Preparation of silica powder from rice husk

Hooman Sharifnasab, Mohammad Younesi Alamooti*

(*Agricultural Engineering Research Institute, Agricultural Research Education and Extension Organization (AREEO), Karaj, Iran*)

Abstract: Silicon dioxide (SiO_2) is the most abundant mineral in the world. Plants are the source of this mineral. In recent decades, with the advent of nanotechnology, silica nanoparticles have been found new applications in chemical industry, textile, packaging, biotechnology, agriculture and medicine. Rice husk and straw are rich sources of minerals. Rice husk (RH) has more than 20% silica. Using thermal process in presence of different gasses, the sample of ash of husk and bran of rice produced. To investigate the effect of heat treatment in different conditions, the properties of the samples were determined. Rice husk ash solution was poured on soda solution. Then silica was separated and sodium silicate was formed. By separation and titration of the solution, silica was obtained. The obtained silica washed by hydrochloric acid and purified. By repeating process on soda solution and titration under controlled conditions and by adding the surfactants silica nanoparticles were obtained. In this study, amorphous silica nanoparticles with an average size of 60 nm were produced by chemical reduction method. Fourier transform infrared (FT-IR) was used for Infrared spectroscopy analysis and X-ray diffraction was used for identification phase. For elemental analysis, the inductively coupled plasma emission spectrometer (ICP/ES) and energy dispersive X-ray analysis (EDAX) were used.

Keywords: silica nanoparticles, rice husk, chemical reduction, rice byproduct

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1 Introduction

Rice is an important nutritional grain in the world. Its production leaves behind a large amount of wastes called rice husk (RH). The by-product is highly important as it accounts for nearly one fifth of the world's annual rice production. About 220 kg (22%) husk is produced per 1000 kg of rice. Burning this amount of RH leaves about 55 kg (25%) rice husk ash (RHA) (Rahman et al., 1997).

Currently, a large portion of this by-product is not actually being used, causing environmental problems. These adverse effects are due to the high level of silica in RH. This high level of silica (22%) in RH renders it useless for human consumption or fodder. High levels of ash production, incomplete combustion, and low efficiency lead to high smoke emissions. Disposal of RHA and unburnt RH gives rise to environmental risks and problems. Accordingly, extensive researches are underway on finding new applications for this pollutant (RHA) (Liu et al., 2012).

Huge amount of rice husk is being generated annually but with no commercial value and extremely underutilized due to low nutritive potentials, resistance to degradation and high ash content. Thus, it is been stacked on farmland or dumped in an open heap near the mill site to be burnt later to ash. This disposal problem constitutes environmental pollution via air pollution, greenhouse gas emission, occupancy of landfill space, as well as energy wastage (Johar et al., 2012; Gu et al., 2015). However, the rice husk has limited applicability in stock-breeding, because it contains more than 70% of lignin-cellulose material and more than 20% of amorphous SiO_2 (Angelova et al., 2011). Therefore, the best application of rice husk appears as a good source of silica. Rice husk upon burning yields 14%-20% ash, which contains 80%-95% silica in the crystalline form together with trace amounts of metallic impurities (Chandrasekhar et al.,

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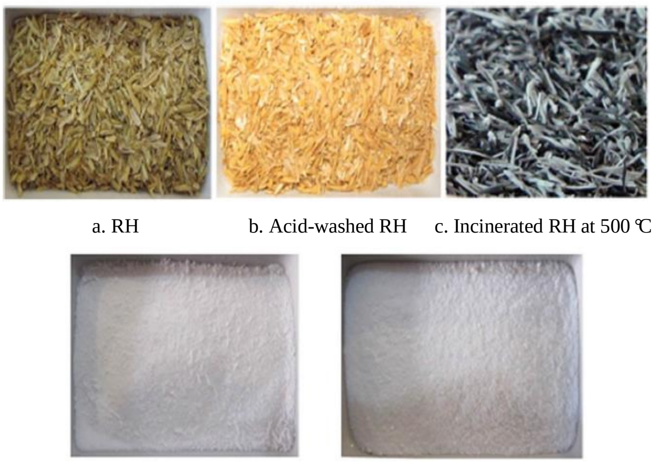
***Corresponding authors: Mohammad Younesi Alamooti,** Agricultural Machinery Department, Agricultural Engineering Research Institute (AERII), Karaj, Iran. P.O. Box: 31585-854, Tel: +98 2612706101, 2753866. Email: mohamadyounesi@yahoo.com.

2005).

Rice Research Institute of Iran reported that the annual paddy production in Iran reaches 3 million tons consisting of 65% white rice, 20% husk and 15% bran. Silica or SiO_2 is available in two forms of amorphous and crystalline. A number of products including colloidal silica, highly-pure silica and silica gel can be obtained by processing silica. Among all silica types, amorphous silica is the one with largest industrial applications. These applications include coatings, plastics, rubbers, electronics, optics, and fire-retardant materials. At the same time, it is widely used in synthesis of chemicals like sodium silicate, zeolite catalysts, aerogel, highly-pure silicon, silicon nitride, and silicon carbide. High-tech industries (e.g. computer, biotechnology, and pharmaceutics) are increasingly requiring nanosilica particles, driven by their unique excellent properties (Kalapathy et al., 2000).

RH is about 13% to 29% minerals, 87% to 97% of which is amorphous silica. Major impurities of RH are sodium, potassium, magnesium, calcium and iron. These RHA impurities (in form of oxides and silicates) account for 3% to 13%. Figure 1 shows RH in different states (Rafiee et al., 2012).



a. RH b. Acid-washed RH c. Incinerated RH at 500℃

d. Incinerated RH at 700℃ e. Nanosilica

Figure 1 RH and RHA images

Different methods have been reported for synthesis of nanosilica particles from RH, including Chemical Vapor Deposition (CVD), sol-gel, and thermal decomposition (Liou, 2004). The high production costs have limited its application (Liou, 2004). The solution-deposition technique is the most inexpensive, straightforward extraction method (Liou, 2004).

Mesoporous SiO_2 nano powder was successfully synthesized from sticky RHA through a simple acid pretreatment method. The micro structural and elemental composition analysis confirmed the spherical shape and purity of the SiO_2 nanoparticles. The scanning electron microscopic (SEM) and transmission electron microscope (TEM) analyses of the silica nano powder revealed that SiO_2had the particle size 50 nm and the surface area 7.1548 m^2 g^{-1}. The absence of sharp peaks in the X-ray powder diffraction (XRD) pattern and the electron diffraction rings in the SAED pattern confirmed the amorphous nature of the material. The bio generated SiO_2nanoparticles synthesized from sticky rich husk ash are considered to be the most compatible material for energy storage and optoelectronic applications (Sankar et al., 2016).

2 Material and methods

The raw material, RH, was obtained from a rice mill in the north of Iran. RH was first washed with distilled water and was then dried at 90℃ for 10 h. An Iranian made oven (Azar brand) with atmosphere air aspirated was used to obtaining the husk ash of rice and also an Iranian experimental refrigerator centrifuge machine model “Universal Z 326” with maximum rotation speed of 18000 r min^{-1}, was used for centrifugation. Speed of 2500 r min^{-1} was used for this study.

One gram of dried RH was incinerated at 700℃ for 3 h. It was then washed with 1 mol L^{-1} acid acetic and distilled water using a centrifuge to reach pH = 7. The result was oven-dried at 90℃ for 12 h (describe the equipment used if fabricated by you, otherwise state the brand and the model). It was then weighed. The dried product’s weight was 0.936 g. It was concluded that the weight loss was due to metallic ions that left during acid washing and sample losses during washing.

RHA was stirred with sodium hydroxide. The resulting solution was heated in a capped beaker under steady rotation and was then filtered. The rest was washed with hot distilled water. The resulting transparent colorless solution was left to cool down to the room temperature. Acid sulfuric was added under steady rotation until reaching pH = 2. Ammonium hydroxide was added to reach pH = 8.5 and was then left to reach the room temperature. The above-mentioned extracted silica solution was refluxed with hydrochloric acid for 4 h to produce nanosilica which was also washed multiple times with distilled water to remove its acidity. It was then solved in sodium hydroxide while being rotated, and sulfuric acid was then added to reach pH = 8. Deposited silica was washed several times with warm distilled water and was washed to remove its alkalinity. It was finally oven-dried at 50 ℃ for 48 h.

FT-IR spectroscopy was done to determine the composition. For analyzing of infrared spectrum, FT-IR model broker tensor 27 was used.

3 Results and discussion

Figure 2 presents SEM images of acid-washed ashes. These images taken with different zooms from the samples clearly show the morphology of particles. As shown, the particles are like connected clots.

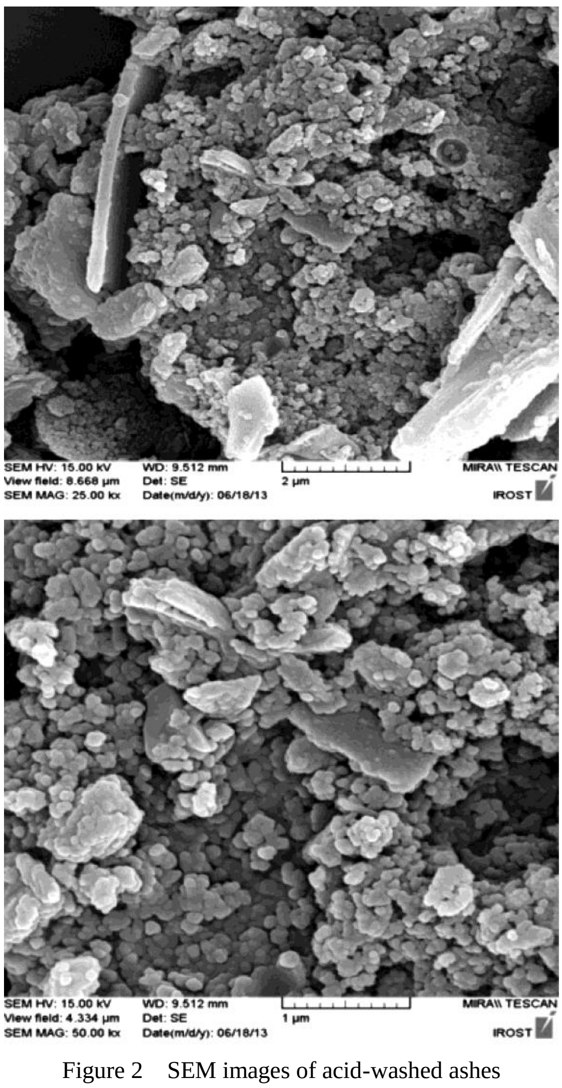


Figure 2 SEM images of acid-washed ashes

FT-IR spectroscopy was done to determine the composition. Figure 3 shows the FT-IR spectrum for a synthesized sample.

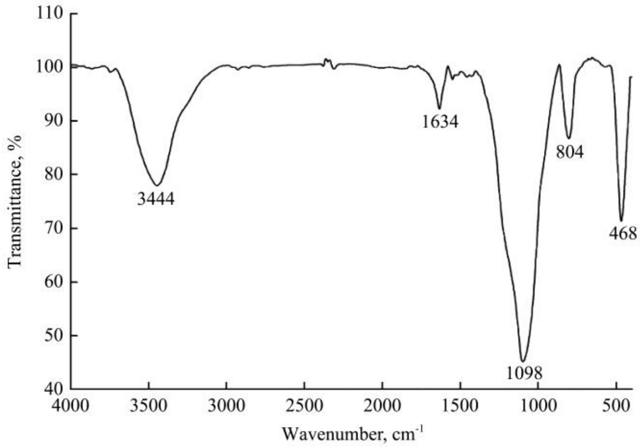


Figure 3 FT-IR spectrum of an acid-washed RHA sample

As shown in Figure 3, the absorption bands (468, 804 and 1098 cm^{-1}) are due to flexural and tensile vibrations of silica. Moreover, the absorption bands at 3444 and 1634 cm^{-1} are associated with tensile and flexural modes of adsorbed water (H-O-H), respectively. The peak points of the spectrum indicated that the white power was silica. Other small peaks of the spectrum show trace amounts of metallic impurities in the sample.

The XRD technique was also applied to identify the crystalline phase of sample. The diffraction pattern indicated a wide peak with its center at $2\theta = 22°$ (Figure 4). This figure shows an amorphous structure with no uniform crystalline pattern.

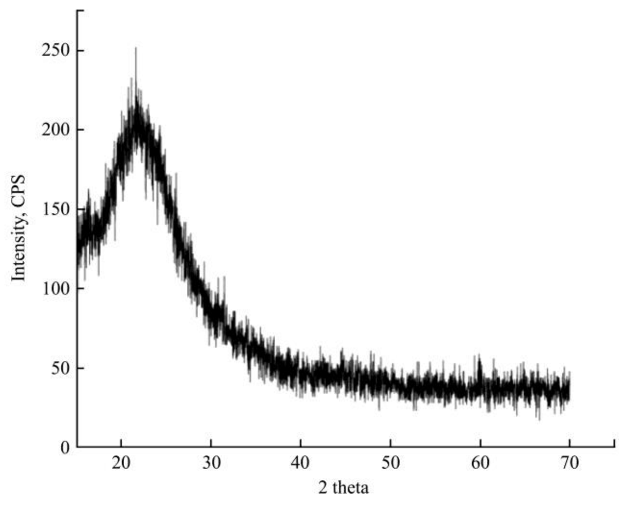


Figure 4 XRD pattern of acid-washed RHA

SEM image of synthesized nanosilica is presented in Figure 5. As shown, the spherical nano-particles have a good distribution and are about 60 nm large.

Figure 5 SEM image of silica nano-particles synthesized from RHA

4 Conclusions

RH, which is an agricultural waste, can be converted into valuable product used for several applications in industry. Therefore the determination of exact quantity of silica powder is important for analyst to study its application. In this study, amorphous silica nanoparticles with an average size of 60 nm were produced by chemical reduction method.

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