

Fluorescent Silica Nanofertilizer for Enhancement of Photosynthesis Rate

Nanofertilizer Silika Fluoresens untuk Peningkatan Laju Fotosintesis

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ABSTRACT

This study aims to synthesize and characterize fluorescent silica nanofertilizers using Mount Sinabung volcanic ash as a silica source. The synthesis method involved extracting silica from ash using a concentrated acid solution, followed by functionalization using triethanolamine (TEA) via the sol-gel method. Characterization included surface morphology analysis using Scanning Electron Microscopy (SEM), crystal structure identification using X-ray Diffraction (XRD), functional group identification using Fourier Transform Infrared (FTIR), and UV absorption measurement using UV-Vis Spectroscopy. The results showed that fluorescent silica nanofertilizers exhibited smooth surface morphology, uniform particle size distribution (particle size: 48.49 ± 1.31 nm), amorphous structure with increased crystallinity after TEA functionalization (crystallinity: 11.88%), and significant enhancement in UV absorption (absorbance: 0.65) and fluorescence after TEA functionalization. The conclusion of this study is that fluorescent silica nanofertilizers can enhance plant photosynthesis efficiency through improved light absorption and fluorescence properties.

Keywords: Sinabung ash, silica, fluorescent silica nanofertilizer, photosynthesis rate.

ABSTRAK

Paku sisik naga (Pyrrosia piloselloides (L.) M. G. Price) adalah salah satu tumbuhan yang tumbuh dan Penelitian ini bertujuan untuk mensintesis dan mengkarakterisasi nanofertilizer silika fluoresens dengan memanfaatkan abu Gunung Sinabung sebagai sumber silika. Metode sintesis melibatkan ekstraksi silika dari abu menggunakan larutan asam pekat, diikuti dengan fungsionalisasi menggunakan trietanolamin (TEA) melalui metode sol-gel. Karakterisasi meliputi analisis morfologi permukaan dengan Scanning Electron Microscopy (SEM), identifikasi struktur kristal dengan X-ray Diffraction (XRD), pengidentifikasian gugus fungsi dengan Fourier Transform Infrared (FTIR), dan pengukuran penyerapan UV menggunakan UV-Vis Spectroscopy. Hasil penelitian menunjukkan bahwa nanofertilizer silika fluoresens memiliki morfologi permukaan yang halus dan distribusi ukuran partikel yang seragam (ukuran partikel: $48,49 \pm 1,31$ nm), struktur amorf dengan peningkatan kristalinitas setelah fungsionalisasi TEA (kristalinitas: 11,88%), serta peningkatan signifikan dalam penyerapan UV (absorbansi: 0,65) dan fluoresensinya setelah fungsionalisasi dengan TEA. Kesimpulan dari penelitian ini adalah nanofertilizer silika fluoresens dapat meningkatkan efisiensi fotosintesis tanaman melalui sifat penyerapan cahaya dan fluoresensi yang ditingkatkan..

Kata Kunci : Abu sinabung, silika, nanofertilizer silika fluoresens, laju fotosintesis.

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1. INTRODUCTION

Agricultural productivity is a critical issue, especially given its impact on global food security. Rapid global population growth exacerbates these productivity constraints, demanding increased efficiency and innovation in agricultural practices (Chugh et al., 2021). Unpredictable climate change, including rising temperatures and changing weather patterns, also affects the intensity of sunlight received by plants, which in turn influences the rate of photosynthesis (Dilnawaz et al., 2023). Moore et al. (2021) have studied the interaction of leaves with rising temperatures and heat stress, which can disrupt the rate of photosynthesis and plant development. High photosynthetic efficiency is crucial for agricultural productivity, and innovative and environmentally friendly technologies are key to achieving this goal sustainably (Dilnawaz et al., 2023).

Excessive use of chemical fertilizers has been shown to damage soil health, the environment, waterways, and reduce farmers' profits (Chugh et al., 2021). Silica nanoparticles with flexible properties and good biocompatibility have been widely used in various biological applications. The ability to combine siloxane precursors with fluorescent dyes in silica nanoparticles enables the engineering of nanomaterials for various fields (Gubala et al., 2020). Nano technology is considered to have great potential for developing innovative products in various aspects, including agriculture (FAO and WHO, 2013). The development of nano technology in Indonesia is still relatively new (Rahayu, 2021). Nano technology is expected to be a solution to improve fertilization efficiency, reduce environmental impact, and optimize plant growth (Li et al., 2023).

Research shows that volcanic ash from Mount Sinabung contains 51.8% silica (SiO_2) in the form of nanoparticles, which have the potential to be used as nanofertilizers (Bariyah and Simatupang, 2020). Silica nanoparticles offer a promising alternative with a large

surface area, enabling them to increase the efficiency of nutrient absorption by plants. The use of nanotechnology in agriculture can stimulate plant growth and enhance disease resistance, as well as improve agricultural productivity through smart and environmentally friendly nutrient release patterns (Chugh et al., 2021; Du et al., 2022).

Silica nanoparticles (SiNPs) have unique properties, including a large surface area, penetration capability, and a structure that allows them to penetrate plants and regulate their metabolic processes (Goswami et al., 2022). Previous research has demonstrated the potential of fluorescent silica nanoparticles derived from coal fly ash as a sustainable and environmentally friendly raw material (Singh et al., 2024). Functionalization with polar groups using the hydrothermal method enhances nanoparticle stability and dispersion, enabling more effective applications (Saita and Kawasaki, 2021). The use of SiNPs as nano-fertilizers helps protect plants against pathogens in leaf tissues and supports increased photosynthesis rates through the transfer of energy emission to chlorophyll (Li et al., 2020).

The significance of this research lies in its innovation in utilizing abundant local natural resources, namely volcanic ash from Mount Sinabung, which has the potential to become a sustainable solution to global agricultural problems. This research aims to develop fluorescent silica nanoparticles from Mount Sinabung geomaterials with modified amino groups to enhance the stability and dispersion of the nanoparticles as a nanofertilizer. The benefits of this research include improved fertilization efficiency, reduced environmental impact from the use of chemical fertilizers, and increased crop productivity through optimized photosynthesis rates.

2. MATERIALS AND METHODS

2.1. Materials

The main materials used in this study were volcanic ash from Mount Sinabung obtained from Namanteran Village, Kabanjahe Regency, sodium hydroxide (NaOH) e-merck, hydrochloric acid (HCl 37%) e-merck, triethanolamine (TEA), ethanol pro analys, and distilled water (H₂O).

2.2. Methods

In this study, the sol-gel method was used for the synthesis of silica and fluorescent silica. The method used refers to the research by Bariyah and Simatupang (2020) and Singh et al (2024) with the following modifications.

2.2.1. Synthesis of Silica from Sinabung Ash

Twenty grams of 200-mesh ash sample were added to a 12 M HCl solution for 24 hours. The volcanic ash was then neutralized with distilled water and dried at 120 °C for 6 hours. After weighing, the Sinabung ash was added to 8 M NaOH and boiled until thickened. The solution was then placed in a furnace at 750 °C for 3 hours, cooled, and mixed with 200 mL of distilled water before being left to stand overnight. The mixture was filtered, and the resulting solution was titrated with 3M HCl while stirring with a magnetic stirrer at 40 °C until the pH approached 7. The precipitate formed was then filtered, washed with distilled water, and dried at 120 °C for approximately 3 hours.

2.2.2. Synthesis of Fluorescent Silica Nanofertilizer

Silica nanoparticles (in variations of 0.5 g, 1.0 g, and 1.5 g) were dissolved in 25 mL of ethanol at a temperature of 40 °C for 30 minutes. Five milliliters of TEA were slowly added while stirring at a temperature of 60 °C for 2 hours. After the reaction was complete, the mixture was cooled and centrifuged at 10,000 rpm for 10 minutes to obtain the amine-modified fluorescent silica nanofertilizer supernatant. The resulting nanofertilizer was then dried at 50 °C, dissolved into a stock solution, and stored at 4°C for further use.

2.2.3. Characterization of Silica and Fluorescent Silica

Morphological and particle size analysis was performed using a Scanning Electron Microscope (SEM) to determine the size distribution and particle shape at various magnifications. Crystallinity or amorphous structure determination was performed using X-Ray Diffraction (XRD) to identify crystalline phases and crystallinity degree. Fluorescence absorption spectra were measured using a UV-Vis spectrophotometer, and functional groups were identified using Fourier Transform Infrared (FTIR) Spectroscopy.

3. RESULTS AND DISCUSSION

3.1 Extraction of Silica from Sinabung Ash

Silica found in nature is impure, so to obtain silica in a material, separation and purification from other elements contained in the sample must be carried out. To obtain pure silica from Sinabung ash, extraction was performed using concentrated HCl 37% (12 M) to dissolve impurity metals such as Fe, Ca, Al, and other metals from the ash sample. After the purification process, 18 grams of ash were obtained from the 20 grams of sample used.

Next, sodium silicate as a silica precursor was made by adding 8 M NaOH to the activated ash. This reaction produced a sodium silicate (Na₂SiO₃) solution, which was then reacted with 3 M HCl to precipitate silica as a stable white gel.

Silica was successfully synthesized from Mount Sinabung ash using the sol-gel method, yielding 9.015 grams of silica with a yield of 49.78% w/w (Figure 1). The yield obtained was quite high and consistent with literature stating that the silica content in Mount Sinabung ash is 51.8%. Sinabung ash has advantages over other raw materials because, in addition to its abundant availability, it was initially considered merely air pollution but

can now be utilized to produce more beneficial silica.

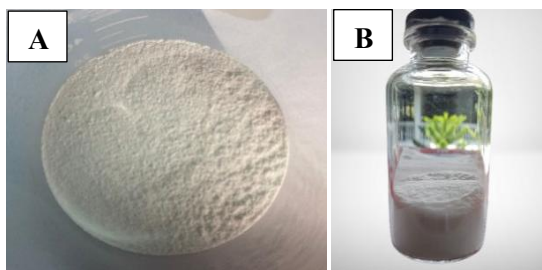


Figure 1. Silica extraction results from Sinabung volcanic ash

3.2 Silica Characterization Results

3.2.1. Silica Particle Morphology and Size

The surface morphology and particle size of silica were characterized using Scanning Electron Microscopy (SEM). The SEM image (Figure 2.a) shows silica particles with a regular and homogeneous morphology. The average silica particle size is approximately 58.80 ± 1.14 nm (Figure 2.b), with a uniform size distribution. These observations indicate that the sol-gel method is effective in producing silica particles with an ideal size for agricultural nanotechnology applications, which are important as precursors to produce nanofertilizers.

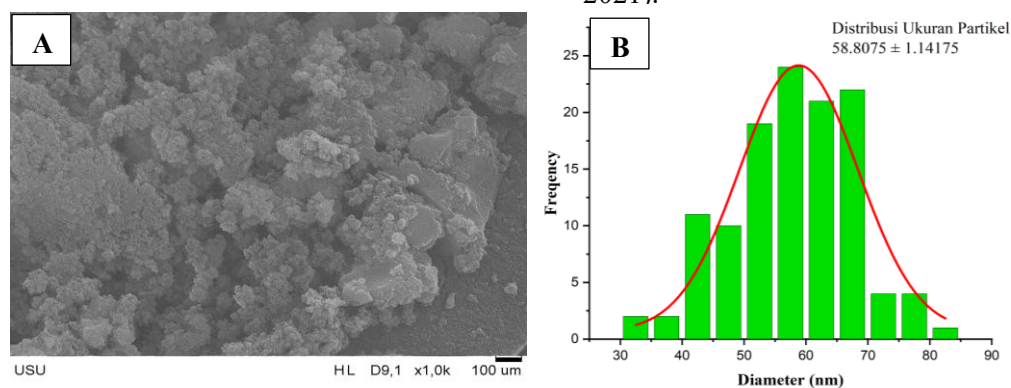


Figure 2. (a) Morphology of silica particles synthesized using SEM, (b) Size distribution of silica particles.

3.2.2. Crystal Structure and Amorphousness

Analysis using X-ray Diffraction (XRD) shows that the produced silica has an amorphous structure. In the XRD pattern (Figure 3.a), a broad diffraction peak (halo) is

observed at an angle of 2θ around 22° , which is characteristic of amorphous materials. The low regularity in atomic spacing confirms that this silica does not have a regular crystalline structure. This is consistent with the characteristics of silica produced from volcanic ash, which tends to have an amorphous structure rather than a crystalline one (Nelson, 2018).

3.2.3. Functionality Analysis with FTIR

The results of characterization using Fourier Transform Infrared Spectroscopy (FTIR) show a spectrum characteristic of silica. Figure 3.b shows the presence of a Si-O-Si stretching band at 1085 cm^{-1} , indicating strong bonds between silicon and oxygen atoms in the siloxane structure. The peak at 810 cm^{-1} indicates Si-OH bending, signifying the presence of hydroxyl groups. The band at 460 cm^{-1} reinforces the presence of Si-O bonds in the silica structure. The FTIR spectrum also shows a broad band in the $3200\text{--}3600\text{ cm}^{-1}$ range, indicating surface hydration of silica, as well as a peak at 1640 cm^{-1} indicating the presence of adsorbed water on the silica surface (Usgodaarachchi et al., 2021).

This analysis confirms that silica produced from Sinabung ash has the appropriate functional groups for application as a nanofertilizer.

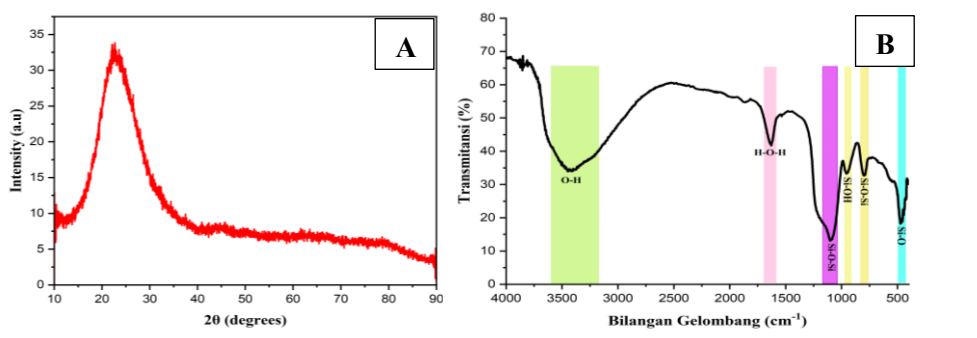


Figure 3. (a) XRD spectrum of silica with an amorphous structure, (b) FTIR spectrum of silica with specific peaks for siloxane and silanol groups.



Figure 4. (a) Silica functionalization process with triethanolamine, (b) Grinding of the synthesized fluorescent silica, and (c) Fluorescent silica variants 1, 2, and 3 that have been ground.

3.3. Synthesis of Fluorescent Silica Nanofertilizer

The synthesis of fluorescent silica nanofertilizer was carried out using the sol-gel method to ensure purity and uniform particle size distribution. Each variant of the nanofertilizer uses different silica concentrations (0.5 g, 1.0 g, 1.5 g) dissolved in ethanol as the solvent. Triethanolamine (TEA) is used as an amine functionalization agent to modify the silica surface.

Silica functionalization with TEA aims to enhance fluorescence and UV absorption properties. This process is illustrated in Figure 4.a, where silica is reacted with TEA at a controlled temperature. After the functionalization process is complete, the resulting product is then ground to ensure uniform size, as shown in Figure 4.b. The final product consists of fluorescent silica in three different variants, as shown in Figure 4.c.

The functionalization process using TEA is

important because the amino groups bound to the silica surface can enhance the stability and dispersibility of nanoparticles in solutions, as well as improve interactions between nanoparticles and plants. TEA also aids in the formation of a more ordered structure on the silica surface, contributing to improved efficiency in UV light absorption and fluorescence.

3.4. Results of Fluorescent Silica Characterization

FTIR spectroscopy was used to confirm the success of the amine functionalization process on silica. In Figure 5.a, the presence of N-H stretching bands in the 3300–3500 cm⁻¹ range and N-H bending bands in the 1500–1650 cm⁻¹ range indicates that the amine groups were successfully bound to the silica surface. Additionally, there is a C-H stretching band in the 2800–3000 cm⁻¹ range originating from the methylene (–CH₂) and methyl (–CH₃) groups of triethanolamine (TEA). The

intensity of these bands varies according to the ratio of silica and TEA concentrations used, with variant 1 showing the clearest intensity, indicating a higher degree of functionalization.

the morphology and particle size of silica. A uniform particle size distribution is crucial in nanofertilizer applications as it ensures consistent absorption by plants, thereby enhancing nutrient utilization efficiency.

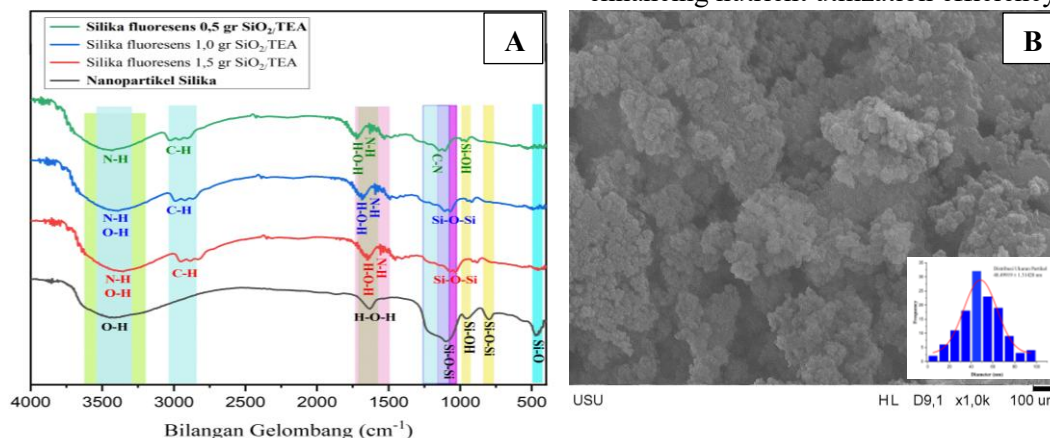


Figure 5. (a) FTIR spectrum of fluorescent silica nanofertilizer variants 1, 2, 3, and silica (b) Surface morphology of fluorescent silica nanofertilizer variant 1

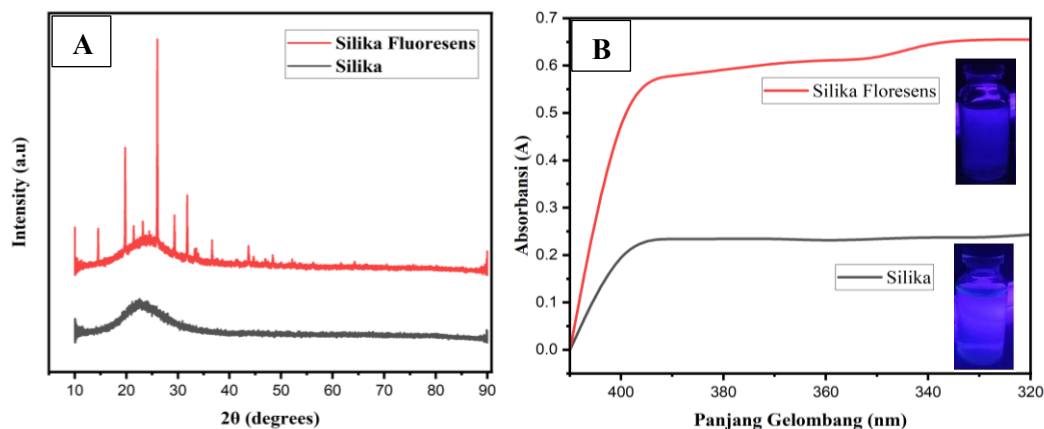


Figure 6. (a) XRD diffraction pattern of fluorescent silica nanofertilizer and (b) increased absorbance spectrum of fluorescent silica nanofertilizer.

Analysis using Scanning Electron Microscopy (SEM) in Figure 5.b shows that modification of silica with TEA results in a smoother surface morphology and more uniform particle size distribution compared to the initial silica. The particle size of the fluorescent silica nanofertilizer is also smaller, approximately 48.49 ± 1.31 nm, and more uniform. This indicates that the functionalization process not only improves the surface chemical properties but also affects

The X-ray Diffraction (XRD) pattern in Figure 6.a indicates an increase in silica crystallinity after modification with TEA. The initial silica had an amorphous structure with crystallinity of approximately 0.87%.

After modification, crystallinity increased to 11.88% with crystal size of approximately 52.01 ± 1.02 nm. TEA helps rearrange the atomic structure within the silica, thereby enhancing crystallinity and improving the resulting material structure.

This increase in crystallinity is important as it enhances the thermal and mechanical stability of the nanofertilizer, making it more resistant to varying environmental conditions when applied to plants.

Figure 6.b shows the increase in absorbance values using UV-Vis spectrophotometry in the UV-A wavelength range (320–400 nm). The absorbance value increased from 0.23 for the initial silica to 0.65 for the fluorescent silica nanofertilizer. This increase in absorbance indicates that the fluorescent silica nanofertilizer is more effective in absorbing UV light. Fluorescence observations under UV light also show that the fluorescent silica nanofertilizer emits a clearer blue light compared to the initial silica. The increased intensity of blue light emission indicates that the fluorescent silica nanofertilizer has better fluorescent properties, which could enhance plant photosynthetic activity by utilizing absorbed UV light and converting it into more effective blue light for plant use.

Overall, the characterization results indicate that the produced fluorescent silica nanofertilizer has superior physical and chemical properties compared to the original silica. The increased crystallinity, uniform particle size distribution, and improved fluorescence properties suggest that this nanofertilizer has significant potential to enhance photosynthetic efficiency and plant growth. The success of the amine functionalization process also indicates that the method used is effective in producing nanofertilizers with desired characteristics. The use of TEA as a functionalization agent has proven to enhance the UV absorption properties and fluorescence capability of silica, making it more beneficial as a nanofertilizer.

This study shows that silica from Mount Sinabung ash, which was initially considered

waste, can be processed into a high-value material with significant applications in agriculture. The resulting fluorescent silica nanofertilizer has the potential to improve nutrient use efficiency in plants, reduce dependence on conventional chemical fertilizers, and support more sustainable agriculture.

4. CONCLUSIONS

This study successfully synthesized silica from Mount Sinabung ash using the sol-gel method, yielding silica with a yield of 49.78%. Characterization of the silica revealed an amorphous morphology with a particle size distribution of 58.80 ± 1.14 nm. Furthermore, fluorescent silica nanofertilizer was successfully synthesized using triethanolamine modification, exhibiting a smooth surface morphology and uniform particle size distribution (48.49 ± 1.31 nm). FTIR analysis confirmed the functionalization of amine on the nanofertilizer, while XRD showed an increase in crystallinity from 0.87% to 11.88% after modification. UV-Vis spectrophotometry showed an increase in absorbance and blue fluorescence intensity in the nanofertilizer, indicating its potential to enhance plant photosynthetic activity more effectively.

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