



King Saud University  
Arabian Journal of Chemistry

[www.ksu.edu.sa](http://www.ksu.edu.sa)  
[www.sciencedirect.com](http://www.sciencedirect.com)



## ORIGINAL ARTICLE

# Effect of carbonization on the processing characteristics of rubber seed shell



L.O. Ekebafé<sup>a,\*</sup>, J.E. Imanah<sup>a</sup>, F.E. Okieimen<sup>b</sup>

<sup>a</sup> Department of Polymer Technology, Auchi Polytechnic, Auchi, Nigeria

<sup>b</sup> Biomaterials Research Centre, Department of Chemistry, University of Benin, Benin City, Nigeria

Received 30 December 2011; accepted 23 July 2012

Available online 8 August 2012

## KEYWORDS

Carbonization;  
Filler;  
Characterization and rubber  
seed shell powder

**Abstract** The effect of carbonization on the processing characteristics of rubber seed shell powder was studied. Rubber seed shells were carbonized at different temperatures and then ground into fine powder. The various powders obtained were then characterized by pH, bulk density, moisture content, iodine adsorption value, yield%, conductivity and loss on ignition. The results show that there was a significant change in the pH as the heating temperature increases. The bulk density and moisture content decrease with increasing heating temperature while the iodine adsorption number and the loss on ignition increase with increasing heating temperature thus showing that carbonization has a significant influence on the processing characteristics of rubber seed shell.

© 2012 Production and hosting by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/3.0/>).

## 1. Introduction

Fillers in rubber compounds play a major role in physico-mechanical properties and exercise control over cost of the products (Bledzki and Gassan, 1999). It is well established that carbon black is one of the most important classical reinforcing fillers, especially for the rubber industry. However, carbon black is expensive and petroleum from which it is derived is non renewable (Mwaikambo and Ansell, 2002). Therefore, considerable research and development efforts are being carried out to investigate the possibility of replacing this filler with

renewable raw materials as fillers (Okieimen and Imanah, 2005).

The use of carbon from agricultural by-products (maize cob groundnut husk, cassava peel, cocoa pod husk, plantain peel, rubber seed shell, etc.) for producing vulcanizate materials that are competitive with synthetic composites has been gaining attention in the last decade because of availability of materials, easy processing, low cost, and high volume applications (Okieimen and Imanah, 2005; Okieimen and Imanah, 2003).

Agricultural residues as by-products and co-products of agriculture and processing of agricultural products represent a large feedstock of underutilized resources which can be used directly or converted by fairly simple chemical processes into higher value added materials.

Rubber seed shell is an agricultural by-product of the rubber tree. The economic importance of the rubber tree has largely focused on the rubber latex with little attention paid to the potential usefulness of its by-product. While significant progress has been made in the development and utilization of

\* Corresponding author. Tel.: +234 8029320114.

E-mail address: [lawekebafé@gmail.com](mailto:lawekebafé@gmail.com) (L.O. Ekebafé).

Peer review under responsibility of King Saud University.



Production and hosting by Elsevier

modified agricultural by-product in water and wastewater treatment, (Toles et al., 1997; Ricordel et al., 2001; Imanah and Okieimen, 2003; Jideonwo and Utuk, 2001) there is little information on the potential for the application of these by-products.

Previous studies (Sun and Jiang, 2010; Ekebafé et al., 2010) revealed that the temperature at which carbonization of agricultural by-products is carried out could affect the characteristics of the carbon obtained and therefore, the physico-mechanical properties of the rubber vulcanizate. The present research work was undertaken, with an objective to explore the influence of carbonization on the processing characteristics of rubber seed shell.

## 2. Experimental

### 2.1. Materials

Rubber seeds were obtained from the Rubber Research Institute of Nigeria, Iyanomo, Benin City, Nigeria. All reagents used were of commercial grade.

### 2.2. Preparation of the rubber seed shell carbon

The shells were separated from the seeds, air-dried and reduced to small sizes.

Eight samples of 1 kg each were weighed and heated to temperatures: 100, 200, 300, 400, 500, 600, 700, and 800 °C for 3 h using the METM-525 Muffle furnace. The carbonized shells were then milled to fine powder and sieved through a mesh size of 150 µm.

### 2.3. The characterization of the rubber seed shell carbon

The rubber seed shell carbon (RSSC) was characterized as follows: loss on ignition was determined gravimetrically and expressed in terms of percentage, it consists of strongly heating (“igniting”) a sample of the material at a specified temperature (850 °C), allowing volatile substances to escape, until its mass ceases to change (Ball, 1964), the moisture content was determined by the method described in ASTM D 1509, by drying the sample to a constant weight and the amount evaluated in percentage (ASTM D 1509, 1983), the bulk density was determined by tamping procedure according to the method described by Ishak and Baker. Accurately weighed samples of

the RSSC were poured into a uniform cylinder of cross sectional area and were then tapped several times until there was no change in the volume occupied. This volume was then recorded and the bulk density calculated (Ishak and Baker, 1995), the pH was determined by using ASTM D 1512 method, based on the pH of water in equilibrium with the active carbon. 1.0 g samples were immersed in 20 ml of deionized water respectively and stirred for 15 min, then, the pH determined using the pH meter (ASTM D 1512, 1983), the method used for the surface area measurement of fillers is the iodine adsorption number, 0.1 M sodium thiosulfate solution was titrated against 20.0 ml of 0.488 M iodine solution containing 0.5 g of RSSC sample using 5.0 ml of freshly prepared starch solution as indicator. Similarly, the thiosulfate was titrated with 20 ml of 0.488 M iodine solution without RSSC samples (blank) and the quantity needed to titrate the blank was determined. The surface area of RSSC was evaluated using the inverse of the iodine value (Ahmedna et al., 1997), the conductivity was determined based on the conductivity of water in equilibrium with the active carbon, by using the Ela conductivity meter, using the same samples as prepared for the pH determination. The calcium and magnesium contents of RSSC were determined by complexometric titration, while the sodium and potassium contents were determined by flame photometry. The yield% is the difference in weight of the respective samples before and after carbonization.

## 3. Results and discussion

The weight of the rubber seed shells on carbonization for 3 h at different temperatures shows a progressive decrease as the temperature increases due to the combustion of the residual materials in the shells. The relationship between weight after carbonization and the carbonization temperature is shown in Fig. 3.1. Most fibers are composed of cellulose, hemicellulose, lignin, waxes, and some water-soluble compounds, where cellulose, hemicelluloses, and lignin are the major constituents (Eichhorn et al., 2001). The percentage composition of each of these components varies for different fibers. Generally, the fiber contains 60–80% cellulose, 5–20% lignin and up to 20% moisture. The moisture content of the rubber seed shell is as shown in Table 1.

The variation of the pH of the rubber seed shell carbon as a function of the carbonizing temperature and the metal content of the seed shell is shown in Fig. 3.2 and Table 1. The pH var-

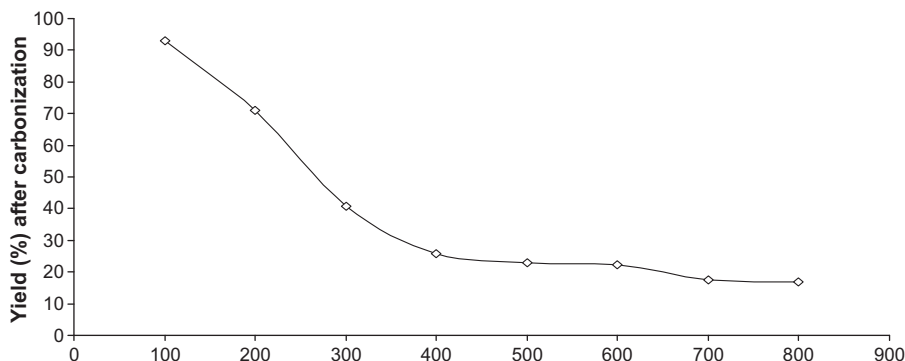


Figure 3.1 Variation of yield of carbon with carbonization temperature.

**Table 1** Metal content and other characterization results of rubber seed shell carbon.

|                                |                   |
|--------------------------------|-------------------|
| Moisture content at 125 °C (%) | 5.78 (2.64, N330) |
| Magnesium (%)                  | 0.673             |
| Sodium (%)                     | 0.014             |
| Potassium (%)                  | 0.01              |
| Calcium (%)                    | 0.32              |

Moisture content of N330 CB is in parentheses.

ied over a range of 4.79–8.77. The results show a progressive increase in pH with increase in carbonization temperature, the possible reason could be that as the residual material is being lost on combustion, the metal content activity is enhanced in view of the Magnesium, Sodium, Potassium and Calcium oxide contents in the seed shell (Table 1) which increase alkalinity and hence pH. These corroborate the results of Sun and Jiang (2010).

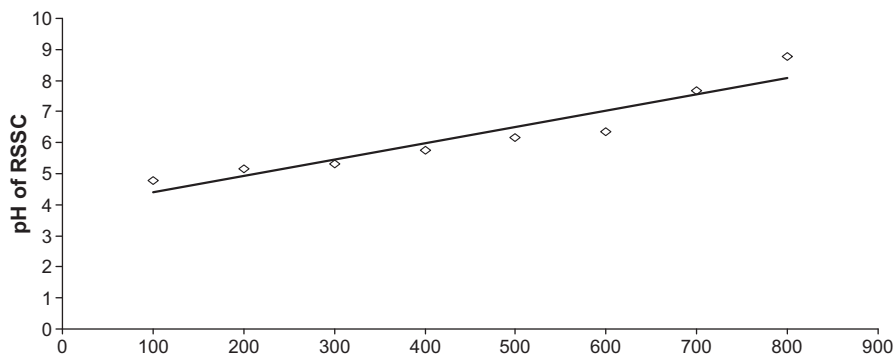
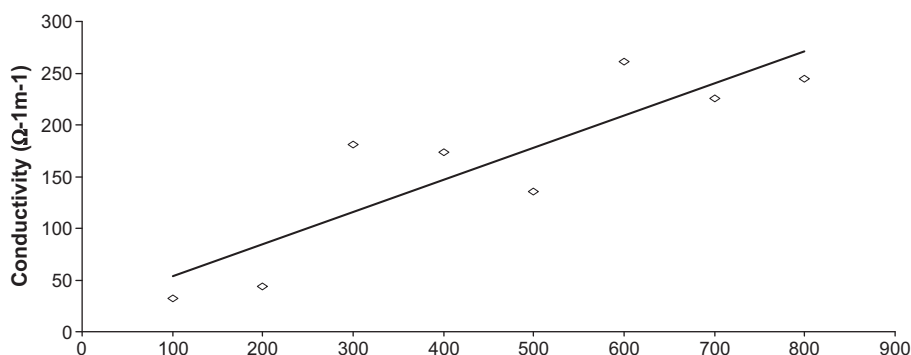
Electrical conductivity is defined as the conducting power of all ions produced by 1 g of electrolyte in solution and it has a reciprocal relationship with resistance (Sharma and Sharma, 2004). The results in Fig. 3.3 show that the electrical conductivity of RSSC increases with the rising of temperature. At 600 °C, the resistance in RSSC becomes very small, meaning good conductivity, while above 600 °C the conductivity descended slightly.

The bulk density is principally influenced by the particle size and structure of the fiber and the lower the particle size

the lower the bulk density and therefore better the interaction between the polymer matrix and the reinforcing filler, this would enhance the processing and improve the quality of the end product as the desirable properties for particulate fillers include excellent tensile strength and modulus, high durability, low bulk density, good mouldability and recyclability (Sharma and Sharma, 2004). This further explains the reasons for the results in Ekebafe et al. (2010). Variation in Fig. 3.4, shows that at high heating temperature, the bulk density reduces which may be due to the opening of the interstitial spaces (microspores) in the carbon residue.

The iodine adsorption number from Fig. 3.5, reveals that as the filler carbonizing temperature increases the amount of iodine adsorbed per 100 g of the material increases. One important application of iodine adsorption number is that it elicits the surface area of the material. It is a parameter that indicates the macrostructure of fillers and reflects the reaction and adsorption abilities (Moloney, 1995). Like wood fiber, at high temperature, all kinds of porosities will form inside RSSC, which bring RSSC a certain specific surface area, reaction and adsorption capacity. The variation between the iodine adsorption number and the filler temperature is shown in Fig. 3.5.

The maximum iodine adsorption number (66.75 mg/100 g) was obtained when the carbonizing temperature reaches 600 °C; eliciting the fact that maximum surface area occurs at this temperature; the surface area value is much smaller when carbonizing under low temperature (200 °C) due to the low porosity resulting from incomplete carbonization.

**Figure 3.2** Variation of pH of RSSC with carbonization temperature.**Figure 3.3** Variation of conductivity of RSSC with temperature of carbonization.

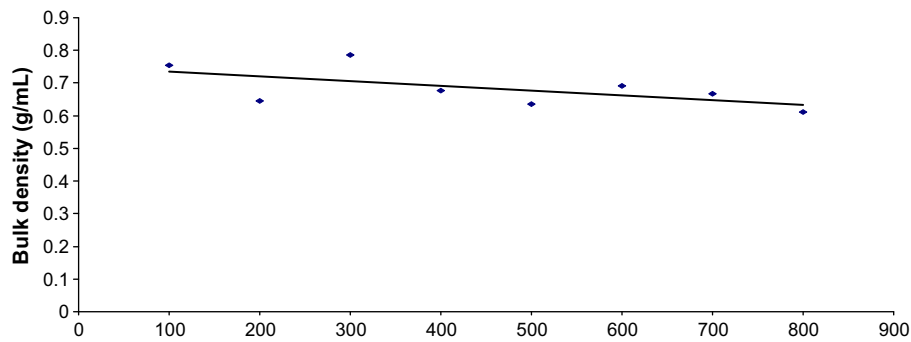


Figure 3.4 Variation of bulk density of RSSC with carbonization temperature.

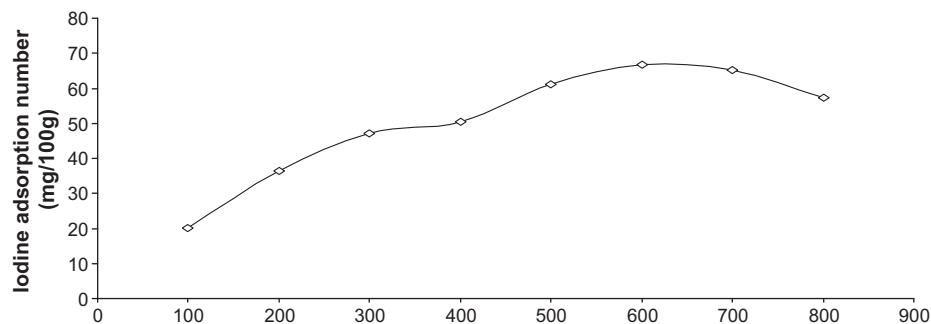


Figure 3.5 Variation of iodine adsorption of RSSC with carbonization temperature.

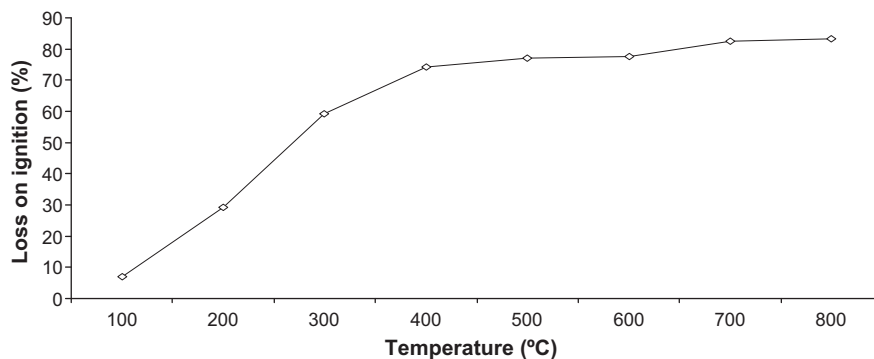


Figure 3.6 Relationship between loss on ignition and the filler temperature.

At higher temperature ( $> 600$  °C), the porosity reduces too, the reason might be that some cavities have been burned and the corresponding surface area reduced. So when carbonizing temperature reaches  $800$  °C, the surface area value is small too.

Fig. 3.6 presents the relationship between loss on ignition of RSSC and the filler carbonizing temperature. It can be seen that the loss on ignition percentage increases from  $7.1\%$  to  $83.2\%$  with increase in the heating temperature, the loss on ignition percentage increased rapidly with the rising of temperature up to  $800$  °C, this might be caused by the almost completed vol-

atilization of the volatile matter at the temperature above  $600$  °C.

#### 4. Conclusion

The work is an attempt at exploring the effect of carbonization on the processing characteristics of rubber seed shell powder. The result shows that heating temperature affects the properties of the rubber seed shell. These results predict the vulcanization rate and the potential application of the filler.

**References**

- Ahmedna, M., Johnson, M., Clarke, S.J., Marshal, W.E., Rao, R.M., 1997. Potential of agricultural by-product based activated carbon for use in raw sugar decolorization. *J. Sci. Food Agric.* 75, 117–124.
- ASTMD 1509, Standard method of testing Moisture content, 1983.
- ASTMD 1512, Standard method of testing for pH, 1983.
- Ball, D.E., 1964. Loss on Ignition of organic matter as an estimate of calcareous soils. *J. Soil Sci.* 15, 4–92.
- Bledzki, A.K., Gassan, J., 1999. Composites reinforced with cellulose based fibres. *Prog. Polym. Sci.* 24, 221–274.
- Eichhorn, S.J., Baillie, C.A., Zafeiropoulos, N., Mwaikambo, L.Y., Ansell, M.P., Dufresne, A., 2001. Current international research into cellulosic fibres and composites. *J. Mater. Sci.* 36 (9), 31–44.
- Ekebafé, L.O., Imanah, J.E., Okieimen, F.E., 2010. Physicochemical properties of rubber seed shell carbon-filled natural rubber compounds. *CE&CIQ* 16 (2), 149–156.
- Imanah, J.E., Okieimen, F.E., 2003. Rheological and mechanical properties of natural rubber reinforced with rubber seed shell. *J. Appl. Polym. Sci.* 90, 3718–3722.
- Ishak, Z.A.M., Baker, A.A., 1995. An investigation on the potential of rice husk ash as fillers for epoxidized natural rubber. *Eur. Polym.* 31 (3), 259–269.
- Jideonwo, A., Utuk, J.P., 2001. Physico-mechanical properties of rubber seed shell filled Natural rubber compounds *Niger. J. App. Sci.* 18, 115–120.
- Moloney, T.M., 1995. In: Lee, S.M., Rowell, R.M. (Eds.), *International Encyclopedia of Composites*. VCH Publishers, New York, p. 656.
- Mwaikambo, L.Y., Ansell, M.P., 2002. Chemical modification of hemp, sisal, jute, and kapok fibers by alkalisiation. *J. Appl. Polym. Sci.* 84 (12), 2222–2234.
- Okieimen, F.E., Imanah, J.E., 2003. Characterization of agricultural waste products as fillers in natural rubber formulation *Niger. J. Polym. Sci. Technol.* 3 (1), 210–216.
- Okieimen, F.E., Imanah, J.E., 2005. Rheological and mechanical properties of natural rubber reinforced with agricultural by-product. *J. Polym. Mater.* 22 (4), 411–415.
- Ricordel, S., Taha, S., Cisse, I., Dorange, G., 2001. Separation purification technol. 24, 389–401.
- Sharma, K.K., Sharma, L.K., 2004. *A Textbook of Physical Chemistry*. Vikas Publishing House, India, 421–449.
- Sun, K., Jiang, J.C., 2010. Preparation and Characterization of activated carbon from rubber-seed shell by physical activation with steam. *Biomass Bioenergy* 34, 539–544.
- Toles, C.A., Marshall, W.E., Johns, M.M., 1997. The effect of activation method on the properties of pecan shell activated carbon. *Carbon* 35 (9), 1407–1414.