

Ultraviolet - Visible Spectroscopy of Silica Gels From Rice Husk

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Abstract

Crystalline silica gel was prepared from leached and un-leached rice husk calcined to 1000°C in a muffle furnace. The prepared gels were dissolved in absolute ethanol and aged for 24 hours. The UV-Visible Agilent Spectrophotometer was used to observe the optical transmission and intrinsic absorbance of the gels. The results obtained were compared to those of the commercial gel. The result of the leached rice husk silica gel showed a higher percentage (up to 91%) than the un-leached rice husk silica gel (82%), and that of commercial gel has the lowest percentage (22%) in transmittance.

Keywords: Silica gel, rice husk, transmission, absorbance, UV-Visible

1.0 Introduction

Burning rice husk as fuel to generate energy results in the product rice husk ash (RHA).

RHA is rich in silica (about 60%) and can be an economically viable raw material for the production of silica gels and powders [1][2].

RHA has been evaluated as an adsorbent of minor vegetable oil components [3][4].

Although various uses for rice husk and RHA have been suggested in the literature, their

disposal or utilization remains a major concern.

Soluble silicates produced from silica are widely used in the glass, ceramics, and cement industries as a component and in pharmaceuticals, cosmetics, and detergent industries as bonding and adhesive agents [5].

Silica has also been used as a major precursor for inorganic and organometallic materials,

which have applications in synthetic chemistry as catalysts and in thin films or coatings for electronic and optical materials [6][7]. Mizuki et al. [8] and Krishnaro et al. [9] have investigated the formation of silica carbide from rice husks at high temperatures, and Maeda et al. [10] studied the semiconductor properties of silicon carbide produced from rice husks. Semiconductor industries require pure silica, currently produced by smelting quartz in a high-temperature furnace to produce ultrapure polycrystalline silicon and silicon hydride. The development of a simple, low-energy chemical method for producing pure silica should lead to a variety of industrial applications for RHA.

Acid leaching (Chakraverty et al. [11]) and gasification (Luan et al. [12]) methods have been investigated for recovering silica from rice husk ash. The aim of this work is to examine the optical properties of rice husk silica gel in the visible transmittance spectrum and compare them with that of commercial silica gel; and to investigate three samples of silica gels and see the one with high transparency for the use of optical materials.

2.0 Method and Material

Two samples of the rice husks were obtained each from different rice stations in Benue and

Kogi States, which are known to be rice producers in the middle belt region of Nigeria. Sample one was leached and came from Abeokuta in Kogi state, while sample two was unleached and came from Makurdi in Benue state.

2.1 Overview of Rice Husk (RH): Rice husk is the protective layer that surrounds a rice grain [2]. It is a yellowish color and has convex shapes. It is slightly larger than a grain of rice, so lengths of up to 7 mm are possible. Typical dimensions are 4 mm x 6 mm. It is lightweight, having a ground bulk density of 340 kg/m^3 to 400 kg/m^3 [4]. Rice is the most commonly eaten daily foodstuff for more than two-thirds of the world's population. 92% of rice is grown in Asia, the majority in Vietnam and Thailand. In 2013, approximately 5.4 million tonnes of raw hush were available from Vietnam [5]. Rice husk is low in density and has an unusual shape. This poses storage and transport challenges [1].

2.1.1 Rice husk ash (RHA): Rice husk ash (RHA) is produced by controlled burning of rice husk and contains a minimum of 75% amorphous silica [6]. The norm is 95%. This can be used for cement production as a percentage substitute for ordinary Portland cement and as an insulator to line ladles in steel manufacturing. It is not as simple as

burning and you have the right product. There are significant property differences between white and black ash. It requires very specific heat and timing to produce the correct product. For every 1000kg of paddy milled, about 200kg (20%) of husk is produced, and when this husk is burnt in the boilers, about 50% (25%) of RHA is generated [7].

2.1.2 Rice husk ash as a precursor to glass:

Silica glass is an interesting material due to its low thermal expansion coefficient, high chemical inertness, and transparency [8]. In this study, low-cost rice husk ash waste containing mainly amorphous silica was used as the starting raw material. The formation of cristobalite, which caused product damage during cooling down, was suppressed by the addition of some sintering aids. Rice husk ash is taken as a silica precursor for the preparation of glass ceramics. The sol-gel route is preferred for synthesizing glass ceramics powder. After calculating, the powder is used for the preparation of the body through the gel-casting process [9]. Thus, rice husk ash is used as a silica source for the preparation of glass-ceramics and can be gel-cast to fabricate scaffolds for bio-application.

2.1.3 Silica gel properties: Silica gel is an amorphous and porous form of silicon dioxide (silica, consisting of an irregular

tridimensional framework of alternating silicon and oxygen atoms with nanometer-scale voids and pores. The voids may contain water or some other liquid, or may be filled by gas or vacuum [5]. Its high surface area (around 800 m²/g) allows it to absorb water readily, making it useful as a desiccant (drying agent). Once saturated with water, the gel can be regenerated by heating it to 120 °C (250 °F) for two hours. Some types of silica gel will "pop" when exposed to enough water [6].

2.1.4 Absorbance, transmittance, and Beer's law:

When light is absorbed by a sample, the irradiance of the beam of light is decreased. Irradiance, P is the energy per second per unit area of the light beam. In a rudimentary spectrophotometer, light is passed through a monochromatic (a prism, a grating, or even a filter) to select one wavelength. Light with a very narrow range of wavelengths is said to be monochromatic ("one color.") [13]. The monochromatic light, with irradiance P_0 , strikes a sample of length b . The irradiance of the beam emerging from the other side of the sample is P . Some light may be absorbed by the sample, so $P < P_0$.

Transmittance, T , is defined as the fraction of the original light that passes through the sample. Therefore, T has a range of 0 to 1.

The percent transmittance is simply $100T$ and ranges between 0 and 100% [14].

Absorbance is defined as when no light is absorbed, $P = P_0$ and $A = 0$. If 90% of the light is absorbed, 10% is transmitted, and $P = P_0/10$. This ratio gives $A = 1$. If only 1% of the light is transmitted, $A = 2$. Absorbance is sometimes called optical density. Absorbance is directly proportional to the concentration, c , of the light-absorbing species in the sample, as described by the Beer-Lambert law, or simply Beer's law. Absorbance is dimensionless, but some people write "absorbance units" after absorbance. The concentration of the sample, c , is usually given in units of moles per litre (M). The path length, b , is commonly expressed in centimetres. The quantity ϵ (epsilon) is called molar absorptivity (or extinction coefficient in the older literature) and has the same units as $M^{-1} \text{ cm}^{-1}$ to make the product dimensionless. Molar absorptivity is a property of a substance that indicates how much light is absorbed at a given wavelength [13].

The equation for Beer's law is written as $A = \epsilon b c$. A is the absorbance, b is the length of the light path, and C is the concentration. The greater the molar absorptivity, the greater the absorbance. The part of a molecule responsible for light absorption is called a

chromosphere. Any substance that absorbs visible light appears colored when white light is transmitted through it or reflected from it. The substance absorbs certain wavelengths of white light. Our eyes detect the wavelengths that are not absorbed [15]. The observed color is called the complement of the absorbed color. For example, bromophenol blue has maximum absorbance at 591 nm and its observed color is blue. Beer's law applies when radiation is monochromatic and the solution is dilute (0.01 M). Beer's law fails in certain cases: high concentrations: In concentrated solutions, solute molecules influence one another as a result of their proximity. When solute molecules get close to one another, their properties (including molar absorptivity) change somewhat. At very high concentrations, the solute becomes the solvent. The properties of a molecule are not exactly the same in different solvents. Interactions with other solvents: Non-absorbing solutes in a solution can also interact with the absorbing species and alter the absorptivity. Concentration-dependent chemical equilibrium: If the absorbing molecule participates in a concentration-dependent chemical equilibrium, the absorptivity changes with concentration. For example, in a concentrated solution, a weak acid, HA, may be mostly undissociated. As

the solution is diluted, dissociation increases. If the absorptivity of A- differs from that of HA, the solution may appear to deviate from Beer's law as it is diluted.

Interference from other absorbing species: diluted species with similar absorbing maxima to the solute of interest may produce false readings [13].

2.2 Flow Chart

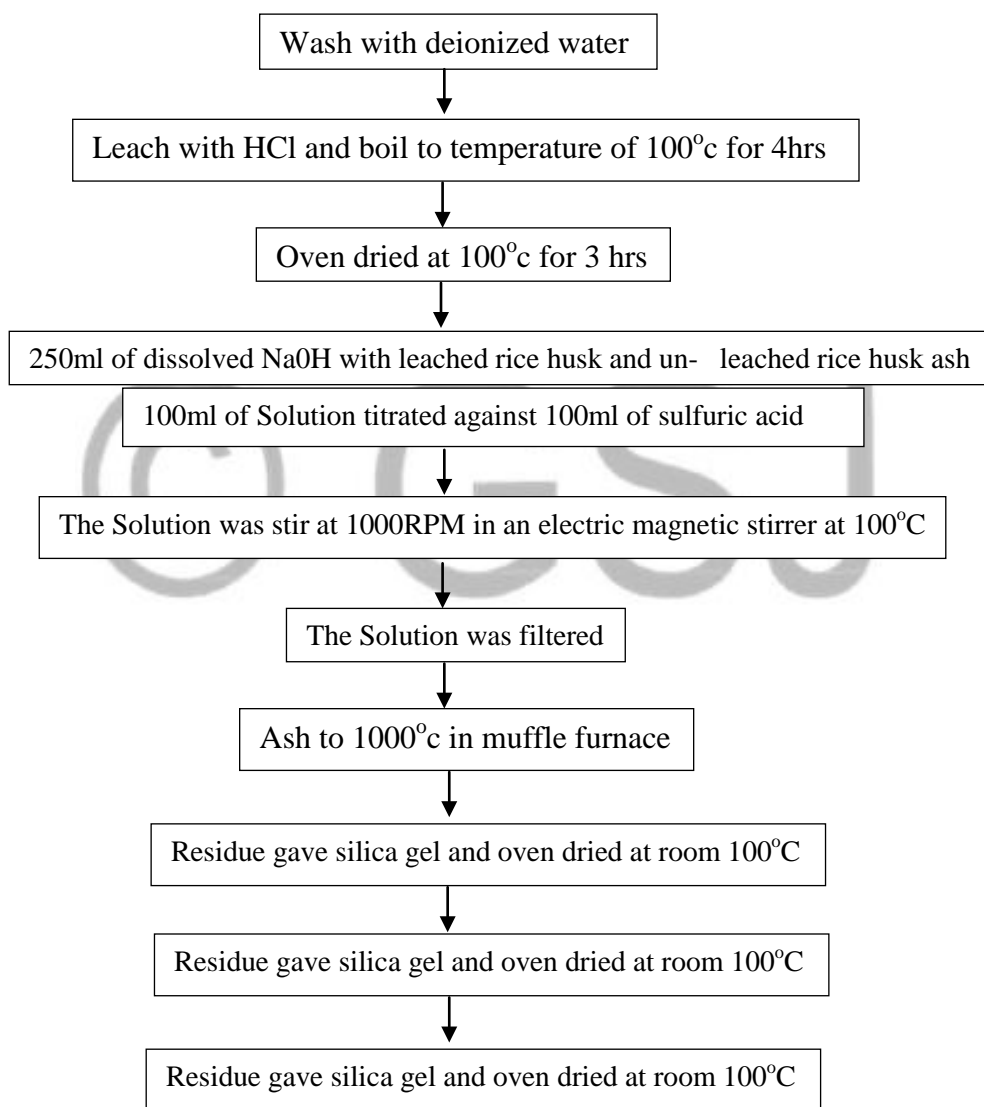


Fig.1 Flow chart showing the process involved in preparation of rice husk (RH) into rice husk ash (RHA) samples.

2.2 Using an ultraviolet spectroscopy meter, produce silica gel from rice husk ash.

On a scale, 80g of sodium hydroxide (NaOH) was measured and dissolved into 1000ml of distilled water. The reaction of water with NaOH emits heat because the content of the NaOH is higher than the water and the reaction that occurs here is called an exothermic reaction.

2.3 Sulfuric acid (H₂SO₄) Preparation

By the following calculation, sulfuric acid was prepared.

$$m = \frac{\text{sulphuric acid in gram} \times \% \text{ in purity} \times 1000}{\text{mass of the weight} \times 100}$$

(1)

where:

Molar mass of Sulfuric acid in grams =

1.84grams

Percentage (%) purity = 98

Mass of the weight = 98.01

2.4 Samples for UV-VIS Spectrophotometry

These are most often liquids, although the absorbance of gases and even of solids can also be measured. Samples are typically placed in a transparent cell, known as a cuvette. Cuvettes are typically rectangle in

shape. Commonly, with an internal with an internal width of 1 cm (this width becomes the path length in the Beer-Lambert law). The type of sample container used must allow radiation to pass over the special region of interest. The most widely applied cuvettes are made of highly quality fused silica or quartz glass because they are transparent throughout the UV-visible and near infrared regions, although glass and most plastics absorb in the UV, which limits their usefulness to visible wavelengths [16].

3.0 Results and Discussion

The result of the NaSO₄ (54.4 cm³) was measured on a scale and diluted with water to make 1000 ml. The reaction taking place here is also an exothermic reaction. After some time, 10 g of a sample leaching rice husk was measured and dissolved into 250ml of NaOH, which was stirred for about 1 hour at room temperature (100°C) with a 1000 rpm by an electrical magnetic stirrer.

The solution of the sample leaching rice husk ash that dissolves into NaOH was measured and titrated against 100ml of sulfuric acid, which reacted. A gel was settled in a solid form where water with sulfuric acid was at the top [17][18]. The solution was filtered to remove the water with sulphuric acid. The

residue is called rice husk silica gel. The wet rice husk silica gel was oven-dried at a room temperature of 1000°C for 1 hour.

The solution of the sample was un-leaching rice husk ash that dissolved into NaOH. 100ml of it was measured and titrated against 100ml of sulfuric acid, which reacted. A gel was settled down in a solid form from where water with sulfuric acid was at the top [19]. The solution was filtered to remove the sulfuric acid. The residue is called rice husk silica gel. The wet rice husk silica gel was oven-dried at a room temperature of 1000°C for 1 hour.

In addition, the silica gel produced was dissolved into ethanol, and then the UV-Visible spectrometer was used. The results for transmittance are shown in tables (fig.1) and for absorbance (fig.2). For the leached sample, as the wavelength becomes progressively shorter, scattering increases, eventually cutting off transmission near 350 nm. The un-leached sample has cut off transmission near 250nm while the commercial sample is near 200nm. The weakest absorbance was observed in the leached sample, followed by the un-leached and in the commercial sample [20][21].

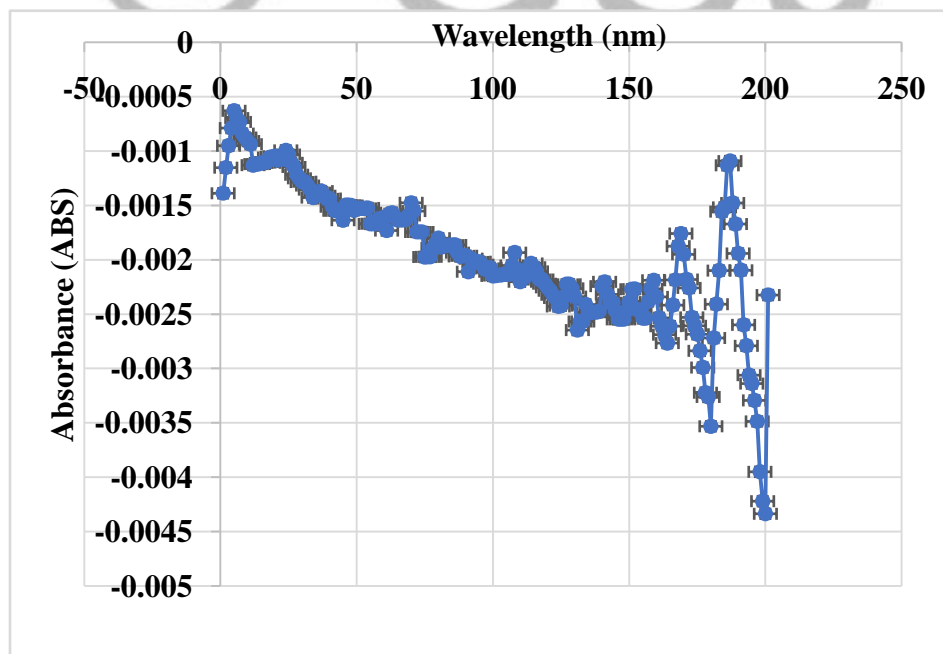


Fig.2 Absorbance plot of Rice Husk Silica.

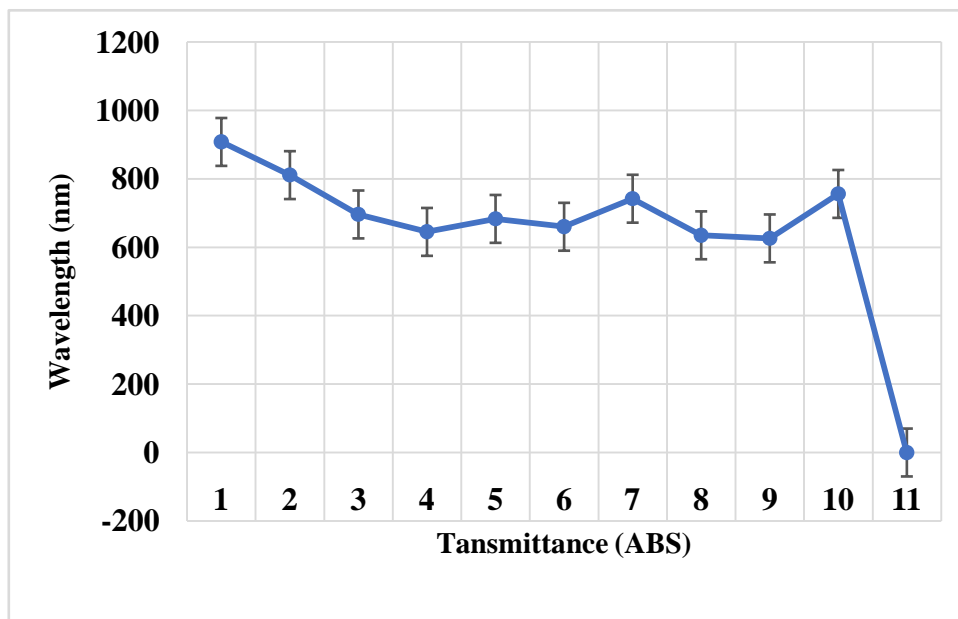


Fig.3 Absorbance plot of Rice Husk Silica.

Conclusion

It can be concluded that for the leached sample, as the wavelength became progressively shorter, scattering increased, eventually cutting off transmission near 350nm; the un-leached sample has cut off transmission near 250nm, while the commercial sample is near 200nm. The weakest absorbance was observed in the leached sample, followed by the un-leached and in the commercial sample. The very high transparency nature of the gels derived from the rice husk ash suggests their possible utilization as optical materials.

References

- [1] Kamath, Savita R., and Andrew Proctor. "Silica gel from rice hull ash: preparation and characterization." *Cereal Chemistry* 75.4 (1998): 484-487.
- [2] Chakraverty, A., and S. Kaleemullah. "Conversion of rice husk into amorphous silica and combustible gas." *Energy conversion and management* 32.6 (1991): 565-570.
- [3] Proctor, Andrew, and Sevugan Palaniappan. "Adsorption of soy oil free fatty

acids by rice hull ash." *Journal of the American Oil Chemists' Society* 67.1 (1990): 15-17.

[4] Palaniappan, Sevugan, and Andrew Proctor. "Competitive adsorption of lutein from soy oil onto rice hull ash." *Journal of the American Oil Chemists' Society* 67.9 (1990): 572-575.

[5] Anon, Soluble Silicates. "their Applications." *Crossfield Publication, Crossfield, Warrington, UK* (1997).

[6] Lender, P. W., and R. Ruiter. "Novel inorganic materials and heterogeneous catalysis." *Inorganic and metal-containing polymeric materials* (1990): 187-195.

[7] Brinker, C. Jeffrey, and G. W. Scherer. "Sol-gel sciences." *The Processing and the Chemistry of Sol-Gel Processing* (1990).

[8] Mizuki, E., et al. "Formation of silicon carbide from rice husks using enzymatic methods for carbon control." *Bioresource technology* 44.1 (1993): 47-51.

[9] Krishnarao, R. V., and M. M. Godkhindi. "Distribution of silica in rice husks and its effect on the formation of silicon carbide." *Ceramics international* 18.4 (1992): 243-249.

[10] E. Maeda, M. Komatsu, The Thermoelectric Performance of Silicon Carbide Semiconductor Made from Rice Hull, Materials Research Society, Pittsburgh, PA 1996.

[11] Chakraverty, A., and S. Kaleemullah. "Conversion of rice husk into amorphous silica and combustible gas." *Energy conversion and management* 32.6 (1991): 565-570.

[12] Luan, Ta Chi, and Tse Chuan Chou. "Recovery of silica from the gasification of rice husks/coal in the presence of a pilot flame in a modified fluidized bed." *Industrial & engineering chemistry research* 29.9 (1990): 1922-1927.

[13] Mäntele, Werner, and Erhan Deniz. "UV-VIS absorption spectroscopy: Lambert-Beer reloaded." *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 173 (2017): 965-968.

[14] Hardy, James D., Harold T. Hammel, and Dorothy Murgatroyd. "Spectral transmittance and reflectance of excised human skin." *Journal of Applied Physiology* 9.2 (1956): 257-264.

[15] Abitan, Haim, Henrik Bohr, and Preben Buchhave. "Correction to the Beer-Lambert-

Bouguer law for optical absorption." *Applied optics* 47.29 (2008): 5354-5357.

[16] Almeida, R. M., and A. S. Rodrigues. "Photonic bandgap materials and structures by sol-gel processing." *Journal of non-crystalline solids* 326 (2003): 405-409.

[17] Krishnarao, R. V., and M. M. Godkhindi. "Distribution of silica in rice husks and its effect on the formation of silicon carbide." *Ceramics international* 18.4 (1992): 243-249.

[18] Luan, Ta Chi, and Tse Chuan Chou. "Recovery of silica from the gasification of rice husks/coal in the presence of a pilot flame in a modified fluidized bed." *Industrial & engineering chemistry research* 29.9 (1990): 1922-1927.

[19] Nocuń, Marek, et al. "Structural studies of TEOS-tetraethoxytitanate based hybrids." *Optical Materials* 27.9 (2005): 1523-1528.

[20] Misra, Prabhakar, and Mark A. Dubinskii, eds. *Ultraviolet spectroscopy and UV lasers*. CRC Press, 2002.

[21] Ogawa, Takayuki, Noboru Murata, and Seiji Yamazaki. "Sol-gel products news." *Journal of Sol-Gel Science and Technology* 27.2 (2003): 237-238.