Preliminary Evaluation of Moringa Oleifera Seed Shells as Precursor for Activated Carbon

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Abstract: The proximate and ultimate characteristics of Moringa Oleifera (MO) seed shells were used to evaluate the suitability of the material as a precursor for activated carbon production. The results showed remarkable influence of particle size on proximate characteristics of the MO seed shell. The variation in weight loss, rates of dehydration and de-volatilization of the material with varying particle size was used to assess the particle size that could be best suited for carbonization. The particle sizes of 400 – 1400 μm have been suggested in this study to be the most suitable for producing good quality activated carbon with good yield; 1400 μm being the recommended optimum particle size of this cellulosic material for carbonization.

Key words: Moringa, Seed shells, precursor, Proximate, Ultimate, Activated carbon.

INTRODUCTION

Moringa Oleifera (MO), also called ‘drum stick’ (because of the shape of its pods) and ‘horseradish’ (describing the taste of its roots) grows rapidly from seed or vegetative propagation in the tropics and can survive long periods of droughts. Fully matured, dried seeds of MO are round or triangular shaped and the kernels are surrounded by a lightly wooded shell with three papery wings (Schwarz, 2000). The use of crushed seed powder of MO as a replacement coagulant for proprietary coagulants meets the need for water and wastewater technology in developing countries because it is simple to use, robust and cheap to install and maintain (Folkard et al., 1994). Planting of MO trees by smallholder farmers is being encouraged because of the potential it holds to improve both their health and income (Folkard et al., 2005). The cultivation and usage of MO have given rise to considerable amounts of its seed shells as waste material, which could be used as a precursor for activated carbon production.

Proximate analysis describes the moisture, ash, volatile matter and fixed carbon while ultimate analysis points at the elemental composition. These analyses have been used for the determination of characteristics of carbonaceous materials that may be used as precursors for activated carbons (Lori et al., 2007). It’s been reported that during carbonization at 250°C and latmospheric pressure, elements such as hydrogen and oxygen or oxides are eliminated from the cellulosic structure of the precursor to produce char as shown in the equation below (Zanzi, 2000, Lori et al., 2007).

\[
(C_6H_{10}O_5)_n + O_2 \rightarrow C_n + nCO + nCO_2 + nH_2O
\]

(Char Carbon)

High moisture content (as steam) of the precursor would aid the conversion of the char carbon as shown in the equation below (Zanzi, 2000):

\[
H_2O + C \rightarrow H_2 + CO
\]

\[
2H_2O + C \rightarrow 2H_2 + CO_2
\]

\[
CO_2 + C \rightarrow 2CO
\]
This results in low yield of the carbon. The reaction of the char carbon with steam is thermodynamically endothermic and the mechanism is reported (Herbert et al., 1975; Kim et al., 1998) as:

\[ \text{C} + \text{H}_2\text{O} \rightleftharpoons \text{C(H}_2\text{O)} \]
\[ \text{C(H}_2\text{O)} \rightarrow \text{H}_2 + \text{C(O)} \]
\[ \text{C(O)} \rightarrow \text{CO} \]
\[ \text{C} + \text{H}_2\text{O} \rightarrow \text{CO} + \text{H}_2 \]

The amount of inorganic components of carbon precursors has been reported to greatly influence porosity-development of porous carbon by blocking pore entrances during the activation process (Yun et al., 2002). Thus, the ash content, which is the inorganic residue after the organic matter is burnt away, should be very low for a high yield of the carbonaceous product of carbonization. The proportion of solid carbon in the carbonaceous product is the fixed carbon. A precursor for activated carbon therefore, requires a high proportion of fixed carbon and high volatile matter content to produce a highly porous carbon.

Heavy metal content of the precursor should be low or removed to avoid possible contamination of the environment, which could arise when used activated carbon materials are disposed.

This work therefore reports the proximate and ultimate characteristics of Moringa Oleifera as it affects suitability for the production of activated carbon.

**MATERIALS AND METHODS**

Dry MO pods were collected from naturally growing plants within Markurdi metropolis in Benue State of Nigeria. The dry MO pods were further air-dried for two weeks to ease removal of the seeds from the pods. The seeds were air-dried for two weeks to ease de-shelling. The seed shells of the MO were air-dried for 3 months on polypropylene sack sheets until they were properly dried for effective pulverization. The dried shells were pulverized using Sprecher Schuh Industrial Control Pulverizer (BICO, USA) and sieved into samples of different particle sizes (90, 125, 180, 250, 355, 500, 710, 1000, 1200, 1400 & 1880 μm) with various Endecotts Laboratory test sieves on Octagon Digital sieve shaker; all at National Metallurgical Development Center, Jos. The samples were packed into polythene bags and labeled accordingly.

Standard protocols described in official methods of analysis by AOAC (1990) were employed for the determination of moisture, ash and volatile matter while the ultimate characteristics were determined using Energy Dispersive X-Ray Fluorescence Spectrometer (ED-XRFS) MiniPAL4 Model © 2005.

20g of the 250μm sieved MO seed shells sample were intimately mixed with Cellulose flakes binder in a ratio of 5:1 and pelletized at a pressure of 197.61kPa (13tons/inch²) and stored in a desiccator. The ED – XRFS machine was warmed-up for 2hrs and the appropriate program for the various elements of interest selected. The machine was loaded with the sample and the result of analysis (% concentration) of the elements was recorded.

**RESULTS AND DISCUSSION**

The results obtained show that particle size has a remarkable influence on proximate characteristics of the MO seed shell as a precursor for activated carbon. Figure 1 shows fluctuations in moisture for particle sizes 90 – 355 μm; while particle sizes greater than 355 μm did not show any appreciable variation in the moisture content. The mean moisture of the MO seed shell varied from 2.67 to 4.05%; with a total mean moisture of 3.56 ± 0.48%.

The correlation between particle size and moisture content was similar to that observed between particle size and rate of dehydration of the MO seed shells (Figures 1 & 2). The rate of dehydration of the MO seed shells fluctuates for particle sizes 90 – 355 μm; while particle sizes greater than 355 μm did not show any appreciable fluctuation in dehydration rate. This observation is consistent with the reported possible shrinkage of matrices of cellulosic fine particles with the formation of a solid mass that could even have a higher

2H₂ + C → CH₄
density; when heated (Gimba, 2001, Lori et al., 2007). Zanzi (2000) reported this observation for dehydration of cellulose to stable anhydrous cellulose. This observation is vital in the consideration of MO seed shells as precursor for production of activated carbon. It suggests that carbonization of this seed shell of particle sizes 90 – 355 μm is prone to low yield since its tendency to show increased and decreased moisture content when heated is likely to support conversion of carbon to CO or CO₂. Particle sizes higher than these are likely to favor uniform dehydration. The total mean rate of dehydration was 6.0 ± 0.01 x 10⁻²% min⁻¹.

Particle size also influenced de-volatilization of the MO seed shells in a similar pattern to Figure 2. Only particle sizes above 355 μm were found to de-volatize uniformly. This observation is consistent with de-volatilization of bagasse, sorghum and millet straw reported by Lori et al., (2007). The non-uniform de-volatilization trend observed with particle sizes less than 355 μm could be as a result of the small particles containing large domains, rich in organic and volatile materials that may tend to form intermediate solids which require slightly higher heat than that required by the original matrix, which may be made heterogeneous in particle sizes by the new intermediate solids (Lori et al., 2007). This observation is also supported by the fact that heat flux and heating rate is higher in small particles than in large particles, and this could reduce the yield of activated carbon (Zanzi; 2000). Weight loss characteristics of cellulosic materials are related to their volatile matter. A mean total volatile matter of 73.78 ± 2.28% and mean weight lost rate of 1.23 ± 0.04% was obtained for the MO seed shells.

The ash content (Figure 5) reveals that, the lower the particle size, the higher the percentage ash content. This result is in consonance with that reported for bagasse, sorghum and millet straws (Lori et al., 2007), and is an indication of high percentage of inorganic, oxidizable and volatile materials. The lower the ash content, the better suited is the material for the production of activated carbon. The variation in ash content is non-uniform for particle sizes below 400 μm while greater particle sizes showed a uniform trend. The total mean ash content of 5.67 ± 0.82% was obtained.
Particle sizes of 90 – 355 μm were also observed to have a considerable influence on the fixed carbon content of the MO seed shells (Figure 6) while sizes greater than 400 μm showed no remarkable influence on the tendency of this cellulosic material to produce high solid contents; following carbonization. High ash content associated with fine particle size enhances the conversion of biomass to gaseous products (Zanzi, 2000), thereby reducing the fixed carbon content (Lori et al., 2007). This could explain the low fixed carbon content obtained for particle sizes 90 – 355 μm. This observation is consistent with that reported for bagasse, sorghum and millet (Lori et al., 2007). The mean fixed carbon content of 17.00 ± 4.04% was obtained for the MO seed shells.
Fig. 6: Effect of Particle Size on Fixed Carbon content of Moringa Oleifera Seed Shell

The result of the ultimate characteristics (Table 1) suggests the need to analyze the activated carbon that may be produced from MO seed shells so as to monitor trace metal residues in the carbon. Baquero (2003) and Lori et al., (2007) reported that washing of the resulting activated carbon with hydrochloric acid and de-ionized water may be adequate for the removal or reduction in level of the heavy metals.

<table>
<thead>
<tr>
<th>Table 1: Ultimate characteristics of Moringa Oleifera Seed Shell</th>
<th>(mg/g shell)</th>
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<tbody>
<tr>
<td>Element</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>0.46</td>
</tr>
<tr>
<td>Si</td>
<td>1.44</td>
</tr>
<tr>
<td>P</td>
<td>38.81</td>
</tr>
<tr>
<td>S</td>
<td>98.17</td>
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<tr>
<td>K</td>
<td>296.35</td>
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<td>Ca</td>
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<tr>
<td>V</td>
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<td>Cr</td>
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<td>Mn</td>
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<td>Fe</td>
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<td>Ni</td>
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<tr>
<td>Cu</td>
<td>21.64</td>
</tr>
<tr>
<td>Zn</td>
<td>4.90</td>
</tr>
</tbody>
</table>

Until recently, proximate and ultimate characteristics of carbon precursors have not been used as a veritable tool for the selection of materials and conditions prior to carbonization. Investigations of new precursor materials, before now, involved carbonizing at different residence times to ascertain their suitability for highly porous activated carbon (Zanzi, 2000; Gimba, 2001; Garcia-Perez, 2002; Jaguaribe et al., 2005). Lori et al., (2007) reported the use of proximate and ultimate characteristics of carbon precursors (bagasse, sorghum and millet) for selection of material and possible conditions for high yield and highly porous carbon during carbonization. This study employs this novel approach to plan for production of activated carbon from MO seed shells.

**Conclusion:**

Results of the evaluation show that particle size has a remarkable influence on the proximate characteristics of MO seed shells. The rates of dehydration and de-volatilization suggest that particle sizes 400 – 1400 μm, which also show low ash content, would favour the production of good quality activated carbon with 1400 μm being the suitable optimum particle size of this cellulosic material. As a result of the presence of heavy metals in the material, it’s recommended that the produced activated carbon should be washed with hydrochloric acid and de-ionized water. Production of activated carbon from the MO seed shells is currently being studied.

**ACKNOWLEDGMENTS**

We acknowledge the assistance of Vershima Aondo (National Metallurgical Development Center (NMDC), Jos, Nigeria) for pulverization and sieving of the samples, Chagga (National Metallurgical Development Center
(NMDC), Jos, Nigeria) for the XRF analysis, the Head of Quality Assurance Laboratory (Nasco Household Products Ltd., Jos, Nigeria) for the Laboratory space and equipment.

REFERENCES


