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Modified Sidoarjo Volcanic Mud as Adsorbent for The Decolourisation of Textile Wastewater

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The Sidoarjo mudflow disaster has persisted for 19 years in Gempol Sari Village, Sidoarjo, East Java, Indonesia. It has resulted in mudflows that have engulfed thousands of hectares of land and affected dozens of villages. Therefore, it is important to explore the positive aspects of this mud disaster, particularly the potential for utilizing the mud. This study investigates the activation of Sidoarjo volcanic mud using sulfuric acid and its application as an adsorbent for decolorizing wastewater from small-scale Batik textile production. The research examines the effects of various operational parameters such as contact time, dosage, pH, and dye concentration. The adsorption process is conducted in a batch system utilizing an orbital shaker. Characterization of the modified Sidoarjo volcanic mud as an adsorbent is performed using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), and Fourier Transform Infrared Spectroscopy (FTIR). Meanwhile, dye concentration is measured spectrophotometrically. Elemental analysis through EDX indicates that acid modification removes certain elements such as chlorine (Cl), potassium (K), calcium (Ca), and titanium (Ti), while increasing the surface area of Sidoarjo volcanic mud from 25.405 m^2/m^3 to 69.734 m^2/m^3. The study achieved a decolorization rate of 95% within 120 minutes of adsorption at an initial color concentration of 880 Platinum-Cobalt (Pt-Co), with an adsorbent dosage of 3 g/L and a pH of 3. The reusability tests showed modest removal efficiency, likely due to pore blockages. The reusability assessment highlighted challenges with the adsorbent's long-term performance, especially regarding irreversible dye deposition.

Keywords: volcanic mud, adsorption, textile wastewater, color removal, isotherm adsorption.

Introduction

Dyes are used in a variety of industries, including textiles, paper, food, and printing/screen printing. One notable industry that employs dyes in its production process is the Batik textile industry. Batik, a craft and art form from Indonesia, was recognized as a cultural treasure by UNESCO in 2009. The batik production process involves multiple steps, including designing, dyeing, washing, and waxing. However, these processes generate liquid waste that contains heavy metals, azo dyes, and other hazardous chemicals, as well as complex organic compounds (Oginawati et al., 2022). If not managed properly, this waste can have detrimental effects on the environment, such as polluting water sources, harming ecosystems, and threatening the health of nearby communities. As batik production continues to grow in various regions of Indonesia, the management of batik waste has become an increasingly urgent issue (Amelia et al., 2025).

Dyes serve various purposes beyond just adding color; they also possess numerous therapeutic properties. Currently, there are over 10,000 dyes available commercially, most of which are non-biodegradable due to their complex molecular structures and synthetic origins (Khan et al., 2023). These dye products are designed to provide lasting color, which means they contain resilient organic compounds that are challenging to treat using conventional biological processes. The characteristics of textile liquid waste often include high concentrations of colorants, organic materials, solids, and heavy metals, which can be harmful to aquatic ecosystems (Fajri et al., 2024).

Typical characteristics of textile wastewater include color (50–2500 Pt–Co), pH (6–10), biological oxygen demand (BOD) (80–6000 mg/L), chemical oxygen demand (COD) (150–12000 mg/L), oil and grease (10–30 mg/L), total suspended solids (TSS) (15–8000 mg/L), chlorine (1000–1600 mg/L), dan sodium (70 mg/L) (Mani et al., 2019; Wang et al., 2022). Wastewater-containing dyes can obscure sunlight, restrict oxygen from entering the air, and have damaging, mutagenic, or carcinogenic effects on aquatic organisms (Jalil et al., 2010).

One of the promising colour removal technologies is adsorption. Adsorption is the process of binding adsorbate (pollutants) on the surface of the adsorbent. Activated carbon is the most popular adsorbent due to its ability to remove various dyes. However, challenges related to expensive price, difficult adsorbent regeneration, and ineffective against some dispersal and vat dyes encourage the development of alternative low-cost adsorbents (Yagub et al., 2014). Many studies have demonstrated the effectiveness of alternative materials as adsorbents for dye removal such as agricultural waste (Yagub et al., 2014; Zahro and Adityosulindro, 2023), sewage sludge (Devi and Saroha, 2017), alum sludge (Adityosulindro et al., 2025; Adityosulindro et al., 2024), walnut shell powder (Uddin and Nasar, 2020), fly ash (Potgieter et al., 2021), algae biomass (Muhammad and Adityosulindro, 2022), clay (Khan et al., 2025) and red mud (An et al., 2023).

An innovative material that can be used as an adsorbent is Sidoarjo volcanic mud. Sidoarjo volcanic mud has been used as a substitute for cement, paving blocks and adsorbents (Dewi et al., 2025). Sidoarjo Volcanic Mud (SVM) originated from the mudflow disaster that began on May 29, 2006, in Porong, Sidoarjo, East Java, Indonesia, and continues to this day. The volume of mud emerging from the center of the eruption is estimated up to 100000 m3/d in 2006–2007 then tending to decline to around 25000 m3/d in November 2012. This volcanic mud has covered an area of over 6.5 km^2 and has displaced more than 30000 people since then (Sajali et al., 2016). Sidoarjo volcanic mud is potentially an effective adsorbent due to its large surface area of over 31 m^2/g and high silica and alumina content, which reaches 32.7% and 11%, respectively (Astuti et al., 2020; Ciptawati et al., 2022). Previous studies have been reported that SVM were effective to remove synthetic methyl orange and acid orange 52 dyes from aqueous solution (Jalil et al., 2010; Kamarudin et al., 2019).

This study examines the effectiveness of Sidoarjo volcanic mud as an adsorbent for removing dyes from wastewater generated by small-scale batik textile operations. Unlike most previous studies, which focused on synthetic dye wastewater, this research investigates real wastewater. The study evaluates the impact of various parameters, including adsorbent dosage, pH levels, and dye concentration, on the decolorization of wastewater.

Materials and Methods

Materials

Sidoarjo volcanic mud (SVM) was collected in Sidoarjo, East Java, Indonesia. SVM was taken at 500 m from the center of the mudflow burst. The SVM was dried under sunlight for several days, oven-dried in 60°C for 3 hours then crushed and sieved through 100 mesh. The modified SVM was prepared by mixing 100 g of powder SVM with 500 mL of 5N sulfuric acid (H_2SO_4) and stirring at 100°C for 2.5 hours. After allowing the mixture to reach room temperature, it was filtered through a 1.2 μm cellulose filter, washed multiple times with distilled water, and then dried overnight in an oven set to 100°C.

Batik textile wastewater

Real textile wastewater was taken from a home-scale batik industry in the city of Depok, West Java, Indonesia. The wastewater sample was filtered with a glass micro-fiber filter (1.2 μm) to remove suspended particles. Wastewater analysis showed a COD concentration of 2205 mg/L, color 880 Pt-co, and pH 9.5.

Experimental setup and protocols

The experiments were conducted in a batch system using an orbital shaker. Each experiment involved a volume of 100 mL of wastewater. Agitation was initiated at 300 rpm following the addition of the adsorbent. After the desired contact time was reached, an aliquot was taken and filtered to remove the adsorbent. The filtered sample was then diluted with distilled water before analysis. All assays were performed in duplicates, and the results are presented as the average values along with the standard deviation.

Analytical

Characterization of Sidoarjo volcanic mud samples as adsorbent was carried out using several methods. Scanning electron microscopy (SEM) coupled to energy dispersive X-Ray (EDX) was performed to evaluate the morphology and elemental composition of SVM and modified SVM samples. Prior to SEM-EDX examination, the powder (100 mesh) was degassed by heating the sample under vacuum at 100–150°C for 4–6 hours, then the analysis was carried out at 20 kV and magnification of 10,000x. Nitrogen adsorption measurements were performed at 77 K utilizing a surface area analyzer (Quantachrome NovaWin). Before the analysis, the sample was degassed under vacuum at 150°C for 12 hours to eliminate any adsorbed impurities. Adsorption isotherms were generated by subjecting the sample to nitrogen gas at varying relative pressures (P/P_0). The obtained data were examined using the Brunauer–Emmett–Teller (BET) method within the relative pressure interval of 0.05–0.30. The point of zero charge (pH_pzc) of the synthesized adsorbent was determined using the pH drift method. A series of 0.01 M sodium chloride (NaCl) solutions were prepared with initial pH values ranging from 2 to 12, using 0.1 M hydrochloric acid (HCl) or sodium hydroxide (NaOH) for adjustment. Approximately 50 mg of the adsorbent was added to each 50 mL solution and agitated for 24 hours at room temperature (Poormand et al., 2017). The concentration of colour and COD in water were measured spectrophotometrically based on Hach method 8025 and 8000, respectively.

Results and Discussion

Characterization of adsorbents

The SEM analysis of both the SVM and modified SVM samples is illustrated in *Fig. 1*. Both samples display irregular shapes and rough surfaces, characterized by a range of particle sizes. Elemental analysis using EDX indicated that the concentrations of Cl, K, Ca, Ti, and Fe in the SVM were lower compared to those in the modified SVM, as shown in *Table 1*. Previous research on SVM adsorbents has reported surface areas ranging from 7.478 to 31.217 m^2/g (Astuti et al., 2020; Sa'diyah et al., 2017; Talib et al., 2016). This study demonstrates that acid activation with 5N H_2SO_4 increased the surface area of SVM from 25.405 m^2/g to 69.786 m^2/g, while the pore diameter remained constant at 1.62 nm (*Table 2*).

Table 1. *Elemental composition of adsorbents*

Element	Wt%	
	SVM	Modified SVM
O	30.32 ± 1.845	44.75 ± 0.77
Na	2.00 ± 0.15	0.34 ± 0.13
Mg	2.33 ± 0.58	1.49 ± 0.09
Al	11.62 ± 1.28	14.91 ± 0.51
Si	30.19 ± 1.16	35.62 ± 0.20
Cl	3.17 ± 0.14	ND*
K	2.11 ± 0.57	ND*
Ca	2.89 ± 0.29	ND*
Ti	1.18 ± 0.23	ND*
Fe	14.17 ± 2.85	± 0.05

**Not detected*

Fig. 1. *SEM Images of SVM (A) and Modified SVM (B)*

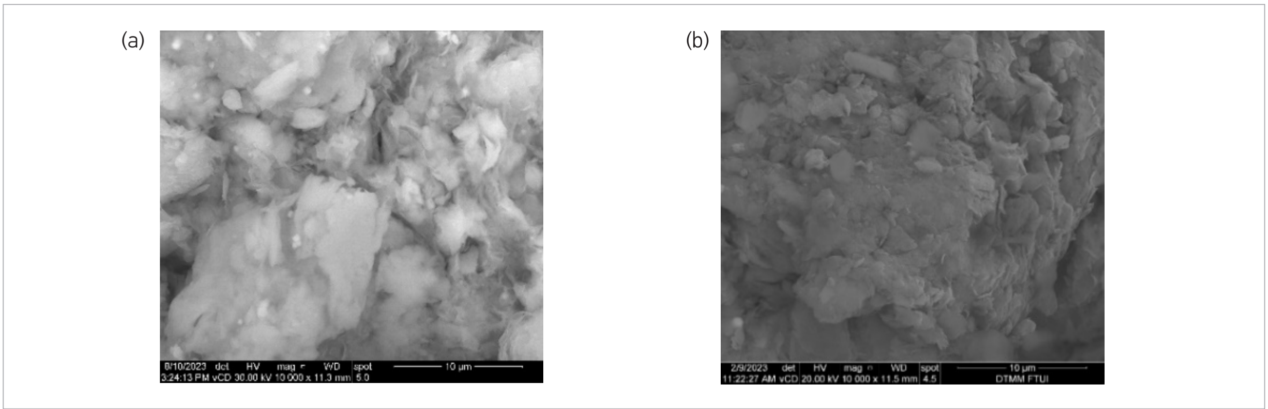


Fig. 2. *EDS of SVM (A) and EDS Modified SVM (B)*

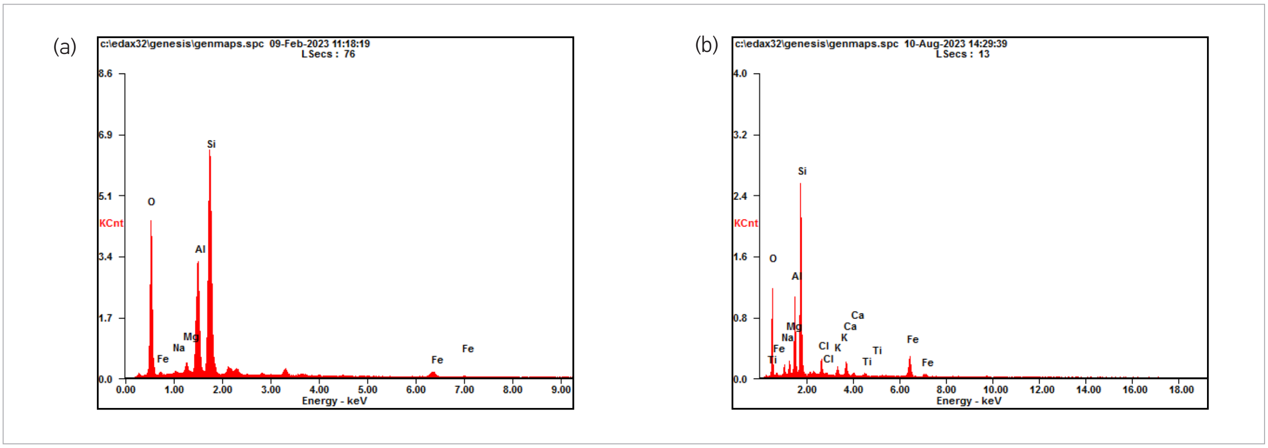


Fig. 3. *FTIR spectrum of modified SVM*

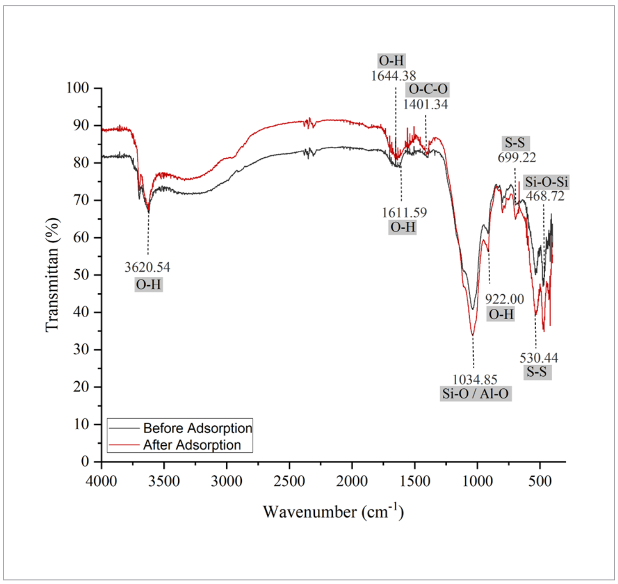


Table 2. *Summary of BET adsorption isotherms*

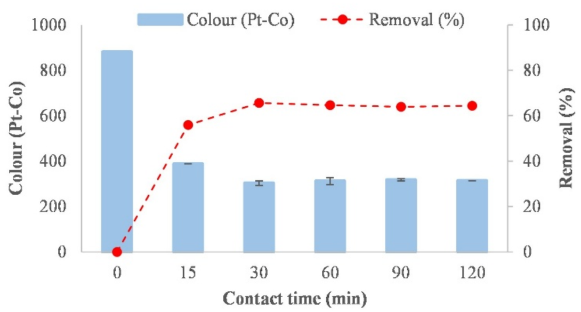
Parameters	SVM	Modified SVM
Slope	137.194	49.205
Intercept	−0.1114	0.6958
r	0.9997	0.9999
C constant	−1230.472	71.718
Surface Area (m^2/g)	25.405	69.789

The FTIR spectrum of the modified SVM displayed several characteristic absorption bands: the O-H bond at 3600 cm^{-1}, the OH group at 1600 cm^{-1}, O-C-O at 1400 cm^{-1}, the Si-Al bond at 1000 cm^{-1}, O-H at 900 cm^{-1}, S-S within the range of 600–500 cm^{-1}, and Si-O-Si at 450 cm^{-1} (*Fig. 3*). The intensity of the functional groups varied within the adsorbent sample. The fingerprint region, which is typically specific and unique, is defined as the wavelength range of 1500–600 cm^{-1} (Nandiyanto et al., 2019). Napthol dyes possess distinctive functional groups that are found between 1700 and 1600 cm^{-1} and 3500 and 3000 cm^{-1} (Suzuki, 2023).

Effect of contact time

Experiments were conducted to investigate the effect of contact time over a duration of 120 minutes. During the reaction, samples were taken every 15 minutes. Decolorization was significant in the first 15 minutes and then remained constant (*Fig. 4*). In the initial 15 minutes of the reaction, dye molecules (adsorbates) occupy the active sites on the modified SVM adsorbent. After 30 minutes, these active sites are likely fully occupied by adsorbates, leading to the establishment of equilibrium (Amran and Zaini, 2021). Equilibrium is reached when the saturated adsorbent can no longer adsorb, resulting in a constant removal percentage (Bentahar et al., 2018).

Fig. 4. *Effect of contact time*

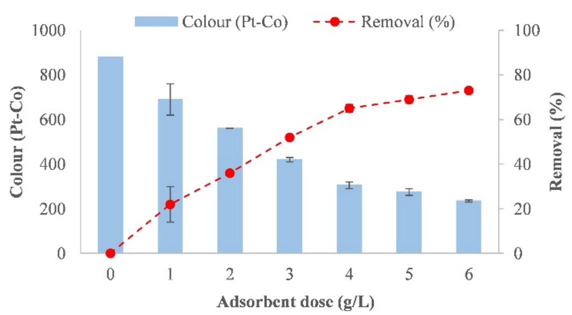


(C_0 = 880 Pt-Co; modified SVM = 3 g/L; t = 120 menit, pH = 9.5, V_{agt} = 300 rpm)

Effect of adsorbent dosage

From *Fig. 5*, it is evident that the colour removal efficiency increases from 22% to 73% as the dosage of modified SVM rises from 1 g/L to 6 g/L. This finding aligns with previous studies that demonstrated a significant effect of adsorbent dosage on colour removal (Devi and Saroha, 2017). The increased efficiency can be attributed to the greater number of binding sites available on the surface of the adsorbent (Devi and Saroha, 2017). However, it is noteworthy that only a slight discoloration (less than 10%) was observed when the adsorbent dosage was increased from 4 g/L to 6 g/L. This phenomenon has also been reported in previous research (Kayranli, 2011). A possible explanation for this behaviour is that the aggregation of adsorbent particles and/or the overlapping of adsorption sites at concentrations greater than 3 g/L leads to a decrease in the overall accessible surface area of the adsorbent (Merrikhpour and Jalali, 2012).

Fig. 5. *Effect of adsorbent dosage*

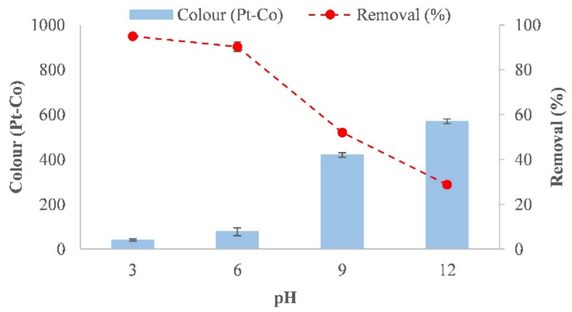


(C_0 = 880 Pt-Co; modified SVM = 1-6 g/L; t = 120 menit, pH = 9.5, V_{agt} = 300 rpm)

Effect of initial pH solution

The pH of the solution critically influences the adsorbent's surface charge and functional groups, as well as the ionization of the adsorbate. Mass titration analysis indicates that the pH point of charge (pH_{pzc}) of the modified SVM was 6.5. Several initial pH of wastewater was tested (3; 6; 9; and 12) to assess the role of pH in adsorption. As shown in *Fig. 6*, the decolourisation of textile wastewater decreases as the pH of the solution increases. Significant color removal (over 90%) was observed at pH levels below the pH_{pzc}. These findings could be explained by the protonation of SiO_2 and Al_2O_3 into Si^{4+} and Al^{3+}, respectively, under acidic pH (Kamarudin et al., 2019)Lapindo volcanic mud (LVM. The phenomenon creates a positive surface charge on the modified SVM surface, attracting negatively charged anionic azo dyes commonly found in textile wastewater. In addition, it is worth noting that the pH of the solutions remained unchanged after 2 hours of adsorption.

Fig. 6. *Effect of pH solution*

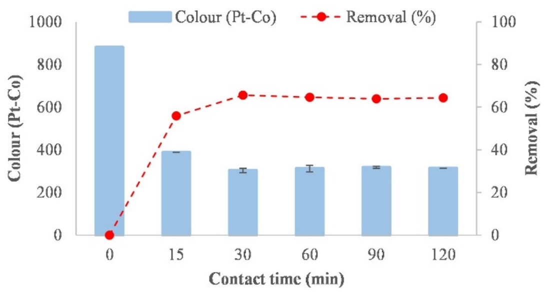


(C_0 = 880 Pt-Co; modified SVM = 3 g/L; t = 120 menit, pH = 3-12, V_{agt} = 300 rpm)

Effect of initial colour concentration

In a recent study, real textile wastewater was carefully diluted using distilled water to create a range of different dye colour concentrations. The results (*Fig. 7*) indicated a notable decline in the efficiency of dye removal as the initial colour concentration increased, dropping from 72% removal at a concentration of 300 Pt–Co to just 54% at a concentration of 880 Pt–Co. This trend can be attributed to the saturation of active sites available for dye adsorption, which becomes overwhelmed by the sheer number of dye molecules present in the wastewater at higher concentrations. When the concentration of dye is elevated, the competition for these active sites intensifies, resulting in less effective removal. This phenomenon of decreasing removal efficiency with increasing dye concentration has been observed in previous studies, confirming the challenges associated with treating densely coloured textile wastewater (Devi and Saroha, 2017).

Fig. 7. *Effect of initial colour concentration*



(C_0 = 300-880 Pt-Co; modified SVM = 3 g/L; t= 120 menit, pH = 9.5; V_{agt} = 300 rpm)

Adsorption isotherms

Adsorption isotherms refer to the relationship between the amount of absorbed by a polymer (adsorbent) at a constant temperature and analyte concentrations in solution. Liquid-phase adsorption on microporous materials is complex, making it difficult to construct a simple equation that encapsulates the equilibrium connection between the liquid and solid phases (Khan et al., 2024). COD was used for adsorption isotherms analysis. The data obtained from experiments were applied to Langmuir and Freundlich isotherm models. The linearised form of these isotherm models is depicted in *Fig. 8*. The Langmuir isotherm model (R^2 = 0.9273) fitted the adsorption data better than the Freundlich model (R^2 = 0.9032) (*Table 2*). Langmuir isotherm model assumes that removal of the sorbate occurs on a specific homogenous surface by monolayer adsorption.

Fig. 8. *Adsorption Isotherm: (A) Langmuir; (B) Freundlich*

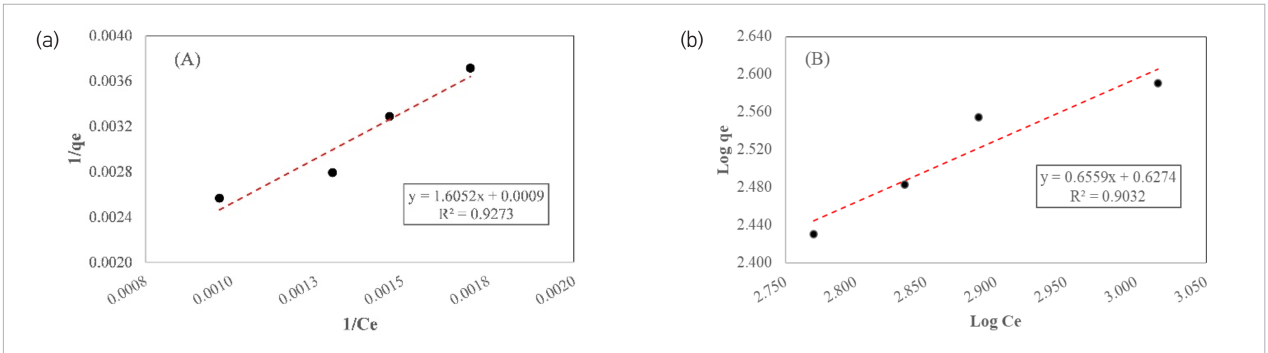


Table 3. *Adsorption parameters*

Model	Langmuir	Freundlich
Equation	y=1.6052x+0.0009	y=0.6559x+0.6274
R^2	0.9273	0.9032
Parameters	q_m= 1111 mg/g K_L= 0.0006 L/mg R_L=0.447	1/n= 0.656 K_f= 4.24 (mg/g)(L/mg)$^{1/n}$

Adsorption mechanisms and reusability test

The point of zero charge (pH_{pzc}) of the adsorbent is 6.5, while the pH of the Batik wastewater solution is 9.0. This indicates that the surface of the adsorbent is predominantly deprotonated and carries a negative charge. Under these conditions, electrostatic attraction may not be particularly favorable. However, surface complexation is likely to play a significant role, as functional groups, such as hydroxyls present on the mud surface, can form chemical bonds with the dye molecules. FTIR analysis confirms changes in these functional groups following adsorption, suggesting a chemical interaction (see *Fig. 3*). Furthermore, the porous structure of the mud enhances physical entrapment, allowing dye molecules to penetrate the micro- and mesopores, where they are retained by van der Waals force (Shikuku et al., 2022).

Evaluating the reuse of adsorbents is crucial for assessing their sustainability, cost-effectiveness, and long-term performance in both environmental and industrial applications. In this study, a decolorization rate of 73% was achieved for batik wastewater using 6 g/L of fresh modified SVM adsorbent. The spent adsorbent was filtered, thoroughly washed with distilled water, and then dried overnight in an oven. After this process, the dried adsorbent was reused to treat batik wastewater, resulting in a dye removal rate of only 23.57%. Lower removal efficiency Pore blockage in adsorbents caused by dye contaminants is primarily due to molecular size, strong chemical interactions, and irreversible deposition within the pore structure. Dye molecules, especially synthetic ones, tend to be large and complex, often containing aromatic rings and functional groups that interact strongly with adsorbent surfaces. These interactions such as hydrogen bonding, van der Waals forces, and electrostatic attractions can lead to deep penetration and tight binding within the pores. Over time, repeated exposure to dye-laden solutions results in accumulation of dye residues, which physically obstructs the pores and reduces available surface area for further adsorption (Rápó and Tonk, 2021).

Conclusions

Sidoarjo Volcanic Mud was successfully modified and utilized as an adsorbent to remove colour from textile wastewater collected from a home-scale Batik textile industry. After undergoing acid modification treatment, changes in the elemental composition and an increase in surface area of the adsorbent were observed. The decolorisation of the wastewater increased in direct proportion to the dosage of adsorbent added, while an increase in the initial colour concentration produced the opposite effect. Additionally, the decolorisation was more effective under acidic pH conditions. A dye removal up to 95% was achieved at an initial colour concentration of 880 Pt-Co, an adsorbent dosage of 3 g/L, and an initial pH of 3. The chemical oxygen demand (COD) removal during adsorption followed the Langmuir isotherm model. The reusability tests indicated modest removal efficiency, which may be attributed to potential pore blockages caused by residual dye. FTIR analysis supported the presence of chemical interactions, and the reusability assessment underscored certain challenges related to the long-term performance of the adsorbent, particularly concerning the irreversible deposition of dye.

Acknowledgements

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