Academic View

Synthesis and Characterization of Nano Silica from Rice Husk

Shanta Pokhrel*, Sanjeeb Dahal
Department of Chemistry, Tri-Chandra Multiple Campus, TU, Kathmandu, Nepal
***Corresponding E-mail:** *shantabhattarai2014@gmail.com*

Abstract

The rice process produces a large amount of rice husk which is most economically important a natural source of silica. In this work, the nano silica was synthesized from dried and cleaned rice husk by treating with hydrochloric acid followed by incineration at 700 ℃ in muffle furnace. Thus, formed silica nanoparticles were characterized through X-ray diffraction (XRD), Fourier- transform infra-red spectroscopy (FTIR) and Energy dispersive X-ray spectroscopy (EDX). The FTIR results showed different peaks at 800 cm^{-1}, 1078 cm^{-1} and 455 cm^{-1} resembling the peaks of Si-O symmetric stretching frequency, Si-O asymmetric stretching frequency and Si-O-Si bending frequency respectively. The average crystallite size was found 39.49 nm from XRD results. Whereas, EDX results showed silica elements with 58.20% composition along with Fe and Cr as major impurities. Thus, agricultural waste i.e. rice husk was found to be a major source of silica.

Keywords: *EDX, FTIR, Incineration, Nano silica, Rice husk, XRD*

Introduction

About 600 million tons of rice paddy is produced every year world widely. Generally, 20% of the rice paddy is husk which gives 120 million tons of annual total production (Chandra, 2007). Nepal is an agricultural country and paddy is a major crop grown here. The average annual rice production of Nepal is about 3.2 million to 3.5 million tons (Pokhrel, 1992). Rice husk ash derived from this production can be used as filler in composites, adsorbent for water purification, additive, oil adsorbent, water purifier, and as a source in the preparation of high-performance silicon and its compounds (Salavati-Niasari, *et. al.*, 2013). As the use of cement produces large amount of CO_2 which increases greenhouse effect, nano silica has the potential to replace cement and can be used as concrete additives (Quercia & Brouwers, 2010). Large scale production of nano silica is possible through rice husk ash. Silica has various applications, such as carriers, medical additives, fillers in composite materials (Liu *et. al*., 2005, Shin *et. al.,* 2008) and as a sources for adsorption materials (Jang, et. al., 2009, Wongjunda & Saueprasearsit, 2009, Lakshmi, *et. al.,* 2009) and become more advantageous when it is at nanometer size. Therefore, the aim of this research is to synthesize nano silica to provide the alternative way for the waste management and also to enhance the research activities in easily available raw material. This research was carried in order to prioritize the green synthesis of nano silica.

Experimental Methods

Silica nanoparticles were extracted from rice husk following the protocol of Nguyen *et al.,* (2017). Firstly, rice husk was washed with distilled water to make it dust free, to remove soluble impurities and other foreign substances. Then it was air dried in oven maintaining the temperature at 60 ℃. After that 900 mL of 1% HCl was added to 50 g of rice husk and stirred for 2 h in magnetic stirring at room temperature. The mixture was kept for overnight, decant out and washed gently with distilled water. Then it was completely dried in the air and was grinded into the powder by using grinder. Thus, obtained rice husk was then placed into a crucible and was calcinated at 700 ℃ for 2 h in a muffle furnace to get the silica nanoparticles.

Synthesized silica nanoparticles were characterized by Energy Dispersive X-ray spectroscopy (EDX), Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffraction (XRD).

Results and Discussion

Energy dispersive X-ray spectroscopy (EDX)

The EDX was done by EDX-8000 in atmospheric air condition gave the following elemental composition which is shown in Figure (1). EDX result showed that silicon was present as major constituent in prepared sample with composition 58.2%. Xiaoyu, *et al.* (2012) reported nano silica with composition 65%. Iron (Fe) and Chromium (Cr) were found as major impurities with their composition 37.4% and 2.8% respectively. However, Ghorbani, *et al.* (2015) prepared silica nanoparticles in purer form with composition 95.5%.

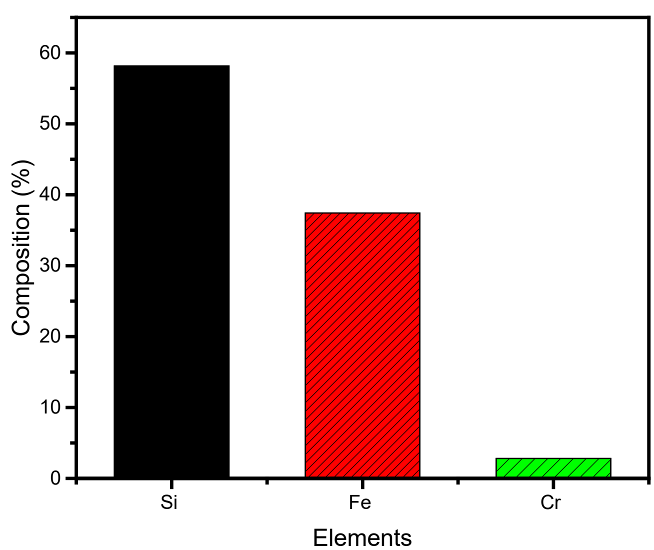

Figure 1: *Elemental composition of prepared samples*

Fourier-transform infra-red spectroscopy (FTIR)

The main functional groups of extracted silica are identified by the FT-IR spectra presented in **Figure 2** and the characteristics peaks were tabulated in **Table 1**.

Table 1: *Observed FTIR peaks with reported range (Launer, 2013)*

Functional group	Observed peak (cm^{-1})	Reported peak range (cm^{-1})
C=N=O	2324	2340-2280
Si-H stretching vibration	2160	-
Si-O stretching vibration	791	850-795
Si-O-Si asymmetric stretching vibration	1078	1130-1000
OH	3611	3700-3600
Si-O bending vibration	455	550-400

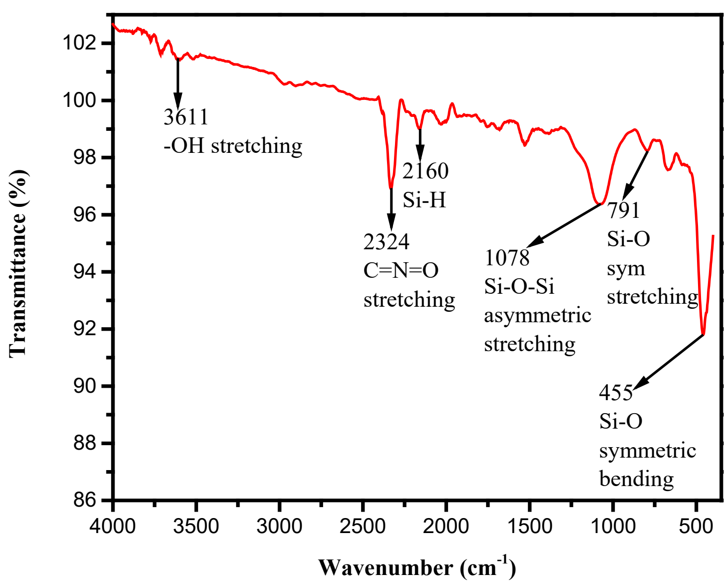

Figure 2: *FTIR spectra of nanosilica ($nSiO_2$)*

The FTIR spectra showed the peaks at 791 cm^{-1} are due to the Si-O symmetrical stretching vibration. Le, *et al.* (2013) assigned the peak at 804 cm^{-1} for the symmetrical stretching vibration of Si-O bond. The peak at 1078 cm^{-1} corresponds to the Si-O-Si asymmetric stretching vibration whereas Patel & Patel, (2014) assigned the peak of Si-O-Si at 1086 cm^{-1}. The peak recorded at the wavelength of 455 cm^{-1} is due to the Si-O bending frequency whereas Yuvakkumar*, et al.* (2012) assigned the peak at 452 cm^{-1}. Thuadaij & Nunita, (2008) and Nayak & Bera, (2009) reported that peaks at 1085, 795 and 450

cm^{-1} are due to the vibration modes of amorphous silica gel network. Moreover, the absorption band at 3611 cm^{-1} shows the presence of -OH group that confirms the presence of silanol (Launer, 2013).

X-ray diffraction (XRD)

XRD pattern of rice husk nano silica (Figure 3) showing sharp lines indicating the crystalline nature of the sample. The characteristic peaks were observed at 20.9, 24.3, 26.8, 33.4, 35.8, 41.0, 49.5, 54.3, 62.8, 64.1° whereas Palanivelu *et al*., (2014) reported signals at 20.6, 26.5, 27.5, 30.2, 36.1° and 20.8, 26.6, 45.8, 50, 60, 65°, respectively. These peaks are allocated for tridymite and quartz crystalline states of nano silica samples pyrolyzed at 800, 850 and 900 °C (Palanivelu *et al*., 2014).

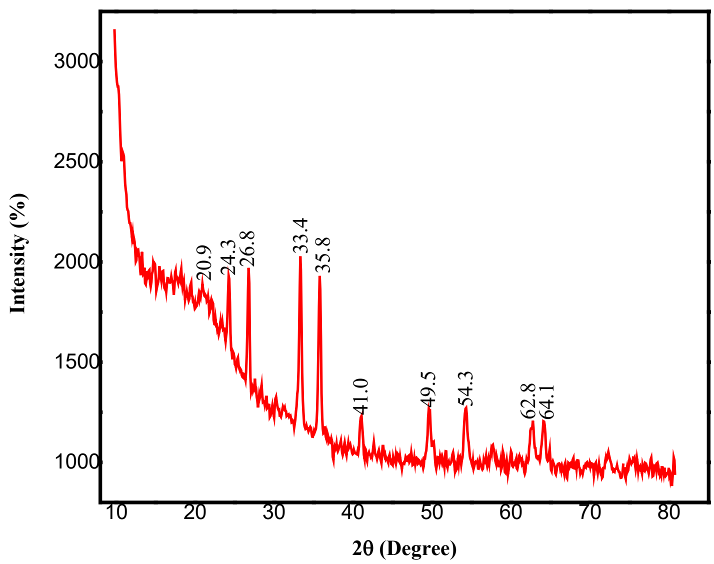

Figure 3. *X-ray diffraction (XRD) patterns of nanosilica (nSiO$_2$)*

Full width at half maximum intensity (FWHM) of different peaks was calculated XRD and the crystallite size of the nano silica was calculated by using Scherer equation (1) (Barati *et al.,* 2019).

$$\text{Crystallite size (D)} = \frac{K\lambda}{\beta \cos\theta} \ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where, K = Scherer constant having value of 0.94, λ = wave length of X-ray used, β = Full width half maximum intensity (FWHM), θ = Bragg angle (peak position). The average crystallite size was calculated as 39.49 nm. Similarly, Hincapié Rojas *et al.,* (2019) synthesized nano silica from rice husk with 37.15 nm crystallite size whereas Hassan reported 26.00 nm average size of synthesized silica nanoparticles (Hassan *et al.,* 2014).

Conclusion

Silica nanoparticles were synthesized from agricultural waste i.e. rice husk by treating with hydrochloric acid followed by incineration at 700 ℃. EDX analysis showed the composition of silica as 58.2% along with Fe and Cr as major impurities present in the given rice husk. FTIR data analysis showed the peak at 800 cm^{-1}, 1078 cm^{-1} and 455 cm^{-1} due to the symmetric stretching of Si-O, asymmetric stretching of Si-O-Si and bending vibration of Si-O bond respectively. From XRD data, the average particle size was found to be 39.49 nm. Thus, this research work showed the rice husk could be a major source of silica from which silica nano particles can be easily synthesized.

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