

Sustainable Removal of Methylene Blue Using an Inverse-Vulcanized Polysulfide Derived from Waste Cooking Palm Oil

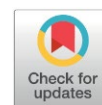
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Abstract

The environmental impact of waste palm cooking oil (WCO) has prompted extensive research into its potential applications, as it remains a significant pollutant, particularly in urban areas. This study investigates the synthesis of polysulfide using elemental sulfur and WCO as a sustainable method to repurpose these waste materials. The synthesized polysulfide showed effective methylene blue (MB) dye adsorption from wastewater, with adsorption performance assessed by varying the sulfur content (wt%) and dosage levels. Characterization techniques, including Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), were employed to analyze its structural and morphological properties. At the same time, UV-Vis spectrophotometry was used to measure the color removal efficiency. The highest removal rate of 78% was achieved at 70 wt% sulfur using 5 g of polysulfide in a 5 mg/L MB solution. The adsorption followed the Temkin adsorption isotherm ($R^2 = 0.96$). Post-adsorption FTIR analysis confirmed that the degradation followed a physisorption mechanism, as no changes in functional bonds were observed. By simultaneously addressing WCO and sulfur waste, this study underscores the potential of polysulfide as an effective, sustainable adsorbent for wastewater treatment.

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Keywords: Waste palm cooking oil; Polysulfide adsorbent; Inverse vulcanization; Methylene Blue removal; Dye adsorption

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1. Introduction

In developed countries, waste cooking oil has been recycled for decades. The annual generation of waste cooking oil varies significantly depending on cooking oil consumption, contributing to environmental concerns and waste management challenges. Recently, waste palm cooking oil (WCO) has gained attention for its potential applications, particularly in the synthesis of bio-based polymers, due to its low cost, abundance, and renewable nature [1]. Among these polymers,

polysulfides have emerged as promising candidates following the discovery that molten sulfur can react with comonomers to form stable polymeric structures [2]. Sulfur, a byproduct of the oil and gas industries, is both inexpensive and widely available. When heated, sulfur melts and undergoes ring-opening polymerization, forming linear sulfur chains. However, as temperature increases, the polymerized sulfur becomes unstable, leading to depolymerization over time. This inherent instability has necessitated the development of copolymerization strategies with organic comonomers, leading to the production of durable sulfur-based polymers [3].

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One such method, known as inverse vulcanization, was first reported in 2013 by Pyun and Chars [4]. This process utilizes sulfur as the primary polymeric backbone and stabilizes it by incorporating a suitable organic crosslinker, such as 1,3-diisopropenylbenzene (DIB). In this approach, sulfur is heated to 185 °C in an oil bath before DIB is gradually introduced, allowing the formation of a stable polymeric structure under continuous stirring. Notably, inverse vulcanization does not require additional initiators or solvents, making it a cost-effective and scalable method. The development of this technique has significantly expanded the potential of polysulfide synthesis, drastically reduced production costs, and enhanced the stability of sulfur-based materials. Among various monomers, palm oil (fresh and used) has been identified as a sustainable alternative for inverse vulcanization [5,6]. It is worth noting that the global palm oil production reached approximately 197 million metric tons in 2017–2018 alone, reinforcing its availability and economic feasibility for polymer synthesis [3].

Dyes are widely used in industries such as textiles, food, paper, plastics, and cosmetics. The wastewater generated from these industries poses significant environmental threats, as dyes are often persistent, resistant to degradation, and highly visible even at low concentrations. These pollutants not only harm aquatic ecosystems but also disrupt the food web, leading to severe ecological consequences. Many dye compounds exhibit resistance to oxidation, light exposure, and microbial degradation, further exacerbating their environmental persistence [7]. Notably, approximately 15% of the global dye production is lost during industrial processes and subsequently discharged into the water systems [8]. Among these pollutants, methylene blue (MB) is a particularly concerning contaminant due to its widespread industrial use and environmental impact. Effective removal of MB from wastewater is therefore a pressing environmental challenge.

Adsorption is one of the most widely used methods for dye removal. Despite the growing interest in inverse-vulcanized sulfur polymers, the current literature has focused predominantly on heavy-metal remediation and on polymers prepared from fresh plant oils or other model crosslinkers, while the use of waste cooking palm oil (WCO) as a low-cost crosslinker for dye adsorption, particularly for methylene blue (MB) removal, remains insufficiently explored. Previous studies have demonstrated the feasibility of sulfur-rich polymers for Fe³⁺, Cu²⁺, and Hg²⁺ removal, but they do not adequately clarify how a WCO-derived inverse-vulcanized polysulfide performs toward cationic dye pollutants or how the sulfur/WCO composition influences the resulting adsorption behavior. In

addition, limited attention has been given to combining waste valorization of two abundant wastes: elemental sulfur and WCO with porogen-assisted synthesis to produce a porous adsorbent for wastewater treatment [5,9]. Therefore, the novelty of this study lies in synthesizing a sustainable inverse-vulcanized polysulfide adsorbent directly from elemental sulfur powder and waste cooking palm oil, systematically evaluating the effect of sulfur loading and adsorbent dosage on MB removal, and further elucidating the adsorption mechanism through post-adsorption FTIR and isotherm analysis. This fills an important gap between earlier sulfur-polymer studies centered on heavy metals and the practical development of low-cost waste-derived adsorbents for dye-contaminated water. This study aims to synthesize polysulfide from WCO via inverse vulcanization and evaluate its potential as a cost-effective adsorbent for methylene blue (MB) removal from wastewater. The synthesized polysulfide underwent physicochemical assessments to determine its structural and morphological properties, while its adsorption efficiency was analyzed by varying sulfur content and dosage levels. The MB removal process was confirmed to follow a physisorption mechanism, ensuring stability and reusability. By addressing both WCO and sulfur waste, this study highlights the potential of polysulfide as a sustainable, efficient wastewater treatment material.

2. Materials and Method

2.1 Materials

Waste cooking palm oil (WCO) was sourced from the university cafe at Universiti Malaysia Pahang Al Sultan Abdullah (UMPSA), located in Gambang, Kuantan, Malaysia. The oil samples were filtered through a 0.224 µm syringe filter to remove unwanted particles. Elemental sulfur (sublimed reagent grade powder, Sigma Aldrich, USA, ± 99.5 %), sodium chloride (NaCl; Sigma Aldrich, USA, ACS reagent, ≥ 99.0%), and methylene blue (MB) (R&M Chemicals, Malaysia) were used as obtained. Deionized water (Millipore Elix 5, USA) was obtained from the Faculty of Chemical and Process Engineering Technology (FTKPP) lab at UMPSA.

2.2. Polysulfide Synthesis via Inverse Vulcanization

The synthesis process was adapted from the methods described in the previous works of this research group, with minor modifications [9,10]. Initially, polysulfides were synthesized using a 70:30 (w/w) ratio of sulfur powder to waste cooking palm oil (WCO). Elemental sulfur powder (7.0 g) was mixed with sodium chloride (NaCl) at 30%

(w/w) relative to sulfur and placed in a 100 mL beaker equipped with a magnetic stirring bar. The mixture was then heated to 195 °C at 450 rpm to induce the ring-opening polymerization of sulfur. The heating process continued for approximately 10 minutes. The appearance of an orange coloration in the solution indicated complete cleavage of the S–S bonds [4]. Subsequently, 3.0 g of WCO was slowly added dropwise via a syringe. The reaction was maintained at the same temperature with continuous stirring for 60 minutes to facilitate crosslinking between the monomers, allowing the polymer to reach the gel point [11]. The resulting mixture was then cooled to room temperature for over 90 minutes before being ground into a fine powder using a mortar and pestle. To remove residual NaCl, which acted as a porogen, the powder was rinsed with deionized water at 600 rpm for 30 minutes. Finally, the polymer was dried overnight in a vacuum oven at 50 °C. The synthesis was repeated for different sulfur-to-WCO ratios of 60:40 and 50:50. The resulting polysulfides were designated according to Table 1 for further analysis in this study.

2.3. Characterization of Synthesized Polysulfides

The synthesized polysulfide adsorbent was further characterized for its morphological properties using Scanning Electron Microscopy (SEM) and elemental composition analysis through Energy-Dispersive X-ray Spectroscopy (EDS) (SEM-EDS; Hitachi TM3030 Plus, Japan). Functional group identification, both before and after adsorption treatment, was conducted using Fourier-Transform Infrared Spectroscopy (FTIR) with Attenuated Total Reflectance (ATR) (Thermo Fisher Nicolet iS5, USA) to analyze the functional bonds present in the polysulfide. The transmittance percentage was recorded over a wavelength range of 400–4000 cm⁻¹, enabling the identification of functional groups responsible for Methylene Blue (MB) adsorption on the adsorbent surface. Additionally, X-ray Diffraction (XRD; Rigaku MiniFlex II, Japan) was employed to investigate catalyst behavior and structural modifications under varying conditions of pressure and temperature. XRD intensity measurements were recorded as a function of 2θ, with a step width of 0.02 °.s⁻¹ over a scanning range of 10° to 80°.

Table 1. Prepared polysulfide samples in this study to remove MB

Sample name	Sulfur (wt%)	WCO (wt%)
PS 1	70	30
PS 2	60	40
PS 3	50	50

2.4. Adsorption of Polysulfide in Methylene Blue (MB)

The experiment was conducted in a 250 mL beaker containing 100 mL of a 5 mg/L methylene blue (MB) solution, using the synthesized polysulfide as an adsorbent. Adsorption efficiency was evaluated through the batch adsorption method. Initially, adsorption was performed for 24 hours using PS 1 to determine the equilibrium time, which was reached at the sixth hour. Consequently, all subsequent adsorption experiments were conducted for 6 hours. The initial MB concentration was maintained at 5 mg/L, with a solution volume of 100 mL, a pH of approximately 7, and at room temperature. The effect of adsorbent dosage was examined using 1, 2, and 5 g per 100 mL of solution. The beakers were stirred at 200 rpm until equilibrium was achieved. MB concentrations were measured at 668 nm using a UV-Vis spectrophotometer (Shimadzu UV-2600 UV-Vis, Tokyo, Japan), while the solution pH was kept constant at 7. Sampling was conducted every 10 minutes during the first hour, then every hour for the remaining 5 hours. The percentage removal of MB was calculated using Equation (1). This procedure was repeated for PS 2 and PS 3. After adsorption, the adsorbent was separated from the solution using filter paper, dried, and analyzed via Fourier-transform infrared spectroscopy (FTIR). The effluent removal percentage was determined using Equation (1).

$$\text{Removal efficiency} = \frac{C_0 - C_f}{C_0} \times 100\% \quad (1)$$

where, C_0 = The initial MB concentration in the sample (mg/L), C_f = The MB concentration of the sample solution after treatment at any time (mg/L)

2.5. Isothermal Study

The isothermal study of methylene blue (MB) removal was performed using the Langmuir (Equation (2)), the Freundlich (Equation (3)), and the Temkin (Equation (4)) isothermal equations [12,13].

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (2)$$

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (3)$$

$$q_e = \frac{RT}{b} \ln A + \frac{RT}{b} \ln C_e \quad (4)$$

C_e and q_e stand for the solution concentration (mg/L) and amount of adsorption (mg/g) at equilibrium conditions, respectively. The maximum adsorption capacity is represented by q_m (% efficiency/min). K_L and K_F are the constants for the Langmuir and the Freundlich isotherms,

respectively. The intensity parameter for the Freundlich isotherm is denoted by $1/n$. The Temkin constant (J/mol) and the Temkin isotherm equilibrium binding constant (L/g) are denoted by b and A in Equation (4). All statistical analyses were performed using Origin 2025 (OriginLab Inc., USA).

3. Results and Discussion

3.1. Characterization of Synthesized Polysulfide

The chemical interactions and physical bonding in the synthesized polysulfides are reflected in the changes observed in the characteristic spectral peaks of the FTIR spectra. Figure 1 presents the FTIR spectra of three different polysulfide samples (PS 1, PS 2, and PS 3), highlighting their fundamental characteristic peaks. Notably, the peak at 3005 cm^{-1} corresponds to the C–H stretching vibration of the vinylic moiety in waste palm cooking oil (WCO) and the stretching vibration of the vinylic C=C bond. In the WCO spectrum, this peak is prominent, signifying the presence of unsaturated structures. However, as sulfur content increases in the synthesized polysulfides (PS 1, PS 2, and PS 3), this peak diminishes and eventually disappears, indicating the conversion of C=C bonds into C–S bonds. This transformation confirms the successful formation of polysulfides. The FTIR

results align with previous studies [14,15], reinforcing the validity of these findings.

All polysulfide samples exhibited nearly identical FTIR patterns. The X-ray diffraction (XRD) analysis further supports these observations by showing a distinct reduction in the characteristic sulfur peak across all synthesized copolymers. The degree of crystallinity in the polysulfides is influenced by the sulfur concentration, as evidenced by the diminished peak intensity. The presence of unreacted sulfur directly affects crystallinity, with polysulfides predominantly being amorphous due to incomplete reaction between sulfur and WCO [10]. Given that WCO contains both saturated and unsaturated fatty acids, its reaction with sulfur is not entirely exhaustive. As a result, the XRD patterns of PS 1, PS 2, and PS 3 (Figure 2) display residual sulfur signals, signifying the presence of unreacted sulfur in the synthesized polysulfides.

Scanning electron microscopy (SEM) images further reveal the coarse and porous surface morphology of the polysulfides, indicating their suitability for adsorption applications. To enhance porosity and improve adsorption capacity, NaCl was ground with sulfur during synthesis. The SEM images of fresh polysulfides exhibit a uniform surface, devoid of signs of depolymerization [16]. As shown in Figure 3,

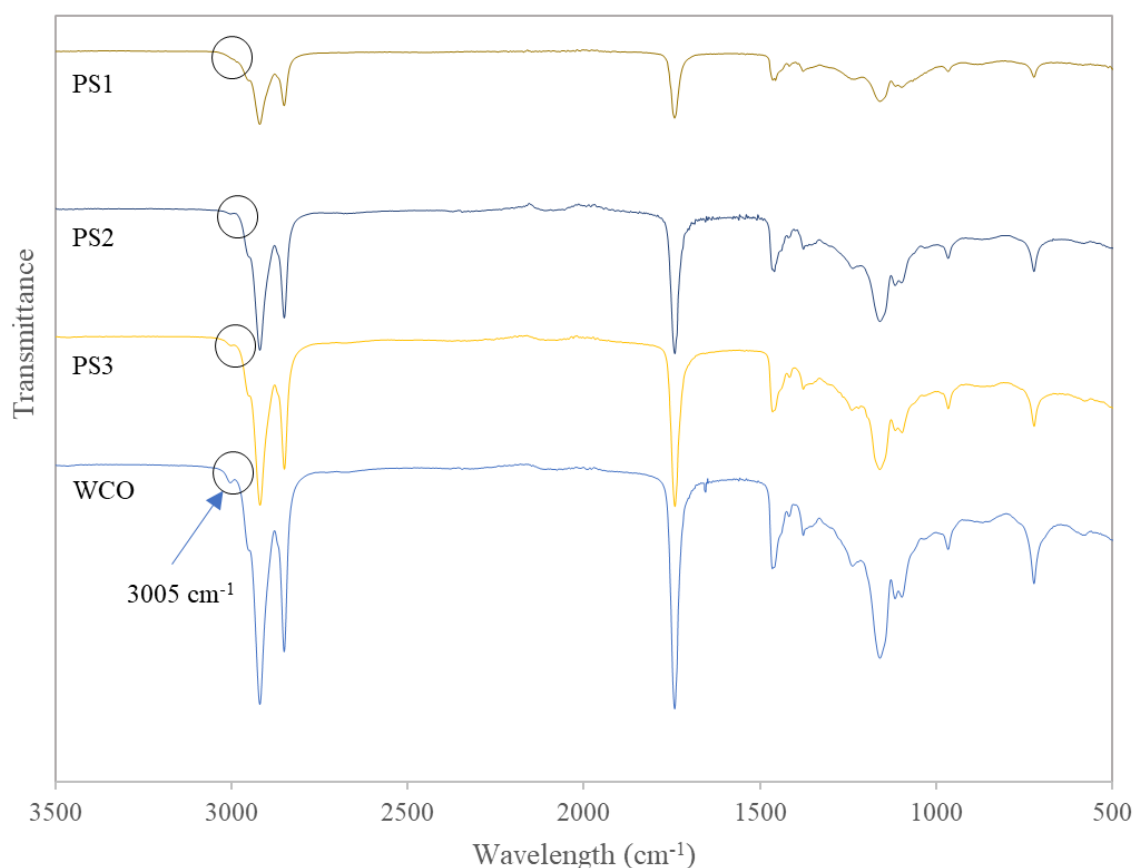


Figure 1. FTIR results of WCO, PS 1, PS 2, and PS 3.

SEM-EDS results for PS 1, PS 2, and PS 3 indicate an increase in surface roughness and density upon the addition of a crosslinker monomer. With higher sulfur content, the polysulfide develops a composite-like texture, characterized by a progressively rougher surface. This surface roughness is indicative of unreacted sulfur in the copolymer, as further supported by the EDS analysis, which aligns with previous findings [5,17].

Elemental analysis via EDS also confirms variations in sulfur content across the different polysulfide samples. The atomic proportion of carbon decreases as sulfur content increases, with PS 1, PS 2, and PS 3 containing sulfur amounts of 3.358%, 5.1%, and 24.944%, respectively. A higher WCO-to-sulfur ratio facilitates increased reactivity between sulfur and the unsaturated C=C bonds. However, at higher sulfur feed levels, excess sulfur leads to incomplete crosslinking, leaving residual unreacted sulfur [9,10]. The black circular regions observed in the SEM micrographs correspond to porous structures created by NaCl incorporation. The porosity of the polysulfides increases with surface area, further enhancing their potential for adsorption applications.

3.2. Investigation of Adsorption Parameters

3.2.1. Effect of sulfur content

The adsorption capability of the synthesized polysulfides (PS 1, PS 2, and PS 3) was evaluated

using 5 g of each sample in 100 mL of a 5 mg/L methylene blue (MB) solution. Among the tested polysulfides, PS 1 exhibited the highest removal efficiency (79%), compared with PS 2 and PS 3, as illustrated in Figure 4. The superior performance of PS 1 can be attributed to the greater likelihood of waste cooking oil (WCO) crosslinking with the polymeric sulfur chains, leading to a more effective adsorbent structure. This enhanced crosslinking may increase surface area or improve the availability of functional groups, thereby facilitating better adsorption of MB molecules.

3.2.2. Effect of dosages and contact time

As illustrated in Figure 5, all three dosages exhibit a positive correlation between increasing

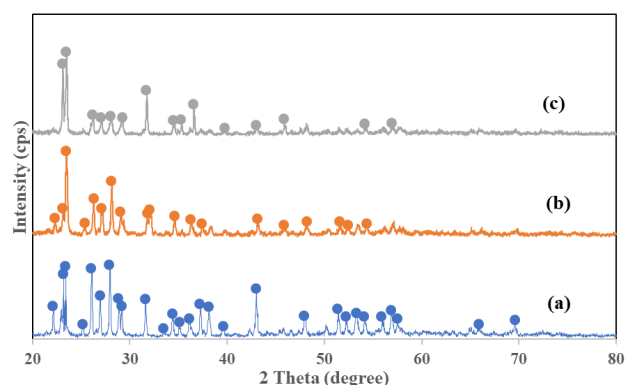


Figure 2. XRD patterns of (a) PS 1, (b) PS 2, and (c) PS 3.

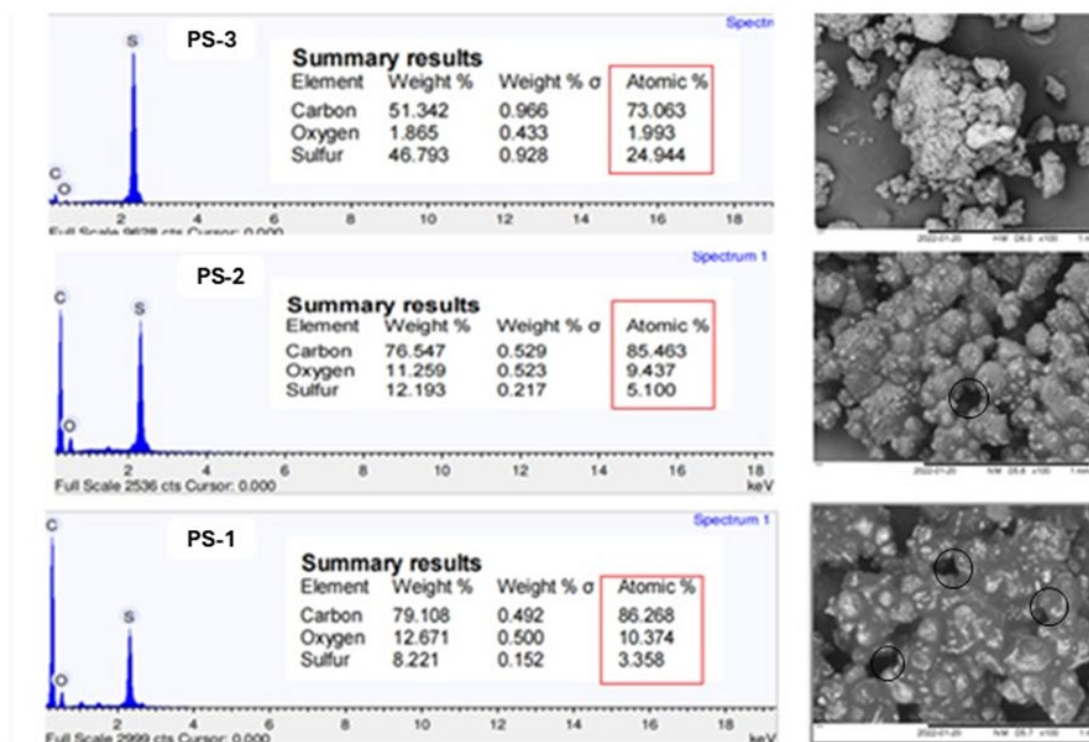


Figure 3. SEM micrographs and EDS analysis of the polysulfide adsorbents: starting from top, PS 3, PS 2, and PS 1.

polysulfide (PS 1) dosage and removal efficiency. At a dosage of 1 g/100 mL, the removal efficiency was 15%, whereas increasing the dosage to 2 g/100 mL and 5 g/100 mL resulted in removal efficiencies of 35% and 78%, respectively. The data indicated that the highest removal efficiency of 78% was achieved at an optimum dosage of 5 g/100 mL. The significant difference in removal performance highlights the importance of adsorbent dosage in improving MB adsorption.

All three curves indicate that MB removal efficiency increases with contact time, but at different rates. For higher dosages (5 g/100 mL and 2 g/100 mL), removal continues to rise steadily, suggesting a prolonged adsorption process. The observed increase in adsorption efficiency with higher dosages can be attributed to the greater availability of active adsorption sites on the polysulfide surface. As the dosage increases, more surface area and binding sites

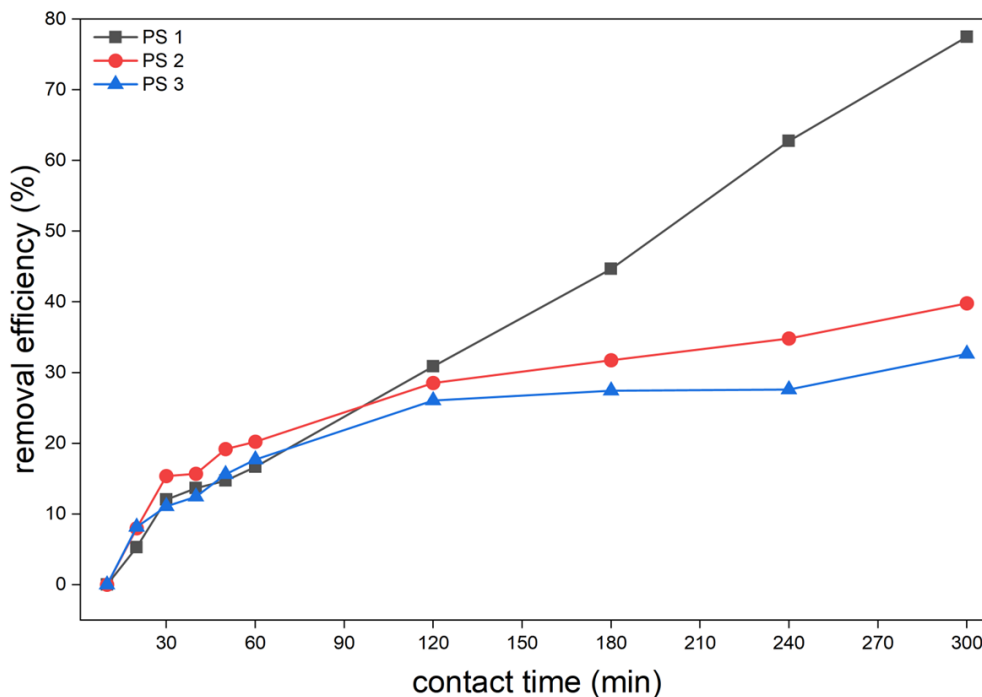


Figure 4. Color removal efficiency of the MB solution using polysulfides: PS 1, PS 2, and PS 3.

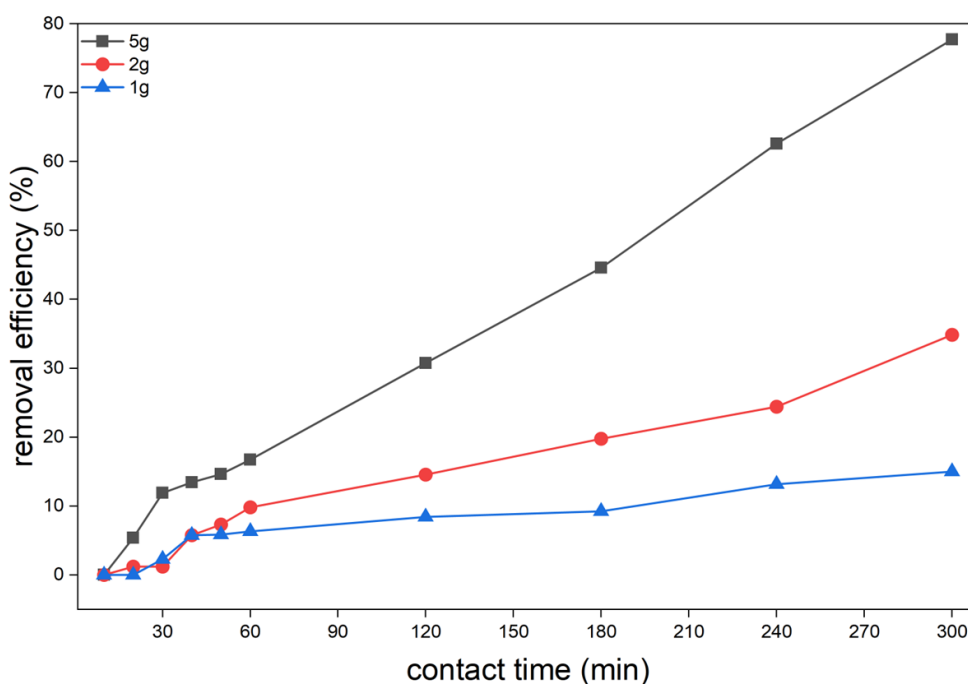


Figure 5. Effect of adsorbent dosage on methylene blue (MB) color degradation in a 100 mL solution (5 mg/L MB) using polysulfide adsorbents at different dosages: 1, 2, and 5 g.

become accessible for pollutant molecules, leading to enhanced adsorption. This trend is consistent with previous studies, which suggest that higher adsorbent concentrations improve pollutant removal by increasing the interaction between the adsorbent and target molecules in solution [15,18]. The lowest dosage (1 g/100 mL) reaches near-equilibrium much earlier, suggesting that adsorption sites become saturated quickly at lower adsorbent concentrations. However, beyond a certain dosage, adsorption may achieve a plateau due to saturation of the available active sites, where all potential adsorption sites are occupied, and further increases in dosage yield minimal improvements.

3.3. Post-analysis of MB Color Degradation

The FTIR spectra presented in Figure 6 for PS 1 after adsorption using 5 g, 2 g, and 1 g of polysulfide exhibit no significant deviations from the FTIR spectra of PS 1 before adsorption. This consistency in spectral patterns suggests that the adsorption process did not induce substantial functional group modifications in the polysulfide structure, confirming its effective adsorption capacity. A comparison between the pre- and post-adsorption spectra reveals a notable reduction in

peak intensity, particularly at 3005 cm^{-1} , which corresponds to the C=C bond stretching vibration. The disappearance of this peak in the post-adsorption spectra suggests the successful interaction between methylene blue (MB) molecules and the polysulfide adsorbent, likely through surface interactions rather than chemical bonding. The absence of newly formed peaks or significant changes in functional groups further supports the conclusion that the adsorption mechanism is predominantly physisorption, in which MB molecules adhere to the adsorbent surface via weak van der Waals forces rather than forming new chemical bonds. This interpretation aligns with findings from previous studies, reinforcing the efficacy of polysulfide as an adsorbent for MB removal from aqueous solutions through a physisorption-driven process [19,20].

3.4. Isothermal Analysis

The polysulfide adsorbent of the highest dosage in this study (5 g/100 mL) showed the highest removal efficiency, which is used to study the isothermal behaviours of the adsorbent where the MB concentration was 5 mg/L, and the solution volume was 100 mL. The calculated data points for the Langmuir, Freundlich, and Temkin isothermal equations have been plotted in Figure 7. The different parameters were calculated from the slopes and the intercepts of the different isothermal equations and are presented in Table 2.

The adsorption behavior of methylene blue onto an inverse vulcanized polysulfide adsorbent was evaluated using Langmuir, Freundlich, and Temkin isotherm models to elucidate the nature of the interaction and the adsorbent's surface characteristics. The Langmuir model, which assumes monolayer adsorption on a homogeneous surface with uniform energy sites, yielded a poor fit ($R^2 = 0.3253$) and a non-physical negative Langmuir constant ($K_L = -1.22\text{ L/mg}$), indicating that the adsorption process does not conform to Langmuir assumptions. This suggests that the

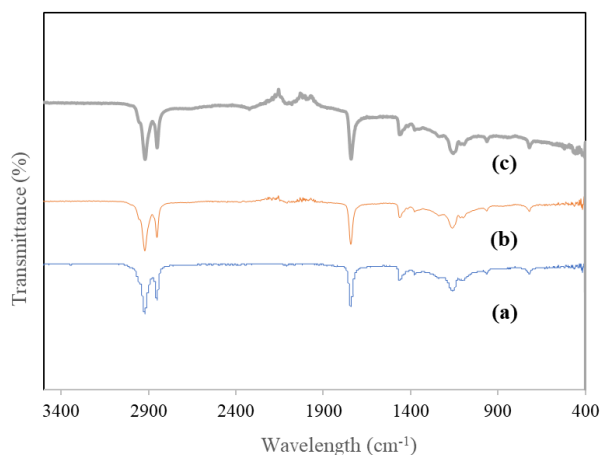


Figure 6. FTIR Analysis of PS 1 after adsorption with (a) 5 g, (b) 2 g, and (c) 1 g.

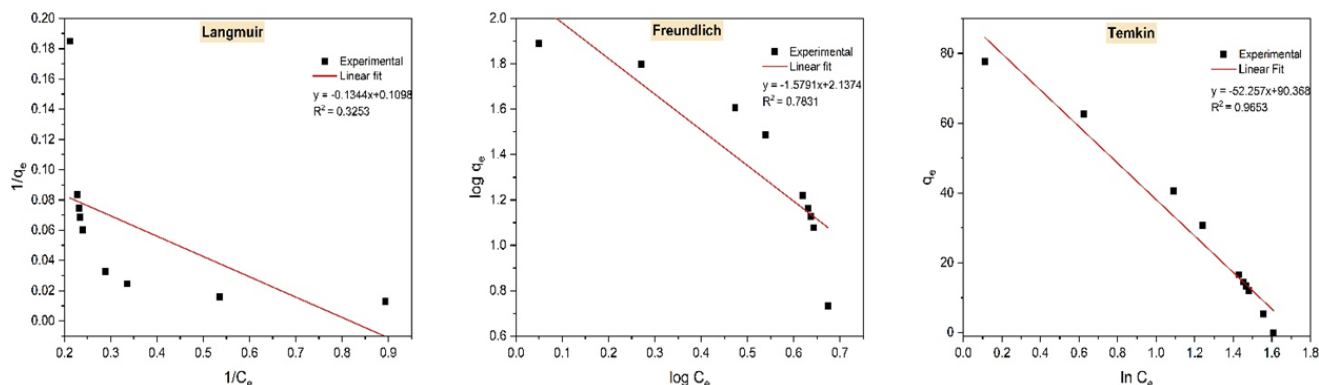


Figure 7. Langmuir, Freundlich, and Temkin isotherms of MB removal using polysulfide adsorbent (5 g/100 mL).

surface of the polysulfide adsorbent is energetically heterogeneous, and that adsorption likely occurs through multilayer formation or variable-energy interactions. In contrast, the Freundlich model provided a significantly better fit ($R^2 = 0.7831$), with a Freundlich constant $K_F = 137.2$ and an adsorption intensity $n = 0.636$, where $n < 1$ implies cooperative adsorption and a high degree of surface heterogeneity. These results are consistent with the structural complexity of inverse vulcanized polysulfide materials, which possess irregular morphologies and sulfur-rich functional groups capable of diverse physical interactions. The Temkin model, which assumes a linear decrease in adsorption energy with increasing surface coverage, showed the highest correlation ($R^2 = 0.9653$). Although traditionally associated with chemisorption, the unchanged FTIR spectra before and after adsorption confirm that no chemical bonding occurred, thereby supporting a physisorption mechanism. The excellent fit of the Temkin model in this context can be attributed to a distribution of adsorption energies arising from physical interactions such as electrostatic attraction between the cationic dye and negatively charged sulfur sites, π - π stacking with any conjugated domains in the polymer matrix, van der Waals forces, and pore-filling effects [21]. These mechanisms collectively contribute to the multilayer, energetically diverse adsorption behavior observed, validating the Freundlich and Temkin models as appropriate descriptors of the system, whereas the Langmuir model fails to capture the complexity of the adsorption process.

4. Conclusion

Polysulfides were successfully synthesized via a sustainable method of inverse vulcanization using waste cooking palm oil (WCO) and elemental sulfur. Characterization via FTIR, XRD, and SEM-EDX of the synthesized polysulfide confirmed the formation of polysulfide. FTIR spectra indicated the disappearance of C=C bonds due to sulfur incorporation, while XRD analysis confirmed the formation of an amorphous polymer structure. SEM-EDX observations revealed a coarse and porous morphology, which

is beneficial for adsorption processes. Among the synthesized materials, the 70:30 sulfur-to-WCO polysulfide composition exhibited the highest adsorption performance, achieving up to 78% MB removal within six hours. Adsorption experiments further demonstrated that increasing the adsorbent dosage significantly improved removal efficiency, indicating the availability of more active adsorption sites. The equilibrium data were best described by the Freundlich and Temkin isotherm models, suggesting a heterogeneous adsorption surface and multilayer adsorption behavior.

From a broader perspective, this work advances the current state of knowledge by demonstrating that waste cooking palm oil can be effectively valorized as a crosslinker for inverse-vulcanized sulfur polymers, producing a functional adsorbent for dye-contaminated wastewater. While previous studies on sulfur polymers have primarily focused on heavy-metal removal, this study shows their potential for organic dye adsorption, thereby expanding the application scope of sulfur-based materials. In addition, the use of two abundant waste resources (elemental sulfur and waste cooking oil) provides a sustainable, low-cost route to developing environmental remediation materials. These findings highlight the potential of inverse-vulcanized polysulfides as promising adsorbents for wastewater treatment and contribute to the growing field of waste-derived functional materials for environmental applications.

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Credit Author Statement

Author Contributions: A. Nayeem: Conceptualization, Methodology, Investigation, Data Curation, Draft Preparation, Review and Editing, Supervision. M. F. Ali: Resources, Review and Editing, Supervision. A. A. Narayanan: Methodology, Data Collection, Data Curation,

Table 2. Comparison of the key parameters of different isothermal models of MB adsorption.

Model	Equation	R^2	Key Parameters	Comments
Langmuir	$1/q_e = -0.1344(1/C_e) + 0.1098$	0.3253	$q_{max} = 9.11$ mg/g, $K_L = -1.22$ L/mg	Poor fit and unphysical K_L
Freundlich	$\log q_e = -1.5719 \log C_e + 2.1374$	0.7831	$K_F = 137.2$, $n = 0.636$	Moderate fit
Temkin	$q_e = -52.257 \ln C_e + 90.368$	0.9653	$b = -47.411$ J/mol, $A = 0.181$	Best fit

Draft Preparation. J.H. Shariffuddin: Conceptualization, Methodology, Resources, Validation, Review and Editing, Project Administration, Supervision. All authors have read and agreed to the published version of the manuscript.

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