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ADSORPTION KINETICS OF METHYL ORANGE ON SILICA DERIVED FROM GAMALAMA VOLCANIC SOIL

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Abstract

Silica gel was successfully synthesized from Gamalama volcanic soil using the sol-gel method and applied as an adsorbent to remove methyl orange (MO) dye. The synthesis process involved treating 20 grams of volcanic soil with 2 M HCl under stirring for 3 hours, followed by a 12-hour soaking period. The solid residue was subsequently filtered, dried, and reacted with 7 M NaOH for 2 hours. The resulting filtrate was neutralized through repeated washing with distilled water, and silica gel formation was achieved by titration with 2 M HCl until a final pH of approximately 3–4 was reached. The synthesized silica was evaluated for its adsorption performance against methyl orange, with optimal conditions identified at pH 4, a contact time of 5 hours, and an adsorbent dosage of 30 mg. UV-Vis analysis showed that the synthesized silica exhibited a higher adsorption capacity (6.7%) compared to raw Gamalama volcanic soil. Kinetic studies indicated that the adsorption process followed a pseudo-second-order model, suggesting chemisorption as the dominant mechanism. The rate constant (k_2) for the synthesized silica was 0.09 $M^{-1}{\cdot}h^{-1}$, compared to 0.077 $M^{-1}{\cdot}h^{-1}$ for the raw volcanic soil. This indicates that the synthesized silica adsorbed MO more rapidly, likely due to its higher purity and greater availability of active sites. The slightly lower k_2 observed in raw volcanic soil might be attributed to the presence of clay minerals such as halloysite and allophane, which possess negative surface charges at neutral to alkaline pH levels, leading to electrostatic repulsion with the negatively charged MO anions. Silica gel synthesized from Gamalama volcanic soil demonstrates promising potential as an eco-friendly adsorbent for dye removal from aqueous solutions.

Keywords: *Adsorption kinetics; Gamalama Volcanic Soil; Methyl Orange; Pseudo-Second-Order Model; Silica Gel*

Introduction

Access to clean, pollutant-free water remains a pressing global health challenge. A significant contributor to declining water quality is contamination by synthetic dyes, which are widely used in industries such as textiles, pharmaceuticals, food processing, and cosmetics (Konicki et al., 2015; Sivakumar et al., 2014). Among these dyes, anionic compounds like methyl orange are particularly hazardous due to their toxic, mutagenic, and carcinogenic properties. Exposure to such dyes can result in respiratory and gastrointestinal disorders, blindness, and even fatal outcomes (Han & Yun, 2007). Due to their high chemical stability and persistence in the environment, it is crucial to develop efficient methods for removing synthetic dyes such as methyl orange (MO) and methylene blue (MB) before they are discharged into natural water bodies.

Various treatment technologies have been explored for dye-contaminated wastewater, including photocatalysis (Ameta et al., 2018), membrane filtration (Makaremi et al., 2015), solvent extraction and bioremoval (Chong & Tam, 2020; Oehmen et al., 2014), ion exchange (Dąbrowski et al., 2004), electrochemical methods (Moura et al., 2016), and adsorption (Agarwala & Mulky, 2023; Foo & Hameed, 2012; Zhao & Liu, 2008). Among these, adsorption stands out due to its simplicity, cost-effectiveness, and environmental compatibility (Luo et al., 2010). The success of adsorption largely depends on the interaction between the adsorbent and the adsorbate, which may involve electrostatic attraction, van der Waals forces, and chemical bonding (Li et al., 2020). Therefore, selecting and developing efficient adsorbent materials are critical to enhancing dye removal performance.

Activated carbon is among the most widely used adsorbents for treating dye pollution (Choo et al., 2013; Foo & Hameed, 2012; Frydel et al., 2023; Huang et al., 2014). However, its widespread use is hindered by high production and regeneration costs (Luo et al., 2010; Lyu et al., 2020). As a result, attention has shifted toward alternative low-cost materials, including silica-based adsorbents (Haider et al., 2022; Haque et al., 2013; Lyu et al., 2020; Sulistiyo et al., 2017). Silica offers a promising solution due to its high surface area, porous structure, and abundance of functional groups such as silanol (Si-OH) and siloxane (Si-O-Si), which contribute to its excellent adsorption capacity (Yuan et al., 2019).

Silica can be synthesized from various natural precursors, including agricultural waste such as rice husks and corn cobs (Dhaneswara et al., 2020), as well as from naturally occurring volcanic soil (Utari et al., 2020). The North Maluku region, which is home to five active volcanoes, offers an abundant and untapped source of volcanic soil rich in silica. Harnessing this local resource could lead to the development of sustainable and affordable adsorbents for water purification. Gamalama volcanic soil, derived from the volcanic activity of Mount Gamalama in North Maluku, Indonesia, is a distinctive geological material rich in silica minerals and volcanic ash deposits. Its high silica content presents a promising yet underutilized resource for the extraction and application of silica-based materials across various industrial and environmental sectors (Calabi-Floody et al., 2011; Cipta et al., 2022). Silica, whether in amorphous form or as crystalline silicate minerals, serves as a key precursor in the production of silica gel, zeolites, silicon-based compounds, and other advanced materials with diverse functionalities. The abundance and accessibility of Gamalama volcanic soil make it a sustainable and cost-effective source of silica, highlighting its potential as a valuable material for environmental remediation, particularly in areas with prevalent volcanic activity and extensive volcanic soil deposits.

Silica can be extracted from Gamalama volcanic soil through several techniques, such as acid leaching, alkaline extraction, thermal treatment, and sol-gel synthesis.

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This research aimed to synthesize silica from Gamalama volcanic soil using the sol-gel method and evaluate its effectiveness as an adsorbent for removing anionic MO synthetic dyes from aqueous solutions. Various adsorption parameters were investigated to determine the optimal conditions for maximum dye removal. The outcomes of this study are expected to contribute significantly to the development of cost-effective and environmentally friendly water treatment technologies, particularly benefiting water quality and public health in the North Maluku region.

Methodology

This study was conducted in two phases. The first phase involved the synthesis of silica from Gamalama volcanic soil, followed by testing its adsorption efficiency toward methyl orange dye.

Silica synthesis was performed by weighing 10 g of Gamalama volcanic soil and placing it into a 250 mL beaker. Then, 50 mL of 7 M NaOH was added, and the mixture was stirred for 2 hours. Afterward, 100 mL of distilled water was added, and the mixture was filtered using Whatman filter paper. The resulting filtrate was titrated with 2 M HCl until silica precipitated at a pH of 3–4. The formed silica was washed five times with 300 mL of distilled water and then oven-dried at 100°C for 5 hours. The obtained silica was subsequently characterized using FTIR (Fourier Transform Infrared Spectroscopy), XRD (X-ray Diffraction), and SEM (Scanning Electron Microscopy) analyses. The second phase was conducted to determine the adsorption capacity of the synthesized silica toward methyl orange. The adsorption process was carried out by stirring 20 mg of silica in 50 mL of methyl orange solution for 30 minutes. The effect of pH on adsorption was studied using pH values of 4, 5, 7, 8, 9, and 10, with contact times of 0.5, 1, 2, 3, and 5 hours. Additionally, the effect of adsorbent dosage was examined by varying the silica mass to 10, 15, 20, 30, and 50 mg.

Results and Discussion

In this study, silica was synthesized using the sol-gel method. The resulting material was then characterized using FTIR and XRD analyses to confirm successful extraction and to identify its structural properties. The particle size distribution and morphology were examined using SEM.

FTIR characterization

FTIR is a powerful analytical technique used to determine materials' chemical composition and structural characteristics by analyzing their interactions with infrared radiation. When applied to silica, FTIR provides valuable information about its molecular structure, functional groups, and bonding configurations. Silica, primarily composed of silicon dioxide (SiO_2), exists in various forms, including amorphous and crystalline polymorphs. The FTIR spectra of silica typically exhibit distinct absorption bands corresponding to the vibrational modes of Si-O bonds and related functional groups. One of the most prominent features in the FTIR spectrum of silica is a broad absorption band between 1000 and 1200 cm^{-1}, which corresponds to the asymmetric stretching vibrations of Si-O-Si linkages within the silica network (Djomgoue & Njopwouo, 2013). The shape and intensity of this band can vary depending on the degree of polymerization and the presence of impurities or dopants in the silica matrix. Another key feature is the Si-O-Si bending vibration band, typically observed between 450 and 700 cm^{-1} (Djomgoue & Njopwouo, 2013; Yuan et al., 2008), reflecting the symmetric and asymmetric bending motions of Si-O-Si units. This band provides insights into the local structural symmetry and coordination environment around silicon atoms in the silica framework. Additionally, FTIR spectra often reveal absorption bands associated with surface functional groups, such as hydroxyl groups (Si-OH and Al-OH) and adsorbed water. These appear as stretching vibrations in the 3200–3600 cm^{-1} range and bending vibrations in the 1600–1800 cm^{-1} region,

indicating the presence of surface hydroxyls and moisture content (Bonelli et al., 2013).

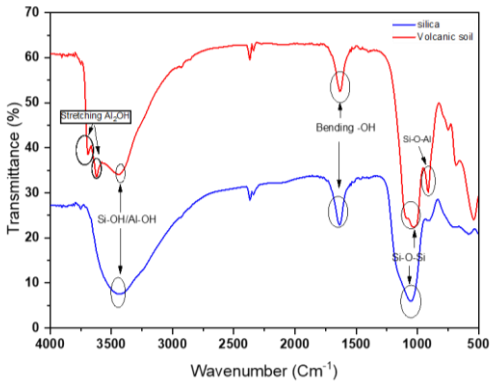

Figure 1. FTIR spectra of Gamalama volcanic soil and silica synthesized from Gamalama volcanic soil

Figure 1 displays the FTIR spectra of raw Gamalama volcanic soil and its synthesized silica. Absorption bands in the volcanic soil spectrum were observed at 3692, 3670, 3448, 1638, 1033, 935, and 871 cm^{-1}. The peaks at 3692, 3670, and 3448 cm^{-1} were attributed to hydroxyl functional groups, likely originating from Si-OH and Al-OH. These peaks disappeared in the spectrum of the synthesized silica, indicating successful silica extraction. The absorption band at 1033 cm^{-1}, associated with Si-O-Si stretching vibrations, remained in the silica spectrum, confirming the presence of silica. Conversely, the Al-O-related bands at 935 and 871 cm^{-1} observed in the volcanic soil spectrum were absent in the silica sample, possibly due to dealumination during acid treatment. Complementary XRD analysis was instructed to validate the FTIR findings and confirm the present mineral phases.

XRD characterization

XRD analysis was conducted to determine the crystallinity and structural properties of the synthesized silica. Amorphous silica typically exhibits a broad halo or diffuse scattering in its XRD pattern rather than the sharp, well-defined peaks characteristic of crystalline materials. This broad feature results from the absence of long-range atomic order in amorphous structures, where atoms are arranged randomly without periodicity. In contrast to crystalline substances, whose XRD peaks correspond to specific crystallographic planes, the lack of distinct peaks in amorphous silica confirms its non-crystalline nature.

The XRD pattern of silica synthesized from Gamalama volcanic soil (see Figure 2) shows a broad peak at 2θ around 9°–12°, consistent with the reference pattern for amorphous silica (PDF No. 00-042-005). Additionally, low-intensity peaks observed at 2θ = 20.13° and 30.89° further support this assignment. The sharp peaks associated with halloysite, particularly at 2θ = 8.26° (10 Å) and 19.69° (4.5 Å) presented in the raw Gamalama volcanic soil, were no longer observed in the synthesized silica. This disappearance indicates the successful removal of crystalline phases such as halloysite during the synthesis process, confirming the formation of amorphous silica.

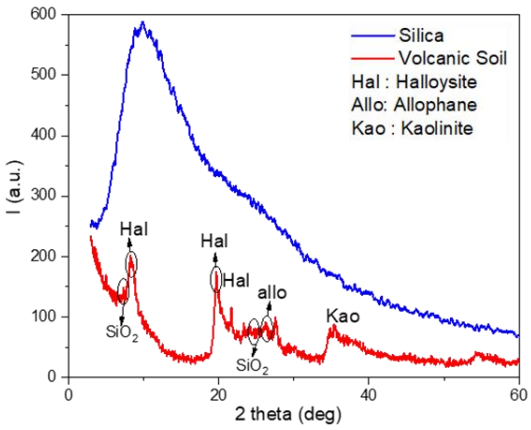

Figure 2. XRD patterns of Gamalama volcanic soil and silica synthesized from Gamalama volcanic soil

SEM characterization

The morphology and particle size distribution of silica extracted from Gamalama volcanic soil were analyzed using SEM. Image processing techniques, including contour detection and equivalent diameter measurements, were employed to determine particle sizes. The results reveal that the silica particles exhibited a fine granular morphology, with an average diameter of approximately 0.151 µm (151 nm). The size distribution lies within the

nanometer to submicron range, which is typical for silica derived from volcanic sources. The small particle size implies a high surface area, enhancing its potential utility in applications such as adsorption, catalysis, and composite material synthesis. These nanoscale characteristics are consistent with previous findings on volcanic soils rich in allophane and halloysite. The measured particle size of 0.151 µm aligns with data reported for other natural volcanic silica sources (Cipta et al., 2022; Takahashi et al., 2001). Furthermore, when compared to synthetic nanosilica obtained from sources such as rice husk ash (Haider et al., 2022), coal fly ash (Yuan et al., 2019), and fumed silica, the particle size of the Gamalama-derived silica fell within a similar range. This suggests that natural volcanic silica may serve as a sustainable and effective alternative to conventionally synthesized nanosilica. Overall, the silica particles obtained from Gamalama volcanic soil demonstrated comparable size and morphology to engineered nanosilica and commercial fumed silica, highlighting their potential for various advanced material applications.

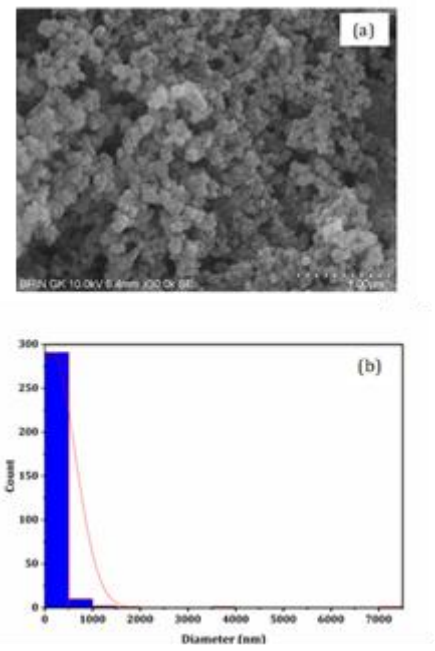

Figure 3. (a) SEM image of silica from Gamalama volcanic soil; (b) Particle size distribution of silica from Gamalama volcanic soil

Methyl orange adsorption

Methyl orange (MO) adsorption using silica derived from Gamalama volcanic soil presents a promising approach for environmental remediation and wastewater treatment. Silica, with its high surface area and porous structure, exhibits excellent adsorption properties, while volcanic soil serves as a readily available and potentially low-cost silica source. Methyl orange, a widely used azo dye in various industrial processes, poses significant environmental hazards due to its toxic and carcinogenic nature (Hanafi & Sapawe, 2020; Wu et al., 2021).

The results of pH optimization experiments, as presented in Figure 4, indicate that the optimal pH for MO adsorption is pH 4. The pH variation was achieved through the addition of HCl or NaOH. The addition of H^+ and OH^- ions significantly altered the surface charge of silica. Under alkaline conditions (high pH), NaOH induced negative charges on the silica surface and the MO dye, resulting in electrostatic repulsion and reduced adsorption. In contrast, at acidic pH levels, the silica surface became positively charged, enhancing electrostatic attraction with the negatively charged MO molecules (Hanafi & Sapawe, 2020; Wu et al., 2021)

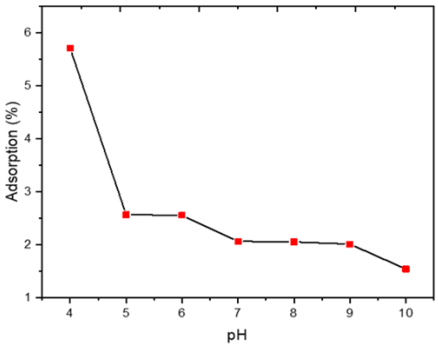

Figure 4. Effect of pH on the adsorption of methyl orange using silica derived from Gamalama volcanic soil

The adsorption efficiency of MO onto synthesized silica and raw volcanic soil was also investigated as a function of contact time and adsorbent dosage, as illustrated in Figure 5. The adsorption percentage increased with extended contact time,

particularly during the initial phase, due to the abundance of available active sites. Over time, the adsorption rate gradually decreased as these sites became saturated. Silica synthesized from Gamalama volcanic soil consistently exhibited higher adsorption efficiency than raw volcanic soil, suggesting improved surface area and chemical reactivity. As shown in Figure 5a, prolonged contact time enhanced the interaction between MO molecules and adsorbent surfaces, thereby increasing adsorption efficiency (Li et al., 2020; Mulana et al., 2015; Zam et al., 2021). Similarly, as depicted in Figure 5b, the adsorption percentage arose with increasing adsorbent dosage, reaching a maximum before plateauing or slightly decreasing. This trend reflects the greater number of active sites available at higher adsorbent concentrations. However, the adsorption percentage slightly declined beyond a certain dosage, such as 50 mg. This was likely due to particle aggregation, reducing the effective surface area and limiting access to active sites. The optimal adsorption was achieved with 30 mg of silica, resulting in approximately 42.32% removal of MO. This finding underscores the superior adsorption capacity of synthesized silica compared to raw volcanic soil. The reduced adsorption capacity of volcanic soil might be attributed to the presence of halloysite. The negatively charged outer surface of the halloysite might cause electrostatic repulsion with MO molecules, thereby hindering adsorption.

Kinetic study

The kinetic study of MO adsorption onto silica extracted from Gamalama volcanic soil and the raw volcanic soil revealed that the pseudo-second-order (PSO) kinetic model best described the adsorption process, as shown in Figure 5c. This model was applied to experimental data obtained by monitoring the adsorption capacity over varying contact times. The PSO model provided the best fit for the experimental data, with a high degree of linearity observed for silica (R^2 = 0.9981) and a moderate correlation for raw volcanic soil (R^2 = 0.955). These results indicate that silica exhibited a more consistent and

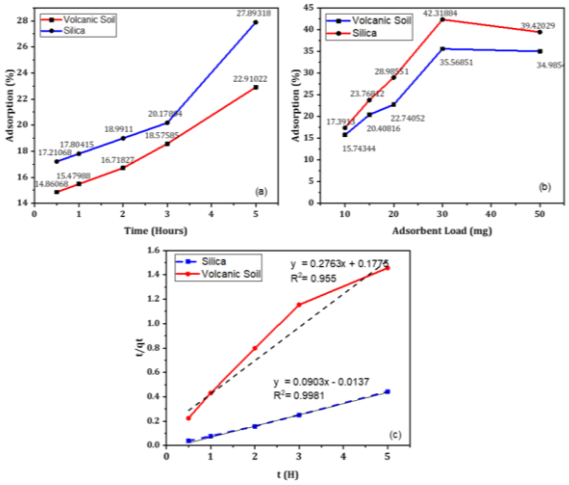

Figure 5. (a) Effect of contact time on MO adsorption; (b) Effect of adsorbent dosage on adsorption efficiency; (c) Pseudo-second-order (PSO) kinetic model graph for MO adsorption

efficient adsorption behavior than the raw material. The calculated rate constants (k_2) were 0.09 M^{-1} h^{-1} for the extracted silica and 0.077 M^{-1} h^{-1} for the raw Gamalama volcanic soil, indicating that the purified silica had a faster adsorption rate. This could be attributed to its higher purity, improved surface area, and more accessible active sites following the extraction process. In contrast, the slightly lower k_2 value for raw volcanic soil might be due to naturally occurring clay minerals such as halloysite and allophane. These minerals typically exhibit negatively charged surfaces at neutral to alkaline pH, which can lead to electrostatic repulsion with the anionic MO dye (Papoulis et al., 2010; Szczepanik et al., 2020; Van Ranst et al., 2020). Such repulsion may hinder effective contact between dye molecules and available adsorption sites, resulting in a slightly slower adsorption rate. Moreover, the complex mineralogical composition of the raw soil may limit the overall availability or accessibility of active sites compared to the more homogeneous structure of the purified silica. Nevertheless, it is important to note that halloysite and allophane, while often negatively charged, possess reactive hydroxylated surfaces. Under certain conditions, they can adsorb anionic dyes via alternative mechanisms such as ligand exchange or bridging through multivalent

cations (Anastopoulos et al., 2018; Szczepanik et al., 2020; Van Ranst et al., 2020). Hence, while their surface charge affects adsorption efficiency, their presence does not eliminate the material's adsorption capability but rather introduces complexity to the overall adsorption behavior of the raw soil.

The strong fit of the PSO model suggests that the adsorption of MO primarily occurred through chemisorption, a process involving stronger, more specific interactions such as electron sharing or exchange between the adsorbent and adsorbate. This contrasts physisorption, which involves weaker van der Waals forces. Chemisorption is typically slower but results in more stable and selective binding, which aligns with the observed gradual increase in adsorption over time. Several mechanisms might be responsible for the observed adsorption behavior. The silica surface is rich in silanol (Si-OH) groups, which can become partially protonated under slightly acidic conditions to form –Si-OH_2^+. These positively charged sites promote electrostatic attraction with the negatively charged sulfonate ($-SO_3^-$) groups on MO molecules (Wang et al., 2021). Additionally, hydrogen bonding between silanol groups and functional groups on MO, such as the azo linkage (–N=N–) and sulfonate oxygens, likely contributes to the chemisorption process (Wang et al., 2021).

These findings are consistent with previous research indicating that MO adsorption onto silica-based materials follows PSO kinetics and is dominated by chemisorption (A'yuni et al., 2024; Boushara et al., 2024; El Foulani et al., 2025; Rahmatpour & Hesarsorkh, 2024). This insight is valuable for the design and optimization of low-cost, sustainable adsorbents derived from natural volcanic materials for practical use in wastewater treatment applications.

Conclusion

This study successfully synthesized silica from Gamalama volcanic soil using the sol-gel method. The synthesized silica demonstrated a methyl orange (MO) adsorption efficiency of 42.32% under optimal conditions: pH 4, 5-hour contact time, and an adsorbent dose of 30 mg. UV-Vis analysis confirmed that the synthesized silica exhibited a 6.7% higher adsorption capacity than the raw volcanic soil. Kinetic studies indicated that the adsorption process followed a pseudo-second-order (PSO) model, suggesting that chemisorption was the dominant mechanism. The rate constant (k_2) for the synthesized silica was 0.09 $M^{-1}{\cdot}h^{-1}$, slightly higher than the 0.077 $M^{-1}{\cdot}h^{-1}$ observed for raw volcanic soil.

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