

# Synthesis of Orange Colored Silica gel based on Rice Husk Ash by Using Iron Salt Indicator

## ABSTRACT

Silica gel is a solid adsorbent known for its unique property of changing color as it adsorbs moisture. The main component in the production of silica gel is sodium silicate, which can be derived from natural raw materials such as sugarcane bagasse ash, rice husk ash and coconut shell. In this study, sodium silicate was extracted from rice husk ash (RHA), followed by the synthesis of silica gel using the sol-gel method. The resulting silica gel was then colored orange using iron (III) chloride salt. FE-SEM image showed a homogeneous distribution of particle size and color on the surface of the silica gel. The BET surface area of orange silica gels synthesized from commercial sodium silicate and RHA (Aging time =24 hours) was found to be 248.6 and 179.86 m<sup>2</sup>/gr, respectively but the high  $S_{meso}/S_{micro}$  in silica gel from rice husk ash contributes to its suitable moisture adsorption capacity compared to silica gel from industrial sodium silicate.

**KEYWORDS:** Adsorbent, Iron (III) chloride salt, Moisture, RHA, Silicagel

## 1.INTRODUCTION

Various moisture-sensitive materials are protected from damage by silica gel desiccant. It is difficult to determine if the gel needs to be regenerated or replaced when saturated with moisture since the gel is usually white or colorless. Hence, it is essential to have a visible indication when the gel is saturated with moisture. Conventionally, this has been accomplished by immersing it in a cobalt salt (usually cobalt chloride), which gives a dark blue color to the dry gel[1]. When the

dry gel adsorbs moisture, its color changes from dark blue to light pink. Due to its color-changing property, it is an effective desiccant for several industrial applications, but cobalt chloride is a carcinogen, which means a much tighter control regime will be needed for the silica gel that contains it. However, due to the sharp change in color, it is preferred for industrial applications excluding food and pharmaceuticals, for this purpose, alternative salts, such as Ferric salts, are used in silica gels to create color change properties [2, 3] .

A common source of silica gel is sodium silicate, which is available commercially. Sodium silicate is produced by melting quartz sand with sodium carbonate at 1300°C[4]. The process is costly because it requires a high temperature, and it emits significant air pollution by creating dust, nitrogen, and Sulphur dioxide [5]. As an alternative, sodium silicate can be generated at much lower temperatures by boiling amorphous silica in sodium hydroxide. Rice husk ash (RHA) is an economical and environmentally friendly source of amorphous silica [6]. In the rice-producing countries, rice husk is abundantly available as a by-product of agriculture. After rice grains are milled, the rice husk is separated from them. Approximately more than 700 million tons of rice are produced in the world annually. About 20% of rice is comprised of rice husk, and the remainder is rice. Commonly, rice husk consists of 50% cellulose, 25-30% lignin, and 15-20% silica [7] . Moreover, researchers have reported that each ton of rice produces approximately 0.23 tons of rice husk [8]. In rice-producing countries, rice husks are either burned or dumped as waste. When rice husk is burned, RHA is produced. The buried rice husks can occupy a great deal of space, cause spontaneous combustion, emitting ash, and damaging the environment. Currently, rice-producing countries struggle with rice husk and are trying to find ways of using it economically. Due to its high internal surface area and porous nature, rice husk is uncommonly high in silica ash, which is 92 to 95% pure [7]. the silica is left behind after cellulose and lignin are destroyed during combustion its high content of silica makes it a valuable material that can be used in a variety of industrial applications. The other compounds in rice ash include  $K_2O$ ,  $Al_2O_3$ ,  $CaO$ ,  $MgO$ ,  $Na_2O$ , and  $Fe_2O_3$ , whose amounts are less than one percent. Many factors affect the properties of ash, such as the temperature and time of combustion [9]. A controlled combustion environment and temperature enhance the quality of RHA, since the particle size and surface area of the ash are influenced by the conditions for combustion [7]. For the production of silica and silicates, RHA is a suitable raw material [8].

Temperature and time of burning are pivotal factors to determine whether silica remains amorphous, as in RHA, or becomes crystalline. The importance of amorphous silica obtained is that amorphous silica is active. Active silica is reactive and participates in reactions, while silica in its crystalline form is inactive and does not participate in reactions [10]. According to Della et al.[10], the ash produced by burning at 700 °C and for 6 hours contained the highest amount of silica compared to burning at 400, 500, and 600 °C, and burning for shorter periods. Ashes created below 773-873 K contain amorphous silica, but above this interval, the amorphous silica transforms into crystallized forms [11]. Kim et al.[12] investigated the silica crystalline forms of RHA produced by burning rice husk for 10 hours at temperatures of 300, 500, 700, and 900 °C by using energy X-ray diffraction analysis. Throughout all temperatures, a wide peak is observed between 15 and 35 degrees, which is characteristic of amorphous materials, illustrating how pyrolysis transformed crystalline cellulose into an amorphous, disordered, random composition. By increasing the temperature from 500 °C to 700 °C, there is a slight increase in peak intensity at 22°. Heating up to 900 °C increased the intensity of peaks at 22°, 36° and additional peaks were observed at 43°, 45°, 47° and 49°. Additional peaks in the pattern represent an example of the crystalline phase of silica. According to many researchers, amorphous ash is crystallized into cristobalite or tridymite at 900 °C [12]. When rice husk is burned under controlled conditions, it produces almost pure silica ash. By pre-treating silica with hydrochloric acid, sulfuric acid, or nitric acid before combustion, metallic impurities like Ferric, manganese, calcium, sodium, potassium, and magnesium can be removed, increasing the purity and color of the silica [13]. A study conducted by Kurama et al.[14] revealed that molarities of acid used during treatment did not have a linear correlation with silica percentage in RHA. However, the silica content of ash increases from 88.02 to 98.5% for 2 M HCl concentration. Also, by increasing the temperature of dissolving rice husk with acid (25, 60, 90°C), the purity of silica obtained from RHA increased. Examining the effect of rice husk dissolution time with acid solution, it was found that two hours of dissolution caused a complete dissolution of impurities and a significant increase in silica content. There were no detectable changes after 2 hours [14]. In the study conducted by Metori et al.[15], the effect of the type of acid used in the rice husk treatment process was investigated. When compared to untreated and other acid-treated rice husks, rice husks treated with HCl had the highest percentage of silica. Furthermore, XRD analysis for untreated and acid-treated ash after heat treatment at 800 °C for 2 hours, revealed that the acid-treated samples are amorphous and

Silica's amorphicity is not affected by the type of acid. In contrast, untreated husk is crystalline in with the crystal phase cristobalite [15]. According to Nayak et al.[2], blue silica gel has been synthesized using RHA and cobalt chloride salt. According to the X-ray energy diffraction analysis, the obtained silica gel shows its amorphous characteristics, and also the lack of washing the gel with deionized water causes the presence of crystalline peaks related to the sodium salt of the consumed acid or sodium silicate. The moisture adsorption rate of blue silica gel was lower than that of colorless silica gel. cobalt chloride salt reduces the adsorption capacity of silica gel because the pores of silica gel are partially filled by  $\text{COCl}_2$  depending on the concentration. The higher the concentration of  $\text{COCl}_2$  in the silica gel, the more likely the pores will be filled [2].

In another study by Nayak et al.[16], the color behavior of silica gels was investigated by immersing the hydrogel in different pH of cobalt chloride aqueous solution. It was observed that the blue color of the dry gel becomes significantly less with the lowering of pH. The gel with a pH equal to 6 has the most prominent color change behavior, from blue to pink. The gel with  $\text{pH} = 2$  is almost colorless in both dry and wet conditions, which may be due to the low absorption of cobalt on the gel because the isoelectric point of silica is close to  $\text{pH} = 2$ [16].

Considering that the primary material for the synthesis of silica gel is sodium silicate and the traditional method of sodium silicate synthesis involves high energy consumption and greenhouse gas production, the purpose of this study is to use RHA as the primary raw material for the synthesis of Sodium silicate and therefore silica gel. This study also uses Ferric salt instead of cobalt salt to turn silica gel orange because cobalt salt is toxic.

## **2.EXPERIMENTAL SECTION**

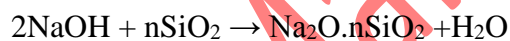
### **2.1. Chemicals**

Sodium silicate, sulfuric acid 98% (Dr. Mojallali company), hydrochloric acid 37% (Dr. Mojallali company), sodium hydroxide (Dr. Mojallali company) for alkali washing in the extraction of silica

from RHA, rice husk (collected from Langrod rice fields), Additionally,  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  were employed for coloring purposes.

## 2.2. Synthesis of silica gel using RHA

The procedure of silica gel synthesis using RHA is schematically shown in Fig 1. As can be seen in this figure, silica gel synthesis has two steps. Firstly, sodium silicate was extracted from the RHA. At a ratio of 10 grams to 100 cc, rice husk was added to 1 M hydrochloric acid solution. after 2 h stirring at 90 °C, Acid-treated rice husk was filtered and washed with distilled water until the pH of the output water reached 7. Finally, it was dried at 110 °C for one night and placed inside the furnace at 700 °C for 3 hours to turn into ash. Afterward, to produce sodium silicate from ash, at a ratio of 5 grams to 100 cc, ash was heated in 1 M sodium hydroxide solution for 2 h at 95°C inside an Erlenmeyer with an aluminum cap, according to the following reaction, liquid sodium silicate was obtained:



In the second stage, silica gel was synthesized with the sol-gel method. At first, the obtained sodium silicate was titrated with sulfuric acid until pH=6 to produce hydrogel. The hydrogel was aged for one night. Finally, the hydrogel was washed with distilled water and dried at 120°C for one night.

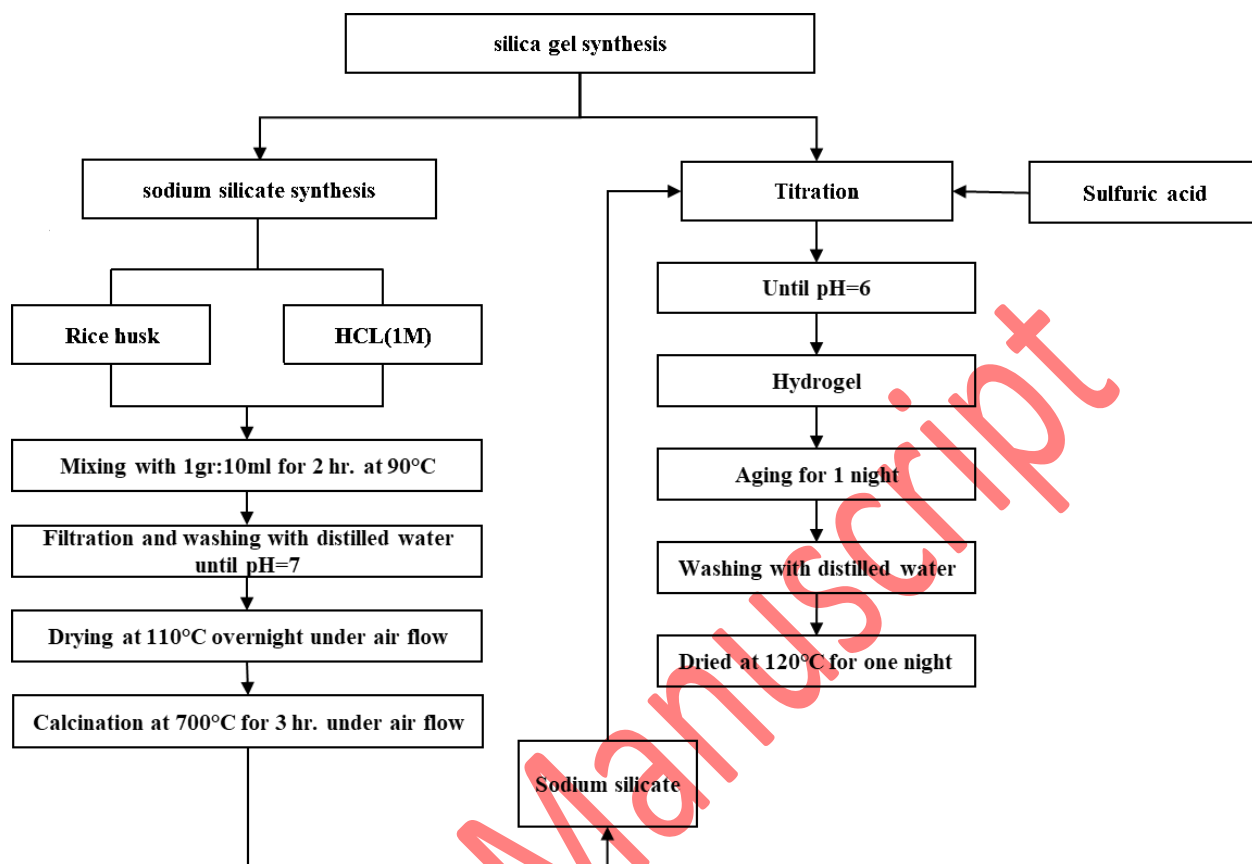


Fig 1. Schematic flow chart for preparation steps of silica gel

### 2.3. Colorization of silica gel

In this study, three methods were used to color silica gel produced from RHA. Methods include; immersing hydrogels in a Ferric salt solution by NAYAK and BERA[2], and dissolving Ferric salt in acid by HAFIZ et al.[17] immersing dry silica gel in Ferric salt solution by MORTON[3]. The coloring was also accomplished using two different types of Ferric salts:  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ .

### 2.4. Characterization of the Humidity Indicating Silica Gel from RHA

X-ray diffraction analysis was performed using an EQUINOX 3000 series device in a scanning range of 10-110 ( $2\theta$ ). The diffraction peaks were detected with High score plus software. BET (Brunauer, Emmett, and Teller) surface area analysis was accomplished using Japan's Belsorp mini device. FESEM analysis was performed using a MIRA3 device manufactured by Tuscan company.

## 2.5. Moisture adsorption capacity

To determine the moisture adsorption capacity of orange silica gel synthesized from rice husk ash and compare it with the moisture adsorption capacity of orange silica gel synthesized from commercial sodium silicate, 2 grams of each sample was placed inside a closed flask with a humid atmosphere (created by the solution Saturated salt (sodium chloride, saturated salt solution provides the required moisture)[18] and after 5 hours and 24 hours, the silica gels were weighed and the amount of moisture adsorbed by them was calculated according to Eq1:

$$X = \frac{x_2 - x_1}{x_1} \quad (\text{Eq1})$$

$X_1$ : weight of dry silicagel

$X_2$ : Weight of moisture silicagel

## 3.RESULTS AND DISCUSSION

### 3.1. Characterization of orange silica gel

#### 3.1.1. *Selecting the appropriate coloring method*

Fig 2 illustrates the orange silica gels synthesized using the three methods mentioned in Section 2.3. It can be seen that the orange silica gel synthesized by the Morton method is more transparent, similar to the commercial orange silica gel samples, and has a good color change from orange to white while silica gels obtained from Nayak and Hafiz are opaque and have no significant color change when exposed to moisture, as a result, Morten method is chosen as the preferred method.



Fig 2: synthesis of orange silica gel with a) Nayak's method, b) Hafiz's method, c) Morton's method

### 3.1.2. *Selecting the appropriate Ferric salt*

The orange silica gels were synthesized by Morton's method and with  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  are shown in fig 3, respectively. As can be seen, the orange silica gels using Ferric chloride salt showed a more suitable color change after being exposed to a humid atmosphere and adsorbing moisture between 20 and 25%.



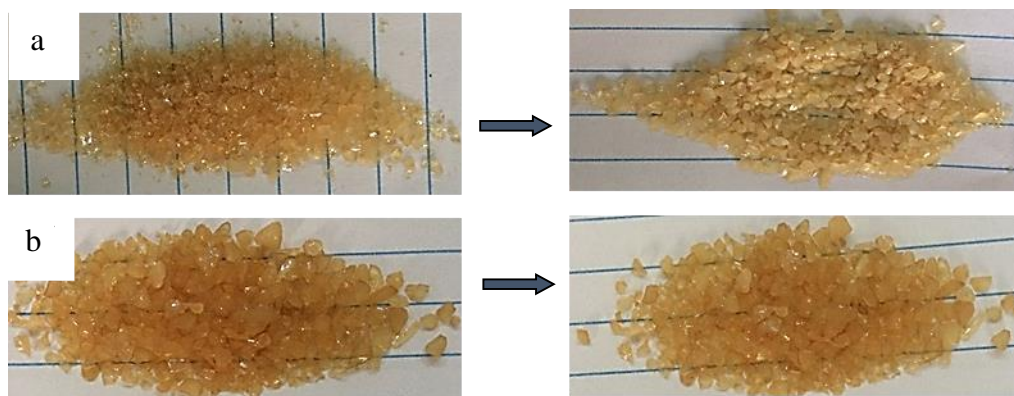


Fig 3: changing color of orange silica gel synthesized with different types of Ferric salt, a)  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , b)  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$

### 3.1.3. XRD analysis

The X-ray energy diffraction spectroscopy of two types of orange silica gel synthesized from RHA, unwashed and washed with distilled water 3 times, respectively, are shown in Fig4. According to the patterns presented, it can be seen that the presence of amorphous silica is quite evident in two samples, which is in the form of a broad peak at  $\Theta=22^\circ$ . Further, other peaks are observed in a sample of unwashed, which are caused by sodium salts such as thenardite (sodium sulfate), at 19.27, 28.03, 32.9, 32.5, 34.2, and 49 degrees. Based on the fact that no other peak is observed in the washed sample, it can be concluded that  $\text{FeCl}_3$  is not detected because of its low concentration. According to the study, for the crystalline composition of the transition metal, the content of at least 1% [19] by weight is sufficient to create sharp peaks in the X-ray diffraction pattern. Here too, the amount of salt used compared to silica gel is approximately one percent by weight, but there is no peak related to its presence.

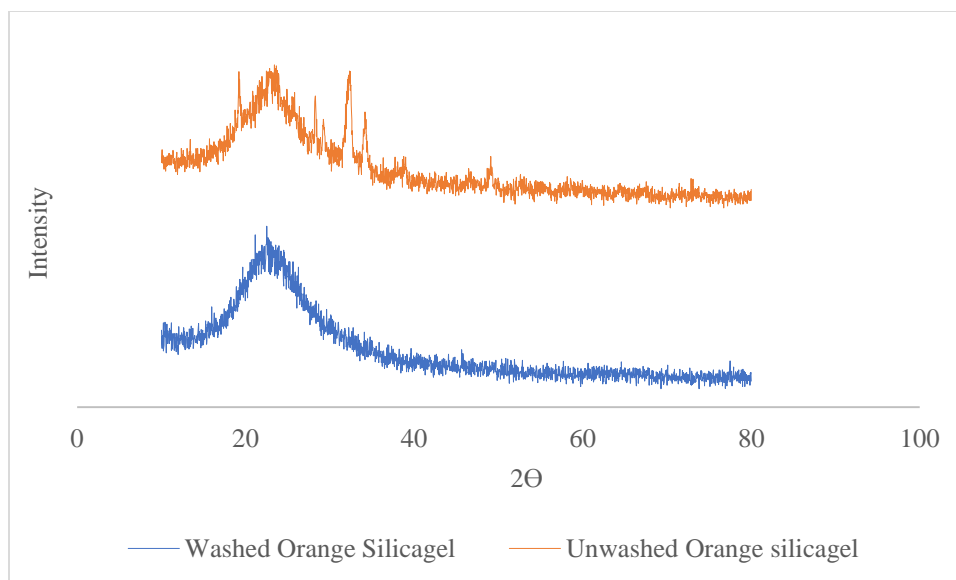


Fig 4: XRD pattern of unwashed and washed orange silica gel based on RHA

#### 3.1.4. BET analysis

Fig 5 shows the  $N_2$  adsorption-desorption of orange silica gel, orange silica gel synthesized from RHA with an aging time of 24 hours, orange silica gel synthesized from industrial sodium silicate with an aging time of 24 hours, and orange silica gel synthesized from industrial sodium silicate with an aging time 2 hours, respectively. Based on the IUPAC classification, all three samples follow the type IV isotherm, which indicates multilayer adsorption on mesoporous solids. The most significant characteristic of this isotherm is the hysteresis loop, which is caused by condensation on the holes. At high relative pressures, limited adsorption results in a flattening of the isotherm, indicating that all voids have been filled. Hysteresis loops of samples are classified by IUPAC as hysteresis type H1, which describes porous materials with holes that are cylindrical-shaped, relatively large, and easily connected.

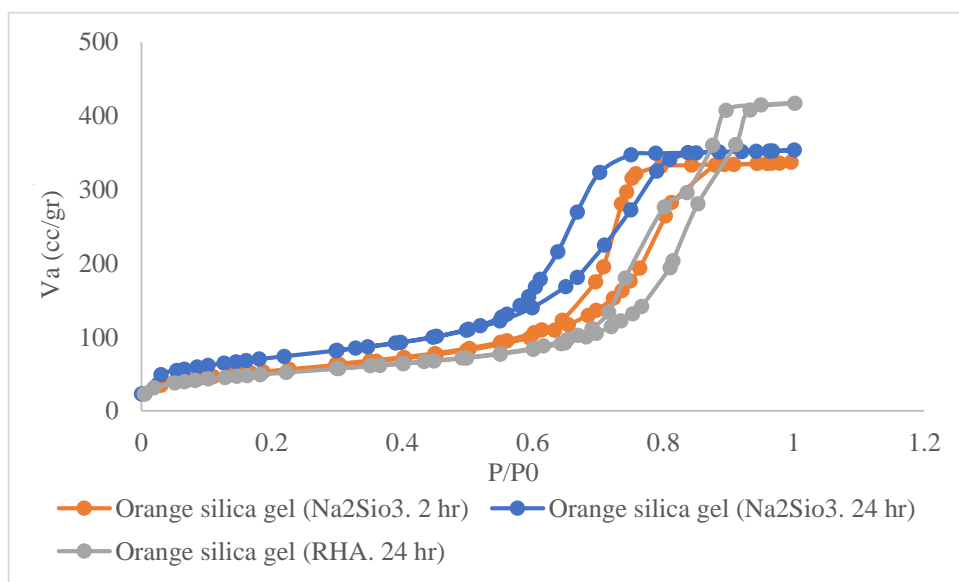


Fig 5: N<sub>2</sub> adsorption-desorption of a) orange silica gel based commercial sodium silicate and RHA

Fig 6 is related to the examination of the size distribution of the holes in the samples. The observations indicate that the size distribution of holes in all three samples is within the range of 2-50 nm due to the mesoporous character of the samples. The presence of mesopores in the silica gel can lead to a wider range of hole sizes in it, resulting in a broader size distribution of holes. On the other hand, if the mesopores are uniformly distributed in the gel, it can lead to a more uniform size distribution of holes. Overall, the mesoporous character of the samples plays a significant role in determining the size distribution of holes in the synthesized orange silica gel, which can influence its properties such as BET surface area [20]. The peak width in the pore size distribution curves indicates the uniformity of the pore size distribution, which means that the smaller the peak width, the more uniform the hole size distribution. The silica gel sample synthesized from rice husk ash has a larger peak width compared to the samples synthesized from commercial sodium silicate, so the distribution of its pore size is more uneven, and this factor causes its surface area to decrease compared to the other two samples. The area under the pore size distribution curves is according to the following pattern:

Orange silica gel (Na<sub>2</sub>SiO<sub>3</sub>. 24 hr) > Orange silica gel (Na<sub>2</sub>SiO<sub>3</sub>. 2 hr) > Orange silica gel (RHA. 24 hr)

This Pattern consists with the total surface area as seen in table1. The surface area also shows the ability of the adsorbent in the amount of adsorption [21].

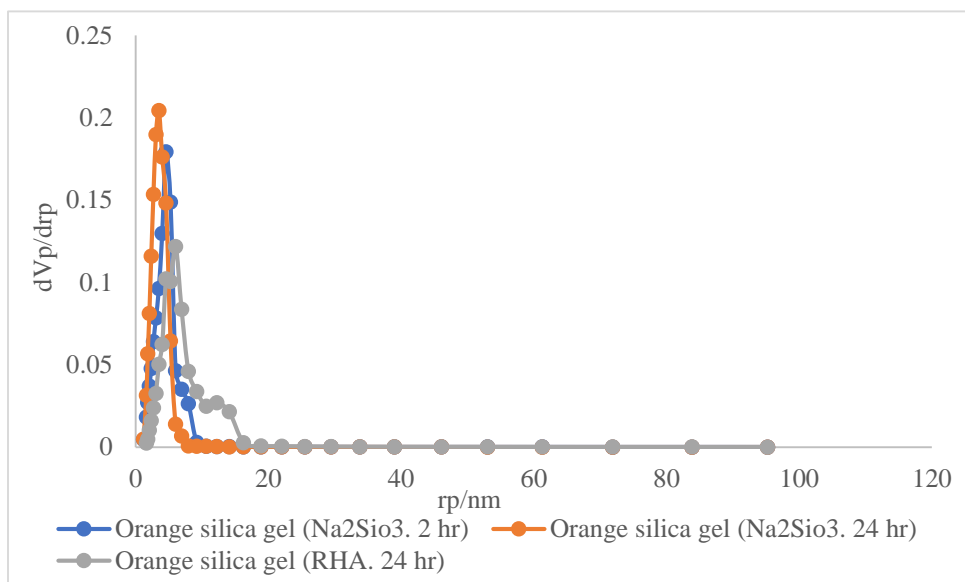


Figure 6: Pore size distribution of orange silicagel based commercial sodium silicate and RHA

Table 1 shows the physical properties of the synthesized orange silica gels. The Orange silica gel synthesized from RHA has a lower surface area rather than other ones. In the synthesis of silica gel, When acid interacts with a sodium silicate solution, it forms orthosilicic acid, an intermediate product of hydrolysis. This substance is highly unstable and quickly undergoes self-condensation to create a colloidal polymer known as silica sol. Upon further standing, the silica sol undergoes additional condensation, resulting in the formation of a gel [17], in addition in the fig 7c, white particles can be seen, which can be attributed to sodium silicate powder (which is usually removed by further washing and washing with hot water), which shows that the total sodium silicate has not interacted with the acid, as a result of which the surface area decreases. The longer the aging time, the greater the surface area of the gel produced. The longer the aging time, the more silica monomers are connected, so the surface area of the produced silica gel increases.

Table 1: Physical properties of synthesis orange silicagel

<i>sample</i>	<i>S<sub>BET</sub></i> ( <i>m<sup>2</sup>/gr</i> )	<i>S<sub>micro</sub></i> ( <i>m<sup>2</sup>/gr</i> )	<i>S<sub>meso</sub></i> ( <i>m<sup>2</sup>/gr</i> )	<i>Pores Diameter</i> <i>Average(nm)</i>	<i>r<sub>p, pic</sub> (nm)</i>	<i>V<sub>p</sub>(cm<sup>3</sup>/gr)</i>
<i>Orange silica gel (Na<sub>2</sub>SiO<sub>3</sub>. 24 hr)</i>	<i>248.86</i>	<i>128.81</i>	<i>120.05</i>	<i>8.781</i>	<i>3.53</i>	<i>0.56</i>
<i>Orange silica gel (Na<sub>2</sub>SiO<sub>3</sub>. 2 hr)</i>	<i>199.56</i>	<i>105.371</i>	<i>94.189</i>	<i>10.442</i>	<i>4.61</i>	<i>0.53</i>
<i>Orange silica gel (RHA. 24 hr)</i>	<i>179.86</i>	<i>82.343</i>	<i>97.517</i>	<i>14.289</i>	<i>6.06</i>	<i>0.64</i>

### 3.1.5. FE-SEM analysis

The FE-SEM images of RH, RHA, and orange silica gel synthesized using RHA are shown in Fig 7 a, b, and c. As can be seen in Fig 7a, a regular strip structure is evident in the morphology of the rice husk. The Outer epidermis consists of parallel and symmetrical ridges made up of silica on cellulose. the entire surface of the epidermis is covered with many small grains. Silica is generally present in all parts of the rice husk, but its distribution varies. Figure 7b shows that rice husk morphology changes after chemical and heat treatment. In the epidermis, small grains have disappeared. A soft, porous, layered structure of the RHA was obtained as a result of the combustion of the carbonaceous compounds in the RH at a temperature of 700°C. A view of the overall morphology and mass aggregation with the geometry of spherical particles of orange silica gel synthesized from RHA with a magnification of 500 nm is shown in Fig 7c. There is no difference in particle size distribution, and the homogeneous color of the silica gel surface is a result of the uniform distribution of Ferric chloride (III) salt indicator in its texture. A view further from the silica gel surface can be seen in Fig 7d. The presence of white particles can be due to sodium silicate powder, most of which has been destroyed by washing. The rest of these particles can be removed by washing more and using hot distilled water.

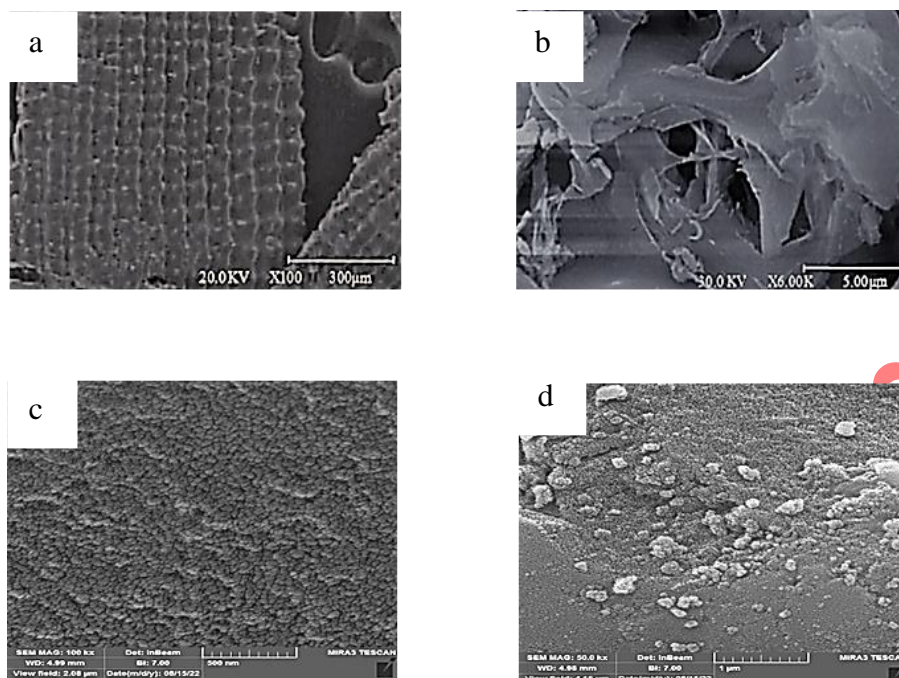


Fig 6: FESEM images of a) RH, b)RHA, orange silica gel based on RHA c) 500 nm d) 1 micrometer

### 3.1.6.EDX analysis

Fig 8 shows the EDX analysis of orange silica gel synthesized from RHA. To prove the orange color of silica gel and the presence of constituent atoms of the Ferric (III) chloride salt indicator, EDX analysis was performed. In addition to the high-intensity peak of silica, there are moderate peaks associated with the presence of Ferric and chloride atoms. These metals confirm their presence on the surface of silica gel.

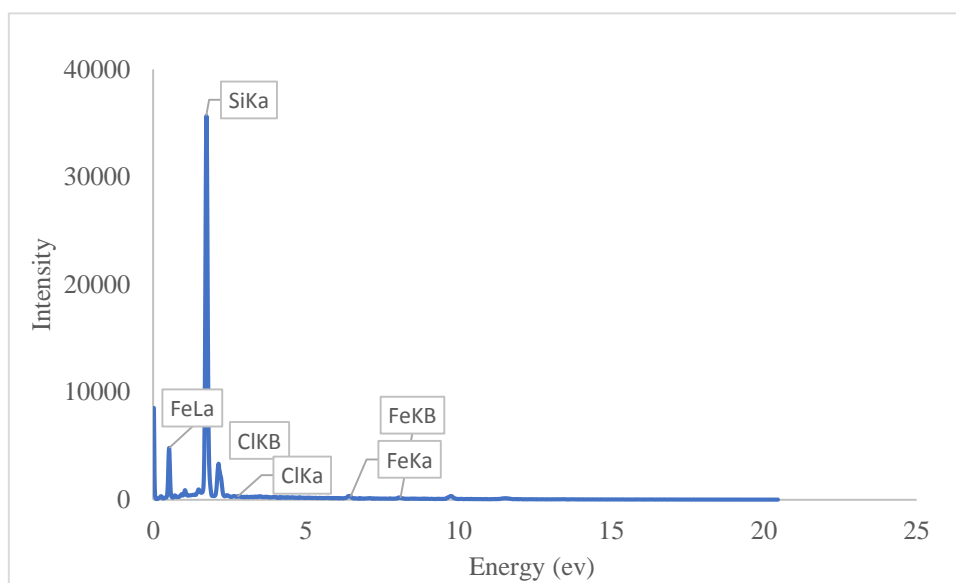


Fig 7: EDX analysis of orange silica gel-based RHA and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

### 3.1.7. Determination of adsorption capacity

In Table 1, it is evident that Orange silica gel ( $\text{Na}_2\text{SiO}_3$ , 24 hr) exhibits a greater BET surface area in comparison to Orange silica gel (RHA, 24 hr), leading to a higher adsorption capacity. In Table 2, it is worth noting that Silica gel synthesized from RHA shows a slight variance in moisture adsorption as compared to Silica gel synthesized from Commercial sodium silicate, which can be attributed to the abundance of mesopores in this adsorbent ( $S_{\text{meso}}/S_{\text{micro}}=1.18$ ). Consequently, sodium silicate synthesized from rice husk ash can serve as a viable alternative to industrial sodium silicate, offering not only eco-friendliness but also adequate adsorption capability.

Table 2: Moisture adsorption capacity of synthesized silica gels

Type of silicagel	Adsorption capacity after 5 hr	Adsorption capacity after 24 hr
Orange silica gel (RHA, 24 hr)	4.5%	24%
Orange silica gel (Na <sub>2</sub> SiO <sub>3</sub> , 24 hr)	5.5%	27.5%

#### 4. CONCLUSIONS

By accomplishing thermal and chemical treatment on rice husk, it can be turned into a suitable raw source for the production of sodium silicate and consequently silica gel. Immersion of silica gel in salt solution according to Morton's method is more suitable for coloring iron (III) chloride salt and shows a more impressive color change than ammonium iron (III) sulfate salt when the silica gel is exposed to a humid atmosphere. The XRD analysis confirmed the amorphous structure of the orange silica gel synthesized from RHA. Although the orange silica gel synthesized from RHA has a lower surface area than the sample synthesized from commercial sodium silicate, its richness in mesopores has caused it to have an acceptable moisture absorption capacity. Considering that it is produced from a natural waste material, it can serve as a viable option for an environmentally friendly adsorbent material.

#### References



- [1]. Korotcenkov, G., Handbook of Humidity Measurement, Volume 3: Sensing Materials and Technologies. 2020: CRC Press.  
<https://doi.org/10.1201/9781351056502>
- [2]. Nayak, J. and J. Bera, A simple method for production of humidity indicating silica gel from rice husk ash. Journal of Metals, Materials and Minerals, 2009. 19(2).  
<https://jmmm.material.chula.ac.th/index.php/jmmm/article/view/230>
- [3]. Moreton, S., Silica gel impregnated with iron (III) salts: A safe humidity indicator. Material Research Innovations, 2002. 5: p. 226-229.  
<https://doi.org/10.1007/s10019-002-0177-3>
- [4]. Aranovich, L., N.N. Akinfiev, and M. Golunova, Quartz solubility in sodium carbonate solutions at high pressure and temperature. Chemical Geology, 2020. 550: p. 119699.  
<https://doi.org/10.1016/j.chemgeo.2020.119699>
- [5]. Foletto, E.L., et al., Conversion of rice hull ash into soluble sodium silicate. Materials Research, 2006. 9: p. 335-338.  
<https://doi.org/10.1590/S1516-14392006000300014>
- [6]. Steven, S., E. Restiawaty, and Y. Bindar, Operating variables on production of high purity bio-silica from rice hull ash by extraction process. J Eng Technol Sci, 2022. 54(3): p. 220304.  
<https://doi.org/10.5614/j.eng.technol.sci.2022.54.3.4>
- [7]. Siddique, R. and M.I. Khan, Supplementary cementing materials. 2011: Springer Science & Business Media.  
<https://doi.org/10.1007/978-3-642-17866-5>
- [8]. Prasad, R. and M. Pandey, Rice husk ash as a renewable source for the production of value added silica gel and its application: an overview. Bulletin of Chemical Reaction Engineering and Catalysis, 2012. 7(1): p. 1-25.  
<https://doi.org/10.9767/bcrec.7.1.1216.1-25>
- [9]. Sigalingging, R., E. Susanto, and S. Pangabea. The effect of rice husk mass on temperature and characteristics of its ash using a pyrolysis equipment organic-inorganic waste. in IOP Conference Series: Earth and Environmental Science. 2020. IOP Publishing.  
<https://doi.org/10.1088/1755-1315/454/1/012043>
- [10]. Della, V.P., I. Kühn, and D. Hotza, Rice husk ash as an alternate source for active silica production. Materials letters, 2002. 57(4): p. 818-821.  
[https://doi.org/10.1016/S0167-577X\(02\)00879-0](https://doi.org/10.1016/S0167-577X(02)00879-0)
- [11]. Yalcin, N. and V. Sevinc, Studies on silica obtained from rice husk. Ceramics international, 2001. 27(2): p. 219-224.

[https://doi.org/10.1016/S0272-8842\(00\)00068-7](https://doi.org/10.1016/S0272-8842(00)00068-7)

[12]. Kim, M., et al., Comparison of the adsorbent performance between rice hull ash and rice hull silica gel according to their structural differences. *LWT-Food Science and Technology*, 2008. 41(4): p. 701-706.

<https://doi.org/10.1016/j.lwt.2007.04.006>

[13]. Bakar, R.A., R. Yahya, and S.N. Gan, Production of high purity amorphous silica from rice husk. *Procedia chemistry*, 2016. 19: p. 189-195.

<https://doi.org/10.1016/j.proche.2016.03.092>

[14]. Kurama, H. and S. Kurama. The effect of chemical treatment on the production of active silica from rice husk. in 18th international mining congress and exhibition of Turkey-IMCET. 2003.

[ISBN 975-395-605-3](https://doi.org/10.1016/j.proche.2016.03.092)

[15]. Matori, K., et al., Producing amorphous white silica from rice husk. *MASAUM Journal of Basic and Applied Sciences*, 2009. 1(3): p. 512-515.

[https://www.researchgate.net/publication/236839923\\_Producing\\_Amorphous\\_White\\_Silica\\_from\\_Rice\\_Husk](https://www.researchgate.net/publication/236839923_Producing_Amorphous_White_Silica_from_Rice_Husk)

[16]. Nayak, J. and J. Bera, Preparation of an efficient humidity indicating silica gel from rice husk ash. *Bulletin of Materials Science*, 2011. 34: p. 1683-1687.

<https://doi.org/10.1007/s12034-011-0377-9>

[17]. Ali, H.A., et al., Synthesis of quality silica gel; Optimization of parameters. *Chemical Engineering and Technology Punjab Lahore University*, 2009.

[https://www.researchgate.net/publication/260982001\\_Synthesis\\_of\\_Quality\\_Silica\\_GelOptimization\\_of\\_parameters](https://www.researchgate.net/publication/260982001_Synthesis_of_Quality_Silica_GelOptimization_of_parameters)

[18]. Bourgault, C., et al., Experimental Determination of Moisture Sorption Isotherm of Fecal Sludge. *Water*, 2019. 11(2): p. 303.

<https://doi.org/10.3390/w11020303>

[19]. Balköse, D., et al., Dynamics of water vapor adsorption on humidity-indicating silica gel. *Applied surface science*, 1998. 134(1-4): p. 39-46.

[https://doi.org/10.1016/S0169-4332\(98\)00244-X](https://doi.org/10.1016/S0169-4332(98)00244-X)

[20]. Wang, Q., et al., Pore size control of monodisperse silica particles by dual template sol-gel method. *Journal of Sol-Gel Science and Technology*, 2020. 94: p. 186-194.

<https://doi.org/10.1007/s10971-019-05152-7>

[21]. Tan, Y.H., et al., Surface area and pore size characteristics of nanoporous gold subjected to thermal, mechanical, or surface modification studied using gas adsorption isotherms, cyclic voltammetry, thermogravimetric analysis, and scanning electron microscopy. *Journal of materials chemistry*, 2012. 22(14): p. 6733-6745.

<https://doi.org/10.1039/c2jm16633j>

Accepted Manuscript