The aim of this research is to prepare various carbonaceous materials with different textural, structural and chemical characteristics, using mango seed a rarely used residue for the preparation of activated carbons, as the precursor material. The mango seed was analyzed by TGA and SEM also methodological data about the preparation of activated carbons are provided. Four activated carbons were prepared using sulfuric acid (H₂SO₄) and calcium chloride (CaCl₂) as activating agents and were characterized by means of TGA, SEM/EDX, Boehm Titration, isotherm determination of N₂ adsorption-desorption at −196 °C and immersion calorimetry. Four carbons were obtained with superficial areas BET between 6 and 33 m² g⁻¹ and different chemical characteristics associated with the changes in the concentration of the activating agents. The activated carbons that were prepared with the highest activating agent concentrations, obtained better results in the amount of oxygenated surface groups, the total acidity and the
amount of fixed carbon. The enthalpy of immersion in water was between 7 and 16 J g⁻¹.
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1. Data

Different characterization data are given in this paper; the termogravimetric analysis is presented in Fig. 1, the Scanning electron microscope (SEM) shows an image of the precursor surface in Fig. 2 and also shows images of the surface of the carbons obtained in Fig. 3. Table 1 shows different values obtained in the proximate analysis, given data about the difference between each activated carbon. EDX data are shown in Table 2. Superficial area data are shown in Table 3, also Fig. 4 shows adsorption isotherm of nitrogen on CAC7 at −196 °C. The data of the characterization of superficial chemistry is shown in Table 4. Table 5 shows the values of enthalpy of immersion of the activated carbons. In Fig. 5, the homemade calorimeter used in this research is shown. Fig. 6 shows the calorimetric curve of the immersion of CAS5.
2. Experimental design, materials and methods

2.1. Materials

A large number of mango seeds were collected in a fruit pulp factory. The calcium chloride was purchased from Merck\textsuperscript{tm}, also the sulfuric acid was purchased from Merck\textsuperscript{tm}.

2.2. Preparation of the activated carbon

Different tropical countries have high mango (\textit{Mangifera Indica}) production. Colombia produces more than 260,000 tons of mango per year. The mango seed has no commercial value and is a common...
residue of the industrial processes to obtain mango pulp [1]. Thus, the mango seed has potential as a precursor material for the elaboration of activated carbons due to its characteristics as lignocellulosic material, this is supported by different scientific reports that have demonstrated its versatility in terms of the textural and chemical characteristics that can be obtained by using a particular activating agent.

![SEM images](image_url)

**Fig. 3.** SEM images a) CAC3 b) CAC7 c) CAS5 d) CAS15. The microphotography was taken with Tescan Lyra III microscope.

| Sample | Moisture | Volatile matter | Ash | Fixed Carbon |
|--------|----------|-----------------|-----|--------------|
| CAC3   | 13       | 33.6            | 5.1 | 48.3         |
| CAC7   | 8.2      | 29.9            | 0.9 | 61.9         |
| CAS5   | 6.2      | 31.8            | 0.3 | 61.7         |
| CAS15  | 8.2      | 32.0            | 1.4 | 58.4         |

**Table 1**
Proximate analysis of activated carbon obtained from mango seed.

| Sample | C % Total weight | O % Total weight | S % Total weight | Ca % Total weight |
|--------|-----------------|-----------------|-----------------|------------------|
| CAC3   | 69.14           | 15.9            | --              | 14.96            |
| CAC7   | 65.61           | 26.75           | --              | 7.64             |
| CAS5   | 66.62           | 32.70           | 0.68            | --               |
| CAS15  | 72.70           | 18.94           | 8.36            | --               |

**Table 2**
Quantitative distribution of the elements present in the activated carbons surface determined by EDX.
In that way, different types of activating agents have been used for the preparation of activated carbons. Several data reports show that calcium chloride allows to obtain materials with mesoporous characteristics and the concentration of calcium can directly affect the carbonaceous matrix [5]. In other way, acid activating agents, such as phosphoric or sulfuric acid, are related to generate high microporosity and a much stronger attack on the lignocellulosic structure of the precursor [2,5,6]. Two concentrations were used for each one of the activating agents, looking for a point of contrast in terms of the concentration of the activant and the characteristics given to the activated materials [5,6]. The mango seed was collected in a fruit pulp processing plant and cleaned before impregnation. The seed was dried in an oven at 90°C and then crushed to obtain a mixture of the three parts of the seed (endocarp, tegument and kernel). Four samples of the mixture were taken and impregnated with each one of the raised solutions during 48 hours. The carbonization was carried out in a transverse furnace in N2 atmosphere with a flow of 80 cm³.min⁻¹ with a heating ramp of 10°C min⁻¹, up to a temperature

Table 3
Analysis of S\textsubscript{BET}.

| Sample | S\textsubscript{BET} (m².g⁻¹) | V\textsubscript{0} (cm³.g⁻¹) |
|--------|----------------------------|-----------------------------|
| CAC3   | 12                         | 0.007                       |
| CAC7   | 33                         | 0.01                        |
| CAS5   | 6                          | 0.003                       |
| CAS15  | 12                         | 0.006                       |

Table 4
Surface chemistry characterization (Boehm Titration).

| Sample | Carboxylic groups (mmol.g⁻¹) | Lactone groups (mmol.g⁻¹) | Phenolic groups (mmol.g⁻¹) | Total acidity (mmol.g⁻¹) | Total basicity (mmol.g⁻¹) | pH/PZC (Point Zero Charge) |
|--------|-----------------------------|---------------------------|---------------------------|--------------------------|---------------------------|-----------------------------|
| CAC3   | 0.137                       | 0.089                     | 1.414                     | 1.462                    | 0.139                     | 7.2                         |
| CAC7   | 0.305                       | 1.318                     | 3.852                     | 2.840                    | 1.739                     | 6.7                         |
| CAS5   | 0.352                       | 0.154                     | 1.478                     | 1.676                    | 0.056                     | 5.0                         |
| CAS15  | 0.500                       | 0.219                     | 2.142                     | 2.424                    | 0.365                     | 5.4                         |

Fig. 4. Adsorption isotherms of nitrogen on activated carbon (CAC7) at –196 °C.
of 450 °C, having a residence time of 2 h. Each of the samples was washed with distilled water until a constant pH was obtained.

The samples obtained were called as:
CAC3 (activated carbon with calcium chloride solution 3% p/v).

Table 5
Values of enthalpy of immersion of the activated carbons obtain into distilled water.

| Sample | $-\Delta H_{im}$ H$_2$O (J g$^{-1}$) |
|--------|----------------------------------|
| CAC3   | 10.77                            |
| CAC7   | 16.06                            |
| CAS5   | 7.209                            |
| CAS15  | 8.371                            |

Fig. 5. Immersion calorimeter used in this research. a-b) Cell socket view c) calorimetric cell d)Multimeter e)computer [4–6].

Fig. 6. Calorimetric curve of the immersion of CAS5 into distilled water.
CAC7 (activated carbon with calcium chloride solution 7% v/v).
CAS5 (activated carbon with sulfuric acid solution 5% v/v).
CAS15 (activated carbon with sulfuric acid solution 15% v/v).

2.3. Experimental design

2.3.1. Mango seed data
Thermogravimetric analysis was performed on each of the parts that make up the mango seed, the data are shown in Fig. 1. The loss of mass between the different parts of the seed is considerable; the mango kernel had the least loss of mass. However, the tegument loses all its mass when reaching temperatures close to 500 °C.
The mango seed were characterized by SEM microscopy, given data about the structure it has in the surface (Fig. 2). The microphotography shows a fibrous structure with little or no porous surface, characteristic of the lignocellulosic materials.

2.3.2. Activated carbon characterization data
The four materials obtained were characterized; textural, structural and chemical characterization data are given below. The proximate analysis gives important information about the composition of the activated carbon. In Table 1 data are shown. CAC3 has the lowest percentage of fixed carbon, this relatedation with the low attack of the activant agent; also it has the highest percentage of ash of all the carbons.

The two materials that were activated with the highest concentration, (CAC7-CAS15) have similar moisture, but has difference in the fixed carbon and the volatile matter. Although CAS5 has the lowest percentage of ash it has one of the highest percentage of fixed carbon.

2.3.2.1. SEM characterization. The SEM images show important data of the difference between the activante agents. The CAS15 microphotography shows a huge attack in the surface, generating a lot of macroporous (Fig. 3).

Despite the difference in the concentration, the activant agents done a good work attacking the surface and the lignocelluloses structure, also proving different superficial chemistry (Table 4). In addition, the EDX results are shown in Table 2.
The data reported by the EDX analysis gives important information about the chemical surface composition of the materials. As can be seen in Table 2, the carbons that were activated with calcium chloride reported the presence of calcium on their surface. Moreover, the materials that were activated with sulfuric acid, obtained the presence of sulfur on its surface.

2.3.2.2. Isotherm determination of N2 adsorption-desorption at −196 °C. Fig. 4. Shows the adsorption — desorption isotherms of N2 at −196 °C for CAC7 which obtained the largest surface area (33 m² g⁻¹). This isotherm is type IV and the presence of the type 3 hysteresis cycle is easily observed [7].
The data obtained demonstrate low surface areas for all materials. Calcium chloride generated greater surface areas being 12 and 33 m² g⁻¹ for materials CAC3 and CAC7. On the other hand, the activation with sulfuric acid generated lower areas of the order of 6 and 12 m² g⁻¹, for CAS5 and CAS15 respectively.
Table 4 shows the data obtained from the chemical characterization of the materials, where changes are observed in the content of surface groups with respect to the activating agent and the concentrations that were used.
The materials that were activated using the highest concentrations, obtained greater changes in their surface chemistry, presenting a greater number of surface groups, both carboxylic and phenolic, this data are important to show the difference between two activant agents with a non-common precursor [2–5]. The two carbons that were activated with sulfuric acid, obtained an acid point of zero charge, on the other hand, the materials activated with calcium chloride obtained a charge point closer to neutral. The presence of a great variety of oxygenated groups like phenolic and carboxylic.
could suggest the use of these activated carbons for the adsorption of certain metal ions in aqueous solution.

2.3.2.3. Immersion calorimetry of activated carbon in distilled water. The four activated carbons obtained were immersed in distilled water using a Tian-type calorimeter Fig. 5 that was built in our laboratory. The intensity of the interaction between the solution and the activated carbon can be determined by means of the enthalpy of immersion of the solid in solutions containing active substances or in this case water. The immersion calorimetry in water, allows to relate the content of oxygenated groups in the surface of the materials and their specific interaction with the polar molecules of water. This technique allows to know the energy that is manifested in the surface as well as relating its results with other data obtained from the chemical characteristics of the materials.

The procedure to obtain the enthalpy of immersion consists of placing 10 ml of solvent in a metal cell Fig. 5 (c). In the heat reservoir of the calorimeter at 298K; the calorimeter registers the output of electric potential as long as it has reached a baseline over time. Then, 100mg of the activated carbon sample is placed in a glass vial and the immersion is performed [8,9]. If the immersion was satisfactory and the baseline is obtained again, the electrical calibration is performed. The variation of the electric potential versus time can be used for the calculation of the immersion enthalpy [8,9]. Fig. 6 shows the data obtain; the first observed peak corresponds to the immersion process, when the solid get contact with the solvent, the second one corresponds to the electrical calibration of the calorimeter.

The data of the immersion enthalpies of the materials obtained are shown in Table 5. The materials that were activated with calcium chloride (CAC3–CAC7) obtained the higher values in the immersion calorimetry; otherwise the materials that were activated with sulfuric acid got the lowest values; these values can be related with the oxygen groups in the surface of each material and also with the total acidity and basicity shown in Table 3, this data is important because it gives information about the several interactions between the surface of the carbons and different molecules, in this case with water.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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