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Green Synthesis of Silica Extracted from Rice Husk Ash

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A B S T R A C T

There has been a significant rise in the scientific, technological, ecological, economic, and social popularity of applications of by-products or waste materials in various industries including the agricultural sector. A by-product of rice4 milling is Rice Husk (RH), which, when burned, produces Rice Husk Ash (RHA). RHA is considered an economically viable raw material used for developing silica-based products since it contains a significant amount of amorphous silica (between 85 and 95 percent). The current research aims to create a green process for producing silica powders from RHA, an inexpensive source rich in biocompatible silica. High-purity silica was successfully generated from RHA through alkaline extraction using the reflux technique and subsequent acidification. For this purpose, RH was burned in an electric furnace for five hours to create RHA at 700 °C. The obtained ash was washed with hydrochloric acid to eliminate the metallic impurities. The sodium silicate solution was then obtained by refluxing the acid-washed RHA in a NaOH solution. The final step was to precipitate silica from sodium silicate solution by adding hydrochloric acid to decrease the pH by 4. Characterizations made in general are Thermo-Gravimetric Analysis (TGA), X-Ray Fluorescence (XRF) for identifying the mineral contents of RHA, Fourier-Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). Simple, efficient, environmentally-friendly, and ideal for mass production are all attributes of the synthetic process.

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1. INTRODUCTION

Silica (SiO_2), as an essential inorganic substance [1], has been used as a major precursor for many materials in applications such as industrial manufacturing, drug delivery, electronics, composites, packaging, ceramic materials, adsorption, biosensing, and catalytic applications [2,3]. Among the remarkable properties of pure SiO_2 are low toxicity, high chemical and physical stability, and great surface chemistry properties that allow SiO_2 to be combined or functionalized with various functional species or molecules. While sodium silicate is the main source of SiO_2 production, it is the production mechanism that determines the energy waste, environmental effects, and purity of the final SiO_2 [4,5]. Both organic and inorganic materials can be used in SiO_2 production. Rice wheat and coffee husks, barley grass, corn cob, sugar can bagasse, and palm oil are the

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potential bio-sources from which sodium silicate can be extracted [6]. Plants acquire and collect SiO_2 in addition to lignin and cellulose, which is then utilized to cover the hard-material components of the plant such as the husk [7]. Ash left from rice husk (RH) burning may be one of the most important sources of making sodium silicate [8] because it is a prominent agriculture industry waste product [9,10]. RH is the hard outer cover of the rice kernel which is removed during the rice milling process [11]. On average, about 20 % of the produced paddy turns to husk on a weight basis [12-14]. The husk itself is mainly composed of lignin, silicon, other inorganic components, and a small quantity of protein. Upon burning RH, organic materials are broken down into carbon dioxide and water vapor, and the remaining ash is mostly of SiO_2 composition [15].

A large amount of RH, about 150 million metric tons per year, as a by-product is being produced worldwide [16]. Depending on the weather, types of rice, and location of the farmland, each ton of RH can yield 0.18 to 0.20 tons of ash [17]. Therefore, a proper waste utilization or disposal method should be adopted for this agricultural waste [10]. Depending on the components of RH, its decomposition time varies that eventually results in a huge amount of waste over time [8]. Moreover, its utilization as fodder should be avoided due to its high SiO_2 content [18]. In this regard, it could be a great alternative source for common methods for sodium silicate production [19] to be used to solve the problem of burying rice in fields [20].

It is possible to obtain sodium silicate solution from rice husk ash (RHA) through a low-temperature chemical process called alkaline extraction which is cost-effective and environmentally-friendly with low energy consumption, compared to other commercial SiO_2 manufacturing methods with multiple steps at high temperatures and pressure [7,10,21,22].

SiO_2 gel is generally produced from acidification of sodium silicate [4]. Amorphous SiO_2 has very low solubility at pH less than 10 [23]. At pH above 10, the solubility increases sharply. Such distinctive behavior allows us to extract pure SiO_2 through the alkaline extraction method and subsequent precipitation by decreasing the pH [6,17]. It turns out that alkali extraction is a practical method that provides a high s SiO_2 yield [24,25] depending on the concentration of NaOH used for extraction as well as the type of acid used for pre-washing RHA before extraction. NaOH provides a slightly high SiO_2 yield that can be utilized even at low concentrations. In this study, 1 M NaOH solution used for extraction provided a SiO_2 yield of more than 85 % [23,26]. In addition, according to the literature, use of HCl to acidify sodium silicate solution and create SiO_2 gel results in final SiO_2 with high purity both in acid leaching and precipitation step. The chemical reaction of precipitation confirms the production of NaCl with high solubility in water which can be easily washed with DI water [23,26,27]. A comparison of the acid-washed RHA with the unwashed one revealed that the percentage yield of the extracted SiO_2 was higher in the washed sample than that in the unwashed one [27]. A number of acids such as HCl, HNO_3, H_2SO_4, etc. can act as the leaching agents. According to several studies, hydrochloric acid is more effective as an RH leaching agent than its other types. Previous studies also confirmed its superiority as a leaching agent in generating high-purity amorphous SiO_2 with enhanced pozzolanic characteristics. Additionally, leaching of RH with hydrochloric acid can essentially eliminate the additional contaminants such as Al_2O_3, Fe_2O_3, CaO, and MgO from RH [17,23]. In this respect, leaching requires an acid concentration of at least 1 M. 1 M HCl which is reported as a suitable leaching agent for this purpose [28,29].

According to the literature, NaOH and HCl with the concentration of 1 M provide a high yield of SiO_2 from RHA, high metal impurity elimination (among inorganic acids), and with soluble and washable products (NaCl) in addition to the SiO_2 during the precipitation. These parameters are separately assessed in the previous studies [23,28,29]. In this paper, both NaOH and HCl solutions with the effective concentrations mentioned in different papers were used.

This study aims to efficiently extract pure SiO_2 with amorphous nature and uniform morphology from RHA waste as a raw material. This eco-friendly synthesis procedure has a high yield. The SiO_2 extraction has been through alkaline extraction (1 M NaOH), followed by selective precipitation and self-assembly of components by acid titration (1 M HCl) to achieve SiO_2 with high purity. The synthesis procedure as well as the physical and structural properties of the produced amorphous SiO_2 are thoroughly investigated in the following sections.

2. MATERIALS AND METHODS

2.1. Materials

The raw RH used in this study was obtained from an Iranian rice processing facility located in Mazandaran, Iran. The used chemicals were concentrated hydrochloric acid (HCl 37 %, Sigma-Aldrich), sodium hydroxide (NaOH, Merck), and deionized (DI) water.

2.2. Methods

In order to dispose of dust, RH was thoroughly washed with DI water and dried for 10 hours at 90 °C. According to the TGA results presented in the following section, the suitable temperature for burning RH is 700 °C. This temperature is also suitable to prevent crystalline SiO_2 formation [30]. Therefore, RH is combusted at 700 °C for five hours to produce RHA. The fundamental steps of the SiO_2 production are the SiO_2 extraction as sodium silicate solution and SiO_2 generation from the solution.

2.2.1. Acid Washing RHA

Five grams of RHA was mixed with 45 ml DI water and 1 M HCl solution dropwise until the pH reached 1. The suspension was stirred for 12 hours. Followed by filtering and washing with DI water, the metallic ions of the suspension were completely removed to maintain the neutral pH. The solid residue, acid-washed RHA, was dried in an oven at 90 °C for 12 hours. Then, ash was used as the primary component in the SiO_2 extraction process.

2.2.2 Silica Extraction as Sodium Silicate Solution

Four grams of RHA was added to 90 ml of 1 M sodium hydroxide (NaOH) solution. The sample was boiled in a covered Erlenmeyer flask for six hours with constant stirring to give enough time for soaking.

The solution was then filtered with filter paper. A tiny amount of boiling water was used to rinse the residue. The filtrate was then allowed to age for 19 hours while being cooled at room temperature.

2.2.3 Silica Precipitation

The obtained yellowish clear and transparent sodium silicate (Na_2SiO_3) solution was transformed into silicic acid (H_3SiO_4) by titration with 1 M hydrochloric acid under constant stirring to reach pH 4. Adding HCl to the obtained Na_2SiO_3 solution caused the formation and condensation of the Silanol group (R_3 Si-OH). To remove the NaCl residue, the final product was rinsed with hot DI water. In case pH was < 10, SiO_2 gel started to precipitate. The slurry gained by introducing DI water to the gel was centrifuged three times for 10 minutes at 4000 rpm. The clear supernatants were discarded. The gel was dried at 70 °C for 14 h to get SiO_2 powder. The obtained power was then washed and dried at the same temperature for five hours to wash the possible residual sodium [21]. The procedure is illustrated through the chemical reactions (1) and (2) [24]:

$$2NaOH + xSiO_2 \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

$$Na_2SiO_3 + 2HCl \rightarrow SiO_2\ (gel) + 2NaCl + H_2O \quad (2)$$

2.2.4 Materials Characterization Techniques

The following techniques were further used for characterization of the synthesized powder: Thermo-Gravimetric Analysis (TGA, Mettler Todelo), X-Ray Fluorescence (XRF, XEPOS), Fourier-Transform Infrared Spectroscopy (FT-IR, Perkin Elmer-Spectrum 65), X-Ray Diffractometer (XRD, PANalytical X'Pert Pro MPD), Field Emission Scanning Electron Microscope (FESEM, TESCAN company, MIRA3).

3. RESULTS AND DISCUSSION

3.1. TGA

TGA was accomplished with the air on the RH at the temperatures ranging from 25 to 800 °C. Figure 1 shows a graph of the weight changes of RH with regard to temperature. Three main steps are depicted in this graph. The first step is done in the temperature range of 145.58-26.48 °C with the weight loss of 5.22 %, the second step in the temperature range of 375.91-145.58 °C with the weight loss of 44.09 %, and the third step in the temperature range of 637.33-373.91 °C with the weight loss of 31.46 %. Therefore, it can be concluded that a total weight loss of 80.77 % leaves 19.19 % residual substance that represents the ash content.

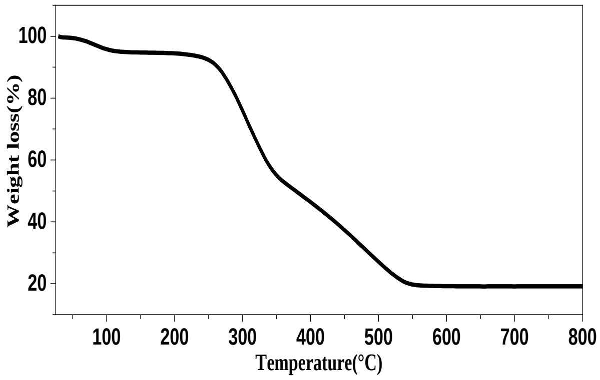


Figure 1. TGA graph of RH

Based on the temperature ranges of these steps, it can be concluded that the weight loss in the first step in the TG curve, which exhibits a weak endothermic response, can be attributed to RH's dehydration. The second weight loss is due to the release of organic and flammable materials. The third and final stage is related to carbonate and carbon decomposition associated with CO_2 emissions. However, at temperatures higher than 637.33 °C, no weight loss can be observed in the sample, indicating that the RH combustion is completed. Consequently, if we heat the husks at this temperature or higher temperatures, we can obtain RHA with relatively highly pure SiO_2 [31,32].

3.2. XRF

XRF test was carried out on RHA prepared at 850 °C. The percentages of oxide compositions of the available elements are tabulated in Table 1, where RHA has a high percentage of SiO_2, i.e., 89.40 wt. %.

3.3. FTIR

The samples as-received RH and extracted SiO_2 were investigated through FTIR. Figure 2 illustrates the FTIR spectra of RH and extracted SiO_2.

TABLE 1. Elements in RHA, in wt. %

Element	wt. %	Element	wt. %	Element	wt. %
SiO_2	89.4	CoO	< 0.01	I	<0.01
K_2O	1.94	NiO	< 0.01	La	<0.01
CaO	0.87	CuO	< 0.01	Cs	< 0.01
P_2O_5	0.64	Ga	< 0.01	Ba	< 0.01
SO_3	0.5	Ge	< 0.01	Ce	< 0.01
MgO	0.45	As_2O_3	< 0.01	Pr	< 0.01
Al_2O_3	0.22	Se	< 0.01	Nd	< 0.01
Fe_2O_3	0.13	Br	< 0.01	Hf	< 0.01
MnO	0.08	Rb_2O	< 0.01	Ta_2O_5	< 0.01
Cl	0.04	Y	< 0.01	WO_3	< 0.01
PbO	0.027	ZrO_2	< 0.01	Hg	< 0.01
TiO_2	0.02	Nb_2O_5	< 0.01	Tl	< 0.01
Cr_2O_3	0.01	Mo	< 0.01	Bi	< 0.01
SrO	0.01	Ag	< 0.01	Th	< 0.01
ZnO	0.01	Cd	< 0.01	U	< 0.01
Na_2O	< 0.01	SnO_2	< 0.01		
V_2O_5	< 0.01	Te	< 0.01		

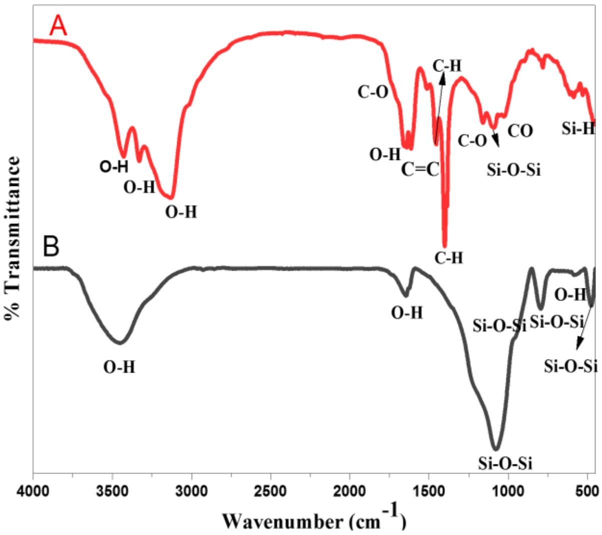


Figure 2. FTIR spectra of (A) RH, (B) silica extracted from RHA

The FTIR spectra of the raw RH shows the peaks at around 3130-3517 cm^{-1} (O-H group), 3015-2770 cm^{-1} (C-H group), 1744 cm^{-1} (C=O group), 1613 cm^{-1} (C=C group), 1455-1401 cm^{-1} (CH_2 and CH_3 groups), 1158 cm^{-1} (CO group), and 953 cm^{-1} reflected the Si–O–Si stretch vibration modes of the SiO_2, and 862.1-476.4 cm^{-1} (Si-H group) [33,34]. In the FTIR spectra of the RH extracted SiO_2, the peaks related to the organic components disappeared as a result of RH combustion.

In addition, removal of inorganic elements from the surface of the RH through base treatment and combustion resulted in the formation of SiO_2 functional group (Si-O-Si) of the RHA-extracted SiO_2 sample appearing more intense than that of the raw RH [35]. The wide peak in the range of 3000-3600 cm^{-1} is attributed to the vibration of O-H functional groups of the water molecules adsorbed on the SiO_2 as moisture.

3.4. XRD

The crystalline structure of the extracted SiO_2 was analyzed using XRD. Figure 3 demonstrates the XRD pattern of RHA-extracted SiO_2.

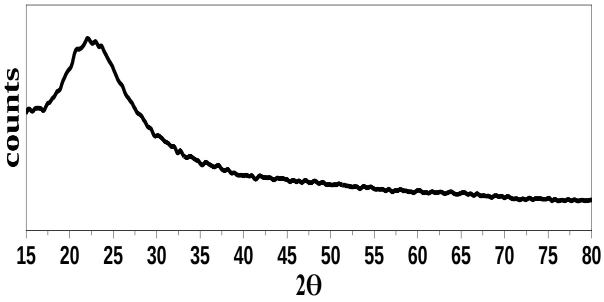
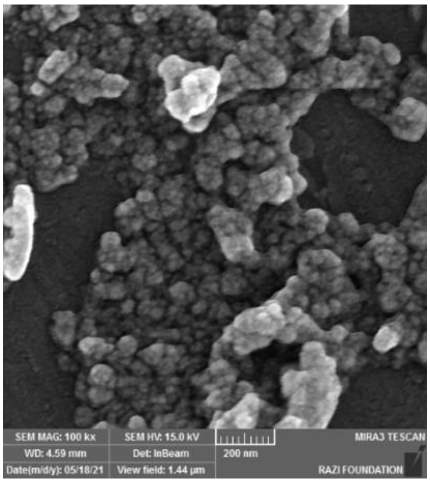


Figure 3. XRD pattern of RHA-extracted silica

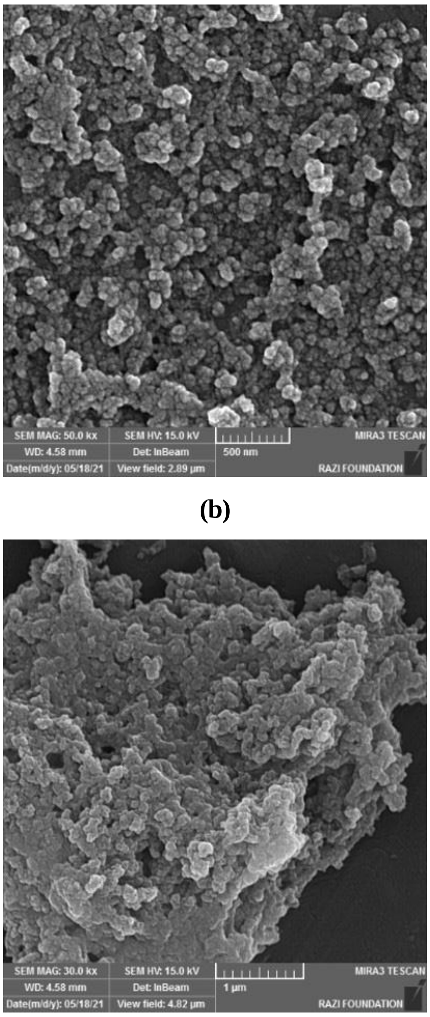
In this figure, the obtained diffraction has no diffraction peak but a wide peak at $2\theta = 23°$, which is considered a characteristic of amorphous SiO_2 [36], thus confirming the extraction and synthesis of amorphous SiO_2.

3.5. SEM

Based on FESEM, the morphology of the produced product was evaluated. Figure 4 depicts the FESEM images of SiO_2 extracted from RHA with different magnifications that show the agglomerated SiO_2 particles of spherical shape with the average particle size of 40 nm.



(a)



(b)

(c)

Figure 4. SEM images of alkaline-extracted silica with magnifications of (a) 100 kx, (b) 50 kx, and (c) 30 kx

4. CONCLUSION

In this study, a simple, low-cost, environmentally-friendly, and low-energy method was employed to produce SiO_2 from the abundantly-in-reach RHA called the alkaline extraction of SiO_2 from RHA and its precipitation through sodium silicate solution acidification. The obtained results revealed that RH was composed of a significant amount of SiO_2. The best combustion temperature and duration to achieve carbon-free RHA were 700 °C and five hours, respectively. The produced SiO_2 was amorphous with no unconventional functional group attached. In addition, the resulting SiO_2 particles were spherical in shape with the average size of 40 nm. Synthesized RHA extracted SiO_2 could be utilized as catalysts in chemical applications as adsorbents as well as in other industries such as chromatograph packing columns, cosmetics, pharmaceuticals, paint and coating, etc.

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