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# Characterization of Rice Hull Ash and Its Performance in Turbidity Removal From Water

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This study characterizes the locally obtained samples of rice hull ash and investigates its performance on turbidity removal from water. Four samples of this material were studied, namely, unwashed parboiled rice hull ash (UPRHA), washed parboiled rice hull ash (WPRHA), unwashed unparboiled rice hull ash (UUPRHA), and washed unparboiled rice hull ash (WUPRHA). Scanning electron microscopy (SEM), x-ray diffractometer (XRD), and Fourier infrared spectroscopy (FTIR) were carried out to characterize these samples. A filtration process was carried out to investigate the effectiveness of the rice hull ash medium in removing water turbidity. The XRD results showed the silica, which is present in the ashes, to be cristobalite, quartz, and tridymite. The silica contents of the UUPRHA and WUPRHA were observed to be 77.10% and 98.24%, respectively, while those of UPRHA and WPRHA were 79.07% and 94.97%, respectively. The SEM images showed agglomeration of ash particles after the ashes were washed. The washed RHA samples showed improved pH, a good percentage of turbidity removal (<5 NTU) from water sample. Washing RHA with distilled water increased the efficiency of RHA in turbidity removal from water and regulated water pH to an acceptable range.

**Keywords:** Chemical composition, rice hull ash, SEM, turbidity, XRD

## Introduction

Water scarcity is known to affect all economic and social sectors and, thus, threatens the sustainability of the natural resources base (Muta'aHellandendu 2012). Poor communities have the tendencies to suffer the greatest health problem from inadequate water supplies and this results in poor health. This is especially true for those living in areas where delivery of services in water and sanitation is significantly difficult and scarce (World Water Day 2007). In order to reduce water scarcity challenges, there is a need to enhance water productivity (the volume of production per unit of water) in all sectors, and to protect and restore the ecosystems that naturally capture, filter, store, and release water, such as wetlands, rivers, forests, and soils so that good-quality water may more readily be available.

Reusing of agricultural waste products is one goal for environmental sustainability (Pandey et al. 2012). Agricultural waste products are used in water treatment and, if well established, will be useful to those in the poor communities.

Rice hulls (RH) are waste products from rice processing. RH have a relatively high content of inorganic compounds, which represent about 20 wt% of the dry hull, of which about 94 wt% is silica and 6 wt% is K<sub>2</sub>O, CaO, MnO, MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, and P<sub>2</sub>O<sub>5</sub>, in decreasing concentrations (Houston 1972). The major organic compounds present in the dry hull are 50 wt% cellulose and hemi-cellulose, and 26 wt% lignin, and 4 wt% other organic compounds such as proteins, oil, etc. The amounts of organic and inorganic compositions present in rice hull depend on weather, soil geographical conditions, soil chemistry, and timeliness of crop production operations, plant variety, fertilizer, and agronomic practices in the paddy growth process (De Souza et al. 2002). RH finds application in the cement industry as lightweight construction products, abrasives, and absorbents due to its high silica content (Farooque et al. 2009). The silica in the RH can be obtained by burning it into white ashes. These ashes may consist of either amorphous silica, crystalline silica, or both depending on the temperature of heating used in the burning process.

The morphology of rice hull ash (RHA) can enable the adsorption of metals and other pollutants due to the irregular surface of rice. The physical characterizations of RH and RHA have shown some properties, such as the presence of carboxyl and silanol functional groups, that can make the adsorption process possible (Nakbanpote et al. 2000). RHA has been reported to exhibit good

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adsorptive properties and has been used by several researchers in different part of the world for the adsorptive removal of metal ions and turbidity from wastewater (Srivastava et al. 2009; Ijadunola et al. 2011). However, the detailed properties and the efficiency of the RHA obtained in Nigeria in the removal of turbidity from water have not been documented.

It was recorded by the Federal Ministry of Agriculture and Rural Development (FAMARD) that Kaduna State was the largest producer of rice in Nigeria, accounting for about 22% of the country's rice output, followed by Niger (16%), Benue (10%), and Taraba (7%) states as of 2001 (Project Coordinating Unit 2001). The north east of Nigeria has the second largest estimated land devoted to rice production in Nigeria in 2007 (Ologbon et al. 2012).

Owing to the geographical factors that have great impact on the properties of RH and RHA and, hence, on their applications, it is important to investigate the properties of RHA obtained in the northern part of Nigeria and its efficiency in turbidity removal from water, which is the main focus of this study.

## Material and Methods

Two types of RH samples were obtained from a rice drying-milling plant at Jimeta main market, in Yola, Adamawa State, in the northern part of Nigeria. One was locally parboiled rice hull (PRH) (temperature and duration not known) and the other was un-parboiled (i.e., as received) rice hull (UPRH). The turbid water sample used in the experiment was obtained from Sangere Village, very close to the Modibbo Adama University of Technology, Yola Nigeria. Its parameters were as follows: pH 6.63, conductivity 199  $\mu\text{S}/\text{cm}$ , and turbidity 88 NTU.

The RH samples (both parboiled and unparboiled) were washed with water to remove dirt and other contaminants present in them and then dried in an oven at 110°C for 24 h. An incineration was carried out on the RH in a porcelain crucible for 4 h under static air condition and then the obtained ash were burned inside a muffle furnace (model: FN 100, NuE ve, Nigeria) for 7 h at 800°C. The RHA obtained was divided into two parts, one part was washed with 750 ml of distilled water and the other was left unwashed. The RHA were ground and classified to obtain 0.2 mm particle size. Samples were classified into four parts, namely, unwashed parboiled rice hull ash (UPRHA), washed parboiled rice hull ash (WPRHA), unwashed unparboiled rice hull ash (UUPRHA), and washed unparboiled rice hull ash (WUPRHA).

## Characterization

### X-Ray Diffraction

Samples were studied for their mineralogical characteristics by using x-ray diffraction (XRD) system (Rigaku UltimaIV, South Africa). Each sample was scanned from  $2\theta$  ranging from 5 to 90°.

### Scanning Electron Microscopy

The morphology of ashes was studied using scanning electron microscopy (SEM; Tescan HRSEM, South Africa). Samples were carbon coated prior to morphological studies to avoid charge effect and to obtain clear images. The compositions of the samples were obtained using electron dispersive spectroscopy (EDX) attached to the SEM machine.

### Fourier Transform Infrared Spectroscopy (FTIR)

The functional groups present in all the samples were confirmed using Perkin Elmer (South Africa) 100 Spectrometer FTIR/ATR system and were recorded with characteristic peaks in wavenumbers from 650 to 3500  $\text{cm}^{-1}$ .

### Application of Rice Hull Ash in Water Purification

The samples were applied in water purification by filtration to investigate water turbidity, pH, and conductivity. The laboratory-scale filtration tests were performed using a Büchner (South Africa) funnel (75 mm in diameter) connected to a graduated column (100 ml and 48 cm diameter). The apparatus was used to measure filtration rate and obtain filtrate for analysis. The filtration took place on a layer of 7 g RHA per  $\text{cm}^2$ .

## Results

### Rice Hull Ash Mineralogy

Figure 1 illustrates the XRD patterns of the RHA samples. The main characteristic signals for UPRHA and UUPRHA can be seen at 21.6, 26.7, 27.9, 34.1, and 35.9° for UUPRHA while, the signals for UPRHA were obtained at 21.7, 26.0, 28.5, 34.1, 35.9, 60.0, and 67.8°. The UPRHA consists of  $\alpha$ -cristobalite, quartz, and tridymite. It was observed that after washing UPRHA, the  $\alpha$ -cristobalite and quartz contents increased from 25% to 41% and 17% to 30%,

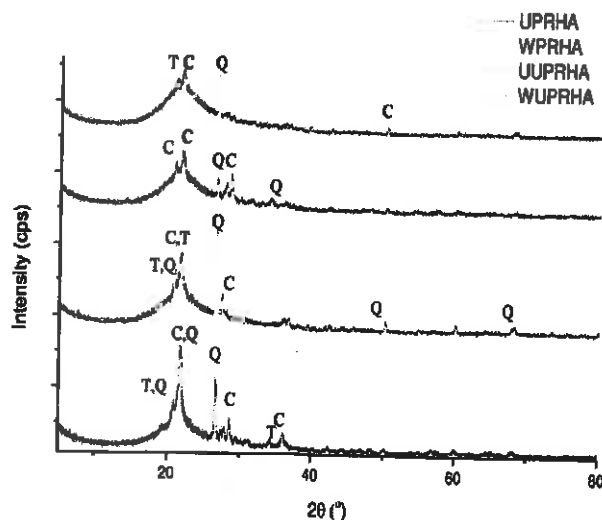


Fig. 1. XRD of RHA: C: cristobalite, T: tridymite, Q: quartz.

respectively. Meanwhile, the tridymite content in the washed ash of UPRHA decreased from 58% to 29%. Researchers have reported that high content of potassium ion acts as reflux for tridymite formation, thus high potassium content in RHA favors the tridymite crystallization (Shinohara and Kohyama 2004; Haslinawati et al. 2008). This explains the decrease in the tridymite content in UPRHA sample (see Table 1).

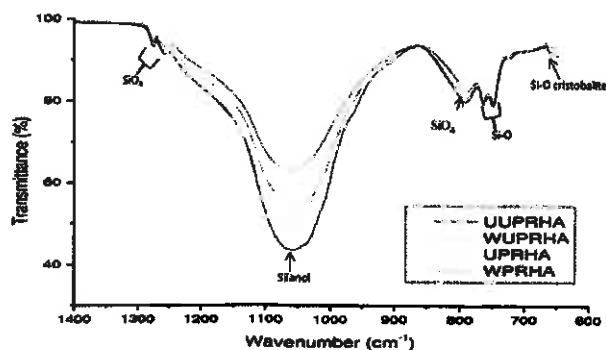
On the other hand, UUPRHA consists of the  $\alpha$ -cristobalite and quartz. After washing the ash obtained from UUPRHA, tridymite was found with  $\alpha$ -cristobalite and quartz. This was due to persistence of cristobalite outside of its thermodynamic stability range, which allows reconstruction of tridymite or quartz from cristobalite transition (Wright and Leadbetter 1975). The decrease in  $\alpha$ -cristobalite content from 89% to 8% and increase in quartz content from 11% to 38% confirms the cristobalite transition. The UPRHA powder was observed to be more crystalline compared to its washed sample. This could be as a result of incomplete evaporation of water from the washed ash. Similar observation was reported by Singh and Singh (2011), whereby the ashes that were treated using water were found to be amorphous in nature.

#### Fourier Transform Infrared Technique

The chemical structure of the RHA plays a significant role in understanding the adsorption process involving RHA. The FTIR technique is an important method of identifying the different functional groups that are influential in the adsorption of various pollutants from water and waste waters. Seven peaks were identified in all cases of UPRH and UUPRH ashes as shown in Figure 2. These are located at 659, 750, 765, 793, 1059, 1259, and 1275  $\text{cm}^{-1}$ . The vibration in the 950–1250  $\text{cm}^{-1}$  region is commonly observed in (aluminosilicates and silica polymorphs due to O-Si-O and O-Al-O stretching modes (Flanigen et al. 1971). The signal at 1059  $\text{cm}^{-1}$  is ascribed to the asymmetric stretching vibrations of tetrahedra  $\text{SiO}_4$  also a band at 793  $\text{cm}^{-1}$  is due to symmetric stretching vibrations of the  $\text{SiO}_4$  (Prasetyoko et al. 2006; Govindarajan and Jayalakshmi 2011). Additionally, the band at 659  $\text{cm}^{-1}$  is a characteristic of the crystalline

**Table 1.** Chemical composition of RHA samples as obtained with EDX

Formula	Compound (%)			
	UUPRHA	WUPRHA	UPRHA	WPRHA
$\text{Na}_2\text{O}$	0.61	0.25	0.54	0.40
$\text{MgO}$	3.51	0.65	3.27	0.78
$\text{Al}_2\text{O}_3$	0.89	0.31	1.43	0.72
$\text{SiO}_2$	77.10	98.24	79.07	94.97
$\text{P}_2\text{O}_5$	10.92	—	10.85	2.49
$\text{K}_2\text{O}$	5.73	0.30	3.71	0.35
$\text{CaO}$	0.52	—	0.41	—
$\text{TiO}_2$	0.05	0.00	0.08	0.03
$\text{MnO}$	0.28	0.19	0.18	0.11
$\text{Fe}_2\text{O}_3$	0.38	0.17	0.45	0.28



**Fig. 2.** FTIR of RHA samples.

cristobalite, while the band at 750  $\text{cm}^{-1}$  may be associated with structures having a Si/Al ratio close to one, such as hydroxysodalite and Si-O absorption is represented by the wave number of 744–765  $\text{cm}^{-1}$  (Hayashi and Oinuma 1965; Novembre et al. 2004). The physical characterizations of RHA have shown that the presence of functional groups such as carboxyl, silanol, silicates, etc. facilitates adsorption processes (Ahmaruzzaman and Gupta 2011).

#### Chemical Composition and Morphological Studies of RHA Samples

The chemical compositions of the RHA samples are shown in Table 1. Two RHA samples (UUPRHA and UPRHA) contained about 77% and 79%  $\text{SiO}_2$ , respectively. The total  $\text{Al}_2\text{O}_3$ ,  $\text{FeO}$ , and  $\text{MgO}$  in UUPRHA and UPRHA were observed to be 4.8% and 5.2%, respectively. The silica ( $\text{SiO}_2$ ) contents of the RHA increased after washing, from 77% to 98% and 79% to 95%, for UUPRHA and UPRHA, respectively, while other components decreased. According to Shinohara and Kohyama (2004), RHA exhibits a characteristic feature of fairly high concentration of potassium about 2–3% as  $\text{K}_2\text{O}$ . However, the RHA investigated in this work exhibits concentration of potassium as  $\text{K}_2\text{O}$  in the range 0.3% to 5.73%. This is due to the difference in climate and regions in which the rice was planted. It was observed that  $\text{TiO}_2$  present in UPRHA was higher than in UUPRHA, this indicates the possibility that UPRHA will perform better if applied in water purification since  $\text{TiO}_2$  has been reported to be useful in water treatment application (Gelover et al. 2006; Rincón and Pulgarin 2006).  $\text{CaO}$  was observed to be absent in both washed ashes. Table 2 shows the composition of rice hull ash as obtained by various researchers from different geographical areas.

Additionally, the chemical analyses of both UUPRHA and UPRHA show that  $\text{SiO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{K}_2\text{O}$ , and  $\text{MgO}$  were the major constituents of both ashes. However,  $\text{SiO}_2$ ,  $\text{MgO}$ , and  $\text{Al}_2\text{O}_3$  were the major contents of washed ash of UUPRH, while the major components of washed UPRHA were  $\text{SiO}_2$ ,  $\text{MgO}$ ,  $\text{P}_2\text{O}_5$ , and  $\text{Al}_2\text{O}_3$ . Silicon dioxide and alumina are known to be among the hardest substances.

The morphologies of UUPRHA and UPRHA are given in Figure 3 at a magnification of 1000 $\times$ . The morphology of RHA can facilitate the adsorption of metals and other

**Table 2.** Composition of RHA as obtained by various researchers from different geographical locations

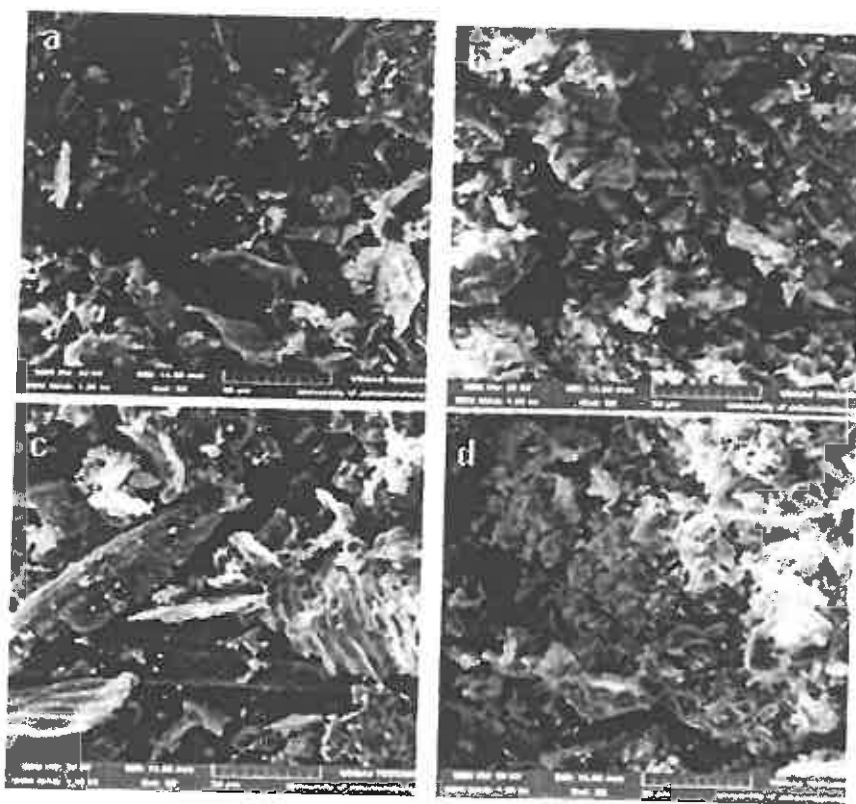
SO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	TiO <sub>2</sub>	SiO <sub>2</sub>	References
—	3.06	0.56	0.15	2.67	0.73	0.36	—	91.57	Singh and Singh (2001)
0.24	0.30	0.21	0.12	2.65	0.31	1.52	0.15	91.30	Farooque et al. (2009)
—	0–0.19	0.25–0.32	—	0.58–1.64	—	—	—	81.09–94	Prasetyoko et al. (2006)
—	0.46	0.67	0.67	2.91	0.44	0.12	—	88.32	Habeeb and Mahmud (2010)

pollutants, because of the irregular surface of rice hull. The RHA particles were observed to be solid in nature with irregular shapes and sizes. The UUPRHA shows rod-like and microporous structures, while the UPRHA exhibits carpet-like structures. The particles of washed ashes show fine morphology and agglomeration, which result in high surface area.

#### Performance of RHA for Water Purification

The performance of RHA in terms of turbidity removal appears promising; turbidity values of 4.05 and 4.15 NTU were recorded for the washed PRHA and UUPRHA respectively (Figure 4). However, the UUPRHA and UPRHA samples show turbidity removal of 9.37 and 5.08 NTU, respectively. This observation is in agreement with the findings of Ijadunola et al. (2011), where their RHA removed turbidity down to 4.00–4.50 NTU for processes without flocculation and 3.50–4.00 NTU for process without

flocculation influent. A turbidity removal of about 95% was achieved for both washed samples of RHA, while 89% and 94% were observed for UUPRHA and UPRHA, respectively. The results show a 6% increase in the turbidity removal of WUPRHA, unlike the 1% increase observed for WPRHA. A study carried out in Sri Lanka by Mampitiyaarachchi (1985) also showed a turbidity removal of 95% at a filtration rate of 0.25 m<sup>3</sup>/m<sup>2</sup> · h and 88% at 2.0 m<sup>3</sup>/m<sup>2</sup> · h filtration rate. The pH values of the filtrates from all the RHA samples were within the accepted pH range for drinking water (6.5–8.5) (Hendrickson 2013). It was observed that the conductivity of the water increased from 199 μS/cm after treatment to 1301 μS/cm, 674 μS/cm, 315 μS/cm, and 602 μS/cm for UUPRHA, UPRHA, WUPRHA, and WPRHA, respectively. This increase in conductivity resulted from the CaO present in the UUPRHA and UPRHA. Wei et al. (2011) reported the effectiveness in adsorption properties of similar material (fly ash) due to an increase in CaO content in the material.



**Fig. 3.** SEM micrographs of (a) UUPRHA; (b) WUPRHA; (c) UPRHA; and (d) WPRHA.

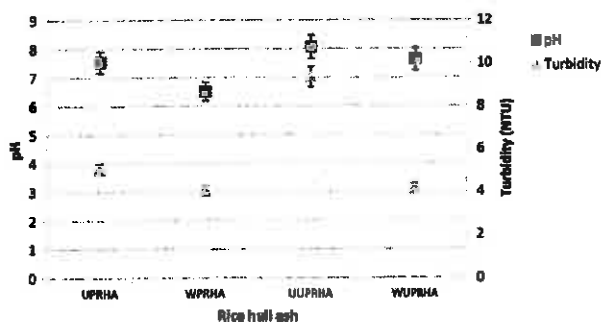


Fig. 4. Samples efficiency in turbidity removal.

## Conclusions

The RHA samples used in this study are efficient as materials for water treatment (turbidity, pH, and conductivity). They are rich in amorphous silica, which are cristobalite, quartz, and tridymite in nature. The washing of the RHA samples resulted in agglomeration of their particles. Increase in silica content and decrease in other compounds present in the RHA samples after washing with distilled water increases their efficiency. A turbidity removal of up to 95% was achieved by using the washed rice hull ashes, while the pH values obtained were in the accepted range for drinking water.

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