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Microwave Oven Assisted Synthesis of Silica Powder from River Sand

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Abstract: Synthesis of Silica (SiO_2) from the Padma River sand of Bangladesh using a novel microwave oven-assisted alkali-fusion reduction method has been presented in this article. Microwave assisted process was considered because of its advantages like lower power consumption, faster reduction kinetics, and reduced production costs. A specialized microwave kiln featuring a porous ceramic body potentially made of alumina with a high-temperature susceptor material coating facilitated the reduction reaction. At first the temperature profile with time inside the kiln was investigated first for various power level of the microwave oven. SiO_2 extraction from river sand was achieved by alkali fusion using NaOH inside the microwave kiln. The resulting solution from the alkali fusion process underwent filtration to separate unreacted sand, followed by conversion of filtered sodium silicate into silica gel through titration and filtration. X-ray diffraction (XRD) analysis confirmed the amorphous nature of the silica powder obtained, while X-ray fluorescence (XRF) analysis indicated a high purity level of 91.4 wt%. This research highlights the efficiency and potential applications of microwave-assisted alkali-fusion methods in sustainable silica production from natural sources.

1. Introduction

Due to the enormous importance of Silica (SiO_2) in many industrial applications, researchers are motivated to synthesize it in green and cost-effective ways (Saha et al., 2024). Synthesis of SiO_2 has been done from crop residues (Kouadri et al., 2023) such as rice husk ash (Bakar et al., 2016), coconut fibrous husk (Anuar et al., 2018), and bagasse ash (Megawati et al., 2018; Norsuraya et al., 2016) with a 99 percent acquisition rate depending on different types of natural resources. Researchers have also synthesized SiO_2 from non-biological elements such as beach sand (Eddy et al., 2015) and carbon waste such as fly ash sediment (Cheng et al., 2018) with 91.19 percent. Coastal areas are the most promising areas for coastal sand with a high silica mineral content (55.30-99.87wt%) (Ishmah et al., 2020).

Silica is widely used as a precursor for a wide range of materials, including catalysts, thin layers, and coatings for electronic and optical materials (McDaniel & Kelly, 2018; Yang et al., 2018). Many industrial sectors, such as glass, mirrors, pottery, silicon carbide, and sandblasting, now use silica. In addition, silica is commonly used as a supporting material in the cast steel, oil, and mining industries, as well as refractory bricks (Munasir et al., 2015). Silica has been combined with calcium to create a new nanoparticle composite as a bioactive material for bone tissue substitution (Tavafoghi et al., 2018). Silica is also being considered as an anti-corrosion agent to reinforce polymer composites (Yeganeh et al., 2019). Since high-purity silica has a melting point of 1700°C, it is expensive to be used in commercial applications (Omar et al., 2016). As a result, one of the ways to minimize production costs is to extract silica using natural materials such as sand.

Microwave-assisted synthesis can be considered as a promising technique in the extraction and synthesis of silica due to its unique ability to heat materials more uniformly and efficiently. Unlike conventional thermal methods, which rely on conduction and convection to transfer heat, microwaves interact directly with the material, resulting in rapid and selective heating (Yadav et al., 2020). This not only accelerates the reaction kinetics but also significantly reduces the overall energy consumption.

Because natural silica gets contaminated with oxides and other minerals, it must be separated to obtain pure silica. Precipitation, electrocoagulation (Zhang et al., 2019), emulsification (Gustafsson & Holmberg, 2017), Stöber method (Meier et al., 2018), sol-gel (Azlina et al., 2016; Saravanan & Dubey, 2020), hydrothermal (Munasir et al., 2015), and alkaline fusion (Munasir et al., 2013) are some of the established ways of acquiring silica. An alkali fusion technique is used in this study to produce high-purity silica. The principle of this technique is to use an alkaline solution such as KOH, NaOH, or Na_2CO_3 to destroy the chemical bonds in the sand, followed by the linking of silicon with oxygen to form SiO_2.

SiO_2 can be synthesized or extracted from both biological and non-biological natural resources. Natural sand has been processed and synthesized into silica nanoparticles with great success (Hoang et al., 2022; Li et al., 2022; Meiliyadi L. A. D. et al., 2023). So far, silica having a purity of more than 99 % has been manufactured from bagasse ash (organic material) (Affandi et al., 2009). Using co-precipitation, a 98 percent purity of nano-silica was manufactured from rice husk ash (Thuadaij & Nuntiya, 2008), as well as a 95.7 percent purity of nano-silica from mud (Munasir et al., 2013). Trabelsi et al. completely synthesized amorphous silica from inorganic material, namely Deuriet natural sand, by reacting sodium carbonate (Na_2CO_3) with a heating temperature of 1030 °C (Trabelsi et al., 2009). Furthermore, Mori suggested another technique for obtaining 99.9% silica by breaking chemical bonds with alkali solutions, such as KOH and NaOH, and then bonding the SiO_2 from waste glasses (Mori, 2003). This scheme is considered as the alkali-fusion method.

2. Experimental Procedure

2.1 Sand preparation

Sand collected from Padma River (comprises 71.05 wt% SiO_2 and various additional chemicals, according to initial XRF analysis) was first washed with distilled water for several times. Then the sand was ground using ball-milling technique to make fine particles of around 200 µm diameter. Minerals like iron is one of the most problematic contaminants in silica sand because it has a negative impact on the production of pure silicon products. When it comes to removing iron and aluminum impurities, there are a few acids that are more effective than others. According to the literature review sulfuric (H_2SO_4), hydrochloric (HCl), and Phosphoric (H_3PO_4) acids were found to better suit for removing Iron and Aluminium. However, we used diluted HCl solution because it is cheaper and more readily available.

In terms of the procedure, it entails mixing a 2M HCl solution into the sand, stirring it for a few hours, then rinsing with distilled water, decanting 10 times (Islam et al., 2019). The color of the acid washed sand becomes much brighter and whiter than it was before. Sand is washed with acid to remove any cations (e.g., K, Ca, Mg, Al and Fe ions) from the cation exchange sites. After that, the Cl^- is removed by rinsing with distilled water. After that the sand was dried again in an oven at 100 °C for 6 hours. By acid washing many of the minerals leave the sand but it doesn't get rid of presence of all minerals.

2.2 Microwave Oven Characterization

The chemical process of synthesis of silica powder from river sand was taken place in microwave oven. Microwave oven can't heat up all the materials in the periodic table by electromagnetic radiation. Hence a microwave kiln was used to heat up the mixtures taking part in the reduction process. Microwave kiln can absorb electromagnetic radiation and produce heat accordingly. Galanz 'Singer D90D23AL-C3' model microwave oven was used in the whole process of extraction. For different power levels and run time of microwave oven, the temperature inside the kiln was varied. Data obtained from the study shows that at power level 'P-20' the ramp rate of rise in temperature was very low. With changing the power level 'P-20' to 'P-Hi' the ramp rate of rise in temperature also goes high. A rapid increase in temperature was recorded for 'P-Hi' power level. Graph for increasing temperature with time at different power levels has been depicted in **Figure 1**.

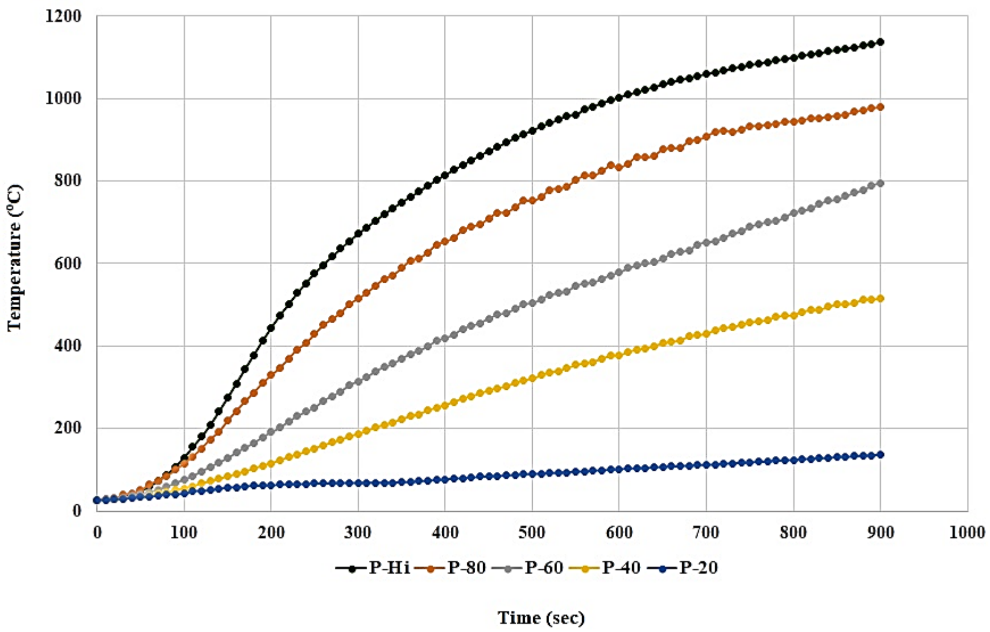

Figure 1. Rise in temperature with time for different power levels of microwave oven.

2.3 Synthesis of Silica (SiO_2) Powder

There are three main steps in extracting silica using microwave assisted alkali fusion process. The first step is to use sodium hydroxide (NaOH) to make sodium silicate (Na_2SiO_3) from silica sand. Treating powdered sand with NaOH produces the Na_2SiO_3. The preparation of silicic acid ($Si(OH)_4$) is the second step (Bao et al., 2007). The Na_2SiO_3 solution reacts with HCl to form a precipitated silica gel. Because $Si(OH)_4$ does not dissolve in strong acids such as HCl, HNO_3, or H_2SO_4, the precipitated $Si(OH)_4$ is separated from its solution of HCl, HNO_3, or H_2SO_4 (NaCl). The third step is to dry the silica gel $Si(OH)_4$ to make SiO_2. In the final step, heat treatment is done in the furnace. The following are some of the possible reaction mechanisms (Affandi et al., 2009; Mori, 2003; Thuadaij & Nuntiya, 2008; Trabelsi et al., 2009).

SiO_2 (sand) + NaOH → Na_2SiO_3 (l) + H_2O (l)
Na_2SiO_3 (l) + 2HCl (l) + H_2O (l) → 2NaCl (s) + $Si(OH)_4$ (aq)
$Si(OH)_4$ (aq) → SiO_2 (s) + $2H_2O$

In our experiment, the concentration of NaOH was varied to determine the effect of its concentration on yield of production of SiO_2. Since the reaction for the production of SiO_2 was carried out in a microwave kiln, which lacks sufficient space to accommodate large-scale materials, the focus had to be on conducting the reaction with a small amount of materials. In an alkaline solution, silica compounds disintegrate rapidly, but in an acidic solution, they stabilize.

At first concentration of sodium hydroxide was chosen 8M for production of silica. In this case 4.6 g of sand was taken into 22 ml of water and then 5.4g of sodium hydroxide was added into the mixture slowly and the mixture was continued to stir so that the pellets of NaOH could be dissolved into the water (**Figure 2**). When NaOH pellets started to be dissolved into the water the temperature of water started rise instantly. Stirring helps to speed up interaction between the solute and the solvent as well as disperse the temperature, and it therefore decreases the frequency of precipitation. The mixture of 8M NaOH and 4.6 g of sand was then placed into a microwave kiln which was the set into a microwave oven. Since the power as well as the temperature of microwave oven is controllable, the mixture was kept at the temperature of 200 ^{o}C for 30 minutes to take place the reaction for producing sodium silicate. The following reactions take place during the extraction process (Ishmah et al., 2020):

SiO_2 (sand powder) + NaOH (aq) → Na_2SiO_3 (aq) + H_2O (l)

After forming sodium silicate, it becomes dry, 50 ml of distilled water was added and the sodium silicate was soaked into the water. Of course, stirring was continued to proceed. Then filtration was done by filter paper. The filtrate is isolated from the residue after the sodium silicate solution (Na_2SiO_3) is formed. The filtrate (Na_2SiO_3) was then mixed with 6M HCl and stirred to form a gel, which was then kept for 18 hours. HCl solution is added to Na_2SiO_3 solution with an initial pH of 11 to achieve pH 7 and form tetraortosilicate acid ($Si(OH)_4$) and sodium chloride, resulting in the formation of silica gel (NaCl) . The following is the reaction:

Na_2SiO_3 (aq) + H_2O(l)+ 2HCl(aq) → $Si(OH)_4$(aq)+ 2NaCl(aq)

The precipitate was then rinsed with distilled water to remove any residual acid before being dried in a 120°C oven for 12 hours. Dehydration of silicic acid occurs at 120°C, resulting in silica gel ($SiO_2.H_2O$), which is then crushed to produce SiO_2 powder (**Figure 2**).

Figure 2. Preparation of silica powder by alkali-fusion reduction of sand.

3. Results and Discussion

3.1 FTIR Transmittance Spectra Analysis

Collected sand sample's FTIR transmittance spectra are shown in **Figure 3**, and the stretching, bending, and out of plane Si-O bonds are represented by the characteristic bands around 1065, 777, and 471 cm^{-1}, respectively. A stoichiometric silicon dioxide structure can be seen in the location and shape of the major Si-O vibrational band at 1065 cm^{-1}. The collected sands' FTIR spectra revealed a significant number of OH groups in the films at around 3420 cm^{-1}. These peaks would be reduced if the heating method was properly followed. The existence of synthesized SiO_2 particles is confirmed by the FTIR transmittance range (225 to 4000 cm^{-1}) shown in **Figure 4**. The existence of the O-H group was attributed to the large peak between 3200 and 3600 cm^{-1}. Similarly, at 1640 cm^{-1}, a peak corresponding to vibration bending can be seen, suggesting the presence of an O-H stretching bond. According to Adam and Chua (Adam et al., 2004), water molecules are normally eliminated completely at higher annealing temperatures. Furthermore, the asymmetric and symmetric Si-O-Si stretching vibration bonding were connected to the strong bands at 1087, 460, and 794 cm^{-1} (Bao et al., 2007).

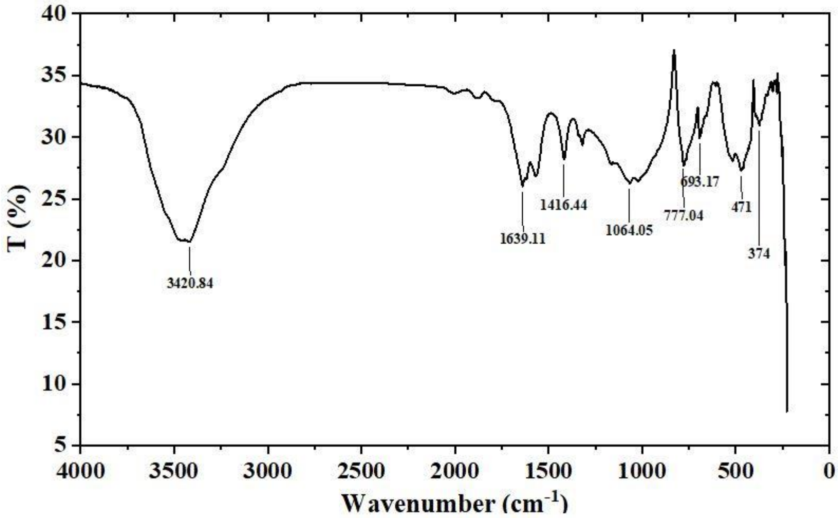

Figure 3. FTIR transmittance spectra of purified sand.

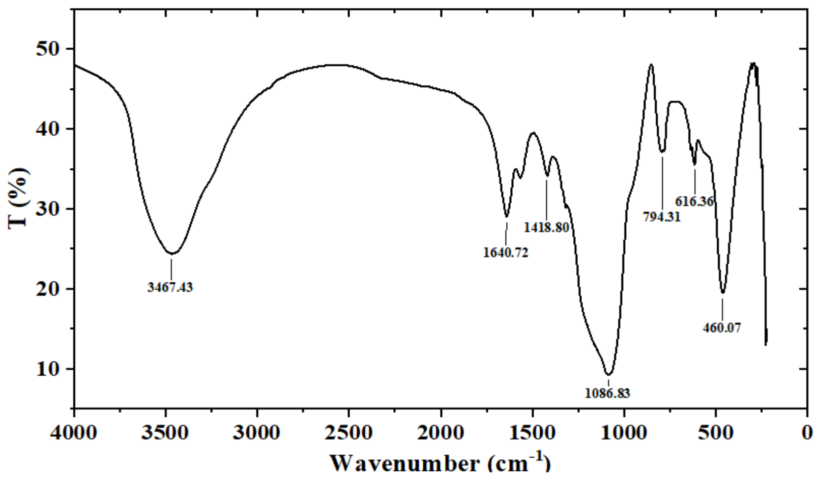

Figure 4. FTIR spectra of silicon dioxide particles.

3.2 X-Ray Fluorescence (XRF) Analysis

The data obtained from XRF has been depicted in **Figure 5**. The purity of silica powder was obtained about 91.4 weight percent. Again, the reactions occurred to synthesize silica powder removes some minerals like magnesium, potassium, calcium, titanium, nickel etc. completely. This reaction introduces presence of Na_2O. More careful washing was needed to minimize these types of impurities and hence to enhance the purification level of silica powder. Moreover, the amount of aluminum oxide and iron oxide were reduced to only 1.83% and 1.1% respectively which was remarkable to achieve. But still there are some contaminants that are needed to be reduced to synthesize pure silica powder. Since synthesis process of silica powder produces sodium silicate and strong acid like HCl was used as the titration of Na_2SiO_3.

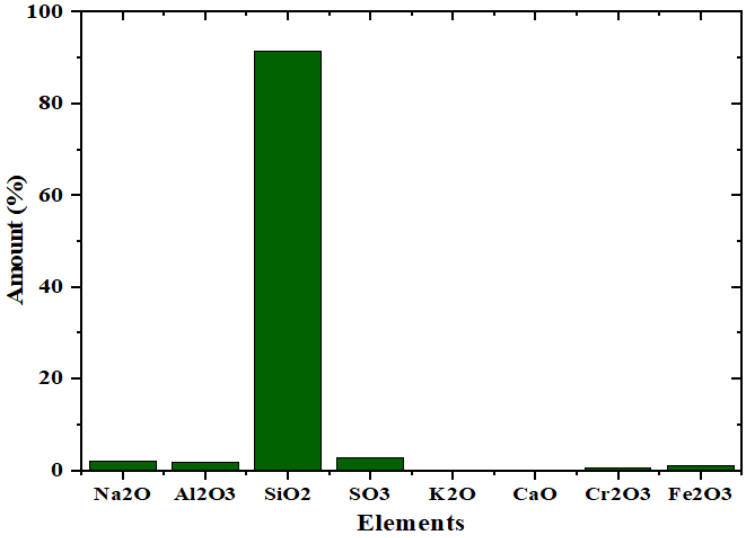

Figure 5. XRF of extracted silica powder.

3.3 X-Ray Diffraction (XRD) Analysis

The X-ray Diffractometer (XRD) was used to classify the synthesized silica particles using CuKα (value of λ is 1.54 Å) as a source of radiation. **Figure 6** shows the XRD patterns of silica particles obtained from alkali-fusion of purified sand. The powder diffraction pattern shows a large peak at 2θ = 23°, indicating that the produced silica particles are amorphous in nature. According to the silica activity index, the non-crystallin phase is again supported by the absence of strong peaks that would have indicated the absorption of crystal structures that were organized. The XRD pattern also shows that there is no ordered crystalline structure.

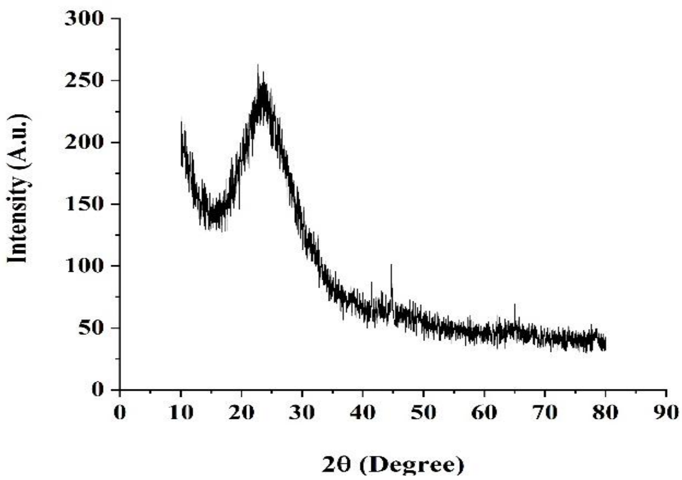

Figure 6. XRD analysis of Silica powder.

3.4 Scanning Electron Microscopy (SEM)

Figure 7 displays a scanning electron microscope (SEM) image of the synthesized white SiO_2 particles. The SEM image reveals a porous surface, indicative of the material's enhanced surface area, which is uniformly and closely distributed across the substrate. The irregular yet cohesive structure, free from observable cracks, underscores the material's robustness and structural integrity. The porous nature of the SiO_2 surface holds significant potential for various applications, particularly in the adsorption of dye molecules. The high surface area combined with the intricate pore network allows SiO_2 to act as an efficient adsorbent, capable of trapping and removing organic dyes from aqueous solutions, a property of great relevance in water purification technologies. This characteristic also positions it as a valuable material in catalysis and environmental remediation, where the ability to selectively adsorb and interact with pollutants or chemicals is essential.

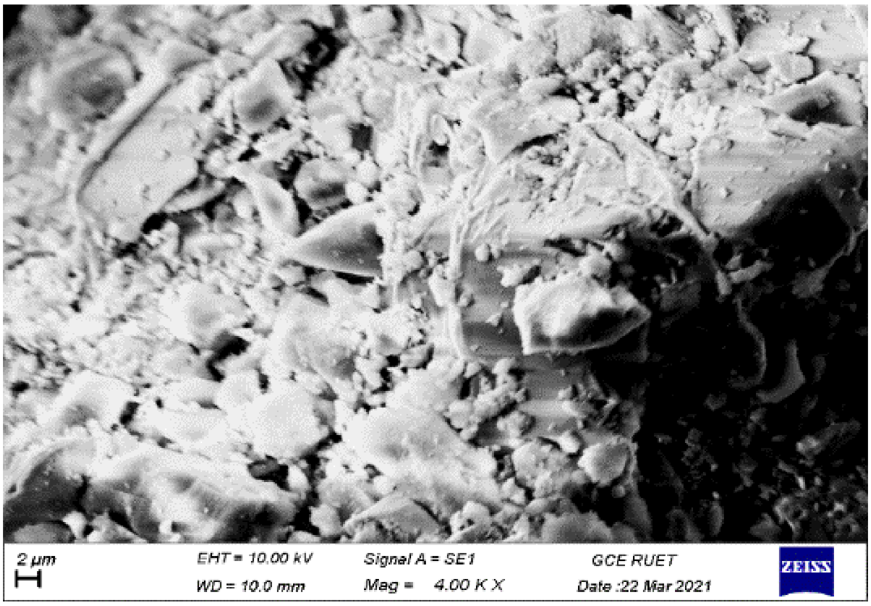

Figure 7. SEM image of silica powder.

Conclusion

The microwave-based alkaline fusion method offers a sustainable approach for silica extraction at low temperatures, ensuring an environmentally friendly process with zero carbon emissions. Analysis using X-ray Fluorescence (XRF) revealed a high purity level of 91.4 wt% for the silica powder, with minimal residual constituents from the original sands. X-ray Diffraction (XRD) confirmed the amorphous nature of the silica powder. Further purification can be achieved by thorough washing of the final product with distilled water, potentially enhancing its purity. This research underscores the feasibility and effectiveness of microwave-based methods in producing high-quality silica with minimal environmental impact.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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