investigative and characterization techniques in order to better understand the mechanisms of adsorption and regeneration to optimize and develop new carbons.

Research in the field of animal charcoal intended for water treatment will undoubtedly lead in the mean term to the development of materials that perfectly suite the resolving of specific problems that not only meet a better ease of implementation of operation, but also to new regulatory requirements. The appearance of new varieties of carbon (nanofibers, nanotubes) also portends vast future developments. Finally, it seems, according to recent results on catalytic processes using charcoals as catalyst supports or catalysts, that these coupling methods are high-performance, economical, and technically feasible processes on an industrial scale. It seems that this path is very promising.

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