Yadanabon University Research Journal, 2019, Vol-10, No.1

A Study on Synthesis of Silica Nanoparticles from Rice Straw

Thida Wynn*

Abstract

Rice straw ash (RSA) is the waste product after burning the rice straws as fuel. In order to develop the waste material recycling process, the silica nanoparticle was prepared from environmental agro waste. This research intended to utilize the waste material rice straw as a valuable material silica nanoparticles. In this study the rice straw sample was collected from Hmawbi Township, Yangon Region. At first, rice straw ash was prepared from rice straw by calcination at 650 °C for 3 h. A chemical method of dissolution-precipitation was applied to produce silica nanoparticles from rice straw ash. The rice straw powder was characterized by using XRD, FTIR, SEM and TG-DTA. Characterization of rice straw ash, silica powder and silica nanoparticles were carried out by the above techniques except TG-DTA. XRD pattern of the silica nanoparticles confirms the amorphous nature of silica. By using Scherrer equation, the crystalline size of the prepared silica nanoparticles was 46.70 nm. Infrared spectral data support the present of hydrogen bonded silanol groups and siloxane groups on the silica. SEM micrograph of silica nanoparticles showed porous structure.

Keywords: rice straw ash, silica nanoparticles, calcination, dissolution- precipitation method, characterization

Introduction

Rice straw, one of the agricultural waste materials, can be economically viable as raw material for the production of silica gel and powders. Rice straw is rich in silica, which is known to be very effective in enhancing the strength and durability of concrete. Rice straw ash contains 76.8 % of silica and small amount impurities (Ca, Mg, K, Na, Al). Commercially, it is the source of elemental silicon and is used in large quantities as a constituent of building materials. It is primarily a raw material for many white ceramics such as earthenware, stoneware, porcelain as well as industrial Portland cement. Nanotechnology refers to understanding and control of matter at dimensions of roughly 1 to 100 nm. Nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale. Nanotechniques are potential applications and just in agriculture but also in medicine, electronic, information technology and environmental monitoring and remediation. It is also applied to prevent waste in agriculture. Silica nanoparticles have a wide range of applications, including ceramic production, chromatography, electrical and thermal insulations, and catalysis. Many common construction materials contain silica including, for example, brick, cement, concrete, stone and sand.

Aim and Objectives

Aim

- To prepare and characterize the silica nanoparticles from the rice straw

Objectives

- To prepare rice straw powder from rice straw
- To characterize the prepared rice straw powder by using TG-DTA, XRD, FTIR, SEM techniques
- To prepare rice straw ash from rice straw
- To characterize the prepared rice straw ash by using XRD, FTIR, SEM techniques
- To prepare silica powder from rice straw ash by precipitation method
- To characterize the prepared silica powder by using XRD, FTIR, SEM techniques

* Associate Professor, Dr, Department of Chemistry, Yadanabon University

- To prepare silica nanoparticles from silica powder and to characterize the prepared silica nanoparticles by using XRD, FTIR, SEM techniques

Material and Methods

All chemicals were of analytical grade, purchased from British drug house (BDH) Chemicals, Ltd., England and conventional and instrumental techniques were used throughout the experimental works.

Sample Collection

Rice straw samples were collected from Hmawbi Township, Yangon Region.

Preparation of Rice Straw Powder

Firstly, rice straw samples were thoroughly washed to remove any extraneous impurities and dried before use. Then straw was cut into shavings and homogenized into small particles by using a grinding mill and then passed over 0.8 mm size screen. After that the rice straw power sample was put in air-tight bottle.

Preparation of Rice Straw Ash

The rice straw powder was preheated on electrical hot plate until it became char. These rice straw chars were burnt in porcelain crucible by using muffle furnace about 1 h at temperatures 450, 550, 650, 750 °C to choose the optimum calcined temperature. After that, rice straw ash was prepared by using muffle furnace about 3 h at temperature 650 °C until it became grey ash. After being burnt, the ash was cooled at room temperature and then passed through the 180 mesh size sieve. It was put in sealed plastic bag and stored in the desiccators at room temperature.

Preparation of Silica Powder

A 15 g of rice straw ash sample and 120 mL of 2.5 N sodium hydroxide solution were mixed in a beaker. The mixed solution was heated at 100°C in a covered 500 mL beaker for 4 h with constant stirring to dissolve the silica (RSA) and to produce a sodium silicate solution. Then the solution was filtered with Whatman No.3 filter paper. The residue on the filter paper was washed with warm distilled water several times. Finally, the obtained viscous, transparent, colorless sodium silicate solution was allowed to cool at room temperature. After that sulphuric acid solution (5 N) was added into the sodium silicate solution from the burette with drop by drop under constant stirring at controlled condition (90-100 °C with normal atmospheric pressure) to obtain the solution of pH 2. Then the gel form silica was filtered with Whatman No.3 filter paper. The residue was washed with warm distilled water several times to remove the sodium sulphate and the filtrate was taken as silica (SiO_2). When the product was dried in a porcelain crucible at 70 °C for 15 h in an oven, it paves the amorphous silica and ground it into powder. The obtained silica powder was white rough powder. It was filtered till white smooth powder is obtained by the use of 150 µm and 600 µm test sieve.

Preparation of Silica Nanoparticles

Silica nanoparticles were extracted from silica powder by refluxing method. Silica with 5 N HCl solution was subjected to 6 h of continuous refluxing at 80-90 °C. The sample was filtered with Whatman No. 3 filter paper and the residue was then thoroughly washed with warm distilled water to become acid free. Afterwards 80 mL of 2.5 N NaOH was added to the silica powder under continuous stirring by using a magnetic stirrer for 10 h. Then concentrated H_2SO_4 was added to the solution until a white precipitate was formed. The precipitate was washed repeatedly with warm distilled water until the filtrate became alkali free. The obtained precipitated silica nanoparticles were dried in a hot air oven for 30 h.

Characterization of Rice Straw Powder by Thermo Gravimetric Differential Thermal Analysis Measurement

By using Thermogravimetry-differential thermal analysis (TG-DTA) (Rigaku Thermoplus TG 8120, Japan) measurement, prepared rice straw powder was carried out to study the weight losses with respect to temperature at a heating rate 10 °C/min and flow rate of 50 mL/min. Heating temperature was changed from 39-600 °C.

Qualitative Analysis of Rice Straw Powder, Rice Straw Ash, Silica Powder and Silica Nanoparticle

X-ray Diffraction Technique

Rice straw powder**,** rice straw ash, silica powder and the filtrate became alkali free. The obtained precipitated silica was qualitatively analyzed by X-ray diffractometer (Rigaku-D-max 2200, Japan). Sample was packed in a glass specimen holder and Cu tube was used as common tube with Ni filer.

Fourier Transform Infra-red Spectroscopy

The procedure was done in accordance with the recommended standard procedure as reported in FT IR instrument, Genesis 2, catalogue.

Scanning Electron Microscopic Examination

Rice straw powder was examined for a visual inspection of external porosity, and morphology by scanning electron microscope (SEM) (JOEL-JSM-5610, Japan).

Results and Discussion

TG-DTA Measurement of Rice Straw Powder

The TG-DTA thermogram of rice straw powder studied at 39 °C-600 °C is shown in Figure 1. At 39 °C - 240 °C, there is no significantly change of TG and DTA line. An exothermic peak was observed between 240 °C-360 °C due to the elimination of organic components and other trace chemicals elements normally present in the rice straw. Also another exothermic peak was observed over 360 °C is due to change in phase composition of rice straw. Exothermic peak observed at temperature above 520 °C is due to residue composed of carbonaceous products and silica. In this way, silica nanoparticles can be obtained at the temperature above 600 °C. In this study, the temperature 650 °C was chosen to get silica nanoparticles.

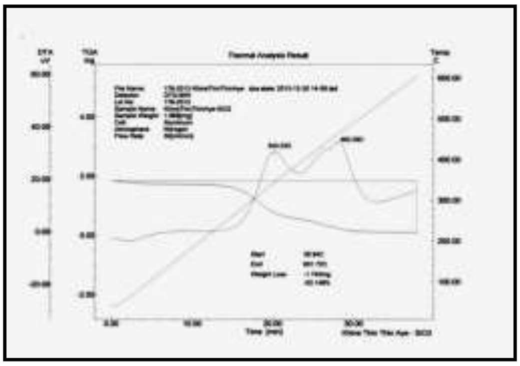

Figure 1. TG-DTA measurement of rice straw powder

XRD diffractogram of rice straw powder

The X-ray diffraction pattern of rice straw powder before heat treatment (Figure 2) indicates broad diffraction peak, extending 2θ value from 20° to 26° which was centered at 23° and is an indication of amorphous silica.

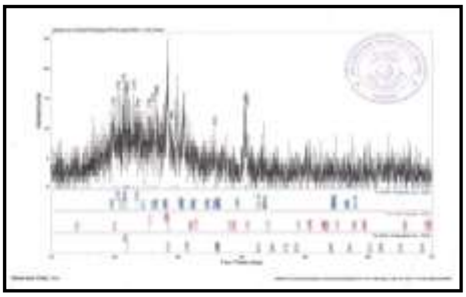

Figure 2. XRD diffractogram of rice straw powder

XRD Diffractogram of Rice Straw Ash

The X-ray diffraction (XRD) patterns of the rice straw ash (RSA) obtained after calcinations at 450, 550, 650, 750 °C for 1 h were shown in Figure 3. This XRD patterns show SiO_2 structure in RSA at temperature 650 °C which start to be more distinct than other temperature. Moreover, crystalline silica was formed by burning rice straw at higher temperature. So, temperature at 650 °C was chosen to prepare silica nanoparticles. The XRD pattern at 650 °C for 3 h in Figure 4 exhibits broad maximum, extending 2θ values from 19° to 31° was the indication of amorphous silica.

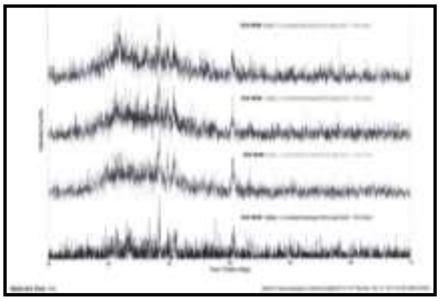

Figure 3. XRD diffractrogram of rice straw ash obtained after calcinations (A) 450 °C (B) 550 °C (C) 650 °C (D) 750 °C

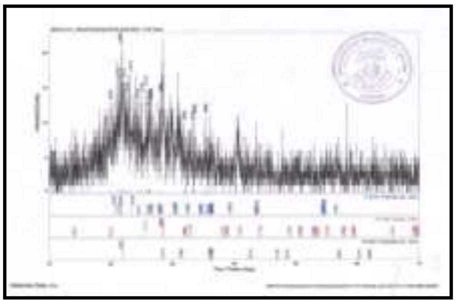

Figure 4. XRD diffractogram of rice straw ash after calcination at 650 °C for 3 h

XRD Diffractogram of Prepared Silica Powder

The X-ray diffraction pattern of the prepared silica sample is shown in Figure 5. Broad peak centered at 2θ of 22°, characterization of amorphous silica is observed in this case.

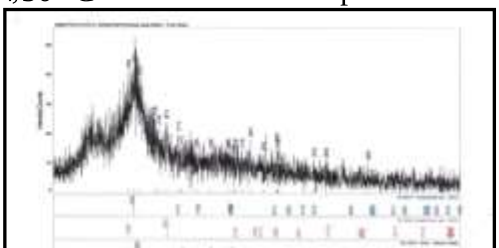

Figure 5. XRD diffractogram of silica powder from rice straw ash

XRD pattern of Prepared Silica Nanoparticle

The X-ray diffraction pattern of silica nanoparticle is shown in Figure 6. The strong broad peaks centered at 24° (2θ) confirm the amorphous nature of silica which is supported to be the characteristic of amorphous SiO_2.

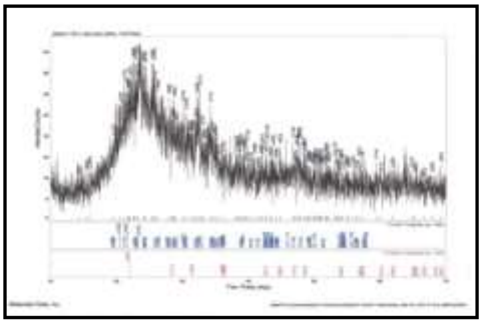

Figure 6. XRD diffractogram of prepared silica nanoparticle

FTIR Spectrum of Rice Straw Powder

FTIR spectrum of the rice straw powder is shown in Figure 7. According to the FTIR data, the absorption peaks at 3338 cm^{-1} and 2918 cm^{-1} originate from –OH stretching and C-H stretching respectively. The absorption peak at 1638 cm^{-1} ascribes to bending vibration of Si-OH.

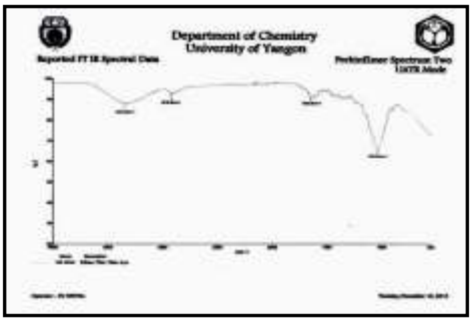

Figure 7. FT IR spectrum of rice straw powder

FTIR Spectrum of Rice Straw Ash

FT IR spectrum and band assignment of prepared ash are presented in Figure 8. The absorption peaks at 3553 cm^{-1}, 3477 cm^{-1} ascribe stretching vibration of O-H bond in Si-OH group. The absorption peaks at 1639 cm^{-1} and 1618 cm^{-1} are related to bending vibration of -OH in Si-OH group. The peaks at 1460 and 1400 cm^{-1} originate from the aromatic C=C stretch of aromatic vibration in bound lignin. The peaks at 993 cm^{-1} and 875 cm^{-1} ascribe bending vibration of Si-OH group. The absorption peaks at 617, 472, 408 cm^{-1} are related to bending vibration of Si-O bond in Si-OH group.

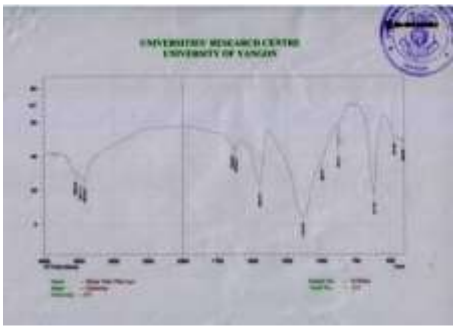

Figure 8. FI IR spectrum of rice straw ash

FTIR Spectrum of Prepared Silica Powder

The FTIR spectrum of the prepared silica powder is presented in Figure 9. According to FTIR data, the absorption peak at 3146 cm^{-1} is related to stretching vibration of -OH bond in Si-OH group. The peak at 1641 cm^{-1} ascribes bending vibration of -OH bond in Si-OH group. The absorption peak at 1116 cm^{-1} is related to stretching vibration of Si-O as-symmetric bond in Si-O-Si group. The peak at 983 cm^{-1} describes bending vibration of Si-OH group. The absorption peaks at 638 cm^{-1} and 617 cm^{-1} originate from bending vibration of Si-O bond in Si-OH group.

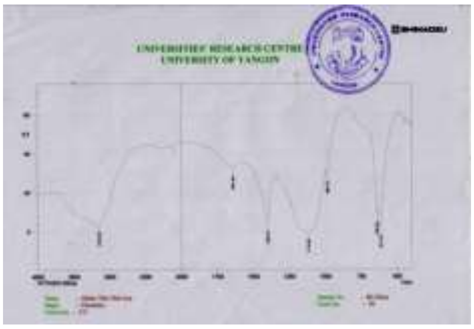

Figure 9. FTIR spectrum of silica powder

FTIR Spectrum of Prepared Silica Nanoparticles

Figure 10 is the FTIR spectrum of rice straw silica nanoparticles. According to the FTIR data, the absorption peak at 1080 cm^{-1} is caused by the presence of siloxane bond (Si-O-Si). The peak at 792 cm^{-1} is related to bending vibration of Si-OH group. The absorption peaks at 619 and 461 cm^{-1} originate from bending vibration of Si-O-H bond in Si-O-Si groups. It can be calculated that the prepared silica nanoparticles consist predominantly of silicon dioxide.

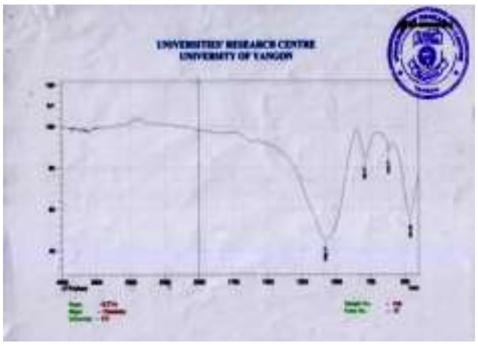

Figure 10. FTIR spectrum of silica nanoparticles

Scanning Electron Microscope Examination on Rice Straw Powder

The micro structure of the rice straw powder was studied by using SEM and described in Figure 11. It can be seen that SEM micrograph of rice straw powder showed flake-like structure.

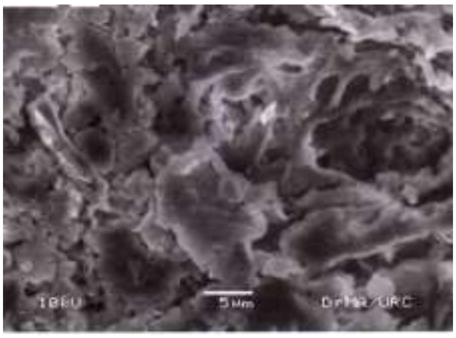

Figure 11. SEM micrograph of the rice straw powder

Scanning Electron Microscope Examination on Rice Straw Ash

The micro structure of rice straw ash was characterized by using SEM technique and described in Figure 12. There are some large size particles in this micrograph.

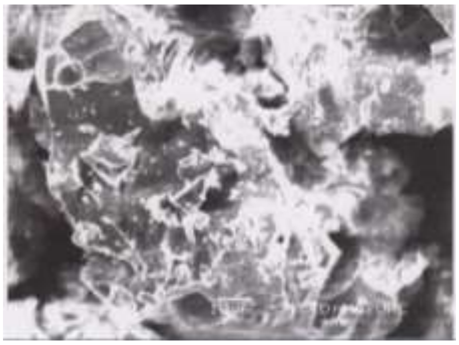

Figure 12. SEM micrograph of the rice straw ash

Scanning Electron Microscope Examination Silica Powder

Morphological feature of the prepared silica powder examined by using SEM is shown in Figure 13, which reveals some irregular and large particles. Prepared silica produces porous silica with a 5 µm average pore size. So, prepared silica powder is porous nature of nano structured morphology and this is different from SEM of rice straw ash.

Yadanabon University Research Journal, 2019, Vol-10, No.1

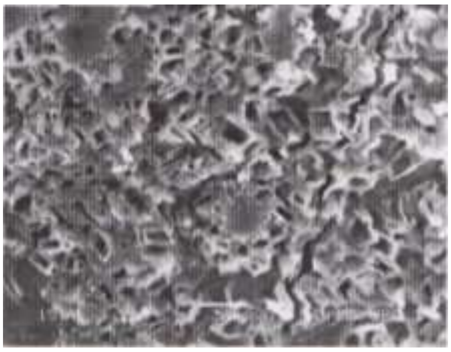

Figure 13. SEM micrograph of prepared silica powder

Scanning Electron Microscope Examination on Silica Nanoparticles

Scanning electron micrograph was used to investigate the morphological property of silica nanoparticles. The SEM images of silica nanoparticles are given in Figure 14. The morphology of silica nanoparticles is agglomerated to a larger extent. It can be seen that SEM micrograph of silica nanoparticles showed porous nature of nano structure morphology.

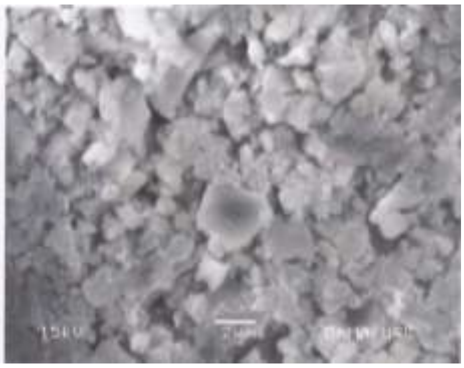

Figure 14. SEM micrograph of prepared silica nanoparticles

Conclusion

In this research, an attempt was made to produce silica nanoparticles from rice straw. By controlling the burning of rice straw ash (RSA) at 650 °C for 3 h, a reactive material relatively free from organic matter is obtained. XRD spectrum shows that the silica is an amorphous nature. By using Scherrer equation, the crystalline size of prepared silica nanoparticle was 46.70 nm. FTIR peak confirms that the silica nanoparticle belongs to the siloxane and silanol groups. SEM micrograph of silica nanoparticle shows that the particles are almost spherical, homogeneous and agglomerated form.

Acknowledgements

I would like to express my sincere gratitude to Rector Dr Maung Maung Naing, Pro-rectors Dr Si Si Khin and Dr Tint Moe Thuzar, Yadanabon University for their kind permission to submit this paper. The author wishes to express special thanks to Professor Dr Hlaing Hlaing Myat, Head of the Department of Chemistry, Yadanabon University for her permission, invaluable comments and encouragement on this paper.

References

Allam, M.E., Gihan, L.G., Hala, G., (2011), "Recycled Chopped Rice Straw Cement Bricks: Mechanical, Fire Resistance & Economical Assessment", *Australian Journal of Basic and Applied Sciences*, **5**(2), 27-33

Deabriges, J., (1991), "Process for the Manufacture of Sodium Silicate", *J. Eur, Ceram.Soc,* **73**, 257-262

FAO, (1997), "Rice By-Products in Asia", RAP Publication, The UN.RAP, Bangkok

Ismail, A.R., and Vejayakumaron, P., (2012) "Synthesis of Silica Nanoparticles by Sol-Gel: Size-Dependent Properties, Surface Modification, and Application in Silica-Polymer Nanocomposites", *Journal of Nanomaterials*, **15**, 35-52

Justin Airyawansa de Silva, M.W., (1980), "Current State of Research on Reactivity of Rice Husk Ash Cement," *Asian Institute of Technology,* Bangkok, 52-55

Lasheva, V., Tzolov, Tz., and Josifov, N., (2009), "Silica Products from Rice Hulls", *Chemical Technology and Metallurgy,* **44**, 257-261

Liou, T.H., (2004), "Evolution of Chemistry and Morphology during the Carbonization and Combustion of Rice Husks", *Carbon*, **42**, 785-789

Nakamoto, K., (1986), "Infrared and Raman Spectra of Inorganic and Coordination Compounds", 4th Edition, A Wiley-Interscience Publication John Wiley and Sons, New York, 1087-1088

Vempati, R.K., Musthyala, S.C., Yousuf, M., Mollah, A., and Cocke, D.L., (1995), "Surface Analyses of Pyrolysed Rice Husk Using Scanning Force Microscopy", *Fuel*, **74**, 1722-1725

Yalcin, N., and Sevinc, V., (2001), "Studies on Silica Obtained from Rice Husk", *Ceram Inter*, **27**, 219-224