Aerogel is a synthetic porous ultra-light material derived from a gel, in which the liquid component of the gel has been replaced with a gas (IUPAC, 2007). It is a mesoporous (Noor et al., 2013), open cell, low-density substance that exhibits density substance that exhibits a density of 1.122. XRD analysis of the RHA fired at 900°C revealed crystalline structure with particle size of 30.34 nm. The nature of surface modification of the aerogel was studied by Fourier Transform Infrared Spectroscopy (FTIR).

Keywords: Physicochemical properties, porosity, silica aerogel, silica, sol-gel

Materials and Methods

Materials

The following materials were used in the course of this research work: electric furnace, electric oven, scanning electron microscope (SEM) model Oxford Instrument, X-ray diffractometer XRD6000, Fourier transform infrared spectrophotometer FTIR-8400S, routine laboratory wares (PYREX), deionize water, stainless steel beakers, rice husk, and systematic modified approaches. Kistler arrived at aerogel which are very similar to silica aerogels prepared today (Stephens, 1932). Aerogel are usually prepared by supercritical drying of wet silica gels (Tang & Wang, 2005). The ambient pressure drying technique is one of the alternative cost-effective processes of aerogel synthesis (Rakesh et al., 2009). Other methods of preparing nanosilica includes the vapour-phase reaction, sol-gel and thermal decomposition technique (Azadeh et al., 2012). The sol-gel process is another method for preparing aerogel using organic silica monomers such as tetramethyloxysilicate (TMOS) and tetraethoxysilicate (TEOS) as precursors. However, report by Kumar et al. (2013) holds that such organic precursors are so expensive and carcinogenic hence the need to produce industrial scale silica aerogel from RH, an inorganic raw material which is inexpensive, biocompatible and nontoxic.
**Preparation of silica aerogel**

About 15 g of ash was mixed with 20 cm$^3$ of 30% $\%_{w}$ NaOH aqueous solution. The mixture was heated up to its boiling point for 90 min with the reflux. The solution was filtered to remove undissolved residues. The filtrate was neutralized with dilute HNO$_3$ to pH of 7 to form silica hydrogel. The prepared gel was aged at room temperature for 24 h under sealed condition. The aged gel was washed using deionized water to remove excess sodium nitrate (Nayak&Bera, 2009). Subsequently, the silica gel was soaked in solution of 20% ethanol for 24 h, maintained at 50°C followed by aging with ethanol at the same condition. The ethanol treated gel was aged in solution of 70% of ethanol for 24 h at 70°C. According to Nayak&Bera (2009) the strength and stiffness of gel may be increased by ethanol washing due to dissolution of silica from the particles and precipitation into the necks between the particles. Precipitation of silica gives an increase in the density of the wet gel and corresponding strengthening and stiffening of the gel network (Einarsrud&Nilson, 1998).

Stated by Nayak&Bera (2009) residual pore water and by-product of condensation is responsible for hydrolysis of ethanol which also takes part in condensation reaction with Si–O group of gel structure according to the polymerization scheme,

**Hydrolysis**

\[ \text{Si(OH)}_3 + 4\text{H}_2\text{O} \rightarrow \text{Si(OH)}_4 + 4\text{C}_2\text{H}_5\text{OH} \quad (1) \]

**Condensation**

\[ \text{Si(OH)}_4 + (\text{OH})_2\text{Si} \rightarrow (\text{OH})_2\text{Si}–\text{O}–\text{Si(OH)}_3 + \text{H}_2\text{O} \quad (2) \]

\[ \equiv\text{Si(OH)}_2\text{H}_3 + \text{HO}–\text{Si}–\equiv \rightarrow \equiv\text{Si}–\text{O}–\text{Si}–\equiv + \text{C}_2\text{H}_5\text{OH} \quad (3) \]

The gel was washed with n-heptane several times to remove ethanol solution from the gel. Then, it was aged inside n-heptane at 50°C for 24 h with four times renewal of fresh n-heptane. Finally, modified gel was aged for another 24 h inside n-heptane at room temperature before air drying. The gel was dried in 24 h interval at 50, 90, 120, and 150°C with partially covered condition and a dried silica aerogel was obtained. A summary of the aerogel preparation steps is as presented in Fig. 1.

**Fig. 1:** Flowchart of silica aerogel preparation from rice husk

The bulk density of the sample was estimated using equation A.

\[ \rho = \frac{M}{V} \quad (A) \]

**Where:** $\rho$ is the bulk density, $M$ and $V$ are the mass and volume of silica aerogel respectively.

The porosity of the aerogel was estimated using equation B.

\[ P = \left(1 - \frac{\rho_{Si}}{\rho_s}\right) \times 100 \quad (B) \]

**Where:** $P$ is the porosity, $\rho_{Si}$ is bulk density of silica aerogel and $\rho_s$ is the specific density of amorphous silica assumed to be 2.0 g/cm$^3$ (Mupae et al., 2015; Adams et al., 2011).

The particle size of the nanosilica was calculated using Debye-Scherrer formula;

\[ d = \frac{0.9\lambda}{\beta\cos\theta} \quad (C) \]

**Where:** $\lambda$ is wavelength of X-ray (0.1541 nm), $\beta$ is FWHM in radian, $\theta$ is the diffraction angle and $d$ is particle size. The refractive index from the aerogel been density dependent was calculated from the formula

\[ n = 1 + k\rho \quad (D) \]

**Where:** $n$ is the refractive index, $k = 2.1 \times 10^{-4}$ a constant and $\rho$ (kg/m$^3$) is the density of the aerogel (Aegerter et al., 2011; Makoto et al., 2012).
Results and Discussion

Chemical Composition of Silica Aerogel

The main aim of converting husk to ash is to utilize its silica for the production of silica aerogel whose percentage varies from one sample to another depending on the climatic and geographical conditions. According to (Nzami, 2003), pyroprocessing of rice husk is frequently carried out to get rice husk ash with maximum percentage of silica with proper processing giving rise to RHA with highest percentage of silica.

High silica content and low levels of elemental impurities are necessary pre-requisite for the synthesis of high purity silicon from RHA (Kingsley, 2010). The chemical composition XRF analysis of the as-prepared RHA revealed 98.32 Wt % SiO$_2$ with other oxides totaling to 1.68 Wt %. This result is in agreement with that obtained by Mohamed et al. (2015) to be 89.00 Wt% SiO$_2$.

A comparative study of the elemental composition of silica gel and aerogel is shown on Table 1. Result shows that the concentration by weight percent (Wt %) of silicon 16.7 in silica gel increased to 17.1 in silica aerogel. This is probably due to removal of impurities after soaking in ethanol and treating with n-heptane to remove ethanol solution from the gel which probably hinders a complete detection of the actual composition of silicon in the gel (Pinghauet al., 2016; Kaviyarasu et al., 2016).

### Table 1: Chemical composition of rice husk ash heated at 900°C

<table>
<thead>
<tr>
<th>Element</th>
<th>Conc. (Wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>98.32</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>0.04</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>0.25</td>
</tr>
<tr>
<td>MgO</td>
<td>0.29</td>
</tr>
<tr>
<td>P$_2$O$_5$</td>
<td>0.32</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>0.18</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0.37</td>
</tr>
<tr>
<td>CaO</td>
<td>0.35</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>0.03</td>
</tr>
<tr>
<td>MnO</td>
<td>0.06</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>0.01</td>
</tr>
<tr>
<td>ZrO$_2$</td>
<td>0.01</td>
</tr>
<tr>
<td>BaO</td>
<td>0.02</td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
<td>0.02</td>
</tr>
</tbody>
</table>

### Table 2: Elemental comparison between silica gel and aerogel

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Number</th>
<th>Conc. (Wt %) Silica gel</th>
<th>Conc. (Wt %) Aerogel</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>7</td>
<td>7.1</td>
<td>7.6</td>
</tr>
<tr>
<td>O</td>
<td>8</td>
<td>65.1</td>
<td>63.1</td>
</tr>
<tr>
<td>Na</td>
<td>11</td>
<td>8.4</td>
<td>5.5</td>
</tr>
<tr>
<td>Si</td>
<td>14</td>
<td>16.7</td>
<td>17.1</td>
</tr>
<tr>
<td>Rb</td>
<td>37</td>
<td>2.4</td>
<td>6.7</td>
</tr>
</tbody>
</table>

### XRD characterization

Phase identification in the RHA was assessed by X-ray diffraction analysis as presented in Fig.2. Generally, silica undergoes structural transformation at different calcinations temperatures (Omotala&Onoja, 2009). Silica obtained at incineration temperatures below 800°C are amorphous (Ramizanianpouet al., 2009) with broad peaks. Crystalline silica begins to form from 800°C and is completed at 900°C (Sugita, 2008). The RHA fired at 900°C shows significant crystallinity with sharp narrow peaks at 28 values of 22$^\circ$ assigned to MnO and 27$^\circ$ attributed to SiO$_2$. Findings are in agreement with (Iyenagba& Othman, 2012). Report by (Kingsley, 2010) also holds that RHA fired at 900$^\circ$ attained significant crystallinity with reflections or sharp peaks of different phases of SiO$_2$ in their XRD pattern. The particle size calculated from Debye- Scherrer formula was 30.34 nm.
Determination of Physicochemical Properties of Silica Aerogel from Rice Husk

The particle size value fits well in the range 20 – 30 nm reported by Young et al. (2008).

Fig. 2: X-ray diffractogram of rice husk ash

FTIR characterization
Figure 3 shows FTIR result of aerogel heated at 900°C. There were characteristic bands of silica aerogel detected. The absorption at 817.85 cm⁻¹ belongs to bending vibration of O – Si – O. The band 946.12 cm⁻¹ is associated with the C – H out-of-plane bending vibration while the band at 1090.78 cm⁻¹ is assigned to C – O as a result of the alcohol and other impurities like ethers, esters, carboxylic acid and anhydrides. Absorption at 1383.97 cm⁻¹ is ascribed to the C – H bending vibration of –CH₃, and that at 1634.73 cm⁻¹ is attributed to the C=C mode with no interpretatively useful vibration. The band 2103.44 cm⁻¹ is also associated with the C=C mode probably due to the alkynes. The 2772.76 cm⁻¹ band can be traced to the C – H mode; while absorption at 2847.03 cm⁻¹ is due to the C – H stretching vibration. And 3469.09 cm⁻¹ is associated with the –OH group of water molecules, bonded by hydrogen bonds or OH groups present in organic compounds.

Fig. 3: FTIR spectrum of silica aerogel

Surface morphology
The nature and form of the samples were examined using a scanning electron microscope. Plates 5 – 7 present the SEM micrograph of the prepared samples. Plate 5 reveals the fused solid nature or honey comb of the RHA. Plate 6 shows the white parts in the surface of the gel the black parts as pores. And Plate 7 shows the compact nature of silica aerogel.

Plate 5: SEM micrograph of rice husk ash
Plate 6: SEM micrograph of silica gel
Plate 7: SEM micrograph of silica aerogel

Conclusion
Rice husk, an important agricultural waste, rich in silica can be used for a number of applications. The paper has demonstrated that thermally treated rice husk yields ash that can be processed under controlled techniques to obtain silica aerogel. The pure SiO₂ obtained was 98.32 by Wt%. X-ray diffraction analysis of the RHA revealed crystalline structure with average article size of 30.34 nm. The bulk density of aerogel was found to be 0.58 g/cm³ and porosity of 71.0%; while its refractive index was 1.122.

References
Determination of Physicochemical Properties of Silica Aerogel from Rice Husk


Qi T &Tao W 2015. Preparation of silica aerogel from rice hull ash by supercritical carbon dioxide drying. J. Supercritical Fluids, 53: 91 – 94.


