

Synthesis of Pure Desiccant Using Rice Husk from Nigeria

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Abstract: Rice husks are forms of agricultural biomass that provide abundant silicon source. They are widely burnt in agricultural fields or disposed indiscriminately because it is difficult to find other uses for them. Despite their non-commercial value and environmental pollution effects, they have ash content that is rich in silica (SiO₂) and therefore could become good source of economic potential. This study utilized this agricultural biomass waste into valuable product, silica (desiccant). Desiccant, Silica (SiO₂) was prepared by using rice husk ash (RHA) and as sources of silicate (SiO₂). Then further reactions of the ash with NaOH at varying times of 1hr, 1½ hrs, 2hrs, 2½ hrs, and 3hrs were carried out to form Sodium silicate (Na₂SiO₃) solution. Then, titration of this solution with H₂SO₄ formed the precipitation of silica-gel. Acid pretreatment was carried out with HCl to remove impurities and other metallic substances. The pretreatment also enhanced the silica yield. Extracted precipitated silica was characterized by Fourier Transform Infrared (FTIR), this also confirmed the presence of some functional groups such as the hydroxyl group and the carbon to carbon double bonds. The silica yield was discovered to increase with increase in time of reacting with NaOH. The percentage yield for RHA was obtained to be 79.20%, 87.78%, 93.64%, 94.00%, 97.60% at 1hr, 1½ hrs, 2hrs, 2½ hrs and 3hrs respectively. The absorption capacity is 20.45%. Also the average moisture content and average ash content are 8.7 and 23.47 respectively.

Keywords: Rice husk, silica, rice husk ash, desiccant, absorption capacity, synthesis

1. Introduction

Farming operations result in the production of agricultural solid waste. It includes other farming and food-related activities as well, so it is not just production. Agriculture produces considerable solid wastes at every level and phase of the food chain. The production of agricultural goods has increased as a result of the world's growing population. Technology developments related to the green revolution and the spread of agriculturally productive soil are further factors that have improved agricultural production (Adejumo et al., 2020).

Food is a requirement for human survival, hence agriculture cannot be eradicated from the planet. However, agriculture has both beneficial and harmful consequences on the ecosystem. A more effective and efficient method of processing agricultural solid wastes is required to boost agricultural production due to the detrimental effects of agriculture on the environment, aquatic life, and human health. Typically, agricultural wastes are burned outdoors or allowed to decompose; while this simply reduces the amount of waste present, it also has an impact on the environment (Obi et al., 2016). While burning creates gases that contribute to air pollution and also forms ash as solid waste, decomposition and putrefaction release methane gas and other harmful chemicals into the atmosphere. As a result, inappropriate agricultural waste management contributes to air pollution, soil and water contamination, and climate change (Adejumo et al., 2020).

The majority of waste agricultural biomass has a significant energy content and other raw materials that can be recovered when managed effectively, so it is crucial to manage it

appropriately. Agricultural wastes are a renewable resource that, if not utilised, generate issues. Therefore, the task is to transform it into an energy source and other useful forms (Bracco et al., 2018). Numerous engineering applications could benefit from the usage of agricultural wastes (Handayani et al., 2022). Fornasiero, et al. (2011) state that silica normally ranges from 98 to 36 weight percent in the ash of agricultural waste materials such rice husks, sugarcane leaves, corn husks, etc. Other metal oxides, such as calcium oxide, magnesium oxide, and potassium oxide, are also present along with silica. Along with some trace elements, after being burned in the air, rice husk releases highly reactive, often nanosized silica particles. During this process, ash that contains various minerals replaces around 20% of the biomass. Depending on the circumstances under which the rice husk was burned, these minerals can either be amorphous or crystalline. In addition, after heating in the presence of activated charcoal, silica can also be changed into silicon. Electronic chips and the production of solar energy are two applications for polished silicon.

There are numerous advantages to making glassware and other products from agricultural waste. It is cost-effective, environmentally friendly, and a renewable supply of silica with a high concentration. It might also be a more efficient approach to manage large agricultural wastes needed to make various engineering materials. In contrast to crystalline silica, which had negative biological consequences, amorphous silica is known to be bioactive, biocompatible, biodegradable, and non-toxic to living human tissue (Martin-Palma et al., 2010). Amorphous silica is now being used more and more in biological applications, medication delivery, and diagnostics. Potential silica-based substances utilized for medication delivery and creating synthetic bone

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tissue include silica spheres, nanospheres, mesoporous silica gel, silica aerogel, and silica stat gel.

The rice husk is an important, accessible source of silica. Ultrapure amorphous silica is obtained from rice husks. It can be used as a cost-effective raw material for the production of silica gels because it contains roughly 60% silica. It has served as a foundation for the creation of a number of silica compounds, including silicon carbide, silicon nitride, amorphous silica, and elemental silicon. Rice husk is leached with hot alkali to produce sodium silicate, which is then processed with mineral acids to produce precipitate silica. It has been described in the literature to first treat rice husk ash with mineral acids such HCl, H₂SO₄, and HNO₃ before thermal treatment to remove contaminants like metals that are detected in traces and can contribute to the production of white ash with a high silica content, resulting in silica with a large surface area. As was mentioned before, rice husk can be used for drying and heat generation in some agricultural areas due to its high calorific value. Additionally, gasification facilities use it to produce electricity on the spot. The ash produced when rice husk is burned to produce electricity can also be used to make silica.

Numerous methods have been documented in the literature for the manufacturing of silica gel from rice husk, all of which adhere to the fundamental process of making silica in the form of alkali-silicate, then precipitating it with a strong mineral acid to obtain silica. It has been demonstrated that both mineral acids and organic acids have been employed in numerous instances when rice husk has been subjected to acid leaching or treatment prior to combustion or pyrolysis. By using acid leaching to remove the inorganic components of the rice husk and breaking down the lignin and organic material, pure amorphous silica with fewer impurities is obtained. HCl, H₂SO₄, HNO₃, and other organic acids including oxalic acid, citric acid, and acetic acid are only a few of the acids that can be employed for leaching.

Akintola et al. (2018) found that agricultural solid wastes are frequently indiscriminately dumped or burned in public spaces, causing air pollution, soil contamination, harmful gas emissions, smoke, and dust. The residue may also be channeled into water sources, polluting the water and aquatic environment. In Nigeria, vast amounts of rice husk are produced as agricultural trash, and managing this material is a significant environmental challenge. The goal of this endeavor is to use the rice husk produced in Nigeria to make desiccants.

Large amounts of rice husk are produced, which is an organic waste. It is a significant by-product of the agro-based biomass sector and the milling of rice. According to Hu et al. (2008), the amorphous form of silica found in rice husk, a cellulose-based fiber, makes up 20% of the material. In addition, it contains 17-23% ash, 10-15% fixed carbon, and 60-65 percent volatile matter (Hu et al., 2008; Mansaray and Ghaly, 1998). Approximately 40% of it is cellulose, 30% is lignin, and 20% is silica (Chindaprasirt et al., 2007). According to Chandrasakhav et al. (2003), the water absorption capacity of rice husk ranges from 5% to 16% of unit weights. About 90% of the rice husk ash is made of silica, a mineral with a high specific surface area, a highly

porous structure, and is lightweight. According to Daoutidis et al. (2013), rice husk ash has been used as an addition in a variety of products and materials including refractory brick, insulation production, and flame retardant materials. This is a result of its very porous structure and effective insulation. Depending on the temperature and duration of the burning, the characteristics of rice husk ash silica change.

The makeup of the rice husks, burning temperature, and burning period all affect how the ash behaves. For instance, burning 100 kilogram of husks in a boiler will produce around 25 kg of RHA. While in some locations it is field-burned as a local fuel, rice husk is sometimes used as fuel for parboiling paddy in rice mills. However, under these situations, the rice husks are only partially burned, which also adds to air pollution (Singh, 2018). Assuming that rice husks have a moisture level of between 8% and 10% and no bran, their calorific value is predicted to be 15 MJ/kg (Tsai et al., 2007). Rice husks have a calorific value that is roughly 50% that of coal.

Combustion of rice hulls generates 'rice husk ash'. This ash is a potential source of amorphous reactive silica, which has a variety of applications in materials science. The majority of the ash is used in the production of Portland cement (Shim et al., 2015). When burnt completely, the ash can have a Blaine number of as much as 3,600 compared to the Blaine number of cement (between 2,800 and 3,000), meaning it is finer than cement. Silica is the basic component of sand, which is used in a variety of materials science applications. This fine silica will produce a concrete that is incredibly dense. Ash is an excellent thermal insulator as well. The ash's fineness also makes it an excellent choice for sealing small gaps in civil structures because it may go deeper than a typical cement-sand mixture. In the Far East's rice-growing regions, such as China and Japan, rice husk ash has long been utilized in ceramic glazes (Oluseun et al., 2017). Since RSA contains almost 95% silica, adding the required amount of silica to the glaze is simple, and the glaze melts more quickly as a result of the small particle size. RHA has a variety of potential applications, including absorbents for oils and chemicals, soil ameliorants, a silicon source, insulating powder in steel mills, "vinegar tar" release agents as repellents in the ceramics sector, and as an insulation material. Using this substance as a catalyst support is one of its more specialized uses.

With the chemical formula SiO₂, silicon dioxide, usually referred to as silica, is an oxide of silicon that is most frequently found in nature as quartz and in a variety of living things. Silica makes up the majority of sand in various regions of the world. One of the most diverse and common groups of materials is silica, which can be found naturally as well as in synthesized forms. Fusion quartz, fumed silica, silica gel, and aerogels are illustrative examples. It is utilized as an electrical insulator in microelectronics, structural materials, as well as pharmaceutical and food industries. Silica gel is a common dehumidifying desiccant, drying agent, adsorbent, filler, and catalyst carrier because of its high surface area (700-800m²/g), great adsorption capacity, and chemical inertness. It can also be found in minerals or ores along with other elements. Silica is still created synthetically for a variety of uses. The SiO₄ tetrahedron

serves as the foundation for the SiO₂ structure. Each silicon atom has a four-atom link with oxygen, while each oxygen atom has a two-atom binding with silicon. Siloxane groups and silanol groups (Si-O-H) are two different forms of functional groups. The siloxane sites on the surface are inert to the majority of activities, while all chemical processes—including physical ones like adsorption—take place on the silanol sites. Additionally, various dicotyledonous plankton husks or seeds, including rice husk and foxtail millet, have been found to contain silica. The three forms of silanol linkages present on the surface of porous amorphous silica are isolated, germinal, and vicinal.

In order to make concrete, silicon dioxide is used in the construction industry (Handayani et al., 2022), the manufacture of glass, as a desiccant, as a sedative, the production of elemental silicon, as an anti-caking agent in spice powders, as a fining agent in juice, beer, and wine, in pharmaceutical tablets, and in toothpaste to remove plaque.

By acidifying sodium silicate solutions, amorphous silica or precipitated silica is produced. Microporous silica is created by dehydrating and washing silica gel. The reaction that takes place when a trisilicate and sulfuric acid are combined (Bogeshwaran et al., 2014). Thermal and chemical processing at higher temperature are required to produce more (reactive or fine-grained) products. Lately, biomass resources such as rice husk as a source of silica are being researched for several industrial applications. Although synthetic silica is produced commercially, the ones produced from plant origins such as rice husks have been noted to have some significant advantages over those from mineral and synthetic origins (Nguyen et al., 2010) which makes them remarkably desirable in applications where high purity silica at humble cost is a necessary requirement. Besides, most biomass resources are waste by-products whose disposal cause environmental nuisance. Hence, developing uses for these waste resources can also lead towards sustainable development.

2. Materials and Methods

2.1 Materials and equipment

Rice husk was sourced from a local rice mill and Distilled water, sodium hydroxide and hydrogen tetraoxosulphate (vi) acid solution were bought from a chemical shop, all located in Awka, Anambra State, Nigeria.

The apparatus and equipment used are Measuring cylinders (500 ml, 100 ml, 50ml, 5ml), Filter papers (Whatmann), Beakers (500 and 250ml), Spatula, Volumetric burett, Crucibles, Reflux condenser, Weighing Scale (1455T 18), Magnetic stirrer hot plate (TMOV – 420), Oven (Heraeus T6060), Heating Mantle (PCE-HM 500), Fourier Transform Infrared Spectroscopy machine (M5-30).

2.2 Methodology

2.2.1 Water Washing

RH was water washed thoroughly with distilled water to remove the soluble particles, dust, dirt and other sand

particles present in the husk. It was then drained of any water and dried in oven at 90°C for 8hrs.

2.2.2 Acid Pretreatment

The acid treatment was carried out with Hydrogen tetraoxosulphate (vi) acid (H₂SO₄) solution in different concentrations with solid to liquid (wt.) ratio of 1:10 to remove soluble elemental and metallic impurities and partial hydrolyzation of the organic content of the rice husk prior to combustion.

50g of dried WWRH and 500 ml of 1M hydrogen tetraoxosulphate (vi) acid (H₂SO₄) solution were mixed in a round bottom flask and heated using a reflux condenser at a temperature of 90°C for 2hr. The solids, after separation by settling and filtration, were washed repeatedly with distilled water, to remove the acid retained, and then dried at 90°C for 8h. The rice husk obtained was light weight and was labelled Water washed treated rice husk WWTRH).



Figure 1: Residual Ash gotten after filtration and drying

2.2.3 Calcination

Calcination was done in a muffle furnace for 12hrs at a temperature of 700°C to produce white ash of rice husk. The weight of the rice husk was measured before and after the calcination process.

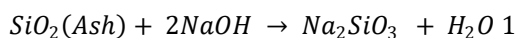


Figure 2: Rice husk Ash after calcinations

2.2.4 Digestion

This involves the digestion of the rice husk ash with caustic soda at S/L (wt. %) ratio of 1:10 at specific conditions. In this process the silica in the ash gets extracted with caustic soda to form a sodium silicate solution. After the completion of the digestion, the solution was filtered to remove the non-reactive impurities and carbon residues for the residual undigested ash present in the solution and the clear filtrate was taken for precipitation.

5g of RHA was added to 50ml of 2.5M NaOH and mixed thoroughly by using glass rod and then heated at 80°C for different time values (1hr, 1.5hrs, 2hrs, 2.5 hrs and 3hrs) to dissolve silica and produce sodium silicate. Silica content of the RHA was leached out to the aqueous phase in the form of soluble sodium silicate according to the following reaction Yuvakkumar et al., 2014).

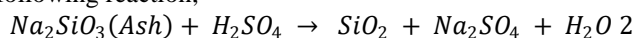


The solution was cooled down and filtered using filter paper. The filtration process was repeated several times to obtain a clear solution. The filtrate contains the sodium silicate solution which was further used to obtain silica.

2.2.5 Precipitation (silica Gel preparation)

This step involves precipitation of silica from the sodium silicate solution with acid. The silica gel is obtained by sol-gel process. The sol is prepared by a silica source solution and gelation is occurred by the addition of an acid. The filtrate, a sodium silicate solution, was allowed to cool to room temperature and then slowly titrated with 1M Hydrogen tetraoxosulphate (vi) acid (H_2SO_4), which precipitated the dissolved silicate in the form of white gelatinous solid (SiO_2) with constant stirring. Silica gels started to form when the PH decreased below 10, and continue by adding of HCl drop wise until the PH of the solution reaches 7, after which no further precipitate was formed.

The precipitation using HCl occurred according to the following reaction;



2.2.6 Aging of the gel

The gel prepared in the above step was aged in its mother solution. When the gel is formed from the sol, it was aged for 14hrs at room temperature. The gels were then filtered using Whatman filter paper and washed repeatedly with distilled water to remove solute salts and wash off any sodium tetraoxosulphate (vi) formed due to the acid base side reaction.

2.2.7 Drying of the silica gel

The solid left on the filter paper in the form of gels (silica gel) is the pure silica. This was further dried in an oven at 80°C for 6h, this yields very pure and crystallized silica. The solids were then ground in to powder using mortar and pestle and stored in air tight plastic bag in sampling bottles for further analysis and characterization.

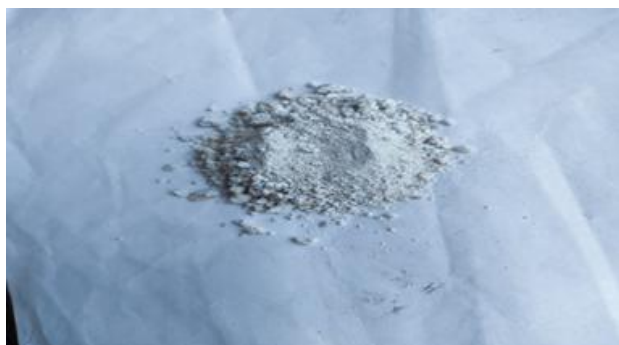


Figure 3: Crystallized Silica

2.2.8 Proximate Analysis

Ash content: Three samples of crucibles each having 5g of rice husk were weighed out, then placed in a muffle furnace at 700°C for 2 hours, after the specified length of time the crucible holding the samples were removed and allowed to cool. The differences in weights were noted and read and the average calculated.

Percentage of ash content was calculated with the formula below,

$$\text{Ash content} = \frac{W_2}{W_1} \times 100 \quad 3$$

Where W_1 = weight of the sample before heating (g)

W_2 = weight of the sample after heating (g)

Moisture content

The percentage moisture content was found by weighing 1g of rice husk and placed in an oven; the samples were dried for 2 hour at 105°C to get rid of the moisture. The resulting sample were removed, covered and allowed to cool. Triplicate experiment was conducted and the weight loss difference was recorded as moisture content, and the percentage of the moisture content was calculated from the data recorded.

The moisture content was calculated with the formula below,

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad 4$$

Where W_1 = Weight of sample before drying (g)

W_2 = Weight of sample after drying (g)

2.2.9 Water absorption capacity

The water absorption capacity was carried out following the ASTM C67-07 method. 10ml of water was measured with a burette and poured into a beaker and 1g of silica produced from both rice husk and cassava periderm was poured into the different beakers respectively. They were allowed to settle and left for 40 minutes. After the scheduled time, the mixture was filtered and the filtrate measured and recorded.

The water absorption capacity was calculated with the formula below:

$$\text{Water absorption capacity} = \frac{V_1 - V_2}{V_2} \times 100 \quad 5$$

Where V_1 = Initial volume of water (ml)

V_2 = final volume of water (ml)

3. Results and Discussion

3.1 Effect of Time on Silica Yield

Figure 4 and tables (1 and 2) show the effect of time on silica yield. The yield increased with increase in time of leaching. The silica synthesized increased from 79.2%, 87.78%, 93.64%, 94.0% to 97.60% as the leaching time increased from 1hr, 1.5hrs, 2hrs, 2.5hrs to 3hrs. This is because of Si-O parts were liberated from the long polymeric chains (Si-O-C/ Si-O-Si) with time (Gorthy and Pudukottah, 2009). This Si-O is removed by alkali extraction and the rate of extraction increases with time. Its yield was

highest at 3hrs with 97.60 hrs while the least was 79.2% which occurred at time interval for 1hours.

From table 1, increase in time gave rise to increase in the mass of silica precipitated which in turn increases the amount of crystallized silica produced, 3.960, 4.389, 4.682, 4.700 and 4.880 at 1hr, 1.5hrs, 2hrs, 2.5hrs and 3hrs

respectively. The mass of residual ash after filtration decreased with time for RHA. It can also be deduced from the figure 5 and table 1 that RHA needs a less volume of acid to precipitate the sodium silicate solution into silica. This implies that increase in the volume of acid used leads to decrease in the precipitation of the sodium silicate solution.

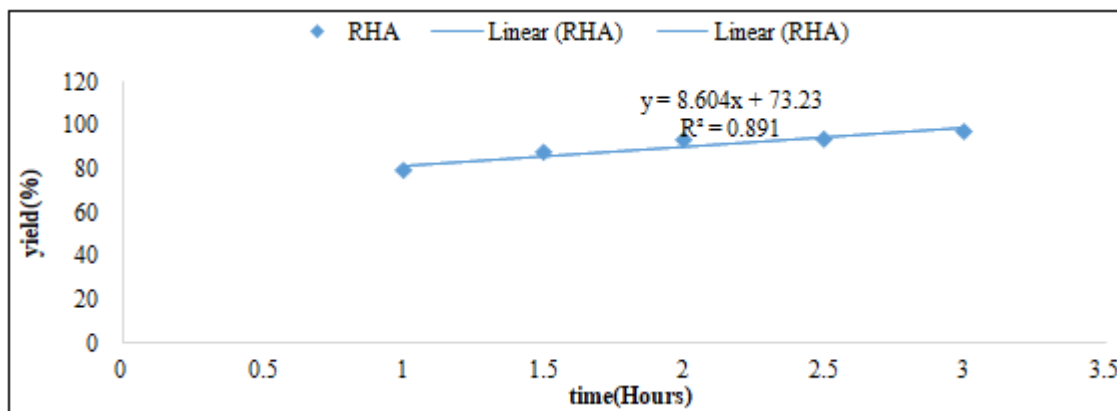


Figure 4: Silica Yield (%) against Time (hours) for RHA

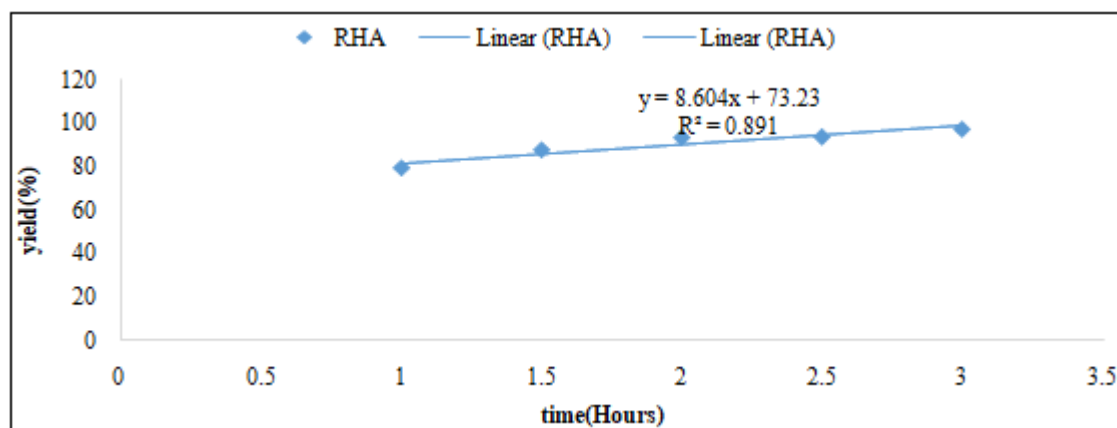


Figure 5: Graph of volume of acid (ml) against time (hrs)

Table 2: Percentage yield of silica for RHA at different time intervals

Time (hr)	Yield of Silica (%) from RHA
1.0	79.2
1.5	87.78
2.0	93.64
2.5	94.00
3.0	97.60

Table 1: Volume of acid, volume of filtrate, mass of residual ash and mass of precipitated and crystallized silica at different time intervals for RHA

Time (hr)	Volume of acid used during titration (ml)	Volume of filtrate (ml)	Mass of residual ash after filtration and drying (g)	Mass of silica precipitated (g)	Mass of crystallized silica after drying (g)
1.0	2.7	35	2.590	9.013	3.960
1.5	2.5	41	2.298	9.122	4.389
2.0	2.0	43	1.980	9.890	4.682
2.5	1.9	44	1.801	11.14	4.700
3.0	1.7	46	1.699	11.52	4.880

3.2 Proximate Analysis

The average moisture content and average ash content of rice husk were calculated to be 8.7 and 23.47 respectively (table 3 and 4)

Table 3: Moisture content of rice husk

Run	Sample Weight (g)			Moisture Content (%)	Average Moisture Content (%)
	W1	W2	(W1-W2)/W1		
1	77.6	70.5	0.0915	9.15	8.73
2	76.9	70.7	0.0806	8.06	
3	74.5	67.8	0.0899	8.98	

Table 4: Ash content of rice husk

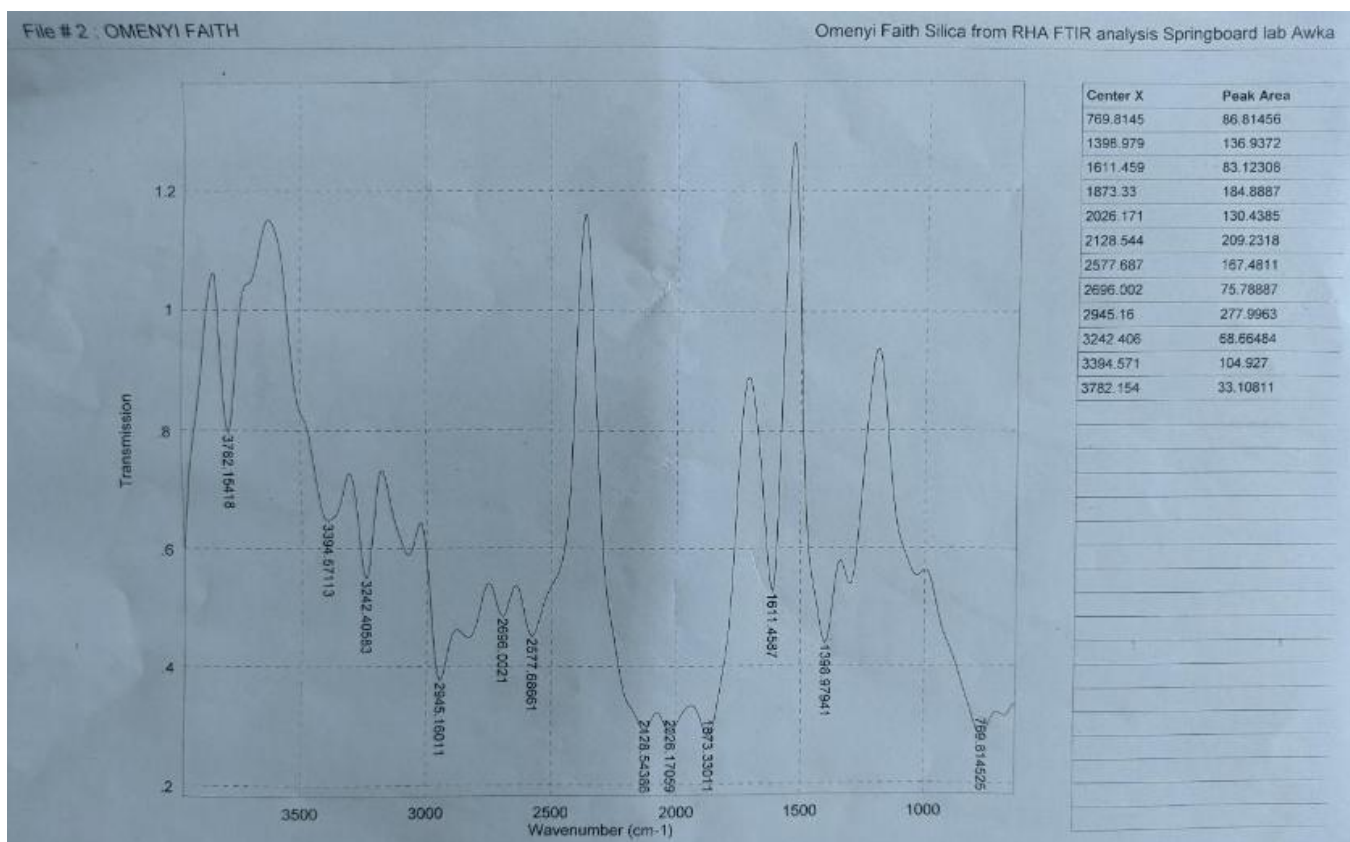
Run	Sample Weight (g)			Ash Content (%)	Average Ash Content (%)
	W1	W2	W1/W1		
1	23.1	5.5	0.2380	23.80	23.47
2	22.9	5.4	0.2358	23.58	
3	23.0	5.3	0.2304	23.04	

3.3 Water Absorption Capacity

The initial volume of water, V_1 , was 10ml and the final volume of water after filtration, V_2 , is 8.3ml. The water absorption capacity for RHA was calculated to be 20.45%. RHA showed a high absorption capacity and this is attributed to high porosity of the RHA derived silica.

3.4 Interpretation of FTIR Spectrum

Figure 6 shows the FTIR spectra of silica from rice husk ash. It showed 12 absorption bands indicating that it is not a simple compound but a complex compound. Six peaks are located at single bond region reflecting six single bond compounds, two triple bond compounds in the triple bond region, two absorption bands signifying two compounds, in the double bond region and two absorption bands (two compounds) fall in the fingerprint region. It has a very strong bonds at 1873.33cm^{-1} and sharp peaks at 3782.15cm^{-1} , 1611.45cm^{-1} and 3242.40cm^{-1} indicating the presence of O-H stretching, C=C stretching and O-H sym-stretching respectively. The -OH groups may include absorbed water, aliphatic primary and secondary alcohols found in cellulose and hemicellulose extractives.

**Figure 6:** FTIR Spectra of silica from rice husk

4. Conclusion

Experiments were conducted to investigate the potential of rice husk as raw materials for the production of silica (Desiccant). Time is found to be an important factor in the extraction of silica from rice husk ash as it affected the yield of crystalline silica. Experimental results showed that it is feasible to produce silica from rice husk ash by acid pretreatment, leaching and titration using sulphuric acid. It was observed that the yield of silica from RHA is high as confirmed from literature (Vaibhav et al., 2015). The mass of residual ash gotten from the reaction of the ashes with NaOH was observed to reduce with an increase in time. The absorption capacity of the RSA derived silica is 20.45% which is high enough and made it a good material for desiccants.

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