Synthesis And Characterization Of Amorphous Silica From Oil Palm Empty Palm Fruit Bunch

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Abstract
The valorization of agroindustrial solid wastes, particularly oil palm residues such as empty fruit bunches (EFB), for sustainable and green chemistry initiatives has gained momentum in recent years. This paper explores the extraction of amorphous silica from EFB ash as a means to repurpose this abundant waste material. Characterization of the silica nanoparticles was conducted through scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), particle size and zeta potential analysis, and X-ray diffractometer (XRD) analysis. SEM images revealed irregular shapes and varied compositions of the nanoparticles, while FTIR confirmed the presence of silanol groups and CTAB molecules on the surface. Particle size analysis indicated a size of approximately 962 nm with a zeta potential of -26.9 mV, suggesting negatively charged surfaces. XRD analysis confirmed the amorphous nature of the silica nanoparticles. Overall, this study demonstrates a novel approach to extract silica from EFB ash, highlighting its potential for various applications such as adsorbents, catalysts, and biopolymers, thus contributing to sustainable waste management and green chemistry practices.

Keywords Empty fruit bunch, amorphous silica, SEM, XRD, FTIR

Introduction

In the pursuit of sustainable and green chemistry, the valorization of agroindustrial solid wastes, particularly oil palm residues, has gained significant attention. These residues, such as oil palm empty fruit bunches (OPEFB), are abundant and can be repurposed into valuable materials, thereby reducing waste and environmental contamination (Abogunrin-Olafisoye et al., 2024). The ash derived from empty fruit bunches is rich in silicon, making it a renewable source for producing value-added materials like silica nanoparticles (Rahim et al., 2023). Improper disposal of this ash can lead to environmental pollution and health hazards, especially when the silica is in crystalline form (Arfiana et al., 2021).

Various studies have explored different avenues for utilizing oil palm residues. For instance, the production of nanocellulose hydrogels from oil palm empty fruit bunch resources has been investigated for versatile applications (Zaffar et al., 2022). Additionally, the application of oil palm fibers in cement composites has shown promise, indicating the potential for incorporating these residues into construction materials (Momoh & Osofero, 2020). Furthermore, the production of hybrid plywood biocomposites using oil palm waste nanoparticles highlights the potential for creating sustainable materials (Nuryawan et al., 2020).

Moreover, the conversion of oil palm empty fruit bunch cellulose into bioplastics demonstrates a step towards eco-friendly alternatives (Isroi et al., 2017). The utilization of oil palm empty fruit bunches as mulch in oil palm plantations not only helps in waste management but also contributes to enhancing soil fertility and plant growth. Furthermore, the gasification of palm empty fruit bunches for syngas production showcases a method to extract energy from these residues (Adu et al., 2022).

In conclusion, the valorization of oil palm residues, particularly empty fruit bunches, presents a significant opportunity to reduce waste, minimize environmental impacts, and create value-added materials. By exploring innovative ways to repurpose these agroindustrial solid wastes, it is possible to address environmental challenges while promoting sustainability in various industries. The current study contemplates the extraction of silica from empty fruit bunch and its characterisation for the chemical and physical properties.

Materials and method

Collection of palm kernel residues

Palm kernel empty fruit bunch (EFB) residues were gathered from the oil palm plantation at Thondamuthur block, Coimbatore. The EFB husks were washed with tap water to eliminate any dirt or impurities. Subsequently, they were dried in hot air oven at 60˚C for 24 hours to decrease the moisture content. The dried residues were preserved for subsequent analysis and experimentation. The moisture content of some of the PKS was measured using the ASTM D4442-92 standard at a temperature of 105 °C in an oven.

Combustion process

The dried residues were crushed into smaller particles using high-intensity ball milling machine and sieved to 0.2 mm size. The crushed residues EFB were burnt in a muffle furnace at 750°C for 2 h to obtain EFB ash (Santana Costa & Paranhos, 2018)**.**

Preparation of sodium silicate solution from ash

50 g of EFB ash along with 500 mL of 3 M NaOH solution and heated for 2 hours on a hotplate with constant stirring (Fig. 1), resulting in the formation of a silicate solution (Ghorbani et al., 2015).

Surfactant-Mediated synthesis of amorphous silica from the obtained sodium silicate

A solution was prepared by dissolving 4.5 g of CTAB in a mixture of 100 mL of water and 100 mL of butyl alcohol (1:1, v/v) in a 500 mL round bottom flask (Fig. 1). The mixture was then heated to 60°C. Under continuous stirring, 40 mL of the sodium silicate solution was added to the emulsion/biphasic system. Afterward, a solution of sulfuric acid with a concentration of 0.5 mol L^{-1} was slowly added until the pH dropped to 4. The gel was subsequently left to age at a temperature of 60°C for a duration of 8 hours. The silica present in the aged gel was carefully cleansed using distilled water, then filtered, and ultimately dried in an oven at 120 °C (Imoisili et al., 2020; Rovani et al., 2018).

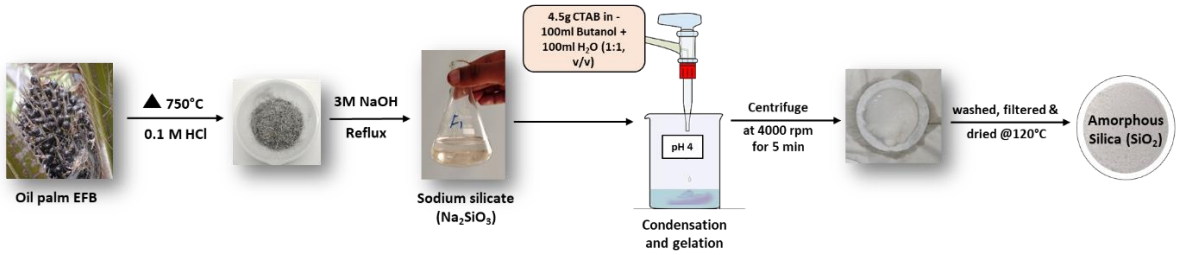

Fig. 1. Synthesis process of amorphous silica from EFB

Results and discussion

A pretreatment procedure was conducted on the EFB ash to remove any excess salts, organic compounds, and low solubility elements. This was achieved by thoroughly washing and eliminating particles (Kalapathy et al., 2000). After conducting the pretreatment procedure, amorphous silica was successfully extracted from EFB ash through a reaction with NaOH at a temperature of 400°C (Alves et al., 2017). By subjecting sodium hydroxide to a temperature of 400°C, the extraction of silicon (Si) was enhanced through the process of melting. This method helped enhance the purity of silicon by releasing elements trapped in the ash structure, making them more soluble. The extraction process resulted in the formation of silicate, which was then dissolved in deionized water, filtered, and stored for the subsequent production of amorphous silica.

There are several methods available for preparing amorphous silica, such as the sol-gel method, hydrothermal synthesis, flame synthesis, and the reverse microemulsion technique. Additionally, the functionalization of amorphous silica can be achieved through grafting or co-condensation methods. During the co-condensation process, the preparation and functionalization of amorphous silica take place simultaneously in a single step. This involves the hydrolysis-condensation reaction, as observed in previous studies (Hoffmann et al., 2006; Wu & Lin, 2013).

For this study, the sodium silicate solution was utilised to create amorphous silica. The generation of amorphous silica was achieved through the hydrolysis (creation of silanol groups) and condensation (formation of siloxane) reactions utilizing sulfuric acid in a biphasic environment with the inclusion of Cetyltrimethylammonium bromide (CTAB) (Wang et al., 2004). According to the literature, the use of CTAB, a well-known micelle maker, was found to be effective in regulating the size of nanoparticles, preventing them from clumping together, and altering their surface properties (Hoffmann et al., 2006; Wu & Lin, 2013). As a result, the co-condensation process involving sulfuric acid produced a solid of a pale color that did not disperse well in either medium. However, it could be easily separated by centrifugation.

Scanning electron microscope (SEM) analysis

The SEM image of EFB ash derived amorphous silica displayed in Fig. 2 illustrates a material with varied composition and irregular shapes. Observations revealed the presence of sizes ranging from 50 to 150 nm and a significant level of roughness. These characteristics are commonly linked to the emission of organic matter that occurs during the EFB burning process, which was utilized for energy generation during pyrolysis. The morphologies obtained from the EFB ash are comparable to other studies (Batra et al., 2008; Faria et al., 2012).

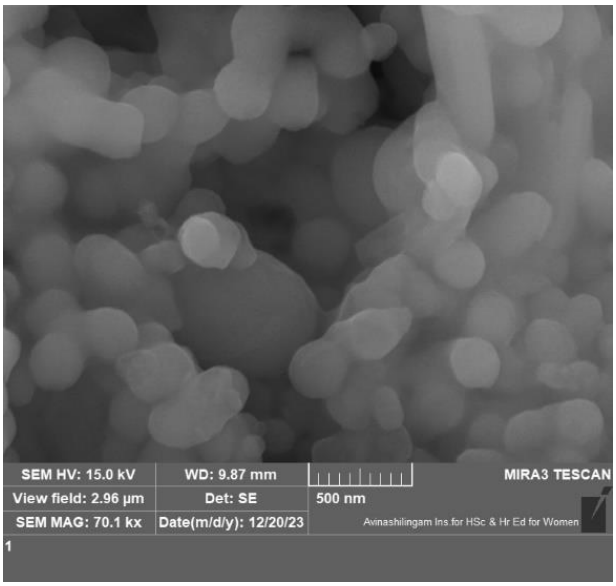

Fig. 2. SEM image of amorphous silica

Fourier transform infrared spectroscopy (FTIR) of amorphous silica

Fig. 3 shows the observation of the presence of CTAB molecule on the surface of the amorphous silica using FTIR. The FTIR spectrum of amorphous silica reveals the existence of four prominent bands of silica at 786 and 447 cm^{-1}, which correspond to the symmetric stretching of siloxane groups (Si−O−Si). Furthermore, a prominent peak at 1064 cm^{-1} is attributed to the asymmetric stretching of Si−O−Si bonds, while another peak at 965 cm^{-1} is a result of the bending of OH groups originating from silanol groups. There were bands detected at 2877 cm^{-1}, which can be attributed to the symmetric and asymmetric stretching of CH_2 (Ruiz et al., 2016; Su et al., 2014; Zhang et al., 2012).

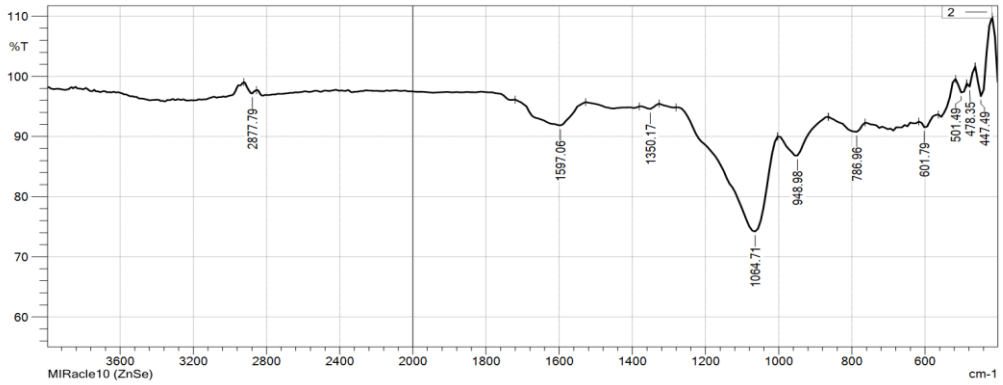

Fig. 3. FTIR spectrum of amorphous silica

Particle size and zeta potential

The size of the particles (Fig. 4) are around 962 nm and the zeta potential of the particles are around -26.9 mV (Fig. 5). The zeta potential of silica show that the surface of silica are negatively charged with oxygen functional groups that was confirmed by the FTIR analysis.

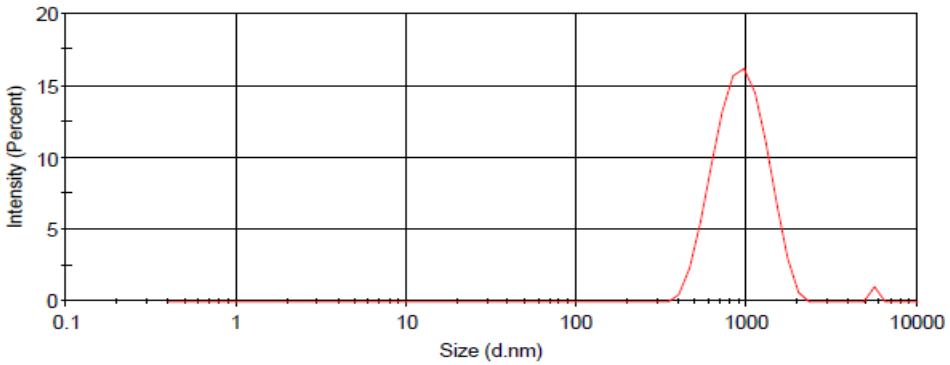

Fig. 4. Size of amorphous silica

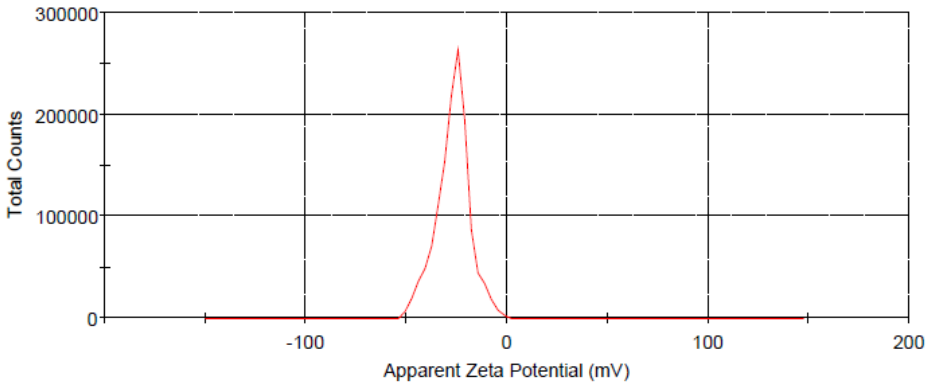

Fig. 5. Zeta potential of amorphous silica

X-ray diffractometer (XRD) analysis

X-ray diffractogram of EFB ash derived amorphous silica nanoparticles is presented in Fig. 6. The broad XRD array of extracted silica nanoparticles at theta = 25◦, which is distinctive of amorphous solid, confirms the formation of amorphous silica; similar results were obtained by other researchers (Athinarayanan et al., 2015; Channoy et al., 2018; Liu et al., 2011; Lu & Hsieh, 2012). The lack of crystallinity nature represented the inoffensive nature of the silica particle for its application for drug delivery systems.

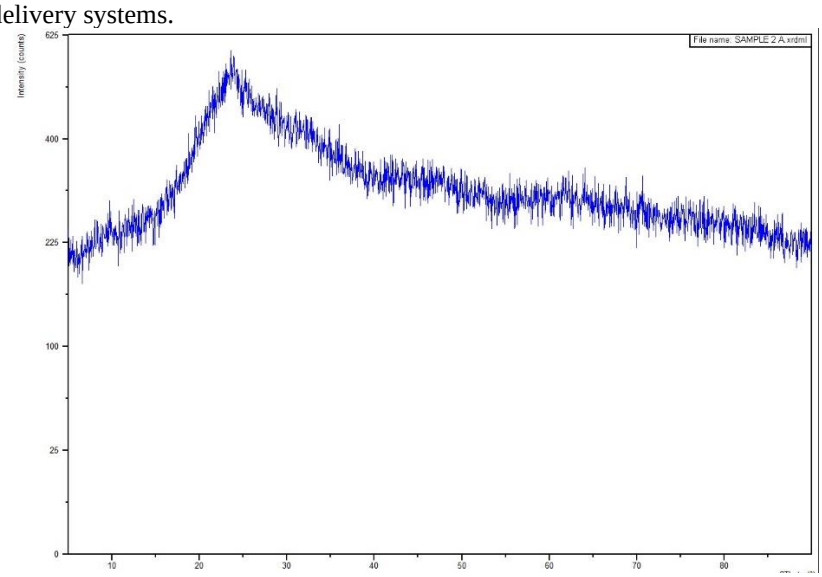

Fig. 6. XRD graph of amorphous silica

Conclusion

This study marked the inaugural utilization of EFB ash to effectively produce amorphous silica. CTAB played a pivotal role in the preparation process, serving as a stabilizer and size-controller. The findings underscore the feasibility of obtaining a commendable green adsorbent from a renewable source at minimal expense. Beyond its role as an adsorbent material, amorphous silica holds promise for diverse applications including catalysts, biopolymers, paints, and more.

Acknowledgment

The authors are thankful to the Department of Environmental Science, Tamil Nadu Agricultural University and the DST-INSPIRE fellowship division for supporting the research.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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