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OPTIMIZATION OF TECHNOLOGICAL PROPERTY OF RICE HUSK ASH (RHA)

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ABSTRACT
Recently, rice husk (RH) has attracted researchers attention for their possibilities to be more than any other crop remnant, this is due to its availability and promising nature. Averagely, 500 million tonnes of rice are produced worldwide each year, leaving behind huge amounts of waste that will be used in our cement and ceramic industries to produce 100 million tonnes of husk as a variety of byproducts.. When rice husk is incinerated, it produced a high quantity of ash known as Rice Husk Ash (RHA). On average, 200 kg complete combustion of rice husk ash is obtained from one tonnes of rice husk. Raw Paddy Rice was bought and blended with motar to extract rice husk. The grinded paddy was extracted locally by spreading the rice on tray and blew into air in an enclosed room. The RH was calcinated at 700°C for 1hour 30mins, grind and sieve to remove agglomeration. The sample was treated with NaOH and HCl acid to remove the unwanted material and enhance the silica production. X-ray fluorescence and X-ray Diffraction analysis results revealed RHA treated with NaOH is rich in silica with about 19.33 (4%wt) molecular structure, which indicated that chemical treatment play a vital role in improving the physico-chemical properties of RHA.

Keywords: Rice husk, Rice husk ash, Silica production

INTRODUCTION
Rice is the seed of the monocot plants *Oryza glaberrima* (African rice) or *Oryza sativa* (Asian rice). Although it can live as a perennial and yield crop for many years in tropical climates, it is often grown as an annual plant. Depending on the kind and quality of the soil, rice plants have a maximum height of 1.8 m (3.3–5.9 ft), and sometimes much higher. It has 50–100 cm (20–39 in) long and 2–2.5 cm (0.79–0.98 in) broad leaves that are long and slender. The tiny, wind-pollinated blooms are produced in a pendulous inflorescence that is 30–50 cm (12–20 in) long and branching. The grain, or caryopsis, that is edible is 3-5 mm (0.20–0.47 in) long and 2-3 mm (0.079–0.12 in) thick. More than half of the world's population, rice is a staple diet. 8 nations in Africa, 17 countries in Asia and the Pacific while 9 countries in North and South America use rice as the main source of dietary energy. 20% of the world's dietary energy is produced from rice, 19% from wheat, and 5% from maize (Shelke et al., 2010).
When burned, no other agricultural residue produces as much ash as from rice (Namiki et al., 2005). Furthermore, several developing nations are interested in using rice husk ash (RHA) as an additional material. According to Mehtra (1992), there is a possible 21 million tonnes of rice husk ash produced worldwide each year in which Nigeria alone produced 121 thousand tons of rice husk ash yearly (Sasu, 2022).
Roughly, it was estimated that the production of paddy in Nigeria stand at about 1.5 million metric tons, producing huge amount of rice husk from rice mills (Sasu, 2022). The husk is typically burned, producing a significant amount of ash in and around the mills, which poses a serious risk to public health and causes pollution. The production of thousands of metric tonnes of pure, high-grade silica, particularly for the ceramic and porcelain industries, is possible with rice husk as the raw material.

Figure 1: Sample of Rice Husk

More than 75 countries cultivate rice (Natarajan et al., 1998a), and developing nations produce more than 97% of the rice husk produced worldwide (Armesto et al., 2002). Depending on the type, rice husk made up 14 to 35 wt % of the harvested paddy, with an average of 20 wt % (Jenkins, 1989). Globally, the predicted output of husk is 80 million tonnes annually (Kapur, 1985).

In 2022, the annual paddy output in Nigeria was around 5.4 million metric tonnes (Sasu, 2022), out of this amount, rice husk accounted for 22 %, which is approximately 1.2 million metric tonnes per annum. Rice husks are regarded as a type of waste from the milling of rice and are frequently burned outside or allowed to decompose slowly. The fact that rice husk is an inexpensive and plentiful source of silica is still mostly unknown, despite the fact that a little percentage of it is utilized to make animal bedding. In several underdeveloped nations, rice husk has been used to some extent as fuel for parboiling and cooking paddy rice, although this use is neither complete nor effective. Such underutilization clearly demonstrates the loss and waste of resources that could actuality generate revenue to extract silica through combustion processes.

Rice husk ash refers to all types of ash formed by burning rice husk. In actuality, the type of ash that is produced changes greatly depending on the temperature at which it burns. The silica in the ash undergoes structural transformations depending on the combustion conditions, such as time and temperature. Crystalline ash forms at temperatures above 800 °C, while amorphous ash forms at temperatures between 550 and 800 °C (Rajeshkumar et al., 2016). Due to its characteristics, rice husk ash (RHA) has a wide range of applications. It is an excellent insulator due to its poor thermal conductivity and has applications in steel foundries and refractory bricks. It is an active pozzolan with a variety of uses in the concrete and cement sectors. It can be used in its refined form as silicon chip (Celestine et al., 2013).

MATERIALS AND METHODS

Raw Paddy Rice was bought at Kure Market, Kpankugu road in Bosso local government area Minna, Niger State. It was grinded in a mortar with pestle to extract rice husk needed. The extraction of rice husk from the grinded paddy was done using local method, the rice was spread on tray and blew into air in an enclosed room. Then weighed and divided into three and placed in a local clay pot for calcination, each pot contains 279.54gram, 246.56gram, and 241.11gram respectively making a total of 767.21gram. It was then placed in an electric muffle furnace at temperature of 700 °C for 1hour 30mins. The sample was then grind and sieve to remove agglomeration. 20grams of NaOH (Sodium hydroxide) pellets was put in 500ml of distilled water and stirred till the pellets dissolved in distilled water, 10grams of ash from the sample was mixed with NaOH solution thoroughly using glass rode and dried using hot air oven at temperature of 100 °C for 90mins and sieved using filter paper to obtain the precipitate silica, the residue was dried at 60 °C in the hot air oven for half an hour. Similarly, 10grams of RHA was dissolved in 500ml of hydrochloric acid (HCl) and mixed homogeneously with glass road and kept in an ice bath for 2 hours. The solution was filtered and dried in over for 1hour 30mins at 100°C. X-ray fluorescence analysis, X-ray Diffraction analysis were conducted to check chemical and physical structure of RHA.

Figure 2: Grinding and Sieving of Raw Rice

RESULTS AND DISCUSSIONS

X-ray fluorescence (XRF) as a spectroanalysical technique is compatible in determining the chemical compounds present in a sample. The XRF machine used for this analysis was operated at 60 KVP and 50 mA. It is pertinently clear that, there are several elements presents in Rice Husk such as S, Fe, Al, Mg, P, Ca, K, Mn, Cl, Si and Nb, this clearly revealed that, RH is promising candidate for varieties of application, such as silica production, as pozzolanic material and in ceramic industry as quartz replacement. Table 1 below shows the elements present in Rice Husk, their concentration percentage, peak and background.

Table 1. Elemental composition of Rice Husk, Concentration Percentage, Peak and Background Count Per Second (cps)

Elements	Concentration (%)	Peak(cps/mA)	Background(cps/mA)
S	0.08739	2002	961
Fe	0.04356	729	-26
Cu	0.000699	39	15
Nl	0.00227	4	9
Zn	0.0068S34	290	-0
Al	0.01275	266	1569
Mg	0.05115	322	166
Na	0.0039	2	49
P	0.8337	8279	-818
Ca	0.10650	594	325
K	0.7600	4202	-250
Mn	0.016705	931	95
Rb	0.00380	18	3
Sr	0.000963	7	2
Br	0.00027	1	2
Cl	0.08061	147	-1
Cr	0.000032	1	61
V	0.001145	11	21
Si	0.77	3	27
As	-0.00400	2	3
Nb	0.1174	11	5
Ta	0.00519	7	33
Ag	0	0	1035

Similarly, after calcination, sodium hydroxide and hydrochloric acid treatment, Rice Husk Ash (RHA) was further subjected to x-ray fluorescence analysis to investigate the effect of the treatments. Evidently, for untreated RHA the compound with highest percentage is silica with about 75.5% and high in peak cps followed by K_2O. This implies that the calcinations of RH enhance its silica content compared to other elements present in the RH.

Treatment have been reported by scientists to improve the workability and mechanical properties of fabricated materials, Di-Bella et al. (2010) reported that, flax fabric-epoxy treated with NaOH revealed better performance. Similarly, Sawpan et al. (2011) studied the influence of NaOH treatment on the tensile and morphological properties of Hemp fibers, they reported that, Sodium Hydroxide treatment produced better results than other chemical treatments. It is therefore important to note that, RHA treated with NaOH shows a tremendous changes on the compositions, it can be seen that, some chemical compounds such as Na_2O. MgO and Al_2O_3 have increased while others such as SiO_2, P_2O_5, K_2O and CaO have drastically reduced, this is a good trend that gives room for research and the flexibility is what researchers have been looking for. Acid treatment is the treatment that is used to remove the small quantities of materials before extracting compounds present in RHA. The best and suitable acid used for the treatment of RHA to obtain pure silica as reported in the literature is hydrochloric acid (HCl) (Durumin-iya et al). Table further revealed that, HCl acid treatment is the most suitable and efficient treatment for the enhancement and improvement of silica content in RHA, the percentage of SiO_3 has increased to 94% which is overall maximum for untreated and NaOH treated RHA. Table 2 below shows Chemical composition of Rice Husk Ash (RHA), NaOH Treated RHA and HCl Treated RHA.

Table 2: Chemical composition of Rice Husk Ash (RHA), NaOH Treated RHA and HCl Treated RHA

Compounds	Untreated RHA (%)	NaOH Treated RHA (%)	HCl Treated RHA (%)
Fe_2O_3	0.2263	0.5096	0.12353
Ni_2O	0.00048	0.00190	0.000632
CuO	0.00631	0.01187	0.00713
ZnO	0.04370	0.08678	0.01429
Ga_2O_3	0.000056	0.000124	0.00008
CeO_2	-0.00500	-0.00400	-0.00400
Ta_2O_5	0.00775	0.0201	0.0104
WO_3	0.149	0.335	0.145
Na_2O	0.131	7.105	0.198
MgO	4.955	5.694	0.480
Al_2O_3	2.419	2.614	1.131
SiO_2	75.456	41.652	94.708
P_2O_5	8.545	1.3585	0.6654
SO_3	0.4942	0.3621	0.3136
Cl	0.818	1.711	1.479
K_2O	6.4375	1.1136	0.2583
CaO	0.5994	1.4272	0.07377
TiO_2	0.01241	0.02959	0.01317
V2O5	0	0.00013	0.00019
Cr_2O_3	0.00037	0.00183	0.00051
MnO	0.13940	0.31832	0.02509
BaO	-0.05000	0.0300	-0.05000
As_2O_3	0.00008	0.00019	0
Br	0.000216	0.000138	0.0007
Rb_2O	0.00774	0.00154	0.00104
SrO	0.0200	0.0478	-0.0600
ZrO_2	-0.04000	-0.04000	0.04000
Nb_2O_5	0.001892	0.001883	0.001927
SnO_2	-0	-0	-0.00
PbO	0.00332	0.00176	0.00357

To determine structure of the RHA samples, it was subjected to X-Ray Diffraction (XRD) analysis using an X-Ray Diffractometer. The samples were analyzed by Cu Kα radiation with a scanning rate of 0.05° per second 40 kV/20A, speed of 0.05°/min and scanning at $3° \geq 2Θ \leq 90°$. The X-Ray Diffractometer (Model Bruker D8 Advance) was used for this research. Table 3 below shows the result for X-ray diffraction of rice husk RH. It is evident that Urea with formula C H4 N2 O has highest molecular structure with 75(19) % followed by graphite C with 25(7)% while cristobalite SiO_2 has lowest molecular structure.

Table 3: X-ray diffraction of RH

Phase Name	Formula	Figure of merit	Space Group	DB Card Number	Weight Fraction wt%
Urea syn	C H4 N2 O	2.881	113 : P-421m	00-008-0822	73(7)%
Graphite	C	1.115	194:P63/mmc	00-008-0415	25(7)%
Chlorite	(Mg,Fe)5(Al,Si)5O10	3.374	15 :C12/c1	00-002-0028	1.2(17)%
Cristobalite	SiO_2	3.051	227:Fd3m:2	00-0010424	0.6(19)%
Albite	Na Al Si_3 O8	3.301	2 : P-1	00-003041	1(2)%

Table 4: Peak list for X-ray diffraction of rice husk RA

2θ°	d, Å	Height,cps	FWHM,°	Int.I.,cps	Int. W.,°	Asymmetry	Decay (Hl/Ml)	Decay (Hh/Mh)	Size, Å
21.670 (4)	4.0977 (8)	580 (70)	8.833 (9)	6344 (6)	10.9 (13)	1.649 (3)	0.000 (3)	0.940 (3)	9.564 (10)

The result of phase diagram (called a diffractogram) is presented in Figure 3 below. A large peak cantered at an angle of 2ϴ indicates that RH is primarily composed of crystalline form. The peak height indicates the phase concentration with higher peak denotes a higher concentration. On the diffractogram, a background hump at peak position of roughly 200 indicates the crystalline structure.

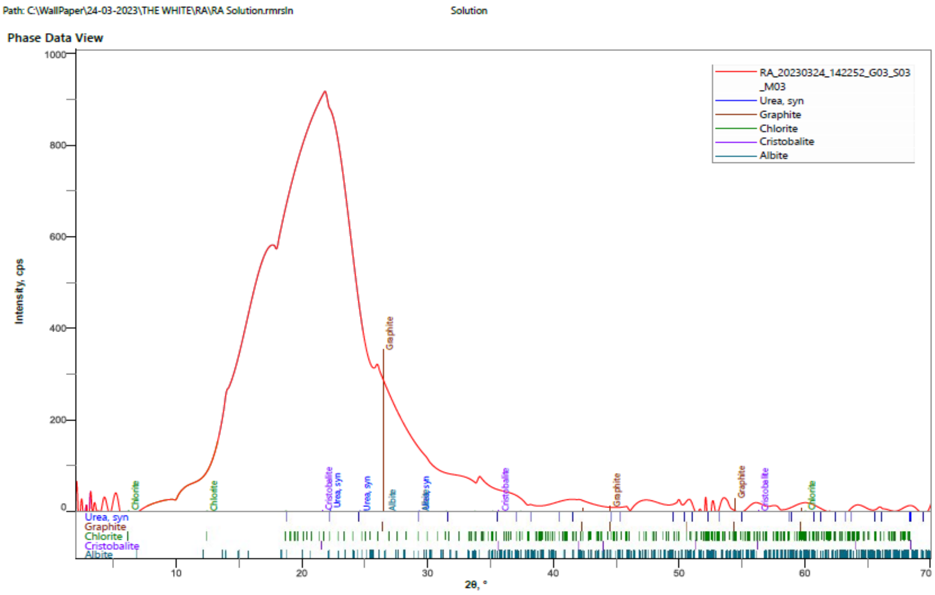

Figure 3: Phase Diagram of RH

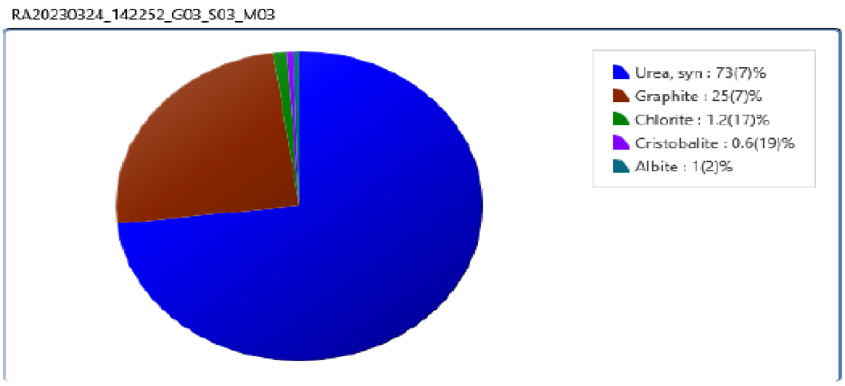

Figure 4: Pie chart indicating high wt% of Urea syn.

Similarly. After calcination of rice husk, the residue is rice husk ash (RHA), Table 5 below shows the XRD of RHA and its pertinent to note that Muscovite has highest molecular structure of 43.8(18) wt% than any other chemical compound present in rice husk ash RHA, followed by Chlorite with 20.6(8) wt%. It is evident that RHA contains high phyllosilicate minerals which are also crystal in nature and of silicate group. Table 5 shows the X-ray diffraction of RHA.

Table 5: X-ray diffraction of RHA

Phase name	Formula	Figure of merit	Space Group	DB Card Number	Weight Fraction wt%
Cristobalite	SiO_2	2.850	227 : Fd-3m:2	00-001-0424	7.9(3)
Graphite	C	1.103	194 : P63/mmc	00-008-0415	9.9(6)
Illite	$K Al_2 Si_3 AlO10(OH)_2$	1.404	15 : C12/c1	00-002-0056	18(3)
Chlorite	(Mg , Fe)5(Al , Si)5O10	2.850	15 : C12/c1	00-002-0028	20.6(8)
Muscovite	KAl_2(Si3Al) O10(OH,F)2	3.114	15 : C12/c1	00-002-0467	43.8(18)

Table 6: Peak list for X-ray diffraction of rice husk ash (RHA).

2θ °	d, Å	Height,cps	FWHM, °	Int. I., cps	Int. W., °	Asymmetry	Decay (ηL/mL)	Decay (ηH/mH)	Size, Å
28.(8)	3.090(7)	118(18)	1.5(2)	186(31)	1.6(5)	5(5)	0.0(10)	0.0(15)	58(9)

The phase diagram result (also known as a diffractogram) in Figure 5 indicates that RHA is primarily composed of crystalline form, as indicated by a broad peak cantered on 2ϴ angle. The peak height indicates the phase concentration, with higher peak denotes a higher concentration. On the diffractogram, a background hump at peak position of 23° indicates the crystalline structure.

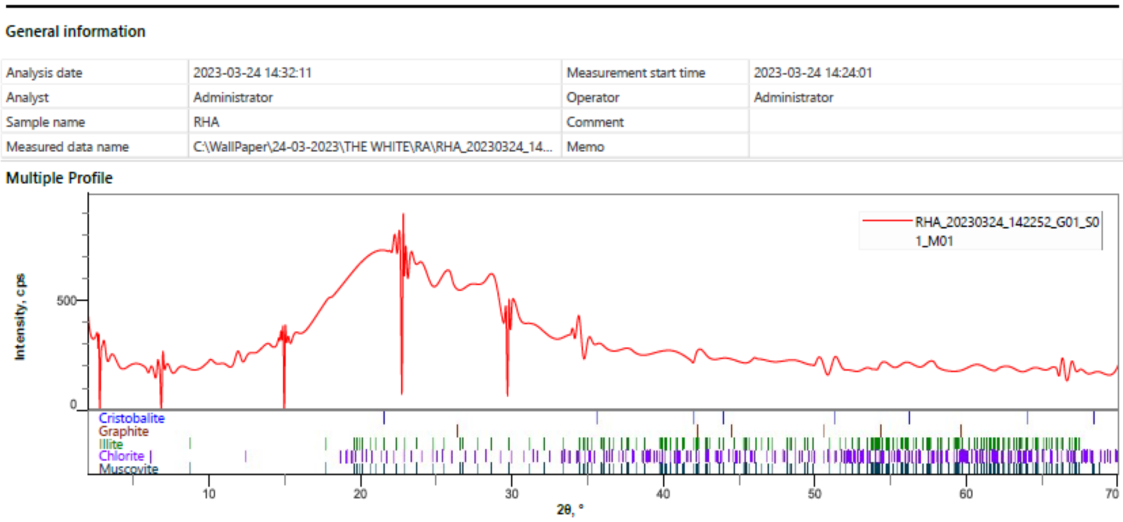

Figure 5: Phase Diagram of RHA

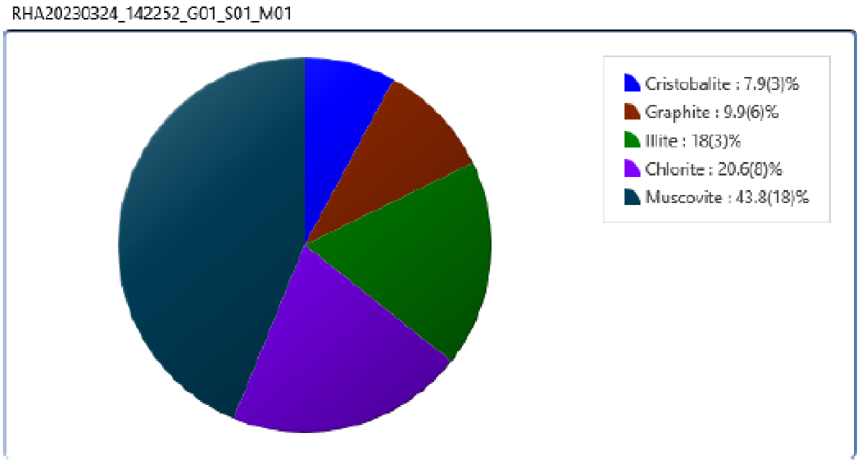

Figure 4: Pie chart of chemical compounds in RHA

Table 7 shows the result of X-ray fluorescence of RHA treated with NaOH. It is clear that after treatment of RHA with sodium hydroxide Muscovite is the highest molecular wt% followed by quartz SiO_2. Therefore, it can be stated that, treatment plays a significant role in improving the percentage of SiO_2 present in RHA. Table 7 shows the X-ray diffraction of RHA treated with NaOH

Table 7: X-ray diffraction of RHA treated with NaOH

Phase name	Formula	Figure of merit	Space Group	DB Card Number	Weight Fraction wt%
Quartz	SiO_2	2.953	154 : P3221	00-001-0649	19.33(4)
Muscovite	H2 K Al3 Si3 O12	3.529	15 : C12/c1	00-002-0055	65.73(10)
Graphite	C	1.157	194 : P63/mmc	00-008-0415	1.804(4)
Urea, syn	C H4 N2 O	2.830	113 : P 421m	00-008-0822	13.09(2)
Albite	Na Al Si3 O8	3.439	2 : P-1	00-003-0451	0.05(15)

Table 8: Peak list for X-ray diffraction of RHA treated with NaOH

2θ, °	d, Å	Height,cps	FWHM, °	Int. I., cps	Int. W., °	Asymmetry	Decay (ηL/mL)	Decay (ηH/mH)	Size, Å
23.629(5)	3.7622(9)	205(28)	9.474(11)	2105(2)	10.3(1)	0.8530(8)	0.000(5)	0.092(4)	8.947(10)

Figure 6 shows the result of phase diagram (known as diffractogram) indicate that RHA treated with NaOH consist mainly of crystalline form as indicated by a broad peak cantered on 2ϴ angle. The phase concentration is indicated by the peak height, with broad peak indicating higher concentration. The crystalline structure is indicated by a background hump at peak position 26° on the diffractogram.

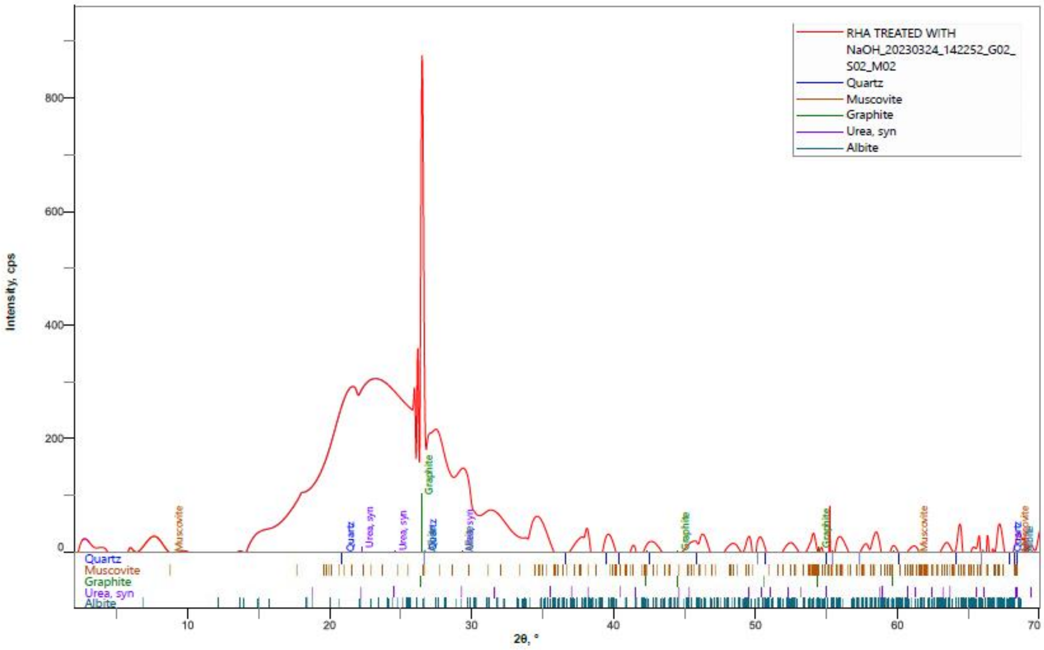

Figure 5. Phase Diagram of RHA treated with NaOH

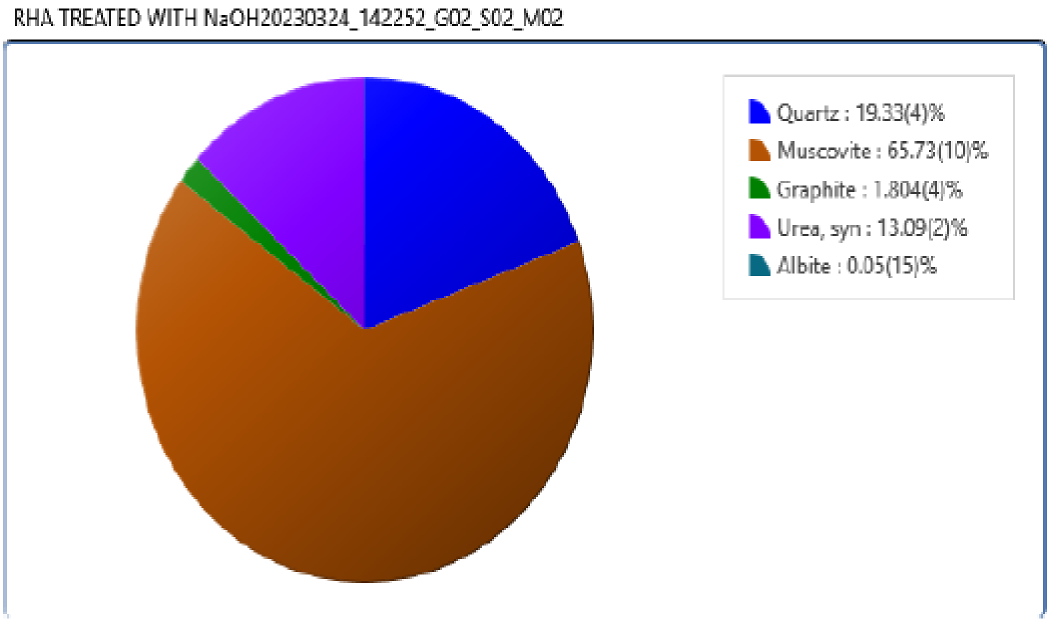

Figure 6: Pie chart of chemical compounds in RHA treated with NaOH

Table 9 below shows X-ray diffraction of RHA treated with HCL acid, it can be noted that Muscovite is still the highest in w% followed by Cristobalite SiO_2. The w % of Muscovite here is much greater compare with table 7 but reduce the w% of SiO_2.

Table 9: X-ray diffraction of RHA treated with HCL acid

Phase name	Formula	Figure of merit	Space Group	DB Card Number	Weight Fraction,wt%
Cristobalite	SiO_2	3.341	227 : Fd-3m:2	00-001-0424	11.20(12)
Muscovite	K Al2 (Si3 Al) O10 (OH , F)2	2 3.367	15 : C12/c1	00-002-0467	77.7(8)
Urea, syn	C H4 N2 O	2.880	113 : P-421m	00-008-0822	8.30(9)
Illite	2 K2 O ·3 Mg O ·Al2 O3 ·24	3.438		00-002-0050	0.06(19)
Graphite	C	1.160	194 : P63/mmc	00-008-0415	2.7(10)

Table 10: Peak list for X-ray diffraction of RHA treated with HCl acid

2θ °	d, Å	Height, cps	FWHM, °	Int.I.,cps	Int.., °	Asymmetry	Decay (ηL/mL)	Decay (ηH/mH)	Size, Å
20.781(9)	4.2710(18)	359(47)	8.947(18)	3433(7)	9.6(13)	0.573(2)	0.000(10)	0.014(7)	9.428(19)

The phase diagram result (also known as a diffractogram) for RHA treated with HCL is displayed in Fig. 7 below. A large peak cantered on a 2θ angle indicates that the majority of the RHA is crystalline form. The peak height indicates the phase concentration with higher peak representing higher concentration. On the diffractogram, a background hump at peak position roughly 2θ ° indicates the crystalline structure.

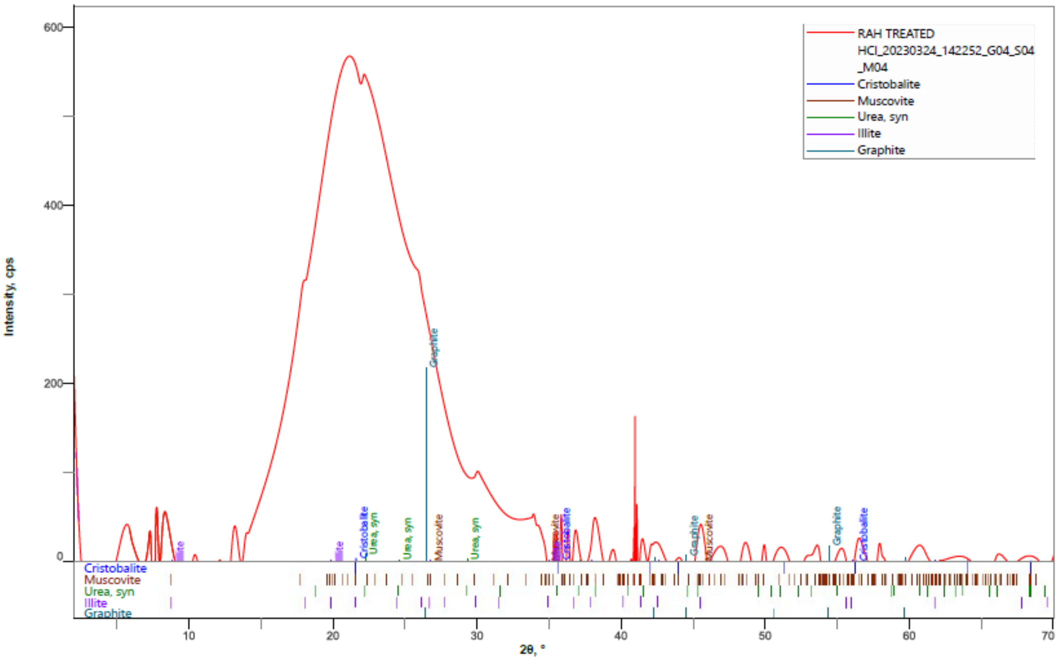

Figure 7: Phase Diagram of RHA treated with HCl acid

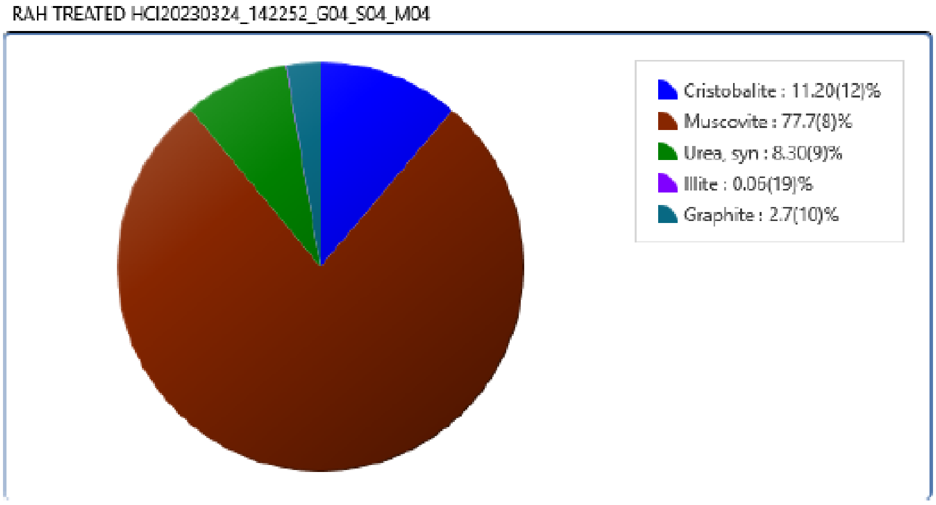

Figure 8: Pie chart of chemical compounds in RHA treated with HCl acid

CONCLUSION

Rice husk is not just a waste material but a promising material of circular economy, thus instead of disposing it off, intensive research can add value by using it through effectively utilizing silica content present in the rice husk ash (RHA) and to produce precipitated silica thereafter. From the findings of this experimental research, it has been observed that RHA approximately contain more than 90% of SiO_2. An accurate cost estimation of this process was not done yet, although rough estimation pointed towards less expensive than the pure silica used by industries, the main advantage of this proposed process is that waste disposal problem of rice husk is eliminated and also valuable product is obtained. The crystalline silica obtained using this method has many applications, e.g. making jewelry items, manufacturing of watches and clocks and raw materials in many industries such porcelain industry as quartz replacement.

From X-ray diffraction analysis it can be concluded that RHA contains mainly crystalline form. The SiO_2 wt% increase, as the RHA undergoes chemical synthesis. Furthermore, it is evident that RHA treated with NaOH has greater SiO_2 wt% compare with the one treated with HCL, thus, this shows that NaOH treatment is a good treatment for the optimum production of SiO_2.

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