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Preparation of a new adsorbent from a biomass

N. Guettaf *[1], O. Kaddour, N. Khettab, A. Gherbi, F. Bouarar

[1]Petrochemical synthesis laboratory (PSL) Oil and Chemistry Faculty
University of M'hamed Bougara, Boumerdes, 35000 Boumerdes–Algeria

*Corresponding author: nguettaf626@gmail.com

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ABSTRACT/RESUME

Abstract: *The objective of this study is to prepare an adsorbent from animal bones using pyrolysis methods by chemical activation with phosphoric acid, , The study was started by optimizing the factors influencing the process as a function of mass yield and the specific surface area of the prepared adsorbent. the results of the processes show that the specific surface area of the prepared adsorbent reaches a value of 171.948 m^2 / g, for a maximum mass yield of 60% to 57% with an acid surface of pH = 4.8. This adsorbent is capable of removing medium-sized aromatic organic molecules, heavy metals,……*

I. Introduction

The bones represent about 20% of total weight of the animals, they consist essentially of the organic matter and the mineral matter, Calcium phosphate (CaP) salts are the main mineral components of vertebrate and tooth bones. Among the CaP salts, hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (HAp), is the most thermodynamically stable crystalline phase in body fluids, the most similar to the mineral phase of bone: in fact, CaP of natural origin is usually carbonated and deficient in calcium with a Ca / P ratio <1.67**[1]** while proteins such as prions are the origin of organic matter.

animal bone has found various uses for a long time; The pharmacist Pierre Figuier shows that, even more than the plant, animal charcoal has the singular property of completely absorbing the color of a large number of vegetable or animal solutions, and to make perfectly clear and colorless the water which was charged with it.

The production of phosphorus, which has been extracted from bones since the year 1770 and finds an industrial outlet in the 1830s. Limouzin, a pharmacist in Albi, uses animal charcoal for the discoloration of grape syrup, a temporary substitute for cane sugar **.**And recent studies use bones as a fuel with high calorific value compared to conventional charcoal [2].

and which makes it possible to use them as a substitute fuel for the baking of clinker according to the works [3,4]:inferior Heating Steam (ICP) between 15.7 and 18.6 MJ / kg and upper heat pump (PCS) between 16.9 and 30 MJ / kg

Activated carbon is the final product obtained by carbonization of any material containing a high percentage of carbon, it is commonly used for centuries, in many domestic and industrial applications because of its very high adsorbent power [5,6]. Among the various existing technologies (adsorption, advanced oxidation, membrane filtration ...) for the removal of unwanted material. Activated carbon adsorption is particularly interesting because of its effectiveness, its simplicity of implementation **[7]** and the fact that it also values biomass [8,9], The synthesis of activated carbons from local biomasses by the chemical activation method has been the subject of numerous studies [10,11,12]. The advantage of chemical activation is to operate at low pyrolysis temperatures and a lower activation cost. the present work is to prepared an activated charcoal from animal bones, in order to have an adsorbent which used for the elimination of organic molecules of medium size.
Since the adsorption capacity of an activated carbon depends in part on the adequacy between the pore size and the size of the adsorbed molecules, it is

therefore essential to optimize the production of activated carbon [13,14,15]. For this purpose we are interested in the valorization of animal bones in activated charcoal by chemical means using phosphoric acid as a chemical activator [16,17]. The study consists in monitoring the variation of the specific surface and the mass yield as a function of the temperature of the pyrolysis, residence times, impregnation rate in phosphoric acid, the concentration of the acid … etc.

II. Materials and methods

II.1. Preparation of an adsorbent from the animal bones by carbonization and phosphoric acid impregnation

The raw material used for this study are slaughterhouse waste (the compact bones (cortical) of a cow) that undergoes cooking to dehydrate them (the water tenure goes from about 80% to 1-2%), and degrease them, then grinding and sieving.

The crushed and sieved bones are impregnated in a solution of phosphoric acid with concentrations ranging from 10% to 80% and a mass ratio ranging from 1 to 4 g of acid per gram of bone. The impregnation is done in an oven at a temperature of 120 ° C for impregnation times ranging from 10 to 72 h any one following the protocol described by some work [18]. The impregnated bones are stored in sealed vials until the carbonization tests.

The carbonization is carried out in an adjustable tubular electric furnace of the PROTHERM type. The thermal pretreatment is performed under a fixed flow nitrogen sweep (50 ml / min). the reactor and its contents are baked to begin pyrolysis. The temperature program followed is defined as follows:

A rise of 5 ° C / min from the ambient temperature to the final temperature of pyrolysisccA step of time is required at this temperature; finally, a free descent in temperature until the ambient temperature

Carbonization is done at temperatures ranging from 200 to 800 ° C and at times ranging from 1 to 72 hours. The carbons obtained are cooled to ambient temperature in a desiccator, after removing any residues from carbonization by washing the coals in 0.1 M hydrochloric acid solutions.Then rinsed several times with distilled water until constant pH [19]. The coals thus washed and rinsed are dried at 105 ° C for at least 8 h in an oven, then cooled in a desiccator and kept away from the air in sealed vials until the characterization tests.

II.2. Determination of the specific surface area

BET analysis: This was performed by the adsorption of N_2 at 77 K using Quantachrome Nova Win2 apparatus. Before measuring the adsorption of N_2, the sample was subjected to degassing for 6 h at a final pressure of 133.32×10^{-4} Pa. The surface area or pore size/volume was estimated from the volume of N_2 (as liquid) held at a relative pressure (P/P0) of 0.99

II.3. Determination of the zero charge pH

The pH at the point of no load (pH PZC) is defined as the pH of the aqueous solution in which the solid exists under a neutral electric potential. It is determined for activated carbon prepared using the method of Lopez-Ramon et al. **[20]** Solutions at 0.1 mol.l-1 NaCl and pH between 2 and 10 (adjusted by adding an aqueous solution of NaOH or HCl) were first prepared. The pH meter was used for pH measurement. 0.1 g of dry activated carbon is brought into contact with 20 ml of each of the solutions contained in capped bottles. The suspensions are stirred for 3 days at room temperature. Each solution is then filtered using a filter paper.

II.4. Determination of mass yield

Mass yield is an important quantitative characteristic for activated carbons. It reflects the loss of mass during pyrolysis. This loss of mass indicates the degree of activation. Usually called burn-off **[21].** The expression of the mass yield is given by the following formula:

$$mass\ yeil\ \% = \frac{initial\ mass - final\ mass}{initial\ mass} \times 100$$

II.5.Surface chemical analysis: surface functions

The measurements are made according to the method of Boehm (1966) **[22].** The basic groups are dosed in their entirety, the acid groups are dosed separately using bases of increasing strength. The experimental protocol is as follows:

1 g of dry activated carbon is brought into contact with 100 ml of each of the aqueous 0.1 M solutions of NaOH, Na_2CO_3, $NaHCO_3CH_3CH_20Na$ and HCl. Each solution is stirred for 24 hours, to ensure that a maximum of surface groupings of the activated carbon has reacted.

After filtration, each of the solutions is determined by pH-metry after stirring. The basic solutions are dosed with 0.1 M hydrochloric acid, the acidic solution with 0.1 M sodium hydroxide.

III. Results and discussion

In this work the quality of the efficiency of the prepared carbons is determined by measuring the mass yield and the specific surface area.

III.1. influence of temperature on carbonization before impregnation in phosphoric acid

The first factor that directly influences the animal bone carbonization reaction is the temperature, the

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results in Table 1 give the variation of the specific surface as a function of the temperature, so the best surface area is given for a temperature of 600 °C.(see table 1)

Table 1. *Specific surface area of coals before impregnation in phosphoric acid:*

Carbonization temperature [°C]	**specific surface area [m²/g]**
400	54,057
500	81,031
600	97,392
700	9,998
800	3,4132

III.2. Influence of phosphoric acid on the carbonization of bones

specific surface results: raw bones (without carbonization), carbonized bones without phosphoric acid and carbonized bones in the presence of phosphoric acid are grouped together in Table 2

Table2. *Influence of phosphoric acid on the carbonization of bones for the same operating conditions of temperature and residence time and particle size.*

Sample	**specific surface area [m²/g]**
Raw bone	88,321
Carbonized bone before impregnation in phosphoric acid	41,049
Carbonized bone after impregnation in phosphoric acid	113,611

III.3. Influence of the operating parameters on the specific surface After the impregnation of the bones in the phosphoric acid

The study of the influences on the mass yield and the specific surface is made by varying one of the parameters of the production while keeping the others parameters fixed.

After the impregnation of the bones in phosphoric acid, the experiments were carried out in order to study how the phosphoric acid influences the carbonization of the bones and that they are the maximum values that can be reached if we modify some parameters (temperature, residence time, bone / acid ratio). The results obtained by successive variation of one of the parameters of carbonization show that:

III.3.1. Effect of temperature on the specific surface area

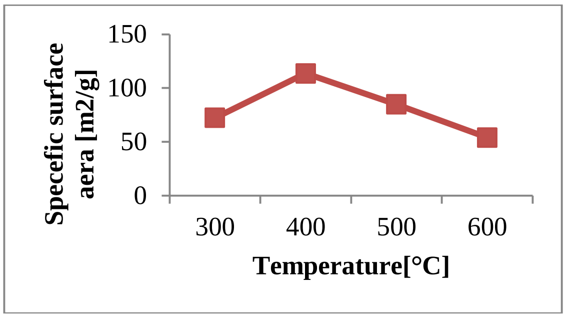

Figure 1. *Effect of temperature on the specific surface area (2.5 mm ≤ Φ, 80 minutes, ratio: bones/acid= 1/1)*

III.3.2. Effect of residence time on the Specific surface area:

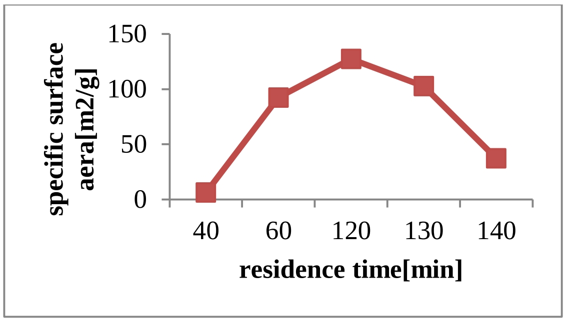

Figure 2. *effect of residence time on the specific surface are (Temperature =400°C , 2,5 mm ≤ Φ ,ratio: bones/acid= 1/1)*

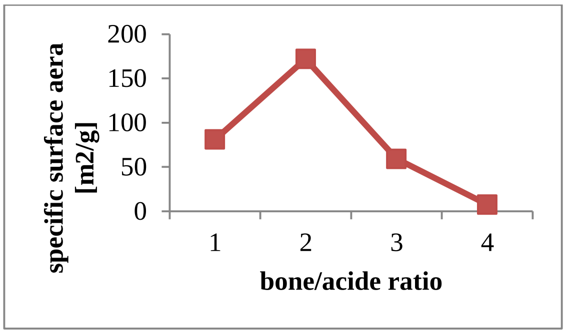

Figure 3. *Effect of bone/acid ratio on the specific surface aera (Temperature =400°C ,2,5 mm ≤ Φ, 120minutes)*

Figure 1: shows that the specific surface increases with the increase of the temperature up to a maximum value of 113.611 m ²/ g for a carbonization temperature of 400 ° C. (fig.1) for 80 minutes, beyond this temperature. the specific surface begins to decrease. a drop in the specific surface area of 84.815 m ²/ g at 500 ° C to41.049 m

^{2}/g at 600 ° C is also noted, more than half for an increase of 100 ° C.
A charring time of 40 min to 120 min, more the residence time increases the surface area increases and it can reach a specific surface of 127.289 m^2/ g for an optimal duration of two hours at a temperature of 400 ° C, (Figure 2) beyond this time the calcination is achieved at high speed, and the coloration of the pyrolysed bones becomes totally white after 20min, this result consistent with some who say that the carbonization time is very short, about 30min**[23]** .and the specific surface area reaches 37,322 m^2/ g for a duration of 140min.
The increase in the bone / acid ratio with an acid concentration of 40% increases promotes carbonization (fig. 3). The results of the experiments give a specific surface area of 81.031 m ^{2}/ g for an bone/ acid ratio = 1 ,while it is equal to the maximum value of 171.481 m^2/g if this ratio (bone / acid) is further increased to 2. For bone / aid ratios greater than 2 The specific surface begins to decrease up to 7.659 m^2/ g for bone/ acid ratio = 4.

as well as large bone particles: bone particles with an average diameter greater than 2.5mm gives a charcoal with a specific surface area of 171.481 m^2/ g while an average diameter of less than 0.8mm gives a specific surface area of 65,072 m^2 / g.

III.4. Influence of the operating parameters on the mass yield

the purpose of studying the influence on the specific surface is to determine the quality of prepared coal; while the influence study on the mass yield is to determine the process performance

III.4.1.Influence of temperature on the mass yield

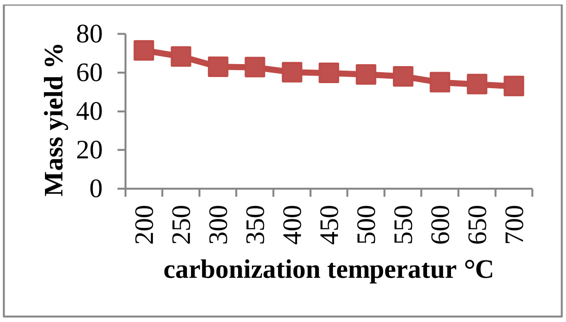

Figure 4. *Temperature effect on the mass yield*

III.4.2.Influence of acid concentration on the mass yield

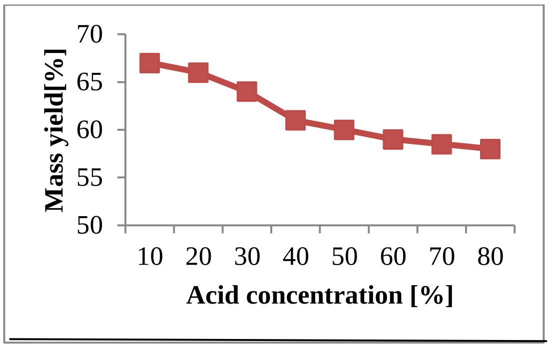

Figure 5. *Acid concentration effect on the mass yield*

III.4.3.Influence of the rate of impregnation on the mass yield

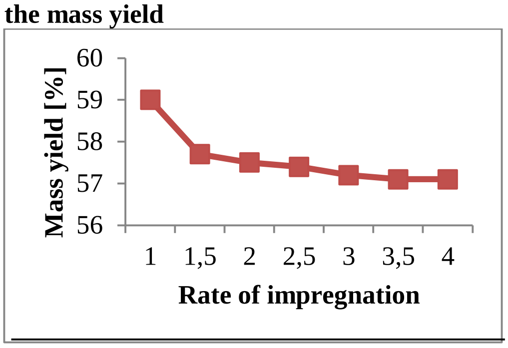

Figure 6. *Rate of impregnation (bone/acid) effect on the masse yield*

III.4.4.Influence of the impregnation time on the mass yield

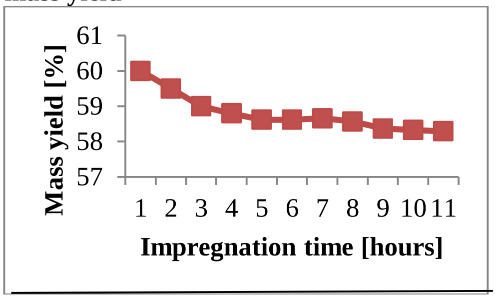

Figure 7. *Impregnation time (of bones in acid) effect on the masse yield*

III.4.5.Influence of the carbonization time on the mass yield

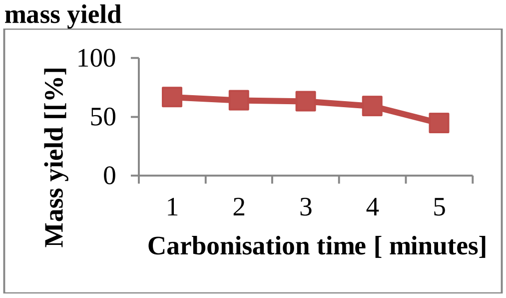

Figure 8. *carbonization time effect on the mass yield.*

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III.4.6. Influence of the particle diameter on the mass yield

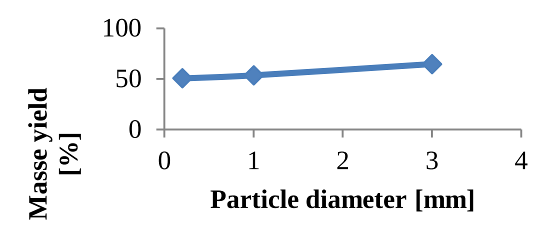

Figure 9. *particle diameter effect on the mass yield*

At a temperature of 200 ° C. for a contact time of 120 minutes, the mass yield went up to 71.5% but the quality of charcoal still remains unsatisfactory, but increasing the temperature to 600 ° C. the yield decreases to at 59.8% and coal quality is improving.(fig.4)

The best mass yield is given by low acid concentrations, low impregnation times and low impregnation rates as well (mass yield of up to 67% for an acid concentration of 10%, a mass yield of 60%).for an impregnation time of 1 hour and a mass yield of 59% maximum for an impregnation rate of 1).(fig.5,6,7)

For a temperature of 400 ° C, the maximum yield for short contact times (for 40min the yield about 66.8%) and the yield decrease with time increasing and becomes between 64% and 44.7% for a time 140min (fig.8)

The larger the particles of the bones, the greater the yield and the maximum yielding to the mass yield of more than 64,5% for the particles of average diameter of 2,5 mm ≤ Φ.(fig.9)

III.5. Surface pH determination

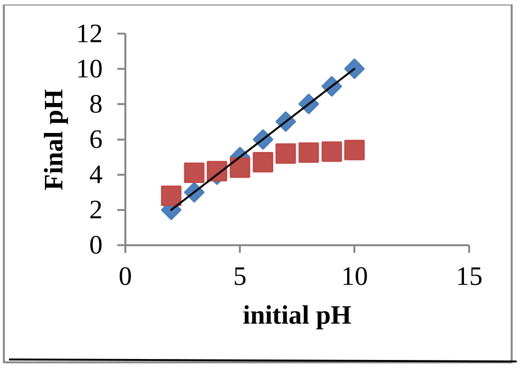

Figure 10. *determination of pH on the surface*

III.5. The influence of the operating parameters on the specific surface

For the results of this study the best charcoal characteristics are obtained for:

The impregnation of the bones in phosphoric acid, because for the same temperature conditions 400 ° C, same residence time of 1 hour and same size of the bone particles (2.5 mm ≤ Φ) the results show that the specific surface increases from 41,049 m^2/ g to 113,611 m^2/ g;

So phosphoric acid is a dehydrating agent for animal matter as dehydrates also the vegetable matter is the result of the work [24,25 ,26] when they studied the activation of vegetable matter (pistachio, walnut, almond and date) by phosphoric acid.

In order to qualitatively analyze the charred animal bones, we used the BET method it is a method that gives an evaluation of the specific surface that directs us towards the adsorption capacity of an activated carbon [24,27] The animal bones are made up of about 95% of the dry matter, which essentially consists of organic matter that is rich in protein such as prions , and hydroxyapatites (PAHs) is the main mineral component of bone, once the phosphoric acid is mixed with bone for a long time and at high temperature, it can react doubly:

First as an agent that promotes crosslinking reactions because of its oxidizing power, followed by condensation and cyclization reactions.

Secondly, it can combine with organic matter to give phosphate and polyphosphate bridges that bind and crosslink biopolymer fragments [24,27],

This induces a formation of grain boundaries that allow the establishment of a solid skeleton through open pores on the outside which increases the specific surface).

In the case of bones the specific surface of the compacted powder increases more than the temperature increases, a group of researchers have noticed that the pores develop as the temperature increases (for plant matter) [28,29] then the specific surface begins to decrease beyond 400 ° C, while the grains are welded with chemical bonds, which are manifested by the appearance of the necks between 600 ° C-800 ° C [30]; The temperature of 400 ° C that is estimated as a carbonization temperature of the bones gives a better specific surface. Finally, the specific surface can also degrade by decreasing the open porosity and appearance of closed pores and is then eliminated by the isolation of the pores of the surface. The growth of grains depends on the thermodynamic and kinetic parameters which is also related to the temperature at which the pores close. A group of researchers have suggested that at higher temperatures the phosphorous compounds are released from the activated carbon surface [24,31]. However, as there is not a formal study that discusses the kinetics of growth of bone grains with respect to temperature, it is difficult to conclude whether time or temperature is predominant.

III.6. Influence of the operating parameters on the mass yield

The mass yield is an important feature of the process performance of the preparation of an activated carbon. It represents the ratio of the mass of activated carbon to the initial mass of raw materials.

The analysis of the results reveals that:

The carbonization temperature has the greatest influence on the mass yield (significant variation in yield). This is explained by the enormous loss of volatiles under the effect of temperature in the presence of phosphoric acid. Theoretically the pyrolysis of animal bones occurs through three essential steps are:

1st step: Dehydration

The first loss of mass is 2 to 8% between 50 ° C and 150 ° C corresponds to the evaporation of water contained in animal bones. [32, 33] and the color of the bones remains almost unchanged during this stage.

2nd: The carbonization and decomposition of all organic matter:

The second step is from 150 ° C to 250 ° C. It results in a significant loss of mass and is associated with the evaporation of low molecular weight compounds. This is explained by the enormous loss of volatile matter (60% -70%) and the high chloride and alkaline content. [33, 34].

This two steps accompanied by a strong smoke release. (For the present study, the large flue gas flow rate was observed around 230 ° C). and the coloring of animal bones begins to change.

3rd step is carbonization:

It is manifested by a loss of net mass between 250 ° C and 550 ° C related to the total decomposition of the organic matter (chain breakage or depolymerization, and breaking C-O and C-C bonds at 240°C to 400 ° C; ; and finally, the beginning of aromatization from 400 ° C, which resulted in the formation of graphitic type. and the coloration of the bones becomes black between 250 ° C and 380 ° C and as the temperature increases the coloring becomes more and more between pink and blue.

4th step is calcination:

It emerges from this study that for a temperature higher than 550 ° C there is total destruction of all the proteins [35,36] that is to say, the pyrolysis or the decomposition of all the organic matter, in fact, the average amount of ash from animal bones is 10 to 31% of the initial mass [37,38, 39], and the yield decreases sharply, which is proven practically by several groups of researchers. For example, the work [40] by activating date kernels with phosphoric acid found that the mass yield decreased from 58% to 9.5% when the carbonization temperature rose from 200°C to 800 ° C. Other groups of researchers have also found that the mass yield decreases as the temperature increases [41].

The duration of impregnation, the carbonization time, the particle size have a lesser influence on the mass yield, while the acid concentration has a significant influence on the mass yield. On the other hand, the degree of impregnation has the smallest influence on the mass yield. Indeed, a huge loss of volatiles occurs causing a rapid decrease in the first hour of carbonization.

III.7. Characteristics of prepared charcoal

The main characteristics of the prepared adsorbent are:

caracteristics	value
1. specific surface aera	171.948 m^2/g
2. pH of surface	4.8
3. surface quality	Acid

IV. Conclusion

The purpose of this work is to prepare an adsorbent from animal bones, based on carbonization in the presence of phosphoric acid. The research was started by studying the influence of some operating parameters on the process mass yield and the specific surface area of the adsorbent produced.

The specific surface of the prepared adsorbent reaches 171.948 m^2/g, for:

A temperature of 400 ° C, beyond this temperature the specific surface area decreases,

A contact time of 120minutes, bone / phosphoric acid impregnation rate of 2, 40% phosphoric acid concentration.

More than the particle diameter of the bones are larger the specific surface area and the mass yield are better.

The mass yield is marked between 57% and 60% for the best carbonization condition.

The value of the surface area of prepared carbons reflects an interesting adsorbent power. Chemical analysis of BOHEM showed the presence of various functional groups on the prepared coal surface.

The prepared adsorbent has an acid surface area of approximately pH = 4.8.

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