Vol. 11(2), pp. 24-30, July-December 2020
DOI: 10.5897/JCEMS2020.0348
Articles Number: E95EDE065092
ISSN 2141-6605

http://www.academicjournals.org/JCEMS

Journal of Chemical Engineering and Materials Science

Full Length Research Paper

Characterization of rice husk ash prepared by open air burning and furnace calcination

Kaleli Mbinda Jonathan[1], Kamweru Paul Kuria[1]*, Gichumbi Joel Mwangi[1] and Ndiritu Francis Gichuki[1,2]

[1]Department of Physical Sciences, Faculty of Science, Engineering and Technology, Chuka University, Tharaka Nithi County, Kenya.
[2]Department of Physics, Faculty of Science, Egerton University, Kenya.

Received 18 January, 2020; Accepted 9 April, 2020

Rice husk (RH) is an agricultural waste, and easily available in rice growing areas. The husk is mostly burnt as a way of getting rid of it. The ash obtained after burning or calcination may have economic application, mainly dependent on its properties. These properties in turn depend on the calcination method. However, for commercial viability, and for many applications, the calcination method should not only be as simple as possible but also cheap. This study characterized the elemental composition, crystallinity, functional bonds present and morphology of rice husk ash (RHA) obtained in two ways, that is, calcination of rice husks in a muffle furnace (FRHA) at a temperature of 700°C and open air burning (ORHA) at uncontrolled temperatures. The elemental composition done by Atomic Absorption Spectroscopy showed a high percentage of silicon that is 81.01 and 79.12% for ORHA and FRHA, respectively. X-ray fluorescence showed a high percentage of silica (SiO_2), 95.45 and 94.85% for ORHA and FRHA, respectively. X-ray diffractograms indicate that the FRHA was crystalline with the highest peak at 21.8°; while ORHA was amorphous in nature. Fourier Transform Infra-Red spectra confirmed the presence of –OH groups and O-Si-O bonds in the two types of ash. Scanning electron microscopy analysis showed agglomerated ORHA, which may be due to the presence of hydrogen bonding between silanol groups on the surface of rice husk ash for FRHA, and presence of –OH groups in ORHA. The study shows that ORHA is as good as FRHA in applications where crystallinity is optional.

Key words: Rice husk ash (RHA), rice husks (RH), silica, calcination, open air burning.

INTRODUCTION

The rice grain, commonly called a seed, consists of the true fruit or brown rice (caryopsis) and the hull, also known as the husk, which encloses the brown rice. The husks are separated from the husked rice through aspiration, making them a byproduct of the rice milling process. The husks are about one-fifth by weight and contain about 20-30% silica, the rest being organic lignin and cellulose (Chaudhary et al., 2004) of about 70-80% of dry hull (Mohamad, 2007). Other studies report that the husk roughly contains 35% cellulose, 35% hemicellulose, 20% lignin and 10% ash (which is 94% silica) by dry weight basis (Prachayawarakorn and Yaembunying, 2005), with the chemical composition dependent of the different geographical conditions and location, type of

*Corresponding author. E-mail: pkamweru@gmail.com.

paddy, climatic variation, soil chemistry and fertilizers used in the paddy growth (Ugheoke and Mamat, 2012). Earlier chemical characterization studies for instance has reported that the husks consists of 66.67% carbon, 22.3% SiO_2, 7.1% H_2O, 0.82% Al_2O_3, 0.78% Fe_2O_3, 1.10% K_2O, 0.78% Na_2O, 0.24% CaO and 0.21 mass% MgO (Chandrasekhar et al., 2003; Babel et al., 2003; Liou, 2004). Therefore, as a result of much contents of cellulose, rice husk is also often considered as cellulosic fiber. The cellulosic fiber tends to degrade at about 200°C and quickly becomes friable with loss of water (Shanks et al., 2004). Besides, these natural fibers have presence of large amounts of hydroxyl groups, which makes the properties of rice husk very much in category of hydrophilic (Mohamad, 2007).

The husk is considered a waste with negligible commercial value, from the agricultural sector. This is due to its high resistance to decomposition in the ground, its low nutritional value to animals and also its being difficult to digest. Rice husks also have low calorific value (3585 kcal kg^{-1}) and high ash content when burnt (Chuah et al., 2005; Ameri et al., 2019). The ash contains nearly 95% silica and is an important renewable source of silica. In addition, the ash contains potassium, sodium, magnesium, calcium, iron, phosphorus and much smaller quantities of other elements (Bakar et al., 2016), the composition of which again depends on the aforementioned factors like geographical location. Burning is a cheap method of extracting the silica from rice husks for possible commercial use. It has been used as a raw material in the production of rice husk ash by the process of calcination (Kenechi et al., 2016).

Calcination of rice husk ash can give rise to two types of ash depending on the type of combustion; white rice husk ash (WRHA) for complete combustion and carbonized rice husk ash (CRHA) for incomplete combustion. The two types have different reactivity. The reactivity of rice husk ash is based on the properties of amorphous silica and the porous structure of ash (Ramezanianpour et al., 2009). Amorphous silica is obtained between temperatures of 500-700°C and crystallization occurs at temperatures above 700°C (Kang et al., 2019). Amorphous silica has a large specific area and the chaotic formation of the structure is open with holes in the network where electrical neutrality is not satisfied. Crystalline silica structure is formed by the repetition of a basic unit. The structure of crystalline silica reduces the surface area of RHA; thus, reduces its reactivity (Leong, 2015).

Once obtained, RHA and silica from RHA has been used and studied for various applications that includes adsorbents for adsorption of dyes, pigments (Lawagon and Amon, 2019; Shukla, 2020) and heavy metal ions (Maingi et al., 2019) from aqueous solutions; catalytic support and catalyst (Ikhlaq et al., 2019); for manufacturing solar cells for photovoltaic power generation and semiconductors (Zamani et al., 2019); in the cement industries as a pozzolone component (Sonat and Unluer, 2019) and fertilizer industries (Sekifuji et al., 2019); in synthesis of advanced materials such as silicon tetrachloride, magnesium silicide, sodium silicate and zeolite (as reported by Genieva et al., 2008); as fillers in rubber (Xue et al., 2019) and plastic (Almirón et al., 2019) composites, due to their low densities, very low cost, non-abrasiveness, high filling levels, recyclability, biodegradability and renewable nature among many others. The aim of the present study is to compare the properties of RHA prepared under controlled conditions that is FRHA with RHA obtained through uncontrolled burning ORHA. The cost and method of obtaining RHA should be as low as possible, to encourage applicability in wide range of applications. The study sought to demonstrate that the properties of the ORHA are not far off different to those of FRHA.

MATERIALS AND METHODS

Rice husks to be used were obtained from Nice rice millers dumpsites in Mwea, Kirinyaga County, Kenya.

Incineration of rice husks to get RHA

Rice husks were washed thoroughly with water and dried in an oven at a temperature of 150°C. Calcination of the dried rice husks was done in a muffle furnace (SX2-2-17TP) for 3 h at a temperature of 700°C to produce FRHA (Ong et al., 2019). On the other hand, rice husks were burnt in open air on a hot plate to produce ORHA. The two forms of RHA were then characterized for their composition and properties.

Characterization of RHA

Determination of the composition of RHA

The FRHA and ORHA samples were analyzed in triplicates following the method done by Kamau et al. (1993). For K, Ca, Fe, Mn, Si and Pb analysis, 0.50 g of sample was digested with 9 ml of a mixture of nitric, sulphuric and perchloric acids (3:1:1). The solution was then filtered on ash less filter paper, residue ignited and treated with hydrofluoric acid to expel silica. The digests were topped to the 100 cm^3 and analyzed using Atomic Absorption Spectroscopy. In addition, the two types of ash were also characterized using X-ray fluorescence technique for their composition.

Determination of the structural properties of RHA using XRD

This was done using Bruker D2 Phasor diffractometer. Finely powdered RHA was mounted on a sample holder and an X-ray diffractogram obtained. The X-ray tube was operated at 30 mA, 30 KV and a scan speed of 2 per minute was used. This was done following the work of Kamau et al. (1993).

SEM analysis of RHA

The morphological characterization of RHA was carried out using a Zeiss ultra plus scanning electron microscope. The samples were first crushed into powder and then trapped on a tape mounted on a sample holder and then gold coated thrice prior to electron microscopy to give the necessary conductivity. This followed the modified procedure of Ayswarya et al. (2012).

Fourier transform Infrared spectroscopy (FTIR)

FTIR spectra of the RHA were recorded on an ATR Perkin Elmer A100. Samples in the form of powder were used. This followed the method of He et al. (2013a).

RESULTS AND DISCUSSION

Elemental composition of RHA

Both methods of RHA preparation as described produced ash of similar physical looks, as shown in Figure 1. It was also noted that the two types of ash were grey in color and similar by sight. The elemental composition of the two types of ashes done by AAS also showed a lot of similarities. As expected, both had higher content of silicon metal, which is 81% for ORHA and 79% for FRHA as shown in Table 1. Potassium, Manganese, Silicon and Carbon had a higher percentage in ORHA; whereas iron had a higher percentage in FRHA. However, much difference was not noted in the percentages since they differ by less than 2%. Lead metal was not detected in either of the two types of ash. Silicon in RHA exists in the form of silica (silicon dioxide) (Ariffin, 2004). This is confirmed by XRF analysis that shows the levels of silica as 94% for FRHA and 95% for ORHA (Table 2).

From the XRF analysis, the two samples of ash had a slightly different percentage of silica. FRHA was composed of SiO_2-94.85%, MgO-1.99% and K_2O-1.74%. Other elements whose composition was less than 1% were CaO, P_2O_5, Mn and Fe among others. ORHA was composed of SiO_2-95.45% and K_2O-2.46%; while those whose percentages are less than 1% are P_2O_5, S, Cl, CaO, Ti, Cr, Mn, Fe, Cu, Zn and Rb. From the XRF results, MgO is found to be in the FRHA and not in the ORHA. The oxides of K, P and Ca exist in considerably higher percentages than other elements present since these elements are essential plant nutrients and they occur in higher quantities due to preferential uptake by the plant. The uptake of minerals by plants is closely related to the soil conditions. It is also evident that most elements have a lower percentage composition in FRHA as compared to ORHA except Mn and Fe. The difference in composition is mainly attributed to the soil chemistry of the rice growing area and paddy varieties.

Structural characterization of RHA

From the XRD analysis, the ORHA has one broad peak between 18 to 30 coupled two theta degrees. This meant that the open air RHA was amorphous in nature. On the other hand, muffle calcined RHA had major sharp peaks at coupled two theta 21.8 and 36.02° and other minor peaks. This meant that the ash was crystalline in nature and this was in agreement with literature. This is shown in Figure 2a and b. This is in agreement with literature where, rice husks calcined at temperatures lower than 700°C produces amorphous silica, while at higher temperatures or equal to 700°C, crystalline silica is formed (Nair et al*.,* 2008). The amorphous silica can be transformed to quartz, tridymite and cristobalite by heating it at high temperature over 900°C (Deshmukh et al*.,* 2012). Under optimized conditions a 99.9% amorphous silica has been formed (Rafiee et al*.,* 2012) and calcination at 1,000°C has been shown to form highly crystalline silica with sharp peaks assigned to cristobalite and tridymite (Deshmukh et al., 2012). It has been reported also that the amorphous silica in RHA is reactive and may be used as a pozzolana (He et al., 2013b); further strengthening the preposition that RHA may be an effective additive in geopolymers (Venkatanarayanan and Rangaraju, 2015).

FTIR analysis of RHA

In the FTIR analysis of RHA shown in Figure 3a and b, greater similarities were noted in the appearance of the peaks. The broad band between 2200.16 and 3398.87 cm^{-1} in the ORHA is due to silanol –OH groups and chemically absorbed water. The band at 1051.24 and 1052.95 cm^{-1} in the FRHA and ORHA, respectively, correspond to Si-O-Si stretching modes. The bands between 443.24 and 792.35 cm^{-1} correspond to O-Si-O bending vibration. The band at 1603.15 cm^{-1} in the ORHA correspond to –OH bending vibration. This band was not detected in the FRHA. These findings are comparable to what is found in other studies, and further shows that the bonds present are dependent on the method of preparation (Singh et al., 2019). Both methods of RHA preparation shows slight difference in peak formation, specifically 3398.87, 2200.16 and 1603.15 cm^{-1} bands.

Surface textural characterization of RHA

From SEM analysis of RHA in Figure 4a and b, it was clear that the particles of RHA consists of fine particles, which appears to have agglomerated to large group of particles creating a dense formation. These particles are of irregular shapes and morphology. The agglomeration in ORHA may be due to the presence of hydrogen bonding between silanol groups on the surface of rice husk ash, while that in FRHA may be due to the hydroxyl groups.

Conclusion

AAS characterization of RHA derived from both methods showed presence of potassium, manganese, silicon,

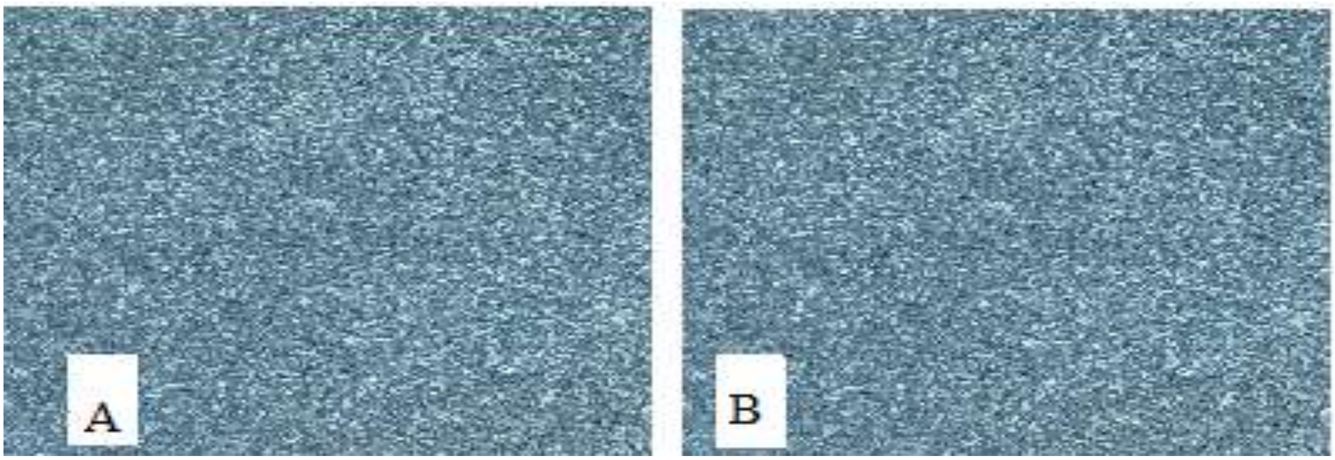

Figure 1. Images of RHA made using Kodak C1530 Digital Camera with 14 megapixels and 3X optical zoom. A. FRHA calcined at 700°C and in B. ORHA burnt on top of a hot metallic plate in open air.

Table 1. Selected elemental composition of ORHA and FRHA done by AAS.

	Si %	K %	Ca %	Fe %	Mn %	Pb %
ORHA	81.01	2.03	0.33	0.13	0.10	ND
FRHA	79.12	1.56	0.21	0.27	0.10	ND

Table 2. XRF results showing analysis of FRHA and ORHA.

Element name	% in FRHA	% in ORHA
MgO	1.985	-
SiO_2	94.852	95.453
P_2O_5	0.383	0.505
Cl	0.023	0.161
CaO	0.527	0.840
K_2O	1.743	2.459
Mn	0.129	0.126
Fe	0.277	0.137
Cu	0.002	0.003
Zn	0.004	0.048
Rb	0.010	0.011
Cr	0.003	0.012
S	-	0.200
Ti	-	0.035
Sr	0.005	-
Y	0.001	-
La	0.058	-
Sn	-	0.007
Au	-	0.003

calcium and iron metals in different percentages in both types of ash. XRF characterization showed a difference in the composition of RHA with the FRHA having 94.85% silica and the ORHA having 95.45% silica, among other

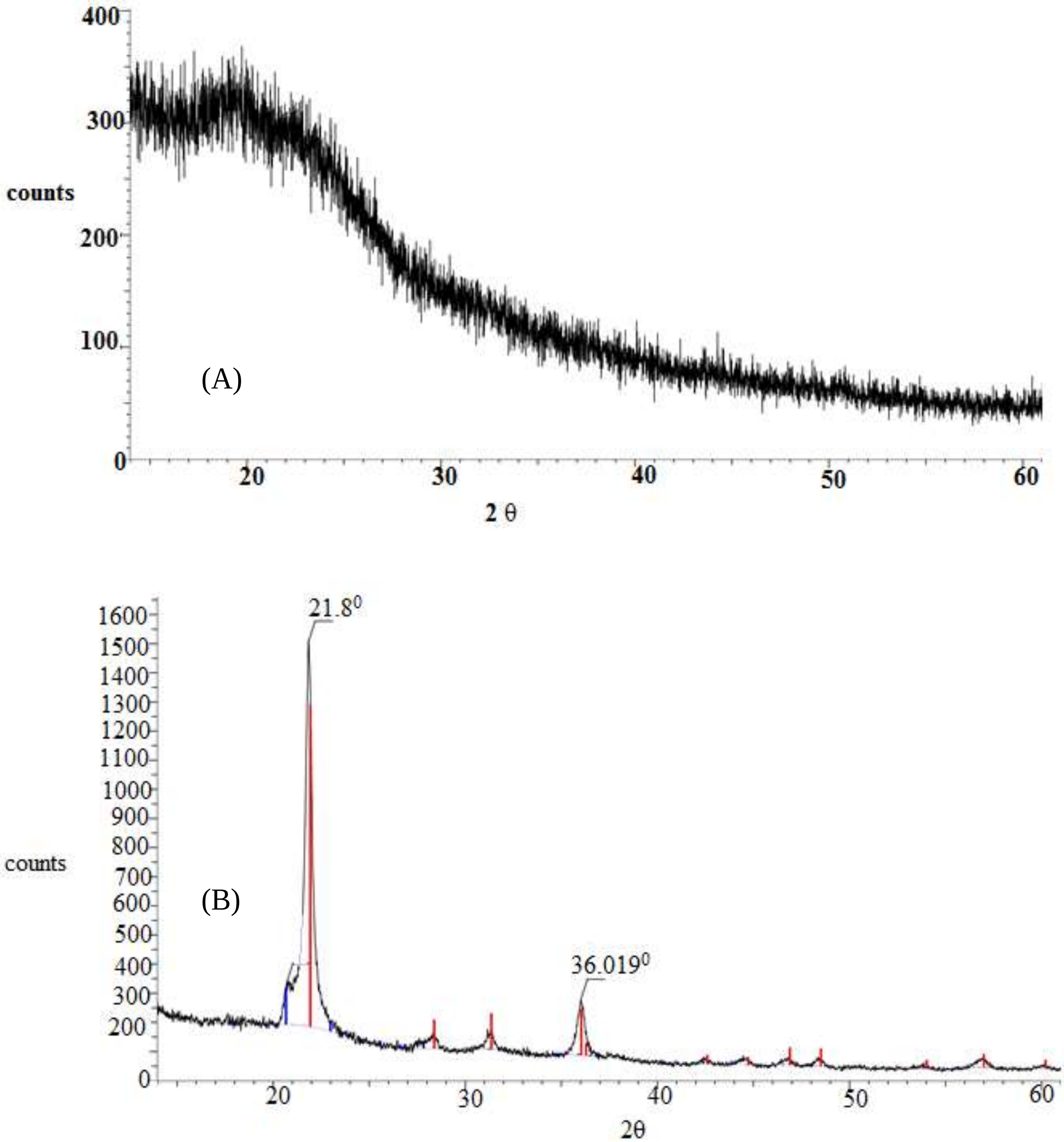

Figure 2. (a) X-ray diffractogram of ORHA. The diffractogram shows that the ash was amorphous, (b) X-ray diffractogram of FRHA. The diffractogram shows that the ash was crystalline.

elements. XRD characterization showed that the ORHA was amorphous; while furnace calcined RHA was crystalline in nature with the cristobalite phase of silica present. FTIR analysis found that there was presence of –OH groups in the ash, while SEM analysis showed that the RHA formed agglomerates.

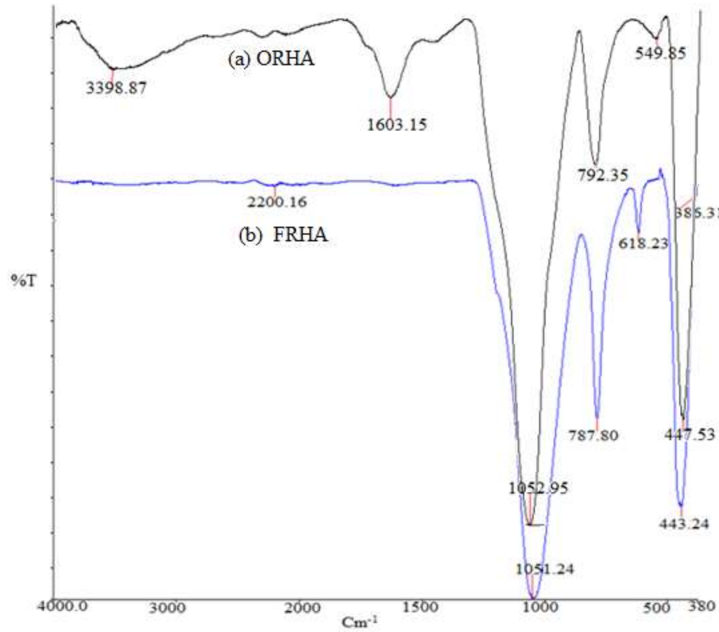

Figure 3. FTIR spectrum for (a) FRHA and (b) ORHA.

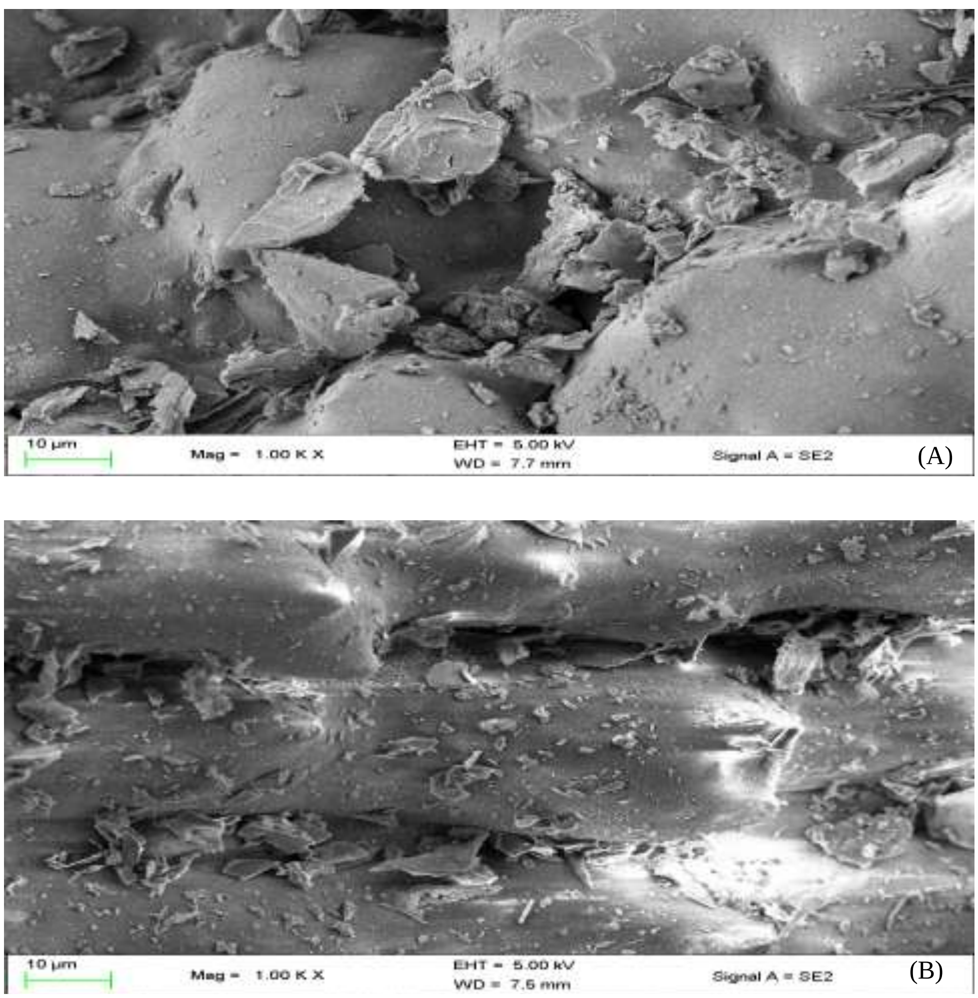

Figure 4. (a) SEM image for FRHA, (b) SEM image for ORHA.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

REFERENCES

Almirón J, Roudet F, Duquesne S (2019). Influence of volcanic ash, rice husk ash, and solid residue of catalytic pyrolysis on the flame-retardant properties of polypropylene composites. Journal of Fire Sciences 37(4-6):434-451.

Ameri F, Shoaei P, Bahrami N, Vaezi M, Ozbakkaloglu T (2019). Optimum rice husk ash content and bacterial concentration in self-compacting concrete. Construction and Building Materials 222:796-813.

Ayswarya EP, Francis KV, Renju VS, Thachil ET (2012). Rice husk ash–A valuable reinforcement for high density polyethylene. Materials and Design 41:1-7.

Babel S, Kurniawan TA (2003). Low-cost adsorbents for heavy metals uptake from contaminated water: A review. Journal of hazardous materials 97(1-3):219-243.

Bakar RA, Yahya R, Gan SN (2016). Production of high purity amorphous silica from rice husk. Procedia Chemistry 19:189-195.

Chandrasekhar SA, Satyanarayana KG, Pramada PN, Raghavan P, Gupta TN (2003). Review processing, properties and applications of reactive silica from rice husk—An overview. Journal of Materials Science 38(15):3159-3168.

Chaudhary DS, Jollands MC, Cser F (2004). Recycling Rice Hull: A Filler Material for Polymer Composites. Advances in Polymer Technology 23(2):147-155.

Chuah TG, Jumasiah A, Azni I, Katayon S, Choong ST (2005). Rice husk as a potentially low-cost biosorbent for heavy metal and dye removal: An overview. Desalination 175(3):305-316.

Deshmukh P, Bhatt J, Peshwe D, Pathak S (2012). Determination of silica activity index and XRD, SEM and EDS studies of amorphous SiO_2 extracted from rice husk ash. Transactions of the Indian Institute of Metals 65(1):63-70.

Genieva S, Turmanova S, Dimitrova A, Vlaev L (2008). Characterization of rice husks and the products of its thermal degradation in air or nitrogen atmosphere. Journal of Thermal Analysis and Calorimetry 93(2):387-396.

He D, Ikeda-Ohno A, Boland, DD, Waite TD (2013a). Synthesis and characterization of antibacterial silver nanoparticle-impregnated rice husks and rice husk ash. Environmental Science and Technology 47(10):5276-5284.

He J, Jie Y, Zhang J, Yu Y, Zhang G (2013b). Synthesis and characterization of red mud and rice husk ash-based geopolymer composites. Cement and Concrete Composites 37:108-118.

Ikhlaq A, Munir HM, Khan A, Javed F, Joya KS (2019). Comparative study of catalytic ozonation and Fenton-like processes using iron-loaded rice husk ash as catalyst for the removal of methylene blue in wastewater. Ozone: Science and Engineering 41(3):250-260.

Kamau GN, Mbindyo JK, Githinji ZP, Tuts RJ, Kinyua AM (1993). Rice Husk Ash and its Application as a Cement Replacement Material in Kenya. International Journal of BioChemiPhysics 2:138-143.

Kang SH, Hong SG, Moon J (2019). The use of rice husk ash as reactive filler in ultra-high performance concrete. Cement and Concrete Research 115:389-400.

Kenechi NO, Linus C, Kayode A (2016). Utilization of Rice Husk as Reinforcement in Plastic Composites Fabrication. American Journal of Materials Synthesis and Processing 1(3):1-5.

Lawagon CP, Amon RE (2019). Magnetic rice husk ash 'cleanser' as efficient methylene blue adsorbent. Environmental Engineering Research 25(5):685-692.

Leong TL (2015). Effects of Rice Husk Ash (RHS) Produced from Different Temperatures on the Performances of Concrete (Doctoral dissertation, UTAR). http://eprints.utar.edu.my/id/eprint/1751

Liou TH (2004). Evolution of chemistry and morphology during the carbonization and combustion of rice husk. Carbon 42(4):785-794.

Maingi F, Mwihaki Ng'ang'a M, Mbuvi HM, Mwangi H (2019). Clay-Rice Husk Ash based Geopolymers for Remediation of Pb (II) and Cd (II) from Wastewater. Egerton Journal of Science and Technology 17(1-139):15-28.

Mohamad FZC (2007). Performance of Recycled High Density Polyethylene (HDPE) (Doctoral dissertation, Universiti Teknologi Malaysia).

Nair DG, Fraaij A, Klaassen AA, Kentgens AP (2008). A structural investigation relating to the pozzolanic activity of rice husk ashes. Cement and Concrete Research 38(6):861-869.

Ong HR, Iskandar WME, Khan MMR (2019). Rice Husk Nanosilica Preparation and Its Potential Application as Nanofluids. In Silver Nanoparticles-Health and Safety. IntechOpen DOI: 10.5772/intechopen.89904

Prachayawarakorn J, Yaembunying N (2005). Effect of recycling on properties of rice husk-filled-polypropylene. Journal of Science and Technology 27(2):343-352.

Rafiee E, Shahebrahimi S, Feyzi M, Shaterzadeh M (2012). Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material). International Nano Letters 2(1):29.

Ramezanianpour AA, Mahdikhani M, Ahmadibeni G (2009). The effect of rice husk ash on mechanical properties and durability of sustainable concretes. International Journal of Civil and Environmental Engineering 10:3.

Sekifuji R, Tateda M (2019). Study of the feasibility of a rice husk recycling scheme in Japan to produce silica fertilizer for rice plants. Sustainable Environment Research 29(1):11.

Shanks RA, Hodzic A, Wong S (2004). Thermoplastic Biopolyester Natural Rubber Composite. Journal of Applied Polymer Science 91:2114-2121.

Shukla SK (2020). Rice Husk Derived Adsorbents for Water Purification. In Green Materials for Wastewater Treatment (pp. 131-148). Springer, Cham. https://doi.org/10.1007/978-3-030-17724-9_6

Singh R, Srivastava P, Singh P, Sharma AK, Singh H, Raghubanshi AS (2019). Impact of rice-husk ash on the soil biophysical and agronomic parameters of wheat crop under a dry tropical ecosystem. Ecological Indicators 105:505-515.

Sonat C, Unluer C (2019). 153001. Development of magnesium-silicate-hydrate (MSH) cement with rice husk ash. Journal of Cleaner Production 211:787-803.

Ugheoke IB, Mamat O (2012). A critical assessment and new research directions of rice husk silica processing methods and properties. Maejo International Journal of Science and Technology 6:430e448.

Venkatanarayanan HK, Rangaraju PR (2015). Effect of grinding of low-carbon rice husk ash on the microstructure and performance properties of blended cement concrete. Cement and Concrete Composites 55:348-363.

Xue B, Wang X, Sui J, Xu D, Zhu Y, Liu X (2019). A facile ball milling method to produce sustainable pyrolytic rice husk bio-filler for reinforcement of rubber mechanical property. Industrial Crops and Products 141:111791.

Zamani C, Mohajerani SS, Ataie A (2019). Synthesis of Three-Dimensional Mesoporous Silicon from Rice Husk via SHS Route. Journal of Ultrafine Grained and Nanostructured Materials 52(2):149-153.