Ultrasonication Temperature Effects on The Green Synthesis of Silica Nanoparticles from Oil Palm Shell

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Abstract. The research interest in vaporizing agricultural waste, especially oil palm empty shells, has increased due to the rising demand for environmentally friendly nanomaterials. This study aims to investigate the environmentally friendly synthesis of silica nanoparticles from oil palm empty shells, emphasizing the impact of varied ultrasonication temperatures using the ultrasonication method on silica nanoparticles yield, and their characteristics (FTIR and SEM analysis). The synthesis involved raw material pre-treatment, silica extraction, and nanoparticles synthesis through controlled ultrasonication with varied temperatures (50, 60, 70 °C), followed by calcination. According to the findings, raising the ultrasonication temperature to 60 °C considerably increased silica nanoparticles and decreased particle agglomeration because of stronger cavitation effects. However, temperatures above this threshold caused morphological abnormalities and minor yield reduction, most likely due to structural disintegration and re-agglomeration brought on by increased cavitation effects. FTIR analysis verified the SiO_2 functional groups. The SEM characterization verified the development of amorphous silica with nanoscale morphology (56.857 nm to 387.6 nm). The optimum temperature was obtained at 60 °C with a yield of 33.13%, and a 56.857 nm size. This study emphasizes the importance of ultrasonication temperature on the environmental nanoparticle synthesis from biomass waste.

1 Introduction

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The interest in utilizing agricultural waste as a renewable resource to produce nanoparticles has increased due to the growing need for sustainable nanomaterials. The agricultural wastes that can be transformed into nanoparticles can be obtained from the rice milling industry, processed fruit and nut industry, aquatic food industry, food processing industry, and oil palm industry [1] [2]. The potential agricultural wastes are such as rice husk, wheat straw, hazelnut tree and shells, cashew nut waste, pomegranate peel, shrimp shell, blueberry waste, orange peel, grape waste, banana peel, coconut shell, egg shell, oil palm shell (OPS), oil palm empty fruit bunches etc [2] [3]. These wastes can produce valuable nanoparticles for pesticide, medicine, food industry, water treatment, coating, and polymer application [1, 2, 4]. Among these biodegradable wastes, oil palm shell (OPS), a solid by-product of the palm oil industry, has drawn a lot of attention because of its significant silica content and abundant availability. The two biggest producers of palm oil, Indonesia and Malaysia, produce more than 9 million tons of OPS a year, most of which is wasted or underutilized as biomass waste. Abdullah et al suggest that 6% of OPS will be produced from processing 1 ton of fresh fruit bunches [5]. However, only a small amount of these wastes is utilized to make a valuable product. It will lead to environmental problems and harm humans and other living beings. The environmental challenges faced by unutilized palm oil waste include particulate emissions, inefficient silica recovery, and greenhouse gas generation [6]. As sustainability and circular economy concepts become increasingly important, there is an urgent requirement to explore value-added utilization pathways for OPS that are both economically viable and environmentally friendly [7].

Traditionally, OPS can be used as biomass fuel in the oil palm industry. The other utilization of OPS is for industrial liquid and solid fuel [8], construction [9], polymer material [10], and catalyst [11]. OPS waste can benefit society using a simple volatilization method [12]. Ash produced by the thermal treatment of OPS can produce OPS ash yielding 40 to 50% silica, which can be utilized for the environmentally friendly production of silica nanoparticles [13]. Compared to other biomass wastes such as rice husk or bamboo, OPS offers superior silica yield and carbon-neutral potential, making it an attractive precursor. Various extraction techniques such as sol-gel [14], hydrothermal [15], precipitation [16, 17], and ultrasonication [13, 18] have been developed to synthesize nano-silica from OPS. However, these methods typically suffer from drawbacks such as long processing times, chemical waste generation, or inconsistent particle morphology [14]. Furthermore, limited studies have addressed the inter-dependence of thermal pre-treatment and downstream processing techniques, particularly the effect of temperature on ultrasonication.

In the synthesis of nanoparticles, ultrasonication has proven to be a successful technique for reducing particle size, improving dispersion, and avoiding agglomeration. Its use in synthesizing silica from biomass waste can greatly increase homogeneity[18]. An effective method for reducing particle size, improving dispersion, and avoiding agglomeration in nanoparticle manufacturing is ultrasonication. It can greatly increase surface area and homogeneity when used in silica [13];

By combining the combustion method, ultrasonication and calcination, this work suggests an efficient and environmentally friendly approach to producing silica nanoparticles from OPS. This work evaluates the effects of varying ultrasonication temperatures (50, 60, 70 °C) on silica yield, particle size, zeta potential, and density. This work fills a major gap in the literature by demonstrating the synergy between initial ultrasonication temperature and subsequent nanoparticle processing. It also provides an affordable and environmentally friendly approach to producing nano silica from waste palm oil. The outcomes are expected to contribute to advanced material design [19], heat transfer applications [20], and lubricant performance [21].

2 Materials and Methods

This work was conducted in six steps, with the variation of ultrasonication temperature as the main step in synthesizing the silica nanoparticles. The steps are raw material preparation, isolation, purification, ultrasonication, calcination, and silica nanoparticles analysis. These phases are a modification method from previous work [13, 14, 22]. The materials used are OPS, distilled water, 32% hydrochloric acid (HCl), and carboxy methyl carbonate (CMC). The step-by-step methodology is explained as follows:

1. Raw material preparation

OPS were collected from a local palm oil mill in Sangatta, West Kalimantan, Indonesia. The raw shells were thoroughly washed with distilled water to remove dirt and impurities, followed by soaking for 2 hours. The shells were sun-dried for 48 hours to ensure moisture removal until the weight was constant. According to Osman et al [22], this pre-treatment is crucial to enhancing the quality of finished silica product and preventing undesirable side reactions during thermal treatment.

2. Isolation process

The dried OPS was subjected to open-air combustion in a furnace at 600 °C for 2 hours to obtain ash rich in silica content. The resulting ash was allowed to cool to ambient temperature in a desiccator to prevent moisture absorption. The selected combustion temperature and time were based on prior research showing that silica retention in ash is optimum at moderate firing temperatures; the combustion temperature was chosen [14]. A 60-mesh screen was then used to sift the ash to achieve a consistent particle size for further processing.

3. Purification Process

After the isolation, the 35-gram OPS ash was dispersed in 10 ml of HCL 32% and 100 ml of distilled water under constant stirring for 15 minutes to eliminate metallic impurities. This amount of 35-gram OPS is named the initial weight (W0) used to calculate the yield percentage. The suspension was cooled, filtered, and washed to reach a neutral pH. This purification process is essential to improve silica quality and lower the concentration of metals like Fe and Al [14]. Before calcination, the resulting material was dried for four hours at 105 °C. The dried silica powder was prepared for the next process.

4. Ultrasonication with temperature variation

The silica powder was dispersed into a 100-ml CMC solution of 5% w/w as surfactant [13]. An ultrasonication of Elma400 (40 kHz, 400 W) was used to ultrasonically treat 10 g of the produced ash at various temperatures (50, 60, 70 °C). The ultrasonication time was set at 60 minutes [23]. A thermostatic water bath integrated with the ultrasonication was used to regulate the temperature. Recent research has highlighted temperature's importance in improving nanoparticle dispersion and cavitation efficiency [24]. To eliminate unreacted residues, the slurry was filtered after sonication, and the residues were repeatedly washed with deionized water.

5. Calcination and final processing

The dried residue was then calcined at 750 °C for 1 hour in a muffle furnace under atmospheric conditions. This final step ensures the removal of any residual organic matter and promotes the formation of amorphous silica nanoparticles. The final product was stored in airtight containers for further characterization. The selected calcination temperature aligns with recent findings recommending thermal treatment above 700 °C to obtain high-purity, amorphous nano-silica from biomass ashes. The silica nanoparticles were obtained and weighed as W_1 to calculate the yield percentage. The synthesis of silica nanoparticles is illustrated in Figure 1.

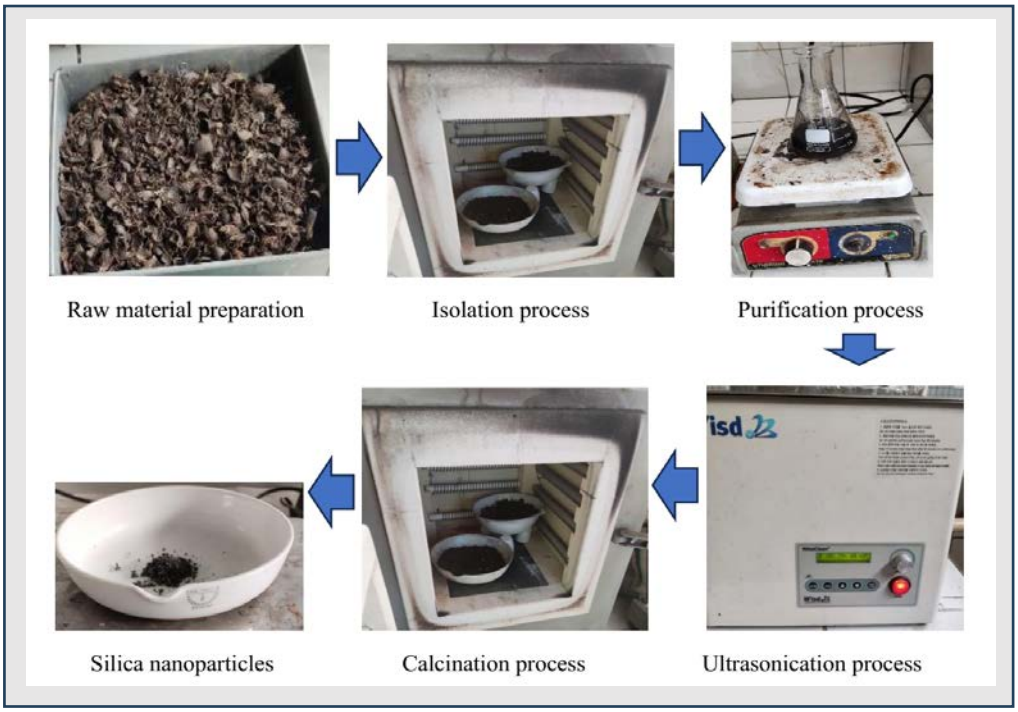

Fig. 1. The synthesis process of silica nanoparticles from OPS.

6. Silica nanoparticles analysis

The analysis was done for yield determination, Fourier Transform Infra-Red analysis (FTIR), and Scanning Electron Microscope (SEM) analysis.

a. Yield determination

The yield of silica nanoparticles was calculated gravimetrically based on the weight of the final silica nanoparticle product obtained (m_1) after synthesis, relative to the raw material dry weight of the OPS ash used (m_0). Each experiment was used to calculate the yield of silica nanoparticles using Equation 1[15].

$$Yield\ (\%) = \frac{W_1}{W_0} x100\% \quad (1)$$

Where W_1 is the weight of final silica nanoparticles (grams), and W_0 is the initial weight of OPS ash used in this work. This calculation provides insight into the efficiency of the synthesis method under different ultrasonication temperatures [15].

b. FTIR analysis

FTIR analysis was performed to verify the chemical structure of the generated silica nanoparticles and identify functional groups. The silica nanoparticle powder was placed in the sample holder for examination. An FTIR spectrometer of Agilest Cary 630 was used to record spectra in the 400 – 4000 cm^{-1} range with a resolution of 4 cm^{-1}. Based on a previous study, the distinctive peaks of silanol groups (Si-OH), Si-O-Si stretching, and other relevant bands were interpreted [14]. FTIR analysis is essential to confirm the amorphous nature and chemical purity of silica nanoparticles synthesized from biomass sources.

c. SEM analysis

The surface morphology and particle size distribution of silica nanoparticles were observed using Scanning Electron Microscopy (JSM-IT300). SEM micrographs were taken with accelerating voltages between 10 and 20 kV and at magnifications of 10,000. Visual proof of particle aggregation, shape (spherical or irregular), and particle homogeneity can be obtained through SEM investigation. Silica nanoparticle surface morphology can be observed and assessed using SEM at a voltage of 20 kV. Due to the unique chamber arrangement, the sample was put on the sample holder without any pre-treatment, even after it had been hydrated [25].

3 Result and Discussion

The analysis of silica nanoparticles obtained was conducted by calculating the yield, analyzing the functional groups by FTIR, analyzing size and surface morphology by SEM, and analyzing the chemical composition by SEM-EDX. The results and discussion are explained in this section.

1. Yield of Silica Nanoparticles

The silica nanoparticles obtained from OPS by varied ultrasonication temperatures are illustrated in Figure 2. The result is black silica nanoparticles resembling OPS in powder form. It is considering the nanoparticles' high carbon content, which is proven by the SEM-EDX analysis. The second explanation is that it is influenced by the significant factor of acidity (pH), according to prior research by Dzulhijjah et al [26]. The yield of silica nanoparticles is a product of the reaction between silica oxide and hydrogen chloride, as presented in equation 2 [27].

$$SiO_{2(s)} + 4\,HCl_{(l)} \rightarrow SiCl_{4(s)} + 2\,H_2O_{(l)} \quad (2)$$

A B C

Fig. 2. Product of silica nanoparticles by varied ultrasonication temperature: A. 50 °C, B. 60 °C, and C. 70 °C

The yield of silica nanoparticles obtained was calculated using Equation 1 after acquiring the powder. Yield shows how effective the silica nanoparticle synthesis method is [28]. This work uses different ultrasonication temperatures to achieve the best yield. The effect of ultrasonication temperature on the yield of silica nanoparticles synthesized from palm oil shell ash is shown in Table 1. It can be seen that from 50 °C to 60 °C, the yield improved with temperature, suggesting improved extraction and dispersion efficiency at moderate temperatures. Because optimal cavitation activity enhances mass transfer and breaks up OPS ash particles to release more silica content, the best yield was achieved at 60 °C. Nevertheless, at 70 °C, there was a modest drop in yield, which might have been caused by the thermal

breakdown of functional groups and the instability of cavitation bubbles at higher temperatures. This condition could have resulted in nanoparticle recombination or agglomeration [18]. These findings highlight the importance of optimizing ultrasonication temperature to enhance the maximum silica nanoparticle obtained, especially for large-scale [29].

Table 1. Yield of silica nanoparticles from OPS by varied ultrasonication temperature.

Sample	Ultrasonication Temperature (°C)	Ultrasonication time (min)	Yield (%)
A	50	60	20.38
B	60	60	33.13
C	*70*	60	29.31

This trend suggests that an optimum ultrasonication temperature exists to maximize silica extraction. This finding is greater than other work using different methods of evaporation and condensation [28]. Dzulhijjah et al. [26] reported a similar finding, stating that excessive heat during ultrasonication decreased nanoparticle recovery because of particle reaggregation.

The ultrasonication process of silica nanoparticles synthesis was influenced by several factors such as frequency, pre-treatment time, particle size, media, time, and temperature [30]. As ultrasonication temperature plays a crucial role in the production of silica nanoparticles, the correlation between ultrasonication temperature and the yield of product should be analysed. The correlation was analysed using the regression method, presented in Figure 3.

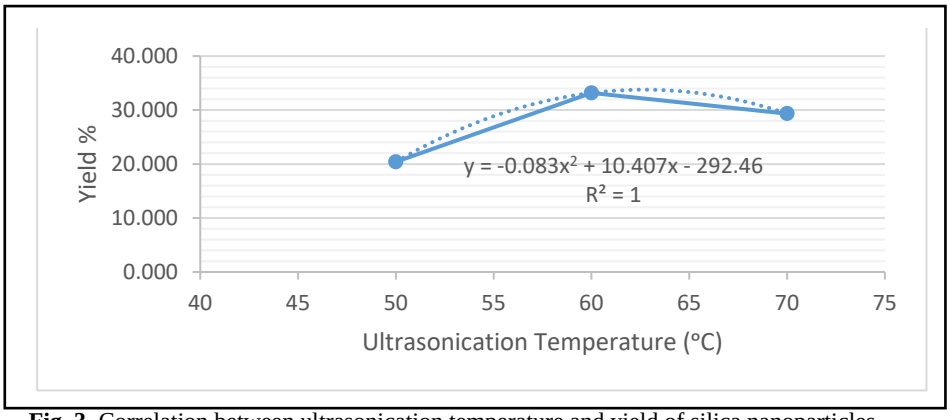

Fig. 3. Correlation between ultrasonication temperature and yield of silica nanoparticles.

Figure 3 displays the correlation utilizing the polynomial equation, which is increased and decreased. The coefficient equation or R^2 value = 1 indicates a strong relationship between ultrasonication temperature and the yield of silica nanoparticles. As the ultrasonication temperature increases, cavitation intensity and the mobility of molecules in

the reaction medium are enhanced, resulting in improved mass transfer and effective dispersion of silica precursors. Moderate increases in temperature (e.g., from 50 °C to 60 °C) have been shown to significantly increase yield due to more efficient breakdown of the biomass matrix, facilitating silica release and solubilization [26].

However, yield may be reduced if the ultrasonication temperature exceeds the ideal threshold (for example, above 60°C). This decrease is because cavitation bubbles become unstable at high temperatures, when the liquid's vapor pressure rises and bubble collapse becomes less severe, lowering the mechanical forces required for particle disintegration [24]. This finding contradicts the hydrothermal method's prior result, in which neither temperature nor time process affected silica nanoparticle yield [15]. To maximize yield while avoiding energy waste and particle aggregation, it is imperative to optimize the ultrasonication temperature.

2. FTIR analysis

The functional groups in the silica nanoparticles were identified using the Fourier Transform Infrared Spectroscopy (FTIR) of the Agilest Cary 630. The absorption band shows the identification of specific functional groups in several samples, which manifests at a particular wave number. Furthermore, the intensity of a certain functional group indicates how many of that group there are in a sample [16]. The FTIR analysis of silica nanoparticles by varied ultrasonication temperature is presented in Figure 4. It can be seen from Figure 4 that the OPS consists of a complex profile of absorption bands in its Fourier Transform Infrared (FTIR) spectra, which suggests the existence of a variety of organic and inorganic functional groups that are mostly derived from its lignocellulosic origin. The absorption bands observed are at 3405 cm^{-1}, 2923 cm^{-1}, 2852 cm^{-1}, 2202 cm^{-1}, 2167 cm^{-1}, 2022 cm^{-1}, 1995 cm^{-1}, 1945 cm^{-1} , 1655 cm^{-1}, 1595 cm^{-1}, 1421 cm^{-1}, 1369 cm^{-1}, 1323 cm^{-1}, 1241 cm^{-1}, 1160 cm^{-1}, 1026 cm^{-1}, 710 cm^{-1}, and 669 cm^{-1}. The broad absorption peak at 3405 cm^{-1} is attributed to the O–H stretching vibrations of hydroxyl groups, characteristic of cellulose, hemicellulose, and residual moisture [31]. This broad and strong band suggests that the biomass is hydrophilic since it shows a lot of hydrogen bonding.

Asymmetric and symmetric C–H stretching vibrations in $–CH_2$ and $–CH_3$ aliphatic groups, which are frequently found in lignin and waxy biomass materials, are shown by the peaks at 2923 cm^{-1} and 2852 cm^{-1} [32]. These bands are indicative of the long carbon chains present in lignocellulosic structures. In aromatic and conjugated systems, bands at 2202 cm^{-1}, 2167 cm^{-1}, 2022 cm^{-1}, 1995 cm^{-1}, and 1945 cm^{-1} are usually linked to C≡C or C≡N triple bond stretches, combination bands, or overtones. These characteristics imply partial heat breakdown or carbonization and are indicative of the complex aromatic structures seen in lignin components [33]. The C=O stretching of conjugated ketones, aldehydes, or perhaps adsorbed water (bending mode of H–O–H) is attributed to the band at 1655 cm^{-1} [34]. In the meantime, aromatic skeletal vibrations at 1595 cm^{-1} verify the existence of lignin's phenolic rings. Cellulosic and hemi-cellulosic backbones are confirmed by the peaks at 1421 cm^{-1}, 1369 cm^{-1}, and 1323 cm^{-1}, which are suggestive of C–H bending and O–H bending (Chen et al., 2021). The C–O–C and C–O stretching vibrations of alcohols, ethers, and esters—which are indicative of cellulose glycosidic connections and hemicellulose acetyl groups—are attributed to the bands at 1241 cm^{-1}, 1160 cm^{-1}, and 1026 cm^{-1}. Suppose there is ash or mineral matter present. In that case, the 1026 cm^{-1} signal is usually strong and sharp, indicating the presence of siloxane linkages (Si–O–Si) Lastly, the low-frequency bands at 710 cm^{-1} and 669 cm^{-1} may be attributed to Si–O bending vibrations and aromatic C–H out-of-plane bending vibrations, respectively, suggesting the presence of silica or inorganic residue in the OPS that may have been left over from the mineral components [35].

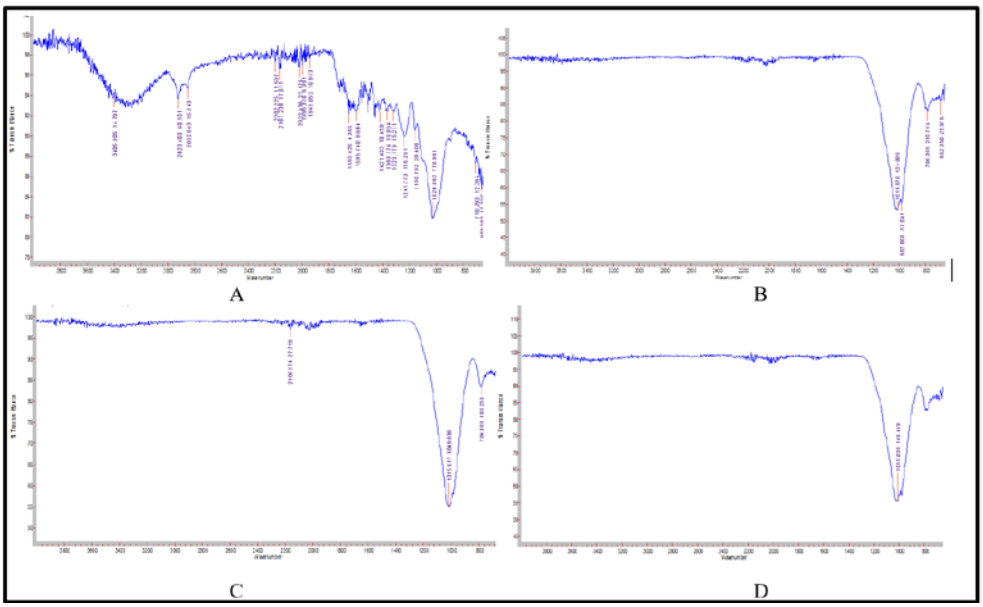

Fig. 4. FTIR result of of OPS (A), and silica nanoparticles by varied ultrasonication temperature: B. 50°C, C. 60°C, and D. 70°C

Despite the OPS samples, all silica nanoparticle samples exhibited the typical absorption peaks for Si-O-Si asymmetric stretching were observed in all samples between 800 and 1100 cm^{-1} for Si-O-Si, Si–O symmetric stretching between 780 and 800 cm^{-1}, and a broad band at 2186 cm^{-1} that corresponded to the O–H stretching vibration from surface silanol groups. Numerous O-H groups on silica are formed by applying acidic conditions. Sample B (60 °C), shown in Figure 4(C), exhibits peaks at 1015.517 cm^{-1} and 784.968 cm^{-1}, undergoing significant spectral changes. This outcome could be interpreted as a possible phase change or decrease in the size of the silica nanoparticles, creating different peaks at specific wavelengths. An increase in O-H groups can impact the quantity of silanol groups on silica. It is simpler to react with silica particles when their silanol group increases [36]. Acids such as HCl and HNO_3 can be added to influence the production of active silanol groups and promote the synthesis of silica nanoparticles [26]. Further evidence of partial dehydroxylation brought on by thermal effects during ultrasonication was the decrease of the 950 cm^{-1} band (Si–OH stretching) at 70 °C, which could lower surface reactivity [24].

Although the two samples' absorption bands are similar (1015 cm^{-1}), their intensities differ. In sample B (50 °C), the intensity of the absorption band was lower than that of sample C (60 °C), suggesting that the siloxane groups at 60 °C were greater than those at 50 °C. The siloxane groups rose and the silanol groups fell at sample C (70 °C), suggesting that the silica compound was undergoing a condensation reaction at that temperature. One silanol group and another underwent a condensation process, producing siloxane groups. The silica components' agglomeration process was what led to the condensation reaction. Due to the reduced agglomeration process at 60 °C, the silanol groups' intensity was higher at 60 °C than at 70 °C. The fact that the siloxane groups' absorption band was less intense at 60 °C than it was at 70 °C demonstrated this phenomenon [16]. These results confirm that the temperature of ultrasonication affects the silanol concentration and surface chemistry of bio-silica, which is important for functional applications.

3. SEM analysis

Determining the optimal ultrasonication temperature is not limited to yield alone. Therefore, other characteristics, such as particle size and morphology, must also be considered to build a complete picture. SEM observations of Sample B (60 °C) were chosen due to the fine powder by naked eye analysis. It revealed a variation in particle size, ranging from 56.857 to 387.06 nm at 10,000 and 30,000x magnification. The SEM result is presented in Figure 5. Despite the smaller particle size, this variation warrants further investigation.

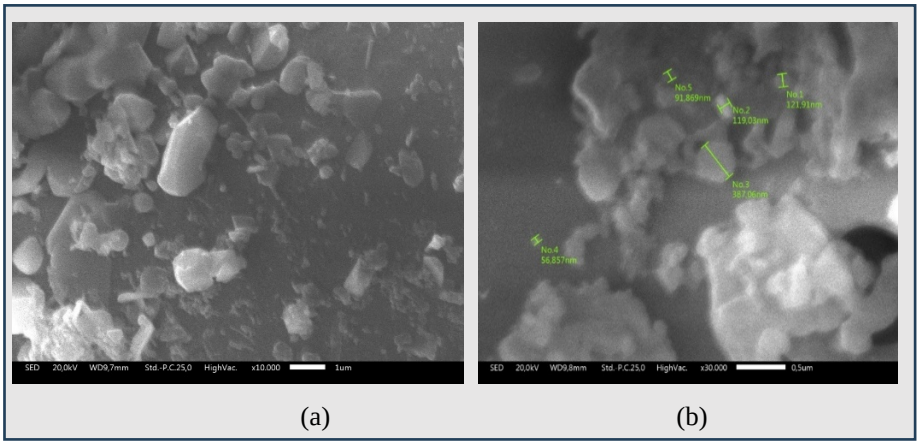

Fig. 5. SEM result of silica nanoparticles by ultrasonication temperature of 60°C at (a) 10,000 magnifications; and (b) 30,000 magnifications

The observed particle morphology included one or more randomly selected nano-silica particles magnified between 10,000 and 30,000 times, as seen in Figure 5. The particle size distribution was observed using a 10,000x magnification, while the morphology of individual particles was observed using a 30,000x magnification. Figure 5 shows the irregular morphology of the resulting silica nanoparticles, and some particles appear larger than the others. These large particles are composed of smaller particles that have undergone agglomeration [13]. Figures 5 (a) show the surface of the silica nanoparticle sample, clearly showing the amorphous structure of the resulting silica nanoparticle. This statement is confirmed by Ismayana [37], who stated that the silica nanoparticle synthesis process using the ultrasonication method produces an amourphous structure because this method generates heat. The resulting heat can encourage the silica nanoparticle crystal structure to become more regular. The distribution of particle sizes that was observed was not consistent. Due to agglomeration, the nano-silica particles' flake and clump shapes were visible by single-particle morphology. This finding is similar to prior research by the precipitation method in flakes particles, yet different in shape [16].

4 Conclusions

This study presents the effect of ultrasonication temperature on the yield of silica nanoparticles. The higher the ultrasonication temperature, the more the yield increases; however, it decreases at 70 °C due to the agglomeration. The findings are also made for the FTIR result of the OPS powder, which exhibits a complex profile of absorption bands, indicating the presence of diverse organic and inorganic functional groups primarily derived from its lignocellulosic nature. The SiO2 functional groups were confirmed by FTIR analysis. The formation of amorphous silica with nanoscale morphology (56.857 nm–387.6 nm) was confirmed by the SEM characterization. With a yield of 33.13 percent and a size of 56.857 nm, the ideal temperature was reached at 60 °C. This study highlights how crucial the

ultrasonication temperature is for the environmentally friendly synthesis of nanoparticles from biomass waste.

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All authors contributed to this work.

The data that support the results of this work are available from the corresponding author upon reasonable request.

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