



Influence of Magnetic Nanoparticle-Modified Sawdust on Dye Removal

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Abstract

Agricultural industry waste is considered a potential raw material for cellulose sources, providing important advantages. The aim of study is to analyze the properties of sawdust powder waste after being reinforced by magnetic (Fe₃O₄) nanoparticles. This research used Sengon (*Albizia chinensis*) wood sawdust waste to produce sawdust cellulose powder. The methods included the crushing process to reduce sawdust waste size, followed by the NaOH and peroxide process. Sawdust powder was reinforced with Fe₃O₄ nanoparticles at 10, 20, and 30wt%. The magnetic powder was analyzed using XRD, SEM, FTIR, and adsorber test for Congo Red and Methylene Blue dye. The XRD analysis indicates that the sawdust structure was identified as cellulose I β with the peak of the diffraction angles of 14°, 16°, and 22°. Fe₃O₄-NPs content reduces the crystallinity of sawdust cellulose powder from 76.05% to 73.31%, while FTIR showed shifts and losses of several cellulose bands that point to interaction between the particles and the cellulose. Incorporating Fe₃O₄-NPs results in the surface texture of the sawdust powder surface looks rougher. Adsorption tests revealed that Fe₃O₄-NPs loading enhanced Congo Red removal with a maximum gain of 17.1% (from 56.0% to 65.6%), whereas Methylene Blue removal slightly decreased from 97.0% to 89.9% due to reduced surface hydrophilicity.

Keywords: Cellulose powder; Dye adsorbent; Magnetite nanoparticles; Sawdust waste.

1. Introduction

Indonesia has 153 million people in urban areas and 117 million in rural areas in 2021. The total volume of wastewater (domestic, commercial, and industrial) is estimated to be 14.3 km³/year, while the treatment capacity is only 0.3 km³/year [1]. Water quality was degraded by the presence of waste pollution and made danger for the human and aquatic environment [2]. Treatment to reduce wastewater should be managed and has been a major challenge in the last decade [3].

Pollution from industry is a long-standing problem that slows development and threatens public health. Organic dyes are among the main pollutants because they are used heavily in textiles, cosmetics, paper, and many other products [4,5]. Many of them are toxic, carcinogenic, or teratogenic [6], and they break down only slowly through light or microbial action [7]. Dye-containing wastewater is commonly treated by oxidation [5] and activated

carbon [8], coagulation–flocculation [9], adsorption [10], or membrane separation [11]. Among adsorbents, synthetic and natural polymers are now used more often than inorganic materials, which tend to be costly, non-selective, and non-biodegradable [12]. A cheaper natural adsorbent that can be disposed of safely after use would lower overall treatment costs, and cellulose recovered from sawdust is one candidate.

Cellulose is the most abundant biopolymer on Earth, and its market was projected to reach about USD 48.37 billion by 2025 [13]. Most commercial cellulose comes from wood pulp. Indonesia produced 48.0 million m³ of logs in 2019 [14], leaving roughly 2 million m³ of sawdust per year [15] which makes sawdust an attractive cellulose source. Sawdust has already been tested as a low-cost adsorbent [16,17], but it is hard and slow to separate from treated water [18]. Cellulose also carries little surface charge density, which limits how it binds dyes [19]. Natural polymers such as cellulose can degrade over time, giving weaker and less stable flocs during coagulation–flocculation [20] and lower removal efficiency [21]. Earlier attempts to improve dye uptake have relied on chemical modification, such as surface functionalization or copolymerization of cellulose ethers [22,23] or on adding particles such as MoS₂ [24].

Over the last few years, research on cellulose nanocomposites has grown at a steady pace. When nanomaterials are added as reinforcement, they can change how the composite behaves and give it functions those cellulose alone lacks. The choice of filler therefore matters, because each one brings its own distinct properties. Fe₃O₄ nanoparticles (Fe₃O₄-NPs) are naturally hydrophilic [25], so the excess electrons on cellulose hydroxyl groups allow these groups to form bonds with oxygen atoms on the particle surface. A second kind of bonding comes from the attraction between the cellulose fibrils and the nanoparticles. The particles also interlock mechanically with the fibrils and further limit their movement, which in turn improves the mechanical properties of the nanocomposite [26,27]. Fe₃O₄-NPs based systems have also been shown to inactivate coliform and enterococcus bacterial communities [28] and to raise the charge capacitance of cellulose-based materials [29]. In wastewater treatment, attaching magnetic Fe₃O₄-NPs to cellulose offers a simple way to recover the material, since it can be pulled out using a magnet after use to be a coagulant or adsorbent for coloration. While various agricultural by-products have been explored as precursors for cellulose nanocomposites, the utilization of regional Sengon wood (*Albizia chinensis*) sawdust from Malang Regency, East Java, presents a unique low-cost and abundant lignocellulosic material with high availability. Unlike previously reported generic cellulose/magnetic composites, this study specifically addresses the regional waste stream of East Java by functionalizing localized Sengon sawdust powder (SWP) with varying Fe₃O₄-NPs loadings (10, 20, and 30 wt%). This work provides novel insights into balancing electrostatic surface charges to simultaneously evaluate the contrasting adsorption behaviors toward cationic (Methylene Blue) and anionic (Congo Red) model dyes, combined with facile magnetic recovery.

2. Materials and Methods

2.1 Materials

Sengon (*Albizia chinensis*) trees roughly five years old provided the sawdust, which was obtained in Malang Regency, East Java, Indonesia, and served as the cellulose source. Magnetite nanoparticles (Fe₃O₄-NPs, 30–50 nm) were purchased from Hongwu Material Technology (China). The alkali and bleaching steps used NaOH and H₂O₂, both supplied by Merck (Singapore). Two model dyes, methylene blue and Congo Red, came from Surya Techno Chemlab (Indonesia).

2.2 Alkalization

Sengon sawdust was first reduced to powder in a mill (10 min). To leach out water-soluble extractives, 100 g of this powder stayed submerged in 4 L of water for seven days, with fresh water supplied every second day. The wet material was then dried for 20 h in an oven kept at 110–120°C. Alkali treatment followed in batches of 4 g: each batch went into 80 mL of NaOH solution (5% w/v) and was heated for 3 h on a hotplate with a set temperature of 180°C. Afterward, the batch was washed with water four times and kept in 500 mL of distilled water for 3 h. This NaOH treatment and washing sequence was performed four times in total.

2.3 Bleaching Sawdust Cellulose

Dry SWP was bleached in 10% H₂O₂ at a ratio of 5 g per 100 mL, and the pH was raised to 11.5 with NaOH before stirring at 80°C for 30 minutes. When the reaction was complete, the SWP was recovered from the solution by filtration. It was then rinsed repeatedly until the pH reached neutral and left to air-dry for 1–3 days.

2.4 Cellulose/Fe₃O₄ - NPs Synthesis

Fe₃O₄-NPs were dispersed in 100 mL of distilled water at loadings of 0 wt% (control, SWP-Fe0), 10 wt% (SWP-Fe10), 20 wt% (SWP-Fe20), and 30 wt% (SWP-Fe30) and sonicated at 20 kHz for 30 minutes. Five grams of dried SWP were then added to each dispersion to form SWP composites. Each mixture was stirred for 30 minutes and sonicated again at 20 kHz for 30 minutes to break up agglomerates. The composites were oven-dried at 60°C for 20 hours.

2.5 Crystalline Analysis

X-ray diffraction (XRD; PANalytical X'Pert PRO, the Netherlands) was used to examine the composites' crystallinity. Each powder was scanned from 5° to 90° (2θ) at 40 kV and 30 mA with Cu Kα radiation (λ = 1.542 Å). The crystallinity index (CI) was obtained from Eq. (1) [30].

$$CI = \frac{I_{[002]} - I_{[am]}}{I_{[002]}} \times 100\% \quad (1)$$

Where: I_[002] is the highest intensity at 22.0° to 23.0° and I_[am] is the lowest intensity at 18°.

2.6 FTIR Analysis

Composite functional groups were identified by Fourier transform infrared (FTIR) spectroscopy (IRPrestige-21, Shimadzu, Japan). Before measurement, each composite was oven-dried at 105°C for 3 hours and ground to a powder. A 0.1 mg sample of the powder was blended with 1.0 mg of KBr and pressed into a pellet. Each spectrum was recorded between 400 and 4000 cm⁻¹ at 2 cm⁻¹ resolution.

2.7 Morphology Observation

Composite powder morphology was examined by scanning electron microscopy (SEM; Inspect S50, FEI, USA) at 25 kV. Before imaging, the samples were sputter-coated with a thin gold layer using an Emitech SC7620 coater (UK).

2.8 Dye Adsorbent

Dye removal was determined from concentrations measured with a UV-Vis spectrophotometer. For Methylene Blue (cationic dye), a 100 ppm stock solution was diluted to 50 mL of test solution at an initial concentration of 2 ppm (C₀). For Congo Red (anionic dye), a separate 2 ppm test solution (50 mL) was prepared at a neutral pH (~7.0). For both dyes,

0.5 g of adsorbent was added to the 50 mL solution, and the mixture was stirred continuously for a contact time of 30 minutes at room temperature (25°C). Absorbance values were recorded at maximum wavelengths of 662 nm for Methylene Blue and 497 nm for Congo Red. The residual dye concentration (C) after adsorption was determined using standard calibration curves. The percentage of dye removed (Rd) was then calculated from the initial and final concentrations using Eq. (2):

$$Rd = \frac{C_0 - C}{C_0} \times 100\% \quad (2)$$

Here, C_0 is the initial dye concentration in mg/L. C is the remaining concentration after adsorption (mg/L).

3. Results and Discussion

3.1 Morphology Observation

Fig. 1 shows how the morphology of the SWP changes once Fe_3O_4 -NPs are added to it.

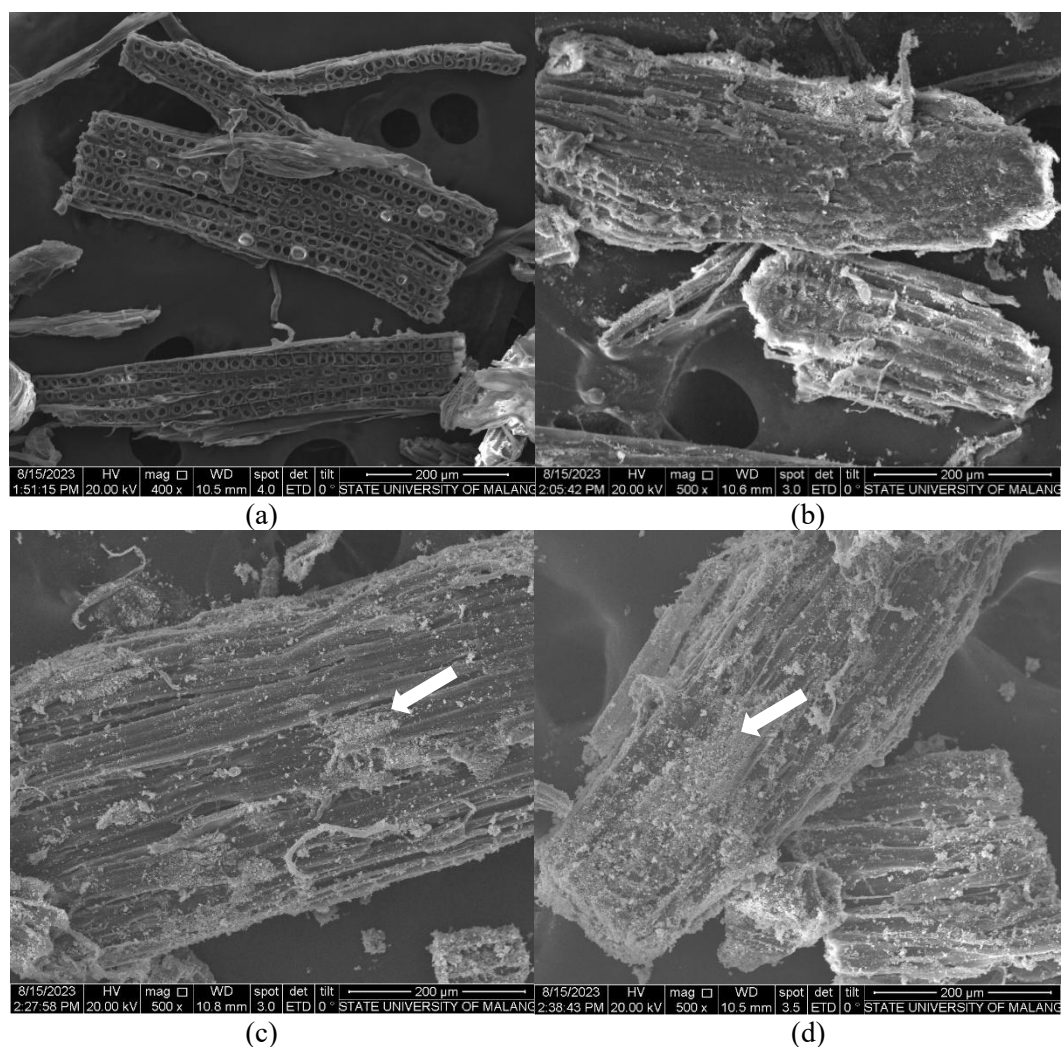


Fig. 1. SEM images of (A) unmodified SWP (SWP-Fe0), (B) SWP-Fe10, (C) SWP-Fe20, and (D) SWP-Fe30; White arrows highlight the formation and growing aggregation of Fe_3O_4 nanoparticles on the cellulose fiber surface.

Unmodified SWP-Fe0 keeps its fibrous structure, with visible cell walls and lumens (Fig. 1A). In SWP-Fe10, scattered Fe_3O_4 -NPs can be seen on the fiber surface (Fig. 1B). The particles begin to cluster in SWP-Fe20, giving it a rougher surface (Fig. 1C). Such clustering is typical of magnetite nanoparticles, which readily stick to one another through magnetic and van der Waals attraction and grow into larger aggregates [31]. Raising the loading to 30 wt% produces even larger clusters on SWP-Fe30 (Fig. 1D). Besides coating the fiber exterior, some Fe_3O_4 -NPs also become embedded in the SWP matrix, where they gather into further clusters below the visible surface. These particles bind well to cellulose because hydrogen bonds form between their surface and the cellulose hydroxyl groups [26]. However, a substantial interaction between the Fe_3O_4 -NPs and cellulose can influence the overall morphology of the SWP.

3.2 Crystallinity of Sawdust Powder

Fig. 2 presents the XRD patterns of all SWP composites. Four main cellulose reflections appear at $2\theta = 15.1^\circ$, 16.5° , 22.8° , and 34.4° , and they correspond to the (110), (110), (200), and (004) lattice planes [33,34], respectively, typical of cellulose I β . The Fe_3O_4 -NPs give rise to a separate set of reflections at $2\theta = 30.5^\circ$, 35.6° , 43.2° , 53.6° , 57.1° , 62.7° , 71.1° , 74.1° , and 86.8° . These reflections match well with the (220), (311), (400), (422), (511), (440), (620), and (622) planes of cubic spinel magnetite (JCPDS No. 19-0629). Between $2\theta = 10^\circ$ and 90° , the overall pattern changed only slightly with Fe_3O_4 -NPs loading, but the magnetite peaks grew stronger as more Fe_3O_4 -NPs were added. These peaks are clearly visible, most likely because many of the particles have accumulated on the outer SWP surface. At higher loadings the particles also aggregate more, so their reflections become more prominent in the pattern. In SWP-Fe10 and SWP-Fe20, the same magnetite peaks are still present but noticeably weaker. This is consistent with SWP-Fe30 containing the most nanoparticles, since it was prepared with the highest amount of Fe_3O_4 -NPs among all samples [35]. The cellulose arrangement becomes random as a result of the greater Fe_3O_4 -NPs content, and the crystallinity value also decreases [36].

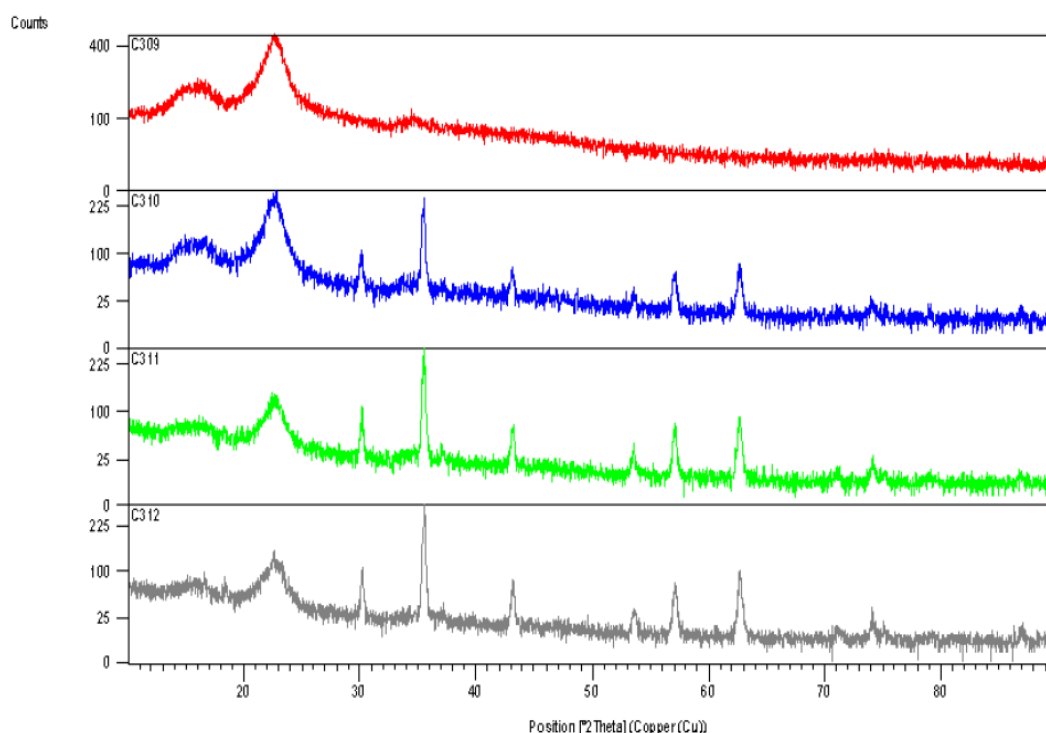


Fig. 2. Diffraction pattern of SWP with Fe_3O_4 -NPs reinforcement

SWP-Fe0 has a CD of 76.05% and a CI of 68.50% (Table 1). With the addition of 10, 20, and 30 wt% Fe₃O₄-NPs, the CD falls to 75.70%, 74.71%, and 73.31%, while the CI drops to 67.91%, 66.15%, and 63.58%, respectively. Crystallite size, by contrast, increases from 12.18 nm in SWP-Fe0 to 13.08, 15.35, and 15.75 nm in SWP-Fe10, SWP-Fe20, and SWP-Fe30, respectively. This increase is probably linked to the Fe₃O₄-NPs taken up into the SWP, since magnetite nanoparticles have large crystallites, typically between 9 and 53 nm, which would raise the average value obtained from the pattern [37]. This set of results collectively illustrates that the integration of Fe₃O₄-NPs played a role in enhancing the overall crystalline characteristics of the SWP, potentially carrying important implications for the eventual properties of the SWP.

Table 1. Crystalline properties of SWP composite

Samples	Intensity (a.u.)		Crystalline Index (%)	Crystallite Size (nm)
	I _{22.5}	I ₁₈		
SWP-Fe0	433.29	136.48	68.50	12.18
SWP-Fe10	244.09	78.336	67.91	13.08
SWP-Fe20	135.15	45.751	66.15	15.35
SWP-Fe30	121.74	44.333	63.58	15.75

3.3 Functional Group Analysis

FTIR was used to compare the chemical structure of SWP before and after Fe₃O₄-NPs addition. The spectra for all four samples appear in Fig. 3. The black curve corresponds to unmodified SWPFe0, and the other three curves show the spectra of SWP functionalized with 10, 20, and 30 wt% Fe₃O₄-NPs (SWP-Fe10, SWP-Fe20, and SWP-Fe30). All four spectra cover the wavenumber range of 400–4000 cm⁻¹.

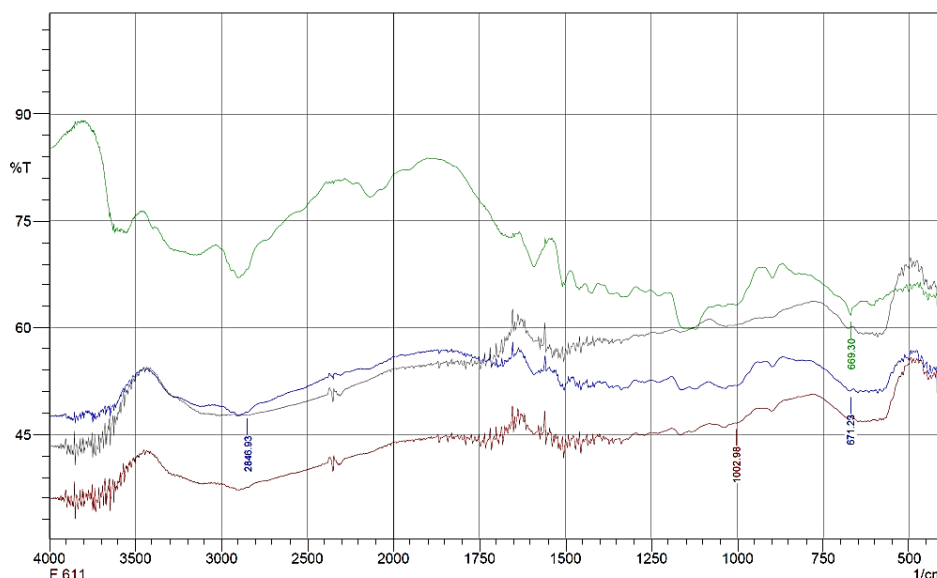


Fig. 3. Infrared spectrum for SWP composite with Fe₃O₄-NPs reinforcement

Several peaks shift or disappear when the Fe₃O₄-NPs loaded spectra are compared with the unmodified SWP spectrum. These changes reflect the intermolecular interactions formed between SWP and the Fe₃O₄-NPs. In the 2700–3600 cm⁻¹ region, the characteristic O–H and

C–H stretching bands of cellulose shift to new positions once Fe_3O_4 -NPs are incorporated into the SWP [38]. The broad band between 3300 and 3600 cm^{-1} comes from O–H stretching vibrations, and its shape and position are strongly sensitive to hydrogen bonding in cellulose. Changes appear at several wavenumbers once the Fe_3O_4 -NPs have been added. The O–H band at 3600 cm^{-1} is no longer visible. This points to a change in the hydrophilic character of the SWP. The C–H stretching band located near 2900 cm^{-1} also fades, and its dip in the transmittance spectrum is almost entirely lost. Between 760 and 1800 cm^{-1} , the spectra contain vibrational modes of various organic groups [39]. From 1520 down to 400 cm^{-1} , the cellulose bands become broader, which is attributed to bending and stretching vibrations of CH_2 , C–H, and C–O groups in the cellulose chains [40]. The C=O band at 1589 cm^{-1} becomes weaker, meaning its transmittance dip is now shallower, which suggests that fewer of these bonds remain [41]. The bands at 1156 and 894 cm^{-1} , assigned to C–O–C stretching and the β -glycosidic linkage of cellulose, also lose much of their intensity. New bands appear at 563 and 430 cm^{-1} in the Fe_3O_4 -NPs loaded samples, and these are characteristic of Fe–O stretching in the magnetite lattice [42].

3.4 Dye Absorption

In the adsorption test results (Fig. 4), unmodified SWP-Fe0 removed 97.0% of the methylene blue (0.194 mg/g), while SWP-Fe10, SWP-Fe20, and SWP-Fe30 removed 95.4% (0.191 mg/g), 93.3% (0.187 mg/g), and 89.9% (0.180 mg/g). Relative to the control, methylene blue uptake therefore fell by 1.6%, 3.8%, and 7.3%, respectively, as the Fe_3O_4 -NPs loading progressively increased. Congo Red behaved differently: SWP-Fe0 removed 56.0% of it (0.112 mg/g). With Fe_3O_4 -NPs present, the removal rose steadily to 58.4% (0.117 mg/g) for SWP-Fe10, 64.0% (0.128 mg/g) for SWP-Fe20, and 65.6% (0.131 mg/g) for SWP-Fe30. These values correspond to relative gains in capacity of 4.3%, 14.3%, and 17.1% over the unmodified powder, respectively.

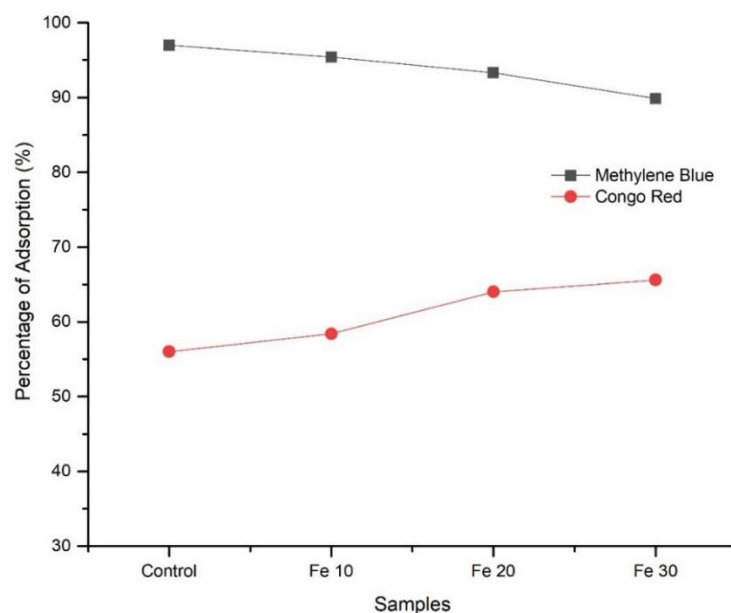


Fig. 4. Adsorption of dye by magnetic SWP

The unmodified and magnetic SWP behave differently as adsorbents, and these differences explain the opposite trends for the two dyes. Unmodified SWP takes up cationic dyes such as methylene blue readily, since hydroxyl groups are distributed along the cellulose chain and provide many binding sites for them [43]. Methylene blue binds to cellulose mainly through

hydrogen bonds and van der Waals attraction [44]. Fe₃O₄-NPs addition lowers methylene blue uptake, probably because the particles make SWP less hydrophilic once they interact with it. The anionic dye Congo Red, however, shows the opposite trend. Its negatively charged sulfonate groups are repelled by the slightly negative SWP surface, yet uptake rises by up to 17.1% once magnetic nanoparticles are added [45]. This increase is attributed to the positively charged surface of magnetic nanoparticles in neutral water. The positive particles attract the negatively charged Congo Red molecules, so the composite captures more of this dye than unmodified SWP.

To demonstrate the practical application and ease of recovery of the magnetic adsorbent, a separation experiment was performed using an external permanent magnet. When dispersed in an aqueous dye solution, the Fe₃O₄-modified sawdust powder remains uniformly suspended during active adsorption. Upon applying an external neodymium magnet to the outer wall of the beaker, the composite particles rapidly migrate toward the magnetic field and clear solution is achieved within 30 seconds. This rapid magnetic separation eliminates the requirement for energy-intensive filtration or centrifugation, confirming that the synthesized nanocomposite possesses effective magnetic responsiveness suitable for convenient post-treatment recovery and potential reusability.

The practical utility of any industrial adsorbent relies heavily on its reusability and operational stability across multiple treatment cycles. Owing to the strong magnetic responsiveness provided by the incorporated Fe₃O₄-NPs, the SWP/Fe₃O₄-NPs composite can be rapidly separated from treated wastewater using an external magnetic field, eliminating the need for complex filtration or centrifugation. Based on similar lignocellulosic-magnetic composite systems reported in the literature, magnetic biopolymer adsorbents typically retain over 80–85% of their initial adsorption capacity after 4 to 5 consecutive adsorption-desorption cycles. The slight decrease in removal efficiency over multiple cycles is generally attributed to minor leaching of magnetite nanoparticles, incomplete desorption of dye molecules from inner pores, or loss of active surface sites during chemical regeneration. Overall, the facile magnetic recovery and robust structural stability of the SWP/Fe₃O₄-NPs composite demonstrate high economic viability and practical potential for scalable wastewater remediation.

4. Conclusions

This study examined how Fe₃O₄-NPs alter sawdust-derived cellulose powder. SEM showed the nanoparticles settling on the SWP and spreading across its surface. At higher loadings, the particles clustered into larger, visible agglomerates on the fiber surface. FTIR changes at 1156–1589 cm⁻¹ and Fe–O bands at 563 and 430 cm⁻¹ indicated interaction between SWP and Fe₃O₄-NPs. Crystallinity degree and index fell slightly with Fe₃O₄-NPs addition, from 76.05% to 73.31% and 68.50% to 63.58%, respectively. In contrast, the average crystallite size grew from 12.18 nm to 15.75 nm. Fe₃O₄-NPs raised Congo Red uptake by up to 17.1%, while methylene blue uptake decreased only slightly. Magnetic SWP is therefore a promising adsorbent for anionic dyes and merits further development and testing for wastewater treatment. Furthermore, the facile magnetic recovery of the composite highlights its strong potential for multi-cycle recyclability and sustainable application in industrial dye wastewater treatment.

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Author Declaration

Data availability statement: The data supporting the findings of this study are available from the corresponding author upon reasonable request.

CRedit authorship contribution statement: JM: Conceptualization, Methodology, Investigation, Writing original draft, Writing – review & editing. RES: Investigation, Data curation, Visualization. RAA: Validation, Resources, Writing – review & editing. AM: Conceptualization, Project administration, Writing – review & editing.

Declaration of Competing Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Artificial Intelligence Statement: During the preparation of this work, the authors used Claude (Anthropic) to paraphrase and improve the language and readability of the manuscript text. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication. All data, analyses, and scientific conclusions were produced by the authors.

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