International Journal on Advanced Science Engineering Information Technology

Vol. 2 (2012) No. 2
ISSN: 2088-5334

A Simple Method of Obtaining Spherical Nanosilica from Rice Husk

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***Abstract* - This paper describes the synthesis of nanosilica from rice husk. Synthesis was done by precipitation using different acids, namely, orthophosphoric acid and nitric acid which yielded nanosilica. The effect of different acids and different solvents (polar and non-polar) on the size and degree of agglomeration of the silica particles were studied. When precipitation was done without the use of solvents, the silica particles obtained were non-spherical in shape, whereas, when precipitation was done using polar solvents (ethanol or 2-propanol), the silica particles were spherical. The use of non-polar solvents (toluene and diethyl ether) resulted in silica particles that were irregular in shape and in the case of orthophosphoric acid, a high degree of agglomeration was also noted. The degree of agglomeration was less when orthophosphoric acid was used with polar solvents as compared to nitric acid in conjunction with polar solvents. As regards the size, it ranged from approximately 70nm to 400nm when either acid was used in conjunction with polar solvents. When non-polar solvents were used, the silica particles were irregular in shape and the particle size was not estimated.**

***Keywords*— Rice husk; Nanosilica; Orthophosphoric acid; Precipitation.**

I. INTRODUCTION

Rice husk is the outer covering of the rice which contains organic materials as the main constituent and the ash of which contains 87-98% silica and a small percentage of metallic elements [1]. It is a by-product of the rice milling process and an agricultural waste and poses an environmental hazard. The commercial applications of rice husk are limited and ways to utilize it in a productive way has been a research priority. Due to its high calorific value, of lately, it has found wide applications as a fuel in power plants [2] and the generated ash ends up will disposal problems.

Malaysia is a major producer of rice and finding ways to put the husk to use is imminent. The high silica content in the rice husk ash has attracted interest in discovering ways to use it commercially. Although silica occurs as a component of cells or cell walls in virtually all arial parts of the rice plant, it is most abundant in the husk [3]. Owing to their small diameter, many technological applications, such as thixotropic agents, thermal insulators, composite fillers, etc., use ultrafine silica powders [4].

Several authors have reported different methods of obtaining silica from rice husk. Reference [5] produced nanosilica particles by worms using a bio-digestion process of rice husk. This paper reports a simple precipitation process for obtaining nanosilica from rice husk.

II. METHODS

A. Materials and Reagents

The rice husk was obtained from Koperasi Pak Badol, Pak Badol, Kelantan. NaOH, Ethanol, diethyl ether (analysis grade), nitric acid, orthophosphoric acid, HCl (37%, analysis grade) and 2-propanol (analysis grade) were obtained from Merck KGaA, Germany. Toluene was obtained from R & M Marketing, UK. The chemicals were used without further purification or dilution except in the case of HCl which was used to remove the metallic impurities from the rice husk. Distilled water was used wherever applicable.

B. Extraction of Silica

The rice husk was soaked in distilled water overnight and washed thoroughly with distilled water the following morning, to remove dirt and soil. It was then dried overnight in a hot air oven at 90°C. The dried rice husk was treated with 1N solution of HCl at 75°C for 1 hour in a hot water bath to remove impurities [6]. The suspension was filtered and the solid residue was washed thoroughly several times by distilled water to remove the metallic ions completely. The rice husk was again dried overnight at 90°C in a hot air

oven. 40 g of rice husk was immersed in 600ml of sodium hydroxide solution in a Teflon beaker and heated in a water bath for 1 hour at 90°C to extract the silica from the rice husk. A resultant sodium silicate solution (SSS) was obtained after filtering the rice husk.

c. Precipitaion of silica in spherical and non-spherical forms

100ml of SSS was taken in a teflon beaker and mixed vigorously with a magnetic stirrer on a hot plate. In the precipitation process, nitric acid was added drop wise till the pH of the SSS dropped to 8. The SSS was further stirred for another 45mins. The SSS was transferred to 4 eppendorf tubes which were centrifuged for 5mins at 4000 rpm. The pellet was washed with distilled water and centrifuged again. This step was repeated several times to get a whitish pellet. The pellets were collected from all the 4 eppendorf tubes and placed in a porcelain bowl and heated at 600°C for 30mins in a furnace. During the precipitation stage, different solvents (polar and non-polar) were also added in different experiments to the SSS and the resultant powder was also characterized. The solvents studied were ethanol, 2-propanol, toluene and diethyl ether. The amount of solvent added in each precipitation process involving each of the acids was 20ml. The above steps were repeated using orthophosphoric acid also as the precipitating agent.

D. Analytical methods

The silica particle morphology was characterized using Scanning Electron Microscopy (Field Emission SEM- Carl Zeiss SMT , Oberkochen, Germany. Image analysis was done using the Olympus 'Cell B' - Soft Imaging Solution GmbH, Munster, Germany.

III. RESULTS AND DISCUSSION

Different researchers have prepared silica from rice husk using different techniques but the resultant silica particles were mostly irregular in shape [2], [7] and [8]. Research on deriving silica has been going on for decades with significant improvements. Some researchers have have obtained silica from rice husk in conjunction with the sol-gel method to come up with better properties. Using sodium silicate derived from rice husk ash as a source of silica, and Pluronic P123 and cetyltrimethyl ammonium bromide (CTAB) as the pore structure-directing agents, reference [9] were able to synthesize mixed-phase bimodal mesoporous silicas (BMS) via sol–gel technique. While some researchers have produced silica in isolation from rice husk, other researchers have obtained hybrids like silica/lignin hybrid, etc. Reference [10] produced mesoporous lignin/silica hybrid from rice husk at different pH values in situ using the sol–gel process.

In the current research, nanosilica particles were obtained from rice husk in the spherical form by the simple inclusion of specific solvents. SEM images were used to characterize the size and shape of the silica. The use of solvent as well as the type can influence the shape of the resultant silica particles. When no solvent was used, the silica particles were irregular as was the case when non-polar solvents were used. Whereas, when polar solvents were used, the silica particles were well rounded and spherical. No significant difference in the size of the silica particles was noted when the two different polar solvents were used. Figures 1 and 2 show the particle size distribution of the silica particles obtained using orthophosphoric acid as the precipitating acid and 2-propanol and ethanol as the solvents respectively. The silica particles, probably, would have been solvinated with the polar solvents thereby increasing the surface charge which may be responsible for attaining the spherical shape of the silica particles.

Different acids used in the precipitation process as well as the solvents can have an influence in the degree of agglomeration of the silica particles obtained from rice husk. In conjunction with polar solvents (ethanol and 2-propanol), the degree of agglomeration was higher when nitric acid was used as the precipitating acid as compared to orthophosphoric acid. When non-polar solvents (toluene and diethyl ether) were used, the degree of agglomeration was significantly higher when orthophosphoric acid was used as the precipitating acid. The SEM images of the silica particles obtained using different acids and solvents are shown in figures 3 to 6.

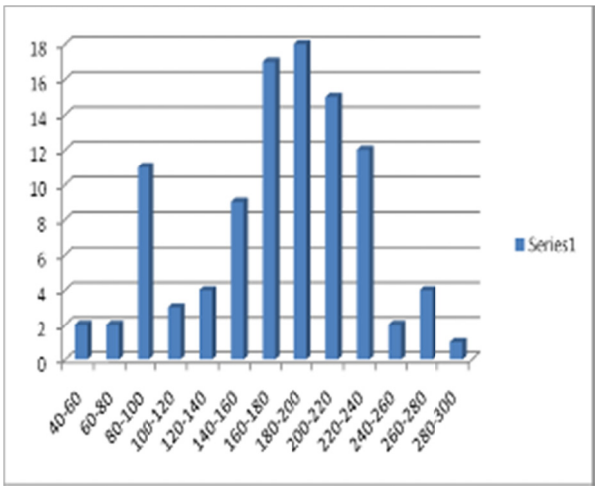

Fig. 1 Particle distribution of silica particles obtained using orthophosphoric acid as precipitating agent and propanol as the solvent

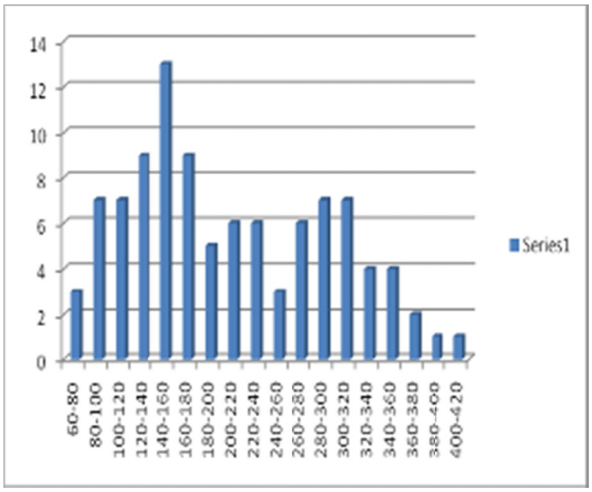

Fig. 1 Particle distribution of silica particles obtained using orthophosphoric acid as precipitating agent and ethanol as the solvent

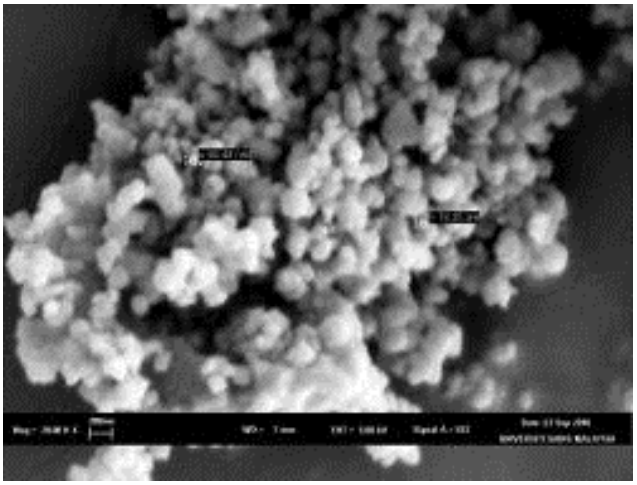

Fig. 3 SEM image shows spherical silica particles obtained by precipitating SSS with Nitric acid and with ethanol as solvent

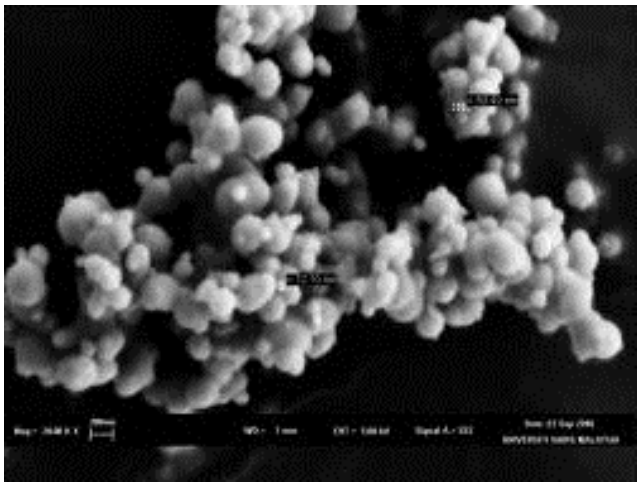

Fig. 4 SEM image shows spherical silica particles obtained by precipitating SSS with orthophosphoric acid and with ethanol as solvent

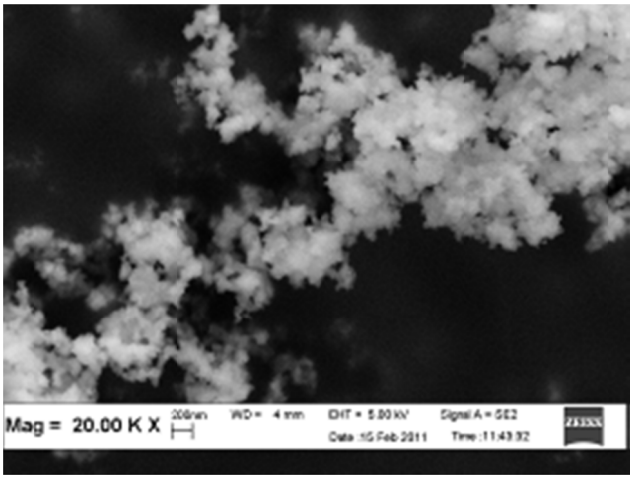

Fig. 5 SEM image shows irregular shaped silica particles obtained by precipitating SSS with Nitric acid and with toluene as solvent

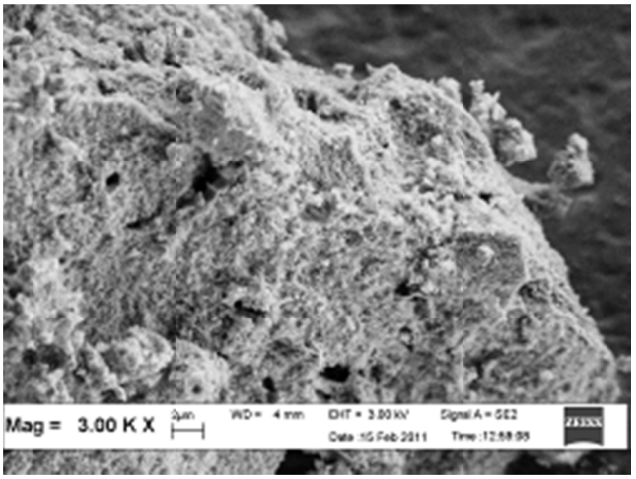

Fig. 6 SEM image shows highly agglomerated silica obtained by precipitating SSS with orthophosphoric acid and with toluene as the solvent

IV. CONCLUSIONS

It was possible to obtain superfine silica particles from rice husk using a simple precipitation method. By the inclusion of specific solvents during the precipitation process, it was possible to directly obtain superfine silica particles in spherical form.

ACKNOWLEDGMENT

The authors would like to thank Universiti Sains Malaysia for the Reaseach University Grant (RU-1001/PPSG/814110) which supported the current research. Mohammed Noushad would also like to thank USM for providing the USM fellowship.

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