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Green Chemistry Preparation and Characterization of Rice Husk Derived Silica Gel in Kenya

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Abstract: Rice is a grass seed from *Oryza glaberrima* species also known as the African rice. In Kenya, rice is mostly grown in Central (Mwea) and Nyanza (Ahero, West Kano, Migori and Kuria) areas. Milling rice produces rice husks as by-products which can be sources of valuable chemical products (silica gel, sodium silicate). In trials to produce silica gel from rice husks, rice husks were charred in a combustion chamber (30 min) then ashed in a Muffle furnace (Advantec KL-420) at different temperatures. The ashes were then leached with distilled water/acids to remove metal oxides. Sixty grams (60 g) of the leached RHA (Rice Husk Ash) was mixed with 300 mL of 3 M NaOH solution in a Pyrex 500 mL beaker and boiled at 100 °C (1 h). The silica gel samples were characterized using several methods. Elemental analysis was done using TXRF (Total X-Ray Fluorescence), while FTIR (Fourier-Transform Infrared Spectroscopy) was used to obtain an infrared spectrum of absorption of the silica sample. Results of the analysis conform to local and international quality standards. The rice husks had an average moisture content of 7.07% and 1.00-2.00 mm diameter. And 1.74% of the rice husk had pore sizes of about 0.710 mm. The average ash content was 22.65%. At 600 °C ,leaching with water yielded 98.2% silica compared with 99.1% (H_2SO_4) and 96.9% (HCl). At 500 °C, leaching with HCl/H_2SO_4 causes a decrease. At 500 °C, the availability of SiO_2 is more for water leached samples. At 400 °C, water leaching gave 98.49% silica while HCl leaching was 97.85% silica and H_2SO_4 was 99.41%. Silica is a precursor of silica gel. Statistical analyses imply water leaching RHA instead of acid leaching at 500 °C will produce a significant amount of silica gel. The open burn samples produced equal or better SiO_2 (silica gel precursor) yields compared with the incineration samples. FTIR analysis of the silica gel sample compared well with adsorption peaks of silica gel in literature. XRD (X-Ray Diffraction) analysis produced a pattern consistent with other XRD patterns of silica gel published by other researchers.

Key words: Rice husk conversion, silica gel synthesis, green chemistry, ideal conditions.

1. Introduction

Rice is a grass seed from *Oryza sativa* (Asian rice) species or *Oryza glaberrima* species also known as the African rice. Rice is grown widely across the globe and is usually a staple food in many countries [1]. As a result, Kenya has embarked on an ambitious program to boost rice production.

Rice has an outer layer known as the rice husk (or hull). The rice husk is normally separated from the rice grains during the process of milling. About 20% of paddy weight is husk. Rice husks have been largely considered a waste product that has been often dumped or burned on landfills [2]. However, rice husks are nowadays considered as an economically viability product [3].

In Kenya, the production of rice has resulted in an increased production of rice husks which have been in many cases considered wastes [4]. Rice husk management in Kenya has not been viable and sustainable and has majorly been via open incineration which has led to environmental pollution. Further, there is a proposition by UNEP (United Nations Environment Programme) for countries to recover valuable materials from the waste stream so as to enhance waste to wealth conversion. So as to help in

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curtailing the danger that rice husks may subject humans to and control environmental pollution, it is important to explore ways to manage waste pollution as a result of disposed rice husks, considerable efforts are being made by many governments and other entities. Such considerable efforts would reduce the costs for waste disposal and would generate revenue from the sale of the recovered materials (chemical materials) and energy [5, 6].

Unfortunately, collection systems are inefficient and disposal systems are not environmentally friendly. The traditional methods for managing rice husk waste in Kenya include: land filling, incineration, open dumping and open air burning. Nevertheless, some of these methods have been found to be unsustainable due to limited land area for expansion, potential environmental risks and increased costs. This has shifted focus to research for more sustainable rice husk waste management strategies with growing interest in chemical treatment of wastes, interest in resource/energy recovery and production of chemical products such as silica gel to solid waste management [6, 7].

The alternative approach to synthesis of low cost solar grade silicon from plant biomass such as rice husk which has a significant source of high purity silica has only been explored by relatively few research groups [3]. However, the narrow and isolated experimental conditions of previous works and lack of an optimized process have limited the engineering application of such results. The lack of a commercially viable route for synthesis of bulk high purity silicon using amorphous silica from rice husk, therefore, provides a good motivation to conduct further research into the feasibility of this approach consequently the urge for the study at hand [8].

As a matter of fact, the focus nowadays is to design sustainable and least cost solid waste management systems considering the variety of management processes that can control environmental pollution. Such a model should serve as a solid waste decision support system taking into consideration both socio-economic and environmental considerations. This waste to wealth conversion will reduce poverty, unemployment and promote rural enterprises in rice growing areas.

Many tons of rice husk go to waste. Therefore, there is need to create value in rice husk waste by generating products for enhancing rural industrial development growth. To ensure development in a country, the needs of present generations should be met without compromising the ability of the future generations to meet their needs [6, 9]. This can be achieved by providing an amicable solution for integrating the environment in socio-economic planning and management by chemically processing rice husks for various chemical products. Increasing environmental concerns and the emphasis on material and energy recovery are gradually changing the orientation of solid waste management and planning in order to boost economic growth [10].

Studies have shown that rice husks consist of 32.24%-21.34% cellulose, 21.44% hemicelluloses lignin, 15.05% mineral ash and approximately 96.34% silica in its mineral ash. This may, however, vary widely, depending on the source of RH and processing conditions. Oxides of Al, Mn, Fe, Ca, K, Mg, Na and P occur in variable proportions in RHA (Rice Husk Ash) in addition to silica. Carbon, sulphates and traces of other elemental oxide have also been reported in RHA [11].

Silica (SiO_2) is one of the valuable inorganic multipurpose chemical compounds [11, 12]. It can exist in gel, crystalline and amorphous forms. It is the most abundant material on the earth's crust. However, manufacture of pure silica is energy intensive. A variety of industrial process, involving conventional raw materials, require high furnace temperatures (more than 700 °C) [12]. In this study, a chemical process is described which uses raw material RHA for extraction of silica (silica gel precursor). RHA is one of the most silica rich raw materials containing about

90%-98% silica (after complete combustion) among the family of other agro-wastes

Adam and Iqbal [13] in their work, produced sodium silicate by reacting RHA and aqueous sodium hydroxide, in open and closed reaction systems. The studied process variables were time, temperature of reaction and composition of the reaction mixture (expressed in terms of molar ratios $NaOH/SiO_2$ and H_2O/SiO_2). About 90% silica conversion contained in the RHA into sodium silicate was achieved in closed system at 200 °C. The results showed that sodium silicate production from RHA can generate aggregate value to this residue.

Adam, Iqbal [13] and Kalapathy, et al. [14] studied RHA, a waste product of the rice industry and its husk. The method based on alkaline extraction followed by acid precipitation was developed to produce pure silica xerogels from RHA, with minimal mineral contaminants. The silica gels produced were heated to 80 °C for 12 h to obtain xerogels. Silica and mineral contents of xerogels were determined by EDX (Energy Dispersive X-Ray) and ICP (Inductively-Coupled Plasma) emission spectrometers, respectively. Xerogels produced from RHA had 93% silica and 2.6% moisture. The major impurities of silica produced from RHA at an extraction yield of 91% were Na, K and Ca. Acid washing prior to extraction resulted in silica with a lower concentration of Ca (< 200 ppm). However, final water washing of the xerogel was more reactive in producing silica with lower overall mineral content (Na < 200 ppm and K < 400 ppm). XRD patterns revealed the amorphous nature of silica xerogel.

The purpose of this study is to evaluate the best conditions for synthesis of sodium silicate (silica gel precursor) and subsequent silica gel and characterize the obtained silica gel.

2. Methods

2.1 Sample Collection and Pre-treatment

About 20 kg of rice husks were collected from Mwea rice millers (Kirinyaga County), Euro rice Millers (Kirinyaga County) and Nice rice millers (Kirinyaga County) in Kenya.

There were two types of rice husks samples prepared:

(a) incinerated samples at different temperatures (Inc400, Inc500, Inc600)

(b) openly burnt samples (OPB1, OPB2 and OPB3)

Samples collected were divided into two portions and leached with water and acid.

The samples were washed for 10 min with tap water to remove soils adhering to the husks and rinsed with distilled water. Drying of the samples was done initially at room temperature and subsequently in the air oven (Memmert UF110) at 110 °C for 24 h. The sample was divided into three equal portions, then charred in a combustion chamber for 30 min to remove carbonaceous matter and subsequently thermally treated in an electric Muffle furnace (Advantec KL-420) at 400 °C, 500 °C and 600 °C for 30 min.

The thermal treated samples were divided into two groups then leached with either 0.5 M HCl or 0.5 M H_2SO_4 (Analar grade). This was done by mixing the sample and acid at 60 °C for 30 min with constant stirring. The acid solution was removed by washing with water, rinsing with distilled water until free from acids. The leached samples were then filtered and then air-dried for 24 h at room temperature. Final drying was then done for 2 h at 110 °C in the oven.

Another three equal portions of thermally treated samples (400 °C, 500 °C and 600 °C) were also leached with distilled water to remove inorganic salts.

2.2 Preparation of Silica Gel

Sixty grams (60 g) of the leached RHA samples were each mixed with 300 mL of 3 M NaOH solution in a Pyrex 500 mL beaker. The solution was boiled at 100 °C for 1 h with constant mixing. To 25 mL of this resulting solution, 2 mL of HCl (37%) was added and heated in a water bath for 30 min.

2.3 Analysis of the Samples

The samples were initially analyzed for silica content using a TXRF (Total X-Ray Fluorescence) S1 TITAN. Since XRF (X-Ray Fluorescence) is a non-destructive technique, it is ideal for analyzing in-process production samples.

It offers a comprehensive, unique and non-destructive characterization of crystalline samples. It also enables qualitative and quantitative phase analysis, polymorphism investigation, the determination of crystallinity, all the way through to structure investigation. The results were thereafter compared to existing marker samples.

The silica gel samples were also characterized by scanning with FTIR (Fourier-Transform Infrared Spectroscopy) IRTracer-100 (SHIMADZU C103-E091A). The samples were pressed in a hydraulic press between smooth stainless steel dies (at 10 t pressure for 2 min) to give a clear KBr disk. The samples were then read using the FTIR spectrophotometer IRTracer-100.

The Silica gel sample was further characterized an XRD (X-Ray Diffraction, D2 PHASER.). It has holders for small sample amounts, low-absorbing and weakly diffracting samples, for filters, for environment-sensitive samples and for examining materials that tend to show a preferred orientation.

3. Results and Discussion

Determination of the % Moisture Content, Particle Size and Ash Content of the Rice Husks.

The preliminary trials are for determining the best conditions for producing sodium silicate (a precursor for silica gel). The following initial trials were done in the food science laboratory using rice husks from Mwea area, Kirinyaga County. The analytical methods were all from the AOAC (Association of Official Analytical Chemists).

3.1 % Moisture Content Determination

Three grams (3.0 g) of rice husk sample was weighed in triplicate into numbered moisture dishes in an analytical electronic balance (model: LibrorAEG-220, NO: D400402854, capacity: 220 g, readability: 0.1 g). The moisture dishes had previously been cleaned, dried in a hot air oven and cooled in a desiccator.

The weighed samples were then dried in an oven (model: Memmert UF 110) at 105 °C for 30 min.

The samples were thereafter cooled in a desiccator and weighed again. This was repeated every 30 min until loss in weight did not exceed 0.1% per drying period. The following results were recorded.

Table 1 shows the average moisture content of the rice husks was an average moisture of 7.07 % after 3½ h. Doorvasan, et al. [15] have reported a moisture range of 8.0%-9.0%.

3.2 Rice Husks Particle Size distribution

Twelve grams (12 g) of the collected rice husks was air dried for 24 h. Ten point eight grams (10.8 g) of sample was placed on the top test sieve of a vibratory feeder Test Sieve BS410/1986 and shaken for 20 min. The vibratory feed is a high speed assembly machine designed and manufactured by VSI Automation Company. It is a stainless steel mesh test sieve. It has five test sieves of 2.00, 1.00, 0.71, 0.50 and 0.25 mm. The five portions trapped in each mesh were weighed. The experiment was repeated two times and the average results tabulated are shown.

From the results in Table 2, it can be observed that most of the sampled rice husks had diameters ranging from 1.00-2.00 mm. Only about 1.74% of the rice husk had pore sizes of about 0.710 mm. Fig. 1 shows the vibratory feeder used to measure the distribution. Studies done have established the pore sizes of rice husks as compared to cement and found that most were over 3.0 mm as shown below in Table 3.

Table 1 Percentage moisture of the samples.

Sample No.	Wt. of dish/sample (g)	Wt. of dish/sample (g) (after 3½ h drying)	% Moisture
I	9.3786	9.1665	7.07
II	7.5596	7.3476	7.06
III	7.6612	7.4662	5.17

Fig. 1 Vibratory feeder. Test Sieve BS410/1986.

Table 2 Percentages of rice husks with different diameters.

Aperture size (mm)	Mass (g)	% Rice husks
2.00	7.9563	73.65
1.00	2.5639	23.73
0.71	0.1877	1.74
0.50	0.0323	0.30
0.25	0.0080	0.07

Particle size distribution graphs showed average diameter of original RHA was about 60.54 µm.

3.3 Determination of Ash Content in Mwea Rice Husks

Twenty grams (20 g) of the collected air dried rice husks were oven dried for 24 h at 110 °C. The samples were left to cool in a desiccator. Eighteen grams (18.0 g) of sample was weighed in triplicate and put in numbered crucibles. The weighed samples were charred in a hot plate in the fume cupboard until no visible smoke was present. The charred samples were placed in a heat resistant mat and transferred to an electronic Muffle furnace (Advantec KL-420). They were then heated at 600 °C for 3 h turning them greyish white. Cooling of the crucibles was done in a desiccator to prevent them from absorbing moisture from the atmosphere. The thermally treated samples were weighed after cooling and the results are as shown.

From Table 4, it can be observed rice husk had an average ash content of 22.65%. The major components of rice husk are organic materials such as hemicellulose, cellulose and lignin totaling about 75%-90% and the remaining is ash content of 17%-20% [16].

Under controlled combustion of rice husks, RHA containing almost pure silica is obtained. The metallic impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) that influence the purity and color of the silica could be eliminated by pre-treatments with hydrochloric acid, sulfuric acid or nitric acid prior to combustion [16, 17]. Strong acid digestion is effective in breaking down rice husks into small fragments thus exposing more organic components. That is, their pyrolysis can be accelerated more rapidly due to the increase of their exposed surface areas.

It has been reported that the function of acid pre-treatment in rice husk contains two aspects. One is to accelerate the hydrolysis and degradation of lignocellulose to monosaccharides by removing

Table 3 Particle size distribution and specific gravity of RHA and OPC (Ordinary Portland Cement).

	Original RHA	Passed 300µm sieve RHA	Ground RHA	Cement OPC
Specific surface (cm^2/g)	1,753.8	4538.88	14,342.53	19,027.98
Diameter at 10% (µm)	26.77	26.34	2.2	1.89
Diameter at 50% (µm)	60.54	59.52	13.43	14.44
Diameter at 90% (µm)	102.65	97.81	35.65	36.68

Source: Hadipramana, et al. [24].

Table 4 Percentage ash content.

Wt. of sample (g)	Wt. of ash (g)	% ash Content
3.0043	0.7040	23.43
3.0005	0.6712	22.36
3.0000	0.6652	22.17

hydrogen bonds or extractives. After polysaccharides are converted to monosaccharides, metal oxides bounding to organic matters are exposed and have a chelate reaction with acids, thus released to acid leaching solutions. The forms of metal impurities in rice husk normally may be classified into three categories: the water soluble part, the acid-leachable part and the residual [17, 18].

Ashes are black with gray particles as a result of different steps of carbon combustion during rice husk burning. The burning of rice husks results in formation of silica ash, which varies from gray to black depending on inorganic impurities.

3.4 Leaching after Thermal Treatment and Elemental Content Analysis

The rice husks were washed for 10 min with tap water to remove soils adhering to the husks and rinsed with distilled water. Drying of the sample was done initially at room temperature and subsequently at an air oven (Memmert UF110) at 110 °C for 24 h. The sample was divided into three equal portions, then charred in a combustion chamber for 30 min to remove carbonaceous matter and subsequently thermally treated in an electronic Muffle furnace (Advantec KL-420) at 400 °C, 500 °C and 600 °C for 30 min.

Each thermal treated sample was then leached with 0.5 M HCl and 0.5 M H_2SO_4 acids. This was done by mixing the sample and acid at 60 °C for 30 min with constant stirring. The acid solution was removed by washing with water, rinsing with distilled water until free from acids. Litmus paper was used to ensure the acidity was removed. The leached samples were then filtered and then air-dried for 24 h at room temperature. Final drying was then done for 2 h at 110 °C in an oven (Memmert UF110). The un-leached are samples leached with water instead of acid.

Table 5 shows that at 600 °C the leaching with water yielded 98.2% silica compared with 99.1% (H_2SO_4) and 96.9% (HCl).

Table 6 indicates that at 500 °C leaching with HCl causes a decrease of 2% while H_2SO_4 causes a decrease of 1%. At this temperature, the availability of SiO_4 is more when RHA is un-leached.

Table 7 indicates at 400 °C, water leaching yielded 98.49% silica. HCl leaching produced 97.85% silica while H_2SO_4 was 99.41%.

F value (26.878) is significantly more than *F* critical value (5.4090). Analysis of Fig. 2 implies the temperature at which the rice husk waste is burnt has a significant relation with the availability of silica. The availability of silica at 600 °C is not very significantly different for both water/acid leached RHAs. At 500 °C, the leaching with water or acid produces the same amount of silica. At 400 °C, the leaching with acid produces more silica content.

Table 5 % Elemental content of acid leached RHA (600 °C).

% Element	Un-leached	HCl-leached	H_2SO_4-leached
SiO_2	***98.191 ± 1.168***	96.926 ± ***1.215***	99.089 ± ***0.896***
MgO	*	2.040 ± ***1.954***	*
Al_2O_3	*	0.016 ± ***0.343***	*
P_2O_5	0.286 ± ***0.050***	0.148 ± ***0.038***	0.113 ± ***0.028***
K_2O	1.617 ± ***0.022***	0.444 ± ***0.020***	0.423 ± ***0.014***
CaO	0.532 ± ***0.020***	0.134 ± ***0.013***	0.127 ± ***0.010***
Mn	0.103 ± ***0.012***	0.061 ± ***0.010***	0.051 ± ***0.009***
Fe	0.158 ± ***0.011***	0.178 ± ***0.011***	0.151 ± ***0.010***
Zn	0.010 ± ***0.002***	0.007 ± ***0.002***	0.005 ± ***0.002***
Cl	*	*	*
Na_2O	*	*	*
S	*	*	*

*not detectable

Table 6 **% Elemental content of acid leached RHA (500 °C).**

% Element	Un-leached	HCl-leached	H_2SO_4-leached
SiO_2	98.041 ± 1.406	95.862 ± 1.126	97.099 ± 1.175
MgO	*	*	*
Al_2O_3	*	0.243 ± 0.387	*
P_2O_5	0.277 ± 0.049	0.104 ± 0.387	*
K_2O	0.592 ± 0.022	0.142 ± 0.015	0.437 ± 0.019
CaO	0.692 ± 0.022	0.251 ± 0.017	0.088 ± 0.012
Mn	0.123 ± 0.013	0.086 ± 0.008	0.044 ± 0.008
Fe	0.188 ± 0.012	0.120 ± 0.009	0.204 ± 0.012
Zn	0.013 ± 0.002	0.007 ± 0.002	0.006 ± 0.002
Cl	0.028 ± 0.027	0.028 ± 0.026	0.029 ± 0.027
Na_2O	*	*	*
S	*	*	*

*not detectable.

Table 7 **% Elemental content of post acid leached RHA (400 °C).**

% Element	Un-leached	HCl-leached	H_2SO_4-leached
SiO_2	98.486 ± 1.149	97.845 ± 1.148	99.409 ± 1.143
MgO	*	*	*
Al_2O_3	*	0.243 ± 0.387	*
P_2O_5	0.222 ± 0.045	0.118 ± 0.027	*
K_2O	0.402 ± 0.019	0.188 ± 0.015	0.193 ± 0.015
CaO	0.485 ± 0.019	0.018 ± 0.011	0.029 ± 0.011
Mn	0.094 ± 0.011	0.086 ± 0.008	0.044 ± 0.008
Fe	0.198 ± 0.012	0.137 ± 0.010	0.173 ± 0.011
Zn	0.008 ± 0.002	0.003 ± 0.001	0.005 ± 0.002
Cl	0.037 ± 0.026	*	*
Na_2O	*	*	*
S	*	*	*

*not detectable.

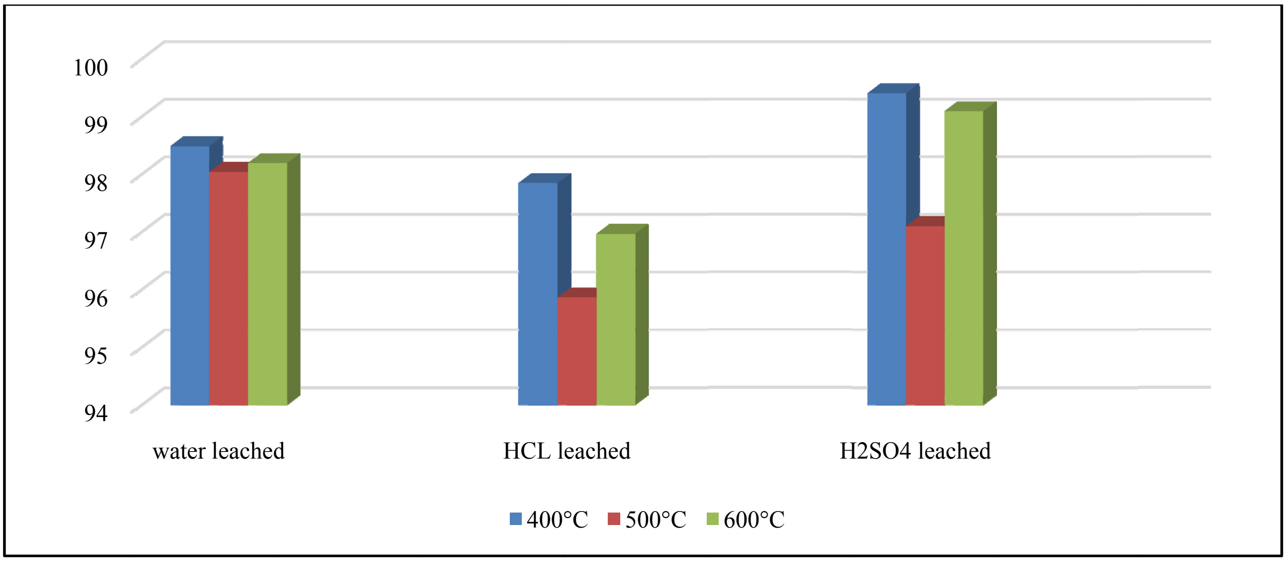

Fig. 2 **% SiO_2 content after different treatments.**

ANOVA: Single Factor						
Summary						
Groups	Count	Sum	Average	Variance		
Column 1	3	1,500	500	10,000		
Column 2	3	294.718	98.23933	0.051258		
Column 3	0	0				
Column 4	3	295.589	98.52967	1.559441		
ANOVA						
Source of variation	SS	df	MS	*F*	*p*-value	*F* crit
Between groups	322,590.1	3	107,530	26.87818	0.001643	5.409451
Within groups	20,003.22	5	4,000.644			
Total	342,593.4	8				

3.5 Silica Gel Preparation

The collected rice husks were washed thoroughly with water to remove the adhering soil and other contaminants present in them and then dried in the sunlight for 24 h. The rice husks were then charred in a combustion chamber for 30 min to remove carbonaceous matter and subsequently ashed at an electronic Muffle furnace (Advantec KL-420) at 400 °C for 30 min.

The sample was then leached with distilled water. Sixty grams (60 g) of leached RHA was mixed with 300 mL of 3 M NaOH solution in a Pyrex 500 mL beaker. The solution was boiled at 100 °C for 1 h with constant mixing. Twenty-five milliliters (25 mL) of the alkali sodium silicate solution (prepared from RHA and NaOH) was measured in a 500 mL beaker. Fifteen milliliters (15 mL) of 37% HCl acid was then added to the mixture and heated for 10 min in a hot water bath (WEB 22ND, DIN/2876-3). Another 5 mL 37% HCl and 15 mL of distilled water was added and the mixture continuously stirred. The mixture was then decanted and a further 5 mL of 37% HCl added while also adding 10 mL distilled water to remove any soluble salt from the silica gel.

FTIS IRTracer-100 (SHIMADZU C103-E091A) was used to scan for the absorption peaks of the dried solid silica gel samples. Two grams (2 g) of sample was pressed in a hydraulic press between smooth stainless steel dies (at 10 t pressure for 2 min) to give a clear KBr disk. The sample was then scanned using the FTIR spectrophotometer IRTracer-100. The results are as displayed below. The absorption peaks 1,101.37 cm^{-1}, 1,081.12 cm^{-1}, 948 cm^{-1} and 799.51 cm^{-1} are a marker for the presence of silica gel.

Fig. 3 shows the FTIR scan results of the silica gel sample. It has peaks at 3,441, 1,101, 1,081, 948 and 799.5 cm^{-1} which is similar to other samples of silica gel in literature (Fig. 4). The peaks are associated with 3,441 (O-H hydroxyl group, H-bonded, OH stretch), 1,101(Si-O-C), 1,081(Si-O-Si), 799 (silicate ion).

The silica gel (Fig. 3) sample compares well with Fig. 4 scanned silica gel.

Both have 1,101 cm^{-1}, 1,081 cm^{-1}, 948 cm^{-1} and 799 cm^{-1} peaks indicating the produced silica gel is characteristically the same.

3.6 Analysis of Silica Gel Using XRF Spectroscopy

The silica gel samples were analyzed using an XRF S1 TITAN spectroscopic meter. The results obtained are displayed in Table 8. The results are extracted from Table 8 and displayed in Figs. 5-9.

The open burn treatments produced equal or better SiO_2 yields compared with the incineration samples as shown in Fig. 5. This means farmers burning their rice husk wastes in the field and using the ashes to produce silica gel will get very good yields.

According to Pinheiro, S., et al.[20], the amorphous silica contained in the RHA is solubilized when treated with a sodium hydroxide solution that has a pH value greater than 10. When solubilized, the silica is in the monomeric form of silicic acid. When sodium

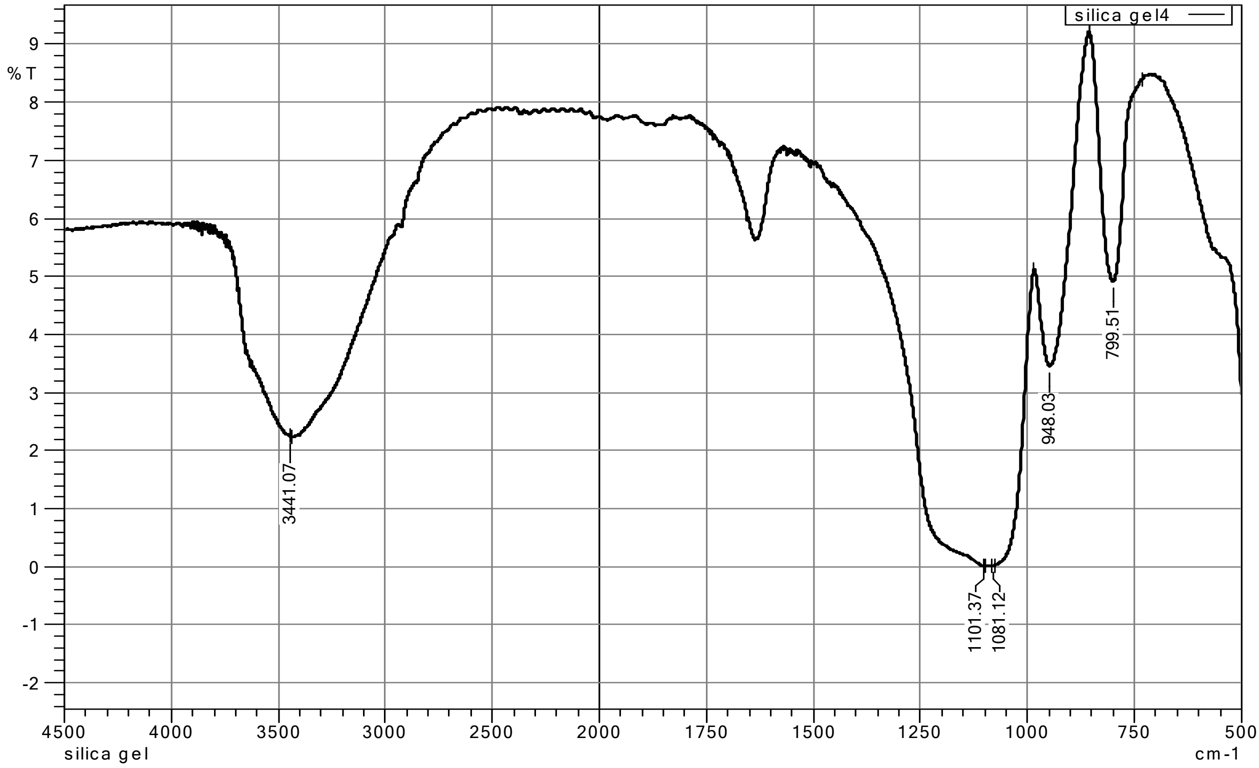

Fig. 3 FTIR spectra of the silica gel sample.

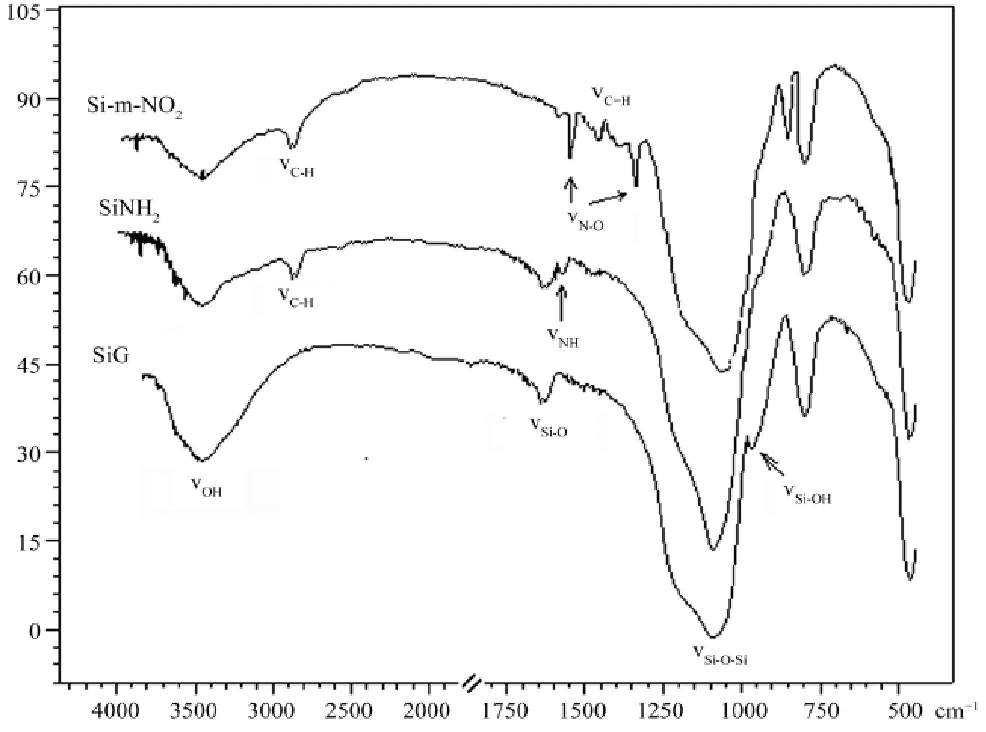

Fig. 4 FT-IR spectra of silica gel (SiG) and of the modified samples ($SiNH_2$) and (Si-m-NO_2) [19].
FT-IR spectra of silica gel (SiG) and of the modified samples ($SiNH_2$) and (Si-m-NO_2).

Table 8 Chemical analysis of silica gel samples results using XRF spectrophotometer.

	% SiO_2	% Cl	% K_2O	% CaO	% P_2O_5
INC 400 (1 M)	88.319 ± 1.089	10.868 ± 0.102	0.157 ± 0.016	0.050 ± 0.013	0.523 ± 0.064
INC 400 (2 M)	96.771 ± 1.088	1.437 ± 0.039	0.073 ± 0.013	0.027 ± 0.011	0.139 ± 0.035
INC 400 (3 M)	96.072 ± 1210	2.006 ± 0.055	0.064 ± 0.013	0.017 ± 0011	0.119 ± 0.049
INC 500 (1 M)	91.022 ± 0.974	8.224 ± 0.067	0.147 ± 0.015	0.072 ± 0.013	0.392 ± 0.055
INC 500 (2 M)	91.354 ± 0.849	7.712 ± 0.057	0.262 ± 0.014	0.092 ± 0.011	0.430 ± 0.047
INC 500 (3 M)	94.048 ± 1.104	5.345 ± 0.070	0.174 ± 0.016	0.071 ± 0.013	0.290 ± 0.053
INC 600 (1 M)	94.784 ± 0.952	2.149 ± 0.035	0.095 ± 0.013	0.021 ± 0.011	0.162 ± 0.043
INC 600 (3 M)	84.368 ± 0.924	14.625 ± 0.086	0.186 ± 0.016	0.033 ± 0.012	0.720 ± 0.064
OPB1 (1M)	95.916 ± 1.091	3.810 ± 0.057	0.030 ± 0.012	0.030 ± 0.011	0.138 ± 0.048
OPB1 (2 M)	88.941 ± 0.882	10.286 ± 0.061	0.071 ± 0.013	0.053 ± 0.012	0.540 ± 0.054
OPB1 (3 M)	82.210 ± 0.847	16.551 ± 0.077	0.141 ± 0.015	0.093 ± 0.013	0.089 ± 0.062
OPB2 (3 M)	96.804 ± 1.048	1.147 ± 0.035	0.030 ± 0.012	0.037 ± 0.011	0.146 ± 0.033
OPB3 (1 M)	96.903 ± 0.918	2.738 ± 0.034	0.095 ± 0.012	0.023 ± 0.010	0.183 ± 0.035
OPB3 (2 M)	88.695 ± 0.970	10.448 ± 0.078	0.129 ± 0.015	0.016 ± 0.012	0.558 ± 0.059
OPB3 (3 M)	93.512 ± 1.056	5.952 ± 0.067	0.090 ± 0.014	0.061 ± 0.012	0.291 ± 0.054
OPB3 2:3	93.400 ± 1.163	6.192 ± 0.084	0.042 ± 0.013	0.004 ± 0.011	0.285 ± 0.053
OPB3 2 M 2:2	94.131 ± 1.184	5.268 ± 0.080	0.151 ± 0.016	0.013 ± 0.012	0.247 ± 0.055

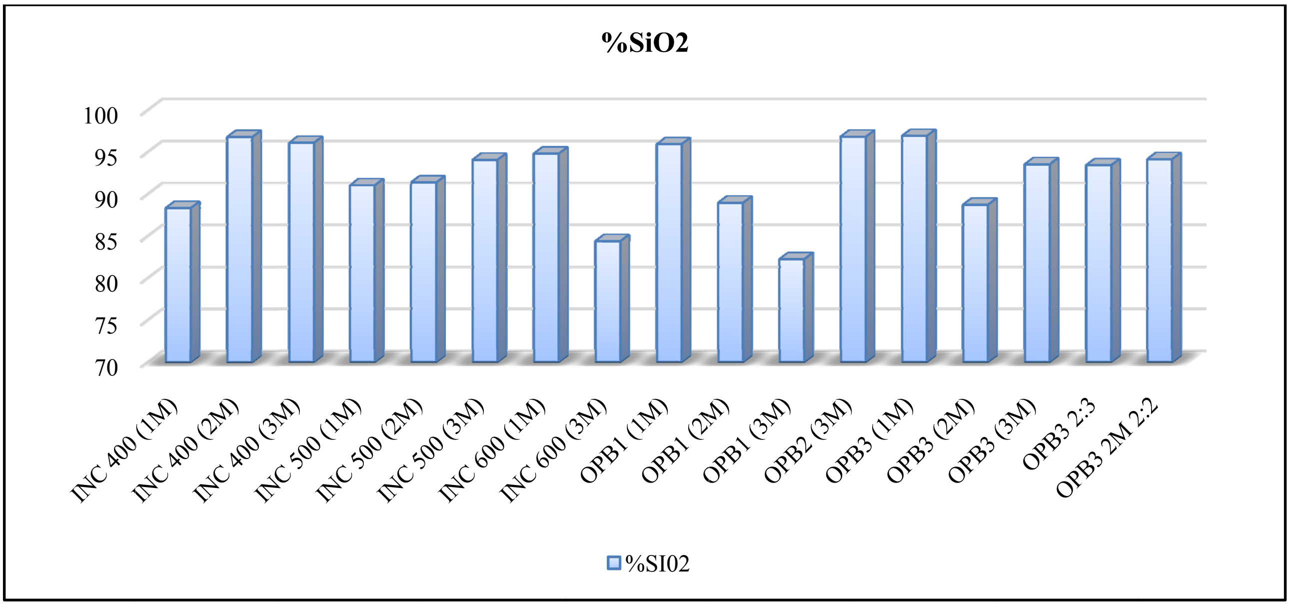

Fig. 5 % SiO_2 yield (silica gel) with different treatments.

silicate is acidified, a supersaturated solution of $Si(OH)_4$ monomers is formed.

Thereafter silica gel is formed during the gelation of a silicic acid solution through a polymerization process, which is divided into three phases: monomer polymerization to form particles; particle growth; and particle union in chains that extend throughout the solution, which increases the viscosity and forms a gel.

Fig. 6 indicates that the 3 M NaOH treated RH had the highest % Cl while the open burn samples had the least. This is probably the result of salt formation (NaCl) by the strong NaOH alkali solution.

Fig. 7 shows the open burn products lost the most % potassium during the silica gel formation process.

The RHA formed as a result of open burning had residual % K (reactive form) which could have been removed by NaOH.

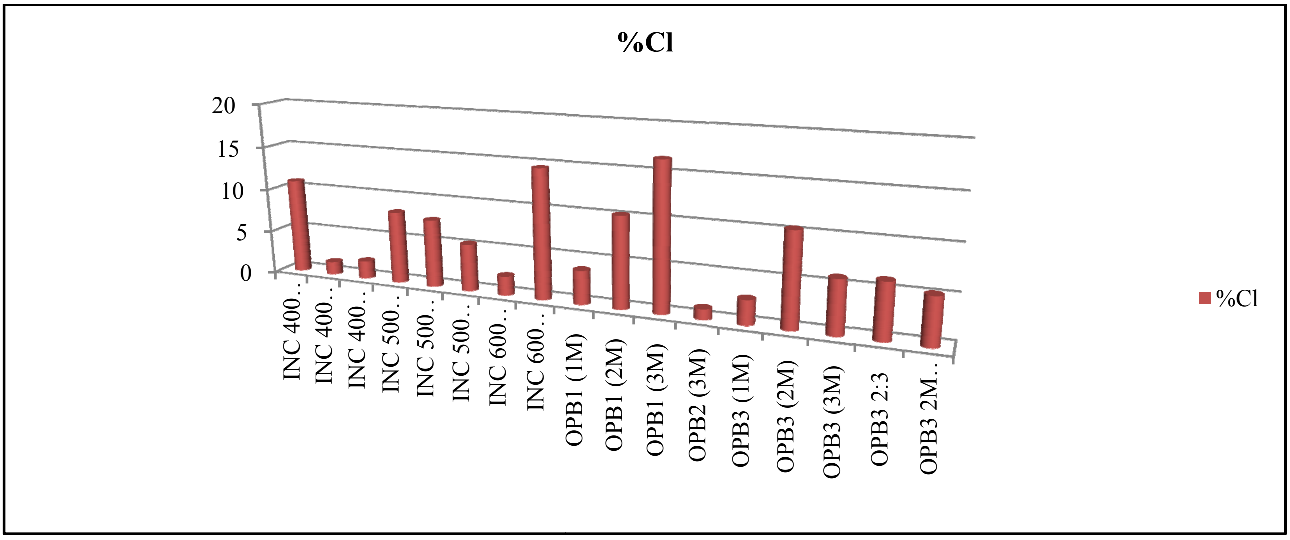

Fig. 6 % Cl content in (silica gel) with different treatments.

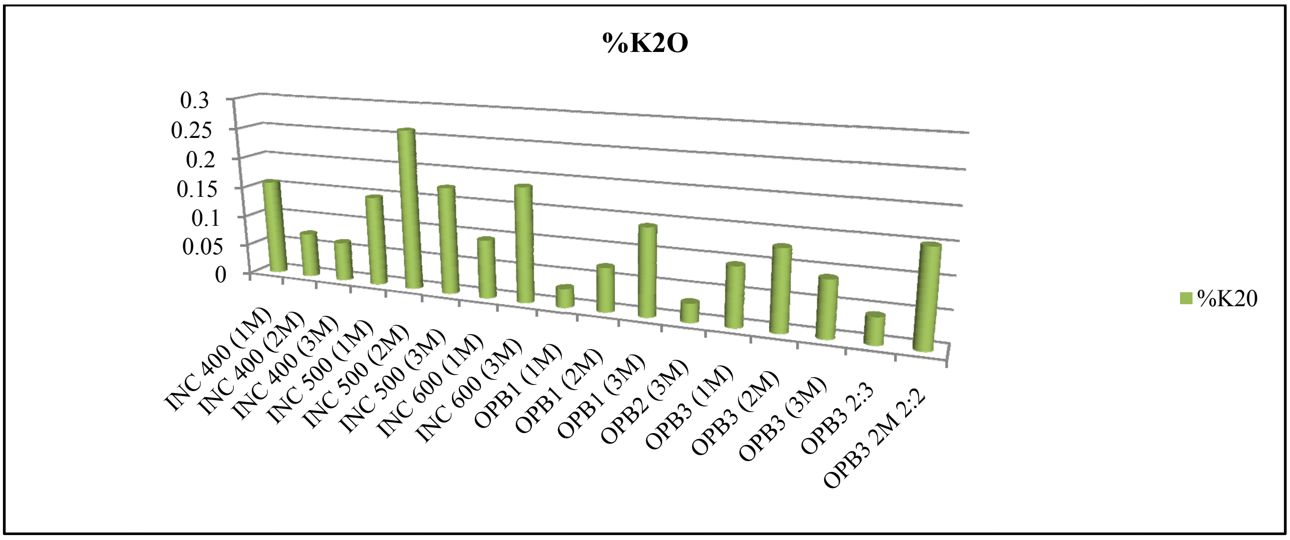

Fig. 7 % K_2O content in (silica gel) with different treatments.

Fig. 8 shows incineration at 500 °C had the most available % CaO while the open burn samples had the least. Studies have found % CaO content decreases with increasing temperatures, the surface areas of RHA obtained at 600 °C are much greater than that of 700 °C. It also can be seen that specific surface area has relationship with K content [21].

According to Fig. 9, incineration of rice husks at 600 °C resulted in most available % P_2O_5 content.

3.7 XRD Analysis of the Silica Gel

The silica gel sample was also analyzed with an XRD D2 PHASER. It offers a comprehensive, unique and non-destructive characterization of crystalline samples. It also enables qualitative and quantitative phase analysis, polymorphism investigation, the determination of crystallinity, all the way through to structure investigation.

It has holders for small sample amounts, low-absorbing and weakly diffracting samples, for filters, for environment-sensitive samples and for examining materials that tend to show a preferred orientation.

Fig. 10 shows peaks for Gupeite and Oldhamite as per library list of the XRD instrument.

The peak pattern at 20° is a marker for Silica gel according to Researchers [22]. Table 9 shows the main minerals of the Silica gel (1).

Fig. 11 shows a pie chart mineral content of Silica gel (1) sample as calculated from the XRD instrument.

Fig. 12 shows the diffraction pattern of the Silica gel (2). The clear peak is an indicator of Silica gel [20].

The high peak indicates it's a higher quality relativity. Table 10 shows a breakdown of its constituent minerals.

Figs. 12 and 13 show XRD scan results of two samples of silica gel. There is a clear peak pattern at 20° which according to literature is an indicator of silica gel [22]. The pattern is consistent with other XRD patterns of silica gel published by other researchers as shown in Fig. 14.

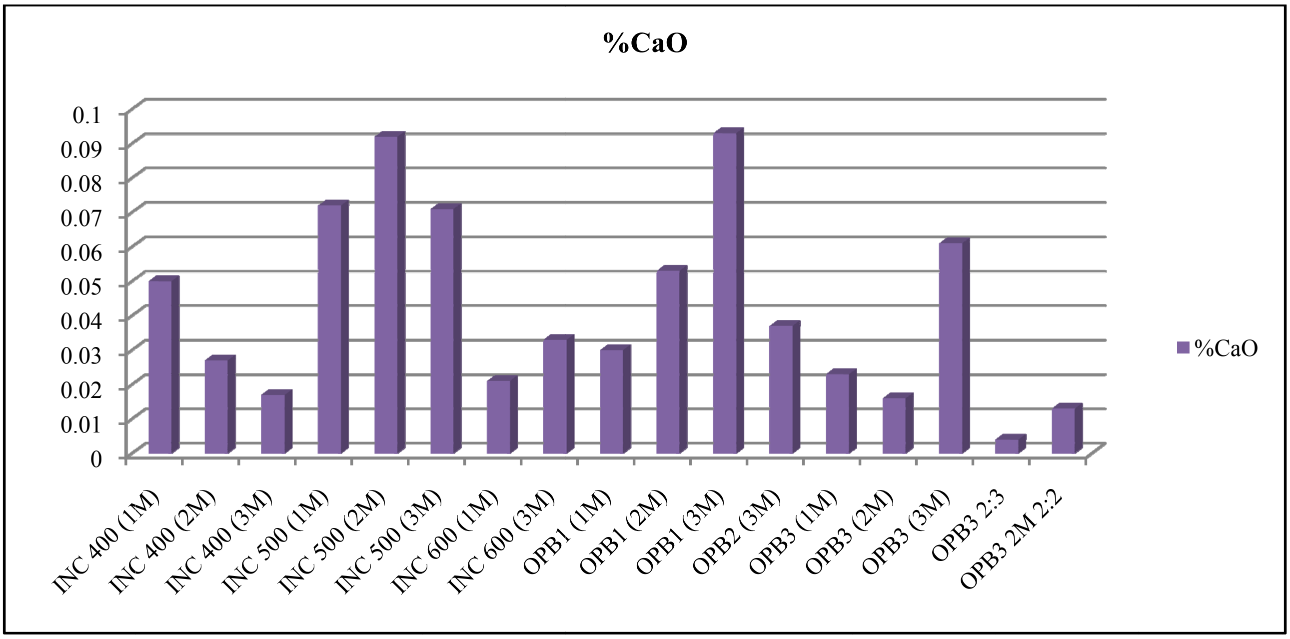

Fig. 8 **% CaO content in (silica gel) with different treatments.**

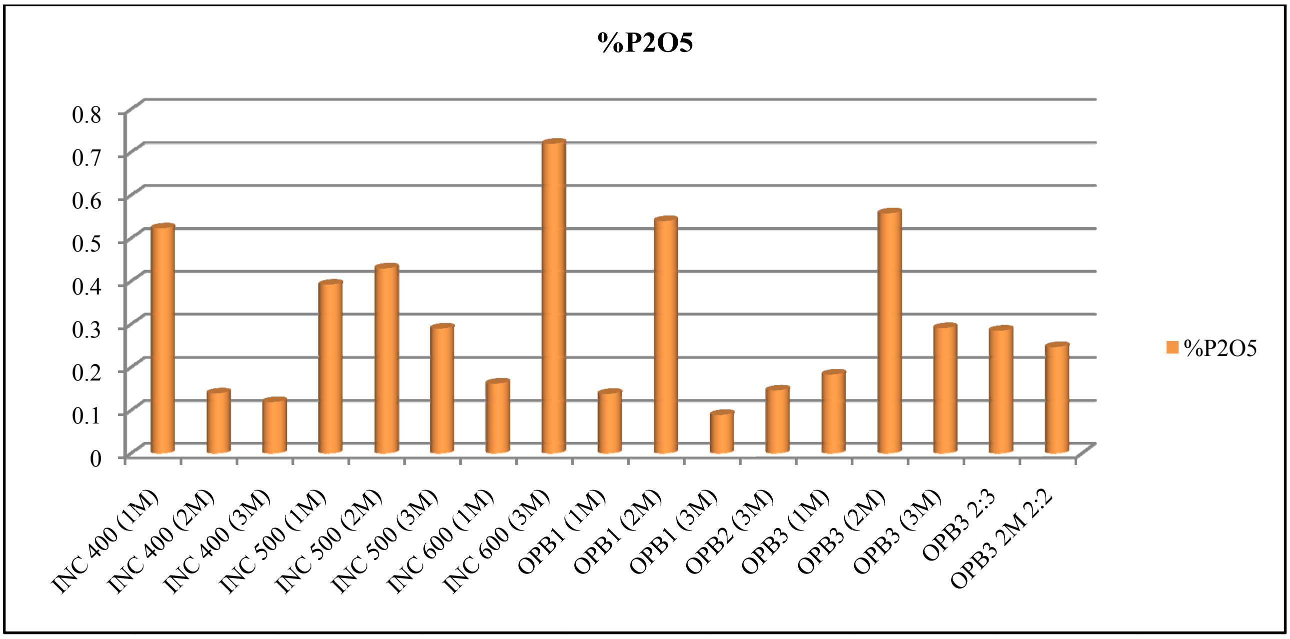

Fig. 9 **% P_2O_5 content in (silica gel) with different treatments.**

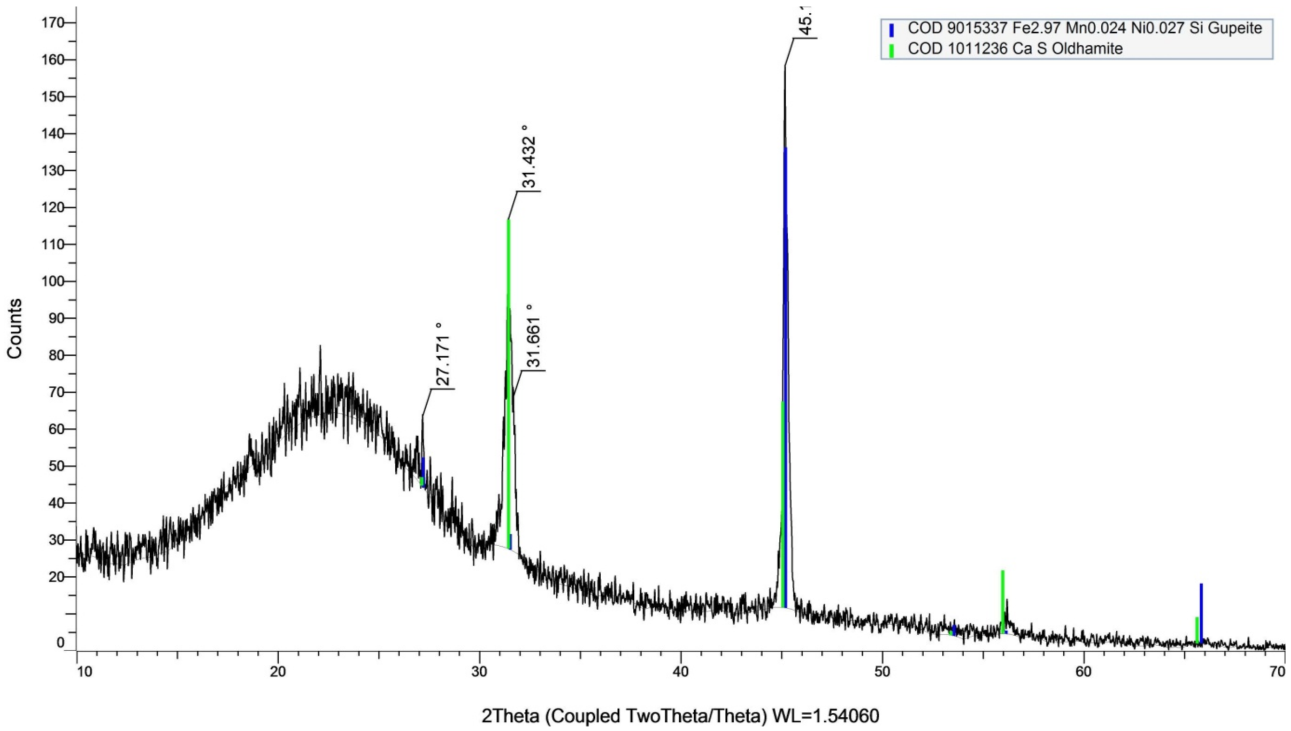

Fig. 10 XRD pattern of the silica gel (1) with mineral list produced from RHA.

Table 9 Silica gel (1) mineral list.

Index	Compound name	Formula	Pattern#	I/IcDB	S-Q
1	Gupeite	$Fe_{2.97}Mn_{0.024}Ni_{0.027}Si$	COD 9015337	11.500	43.6%
2	Oldhamite	CaS	COD 1011236	6.360	56.4%

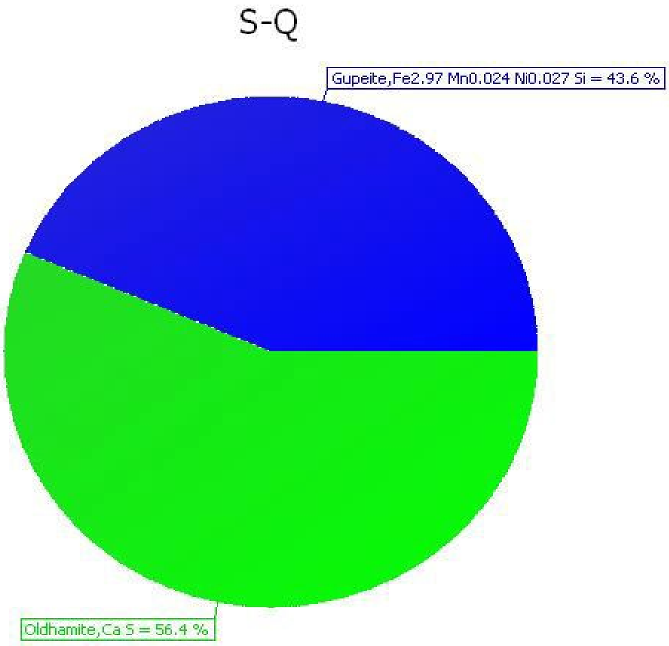

Fig. 11 XRD mineral list of the silica gel (1) with mineral list produced from RHA.

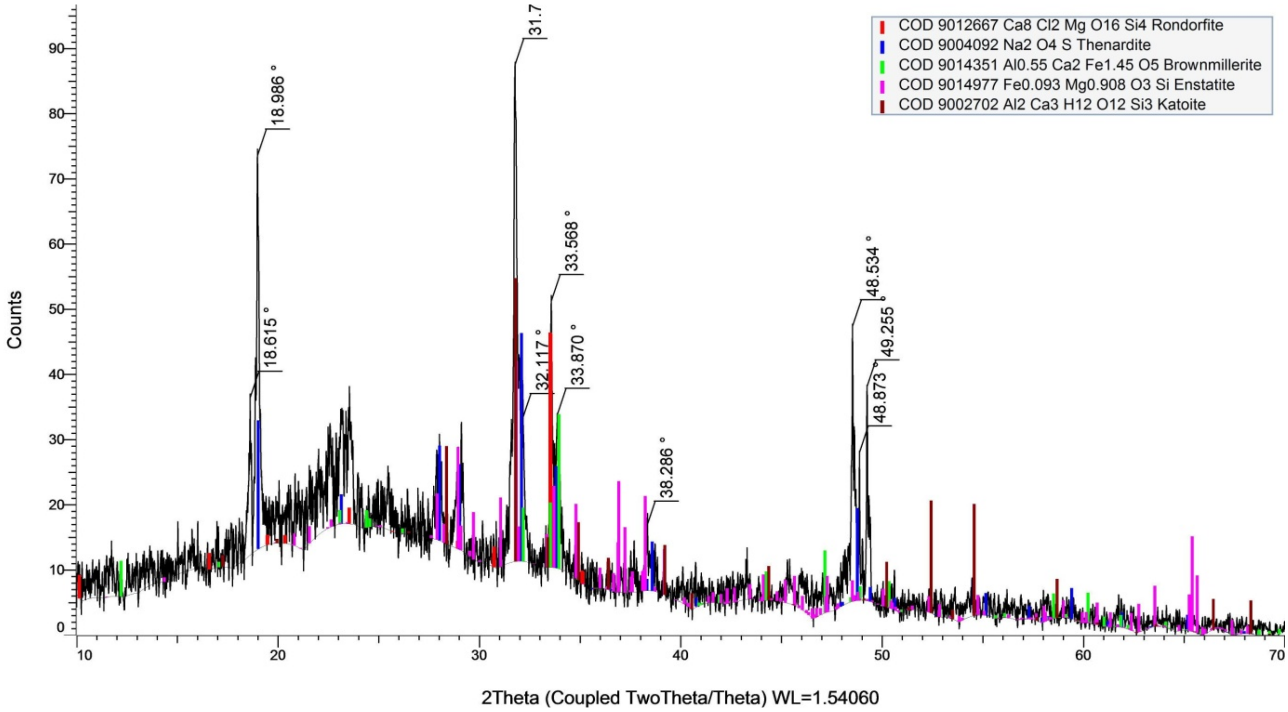

Fig. 12 XRD pattern spectra of silica gel (2) from RHA.

Table 10 Silica gel (2) mineral list.

Index	Compound name	Formula	Pattern#	I/IcDB	S-Q
2	Thenardite	Na_2O_4S	COD 9004092	1.770	11.6%
4	Enstatite	$Fe_{0.093}Mg_{0.908}O_3Si$	COD 9014977	0.550	32.9%
1	Rondorfite	$Ca_8Cl_2\ MgO_{16}Si_4$	COD 9012667	0.550	38.5%
5	Katoite	$Al_2Ca_3H_{12}O_{12}Si_3$	COD 9002702	2.130	12.0%
3	Brownmillerite	$Al_{0.55}Ca_2Fe_{1.45}O_5$	COD 9014351	2.820	4.9%

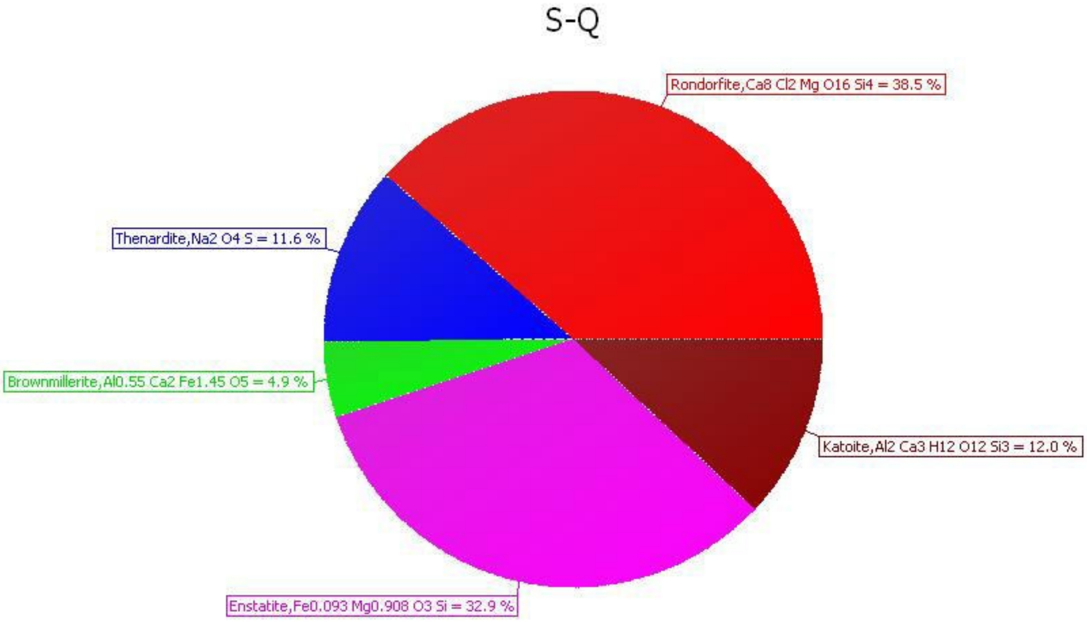

Fig. 13 XRD pattern list of silica gel (2) from RHA.

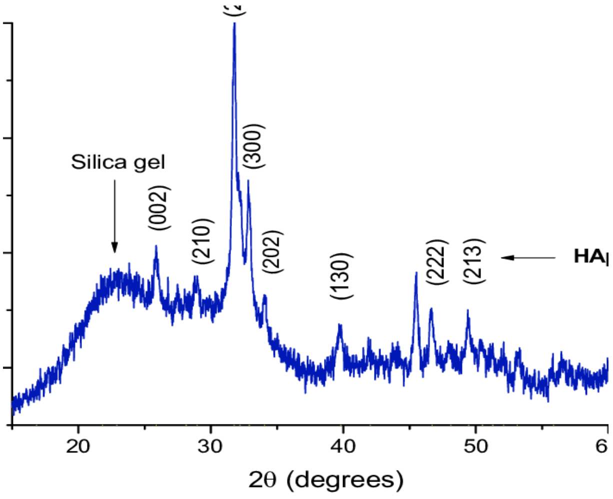

Fig. 14 XRD pattern of silica gel (literature) [22].

4. Conclusions

Analysis of moisture content of sample rice husks from Mwea, Kenya found an average moisture content of 7.07% after 3½ h. Doorvasan, et al. [15] have reported a moisture range of 8.0%-9.0%. It was also observed that most of the sampled rice husks had diameters ranging from 1.00-2.00 mm. Only about 1.74% of the rice husk was within a range of 0.710 mm. The average ash content was 22.65%.

In a trial to evaluate water as a leachate to remove inorganic minerals it was found post leaching with water after incineration at 600 °C yielded 98.2% silica compared with 99.1% (H_2SO_4) and 96.9% (HCl). The availability of silica at 600 °C is not very significantly different for both water/acid leached RHA. At 500 °C, the leaching with water or acid produces the same amount of silica. At 400 °C, the leaching with acid produces more silica content.

This implies we can go ahead and leach the RHA with water instead of acid at 500 °C and still produce a significant amount of silica content. FTIR analysis of the silica gel from rice husks had peaks at 3,441, 1,101, 1,081, 948 and 799.5 cm^{-1} which is similar to other samples of silica gel in literature. The peaks are associated with 3,441 (O-H hydroxyl group, H-bonded, OH stretch), 1,101 (Si-O-C), 1,081 (Si-O-Si), 799 (silicate ion). XRD analysis shows a clear peak pattern at 20° which according to literature is an indicator of silica gel. The pattern is consistent with other XRD patterns of silica gel published by other researchers.

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