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# Synthesis and Characterization of Silica Gel Produced from Rice Husks

Nneka, B. EKWE[1*] and Helen, A. IKE[1]
[1]*Department of Chemical Engineering, University of Abuja, Nigeria.*
[*] Corresponding author: blessing.ekwe@uniabuja.edu.ng

***Abstract***
*In this work, sol-gel method was used to synthesize silica gel from rice husks and was characterized. The rice husks were leached with HCl and calcined to produce rice husk ash (RHA). The RHA was treated in NaOH followed by neutralization with HCl to obtain silica gel. Structural and compositional analysis were carried out using X-Ray Fluorescence Spectroscopy (XRF), Fourier-Transform Infrared Spectroscopy (FT-IR), X-Ray Diffraction spectroscopy (XRD) and Nitrogen sorption analysis. 74.54% amorphous silica with surface area of 283 m3/g, 0.16 cm3/g and 2.12 nm of pore volume and pore diameter respectively was recovered from RHA. The purity of the silica obtained showed the importance of aging after neutralization to completely eliminate attached product of neutralization to the silica gel.*
***Keywords***
*Characterization; Purity; Rice husk ash; Silica gel.*

## 1.0 Introduction

Globally, agricultural processes produce very big waste on a daily basis. Inappropriate management of these agricultural wastes causes environmental pollution and resultant health challenges (Bernal, 2017; Kumar & Joshi, 2013). However, agricultural wastes have unimagined potentials (Afolayan, Olofinade, & Akinwumi, 2019; Bala et al., 2023; Jena & Singh, 2022; Sadh, Duhan, & Duhan, 2018) that can be converted, transformed and recycled into beneficial use. One of such agricultural wastes is crop residue (rice husks, groundnut shells, coconut shells and husks, rice straw), pulps (tomato, orange, pawpaw), poultry waste (droppings, feathers) etc.

Rice husk is an agricultural waste produced from rice farm. Rice husk consist of cellulose (40–50%), hemicellulose (15–20%), lignin (25–30%), (Gao et al., 2018; Ugheoke & Mamat, 2012) as the main components. It has been used to produce sodium silicate (Handayani et al., 2022; Lima et al., 2021) insulating and particle boards (António, Tadeu, Marques, Almeida, & Pinto, 2018; Temitope, Onaopemipo, Olawale, & Abayomi, 2015) silica (Bakar, Yahya, & Gan, 2016; Guo, Li, Zheng, & Li, 2021) oil (Pal & Pratap, 2017), fertilizer and substrate (Badar & Qureshi, 2014; Haider, Yousaf, Malik, & Visvanathan, 2015; Lim, Wu, Sim, Lim, & Clarke, 2012), briquettes (Saeed et al., 2021), composites for building (Bisht, Gope, & Rani, 2020; Chabi, Doko, Hounkpè, & Adjovi, 2020; Choi, Mori, & Ohama, 2006; Suhot, Hassan, Aziz, & Md Daud, 2021) etc. Rice husk is also used as fuel to parboil rice paddy in rice mills due to its satisfying calorific value (Awulu, Omale, & Ameh, 2018; Yank, Ngadi, & Kok, 2016). Managing rice husk waste at times pose difficulties. Often times, it is indiscriminately dumped in open landfill and are left to putrefy, generating green-house gases (GHGs) like methane, carbon dioxide and nitrous oxide; and releasing of foul smell to the environment (N B Ekwe, 2013; Nneka Blessing Ekwe, 2012). Also, when thermally degraded, it causes environmental pollution and endangers lives.

Rice husk ash produced from thermal degradation of rice contains about 18-20 % of ash (Ardiantoro, Sunarsih, & Sucipto, 2021). Over 70% of the rice husk ash component is silica (Sawasdee & Pisutpaisal, 2022; Zainal et al., 2018). Silica ($SiO2$) is one of the most common minerals in the earth crust. It is an inorganic multi-purpose chemical compound with several industrial applications, and generally exist as amorphous or crystalline silica. Silica has many industrial applications which includes as an excipient in drugs and vitamin (Martin, 2007; Schalau & Ulman, 2009), for collagen production to improve skin elasticity (Araújo, Addor, & Campos, 2016), as coatings solutions (Gąsiorek et al., 2018; Hossain et al., 2021; Kócs et al., 2022; Xia et al., 2018), in glass industry (Onodera et al., 2020), in electronics, optical fibre and refractories (Chen, Xiong, Xu, & Lu, 2021; Kapur, 1974; Laskowski, Laskowska, Vila, Schabikowski, & Walcarius, 2019), as absorbent in nanomaterial production (Rovani, Santos, Corio, & Fungaro, 2018).

Silica has been produced through various methods using soda ash, quartz or sodium silicate. These methods include sol-gel, fluidized bed technology, precipitation, chemical vapor deposition, electrocoagulation, hydrothermal and alkaline fusion. It can also be produced from alkyl orthosilicates ore using a catalyst. In the present study to synthesize silica gel from rice husk, the objective is to use sol-gel method to produce high purity silica gel. The silica gel produced will be analysed for composition and structural silica gel for use.

## 2.0 Materials and Methods

### *2.1 Materials*

Rice husk was collected from Gwagwa farms in FCT, Nigeria. The rice husk was washed with water to remove dirt and impurities. It was sun dried for 48 hours and sieved to remove heavy impurities. Hydrochloric acid and sodium hydroxide of analytical grade were purchased from a chemical vendor in Abuja-FCT. All chemicals were used as received.

### *2.2 Materials*

400 ml of 1M HCl was mixed with1 kg rice husks in a beaker and boiled for 90 minutes at 80°C with constant stirring. The acid leached rice husk was filtered and washed with distilled water till a pH 7 of the filtrate was obtained. The washed rice husk residue was oven dried at 105°C for 48 hours; calcined for 3hrs at 600°C and allowed to cool. 100 g rice husk ash obtained was dissolved in 1M NaOH with the beaker covered. The mixture of rice husk ash was boiled for 90°C for 90 minutes. The solution was allowed to cool. The cooled solution was filtered, and the filtrate was titrated with1M HCl to a pH of 7 to form gel. The gel was washed several times till the pH of the filtrate was 7. The silica gel obtained was oven dried at 105°C for 5hrs.

### *2.3 Characterization of sample*

#### *2.3.1 X-Ray Fluorescence Spectroscopy*

The chemical compositions of the silica were obtained using Thermo Fisher Scientific Energy Dispersive X-ray Fluorescence (EDXRF), model-ARL. QUANT'X. EDXRF Analyzer (Thermo Fisher Scientific Company, Switzerland).

#### *2.3.2 Fourier Transform Infra-red Spectroscopy*

Fourier transform infra-red (FTIR) spectra of RHA and silica gel samples were obtained using Agilent Fourier-transform infrared spectrometer equipped with attenuated total reflectance (ATR) accessory. The spectrum was averaged from 64 scans. The resolution was $16 cm^{-1}$ over the wavenumber range from 650 to 4000 $cm^{-1}$.

#### *2.3.3 Nitrogen sorption analysis*

The surface area was estimated by Brunauer–Emmett–Teller (BET), while pore volume and pore diameter were analysed by Barett-Joyner-Halenda (BJH). The nitrogen gas adsorption-desorption were carried out using Quantachrome NovaWin Version 11.03 (Quantachrome Instruments, Boynton Beach, FL, USA), at 77.350 K and degassed under vacuum at 250 °C for 180 min prior to analysis.

#### *2.3.4 X-Ray Diffraction Spectroscopy (XRD)*

X-ray diffractometer of the produced silica was obtained from Thermo Fisher Scientific, model-ARL. QUANT'X. X-ray. XRD (Thermo fisher Scientific Company, Switzerland), using Cu Kα ($\lambda$ = 1.540593 Å) at 40 kV and 15 mA. The sample was scanned over a range of 2 theta angles from 2° to 70° at a step size of 0.02° and scan speed of 20 degrees/min.

## 3.0 Results and Discussions

### *3.1 X-Ray Fluorescence Spectroscopy*

Quantitative identification of chemical composition and the purity of silica produced were analysed with the use of X-Ray Fluorescence. From **Table 1**, it was observed that silica was the major constituent of both the rice husk ash and silica gel. Evidently shown is the decrease in the silica obtained when compared with the rice husk ash and significant increase in chlorine from the silica in comparison to the race husk ash. The result suggest that the neutralization process was not thoroughly washed accounting for the impurity increase.

**Table 1:** Chemical composition of the Rice Husk Ash and Silica Gel

| S/no | Chemical Compositions | RHA (%) | SG (%) |
|---|---|---|---|
| 1 | $SiO_2$ | 81.35 | 74.54 |
| 2 | CaO | 1.39 | 0.03 |
| 3 | $Al_2O_3$ | 1.25 | 0.92 |
| 4 | $Fe_2O_3$ | 0.52 | 0.02 |
| 5 | MgO | 0.80 | 0.38 |
| 6 | $P_2O_5$ | 1.05 | 0.19 |
| 7 | $SO_3$ | 0.35 | 0.32 |
| 8 | Cl | 0.01 | 0.80 |
| 9 | $K_2O$ | 0.06 | 0.67 |
| 10 | BaO | 0.46 | 0.47 |
| 11 | SrO | 0.54 | 0.19 |
| 12 | $Nb_2O_5$ | 0.12 | 0.12 |

### *3.2 Fourier Transform Infra-Red Spectrometry*

To gain insight about the chemical changes that occurred in the process of silica gel synthesis, FT-IR was used to analyse the rice husks ash and silica gel produced. FT-IR spectra are as presented in Figure 1. A considerable observation from Figure 1a shows an absence of the functional group band but with prominent silica bands within the fingerprint region of the rice husk ash. The two vibrations of 1051 $cm^{-1}$ and 797 $cm^{-1}$ in the fingerprint region are attributable to Si – O- Si and O-Si-O vibration mode of $SiO_2$ respectively. The spectrum in Figure 1b has similar trend recorded in Figure 1a but with an additional vibration appearing in band 954 $cm^{-1}$ attributable vibration mode of Si – OH. The spectrum of silica in Figure 1b clearly shows that the vibrational bands of Si – O- Si, O-Si-O and Si – OH are characteristics of amorphous silica.

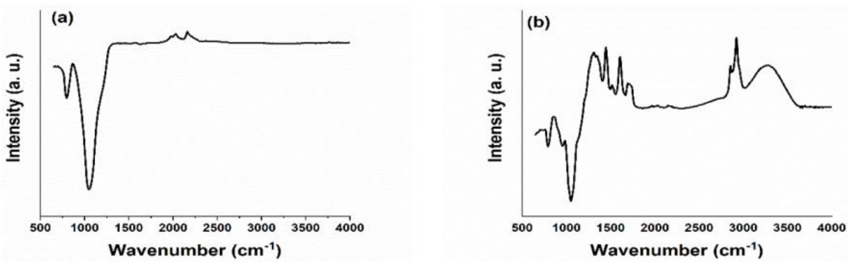


***Figure 2:*** *FTIR spectra of (a) Rice Husk Ash and (b) silica*

### *3.3 Nitrogen sorption analysis*

From the Nitrogen sorption analysis of the synthesized silica gel, the estimated surface area was 283. $m^3/g$. the pore volume and pore diameter were evaluated to be 0.16 $cm^3/g$ and 2.12 nm respectively.

### *3.4 X-Ray Diffraction Spectroscopy (XRD)*

Silica gel produced from rice husks ash were subjected to XRD analysis. Figure 2 shows the diffractogram of $SiO_2$. The diffractogram of $SiO_2$ consists of broadened peak at 22.57$^o$ which indicates critical amorphous $SiO_2$. This is consistent with the silica bands already reported in FT-IR spectroscopy. Remarkebly observed from Figure 2 are the sharp crystalline peaks at 32.20$^o$ and 45.96$^o$. These peaks are as a result of impurities in the form NaCl in the silica gel. The impurities are indication that the silica gel was not soaked for too long to allow for complete dissolution of NaCl formed after neutralization of the filtrate with HCl. Accordingly, the presence of impurities explains the decrease witnessed in the percentage of silica produced in comparison with the silica content of RHA and the significant increase in the chlorine content of the silica when compared to its composition in RHA.

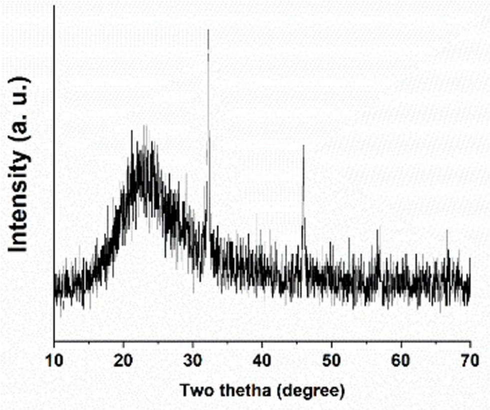


***Figure 2:*** *XRD diffractograms of the silica gel*

## 4.0 Conclusion

In this study, silica gel was synthesized from rice husks through sol-gel method and characterized. The success of the synthesis was demonstrated by the different characterization techniques (FTIR, XRD, XRF) employed. The amorphous silica gel produced exhibited some impurities thus impacting on the purity level. The study realized the importance of neutralization products removal.

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