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PERFORMANCE OF $FeCl_3$ AND CHIA SEED (*SALVIA HISPANICA L.*) AS A COAGULANT–FLOCCULANT COMBINATION FOR REMAZOL RED DYE REMOVAL

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Abstract

Textile dye wastewater contains persistent and potentially carcinogenic pollutants that threaten aquatic ecosystems. This study applied a coagulation–flocculation technique to evaluate the treatment efficiency of $FeCl_3$ as a synthetic coagulant combined with a natural flocculant derived from chia seeds (Salvia hispanica L.) for the removal of Remazol Red dye. Operational parameters, including pH, initial dye concentration, and coagulation stirring speed, were varied to assess treatment effectiveness. The results showed that the addition of chia seed flocculant significantly increased dye removal efficiency, reaching 99.01% under the optimum conditions of pH 5, an initial dye concentration of 50 mg/L, and a stirring speed of 700 rpm. The percentage removal of Remazol Red dye was determined using a UV-Vis spectrophotometer, while a Fourier transform infrared (FTIR) analysis was used to identify the functional groups present in the resulting flocs. The FTIR results confirmed the proposed mechanism, suggesting that $FeCl_3$ facilitated charge neutralization and subsequently promoted a polymer-bridging mechanism through natural chia seed polymers. This combined system provides a highly efficient and environmentally friendly alternative for textile wastewater treatment, thereby reducing dependence on purely synthetic chemicals.

Keywords: *Chia seeds; Coagulation-flocculation; $FeCl_3$; Remazol Red; Textile wastewater*

Introduction

Synthetic dyes are used in various sectors, including cosmetics, plastics, paper, and textiles. However, improperly managed dye wastewater is often discharged directly into water bodies, potentially endangering aquatic ecosystems [1]. These dyes can damage aquatic environments and are also associated with cancer risks in humans. Furthermore, dye wastewater can increase biochemical oxygen demand (BOD) and chemical oxygen demand (COD) to hazardous levels [2], [3], [4]. Synthetic dyes are extensively used in global industries, particularly in the clothing and textile sectors. Therefore, efficient and sustainable management of dye wastewater generated by these industries is crucial [5], [6].

The production of azo dyes increases every year. Remazol Red is one of the azo dyes commonly used in textile applications [6], [7], [8]. The chemical formula of Remazol Red is $C_{20}H_{22}N_2NaO_{11}S_3$ [9], [10]. Azo dyes contain N=N groups and, because of their stable and complex structures, are difficult to decompose. In addition, this dye is harmful to aquatic systems because it inhibits photosynthesis by reducing light penetration into water [11]. To reduce these negative effects, wastewater generated during the textile dyeing process must be properly treated.

Several chemical, biological, and physical methods can be used to remove azo dyes. Coagulation–flocculation is one of the most commonly used methods in textile wastewater treatment because of its efficiency and ease of operation. Coagulants are generally classified into two types: inorganic coagulants, often called synthetic coagulants, and natural coagulants. Inorganic coagulants, such as ferric chloride ($FeCl_3$), can effectively remove colloidal particles, including dyes. This is because Fe^{3+} ions have high hydrolytic capacity, as indicated by their large hydrolysis constant (K_a). The hydrolysis process produces positively charged species and $Fe(OH)_3$ flocs, which destabilize colloidal particles by neutralizing their charges and facilitating their removal through adsorption and coagulation [12]. However, the use of synthetic chemicals alone often leads to significant corrosion and large sludge volumes [13], [14]. Residual metal particles in treated water may also pose health risks. To mitigate the negative effects of excessive chemical use, more environmentally friendly treatment agents should be explored.

To improve the efficiency of Remazol Red removal while reducing environmental impact, the use of biopolymers from natural materials as bioflocculants has become a major research focus. Recent studies have shown that combining synthetic coagulants with natural flocculants, rather than using them separately, can substantially improve dye removal efficiency [15]. Chia seeds (*Salvia hispanica* L.) are a potential candidate because of their high polysaccharide mucilage content, which facilitates effective interparticle binding through a bridging mechanism [16]. Despite numerous investigations of natural flocculants, limited information is available regarding the specific synergy between $FeCl_3$ and chia seeds for the removal of reactive azo dyes such as Remazol Red.

Operational factors such as pH, coagulant dosage, and stirring speed significantly influence the efficiency of the coagulation–flocculation process [17]. However, research on the effects of stirring speed and initial solution concentration in combined synthetic–natural systems remains limited [18], particularly regarding the relationship between iron hydroxide species and natural biopolymers. Therefore, this study aimed to evaluate the ability of a combined $FeCl_3$ and chia seed flocculant system to remove Remazol Red dye. This research contributes to the development of more economical, efficient, and sustainable textile wastewater treatment techniques by examining how solution pH, dye concentration, coagulation speed, and removal mechanisms affect floc formation.

Experimental Section

Materials

Remazol Red dye was used to prepare synthetic wastewater stock solutions for all experiments. The other chemicals and materials used included ferric chloride ($FeCl_3$), chia seeds used as mucilage/gel, sodium hydroxide (NaOH, >99%), hydrochloric acid (HCl, 37%), filter paper, universal pH indicator strips (Merck), and distilled water. All chemical reagents used were of analytical grade.

Instruments

A UV-Vis spectrophotometer (Genesys 10S, Thermo Scientific) was used to measure absorbance and determine the residual concentration of Remazol Red dye. The functional groups of the resulting flocs were analyzed using a Fourier transform infrared (FTIR) spectrometer (Shimadzu 8400S, Japan).

Experimental Procedure

Preparation of Coagulant and Flocculant

Commercial ferric chloride ($FeCl_3$) powder was used as the synthetic coagulant at a dosage of 550 mg/L for the coagulation–flocculation process [19]. Twenty grams of chia seeds were soaked in distilled water at 50°C to obtain the natural flocculant. The resulting mucilage was separated by centrifugation and blending. The gel-like mucilage extracted from chia seeds was subsequently used as the natural flocculant in the coagulation–flocculation process (see Figure 1).

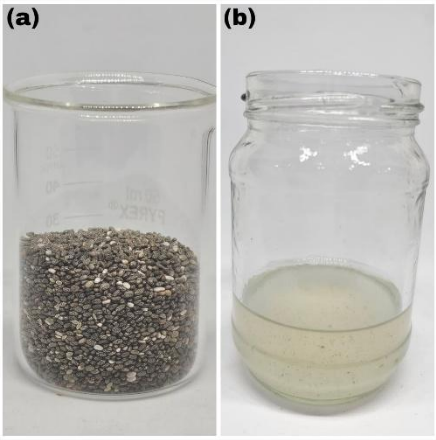

Figure 1. (a) Chia seeds and (b) chia seed gel.

Preparation of Dye Solutions

A 100 mg/L Remazol Red stock solution was prepared and stored in a sealed container. The stock solution was diluted with distilled water to obtain 200 mL of a 50 mg/L test solution for the pH and coagulation-rate experiments. For the initial concentration variation test, 200 mL solutions containing 30, 40, 50, 60, and 70 mg/L Remazol Red were prepared. Before treatment, the pH of the test solution was adjusted to 3, 4, 5, 6, 7, 8, and 9 using either HCl or NaOH.

Coagulation–Flocculation Process

A combination of rapid and slow mixing stages was applied in the treatment procedure. The $FeCl_3$ coagulant was added to the test solution during the coagulation phase, which involved rapid mixing at varying coagulation speeds of 500, 600, 700, 800, and 900 rpm for 4 minutes. Flocculation, or slow mixing, was then initiated by adding 300 mg/L of chia seed flocculant to the mixture. This stage was performed for 30 minutes at a speed of 50 rpm.

Analytical Measurements

After the mixing phase was completed, the suspension was allowed to settle for 30 minutes to ensure complete floc sedimentation. The supernatant was then filtered using filter paper. A UV-Vis spectrophotometer was used to measure the final absorbance of the filtrate and determine the residual dye concentration. The removal efficiency of Remazol Red dye was calculated using the following equation:

$$\% \text{ Removal} = \frac{(C_0 - C)}{C_0} \, x \, 100\%$$

where C_0 represents the initial concentration of the dye solution before treatment, and C represents the final dye concentration after treatment.

Fourier Transform Infrared Characterization

FTIR spectroscopy was used to identify the functional groups involved in the treatment process. Pure Remazol Red dye, $FeCl_3$ coagulant, and extracted chia seed mucilage were examined directly to determine their baseline spectra. The flocs generated during the coagulation–flocculation process were also collected and dried appropriately before FTIR analysis.

Data Analysis

All experimental measurements were performed in triplicate (n = 3), and the results are presented as the mean ± standard deviation (SD). Microsoft Excel and SPSS were used to perform statistical analyses. One-way analysis of variance (ANOVA) was used to evaluate the effects of test solution pH, initial dye concentration, and coagulation stirring speed.

Results and Discussion

Effect of test solution pH

The efficiency of Remazol Red dye removal using the combined system of 550 mg/L $FeCl_3$ coagulant and 300 mg/L chia seed natural flocculant was strongly influenced by the initial pH of the test solution. As shown in Figure 2, pH 5 produced the highest dye removal efficiency, reaching 98.98%. In contrast, removal performance decreased significantly under strongly acidic and strongly alkaline conditions. This trend demonstrates the important role of pH in the overall effectiveness of the coagulation–flocculation process.

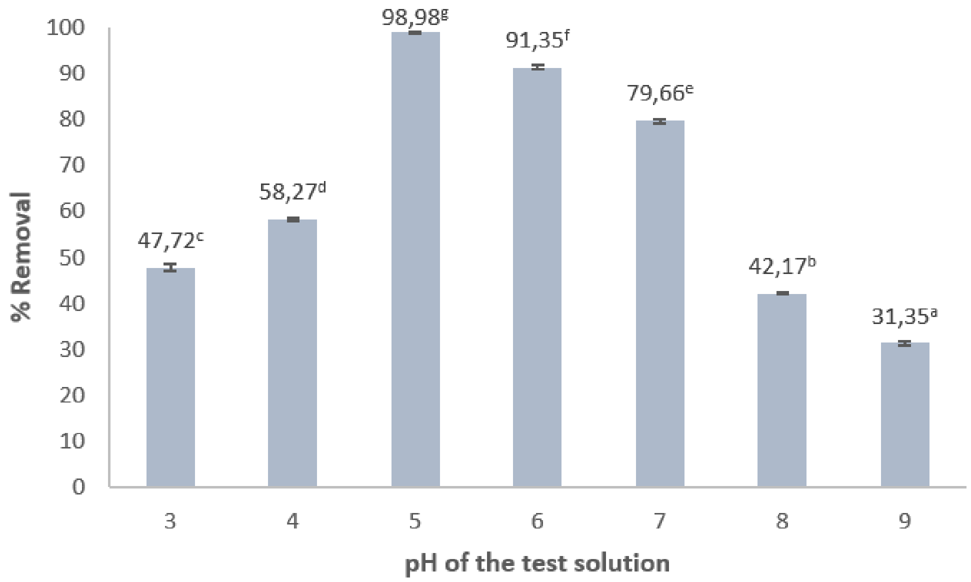

Figure 2. Effect of test solution pH on Remazol Red dye removal efficiency. Conditions: $FeCl_3$ dosage = 550 mg/L, chia seed dosage = 300 mg/L, and initial dye concentration = 50 mg/L.

When metal-based synthetic coagulants such as ferric chloride ($FeCl_3$) and alum are used, the initial pH of the solution is highly important. The pH of the solution usually decreases when these metal coagulants are added, and this effect is more noticeable at higher coagulant dosages and lower water alkalinity [20], [21], [22]. Previous studies evaluating $FeCl_3$ as a single coagulant reported a maximum removal efficiency of 96.6% at an optimum pH of 6. This suggests that, even under optimal pH conditions, the use of synthetic coagulants alone limits dye removal performance. Conversely, combining a synthetic coagulant with a natural flocculant enhances removal performance. Due to the active polysaccharides in the natural flocculant, the removal efficiency of Remazol Red increased to 98.98%.

Table 1. Effect of test solution pH

Coagulant dosage (mg/L)	Flocculant dosage (mg/L)	Coagulation speed (rpm)	Flocculation speed (rpm)	pH of test solution	Removal (%)
550	300	700	50	3	47.72
550	300	700	50	4	58.27
550	**300**	**700**	**50**	**5**	**98.98**
550	300	700	50	6	91.35
550	300	700	50	7	79.66
550	300	700	50	8	42.17
550	300	700	50	9	31.35

Table 1 shows that dye removal efficiency increased as the pH increased from 3 to 5. At this pH, positively charged iron species neutralized the negatively charged Remazol Red dye molecules. Under acidic conditions, free protons (H^+) attach to functional groups on the dye surface, thereby increasing Remazol Red dye removal efficiency. The main iron species, $FeOH^{2+}$ and $Fe(OH)_2^+$, were found at pH 3 and pH 5. The highest Remazol Red removal efficiency occurred at pH 5 due to the presence of $Fe(OH)_2^+$ species [23]. These species have a significant positive charge, making them more likely to interact with negatively charged dyes through electrostatic forces and thereby supporting microfloc formation. The polymer-bridging mechanism then helps these microflocs bind to the natural chia seed flocculant, resulting in larger flocs that settle more easily.

In contrast, at pH levels of 6, 7, 8, and 9, removal efficiency decreased significantly. A substantial increase in pH reduces charge-neutralization efficiency by altering $FeCl_3$ hydrolysis and decreasing the availability of positive charges [24]. Amorphous iron hydroxide species, $Fe(OH)_3$, are mostly formed under slightly acidic to basic conditions, ranging from pH 6 to pH 9. Once formed, these precipitates remain partially suspended and have a neutral charge [23], [25]. The reduction in positively charged coagulant species weakens electrostatic attraction to the dye, leading to decreased Remazol Red removal.

In this study, the coagulation–flocculation process was carried out at pH 5 with a Remazol Red concentration of 50 mg/L, using a synthetic $FeCl_3$ coagulant dosage of 550 mg/L and a chia seed natural flocculant dosage of 300 mg/L. The coagulation stirring speed was 700 rpm. Under these conditions, the optimal Remazol Red removal efficiency was 98.98%. This value is higher than that reported in previous studies using papaya seed biocoagulant and aloe vera bioflocculant, which achieved 97.41% dye removal. The optimum conditions in that previous study were pH 2, with biocoagulant and bioflocculant dosages of 2 mg and 5 mg, respectively [26].

Effect of initial solution concentration

Figure 3 shows the effect of the initial Remazol Red dye concentration on overall removal efficiency. This test was conducted under controlled conditions using 550 mg/L $FeCl_3$ as the synthetic coagulant and 300 mg/L chia seed mucilage as the natural flocculant. In this study, an optimal initial dye concentration of 50 mg/L resulted in a dye removal efficiency of 98.54%. In contrast, previous research using slightly lower concentrations of biocoagulants and bioflocculants reported a dye removal efficiency of 97.55% [26]. These results indicate that the combination of a synthetic metal coagulant and a natural polysaccharide flocculant was more effective than the combination of a biocoagulant and bioflocculant.

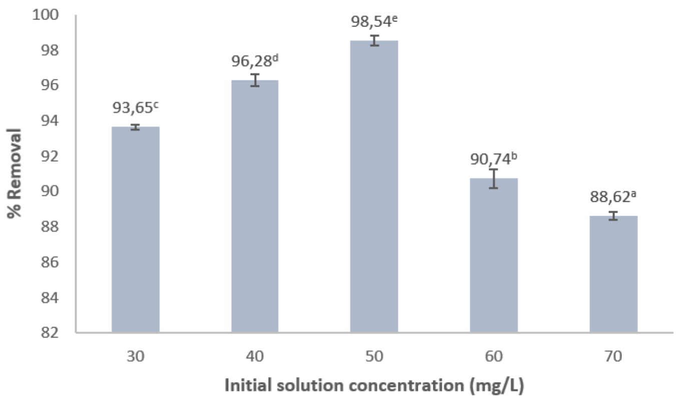

Figure 3. Effect of initial solution concentration on Remazol Red dye removal efficiency. Conditions: $FeCl_3$ dosage = 550 mg/L, chia seed dosage = 300 mg/L, and pH = 5.

The highest removal efficiency was obtained at a Remazol Red concentration of 50 mg/L, with lower efficiencies observed at initial concentrations of 30 and 40 mg/L. However, when the initial concentration was increased to 60 and 70 mg/L, removal efficiency decreased substantially. This decrease may be due to the rapid saturation of available active binding sites on the flocculant and coagulant surfaces by the excess dye molecules. The dense accumulation of these molecules causes strong electrostatic repulsion among similarly charged Remazol Red anions, preventing further aggregation and efficient floc formation [18].

Table 2. Effect of initial solution concentration

Initial solution concentration (mg/L)	Coagulant dosage (mg/L)	Flocculant dosage (mg/L)	Coagulation speed (rpm)	Flocculation speed (rpm)	pH of test solution	Removal (%)
30	550	300	700	50	5	93.65
40	550	300	700	50	5	96.28
50	**550**	**300**	**700**	**50**	**5**	**98.54**
60	550	300	700	50	5	90.74
70	550	300	700	50	5	88.62

Table 2 shows that lower initial dye concentrations of 30 and 40 mg/L produced removal efficiencies of 93.65% and 96.28%, respectively. The highest efficiency was achieved at 50 mg/L. However, the efficiency decreased beyond this concentration, declining to 90.74% at 60 mg/L and 88.62% at 70 mg/L. At lower concentrations, the amount of Remazol Red dye was balanced with the binding capacity of the $FeCl_3$ coagulant and chia seed flocculant. Charge neutralization and subsequent floc formation therefore occurred effectively under these conditions. Conversely, the lower removal efficiency at higher concentrations indicates that the number of dye molecules exceeded the available active binding sites at the given coagulant and flocculant dosages. As a result, unbound dye particles remained in suspension, reducing the overall effectiveness of the coagulation–flocculation process.

Effect of coagulation stirring speed

The overall efficiency of the coagulation–flocculation process is significantly influenced by system hydrodynamics, particularly the rapid mixing speed during the coagulation phase. The stirring speed directly regulates the frequency of particle collisions and coagulant dispersion. The effect of varying coagulation stirring speeds on Remazol Red dye removal efficiency is shown in Figure 4.

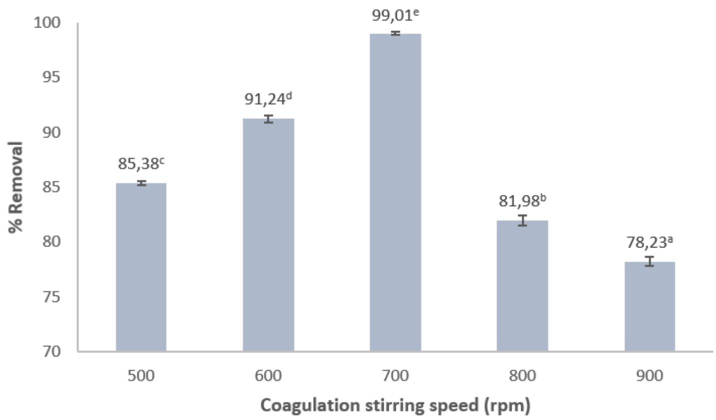

Figure 4. Effect of coagulation stirring speed on Remazol Red dye removal efficiency. Conditions: $FeCl_3$ dosage = 550 mg/L, chia seed dosage = 300 mg/L, initial dye concentration = 50 mg/L, and pH = 5.

The results in Figure 4 indicate that 700 rpm was the optimal coagulation stirring speed. The tests were conducted under constant operating conditions, with an initial Remazol Red dye concentration of 50 mg/L, an $FeCl_3$ coagulant dosage of 550 mg/L, and a chia seed flocculant dosage of 300 mg/L. After 4 minutes of rapid mixing during coagulation, the process was followed by a slow-mixing phase to promote flocculation.

Table 3. Effect of coagulation speed

Coagulant dosage (mg/L)	Flocculant dosage (mg/L)	Coagulation speed (rpm)	Flocculation speed (rpm)	pH of test solution	Removal (%)
550	300	500	50	5	85.38
550	300	600	50	5	91.24
550	**300**	**700**	**50**	**5**	**99.01**
550	300	800	50	5	81.98
550	300	900	50	5	78.23

Previous research using only biocoagulants and bioflocculants at 700 rpm reported a maximum removal efficiency of only 87.62% [27]. This limitation may be attributed to suboptimal adsorption capacities and less effective particle collisions when purely biological agents are used. In contrast, under the same coagulation speed, the synergistic combination of a synthetic coagulant and a natural flocculant in this study achieved significantly higher performance. As shown in Table 3, the optimal coagulation speed of 700 rpm produced a maximum Remazol Red removal efficiency of 99.01%.

The treatment efficiency initially increased as the coagulation speed increased from 500 to 600 rpm, before reaching its maximum at 700 rpm. Statistical analysis indicated significant differences among the various stirring-speed treatments. The increased mixing intensity initially improved removal efficiency by dispersing the $FeCl_3$ coagulant uniformly throughout the solution. This rapid and uniform dispersion enhanced the frequency of particle collisions and accelerated the hydrolysis of Fe^{3+} into highly active species, such as $Fe(OH)_2^+$, which effectively neutralized the negative charge of Remazol Red molecules. This process resulted in colloidal destabilization between the Remazol Red dye and $FeCl_3$ coagulant. Furthermore, slow stirring with the addition of chia seed flocculant promoted polysaccharide polymer bridging, which bound the microflocs formed during coagulation into larger macroflocs during the flocculation process [28], [29]. However, as shown in Table 3, increasing the speed to 800 and 900 rpm caused the removal efficiency to decrease. Excessive agitation at high speeds generates strong hydrodynamic shear stress, which can break the newly formed microflocs and release trapped dye molecules back into the suspension. Consequently, the overall treatment performance decreases [30].

Fourier Transform Infrared Analysis

Chemical interactions and functional groups associated with the dye removal process were identified using FTIR spectroscopy. Four distinct samples were used for spectral characterization: pure Remazol Red dye, $FeCl_3$ coagulant, natural chia seed flocculant, and solid flocs formed after the complete coagulation–flocculation process.

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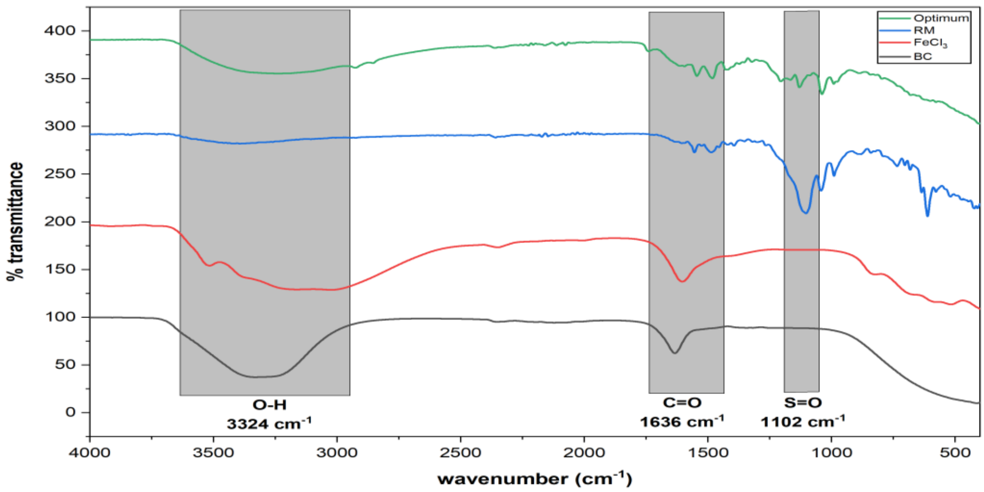

Figure 5. Fourier transform infrared characterization.

The FTIR spectra show that the O–H stretching vibration in pure chia seeds initially appeared at 3324 cm^{-1}. In the spectrum of the flocs formed under optimal conditions, this peak shifted to 3309 cm^{-1}, which falls within the typical broad O–H stretching range of 3200–3500 cm^{-1}. This distinct shift indicates that the hydroxyl groups in chia seed polysaccharides participated in binding interactions during treatment [31].

In addition, a change in peak intensity around 1636 cm^{-1}, corresponding to the C=O functional group, indicates that free dye molecules were successfully trapped in the flocs and removed from the water. In the post-treatment floc spectrum, the sulfonate group (S=O) in Remazol Red dye showed a distinct absorption band centered at 1106 cm^{-1}, which subsequently decreased in intensity. This substantial decrease in the sulfonate peak further demonstrates that the combined coagulation–flocculation process effectively removed Remazol Red dye.

Conclusion

Overall, the synergistic combination of $FeCl_3$ coagulant and natural chia seed flocculant effectively removed Remazol Red dye from aqueous solutions. The removal mechanism was primarily driven by colloidal charge destabilization followed by polysaccharide-mediated polymer bridging. Overall treatment efficiency was highly dependent on the initial solution pH, dye concentration, and coagulation stirring speed. A maximum removal efficiency of 99.01% was obtained at pH 5, an initial dye concentration of 50 mg/L, and a coagulation speed of 700 rpm. FTIR spectral analysis also confirmed the binding process, showing that the O–H, C=O, and S=O functional groups actively participated in the coagulation–flocculation process. Although the results are very encouraging, the present study was limited to laboratory-scale synthetic dye solutions. Future research is therefore strongly recommended to scale up the process and assess its technical and financial feasibility using actual textile industrial wastewater.

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