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Properties of Carbon Black from *Jatropha* Seed Shell as A Potential Source of Filler Enhancement in Biocomposites

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Abstract - The use of biomass as filler in biocomposites are becoming widespread in the present decade because its variety of advantages. *Jatropha* (*jatropha curcas* L) seed shell after separated from kernel can be used to produce carbon black because of its higher carbon content, and have the potentiality to use as reinforcement in composites. In this study, carbon black was produced from *jatropha* seed shell by carbonization in furnace method for 1 hour at 600°C. Thermo gravimetric analysis to analyze seed shell of *jatropha* as raw material. The morphological properties of the carbon black were analyzed by elemental analysis by X-ray (EDX), SEM, TEM, XRD, SEM, and TEM. It was found that the yield of carbon black was 40% which included 4 different sizes ranging from 70 – 300 mesh in distribution of particle size analysis. Carbon black suggested that it could be a good filler or reinforcement in biocomposites.

Keywords—*Jatropha* seed shell, carbonized particles, morphology, properties and particle size distribution

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I. INTRODUCTION

Biomass are materials renewable and abundant in the world. Reinforcement of biomass in composite will increase mechanical properties for many application. Carbon black is very important material widely used as filler in composites. Structure and configurations of particles influence properties of carbon black (Stabik *et al.*, 2012). The principal uses of carbon black are as a reinforcing agent in rubber compounds (especially tires) and as a black pigment in printing inks, surface coatings, paper, and plastics, Antal *et al.*, 2003. Carbon black is a product of incomplete combustion. It is the dark component of smoke and in fact all carbon black processes start with the production of a smoke.

To achieve the green composites, many researcher concern to agriculture biomass resource. Agricultural by products and waste materials used for the production of carbonized particle have studied by several researcher include olive mill waste and sewage sludge (Gokce *et al.*, 2009), coconut shells (Young *et al.*, 2010, Li *et al.*, 2009). *jatropha* hull (Xin-hui *et al.*, 2011), *jatropha* fruit shell (Tongpoothorn *et al.*, 2011, Ramakrishnan *et al.*, 2009, Karthikeyan *et al.*, 2008,

Sricharoenchaikul *et al.*, 2007), *jatropha curcas* pods (Sathishkumar *et al.*, 2012), *Delonix Regia Seed Shell*, *Ferronia Seed Shell*, *Ipomea Carnia Stem* (Karthikeyan *et al.*, 2008), olive bagasse (Demiral *et al.*, 2011), rice husk (Wang *et al.*, 2011) this carbon black have activated and successfully used for the adsorption processes and another application.

Several researcher have studied of carbon black from biomass as a filler in biocomposites. Bioresource that used in biocomposite are bamboo (Khalil *et al.*, 2007), coconut shell, oil palm empty fruit bunches (Khalil *et al.*, 2010, 2012), carbon residue derived from woody biomass (Pries *et al.*, 2010), carbonized bagasse filler in rubber product (Osarenmwinda and Abode, 2010) have been investigated.

The increasing areas of plantations of *jatropha curcas* L. in India, China, and Indonesia as well as in South America and Africa will lead to an increased amount of *jatropha curcas* in future. The shells of the seeds (hulk) are promising to be an alternative fuel for these countries, because they appear as a by-product during the processing of *jatropha* oil and can not used for any other application until now. The *jatropha* seed contains about 42% seed husks (seed shell). Analysis of the

husks by (Singh *et al.*, 2008 and Vyas and Singh, 2007) showed that the husks contained 4% ash, 71% volatile matter and 25% fixed carbon. The chemical analysis of *jatropha* shell are 34%, 10% and 12% cellulose, hemicellulose and lignin, respectively (Singh *et al.*, 2008). Volatile matter, ash and fixed carbon content of the shell have been shown to be 69%, 15% and 16% (Singh *et al.*, 2008 and Jingural *et al.*, 2010). On the other hand, the by product such as seed shell contain fixed carbon can be an alternative to make carbon black as bio filler in biocomposites (Khalil *et al.*, 2013).

Two main reasons to employ particle size characterization, e.i., better control of product quality and better understanding of products, ingredients and processes. Filler from biomass is a diluent and is used primarily to lower volume cost. With use, all fillers modify certain physical properties of the compound in addition to lowering cost. Therefore, lower cost is generally achieved at the expense of other desirable properties and all compounds are compromises with various trade-offs considered and balanced by the compounder (Sri Aprilia *et al.*, 2014).

The objective of this study was investigated to get an excellent carbon black from *jatropha* seed shell as raw material filler for reinforcement in biocomposite and characterization physico-chemical properties of carbon black to determine elemental analysis by X-ray (EDX), SEM, TEM, XRD.

II. MATERIAL AND METHOD

Procedure

Jatropha curcas L seed shell originated from Aceh Province, Indonesia. The kernel were manually removed from the seed. The seed shell were washed with distillate water to remove the foreign materials and oven dried for 24 hours at 105 °C. The dry materials were sieved to the particle size of 2 to 5 mm and stored in airtight container for further experiment. The average proximate analysis of *jatropha* seed shell represented in weight percent, shows a moisture content of 5.68%, a volatile matter of 64.63%, fixed carbon of 25.34% and ash content of 3.82%.

Thermogravimetric analysis

Thermal degradation of *jatropha* seed shell was determined using a pyris Diamond TG/DTG thermal analyzer (Perkin-Elmer). The sample amount 3 mg (± 0.5) of *jatropha* seed shell was heated from 30 to 800°C at a rate of 20 °C/min under nitrogen gas atmosphere.

Experimental methods

Carbonization process of *jatropha* seed shell carry out from dry materials used in electric furnace Gotech Testing Machines Inc. method for 1 hours at 600°C. After carbonization the sample was cooled to room temperature. The carbonized product were ground in different grinder. A big grinder (Mukmin Enviro) and than continue with small grinder (Retsch). Sieving process to get particle size of carbon black as a filler. Carbon black were sieved in 4 size, 70-100 mesh, 100-150 mesh, 150-300 mesh, and > 300 mesh.

Products characterization

The energy dispersive analysis X-ray (EDX) was used in

analysis elemental of carbon black. This machine also conducted with this FE SEM. Microscopic images of carbon black were obtained by scanning electron microscope (SEM model EVO MA10, Carl-ZEISS SMT, Germany). Transmission electron microscopy (TEM) images of carbon black of *jatropha* seed shell were taken by placing a drop onto a carbon film supported by a copper grid. A Philips CM12 instrument. The carbon black obtaining after UV cured were cut by means of a microtome (LKB model 8800) and placed on the observation grid to get them TEM images. The diffractograms for carbon black specimens were measured by a Philips pw 1050 x-pert diffractometer recorded using Cu $k\alpha$ radiation ($k\alpha = 1.54 \text{ \AA}$). X-ray unit operated at 40 kV and 25 mA. An X-ray diffractometer (XRD) was used to investigate the surface inorganic components of the prepared carbon black. The X-ray pattern was recorded in an angel of 20° to 80° of 2 θ . Distribution of particle size analysis on carbon black was used by the long Benc Mastersizer S (Malven Instruments) fitted with the Qspec Dry Powder Feeder. Sample weight in 2 gram were prepared for this analysis. The number distributions at 10%, 50% and 90% cut-off point as well as the number mean diameter.

III. RESULT AND DISCUSSION

Thermal Degradation of Jatropha Seed Shell

Thermogravimetry analysis is study a formation on thermal behavior of the starting *jatropha* seed shell to form carbon black. Figure 1 show thermogravimetry (TG) and differential thermogravimetric (DTG) curves of mass loss of *jatropha* seed shell in the range 30–800 °C.

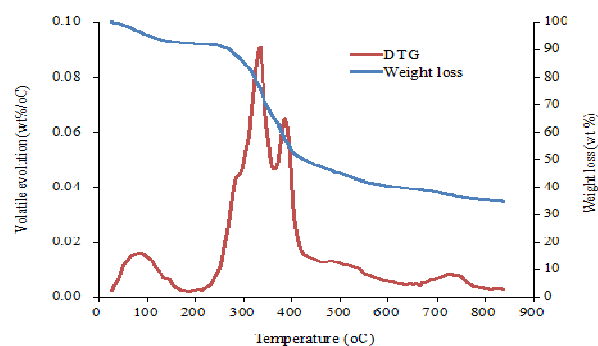


Figure 1. Thermogravimetric analysis of *jatropha* seed shell showing weight loss and DTG plot.

From this analysis show the initial weight loss corresponds to moisture removal at 100°, degradation of biomass at 200-375 °C, and degradation of lignin at >375 °C. Tongpoothorn *et al.*, 2011 and Sricharoenchaikul *et al.*, 2008 has mentioned that the first stage of mass loss was verified by heating the biomass, resulting in moisture elimination. The second stage is primary of carbonization, in this zone had a greater mass loss of biomass. This stage presented the evolution of light volatile compounds occurs from degradation of cellulose and hemicelluloses. The third stage indicating the decomposition of lignin, which was a structure with higher stability.

Elemental composition of carbon black

Table 1 shows the major compound in carbon black from *jatropha* seed shell at 600°C in furnace methods at 1 hour by Energy Dispersive Analysis of X-Ray (EDX).

Table 1. Elemental composition carbon black *jatropha* seed shell

Element	Weight%	Atomic%
C	74.25	81.97
O	20.85	17.28
K	1.57	0.53
Au	3.33	0.22

Carbon is a main compound in carbon black of *jatropha* seed shell carbonization, this is about 74.25 weight % and 81.97 atomic %. Carbon is main product degradation of hemicellulose, cellulose and lignin from biomass. Oxygen is the important compound, and in determines the properties of carbon black. Oxygen present during the carbonization process to produce carbon black.

Morphology of carbon black

Surface structure of *jatropha* seed shell and carbon black *jatropha* seed shell has investigated by SEM in Figure 3. The surface is not smooth and rough on the surface shell. After carbonization of lignocellulose and attractive compound in biomass at 600°C at one hour in furnace method, the black color, sponge shape was occurred.

Figure 4 shows the surface structure of carbonized *jatropha* seed shell after sizing in four sizes. This figure revealed change in surface morphology of char as a function of size after carbonization. The surface area shows relatively smooth as well as cracked and pitted morphology. The four sizes of carbonized particles are not different in structure.

TEM has long been employed in the study of carbon black micro-structure. TEM images of carbon black from *jatropha*

seed shell is given in Figure 5. Regardless of varying average particle size from carbon black, some common features can be extracted from the observations. Zhu et al, 2004 has stated in their writing that particles in the carbon blacks are commonly fused together to form aggregates so that clear boundaries among particles are difficult to define (Figure 4a and 4b). Aggregate is commonly the primary structural unit for carbon black. One notable tendency is that the same aggregate appears to comprise particles of comparable size. The particle shapes are always nearly spherical. Pores are present within and among the aggregates.

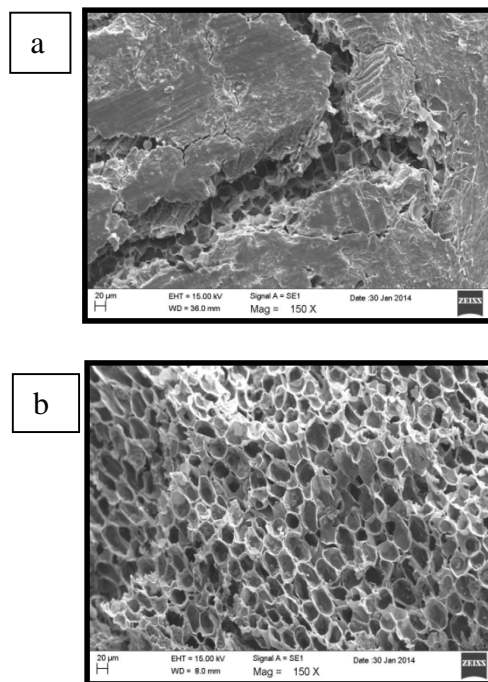
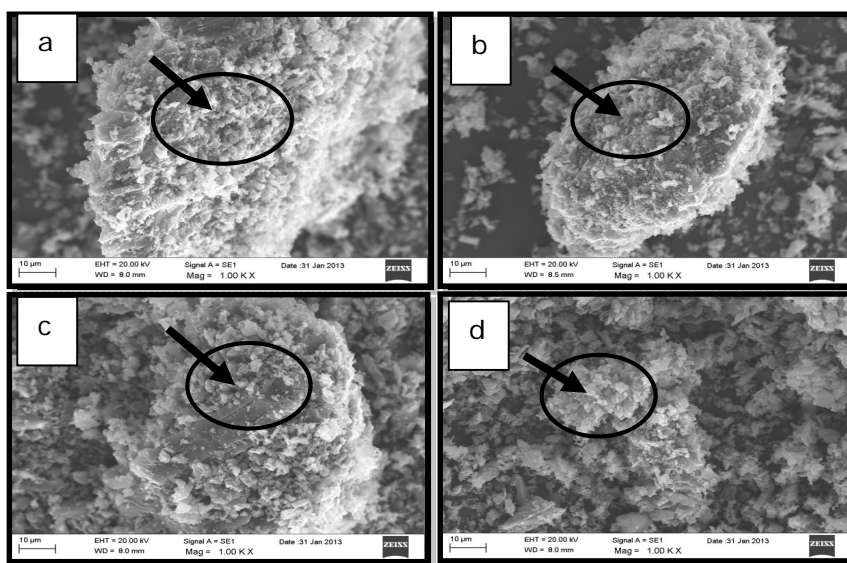


Figure 3. Surface structure of: a. *Jatropha* seed shell and b. carbonized *jatropha* seed shell



pha seed shell in size: a. 60 mesh, b. 100 mesh, c. 200 mesh, and d. > 300 mesh.

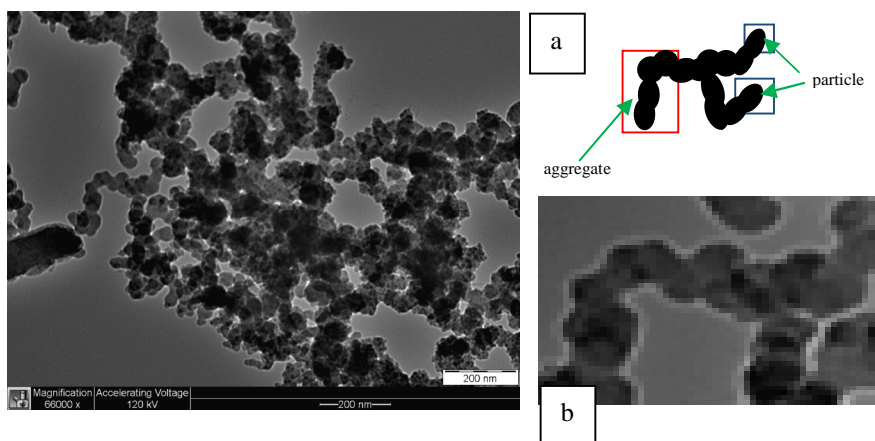


Figure 5. TEM of carbon black from *jatropha* seed shell; a. Image of a particle and aggregate, b. TEM part of carbon black

XRD of carbon black *jatropha* seed shell

Figure 7 show X-ray diffraction patterns that were recorded in order to determine the degree of crystallinity of the carbon black. The appearance of broad peak centered at the 2θ angle of 23° in the X-ray diffractograms of carbonization. Sricharoenchaikul et al, 2008 has indicated the presence of silica in this peak. At 2θ angle of 43° , the graphitic basal panel could be seen in the spectrum of carbonization of carbon black. This is also imply by Chiravoot Pechyen et al, 2007 and Wimonrat et al, 2011, which confirm the structure of graphitic basal plans of char crystallite in the low angle region and radial spread of crystalline structures in high angle region in the similar broad peak. Hence, the product carbonization from *jatropha* seed shell obtain are believed to be in the form primary particles of small graphitic sheets.

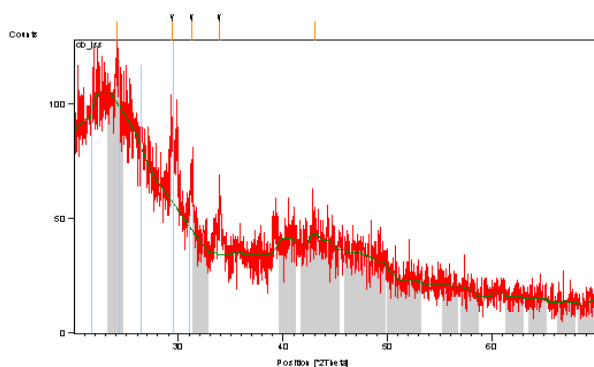


Figure 7. X-ray diffraction analysis of carbon black from *jatropha* seed shell

IV. CONCLUSIONS

It can be concluded that by-product of palm oil plantation and mill had good nutritional value. Therefore, this feedstuff can be used to formulate complete feed for sheep. It also proved that this effort successfully increased the nutrient digestibility.

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