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CHARACTERIZATION OF RISE HUSK AS REINFORCEMENT IN COMPOSITE MATERIALS

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ABSTRACT

Rice husk (RH) as a silica source was studied, resulting from the husk removed in the lab (RHA) and that collected from commercial rice milling area (RHB). Rice husk consists of inorganic, combustible matter in the rice that has been fused into an amorphous structure. A composting treatment was employed in an effort to improve the processability of rice husks. Changes in the chemical composition and physical structurewere analysed. Microscopic techniques, such as X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to observe the surface and internal structure of the RHA and RHB. The results among other things revealed that RHA and RHB both did not contain SiO_2, as they are not in ash form. Microscopic examination showed that it amorphous and has a porous cellular structure and consists of irregular-shaped particles. However, scanning electron microscopy images showed that the epidermis became rugged and lumpy because the composition of rice husks (cellulose, hemicellulose, lignin, and pectin) was partially decomposed due to controlled burning conditions, an effect confirmed by the chemical composition. All results indicated that the rice husk prepared and treated in the lab has better physical and chemical properties that randomly selected at the rice processing area, which are beneficial for its utilization in engineering application.

Keywords: Rice husk, Composite materials, XRD, XRF, SEM

1.0 INTRODUCTION

Due to the present Nigerian economic situation, government is encouraging farmers and other Nigerian to improve the level of productivity of food and other farm product. This make the rice production more ruminant and making its husk available in abundance without proper utilizations where part of it is taking to farm in mixture of fertilizer and little of it is utilize in animal food. In the course of a typical milling process, the husks are removed from the raw grain to reveal whole brown rice which upon further milling to remove the bran layer will yield white rice. Among the different existing residues and by-products, the possibility of using rice husk (RH) has attracted more attention of the researchers than any other crop residues. First due to the excess of this residue, 100 million tonnes of husk are obtained from an annual world production of 500 million tonnes of rice, a huge quantity of residue that can only be consumed by the cement, and ceramic industries that use a wide range of by-product (Akindoyo, J.O.; Husney, N.A.A.b.; Ismail, N.H.; Mariatti, M., (2020); Chapagain, A. K., and A. Y. Hoekstra (2011);Athira, G.; Bahurudeen, A.; Appari, S., (2019). Secondly, RH is not a proper feed for animals due to its few nutritional properties and its uneven abrasive surface is resilient to natural degradation, which stances serious accumulation difficulties (Kargarzadeh, H.; Johar, N.; Ahmad, I., (2017); Jiang, L.; Fu, J.; Liu, L.; Du, P., (2021)). When it is incinerated, it produces a great quantity of ash. On average, each one tonne of RH on complete combustion produces 200 kg of RH ash. No other crop residue generates such quantity of ash when it is burnt Athira, G.; Bahurudeen, A.; Appari, S.,(2019). Thirdly, the use of RHA as a supplementary material is of interest to many developing countries.

The potential global rice husk ash production is estimated at 21 million tonnes per year which in Malaysia alone, a total amount of 78 thousand tonnes is produced annually (Mehta, P. K., (1992); Memon, S. A., A. S. Muhammad, and A. Hassan (2011); Nuruddin, M. F., N. Shafiq, and M. L. Kamal (2009)). Within recent decades, the emission of rice husk ash into the ecosystem has attracted huge criticisms and complaints, mainly associated with its persistent, carcinogenic and bio-accumulative effects, resulting in silicosis syndrome, fatigue, shortness of breath, loss of appetite (respiratory failure) and even death (AyswaryaE.P., K.F. Vidya Francis, V.S. Renju and E. Thomas Thachil, (2012); Bˇehálek, L.; Bor ˚uvka, M.; Brdlík, P.; Habr, J.; Lenfeld, P.; Kroisová, D.; Veselka, F.; Novák, J., (2020); BalajiA., B. Karthikeyan, and C. Sundar Raj,(2015); Goh, C.S.; Tan, K.T.; Lee, K.T.; Bhatia, S., (2010).With the price of the ash disposal cost (either in landfills or ash ponds) hitting as high as $5/ton in developing countries and $50/ton in developed countries, the urgency of transforming the residue into a more valuable end product has been promulgated (Goh C.S et al., (2010)).

Rice husk constitutes about 20% of the weight of rice and its composition is as follows: cellulose (50%), lignin (25%–30%), silica (15%–20%), and moisture (10%–15%). Bulk density of rice husk is low and lies in the range 90–150 kg/m^3(Patricio Toro, Rau´ Quijada, Omar Murillo and Mehrdad Yazdani- Pedram (2006); Mohamed, R.; Mohamed, M.M.F.; Norizan, M.N.; Mohamed, R.R.R., (2017); Muthuraj, R.; Lacoste, C.; Lacroix, P.; Bergeret, A., (2019)). The large amount of silica freely obtained from this source provides an abundant and cheap alternative of silica for many industrial uses. The reactivity of the silica depends on the preparation and temperature (Patricio T. et al., (2006)).

Shafie, S. M., T. M. I. Mahlia, H. H. Masjuki, and A. Andriyana (2011) in their review work study thestructure and surface morphology of the extracted silica of the RHA from Muar Johor and compare it with that obtained from India.The result of the analysis of RHA burned at a temperature of 700^{o}C using X-ray fluorescence (XRF), Scanning electron micrographs (SEM), X-ray diffraction (XRD), and Differential Thermal analysis (DTA) revealed that the RH ash from the two countries has some similarities and differences which is due to environmental factors. In this paper a deeper characterization of rice husk was conducted with the aim of investigating its suitability as a proper reinforcement in composite materials for Engineering applications with environmental factors into considerations.

2.0 EXPERIMENTAL METHOD

The following tests were conducted to characterize the rice husk which has been collected from Namouwa farm Zaria, Kaduna State.

2.1 Scanning Electron Microscopy (SEM)

Scanning electron microscopeJOEL-JSM-6380 Instrument was used to study the morphology of the RHA and RHB which is available at Mechanical Laboratory, Umar Musa Yar'adua University, Katsina. It was used to observe the RH at different magnification under the same working condition to have a clear morphology of this important agro waste material.Required amount of RH powder was poured on the carbon tape which is attached to the holder and then the excess powder was blown with air gun to ensure that small pieces of the powder remain on the tape. After that it was put into in the SEM chamber for analysis. The SEM machine was operated at 10 kV. The magnification of X100 is used to capture a photo of the sample and then subsequently changes the magnification to get the desired morphology.

2.2 X-ray Fluorescence (XRF)

The RH (Rice husk) was thoroughly washed with distilled water in order to remove adhering soil and dust. After that it was dried in an oven at 100^{o}C for 24 hours. Then the dried husk was subjected to the chemical treatment; 2 M HCL, 5% solid at 25^{o}C before calcinations to increase silica content. After the leaching process, the treated husks were washed with distill water and then dried again. The treated husk was then subjected to calcinations at 700^{o}C for six (6) hours, after which it was subjected to the XRF analysis (Johar, N., I. Ahmad, and A. Dufresne (2012)). The machine used for the analysis was XRF Bruker S4 Pioneer which was operated at 60 KVP and 50 Ma.

2.3 Husk through X-Ray Diffraction (XRD)

X-ray diffraction analysis (XRD) technique was used in this work to determine the crystallographic structure of the two rice husk samples RHA and RHB by irradiating the two samples with incident X-rays. The rice husk samples were subjected to X-Ray Diffraction (XRD) analysis using an X-Ray Diffractometer to determine their silica structure. The X-

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Ray Diffractometer (Model Bruker D8 Advance) was used. Prior to analysis, the husk samples were ground to a powder form by simple pounding using a mortar and pestle due to its brittle nature. The ground samples were analyzed by Cu Kα radiation with a scanning rate of 0.05^{o} per second 40 kV/20A, speed 0.05^{o}/min and scanning at $3^{o} \geq 2\Theta \leq 90^{o}$.

3.0 RESULTS AND DISCUSSION

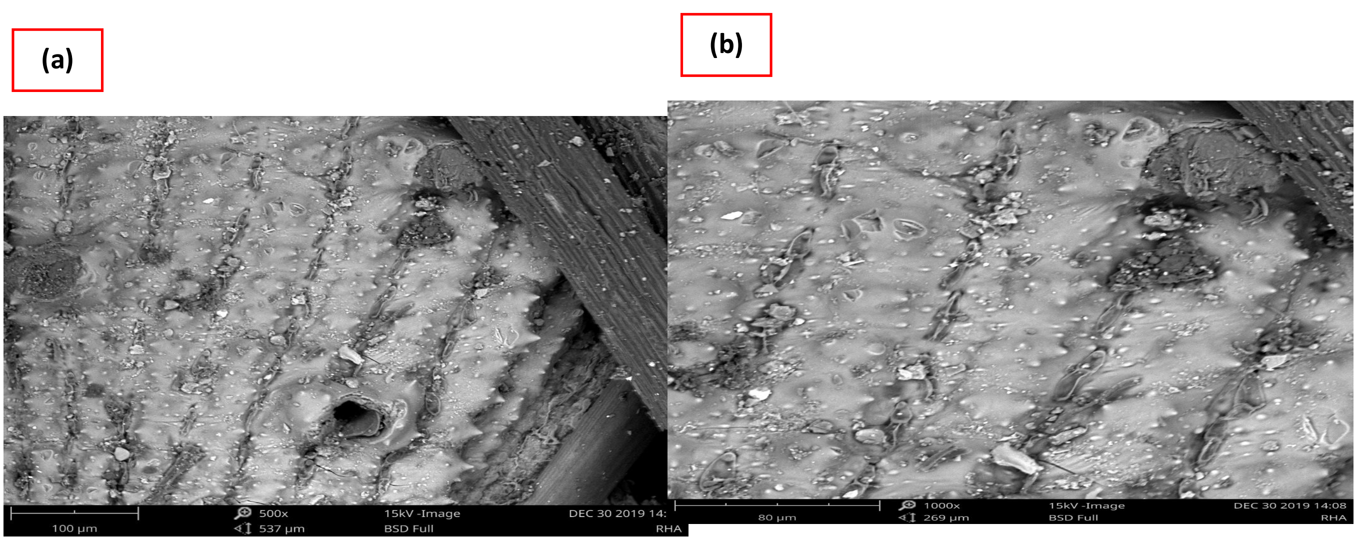
3.1 As- received images of rice husk

The RH was observed to be brown in colour and between 1.9-2.2mm in length with a total average length of 2.0mm. It is lighter in weight and it contains about 30%–50% of organic carbon (Johar, N., I. Ahmad, and A. Dufresne (2012)). The husks are removed from the raw grain for further experimental actions as shown in Figure 1.

3.2 Morphological features of RH using SEM

Rice husks are the hard protective coverings of rice grains which are separated from the grains during milling process and is an abundantly available waste material in all rice producing clusters within the county, and it contains about 30%–50% of organic carbon. It was revealed from Fig. 1-3, the SEM images under the same working voltage showing (a) the exterior surface morphology of rice husk at 500x , and (b), (c) are magnified view of the surface bumps at 1000x and 1500x respectively.

Fig. 1.0: Rice husk at different magnification (a) 100x, (b) 200x & (c) 300x



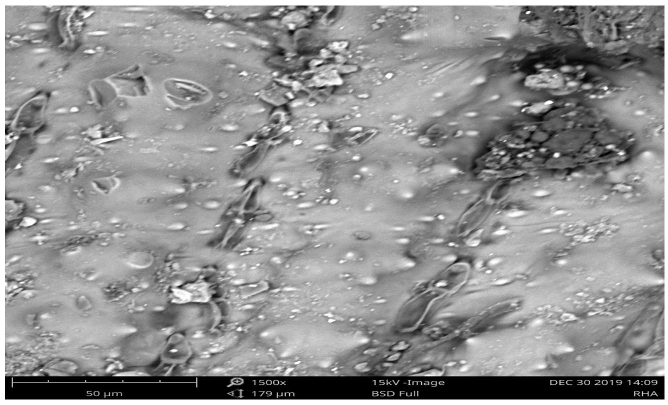


Fig. 2: SEM images showing (a) the exterior surface morphology of RHA 500x, and (b) 1000x, (c) 1500x at magnified view of the surface bumpsr

From the images observed in Fig. 2, 3 and 4 using SEM, it was clear that, the RHA processed under researchers care in the laboratory and clean before the experiment has some different morphological view with that of RHB collected from commercial rice milling area. The particle size in RHA was arranged in column like pattern whereas that from commercial area RHB has a conical shape well arrange pattern in columns and rows. Both RH has the same colour under the SEM but with different particles arrangement as observed. Some crack was observed on RHB which may be due to excessive milling condition and there is evident of some particles as impurities in the material.

3.3 X-ray Fluorescence (XRF)

It is evident from Tables 1 and 2 that, there was no trace of silica in both RHA and RHB, which means that, for any research that is concern about the presence of high percentage of silica is found in the rice husk should be in ash form not as it is from the rice milling area. Fig. 5 & 6 shows the plot of(a) peak values of elements & compounds and (b) the compound plot at a particular % concentrationand (a) background count and (b) the % concentration against the elements and compounds for RHA respectively. Fig. 7 &8 also revealed the plots of (a) peak values of elements & compounds and (b) the compound plot at a particular % concentrationand (a) background count and (b) the % concentration against the elements and compounds for RHB respectively.

Table 1.0: Chemical analysis of RHA

Elements	Concentration (%)	Peak (cps/mA)	Background (cps/mA)
Fe_2O_3	0.11204	1288	-36
CuO	0.000672	22	10
NiO	0.0147	2	10
ZnO	0.001330	77	10
Al_2O_3	0.14763	2063	1694
MgO	0.3375	196	230
Na_2O	0.0765	9	51
S	0.07917	1331	1071
P_2O_5	0.3511	2000	342
CaO	0.18095	740	103
K_2O	0.32368	1419	-108
MnO	0.01956	914	103
Rb_2O	0.000585	4	1
SrO	0.000608	4	1
Br	0.000156	0	1
Cl	0.04556	62	4
Cr_2O_3	0	93	
V_2O_5	0.000278	17	112

Table 2.0: Chemical analysis of RHB

Elements	Concentration (%)	Peak (cps/mA)	Background (cps/mA)
Fe_2O_3	0.06844	787	-25
CuO	0.002353	77	9
NiO	0.0351	4	10
ZnO	0.007993	461	-2
Al_2O_3	0.01291	180	1611
MgO	0.0461	27	228
Na_2O	0.0276	3	40
S	0.03530	593	1138
P_2O_5	0.17359	989	486
CaO	0.15560	636	-5
K_2O	0.06576	288	-18
MnO	0.014569	681	110
Rb_2O	0.000353	2	2
SrO	0.000557	4	2
Br	0.000281	1	1
Cl	0.01036	14	5
Cr_2O_3	0	70	
V_2O_5	0.000286	10	55

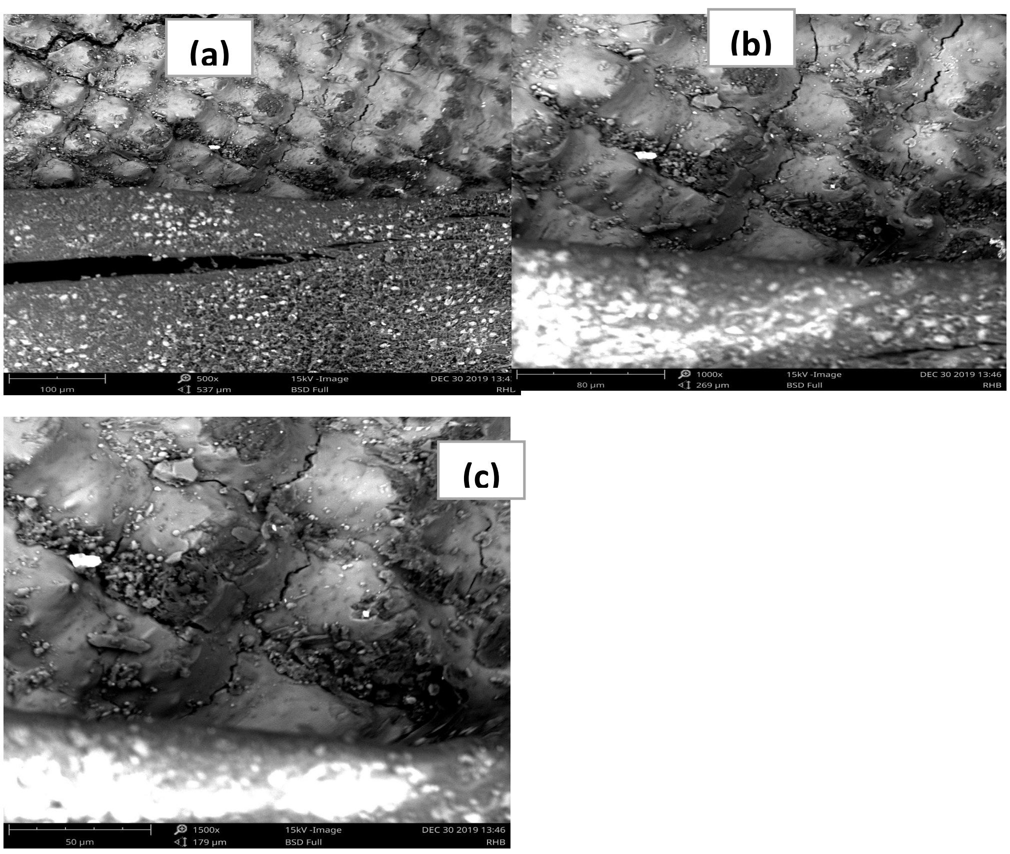


Fig. 3: SEM images showing (a) the exterior surface morphology of RHB 500x, (b) 1000x &, (c) 1500x magnified view of the surface bumps

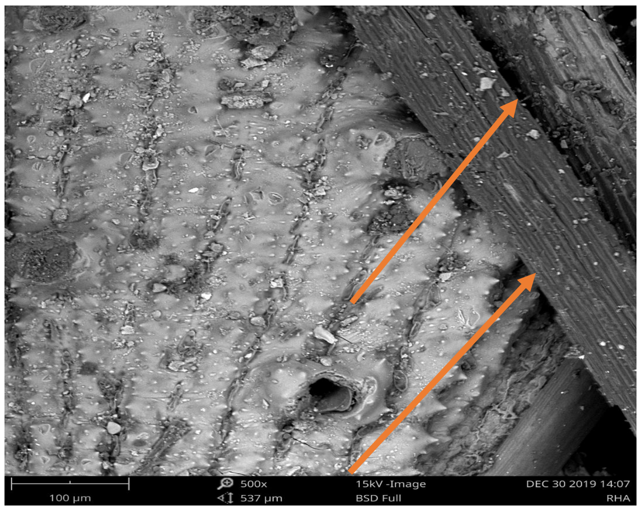


Fig. 4: SEM images showing the cross-section of the of rice husk RHA at 500x magnification with the hypodermis section fiber (H-fiber) portion emphasized by arrows

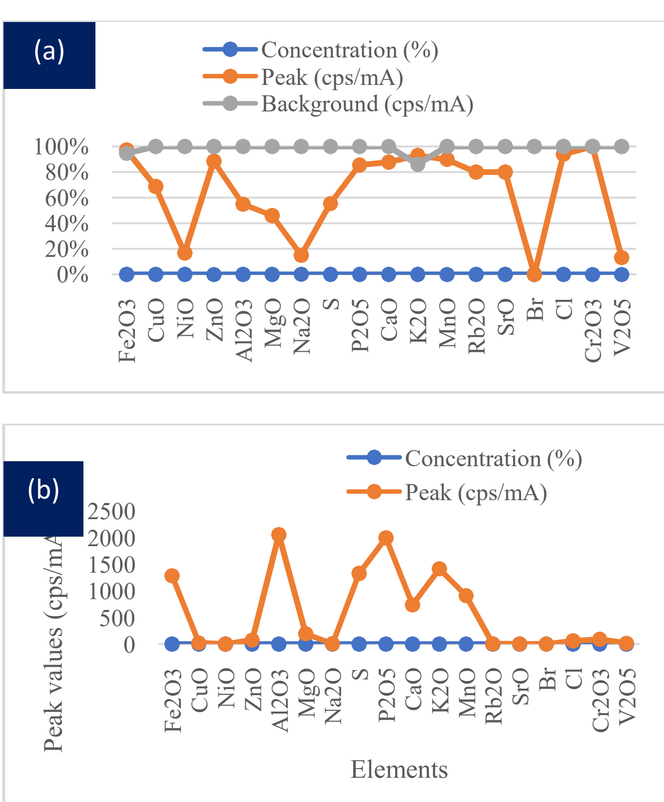


Fig. 5: A graph of XRF spectra showing (a) plot of peak values of elements & compounds and (b) the compound plot at a particular % concentration for RHA from table 1.

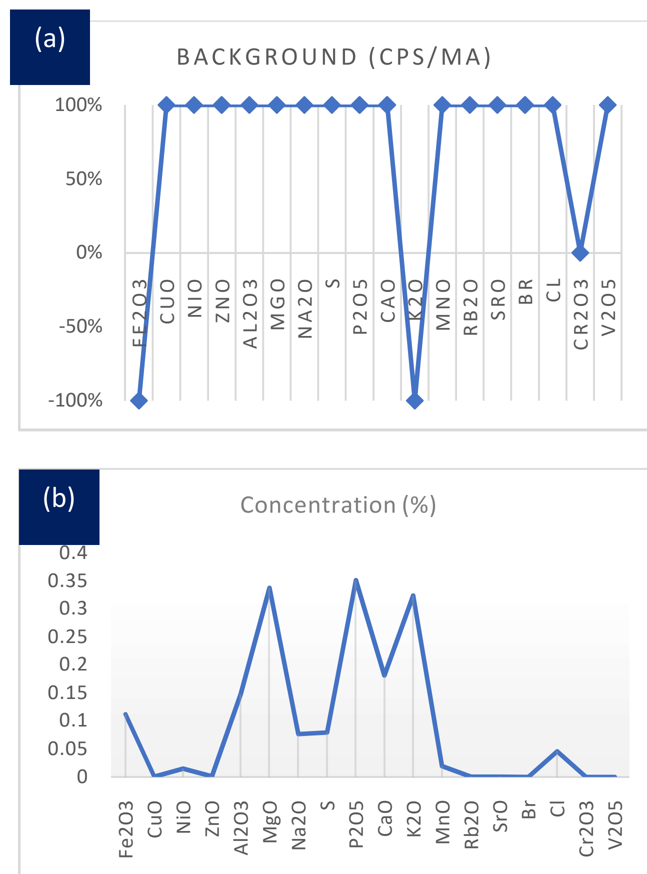


Fig. 6: A graph of XRF spectra showing (a) plot of background count and (b) the % concentration against the elements and compounds found for RHA from table 1.

The heights of the peaks are proportional to the number of x-rays counted, which in turn is proportional to the mass of the element present in the sample. The width of the peaks, in general, is an indication of the detector's ability to "resolve" x-ray energies it observes, or in other words, to correctly identify the energy level of the x-ray it detected. The better the resolution, the tighter these peaks will be, the better the XRF will be in terms of performance (i.e., correctly identifying and quantifying the presence of a particular element). This spectrum has a couple of features of interest. As this spectrum demonstrates, any particular element or compound can have more than one peak associated with it, for example lead, or zinc, or iron in this spectrum. As this spectrum also demonstrates, peaks for individual elements may be so close that for all practical purposes they are indistinguishable. The Al, Fe, P, K peak at higher values is a good example. This is what causes what is known as interference.Jamo Usman Hassan, Mohamad Zaky Noha, Zainal Arifin Ahmad, (2014) also have similar observations in their work to study the cchemical and Mineralogical Properties of Rice Husk Ash (RHA). They observed that, the ash was mainly amorphous form as indicated by a broad peak cantered on 2Ɵ angle. The phase concentration is indicated by the peak height, with higher peak representing higher concentration. The amorphous structure is indicated by a background hump at peak position of approximately 25o on the diffractogram. This is because the rice husk ash was found to be reach in silica, whereas our sample has no trace of silica content as it is not in ash form.

The better the resolution, the tighter these peaks will be, the better the XRF will be in terms of performance (i.e., correctly identifying and quantifying the presence of a particular element). This spectrum has a couple of features of interest. As this spectrum demonstrates, any particular element can have more than one peak associated with it, for example lead, or zinc, or iron in this spectrum. As this spectrum also demonstrates, peaks for individual elements may be so close that for all practical purposes they are indistinguishable (Jamo U. H. et al., (2014)). In the RHB sample it was observed that, the Al, Fe, P, K peak is at higher values but almost 60% lower than that of RHA, which indicated that RHA is far better than RHB quantitatively. It is recommended to remove the rice husk samples for any experimental needs in the lab than to take it from commercial rice milling clusters.

3.4 X-Ray Diffraction (XRD) Analysis

The intensities and scattering angles of the X-rays that leave the sample materials were measured and plotted graphically as shown below in Fig. 9 (a) and (b) for RHA and RHB respectively. It is clear from Fig. 9 that a very broad humped peak on the plot with short range ordering indicated that, the RH is non crystalline in nature. Bˇehálek, L.; Bor ˚uvka, M.; Brdlík, P.; Habr, J.; Lenfeld, P.; Kroisová, D.; Veselka, F.; Novák, J., (2020) also found the same thing in their work to study the non-isothermal crystallisation behaviour and thermal properties of poly(lactic acid) (PLA)-based biocomposites filled with technical cellulose fibres (CeF) and rice husks (RHs) at 10-30 mass% loadingprepared by twin-screw extrusion and injection moulding to enhance stiffness of resulting biocomposites through differential scanning calorimetry (DSC) under various cooling rates (5, 10, 20 and 40 degrees C min(-1)). They observed that, the influence of RH and CeF on transformation behaviours of PLA alpha MODIFIER LETTER PRIME-/alpha-polymorphs leads to the total elimination of imperfect alpha MODIFIER LETTER PRIME-crystals with increasing amount of RH and CeF. This is as a result of the non-crystalline nature of the RH as filler that has direct effect on the matrix material.
Table 2 shows the XRF result of the RHB. Here also it is the same with the result observed in the case of RHA as the heights of the peaks are proportional to the number of x-rays counted, which in turn is proportional to the mass of the element present in the sample. And the width of the peaks, in general, is an indication of the detector's ability to correctly identify the energy level of the x-ray it detected.

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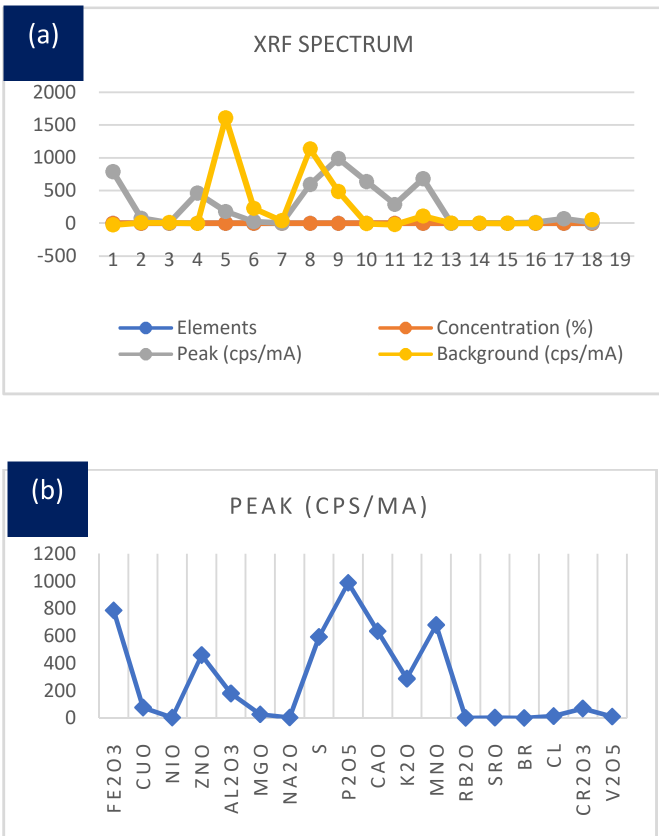


Fig. 7: A graph of XRF spectra showing a (a) the peaks and background at a particular % concentrationand (b) plot of peak values of uncommon elements

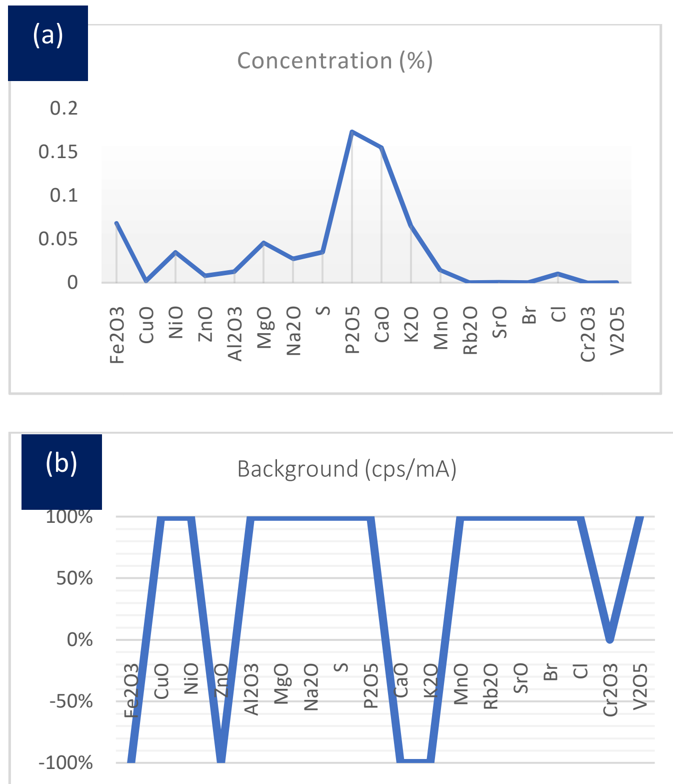


Fig. 8: A graph of XRF spectra showing a (a) plot of background count and (b) the % concentration against the elements found

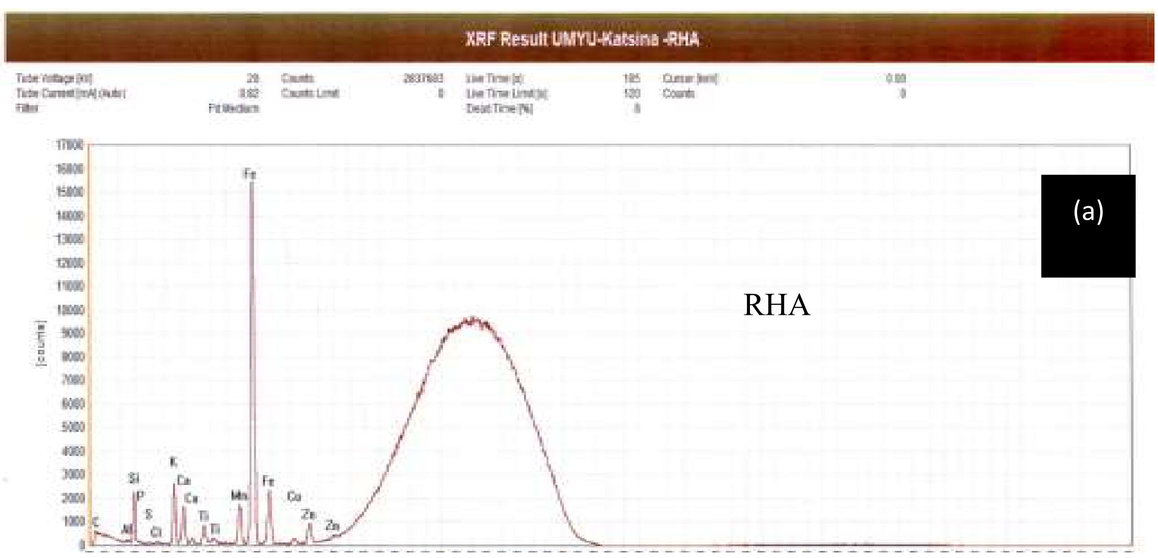
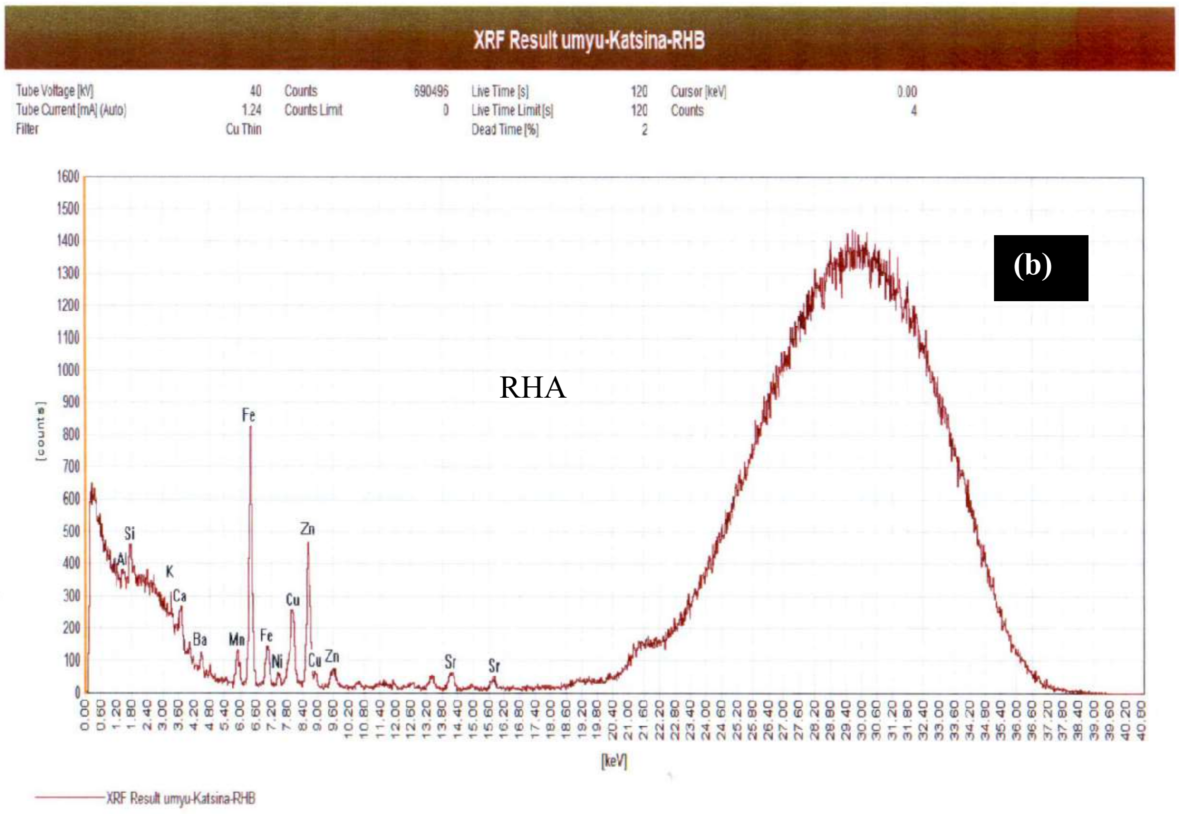


Fig. 9: The diffraction peak width for (a) RHA and (b) RHB using XRD



4.0 CONCLUSION

It was revealed from the experimental results that, rice husk as a fiber and rice husk ash as a silica source can serve an important role as reinforcement in a composite material. It also consists of inorganic, combustible matter in the rice that has been fused into an amorphous structure. The chemical composition and physical structureof treated rice husks were analysed. Microscopic techniques, such as X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to observe the surface and internal structure of the RHA and RHB. The results among other things revealed that RHA and RHB both did not contain SiO_2, as they are not in ash form. Microscopic examination showed that it amorphous and has a porous cellular structure and consists of irregular-shaped particles. However, scanning electron microscopy images showed that the epidermis became rugged and lumpy because the composition of rice husks (cellulose, hemicellulose, lignin, and pectin) was partially decomposed, an effect confirmed by the chemical composition.

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