PRODUCTION OF SILICA GEL FROM RICE HUSK FOR LABORATORY APPLICATION AS A DESICCANT

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ABSTRACT

Silica of high purity and good surface area was beneficiated from rice husk by the application of heat in a standard solution of hydrochloric acid to produce rice husk ash after calcination at a temperature of 750 °C for 2 hours. Silica gel was then synthesized using sodium hydroxide (NaOH) to solubilize the silica present in the ash with the application of heat on the rice

Introduction

Rice is a grass seed that comes from the *Oryza sativa* (Asian rice) or *Oryza glaberrima* (often referred to as the African rice) species. Around the world, rice is widely farmed, and it is typically a staple diet in many nations (Raphael et al., 2019). Nigeria has therefore started ambitious plans to increase rice output as result; Nigeria is currently the largest rice producing country in Africa. Similar to that, because rice is a basic food in Nigeria, it is predicted that by 2050, there would be a demand for 36 million metric tons (MMT) of rice (Johnson, 2016)

The rice husk is the outer coat of rice (or hull). During the milling process, the rice grains are typically separated from the rice husk. Hull makes up about 20% of paddy weight. The majority of the times, rice husks have been seen as a waste material that should be burned or discarded in landfills. But today, rice husks

husk ash. Therefore, Rice Husk Ash (RHA), Cleaned Rice Husk Ash (CRHA), and Silica Gel (SG) tagged Samples A, B, and C respectively were summarily developed from the synthesis processes. The samples were tested for moisture adsorptivity using laboratory desiccator. Sample C had the highest moisture adsorptivity of 24.34%, followed by Sample B with 15.29% adsorptivity, while the percentage adsorptivity for Sample A was 3.52. In addition to moisture Adsorptivity test, the various characterizations carried out on the samples proved their potential of applicability as desiccants; such as the presence of Silanol functional group, and good surface area especially for the SG sample. Also from the BET analysis of the SG Sample it was found to be a mesoporous material having a pore width value of 2.92 nm, pore volume of 0.1886 cm³/g, and the surface area of 407.6 m²/g. X-ray fluorescence (XRF) Spectrometer was used to determine the elements and the oxides present in the Rice husk ash (RHA), Cleaned rice husk ash (CRHA), as well as in the Silica gel samples.

Keywords: *Adsorptivity, Desiccant, Rice husk ash, Silanol, Silica gel*

are viewed as a commercially viable product. (Raphael et al., 2019) The production of large amounts of garbage is one effect of rice production activities; each ton of processed paddy rice yields 200 kg of rice husk. Hence husk straw, husk, ash, bran, and broken rice are the main solid wastes produced during this process. Because they take up a lot of space, they can be difficult to dispose of, and if not handled properly, they can be hazardous to the environment and the general public's health. (Aderonke et al., 2020)

The combustion of rice husks yields rice husk ash (RHA), which is widely and abundantly available, particularly in developing nations. Surprisingly, the silica content of RHA is estimated to be between 80 and 95 percent weight (Soen et al., 2021), which encourages its use for industrial purposes. Green silica, or silica derived from biomass, is often made by calcining rice husks and then extracting RHA with an alkaline. Before calcination or before extraction, acid leaching is

typically done to rice husk or RHA. When acid leaching is not employed, silica purity seldom surpasses 95%, and the surface area is typically approximately 200 m²/g, which does not meet the requirements for industrial silica gel (Azat et al., 2019)

The use of rice husk as moisture absorber for active food packaging would be very interesting to be developed, even more, the concern of food industries lately seem to look for natural moisture absorber sources as a substitute for synthetic of the silica gel one. Nowadays, moisture absorber, also called desiccant, is widely attached into such food packaging to prolong the product self-life. This absorber would help the package dry thus it could maintain the crispiness of the product inside and also inhibit the growth of mold and other undesirable microbes. The development of desiccant so far is finding the more efficient moisture absorber by combining more than one active ingredient to improve the performance of the moisture absorbers in absorbing and controlling water vapour. Meanwhile, a desiccator is an airtight container that maintains a low humidity environment using a suitable drying agent that fills the desiccator's bottom portion. It is used to store dry materials that must not be exposed to the moisture that is generally present in the atmosphere as well as to cool heated objects. (Warsiki et al., 2021)

Sodium silicate applied as a silicon source in industrial production of silica. However, sodium silicate produced by smelting quartz sand and sodium carbonate at 1300°C not only requires a large quantity of energy, but also further purification (Affandi, et al., 2009) and also it can be cause of widespread environmental pollution. In contrast, low temperature extraction of amorphous silica from plant biomass yields high quality, environmental friendly and cost effective product as opposed to the high energy processing of the inorganics

MATERIALS, EQUIPMENT, AND EXPERIMENTAL METHODS
Materials and Equipment

Rice husk was obtained from Imaab rice milling factory located at *Badiko* layout, Western bypass in Kaduna South Local Government Area of Kaduna

State, Nigeria. The muffle furnace at Step B Laboratory Kaduna Polytechnic was used for the ashing of the rice husk. An analytical grade of sodium hydroxide and hydrochloric acid were procured from the Cybex Chemical Store in Kaduna. Deionized water was provided from Chemical Engineering Laboratory likewise, Kaduna Polytechnic Kaduna.

Beneficiation

Beneficiation was carried out during the processing of the Rice Husk (RH) to Rice Husk Ash (RHA). Rice husk was weighed into the 500 ml beaker, and 1mole of HCl was prepared and then added to the sample. This was heated for 2 hours at an average temperature of 100 °C. After heating; the sample was then thoroughly washed with deionized water until a pH value of 7 (neutral) was achieved. The content was then dried in a drying oven operated at temperature of 110 °C for 2 hours.

Ashing

The ashing process was carried out by loading the dried beneficiated rice husk into a clay crucible and then placed in a muffle furnace operated at a temperature of 750 °C for the period of 2 hours. The ash was then allowed to cool for 5 hours and then packaged in an air tight container and labelled Sample A.

Cleaning

The cleaning process was carried out by adding 3% v/v HCl into RHA and this was heated to a temperature of 80 °C and kept for a period of 2 hours. The content was then allowed to cool to a room temperature and filtered using a Whatmann filter paper size 1. The filtrate was discarded and the cake was then dried in a drying oven operated at a temperature of 110 °C for 2 hours. The resultant product is the cleaned rice husk ash (CRHA). It was then packaged and labelled Sample B.

Extraction

During the silica extraction process 1m of NaOH was poured into the cleaned rice husk ash and then heated for 100 °C for 2 hours with constant agitation

using a glass stirrer. After the heating, the content was then allowed to cool to room temperature. NaOH was used in order to solubilize the silica present in the sample.

Filtration and Gelification

Whatmann size 1 filter paper was used in conjunction with a conical flask together with a funnel to carry out the filtration process. The filtrate being the silica gel liquid was then used for the next process and the cake was discarded. The filtrate obtained was then transformed into gel through a process known as gelification. During this process, 1 mole of HCl was prepared, and then added in drop wise into the filtrate with continuous stirring using a glass stirrer and the pH was also monitored. The addition of the acid continued until the gels were formed and the pH values also dropped to neutral value.

Washing, Drying and Size Reduction

The washing step was performed by adding deionized water and agitating the solution. The water was discarded, and this process was repeated (four times) until the sodium chloride (produced at the end of the process) was removed from the sample.

Drying was then carried out on the wet gel using the drying oven, and it was dried for a period of 2 hours at a temperature of 110 $^{\circ}$C.

The silica gel obtained after drying was then subjected to size reduction using a laboratory scale ball milling machine. Different sizes were obtained including the powder. This was then sieved using a laboratory sieves. The silica gel of the sane sizes relatively was obtained, packaged, and labelled Sample C.

Moisture Adsorptivity Test

In other to test the moisture adsorptive properties of the samples, they were subjected to laboratory experiment using a desiccator in the Department of Chemical Engineering, Laboratory Kaduna Polytechnic. Water of known weight was poured in a laboratory petri dish which acted as the CONTROL, and Samples A, B, and C which were of the same weight were then placed inside

the desiccator along with the CONTROL sample. The set up was allowed to stay for five days, however; every 24 hours all the samples including the CONTROL were weighed using an electric weighing balance, and their values were recorded.

The Percentage of water vapour sorption for each sample were calculated using equation 1, according to Puri et al., 2020

$$\%Moisture\ sorption = \frac{W_f - W_i}{W_i} \times 100 \qquad (1)$$

Description: W_i = initial weight of sample (g) at time of 0

W_f = final weight of sample (g) at time of t (day)

RESULTS AND DISCUSSION

X-Ray Fluorescence Analysis

X-ray fluorescence (XRF) Spectrometer was used to determine the elements and the oxides present in the Rice husk ash (RHA), Cleaned rice husk ash (CRHA), and Silica gel (SG) as shown in Table 1

Table 1 XRF result of the samples

S/N	COMPONENTS	RHA	CRHA	SG
1	CaO	0.629	0.529	ND
2	SiO_2	90.87	94.873	97.79
3	Al_2O_3	1.112	0.101	0.09
4	Cr_2O	0.121	0.001	ND
5	CuO	0.102	ND	ND
6	MgO	0.471	0.379	0.211
7	P_2O_3	0.204	0.214	0.07
8	NiO	0.001	ND	ND
9	SO_3	1.011	0.002	0.06
10	K_2O	1.322	0.22	0.12

11	TiO_2	1.154	0.087	0.057
12	MnO	1.13	0.04	0.036
13	Fe_2O_3	0.092	0.083	0.082
14	Na_2O	1.002	ND	ND
15	ZnO	0.12	0.05	0.02

ND= Not detected

It is observed that the percentage weight compositions of most of the oxides in CRHA and SG are lower compared with that of RHA; this is as a result of chemical and thermal treatments given to the RHA by addition of 3% v/v HCl which has successfully reduced some metal oxide impurities. This is in agreement with the finding of Gad *et al.,* (2012) which reported that acid treatment reduces the cellulose and hemicelluloses as well as the traces of the metals in the husk. Hence silica oxide (SiO_2) was seen to have improved from 90.87% In RHA to 97.79% in SG

Fourier-Transform Infrared Spectroscopy (FTIR) Analysis
To determine the samples' molecular compositions and structure otherwise known as functional groups, the RHA, CRHA, and SG were subjected to the FTIR analysis. The FTIR study has been also to observe the alterations in the spectra due to acid and thermal treatments. The spectra of the FTIR images are shown in Figure 1. The peaks represent the absorbance bands and the bands correspond with the various vibrations of the samples atoms when exposed to infra-red spectrum.

Most of the peaks that happen to be present initially on the CRHA have diminished; nonetheless, others have changed location thus, validating the effect of chemical and thermal treatment in reducing the cellulose and hemicelluloses that originally present in the RHA. Table 2 displayed some extracted frequency ranges from the FTIR spectra library and their corresponding bond type or functional groups.
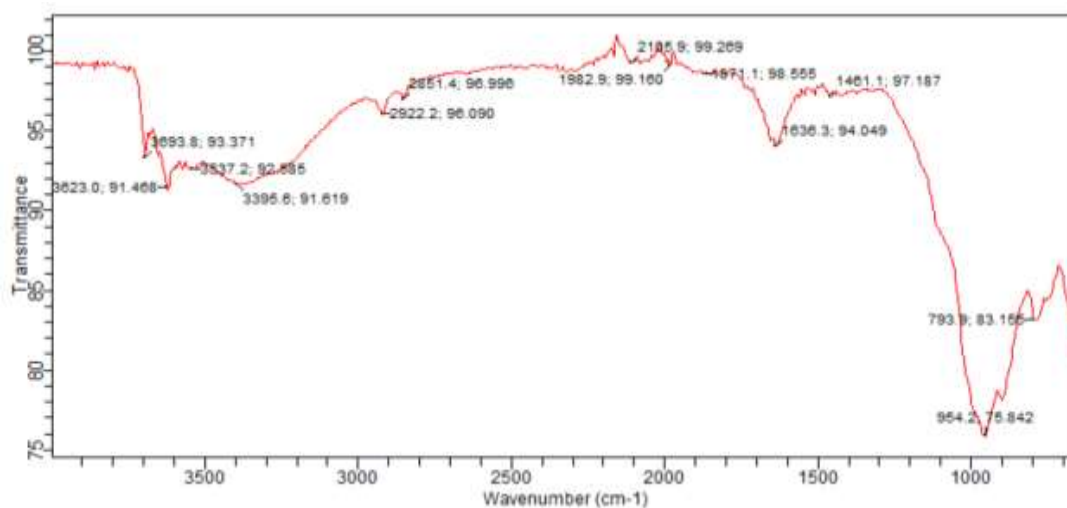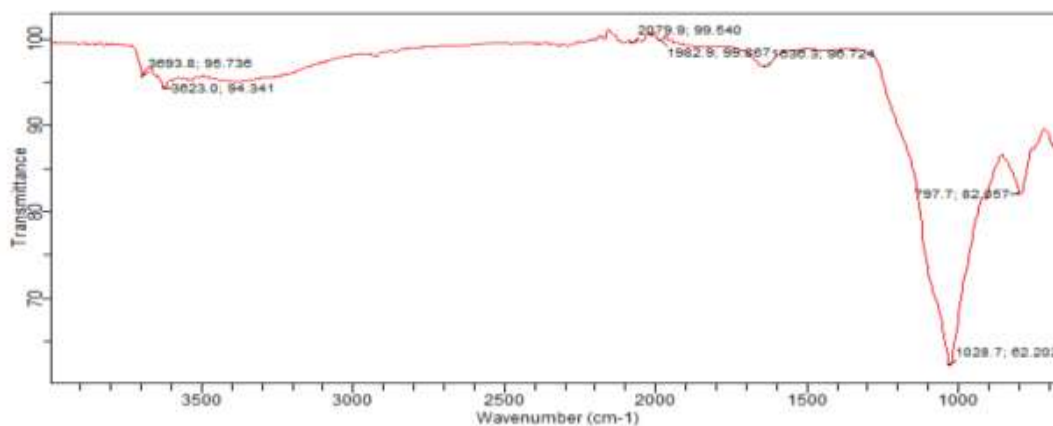
Table 2 Bonding group analysis for FTIR study

S/No.	FREQUENCY RANGE	BOND
1.	3,452 – 1,635	Stretching O-H
2.	1,982.9	C-H
3.	1,982.9 – 1,636.3	C=C, H-O-H
4.	1,461.1 – 453	Si-O

Fourier-transform infrared (FTIR) was also employed to establish phase conversions in the rice husk as well as the surface functional groups.
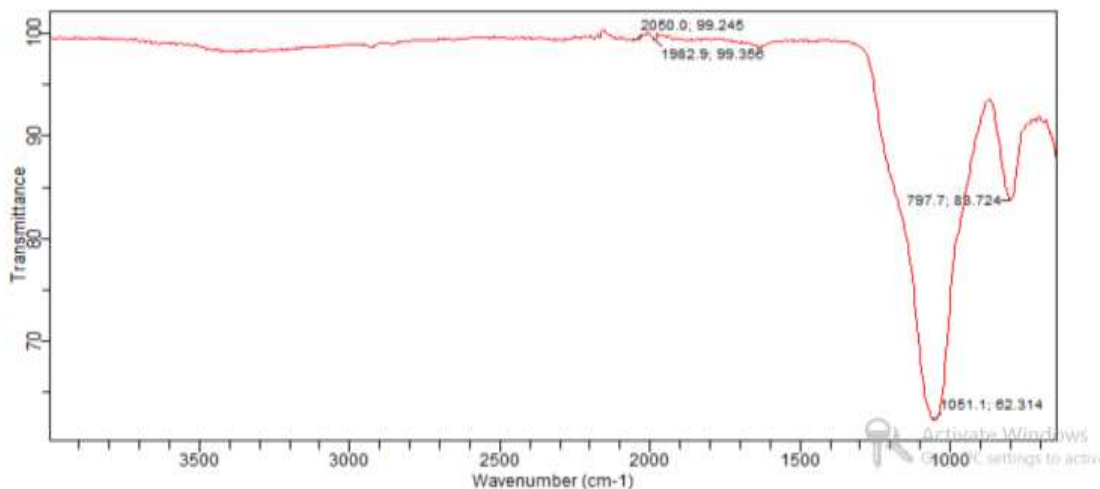

(a)

(b)

(c)

Figure 1: FTIR images for (a) RH, (b) RHA, and (c) SG

The frequency ranges belonging to hydroxyl, carbonyl, and carboxyl are evident in the FTIR spectral shown in the Figure 1 a, b and c. The high content of silica present in ash necessitates the presence of Silanol functional group (Si-O-H) which is related to hydroxyl functional group(C-O-H).

Brunauer - Emmett - Teller (BET) analysis

The pore size distribution and surface area analysis were carried out using BET as shown in Table 3. The BET analysis is considered to be the most extensively used method for estimating surface area (Naveen *et al*, 2014). The results in Table 3 revealed that the SG is mesoporous (2-50 nm) in nature.

Table 3 BET results for silica gel (SG)

S/N	Parameters	Value
1	Surface area (m^2/g)	407.6
2	Pore volume (cm^3/g)	0.1886
3	Pore width (nm)	2.920

According to International Union of Pure and Applied Chemistry (IUPAC) nomenclature, the pore size classification range of 2-50 nm is a mesoporous material, if the pore size of a material is less than 2 nm; that material is microporous in nature, however, if it is above 50 nm, the material is macroporous in nature (Katsuhiko *et al.*, 2012). Therefore; based on this IUPAC classification of pore size and with the value of pore width of 2.92 nm, hence SG is a mesoporous material with a large surface area of 407.6 m²/g, and this value is close to 409 m²/g reported by Supitcha et al (2009)

Moisture Adsorptivity Result
Adsorptivity is the ability to adsorb. The greater the adsorptivity of a substrate, the more readily and strongly a substance will bind to it.
Table 4 shows the result of moisture adsorptivity test carried out on the samples.

Table 4 Moisture absorptivity test

DAY(S)	0	1	2	3	4	5	WG	Absorp.
CONTROL (g)	3	2.127	2.054	1.123	0.912	0.902	-2.098	-69.93%
RHA (g)	5	5.009	5.055	5.074	5.176	5.176	0.176	3.52%
CRHA (g)	5	5.283	5.341	5.693	5.702	5.765	0.765	15.29%
SG (g)	5	5.581	5.550	6.110	6.209	6.217	1.217	24.34%

From Table 4, Control sample with an initial weight of 3 g can be seen to have decreased in weight progressively with respect to the days. After 5 days the control reduced from 3 g to 0.902 g which is close to the original weight of petri dish without water. The essence of placing the petri dish with water inside the desiccator was to saturate the atmosphere within with moisture The weight gain (WG) for the control appeared negative indicating the decrease in weight, likewise the -69.93% absorptivity indicting no absorptivity of moisture but rather lost moisture to the immediate environment within the desiccator. The three other samples were seen to have gained weight as the day progresses,

however, the increase in weights of the samples were not the same. That is, at the end of day 5, SG adsorbed more moisture from the control compared to other samples, followed by CRHA. Hence, SG had the highest ability of water adsorption. This can be further explained from the XRF results obtained where silica content of the SG happened to be highest among the other two samples. Also its large surface area of 407.6 m^2/g, allows the silica gel for showing great water adsorption ability via chemisorption on Si-OH groups.

Conclusions

An amorphous and mesoporous silica gel with 407.6 m^2/g of surface area was successfully produced from RH, and tested as a desiccant on a moist soil sample in a laboratory desiccator. Because of the SG high surface area of 407.6 m^2/g and the presence of OH groups and the polar side of cellulose makes the lignocellulose material naturally hydroscopic, so it has a high water-absorbing ability. This is due to the formation of hydrogen bonds of hydroxyl groups in the polymer chain with water molecules. Hence, from the absorptivity test carried out on the samples; SG performed better with 24.34% absorptivity while, CRHA and RHA absorptivity were 15.29% and 3.52% respectively. Therefore, SG developed from rice husk can conveniently serve as a desiccant in the laboratory. It's therefore recommended that the bio-desiccant (SG) developed should always be stored in high moderate temperature before and after use for maintaining good performance. Also test on the longevity as well as the thermal resistivity of the sample are recommended.

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