

A Study on the Water Absorption Efficiency of Porous Silica Gel Prepared from Rice Husk Ash

U. Parida, T.K. Bastia and B.B. Kar*

Department of Chemistry, School of Applied Sciences
KIIT University, Bhubaneswar
✉ karpublishations@gmail.com

Received April 17, 2016; revised and accepted December 2, 2016

Abstract: In the present study, an attempt has been made to prepare porous silica gel from rice husk ash and use it as a water absorption medium. In the process, raw rice husk was calcined at an elevated temperature and turned into ash. The ash sample was characterized and then treated with concentrated alkali medium to using dielectric heating in microwave. The product thus obtained is sodium silicate, which when treated with concentrated sulphuric acid leads to the formation of silica gel. The process parameters are optimized and the gel thus obtained was subjected to the water absorption study and results are represented.

Key words: Silicagel, ash, absorption, water.

Introduction

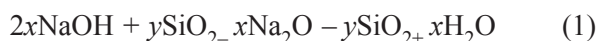
Rice husk (RH) is one of the by-products obtained during milling of rice. It has been estimated that the annual rice husk production in India is 12 million tonnes but in world rice production is approximately 645 million tonnes. Researchers analyzed that rice husk produced from cellulose and sugar contents are low, so it cannot be used as cattle food. In certain countries, it is sometimes used as a fuel for parboiling paddy in the rice mills and to power steam engines (Real et al., 1996; Kalapathy et al., 2000, 2002; Foletto et al., 2006; Rungronmitchai and Supjaroenkul, 2009). When rice husk is subjected to heating at a temperature 750°C one gets rice husk ash, which is the cause of environmental pollution. It would be beneficial to the environment to recycle the waste to produce eco-material having high end value. Rice husk ash is the major source of silica (95-98%) and can be an economically viable raw material for production of silica gel and powders.

Silica gel is the amorphous (non-crystalline) form of SiO_2 . It has a large surface area ($\sim 500 \text{ m}^2/\text{g}$), which allows silica gel for showing great water absorption ability via chemisorptions on Si-OH groups. Silica gel is a granular, vitreous, porous form of silicon dioxide obtained from sodium silicate ((Novotny et al., 1991; Yalcin and Sevinc, 2001; Pijarn et al., 2010; Rao; 1980). It is a three dimensional network. Silica gel is used as desiccant having pore size 2.4 nm and strong affinity for water molecules.

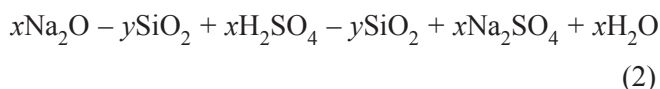
Silica gel from rice husk ash can be prepared by two methods. The first method is thermal treatment with temperatures 1400°C. This method requires high temperature. The second way of preparation is leaching by acid or acidic solutions (Kalapathy et al., 2000, 2002) and then neutralization by acid to produce silica gel. The second method consumes low energy, and is cost-effective compared to the current melting method (Sharma et al., 1984; Patel et al., 1987; Patel and Karera, 1991; Ding, 1992; Krishnarao and Godkhindi,

*Corresponding Author

1992; Chandrasekhar et al., 2003). Besides this advantage, the process may decrease CO₂ emission due to the current manufacture of sodium silicate from the reaction of Na₂CO₃ and SiO₂. The second method was performed by microwave heating. Because microwave heating is dielectric heating, which directly interacts with the molecules and usually gives higher reaction efficiency with traces amount of by product. It is useful to calculate the SiO₂/Na₂O ratio of the system. The reaction between NaOH and SiO₂ of rice husk ash can be expressed as the following reaction (Krishnarao, and Godkhindi, 1992; Ismail and Waliuddin, 1996; Proctor et al., 1995; Proctor and Palaniappan, 1990; Sun and Gong, 2001; Krishnarao et al., 2001).



And silica gel can be produced by neutralization of the obtained sodium silicate (xNa₂OySiO₂) as follows:



Material and Method

For the above study, rice husk have been imported from the nearby village and is subjected to burning to get rice husk ash. The physicochemical composition of the ash sample is given in Table 1. Before burning the husk, the material has been dried up at 150 °C for 12 hrs to extract absorbed moisture. The ash thus obtained is being used as the raw material. Reliance chemical grade (analytical grade) chemicals were used for the study. The microwave used for the study is Electro

Table 1: Physico-chemical composition of rice husk ash

<i>Physical properties</i>	
Length	7.2 mm
Width	3.3
Thickness	0.6
Bulk density	118 kg/m ³
Moisture content	65%
<i>Chemical properties</i>	
SiO ₂	65.8%
Fe ₂ O ₃	1.00%
CaO	2.36%
Al ₂ O ₃	5.12%
MgO	2.08%
LOI	23.64%

hub EMM 2006 model whose power could vary in the range of 300-800 Watt. The reactions are to be carried out with container which should be thermo-resistance fibre container (Tang and Wang, 2005; Bhattacharya and Wu, 1989; Boaterg, 1990; Huang et al., 2001). The rice husk along with the dilute activator is heated at different microwave irradiation at frequency range of 200 W, 400 W, 600 W and 800 W by heating the microwave at 100 °C for 10 mins.

Preparation of Silica Gel by Microwave Heating

Rice husk = 10 gms

Sodium hydroxide (as activating material)

Sodium hydroxide solution is made of different concentrations

3.0 M = 82.5 ml

2.0 M = 165 ml

1.0 M = 330 ml

0.5 M = 660 ml

These concentrations provide a minimum content of NaOH to synthesis sodium silicate with SiO₂:Na₂O = 1. Silica gel is prepared by taking rice husk as raw material because of its availability, cost effective and to be eco-friendly. Ten gram of rice husk ash was calcined at 750°C for four hours. Reflux the solution with 2M sodium hydroxide solution for two hours. Then the solution was filtered through Whatmann filter paper and the residue containing carbon was washed. Boil the filtrate known as sodium silicate and cool the solution at a room temperature. The solution was treated with 2N H₂SO₄ with constant stirring to make pH 7. Then the solution was heated by microwave irradiation 100 W, 300 W, 450 W, 600 W, 800 W, 900 W for 10 minutes. The solution was filtered and residue was washed with de-ionized water. The filtrate was cooled at room temperature. It was kept in incubator for 48 hours to get silica gel. Silica gel was dried at 100°C for 24 hours. Silica gel was treated with 1M hydrochloric acid for XRD Analysis. The amount of silica gel generated using different alkali medium with time and variable capacity of microwave roasting is represented in Table 2.

Result and Discussion

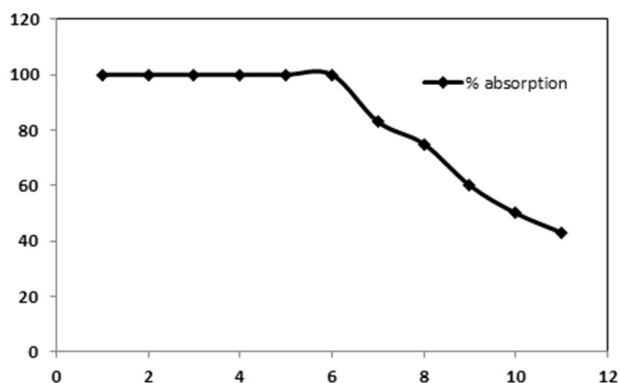
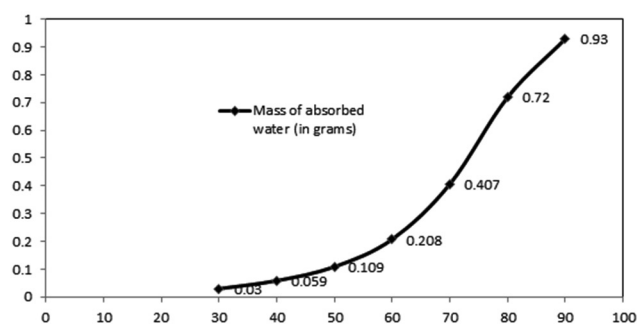
Mechanism of Absorption

When a substance is uniformly distributed throughout the bulk of another substance, it occurs at the uniform rate throughout the body of the material. Silica gel is

Table 2: Synthesis of silica gel with variable parameters

<i>NaOH</i>	<i>Time</i> (min)	<i>800 W</i>	<i>600 W</i>	<i>400 W</i>	<i>200 W</i>
0.5M = 660 ml	5	0.120	0.084	0.017	0.008
	10	0.175	0.098	0.043	0.017
	15	0.240	0.120	0.067	0.022
	20	0.280	0.144	0.083	0.028
1.0M = 330 ml	5	1.410	1.123	0.091	0.070
	10	2.140	1.274	0.807	0.378
	15	2.780	1.343	1.008	0.642
	20	3.490	2.180	1.342	0.980
2.0M = 165 ml	5	1.890	1.524	1.290	1.004
	10	3.180	2.027	1.178	1.096
	15	6.820	3.180	2.014	1.106
	20	9.472	5.332	2.108	1.478
3.0M = 82.5 ml	5	4.76	6.556	2.46	1.502
	10	8.44	7.689	3.05	1.599
	15	13.61	8.732	3.92	1.866
	20	18.48	9.940	4.088	1.998

hygroscopic material which shows that they readily absorb moisture from their surrounding environment. Silica gel attracts moisture and dissolves the process into watery brine and it attracts moisture through the process of absorption in which water was attracted to glassy surfaces through its internal pore. The data obtained are given in Figure 1, which represents the volume of water against the mass of water getting absorbed in the absorbent medium. The data also revealed that water absorption is directly proportional to the surface absorption. Silica gel shows water absorption effectively due to its non-corrosive nature and the crystallization makes it sensitive to shocks. The amount of water absorbed by this silica gel micro porous solid is directly proportional to the mass of the sample, temperature of the operating medium and pressure of the system. The data are plotted in Figure 2. The supporting parameters which contribute to water absorption are the nature

**Figure 1: The percentage absorption of water on silica gel.****Figure 2: Mass of absorbed water (in grams) vs volume of water in ml.**

of the absorbent (silica gel) and liquid to solid ratio. During the study, all the above mentioned parameters have been optimized.

In order to study, the silica gel (powdered form) is taken and water is added to it at different ratio. The silica gel is taken with variable particle sizes and experiments were carried out at room temperature. The data revealed that during preparation about 75% of the silica gel were found to be present in the fraction of +250 μm . The silica gel of variable particle size is put into the beaker at different quantity and water absorption capability of the silica gel is being studied under variable conditions. The data obtained are given in Table 3. The study revealed that dry silica gel has water absorption capacity almost three times more than the wet silica gel produced without drying.

Table 3: Volume of water absorbed in saturated and dry silica gel

<i>Mass of saturated silica gel (kg)</i>	<i>Maximum water absorption (%)</i>	<i>Mass of dry silica gel (kg)</i>	<i>Maximum mass of absorption (%)</i>
5.817	13.7	4.454	30.6
1	2.355	1	6.87

Conclusions

The silica gel is a highly porous solid produced when rice husk ash is treated with sodium hydroxide followed by sulphuric acid and sodium silicate. It consists of porous particles with diameters varying between 2 and 20 nm. Each 1 m^3 of silica gel contains pores that if added would account for a surface area of about $2.8 \times 10^7 \text{ m}^2$. The silica-gel has a great capacity to absorb vapour water of around 35 to 40% of its dry mass, along with low regeneration temperatures. Silica gel is safe, non-corrosive, cheap, and can be synthesized in abundance. It has been widely used due

to its great adsorption capacity and chemical stability. The knowledge of the pair behaviour in different conditions of temperature and pressure and the relation to the absorbate or absorbent mass is essential in the project of refrigeration systems by absorption. In the characterization of the pair silica-gel/water in this work, the maximum experimental absorption was found to be 30.6%.

References

- Bhattacharya, S.C. and W. Wu (1989). Fluidized bed combustion of rice husk for disposal and energy recovery. *Energy from Biomass and Wastes*, **12**: 591-601.
- Boaterg, S.D.A. (1990). Incineration of rice hull for use as a cementitious material. *Cem. Concr. Res.*, **20(5)**: 795-802.
- Chandrasekhar, S., Satyanarayana, K.G., Pramada, P.N. and P. Raghavan (2003). Review Processing, properties and applications of reactive silica from rice husk—An overview. *J. Mat. Sci.*, **38**: 3159-3168.
- Ding, M. (1992). Rice husk silicon and its applications. *Inorg. Chem. Ind.*, **24(6)**: 36-42.
- Foletto, E.L., Gratieri, E., de Oliveira, L.H. and S.L. Jahn (2006). Conversion of rice hull ash into soluble sodium silicate. *Mater. Res.*, **9(3)**: 335-338.
- Huang, S., Jing, S., Wang, J., Wang, Z. and Y. Jin (2001). Silica white obtained from rice husk in fluidized bed. *Powder Tech.*, **117**: 232-238.
- Ismail, Muhammad Shoaib and A.M. Waliuddin (1996). Effect of rice husk ash on high strength concrete. *Const. and Build. Mat.*, **10**: 521-526.
- Kalapathy, U., Proctor, A. and J. Shultz (2000). A simple method for production of pure silica from rice hull ash. *Bioresour. Technol.*, **73(3)**: 257-262.
- Kalapathy, U., Proctor, A. and J. Shultz (2002). An improved method for production of silica from rice hull ash. *Bioresour. Technol.*, **85(3)**: 285-289.
- Krishnarao, R.V. and M. Godkhindi (1992). Effect of Si additions on the formation of SiC whiskers from rice husks. *Ceram. Int.*, **18**: 185.
- Krishnarao, R.V. and M.M. Godkhindi (1992). Distribution of silica in rice husks and its effect on the formation of silicon carbide. *Ceram. Int.*, **18**: 243-249.
- Krishnarao, R.V., Subrahmanyam, J. and T.J. Kumar (2001). Studies on the formation of black particles in rice husk silica ash. *J. Eur. Ceram. Soc.*, **21(1)**: 99-104.
- Novotny, R., Hoff, A. and J. Schuertz (1991). Process for hydrothermal production of sodium silicate solutions. US Patent no 545000933.
- Patel, M. and A. Karera (1991). SiC whisker from rice husk: Microscopic study. *Powder Metall. Int.*, **23(1)**: 30-32.
- Patel, M., Karera, A. and P. Prasanna (1987). Effect of thermal and chemical treatments on carbon and silica contents in rice husk. *J. Mater. Sci.*, **22**: 2457-2464.
- Pijarn, N., Jaroenworuluck, A., Sunsaneeyametha, W. and R. Stevens (2010). Synthesis and characterization of nanosized-silica gels formed under controlled conditions. *Powder Technol.*, **203(3)**: 462-468.
- Proctor, A. and S. Palaniappan (1990). Adsorption of soy oil free fatty acids by rice hull ash. *J. Am. Oil Chem. Soc.*, **67**: 15-17.
- Proctor, A., Clark, P.K. and C.A. Parker (1995). Rice hull ash adsorbent performance under commercial soy oil bleaching conditions. *J. Am. Oil Chem. Soc.*, **72**: 459-462.
- Rao, V.M.H.G. (1980). Utilization of rice husk—A preliminary analysis. *J. Sci. Ind. Res.*, **39**: 495-515.
- Real, C., Alcala, M.D. and J.M. Criado (1996). Preparation of Silica from rice husks. *J. Am. Ceram. Soc.*, **79(8)**: 2012-2016.
- Rungrodnimitchai, S. and U. Supjaroenkul (2009). Modification of rice straw for heavy metal ion adsorbents by MW heating. Prague Meetings on Macromolecules. Prague, Republic of Czech.
- Sharma, N.K., Williams, W.S. and A. Zangvil (1984). Formation and structure of silicon carbide whiskers from rice hulls. *J. Am. Ceram. Soc.*, **67**: 715-720.
- Sun, L. and K. Gong (2001). Review, silicon-based materials from rice husks and their applications. *Ind. Eng. Chem. Res.*, **40**: 5861-5877.
- Tang, Qi and Tao Wang (2005). Preparation of silica aero-gel from rice hull ash by supercritical carbon dioxide drying. *J. Supercritical Fluids*, **35**: 91-94.
- Yalcin, N. and V. Sevinc (2001). Studies on silica obtained from rice husk. *Ceram. Inter.*, **27**: 219-224.