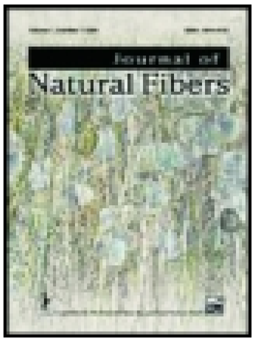

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ARTICLE

Effect of Alkaline Surface Modification and Carbonization on Biochemical Properties of Rice and Coffee Husks for Use in Briquettes and Fiber-Reinforced Plastics

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ABSTRACT

Effects of alkaline surface modification and carbonization on biochemical properties of selected rice and coffee husk varieties in Uganda were studied. Alkali pre-treatment was done by washing the husks in NaOH at a 15:1 liquor ratio. Pre-treatment is used in biomass conversion to overcome chemical and structural difficulties. Carbonization resulted in production of bio-char. Results showed increased cellulose contents for both the husks and their bio-chars after pre-treatment with NaOH solution. Lignin and hemi-cellulose compositions were reduced after pre-treatment in NaOH solution. Bio-chars for all rice husk varieties showed an increase in lignin content after carbonization. Lignin contents for *Robusta* coffee husks also increased after carbonization. Cellulose content in bio-chars of both rice and coffee husks reduced drastically after carbonization. A simple regression model relating bulk density and biochemical composition was developed at an accuracy of 70%. Alkaline surface modification and carbonization effects on biochemical properties are due to their impact on the non-cellulosic material and hydrophobicity, respectively, on the fibers.

KEYWORDS

Bio-chemical; Briquettes; Bulk density; Coffee husks; Fiber-reinforced plastics; Hemicellulose.

关键词

关键词; 生物化学; 型煤; 体积密度; 咖啡皮; 纤维增强塑料; 半纤维素

摘要

研究了碱性表面改性和炭化对乌干达部分稻壳和咖啡壳生物化学性质的影响.碱预处理是以15:1的液比在NaOH中对稻壳进行清洗.预处理用于生物质转化，克服了化学和结构上的困难.炭化导致了生物炭的产生.结果表明，经氢氧化钠溶液预处理后，稻壳及其生物炭的纤维素含量均有所增加.在氢氧化钠溶液中预处理后木质素和半纤维素组分降低.所有稻壳品种的生物炭在炭化后木质素含量均呈上升趋势.经过炭化处理后，罗布斯塔咖啡皮的木质素含量也有所增加.炭化后，稻壳和咖啡壳的生物特性中纤维素含量均显著降低.建立了一个体积密度与生物化学成分的简单回归模型，计算精度为70%.碱表面改性和炭化对生物化学性质的影响是由于它们分别对非纤维素材料和纤维的疏水性的影响.

Introduction

In many developing countries, commercial agricultural production is essential for economic development as populations grow (Nzila et al. 2015). Increasing agricultural production has resulted in huge amounts of wastes being generated during processing (Lin et al. 2013; Yank, Ngadi, and Kok 2016). Over 20% of paddy rice is husk (Giddel and Jivan 2007) and over 50% of coffee is husk (Saenger et al. 2001). With exponentially increasing population, the demand for rice and coffee is

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predicted to increase even higher. With an ever-increasing trend in the production of these crops in Uganda, their husks are only expected to exponentially increase (Ministry of Agriculture Animal Industry and Fisheries 2016). Disposal of agricultural wastes is a major problem in many developing countries with open-burning being the main method of choice, which creates negative environmental impacts (Haykiri-Acma, Yaman, and Kucukbayrak 2013). Agricultural residues, particularly rice and coffee husks have vast applications in briquetting for domestic cooking applications, fiber-reinforced plastics production, soil remediation and wastewater treatment (Ahmad et al. 2014; Lubwama and Yiga 2017, 2018; Raju, Kumarappa, and Gaitonde 2012; Tsou et al. 2015; Yao et al. 2012; Yiga et al. 2019; Yu et al. 2010; Yu, Ying, and Kookana 2009).

The use of materials containing natural fibers depends strongly on the biochemical properties of the constituent fibers (Mohanty, Misra, and Drzal 2001; Liu et al., 2013; Saidur et al. 2011). Lignocellulosic fibers mainly consist of cellulose, hemicellulose and lignin, which are strongly linked by covalent bonds and networks (Saidur et al. 2011; Zhang, Xu, and Champagne 2010; Zheng et al. 2014). Cellulose ($C_6H_{11}O_5$) is a hydrophilic glucan polymer consisting of a linear chain of 1, 4-β-bonded anhydro-glucose units that contain alcoholic hydroxyl groups (Khalil, Bhat, and Yusra 2012). The nature of bonding in cellulose necessitates the micro-fibril structure to develop strong inter-molecular and intra-molecular hydrogen bonding (Keshwani 2010). Hemicelluloses are a branched polysaccharide comprised of different sugar monomers (Pérez et al. 2002). Hemicelluloses can form hydrogen bonds with the cellulose and lignin and hence they are referred as “cross-linking glucans”. Lignins are complex hydrocarbon polymers with both aliphatic and aromatic constituents. It is the compound that gives rigidity to the plants (Bledzki and Gassan 1999; Maya and Sabu 2008).

In order to utilize agricultural wastes, in particular, rice and coffee husks, in briquette and bio-composite plastics development, several modifications may have to be done on the residues. One major modification that can be applied to these husks is carbonization, which produces liquid, gaseous and solid fuels (Mohan, Pittman, and Steele 2006). Bio-char is a stable carbon-rich- by-product synthesized through pyrolysis/carbonization of plant and animal-based biomass (Ahmad et al. 2014; Haykiri-Acma, Yaman, and Kucukbayrak 2013). Alkaline surface modifications on fibers are quite commonly done in order to enhance adhesion between the fiber and polymer and between the fibers themselves (Herlina Sari et al. 2018; Sheshmani, Ashori, and Farhani 2012).

Two common agricultural residues in Uganda are rice and coffee husks (Okello et al. 2013). Very few studies exist on the effect of alkaline surface modifications and carbonization on biochemical properties (Lubwama and Yiga 2017, 2018). This limits potential utilization and application of these agricultural wastes. Therefore, this study is aimed at investigating the effects of alkaline surface modification and carbonization on biochemical properties of different rice and coffee husk varieties for potential application in briquettes and fiber-reinforced plastics. A simple regression model relating bulk density and biochemical properties has also been developed.

Materials and methods

Rice husks (*Wita-9* and *Pussa*) were obtained from Kibimba Limited in Uganda. *Arabica* coffee husks were collected from Mbale district in Eastern Uganda. *Robusta* coffee husks were obtained from Mubende district in Central Uganda. Over 100 kg of each category of husks were collected.

During carbonization the husks were placed in a carbonizer of 200 l capacity that had holes of diameter 0.02 m for air-flow regulation. As the carbonization process took place, these holes were covered to limit oxygen supply in the reactor. Equation 1 shows the mechanism of carbonization (Eastop and McConkey 1993). Details of these carbonization process have been described elsewhere (Lubwama and Yiga 2017, 2018).

$$C_xH_yO_z + O_2 \xrightarrow{\text{limited air supply}} H_2O + CO + C \qquad \text{(Eq.1)}$$

Alkaline surface modification was done for rice and coffee husks and their bio-chars. It involved soaking half of these husks in 3% NaOH solution. The reaction of NaOH with natural fiber is given by Equation 2 (Sepe et al. 2018). For comparison some husks were soaked in distilled water. Soaking took place for 3 h at ambient temperature. A liquor ratio of 15:1 was used. The samples were then dried for 48 h at room temperature and 60°C in an oven overnight to reduce the moisture content to less than 5%.

$$Fiber - OH + NaOH \rightarrow Fiber - O^{-}Na^{+} + H_2O \quad \text{(Eq.2)}$$

The hemicellulose, cellulose and lignin contents were obtained according to the methods described by Van Soest, Robertson, and Lewis 1991. The cell wall constituents comprising neutral detergent fiber (NDF), acid detergent fiber (ADF) and acid detergent lignin (ADL) were determined (Olupot et al. 2016). Bulk density of the husks and their bio-chars were determined from the ratio of mass of unified material to volume of a standard cylinder (Lubwama and Yiga 2017).

Results and discussions

Lignin content for *Pussa* and *Wita 9* rice husks was 17.6% and 21.5%, respectively. *Arabica* and *Robusta* coffee husks had contents of 19.6% and 22.5%, respectively. Hemicellulose content was 15.6% for *Wita 9* and 20.5% for *Pussa*, respectively, and 24.5% and 8.8% for *Arabica* and *Robusta* coffee husks, respectively. Cellulose contents in the unmodified rice and coffee husks were 28.4% and 34.3% for *Pussa* and *Wita 9*, respectively, and 42.1% and 25.4% for *Arabica* and *Robusta* coffee, respectively (See Figure 1). Generally, modification of the husk raw material with NaOH solution resulted in an increase in cellulose for both the husks and their bio-chars (See Figures 1 and 2). The highest cellulose content of 37.6% was observed when *Pussa* rice husks were washed in 3% NaOH solution. Briquette development and adhesion between polymer matrix and natural fiber are enhanced by high cellulose contents (Valadez-Gonzalez et al. 1999). Because cellulose content has a positive effect on briquette development, rice and coffee husks can easily be used as raw material to obtain energy for burning. Cellulose improves the mechanical properties, hemicellulose has negative effects on the composite properties while lignin can reduce the water absorption and increase thermal stability (Liu, Quek, and Balasubramanian 2014a). Considering that cellulose improves mechanical properties (Young's modulus, Elongations at break, water absorption retardation, tensile and impact strengths), developed rice and coffee husks fiber-reinforced plastics can find suitable

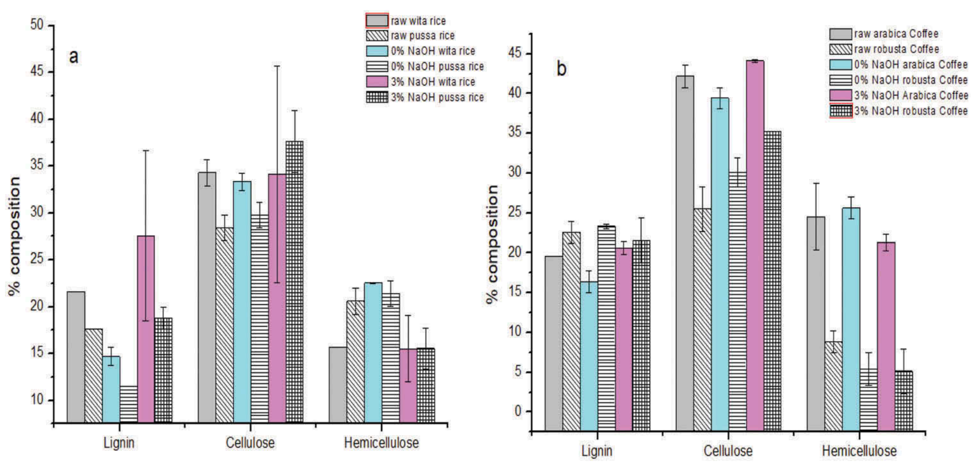

Figure 1. Biochemical analysis of un-carbonized husks a) rice b) coffee.

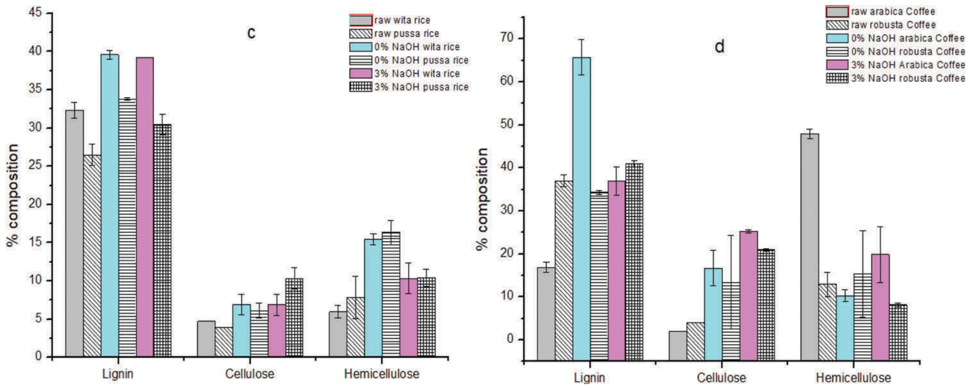

Figure 2. Biochemical analysis of bio-char c) rice d) coffee.

application in packaging composites which require such properties. Additionally, it is possible to isolate these micro and nano-cellulose components which are more environmentally friendly (Ravindran, Sreekala, and Sabu 2019).

However, Lignin and hemi-cellulose compositions were reduced after pre-treatment in NaOH solution (See Figures 1 and 2). Lignin content of 16.3% was observed for *Arabica* coffee husks after washing in distilled water. Lignin content increased to 20.5% when 3% NaOH solution was used. Lignin in *Robusta* coffee husks was 23.3% after washing in distilled water and 21.5% after washing in 3% NaOH solution. Hemicellulose content for *Wita 9* and *Pussa* rice husks after washing in distilled water was 22.5% and 21.4%, respectively. Washing in 3% NaOH resulted in a decrease to 15.5% and 15.4% for *Wita 9* and *Pussa* rice husks, respectively. Hemicellulose content recorded after washing in distilled water was 25.6% for *Arabica* coffee husks. After washing in 3% NaOH solution, hemicellulose content of 21.3% was measured. High lignin contents signify increased resistance by the rice and coffee husks to hydrolyze (Zhao 2013). Lignins increase resistance of the biomass to chemical and biological degradation (Zheng et al. 2014). Pre-treating rice and coffee husks with NaOH removes not only lignins, but also waxes and resins covering the external surface of the fiber walls and exposes the hydroxyl groups which exposes the cellulose and hemicellulose for enzymatic action (Essabir et al. 2016). This is of particular importance for briquettes and fiber-reinforced plastics which are prone to moisture absorption by the natural fibers which affect their mechanical and thermal properties (Bledzki and Gassan 1999; Lubwama and Yiga 2018). Lignin is thermally stable because of crosslinking between monomer units (Chen, Lu, and Tsai 2012; Pasangulapati et al. 2012). Thermal stability is an important parameter in the development of flame resistant fiber-reinforced plastics (Zhao et al. 2009). With the lignin contents in rice and coffee husks being high above 10%, packaging of hot substances/materials can be done with rice and coffee husks reinforced plastics.

Treatment of the carbonized husks in distilled water resulted in lignin contents of 39.6% and 33.8% for *Wita 9* and *Pussa* rice husks. Washing the carbonized rice husks in 3% NaOH solution resulted in lignin contents of 39.2% and 30.4% for *Wita 9* and *Pussa* rice husks respectively. A similar trend was observed in results for hemicellulose content (See Figure 2). However, cellulose content increased significantly after washing in 3% NaOH solution. Cellulose content of carbonized coffee husks was 1.9% and 4% for *Arabica* and *Robusta* coffee husks, respectively. The content of cellulose increased significantly to 16.6% and 13.4%, respectively, after washing in distilled water. Washing in 3% NaOH solution resulted in even higher values of cellulose content at 25.2% and 20.9% for *Arabica* and *Robusta* coffee husks, respectively (See Figure 2). Higher lignin content is advantageous for briquette development due to production of very little ash and longer burn times (Lubwama and Yiga 2017, 2018). The carbonization process improves the hydrophobic properties of lignocellulosic materials (Felfli et al. 2005). This implies that carbonized natural fibers can be used as

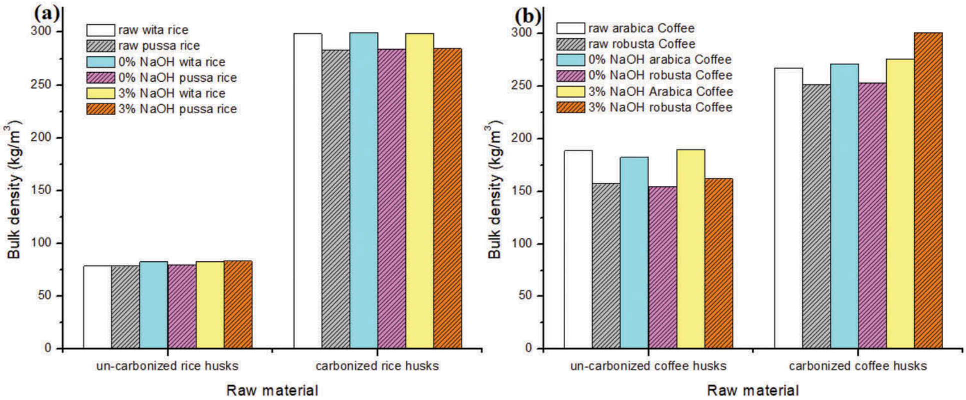

Figure 3. Bulk density of the raw materials a) rice husks b) coffee husks.

filler material in development of fiber-reinforced plastics and briquettes (Zhao et al. 2009). Cellulose content reduction after carbonization is an indicator that moisture absorption by bio-chars is reduced, making potential application in briquettes and fiber – reinforced plastics worth considering (Das et al. 2016; Muazu and Stegemann 2015).

Bulk density of raw material husks was 78.6 kg/m^3 for the rice husks and 188.6 kg/m^3 and 157.2 kg/m^3 for *Arabica* and *Robusta* coffee husks, respectively (See Figure 3). Treatment of the husks in distilled water resulted in a decrease the bulk densities for *Arabica* and *Robusta* coffee husks by 3.2% and 1.7%, respectively, while slight increases were obtained in both *Wita 9* and *Pussa* rice husks to 82.5 kg/m^3 and 79.6 kg/m^3 respectively. Further pre-treatment in 3% NaOH solution increased the bulk densities to 190.0 kg/m^3, 162.0 kg/m^3, 83.1 kg/m^3 and 83.2 kg/m^3 for *Arabica* coffee, *Robusta* coffee, *Wita 9* rice and *Pussa* rice husks, respectively. This increase is attributed to an increased cellulose content due to the alkali pre-treatment (See Figures 1 and 2). A small reduction in particle diameter associated to the removal of some non-cellulosic components also plays a role (Laaziz et al. 2017).

Carbonized *Wita* 9 and *Pussa* rice husks had bulk densities of 298.7 kg/m^3 and 283.0 kg/m^3 respectively. The bulk densities for carbonized raw coffee husks were 267.3 kg/m^3 and 251.5 kg/m^3 for *Arabica* and *Robusta* coffee husks, respectively. After washing on distilled water, bulk densities increased to 299.5 kg/m^3 and 283.9 kg/m^3 for Wita 9 and *Pussa* rice husks, respectively, and to 271.2 kg/m^3 and 253.19 kg/m^3 for *Arabica* and *Robusta* coffee husks bio-char, respectively. The highest bulk density was obtained for *Robusta* coffee bio-char at 300.8 kg/m^3 after washing in 3% NaOH. Increased bulk densities are explained by an increased weight due to size reduction in the particles (Chevanan et al. 2010; Deng et al. 2009). Bio-chars of rice husks had higher bulk densities than those of coffee husks because of the presence of silica oxides in the rice husks (Liu, Quek, and Balasubramanian 2014a). Bulk density plays a major role in utilization of material as fuel energy. The higher the bulk density, the easier it becomes to package, transport and store fuel energy (Lubwama and Yiga 2017). Therefore, the agricultural residues used in this study have a huge potential for use in providing energy for burning/cooking in domestic applications. Bulk density also plays a major role in determining packaging requirements (Abegunde et al. 2019). Additionally, bulk density affects the compatibility of materials in a composite, therefore high bulk density leads to high mechanical properties (Tensile strength, Young's modulus and impact strengths). In fact, Binoj, Edwin Raj, and Daniel 2017 discussed the role of fiber density in improving tensile strengths and Young's moduli of composites.

Strength is a high impact factor when working with fiber-reinforced plastics development and briquettes production. Because of high bulk densities obtained for bio-chars, briquettes can be suitably produced (Menya et al. 2018). This is because increased bulk density of residual biomass makes the materials obtained out of them compact and less humid, thus increasing the energy capacity of these materials to a level higher than that of less dense materials. (Paula et al. 2011; SelSellin et al. 2013). In fact, for briquettes, a positive correlation has been noted between bulk density and the energy/volume ratio (Brand et al. 2019). Previous studies on density of briquettes made from biomass found out that it varied between 200 kg/m^3 and 1200kg/m^3, depending on the compaction pressures and feedstock nature (Lubwama and Yiga 2017).

Correlation between bulk density and biochemical composition

In order to establish mathematical relationships/equations between the bulk density values of the rice and coffee husks samples and their biochemical compositions, a simple regression analysis was done to obtain coefficients and a constant for the lignin, cellulose and hemicellulose contents in rice and coffee husks. Data from Figures 1–3 were used for this purpose. The developed Bulk density model is shown in Equation 3.

$$Bulk\ density\left(\frac{kg}{m^3}\right) = 180.431 + (3.072L) + (0.823HC) - (3.385C) \quad \text{(Eq.3)}$$

where L, HC and C are the lignin, hemicellulose and cellulose compositions, respectively.

The numerical and analytical bulk densities were fitted according to ordinary least squares (OLS) method (See Figure 4) to develop a relationship between the numerical and analytical bulk densities (See Equation 4).

$$y = (0.684 * x) + (64.457) \quad \text{(Eq.4)}$$

where y and x are the numerical and analytical bulk densities.

The mean absolute error, average absolute error and average bias error for the correlation were computed using Equations 5, 6 and 7 (Nhuchhen and Salam 2012). The developed model had a 0.296 mean absolute error, meaning bulk density can be determined from biochemical analysis with an

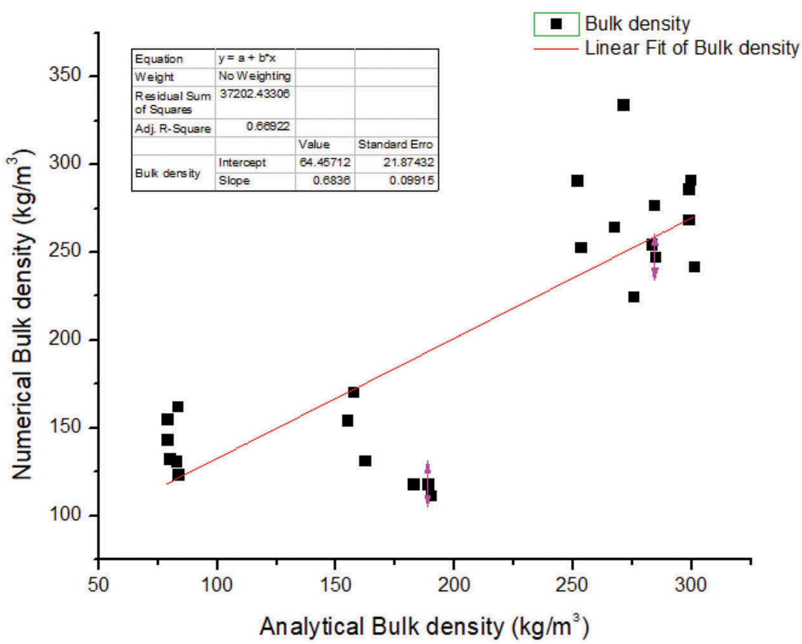

Figure 4. Plot of numerical bulk density versus analytical bulk density.

accuracy of over 70% (R^2 = 0.7). The developed model can be used for bulk density calculation of any lignocellulosic material, from its biochemical analysis. The main advantage of this model is that bulk density determination of lignocellulosic material can be determined very rapidly and easily based on only the biochemical compositions.

$$\text{Mean Absolute Error} = \left(\frac{1}{n}\right)\sum_{i=0}^{n}\left(\left|Numerical\ Bulk\ density - Analytical\ Bulk\ density\right|\right) \quad \text{(Eq.5)}$$

$$\text{Average Absolute Error} = \left(\frac{1}{n}\right)\sum_{i=0}^{n}\left(\frac{\left|Numerical\ Bulk\ density - Analytical\ Bulk\ density\right|}{\text{Analytical Bulk density}} \times 100\%\right) \quad \text{(Eq.6)}$$

$$\text{Average Bias Error} = \left(\frac{1}{n}\right)\sum_{i=0}^{n}\left(\frac{\text{Numerical Bulk density} - \text{Analytical Bulk density}}{\text{Analytical Bulk density}} \times 100\%\right) \quad \text{(Eq.7)}$$

Conclusions

In this research, the effects of alkaline surface modification and carbonization on biochemical properties and bulk density of selected rice and coffee husk varieties in Uganda were studied. Biochemical composition of unmodified and modified rice and coffee husks as well as their bio-chars were determined. The results suggest that modification with NaOH greatly improved the cellulose content of the fibers. Alkali treatment with NaOH removed hemicellulose and lignin from the fibers. Biochemical contents of cellulose, lignin and hemicellulose have been used to predict develop a simple model relating bulk density and biochemical composition. The numerical bulk densities of rice and coffee husks varied between 112kg/m^3–334kg/m^3 while the analytical bulk densities ranged between 79kg/m^3 and 301kg/m^3. The Lignin and Cellulose contents increases with modification in distilled water and 3% NaOH is an indicator that moisture absorption by the rice and coffee husks will be reduced thus enhancing their potential usability in the production of briquettes and as fiber/fillers in polymer composites. Carbonization results in higher cellulose content which enhances mechanical properties of potentially developed briquettes and fiber-reinforced plastics. A model relating bulk density and biochemical composition of rice and coffee husks was presented. Notice should be taken to determine how the developed model is affected by the different varieties of these fibers.

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