Chiang Mai J. Sci. 2008; 35(1) : 206-211
www.science.cmu.ac.th/journal-science/josci.html
Contributed Paper

Preparation of Nanosilica Powder from Rice Husk Ash by Precipitation Method

Nittaya Thuadaij* and Apinon Nuntiya
Department of Industrial Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand.
*Author for correspondence, e-mail: anuntiya@chiangmai.ac.th

Received : 24 September 2007
Accepted : 15 October 2007

ABSTRACT

This work presented a laboratory studies on the preparation of rice husk ash by burning at 700 °C for 3 and 6 h, respectively. Consequently, silica content obtained after heat treatment at 700 °C for 6 h was 98.14%. Rice husk ash (RHA) was purified by alkaline extraction method used 2.0, 2.5 and 3.0 N sodium hydroxide. Percent yield of silica extracted by 2.5 N. NaOH, was 90.3% and the infrared spectral data supported the presence of hydrogen bonded silinol group and the siloxane groups in silica. Subsequently, the RHA was subjected to precipitation method in order to produce nanosilica. The precipitation was done by refluxing silica from RHA in boiling 2.0, 2.5 and 3.0 N NaOH, respectively. TEM results showed that 2.5 N NaOH for 10 h provided agglomerate particles with dimension of 5-10. The specific surface area was found to be 656 m^2/g. From X-ray diffractograms and diffraction pattern showed that the obtained products were amorphous nanosilica.

Keywords: Nanosilica, Precipitation Method, Rice Husk Ash (RHA), TEM.

1. INTRODUCTION

The beneficiation of rice husk has been used in many applications. Burning rice hull as fuel to generate energy resulted in the waste product, rice husk ash (RHA). RHA is rich in silica (about 60 %) and can be an economically variable raw material for production of silica gels and powders. The amorphous nature of RHA silica makes it extractable at lower temperature, and hence provides a low energy method[1,2].

Recently, nanotechnology has attracted considerable scientific interest due to the new potential uses of particles in nanometer scale. Thus industries may be able to re-engineer many existing products that function at unprecedented levels. There are few reports on mixing nanosilica in cement-based building materials. Chimera Research Institute (2002) addressed functional applications of SiO_2 in nano scale. However, up to now, research performed over the years has been largely aimed at achieving high mechanical performance with cement replacement materials in micro size. The development of an ultra high strength concrete was made possible by the application of densified system containing homogeneously arranged ultra fine particles[3, 5-6].

The objectives of this work are to prepare stable nanosilica using precipitation method from rice husk ash and to characterize of as-precipitated nanosilica.

2. MATERIALS AND METHODS

2.1 Material

Rice husk ash from brick factory was washed by distilled water and burnt at 700 °C for 3 and 6 h. Chemical composition of RHA was accomplished by X-ray fluorescence (Horiba Mesa-500w).

2.2 Synthesis pure silica from rice husk ash

Ten grams of RHA samples were stirred in 80 ml distilled 2.0, 2.5 and 3.0 N sodium hydroxide solution, respectively. RHA was boiled in a covered 250 ml Erlenmeyer flask for 3 h. The solution was filtered and the residue was washed with 20 ml boiling water. The filtrate was allowed to cool down to room temperature and added 5 N H_2SO_4 until pH 2 and then added NH_4OH until pH 8.5 allowed to room temperature for 3.5 h. The filtrate was then dried at 120 °C for 12 h. As-received product was finally investigated by Fourier transform infrared (FTIR- model tenser 27).

2.3 Preparation of nanosilica

Pure silica was extracted by refluxing with 6 N HCI for 4 h and then washed repeatedly using deionised water to make it acid free. It was then dissolved in 2, 2.5 and 3.0 N NaOH by continuous stirring for 10 h on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5. The precipitated silica was washed repeatedly with warm deionised water until the filtrate became completely alkali free. The washing process continued by deionised water repeatedly and dried at 50 °C for 48 h in the oven.

2.4 Characterization of Nanosilica

2.4.1. Morphology of nanosilica

Particle size and morphology of synthesized silica were examined by TEM (Jeol, JSM2010) using a 200 keV electron beam on the sample mounted on a carbon coated copper grid. With the assumption of spherical shape, the size of the particle is determined from the number averaged particle radius. Silica sample (10 mg) was sonicated for 3 h in isopropyl alcohol (5 ml). About 50 ml of the silica suspension was taken using a dropper and spread on the carbon coated copper grid and allowed to dry in room temperature. The copper grid was introduced into the instrument and the sample chamber was evacuated. The sample was scanned along the path of the electron beam and photograph of the sample was taken at 200,000 magnification.

2.4.2. Surface area measurement

Specific surface area of nanosilica was measured by nitrogen adsorption using BET equation. Samples were degassed in an oven at 383 K for 6 h and a five points BET analysis was conducted by using Quantachrome Autosorb-1 to obtain the specific area of the nanosilica powder.

2.4.3. Phase analysis by X-ray diffraction

X-ray diffractometer (D8 advanced: Bruker) was used to determine the phases of nanosilica. The scanning rate was 10/min in the 2θ diffraction angle between 10^o and 60^o.

3. RESULTS AND DISCUSSION

The rice husk ash (RHA) sample after being burnt at 700 °C for 6 h presented higher silica content compared to the other sample 700 °C for 3 h, as shown in Table 1.

The rice husk ash (RHA) sample after being extracted by 2.5 N sodium hydroxide generated the yield of pure silica up to 90.3% as shown in Table 2. The concentration of sodium hydroxide had strongly effect on the dissolution of silica from as-received rice husk and it also removed some impurities which were not dissolved from the main product.

The major chemical groups presented in silica were identified by the FTIR spectra shown in Figure 1. The broad band between 2800 and 3750 cm^{-1} was due to silinol OH

Table 1. Chemical composition of RHA before after burning out at 700 °C for 3 and 6 h.

Components expressed as oxides	RHA as-received	RHA after burning out at 700 °C for 3 h	RHA after burning out at 700 °C for 6 h
SiO_2	96.51	97.86	98.14
Al_2O_3	0.15	n/a	n/a
Fe_2O_3	0.17	0.07	0.07
CaO	0.66	0.52	0.46
ZrO	0.05	0.01	0.03
MgO	0.77	0.29	n/a
P_2O_3	0.21	n/a	n/a
Mn_2O_3	0.21	0.16	0.16
SO_3	0.04	0.07	0.07
LOI	n/a	0.01	0.02

Table 2. Effect of concentration of sodium hydroxide on the percent yield of pure silica.

Concentration of sodium hydroxide (Normal/N)	Yield of pure silica (%)
2.0	64.2
2.5	90.3
3.0	91.1

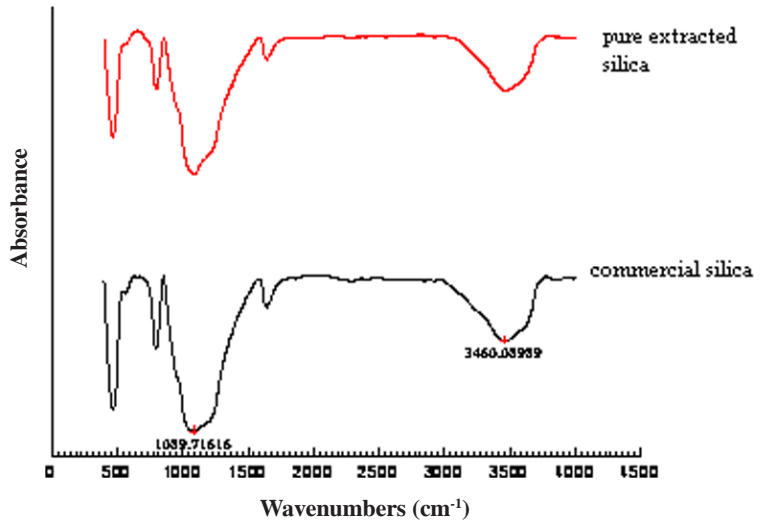

Figure 1. Fourier transform infrared spectra of spectra of pure silica produced from RHA.

groups and adsorbed water. The predominant absorbance peak at 1320 cm^{-1} was due to siloxane bonds (Si-O-Si). The peaks between 1200 and 700 cm^{-1} are attributed to vibration modes of the gel net work [4]. IR spectrums were not clearly show the difference between pure silica and commercial silica. The characteristic and position of the peaks are identical.

3.1. Effect of concentration of sodium hydroxide on nanosilica production

The particles size of the nanosilica after treated by 2.0, 2.5 and 3.0 N sodium hydroxide was determined by TEM. Sodium hydroxide had an effect on the particle size and specific surface area of the nanosilica particles, as summarized in Table 3. TEM micrographs showed that the particle sizes are in nanometer scale within agglomeration form. The shape of the particle was found to be uniform and agglomerated species. The diffuse ring pattern indicative of an amorphous phase of the main phase was shown in Figure 2.

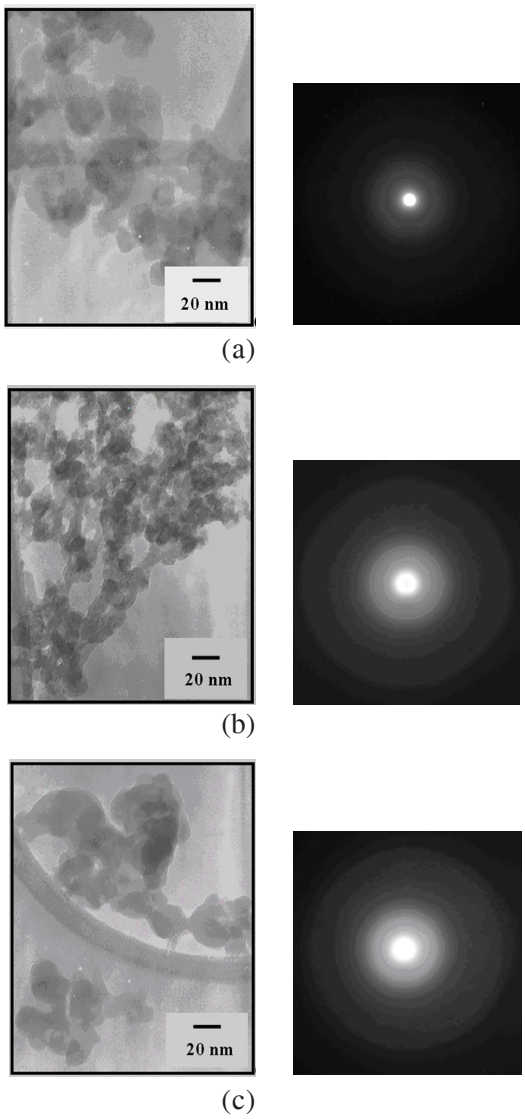

Figure 2. TEM micrograph and diffraction pattern of samples (a) treated by 2.0 N NaOH, (b) treated by 2.5 N NaOH (c) treated by 3.0 N NaOH.

Table 3. Effect of sodium hydroxide on specific surface area and particle size of nanosilica.

Concentration of NaOH (Normal/N)	Particle size (nm)	Specific surface area (m^2/g)
2.0	15-20	187
2.5	5-10	656
3.0	20-50	184

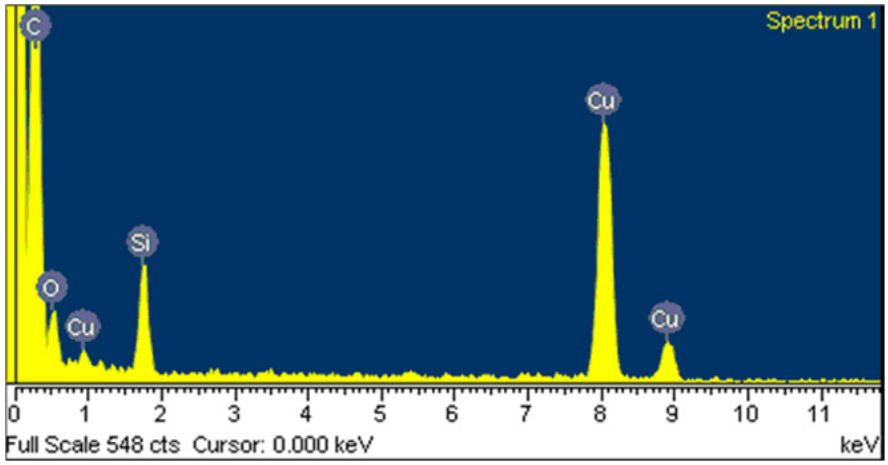

Figure 3. EDS of nanosilica particles.

EDS profile (Figure 3) of nanosilica particles contained predominantly the elements of Si, O, C and Cu. Both Si and O peaks correspond to the silica. The dominant signals originate from copper and carbon due to TEM copper grid and carbon coating

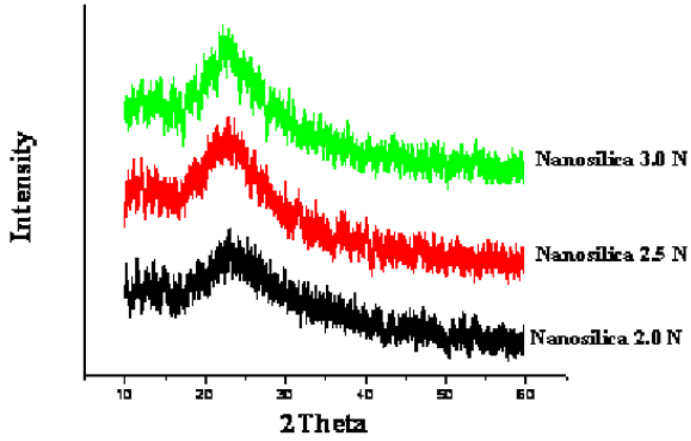

Figure 4. XRD diffractograms of nanosilica after treated by different concentration of sodium hydroxide.

XRD diffractograms of nanosilica (Figure 4) showed strong broad peaks between 22° and 23° (2θ). These strong broad peaks suggested characteristic of amorphous SiO_2.

4. CONCLUSION

The study revealed that silica with 90.34% silica content could be produced from RHA using a simple heat treatment. The 2.5 N sodium hydroxide treatment resulted in the highest SiO_2 content. Nanosilica particles which obtained from the rice husk ash were in the agglomerate form which dimension of 5-10 nm and specific surface area 656 m^2g^{-1}. The particle shape distribution is found to be uniform. The diffraction pattern of the particles showed a diffuse pattern which indicative of amorphous phase and supported by XRD patterns.

ACKNOWLEDGEMENTS

The author is grateful to the Thailand Research Found for fundings this research and financially supported by Grad Research Chiang Mai University.

REFERENCES

[1] Della V.P., Kuhn I. and Hotza D., Rice husk ash as an alternate source for active silica production, *Materials Letters,* 2000; **57**: 818-821.

[2] Kalapathy U., Proctor A. and Shultz J., A Simple method for production of pure silica from rice hull ash, *Bioresource Technology,* 2000; **73**: 257-262.

[3] Hui L., Hui-gang X., Jie Y., and Jinping O., Microstructure of cement mortar with nano particles, *Composites part B : engineering,* 2003; **35**: 185-189.

[4] Kalapathy U., Proctor A. and Shultz J., An Improved method for production of silica from rice hull ash, *Bioresource Technology,* 2000; **85**: 285-289.

[5] Qing Y., Zhang Z., Kong D., and Chen R., Influence of nano-SiO_2 addition on properties of hardened cement paste as compared with silica fume, *Contruction and Building Materials,* 2007; **21**: 539-545.

[6] Jal P K., Sudarshan M., Saha A., Sabita P., and Mishra B.K., Synthesis and characterization of nanosilica prepared by precipitation method, *Colloidals and Surface,* 2004; **240**: 173-178.