INFLUENCE OF THE PRECURSOR ON THE RESULTS OF CARBONIZATION

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Introduction

Lignocellulosic materials are derived from plant tissues. They are composed of varying percentages of the major botanic components: holocellulose (cellulose and hemicellulose), lignin, which compose the cell walls and, intercellular substances commonly called extractives in reference to their analytical determination procedure. The use of lignocellulosic materials for preparation of activated carbons is well known [1]. So, Guava (Psidium Guajava [2]) seeds, Tropical almond (Terminalia Catappa [2]) and Dende (Acrocomia Karukerana [2]) shells were selected on account of their abundance as by-products from tropical agricultural industries and their low cost to be converted in activated carbons. Physical activation involves carbonization of the raw material in an inert atmosphere followed by the activation of the resulting char in the presence of an oxidizing gas. Here, we report the results of a study directed toward carbonization.

Experimental

Approximately 5 g samples of Guava seeds, Tropical almond and Dende shells are introduced, respectively, in an alumina sample holder. Then, the whole is placed in the reactor made of a cylindrical quartz tube crossing an electrical furnace, whose, temperature is recorded to ± 2°C. The sample is then pyrolysed at a constant heating rate (10°C/min) to the desired temperature for 2 hours, under a constant flow of nitrogen (90 mL/min). The results are analyzed according to the char yield obtained and according to the micropore volume developed during pyrolysis. Yield is defined as the ratio of the char mass obtained to the seed or shell mass introduced. D-R micropore volume of the char is determined by gas-adsorption analysis using N2 at 77 K (Sorptomatic 1900 apparatus). Botanical characterization of the precursors are performed using extraction methods [3] and the textural analysis is realized by pycnometer measurements.

Results and discussion

Figure 1 displays the char yield as a function of the temperature treatment for the three precursors. The slope of the plot is more important for Tropical almond (1.7.10⁻² %/°C) and Dende shell chars (1.3.10⁻² %/°C) than for Guava seed chars (0.7.10⁻² %/°C). The loss of weight is regular for Guava and Dende chars, accelerates roughly between 700-850°C for Tropical almond shell char. Carbonization of Dende shells gives the best char yield whatever the temperature. At 800°C, the char yield for Tropical almond, Dende shells and Guava seeds is respectively 28.2, 31.5 and 28 %. The results show that the char yield is as high as the precursor major components (holocellulose and lignin) are thermally stable and as the stabilization reactions between components are favored. According to table 1, the whole precursors are rich in holocellulose, moderately rich in lignin and have a low ash content. Tropical almond shell is characterized by the highest content in holocellulose whereas Dende stone is by the highest content in lignin. Only Guava is rich in extractives. Cellulose for Tropical almond shell must be so much less orientated, so much less crystalline that it can not be compensated by the presence of even a good binder (lignin) in high quantity during the pyrolytic process. The high char yield for Dende shell is explained by the good quality of its components and that of Guava seed by the good stabilization reactions between its components. Figure 1 also shows how temperature affects microporosity development accessible to measurement. When temperature increases, microporosity increases too. Tropical almond shell char exhibits the strangest behavior having a micropore volume as early as 700°C whereas it begins to appear at 900°C for Guava seed char. The porosity development is the consequence of the achievement of scission reactions and of internal recombinations of the char. It is dependent upon the original texture, structure, density, volatile matter content of the precursor. Tropical almond shell has the more developed inherent porosity, it is also the least dense precursor, then, the development of porosity is facilitated. Some inorganics can also exert an effect on char yield and on porosity development. For example, the presence of Silicium in Dende shell increases the char yield. And, for Tropical almond shell, the presence of potassium favors the development of
porosity. However, the minimal and the critical contents of a catalytic species affecting the char yield are different for each material (for example, the proportion of calcium must be not sufficient to calalyse the development porosity for Guava seed char).

Conclusion

The parameters affecting the char structure and texture (quantity/quality of the components, presence of catalytic species, inherent porosity) are studied in order to determine which properties are necessary for the precursor and consequently for the resulting char to obtain an optimum activated carbon (high char yield and porosity). The high char yield and the good development of porosity obtained during pyrolysis, without shrinking core of the particle, make Guava seed an ideal material for the preparation of activated carbons.

References

Table 1. Botanical analysis of the three precursors.

<table>
<thead>
<tr>
<th>precursor</th>
<th>Guava seeds</th>
<th>Dende stones</th>
<th>Tropical almond shells</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lignin (% weight)</td>
<td>41.7</td>
<td>45.4</td>
<td>40.3</td>
</tr>
<tr>
<td>Cellulose</td>
<td>28.0</td>
<td>24.9</td>
<td>37.9</td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>15.5</td>
<td>27.0</td>
<td>21.6</td>
</tr>
<tr>
<td>Extractives</td>
<td>16.8</td>
<td>1.1</td>
<td>1.6</td>
</tr>
<tr>
<td>Ash</td>
<td>0.4 (Ca: 0.08)</td>
<td>0.9 (Si: 0.34)</td>
<td>0.2 (K: 0.01)</td>
</tr>
<tr>
<td>total porosity (cm³/g)</td>
<td>0.2</td>
<td>0.2</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Figure 1. char yield and micropore volume evolutions as a function of temperature
(Discontinued lines and blank captions are for the evolution of micropore volume calculated by N₂ adsorption isotherm; full lines and dark captions are for the evolution of char yield).