



## Synthesis of Silica Nanoparticles from Silica Sand via Vibration Assisted Alkaline Solution Method

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### ABSTRACT

The effect of frequency of speaker membrane vibration on the grain size of the silica nanoparticles (SNP) was investigated. SNP was synthesized using the alkaline fusion method under the vibration of the membrane speaker. Variations of membrane vibration used in this research were 0, 50, 100, and 200 Hz. The material compositions, crystal structure, and morphology of the synthesized SNP were characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), and transmission electron microscopy (TEM), respectively. Meanwhile, its dielectric property was determined using impedance spectroscopy. The results showed that the SNP consisted of 99.35% silica and corresponded to the crystalline structure of quartz silica. The SNP size was decreased with increasing vibration frequencies. The smallest size of SNP ( $9.04 \pm 1.9$  nm) was obtained at a frequency of 200 Hz. Moreover, the dielectric constant and dielectric losses were increased with an increase in membrane vibration frequency due to the decrease of SNP size.

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## 1. INTRODUCTION

Indonesia is a country that is rich in potential natural minerals, including oxide materials such as silica sand [1, 2]. There are many silica grains of sand in Indonesia, spread all over the country regions, such as in Poso Regency, Central Sulawesi. Silica sand obtained from Poso has a high SiO<sub>2</sub> content that can be utilized in industrial materials [3]. It can be processed into silica nano particles (SNP) through a top-down or bottom-up method [4]. For the last-mentioned method, SNP was obtained through chemical reactions using precursors. Particles in nanometer size provide advantages such as good electrical, optical, and magnetic properties [5]. SNP is widely used in industry because of its high productivity and low production costs [6]. Previous researchers have reported various methods in synthesizing the SNP, including hydrothermal [7, 8], sol-gel [9], sodium silicate solution [10, 11], alkaline fusion [12-14], and precipitation method [15]. These methods are generally

carried out with stirring to inhibit grain growth; however, the resulting particles have still lacked. Other researchers have also developed several methods to obtain smaller sizes of particles. Indira and Malathi [16] reported the synthesis of hydroxyapatite nanoparticles for biomedical applications using ultrasonic and microwave methods. Rusianto et al. [17] reported the synthesizing of magnetite nanoparticles assisted by mechanical vibration. Yu et al. [18] reported the method of utilizing ultrasonic/mechanical vibrations in metallurgical processes such as welding and metal casting to control metal particle size. It was also reported that ultrasonic vibrations in the welding process can smooth particles and improve the mechanical properties of welds [18-20].

SNP can exist in three crystal structures, namely quartz, tridymite, and cristobalite. It has a large surface area, good heat resistance, high mechanical strength, and can be used as catalyst precursors, adsorbents, and composite filters, easy modification, good chemical stability, and low cytotoxicity [5, 21, 22]. SNP can be

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used to improve the cooling effect and efficiency of the vapor compression refrigeration cycle [23]. Nowadays, SNP is commonly used in various industries such as rubber, filler, catalyst carriers, [24-27], food, automobile, energy storage materials, piezoelectric materials, paints, medical, electronics, and others [28, 29]. A dielectric material is an insulator with high resistivity that can be polarized if there is an electrostatic dipole or under an external electric field [30]. Previous researchers have reported the impact of the nano-silica amount on its dielectric properties [31, 32]. The combination methods between alkaline fusion and membrane vibration can be used in synthesizing silica nanoparticles by heating the sample at a specific temperature, depositing it and then it followed by vibrating and stirring until the pH is neutral. This method is advantageous because it does not require a long time and does not require high energy [13]. As a result, the synthesis process takes place effectively and efficiently to produce nanoparticles. The SNP synthesis method that has been used so far has limitations, including the lack of nanoparticles produced, and the particle size is not controlled, so a new approach is needed.

This study reports the new method of SNP synthesizing by combining the alkaline fusion with membrane speaker vibration, which offers mass production of SNP with controllable size. This study aimed to investigate the effect of the frequency of speaker membrane vibration on the particle size and the dielectric properties.

## 2. MATERIALS AND METHODS

The raw material used to synthesize silica nanoparticles was obtained from silica sand on Poso Island, Central Sulawesi, Indonesia [33]. Sodium hydroxide and hydrochloric acid used in this work were bought from Merck (Germany).

Silica nanoparticles (SNP) was prepared by using a combination of alkaline fusion and speaker membrane methods as follows. Briefly, the raw silica sand was cleaned and dried in an oven. The metallic elements within the silica sand were removed using several permanent magnets to obtain non-metallic compounds with high concentrations. Silica sand was crushed using a ball mill to find smaller particle sizes. Then, the crushed silica sand was sieved into < 200 mesh using a sieve shaker. Silica sand concentrates were mixed with pro-analyst NaOH in a ratio of 1: 1 by weight fraction, then heated at 600°C for 1 h. The heated mixture of silica sand and NaOH was put into a beaker glass which was filled with distilled water. The beaker glass was placed on a hot plate magnetic stirrer and heated at about 75°C with a stirring speed of 500 rpm. Stirring was combined with the vibration of the speaker membrane with different

frequencies of 0, 50, 100, and 200 Hz with a duration of 30 min, respectively. The solution was stirred for 16 h while the pre-analytical HCl solution was titrated with a concentration of 37% at 2 M. The solution would form a residue (silica gel) at the pH of 7-8. The silica precipitates were filtered using Whatman glass microfiber filter (grade 42, 2.5 µm) and then washed with distilled water until it turned white. The washed silica residues were dried in an oven at 100°C for 15 h and then characterized. Variations of membrane vibration used were 0, 50, 100, and 200 Hz, respectively, then referred to as SNP0, SNP50, SNP100, and SNP200. The schematic of the synthesis of SNP with the alkaline fusion method combined with a speaker membrane can be seen in Figure 1.

The chemical composition and microstructure of SNP were characterized using X-ray fluorescence (XRF) (RIGAKU-NEX-QC+QuanTES) and transmission electron microscope (TEM) (JEOL JEM-1400), respectively. Images of each SNP from the TEM image were analysed using Image J software to determine the grain size distribution. X-ray Diffraction analysis (Bruker D2 Phaser) was used to identify the crystalline phase of each SNP. Then, the synthesized SNPs at various frequencies of 0, 50, 100, and 200 Hz were compacted in a 15 mm diameter cylindrical die with pressure of 75 MPa to produce green bodies. The green bodies were sintered at 1360°C for 2 h with a heating rate of 10°C/min. The dielectric properties of the sintered samples were tested using a computerized impedance spectroscopy device with a sine frequency generator that produced a modulating frequency in the range of 10 to 500 kHz.

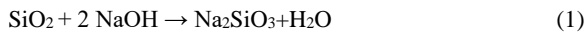
## 3. RESULT AND DISCUSSION

**3. 1. Synthesis of Silica Nanoparticles** The main mineral in silica sand is quartz which has a tetrahedron



**Figure 1.** Schematic illustration of the synthesis method of SNP

structure, where at high temperatures, each tetrahedron will be separated from each other because the bonds between the anions and cations are not very strong [34], so that NaOH binds silica to form sodium metasilicate that was separated from other minerals. Consecutively, sodium metasilicate was titrated with HCl to produce hydrous silica and silicic acid. Then, the white residue in a gel was washed with distilled water to remove the remaining solution. The chemical reactions of this synthesis process are presented in Equations (1) and (2) [35].



### 3. 2. Composition

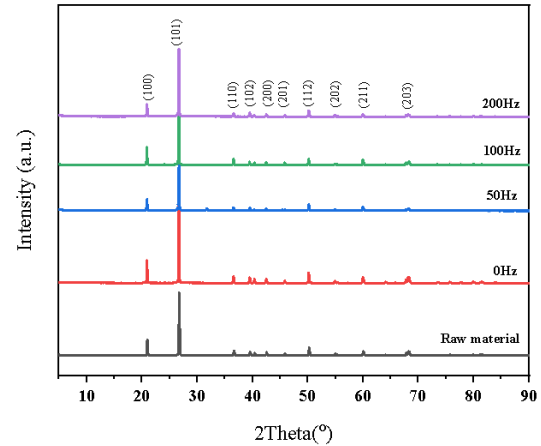
Table 1 shows the chemical composition of the silica sand and SNP as revealed by XRF. It was found that both silica sand and SNP have the main component of SiO<sub>2</sub> around 99.35-99.64%. From Table 1, it can be also observed that the content of the same components is indicated by both silica sand and SNP. It can be concluded that the synthesis of SNP did not change the composition of silica sand.

### 3. 3. Crystal Structures

The XRD diffractogram of the raw material (silica sand) and the obtained SNP are presented in Figure 2. As observed, 2θ of the peaks of raw material and SNP are not significantly different. As compared to JCPDS (33-1161 card number) for silica quartz, the appeared sharp peaks show that the raw material and SNP are in the quartz phase with the related 2θ are 20.90°, 26.70°, 36.58°, 39.51°, 42.49°, 45.83°, 50.18°, 54.91°, 60.64°, 68.16°. In line with the literature, quartz is one of the crystalline phases of silicon oxide that formed at temperatures below 870°C [36]. From Figure 2, it can be seen that various vibration frequencies of the speaker membranes (0, 50, 100, and 200 Hz) did not affect the SNP crystal structure. The SNP synthesized with various frequencies exhibits a quartz phase. Furthermore, X-ray diffraction is not only used to identify the crystal structure but also is used to determine the crystal size by using the Scherer equation as shown in Equation (3) [17]:

**TABLE 1.** Chemical composition of silica sand and the SNP

Composition	Silica sand (wt%)	SNP (wt%)
SiO <sub>2</sub>	99.64	99.35
TiO <sub>2</sub>	0.20	0.18
Fe <sub>2</sub> O <sub>3</sub>	0.06	0.06
NiO	0.03	0.02
CuO	0.01	0.01
ZnO	0.01	0.01
Others	0.05	0.35



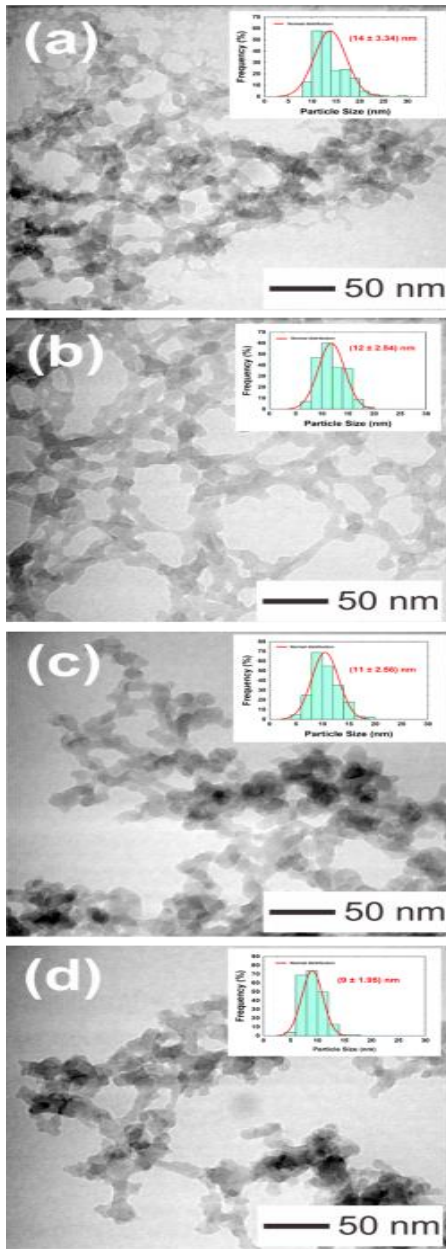
**Figure 2.** XRD diffractogram of synthesized SNP with various frequencies of speaker membrane vibration

$$D = (0.9 \lambda) / (\beta \cos \theta) \quad (3)$$

where D is the size of the crystallite diameter (nm), λ is the wave length of the x-ray used, θ is the angle (1/2 peak angle), and β is the full width of half maximum (FWHM). The four peak at 2θ of 20.90°, 26.70°, 36.58°, and 50.18° are used as the basis for determining the size of the crystal diameter. It was found that the SNP crystal size obtained at different frequencies (0, 50, 100, and 200 Hz) are 10.8, 5.0, 1.04, and 0.9 nm, respectively. Based on the XRD results, it can be concluded that the synthesis of SNP by the alkaline fusion method combined with a speaker membrane did not change the crystal structure of silica sand. This finding is in consistent with XRF results as previously discussed. In addition, increasing the vibration frequency reduces the crystal size of the SNP. This is associated with an increase in frequency can increase the nucleation rate and then result in smaller crystal size [37].

### 3. 4. Morphology Studies

Figure 3 shows the TEM images and the size distribution of SNP for various speaker membrane vibration frequencies. The size of silica particles is in the nanometer order, which depends on the frequency of the speaker membrane vibration. From Figure 3, it was found that the particle sizes of SNP at different frequencies of 0, 50, 100, and 200 Hz are 14±3.34, 12±2.54, 11±2.56, and 9±1.95 nm, respectively. The SNP size decreases with the imposition of speaker membrane vibrations. This indicates that the higher vibration frequency leads to a smaller size of produced SNP. Thus, the SNP size decreases with the application of speaker membrane vibrations. This is because the vibrational frequency of the membrane can inhibit crystal growth. After all, the arrangement of atoms becomes disturbed or unstable when speaker membrane vibration is applied. In comparison, the increase in frequency will increase the vibration energy, where the vibration energy

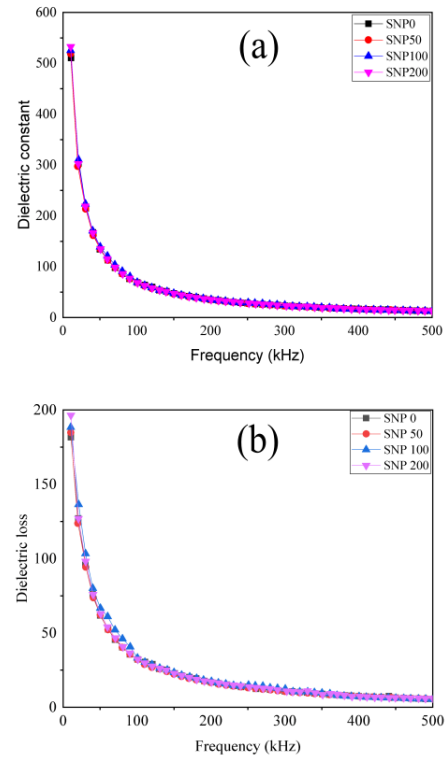


**Figure 3.** TEM images of SNP and its particle distributions with various speaker membrane vibration frequencies: a). 0 Hz, b). 50 Hz, c). 100 Hz, and d). 200 Hz

can inhibit the growth of crystals to form smaller particles [17]. The TEM results are consistent with the XRD results as mentioned before, where increasing the vibration frequency decreased the SNP crystal size.

**3. 5. Dielectric Properties**

Figure 4 shows the frequency dependence of real dielectric permittivity ( $\epsilon'$ ) and dielectric loss ( $\epsilon''$ ). The dielectric permittivity and dielectric loss of SNP0, SNP50, SNP100, and SNP200 are decreased with the increase of frequency. Meanwhile, the decrease in real dielectric permittivity and dielectric



**Figure 4.** Frequency-dependent plot of permittivity for SNP0, SNP50, SNP100, and SNP200, (a) dielectric constant, and (b) dielectric loss

loss is significant in the range of 10 kHz to 200 kHz and be saturated over 200 kHz. The real dielectric constant and the imaginary dielectric constant are calculated by Equations (4), (5) and (6).

$$\Phi = \arctan (V_c/V_r) \tag{4}$$

$$\epsilon' = (d \sin \theta)/(2\pi f \epsilon_0 A|Z|) \tag{5}$$

$$\epsilon'' = \epsilon' \tan \delta \tag{6}$$

where  $\phi$  is the capacitance impedance which determined by  $|Z| = V_c \max/V_r \max \times R$ ,  $\epsilon'$  is proportional to the energy stored and  $\epsilon''$  is proportional to the energy lost or dissipated. While the loss tangent value is the ratio between the permittivity of the imaginary dielectric to the permittivity of the real dielectric. The two parts of the permittivity (real dielectric and dielectric loss) in the low-frequency region have strong dispersion. The real dielectric constant and dielectric loss show low values at high frequencies and increase with decreasing frequency. This phenomenon is comparable to that reported in the literature [37, 38].

The sample exhibits strong dielectric dispersion behavior at low frequencies due to the significant contribution of space charge polarization to dielectric properties, and the dielectric constant remains nearly

constant at higher frequencies [39, 40]. Another thing that causes is the inability of electrons to align their position with a given electric field so that the polarization between grain decreases which affects the dielectric value.

The decrease in grain size causes an increase in the number of grains and grain boundaries, which directly reduces the number of dipole moments. The poling plane does not have many domains to exchange, so the poling process is inefficient in a particle. Similar types of results have been reported for oxide ceramics [41]. Furthermore, four types of polarization, i.e., dipolar, ionic, electronic, and interfacial polarization, contribute to the total polarization of the dielectric material [42]. The different magnitude of the real dielectric value in each sample is due to differences in grain size. Decreasing the grain size leads to a higher dielectric value [39, 43]. Moreover, the highest dielectric occurs in SNP200, which is in agreement with the TEM results. Other factors that affect the dielectric value are defects, residual stress at the interface, porosity, grain boundaries, and variations in crystal structure parameters [44].

#### 4. CONCLUSIONS

Silica nanoparticles sourced from natural sand were successfully synthesized using the alkaline fusion method assisted by speaker membrane vibration. XRF analysis showed SiO<sub>2</sub> content of 99.36% by weight and a similar to the silica content of silica sand. XRD analysis indicated that the identified phase of SNP was quartz. TEM observations showed that the silica nanoparticle's size was decreased with an increase in speaker membrane vibration frequency. The dielectric constant and dielectric loss show high values at low frequencies and decrease at high frequencies, and this occurs in all samples. With an increasing frequency of the speaker membrane vibration during synthesis, the value of the dielectric constant was found to be higher, and this was the effect of decreasing the grain size of silica nanoparticles.

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**Persian Abstract**

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**چکیده**

اثر فرکانس ارتعاش غشای بلندگو بر اندازه دانه نانوذرات سیلیس (SNP) مورد بررسی قرار گرفت. SNP با استفاده از روش همجوشی قلیایی تحت ارتعاش بلندگوی غشایی سنتز شد. تغییرات ارتعاش غشا در این تحقیق ۰، ۵۰، ۱۰۰ و ۲۰۰ هرتز بود. ترکیبات مواد، ساختار کریستالی و مورفولوژی SNP سنتز شده به ترتیب با استفاده از فلورسانس اشعه ایکس (XRF)، پراش پرتو ایکس (XRD) و میکروسکوپ الکترونی عبوری (TEM) مشخص گردید. در همین حال، ویژگی دی الکتریک آن با استفاده از طیف سنجی اهدانس تعیین شد. نتایج نشان داد که SNP از ۹۹/۳۵ درصد سیلیس تشکیل شده و با ساختار کریستالی سیلیس کوارتز مطابقت دارد. اندازه SNP با افزایش فرکانس ارتعاش کاهش یافت. کوچکترین اندازه  $9.04 \pm 1.9$  SNP نانومتر در فرکانس ۲۰۰ هرتز به دست آمد. علاوه بر این، ثابت دی الکتریک و تلفات دی الکتریک با افزایش فرکانس ارتعاش غشا به دلیل کاهش اندازه SNP افزایش یافت.

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