JJEES

JJEES (2024) 15 (4): 287-297
ISSN 1995-6681

Jordan Journal of Earth and Environmental Sciences

# Removal of Color from Textile Dyeing Effluents Using Coagulation-Flocculation Process Coupled with Adsorption on Nanoparticles Process

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*Received on August 28, 2023; Accepted on November 6, 2024*

**Abstract**

Textile dyeing industries discharge large volumes of effluents that threaten sustainable water resources and environmental management. This study aimed to explore the feasibility of treating discharge textile dye effluents by combining coagulation and adsorption processes to achieve the required discharge wastewater standards. The X-RD analysis results confirmed that the synthesized iron-oxide particles were found to be within the nano-size range (10-20 µm). The chemical coagulation process showed about 75% color removal efficiency, and the cumulative color removal efficiency achieved was 99.5% in combined coagulation, followed by the adsorption process with iron-oxide nanoparticle adsorbents (IONPs). The SEM images showed the rough and porous surface of virgin IONPs, having a slightly hazy and smooth surface for the adsorbed IONPs. The EDX analysis confirmed the presence of various metal ions on the IONPs surface. The study illustrated that the combination of coagulation-flocculation (C-F) and adsorption onto IONPs was more efficient for decolorizing textile dyeing effluents than the single coagulation process. The study observed that the coagulant $FeSO_4$ and the adsorbent IONPs have the potential to treat textile dyeing effluents and achieve the required standards for discharging the effluent into the environment.



**Keywords:** *Dyeing Effluents, Coagulation-Flocculation, Adsorption, Color Removal, Nanoparticles, Water*

## 1. Introduction

Bangladesh, a developing country, has been industrialized over the last two decades in various sectors. Thousands of textiles in Bangladesh use a huge amount of water, chemicals, and dyes in the finishing and dyeing processes. Particularly, synthetic dyes are extensively used in various branches of the textile industry (Islam and Mostafa, 2018b; Slama et al., 2021), the leather industry (Chowdhury et al., 2015; Monira et al., 2023), the food industry ( Zahedi et al., 2020), and so on, producing a huge volume of dyeing effluents. In textiles, 93% of the raw water, used for production, comes out as strongly colored wastewater due to dyes containing high chemical oxygen demand (COD), biological oxygen demand (BOD), a large number of suspended solids, total dissolved solids (TDS), extreme pH, concentrated organic compounds, and heavy metals (Hossen and Mostafa, 2023; Wijannarong et al., 2013). Crops, grown on polluted soils, may cause trace metals to be absorbed and, subsequently, accumulated along the food chain posing potential threats to animal and human health (Dahnoun and Djadouni, 2020). More than 70,000 tons of approximately 10,000 dyes and pigments are produced annually worldwide, of which approximately 20-30% are discharged as effluents from the curing and finishing processes of textiles (Patil and Shrivastava, 2015). About 20–30% of the 10,000 dyes and pigments, used in the textile industry, are reactive dyes, which include azo, anthraquinone, phthalocyanine, formazine, oxazine, etc. (Islam and Mostafa, 2022; Papić et al., 2004). The overwhelming majority of synthetic dyes exhibit a considerable structural diversity, and most of them are azo derivatives. Azo dyes represent about 60% of all reactive dyes utilized by the textile industry. Due to the presence of carcinogenic compounds such as naphthalene, benzamine, and other aromatic compounds, the dye effluent becomes toxic even at its lower concentrations (Gil et al., 2011). The exhaustion properties of azo dyes are poor, and they obstruct light penetration, interrupt photosynthetic movement, and prevent the growth of biota (Tareque et al., 2023; Islam and Mostafa, 2018a). The color of the effluents is aesthetically unpleasant to aquatic bodies. It hinders the oxygenation ability of water, disturbs the whole aquatic ecosystem and food chain, and poses a thoughtful risk to human health. The problem is even more acute in developing countries like Bangladesh, where rapid population growth and industrialization have increased the complexity of wastewater (Rahim and Mostafa, 2021; Saha el al., 2021; Ntuli et al., 2011; Qasim and Mane, 2013).

Hence, there is an urgent requirement for the development of innovative, but low-cost techniques, by which dye molecules can be removed. Many techniques, including coagulation-flocculation, electro-coagulation, adsorption, ion exchange, advanced oxidation processes ($O_3/H_2O_2$, $H_2O_2$/UV, and photo-catalysis), flotation, membrane techniques (ultra-filtration, nanofiltration, and reverse osmosis), ozonation, radiolysis, and biological degradation, have been successfully used to remove the color from wastewater (Sayed and Mostafa, 2021, 2023; Deng and Zhao, 2015; Fan et al., 2008; Mostafa and Hoinkis, 2012). However, due to

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some drawbacks of the aforementioned single process, as wastewater contains a complex mixture of variable contents, conventional techniques could not be implemented at scale in the industry. The C-F technique, coupled with adsorption, offers high efficiency of dye removal, less produced sludge, coagulant savings, and economic feasibility (Papić et al., 2004). Various adsorbents, such as montmorillonite, bentonite clay, nanoparticles, petroleum wastes, tannin-rich materials, sawdust, fly ash, sugar industry wastes, chitosan, peat moss, scrap tires, etc. (Kandisa et al., 2016) and agricultural unused materials include banana peel (Annadurai et al., 2002), rice husk (Malik, 2003), date pit (Banat et al., 2003), almond shell (Ardejani et al., 2008), etc. are being studied for removing of color, COD, and BOD at changed operational settings (Rafatullah et al., 2010). Among the various adsorbents, nanoparticles have drawn great attention due to their outstanding physicochemical properties, including small size, larger surface area, and magnetic properties. Adsorption by nanoparticles is a promising and attractive alternative for the treatment of azo-containing dyes and is inexpensive and readily available. The cost-effectiveness, higher environmental stability, higher adsorption capability, and nontoxic nature of IONPs make them suitable for wastewater treatment (Crane and Scott, 2012). Another exclusive property that favors using IONPs in the field of adsorption is magnetism, which makes the separation process easier. Therefore, IONPs can be easily separated with the help of a magnetic field after the adsorption. Several chemical methods can be used to synthesize IONPs, such as micro-emulsions (Salvador et al., 2021), hydrothermal synthesis (Ge et al., 2009), thermal decomposition (Unni et al., 2017), co-precipitation (Al-Alawy et al., 2018), sol-gel method (Kayani et al., 2014), and the colloidal chemistry method (Krans et al., 2020). Among these, chemical co-precipitation is probably the simplest and most promising method for the production of nanomaterials, as the procedure is relatively simple and a large amount of controlled particle size of IONPs can be synthesized (Al-Alawy et al., 2018). The study synthesized IONPs by co-precipitation of ferric and ferrous ions in an alkaline solution at 80 °C, and, then, it was used for the removal of color, COD, and BOD from textile dyeing effluent. The study aimed to investigate the feasibility of a coagulation process coupled with nanoparticle adsorbents to achieve a higher color removal efficiency in treating textile dye effluents.

## 2. Materials and Methods

### *2.1 Sample collection and analysis for characterization*

The textile dyeing effluent was collected in a five-liter pre-washed plastic container from Sirajganj District in Bangladesh. Before sampling, the containers were washed with diluted acid and double distilled water, and just before sampling, they were rinsed with the effluents to be sampled. pH, dissolved oxygen (DO), and electrical conductivity (EC) were measured on the spot by a portable multimeter. A few mL of concentrated $HNO_3$ and HCl were added to the effluent to prevent the growth of microbial bacteria, and the containers were sealed to prevent air oxidation. The effluents were analyzed before and after treatment using different analytical techniques, including titration, gravimetric, and spectrophotometric methods. Some of the physicochemical parameters were determined as per standard methods (APHA, 2012).

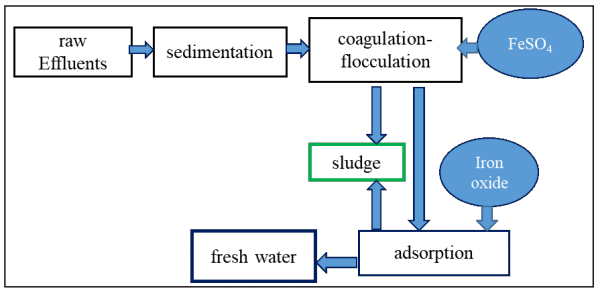


**Figure 1.** Flow chart of the experiment

### *2.2 Coagulation-flocculation experiments*

Figure 1 shows a simplified experimental setup that describes different stages of C-F and adsorption processes. The collected effluents were kept overnight for sedimentation. It was then placed in the jar test apparatus for C-F experiments, and the supernatant, obtained after C-F, was subjected to the adsorption process to get fresh water. The C-F process was conducted to obtain optimal process parameters, such as, coagulant doses (100 mg/L to 1200 mg/L), pH (3 to 12), temperature (25 to 50 °C), contact time (15 to 180 min.), and mixing speed (30 to 180 rpm). Each beaker was filled with the desired volume of effluent and added the desired amount of coagulant dose. The solution was stirred rapidly for 1.5 min. to ensure complete dispersion of the coagulants, followed by slow mixing to aid in the formation of flocs. The pH of the sample was adjusted in the range of 3-12 with 0.01 M HCl and/or 0.01 M NaOH solution before being subjected to the jar test. At the end of the sedimentation period, the supernatant was collected from the top of the beaker and the absorbance was measured using UV-visible spectrophotometer (SHIMADZU UV-mini1240) of the treated effluents at its $\lambda_{max}$. All experiments were conducted thrice and mean values were taken. The color removal percentage was calculated using the following equation (Hoong and Ismail, 2018):

$$\text{Color removal (\%)} = \frac{A_0 - A_f}{A_0} \times 100 \quad (1)$$

where $A_0$ and $A_f$ are the initial and final absorbance of the untreated and treated effluents, respectively.

### *2.3 Batch adsorption experiments*

After C-F experiments, batch adsorption studies were carried out at different doses (0.5 to 3.0 g/L) of synthesized IONPs adsorbent, keeping other parameters constant. Then, the adsorption of color was studied at pH values of 2.0 to 12.0 at different contact times (10, 15, 20, 30, 45, 60, 90, 120, and 180 min.) at 30 °C to get the optimum values. The experimental procedure is as follows: 100 mL of effluent was taken in 250 mL beakers loaded with a definite dose of IONPs after adjusting the pH of the effluent with 0.01 M HCl and/or 0.01 M NaOH solutions. The solution was stirred vigorously in an electric shaker at 180 rpm for a predefined time, and the beaker was, then, placed on the magnet. The effluents became colorless due to the magnetic separation of dye-loaded adsorbents. The mixture was decanted to measure its concentration with a UV-visible spectrophotometer by

measuring the absorbance at a wavelength corresponding to the maximum absorbance of the sample. All batch adsorption studies were conducted three times, and mean values were taken. The color removal efficiency of the adsorption process was calculated using the same equation used in the coagulation process stated above.

### *2.4 Adsorption kinetics*

The pseudo-first-order equation and pseudo-second-order equation are used to illustrate the adsorption kinetics of the adsorbent surfaces. The pseudo-first-order equation can be expressed in the following form (Ho and McKay, 1998; Mostafa et al., 2011):

$$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303}t \quad (2)$$

where $q_e$ and $q_t$ are the amounts of adsorbate adsorbed (mg $g^{-1}$) at equilibrium and at time $t$ (min.) respectively, and $k_1$ is the pseudo-first-order rate constant (L $min^{-1}$).

The pseudo-second-order equation can be stated as the following (Ho and McKay, 1999):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t) \quad (3)$$

where $k_2$ is the rate constant of pseudo-second-order adsorption (g $mg^{-1}min^{-1}$).

## 3. Results and Discussion

### *3.1 Characterization of the effluents*

Table 1 shows the physicochemical characteristics of the effluent. The collected effluents were highly colored and aesthetically unpleasant, and the values of all the parameters exceeded the DoE-BD standard, indicating that the wastewater was extremely polluted. Due to its color and higher levels of pollutants, the wastewater cannot be disposed of in any treatment plants before a pre-treatment stage. The untreated wastewater discharge has potential threats to surface water quality, aquatic life, and the entire environment. Therefore, it is imperative to improve the treatment method to reduce the impact of the effluents on the environment. Hence, the study looked for ways to improve the treatment of dyeing effluents through coagulation with $FeSO_4$, coupled with adsorption on nanoparticles.

**Table 1.** Physicochemical characteristics of the effluents

| Parameters | | Color | pH | EC, µS/cm | TDS, mg/L | TSS, mg/L | DO, mg/L | COD mg/L | BOD mg/L |
|---|---|---|---|---|---|---|---|---|---|
| Results | Ef1 | pink red | 11.3 | 6240 | 4337 | 475 | 1.4 | 3732 | 1156 |
| | Ef2 | greenish black | 11.2 | 6082 | 4025 | 400 | 1.6 | 3480 | 1065 |
| DoE standard | | - | 6-9 | 1200 | 2100 | 150 | - | 200 | 50 |

### *3.2 Optimization of Coagulant*

#### *3.2.1 Effect of coagulant dose*

In the C-F process, the dose is one of the most important factors to consider. Essentially, a small dose or overdosing would result in poor flocculation. The effects of coagulant doses (100-1200 mg/L) on the removal of color were shown in Figure 2, which illustrates that for small dosages (especially below 500 mg/L), color removal was rather low, while for 700 mg/L dosages, the removal efficiency of color was around 73%. The color removal efficiency of $FeSO_4$ coagulant increased gradually with dose due to the higher positive charge and polymeric effects of $FeSO_4$ (Joo et al., 2007). Furthermore, the high concentrations (>700 mg/L) of the coagulant may confer positive charges on the particle surface (a positive zeta potential), thus re-dispersing the particles, resulting in a decrease in color removal (Patel and Vashi, 2015). A similar result was observed in the ferrous sulfate coagulant, which showed more than 90% color removal efficiency at a coagulant dosage of 1600 mg/L (Jindal et al., 2016).

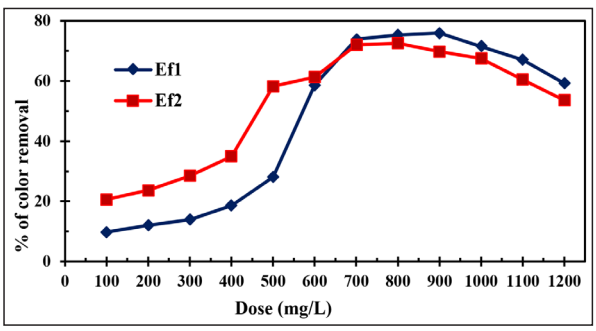


**Figure 2.** Effect of coagulant dose on color removal efficiency [pH 11.30, T 30 °C, contact time 30 min., and mixing speed 30 min.]

#### *3.2.2 Effects of pH*

One of the most important variables in the coagulation process using inorganic salts is the pH. This is because the inorganic coagulant is converted into different ionic species as the pH value changes, thus influencing the coagulation (Rodrigues et al., 2013). Figure 3 shows a decrease in percentage removal with an increase in pH for Ef1, but the percentage removal is increasing for Ef2 at pH ranges from 3 to 5. This is because, in the acidic region, Fe salt hydrolyzes to form monomeric Fe hydrolyzed species. Hence, soluble $Fe^{2+}$ and $Fe(OH)^+$ cations play an important role in destabilizing the negatively charged dye particles via charge neutralization, and thus color removal takes place at lower pH but too little because of the lack of formation of neutral $Fe(OH)_2$ precipitates (Wong et al., 2007). The formation of $Fe^{2+}$, $Fe(OH)^+$, and neutral $Fe(OH)_2$ hydrolysis species with large surface areas capable of adsorbing soluble dye is maximized at an alkaline pH, and color removal occurs via charge neutralization and sweep flocculation (Perng and Bui, 2014; Suman et al., 2018). The maximum color removal efficiency of $FeSO_4$ was about 75% at the optimum pH of 11.0. A study showed that each coagulant was effective in decolorization within a specific pH range, which depended strongly on the nature of the wastewater, and the color removals were 91.4% for cotton at pH 9.4 and 93.8% for acrylic effluents at pH 8.3, respectively, using $FeSO_4$ coagulant (Rodrigues et al., 2013). At another pH, the complexes of hydrolysis products caused a decrease in the removal efficiency (Perng and Bui, 2014).

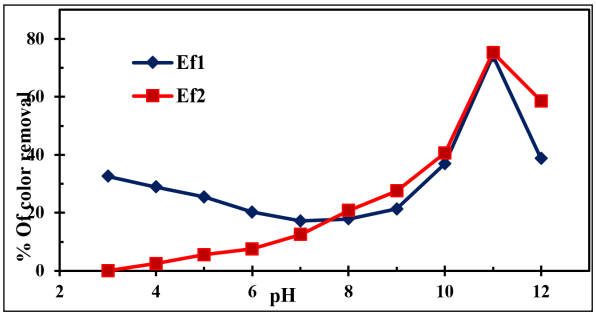


**Figure 3.** Effects of pH on color removal efficiency for Ef1 and Ef2 samples [coagulant dose 700 mg/L, temp 30 °C, contact time 30 min., and mixing speed 30]

*3.2.3 Effects of temperature*

Temperature affects the solubility of the metal hydroxide precipitate and the rate of formation of the metal hydrolysis products. Low temperature affects coagulation processes by altering coagulant solubility, increasing water viscosity, and retarding the kinetics of hydrolysis reactions. As shown in Figure 4, the effect of temperature on color removal is marked, increasing from 58% at 25°C up to 73% at 35°C for Ef1 and 68% to 76% for Ef2, and the % removal was not increased further up to 50°C, rather slightly decreasing for both effluents. This is because coagulation with hydrolyzing metals is less efficient at lower temperatures and has a pronounced detrimental effect on flocculation kinetics (Duan and Gregory, 2003). It has been reported that a decrease in temperature, (0-24°C), impairs the flocs› strength and virtually the flocs› formation efficiency, which results in a decrease in aggregation rate and bad settling (Fitzpatrick et al., 2004). The increased performance with temperature may be a consequence of the improved kinetics as occurs in most chemical reactions (Rodrigues et al., 2013). As the temperature increases from 30 to 35°C, the viscosity of the water decreases, and brown movement becomes fierce gradually as a result of the hydrolysis of the Fe(II) ion and increased competition for bonding by the macromolecules, and this accelerates the coagulation processes (Misau and Yusuf, 2016). The reasons behind decreased performance above 35°C were floc breakage increases, and floc reformation decreases at higher temperatures. Warmer temperatures generally produce bigger flocs that break more easily and reform less well, suggesting a weaker floc settlement. At high temperatures (above 35 °C), breakage in terms of floc size reduction is greater (Brabty, 2006).

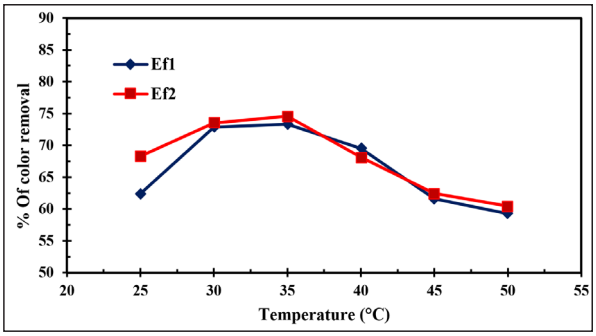


**Figure 4.** Effect of temperature on color removal efficiency for effluent Ef1 and Ef2 [coagulant dose 700 mg/L, pH 11.00, mixing speed 30 and settling time 30 min]

*3.2.4 Effect of contact time*

Contact time plays a vital role in flocs formation and growth in the flocculation process. The effect of contact time on the coagulation of dyeing effluents was studied using a time range of 15 min. to 180 min. and keeping other parameters constant. The trends that had been illustrated in Figure 5 showed that a longer or shorter contact time would result in poor performance of $FeSO_4$ for binding and bridging in the case of both effluents. In a short period, the collisions between the flocculants and colloids are not efficient to precipitate suspended solids. The color removal efficiency significantly dropped from 73% to about 56% with increasing reaction time from 30 min. to 180 min. for Ef1 and 73% to 53% for Ef2, respectively. This is because longer mixing times lead to an increase in floc breakage and limit the size of the flocs formed. The small-size flocs are not dense enough to settle down and thus, indirectly cause the sample to be turbid again (Hassan et al., 2009). This phenomenon is observed in Figure 5, which shows a lower percentage of reductions at longer contact times (i.e., 180 minutes). Similar results were reported as the restabilization phenomenon by Klimiuk et al. (1999).

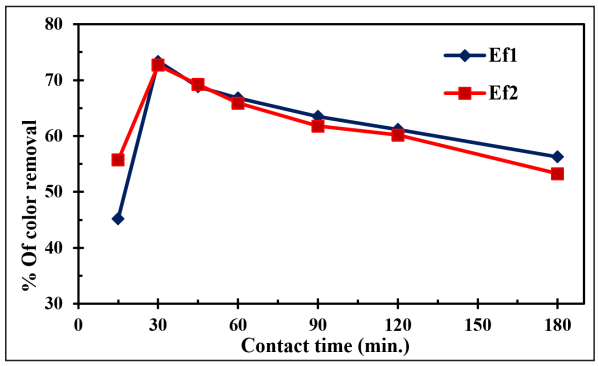


**Figure 5.** Effects of contact time on color removal efficiency for effluent Ef1 and Ef2 [coagulant dose 700 mg/L, pH 11.00, temp. 30 °C, mixing speed 30 and settling time 30 min.]

*3.2.5 Effects of mixing speed*

Mixing speed is one of the important factors in achieving higher color removal efficiency of the coagulants during the coagulation process. In this study, the effects of agitation speeds between 15 and 180 rpm were investigated. The results showed that the color removal efficiency of $FeSO_4$ increased with increasing mixing speeds between 15 and 45 rpm. These results might be caused by the fast flocs formation and high sludge precipitation rates of the inorganic coagulant. The color removal efficiency gradually decreased as the agitation speed increased. This trend showed that floc formation and breakage were intensely affected by the mixing rate (Xu et al., 2010). The highest color removal might be caused by increasing shear stress and breakage of flocs with increasing agitation speed. Figure 6 shows that the color removal efficiency was about 75% and 76% for Ef1 and Ef2, respectively, at an optimum mixing speed of 45 rpm. Similar results were observed in an experiment, which showed that about 74% of color removal was obtained by $FeSO_4$ for the cotton dyeing effluent (Xu et al., 2010).

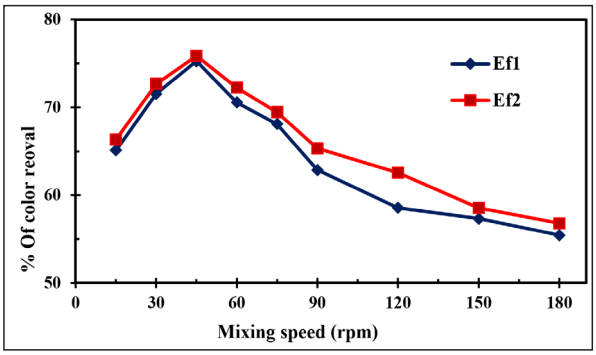


**Figure 6.** Effects of mixing speed on color removal efficiency for Ef1 and Ef2 [dose 700 mg/L, pH 11.00, temp. 30°C, and contact time 30 min.]

### *3.3 Adsorption followed by coagulation*

Adsorption can be an effective and versatile method for wastewater treatment, particularly when combined with chemical treatment methods. For better effluent treatment, optimal adsorption conditions must be determined. Several studies have illustrated that optimizing the parameters greatly influences the adsorption efficiencies of color and COD (Nayl et al., 2017; Nure et al., 2017; Wasti and Awan, 2016).

#### *3.3.1 Effects of adsorbent dose*

Figure 7 shows the effect of varying the adsorbent dose on the adsorption of color from effluents. The percentage of color removal increased with increasing doses from 1.0 to 3.0 g/L up to a maximum, after which increasing the dose of IONPs did not improve color removal. This is because the active sites could be effectively utilized when the dose was low. But at higher adsorbent dosages, it is more likely that a significant portion of the available active sites remains uncovered, leading to lower specific uptake (Patel and Vashi, 2010). The number of active sites for the sorption also increases by increasing the adsorbent dose, thus increasing the amount of adsorption. When the surface active sites are covered completely, the extent of adsorption reaches equilibrium (Zobayer et al., 2013). So, it is clear that equilibrium was attained after a dose of 1.5 g/L. The maximum removal efficiencies found were 94% and 95% for Ef1 and Ef2, respectively, at doses of 1.5 g/L for both effluents.

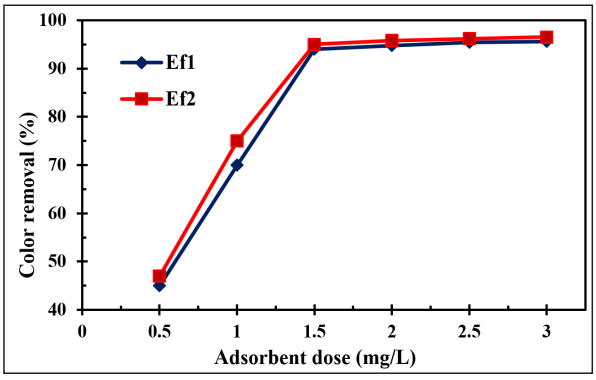


**Figure 7.** Effects of adsorbent dose on color removal efficiency (pH 7.0, contact time 30 min., temp. 30°C, and mixing speed 180 rpm)

#### *3.3.2 Effects of pH*

pH is an important parameter that affects the adsorption of dye molecules. The effect of the initial pH of the textile effluents on dye adsorption onto IONPs was assessed at different pH values, ranging from 2 to 12, with an optimum dose. Figure 8 shows that the maximum color removal was obtained around the neutral pH. Nevertheless, at optimum pH 6.0, more than 95 % and 96% of the color for Ef1 and Ef2, respectively, was removed by IONPs. The results showed that the adsorption was favorable in both acidic and basic environments. The electrostatic interactions between the nanoparticle surface and the functionalized dye molecule play a crucial role in the adsorption of dyes by IONPs. These interactions are greatly influenced by the pH of the sample, as it directly affects the surface charge of the nanoparticles. As the pH of zero-point charge (pHpzc) of IONPs is around 7.5 (Nassar and Ringsred, 2012), the adsorption of ionized dye on iron oxide surfaces could be due to electrostatic attraction. The surface charge is positive at pH values lower than pHzpc, neutral at pHzpc and negative at pH values higher than it. Therefore, in a relatively basic solution, pH >pHpzc, there is a high electrostatic attraction between the negatively-charged iron oxide and the positively-charged dye molecules. In contrast, as the pH of the solution falls, the proportion of positively charged sites rises while the proportion of negatively charged sites falls. This led to increased negatively charged dye adsorption but a decrease in positively-charged dye adsorption (Nassar et al., 2015). Simply to say, lower pH favors anionic dye adsorption and higher pH cationic dye adsorption and vice-versa (Salleh et al., 2011). In this study, the adsorptive dye removal was not significantly affected by solution pH, which suggests that the dye present in the textile wastewater sample might be multifunctional, and accordingly, it could be adsorbed in either basic or acidic environments (Nassar et al., 2015).

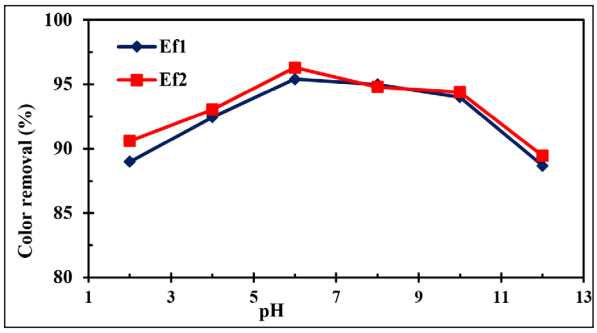


**Figure 8.** Effects of pH on the color removal efficiency of adsorbent (dose 1.5 g/L, contact time 30 min, temp. 30°C, and mixing speed 180 rpm)

#### *3.3.3 Effects of contact time*

Figure 9 shows the adsorption of color by IONPs as a function of contact time from 05 to 180 minutes at an adsorbent dose of 1.5 g/L at 30 °C for both effluents at pH 6.0. As seen, the adsorption was very rapid at the initial stages and then approached equilibrium just after 45 minutes i.e., the adsorption capacity increases with the increase in contact time until equilibrium is reached (Al-trawneh, 2015). This may be due to the larger surface area of IONPs at the very beginning of color adsorption. As the adsorption sites are depleted, the rate at which the dye molecules are

transported from the exterior to the interior sites of the adsorbent particles controls the dye uptake rate (Patel and Vashi, 2010). At equilibrium, about 96% and 98% of color were removed from Ef1 and Ef2, respectively (Figure 9). The relation between the removal of dyes and contact time was studied by (Hashemian et al., 2013), where adsorption increased with an increase in contact time. It was found that more than 75% of the dye removal occurred in the first 45 min., and thereafter the rate of adsorption was found to be slow. The rapid adsorption was due to the availability of the surface porosity of the adsorbent, which led to fast adsorption of dyes, the later slow rate of adsorption is due to the slow pore diffusion of the solute ion into the bulk of the adsorbent. El-Sayed et al. (2014) studied that the removal rapidly increased in the first 10 min. but then slowly increased to reach equilibrium because of the strong attraction forces between methylene blue dye and the adsorbent.

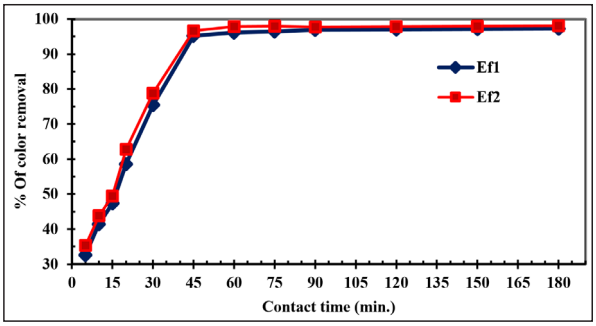


**Figure 9.** Effects of contact time on color removal efficiency for Ef1 and Ef2 (dose 1.5 g/L, temp. 30°C, and mixing speed 180 rpm)

### *3.4 Adsorption kinetics*

The experimental data were fitted to the pseudo-first-order and pseudo-second-order equations as shown in Figure 10 and Figure 11. The higher $R^2$ values of the experimental data (greater than 0.99) and good agreement of the calculated $q_e$ values with that of the experimental qe values at 30 °C (Table 2), recommended that the adsorption procedure followed a pseudo-second order kinetic reaction mechanism (Jafar et al., 2015).

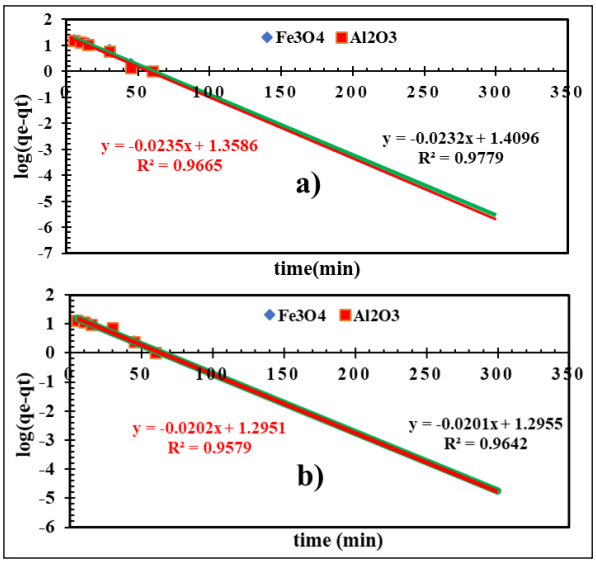


**Figure 10.** Pseudo -1st order kinetic plots of adsorption for textile dyeing effluents a) Ef1, and b) Ef2 onto IONPs

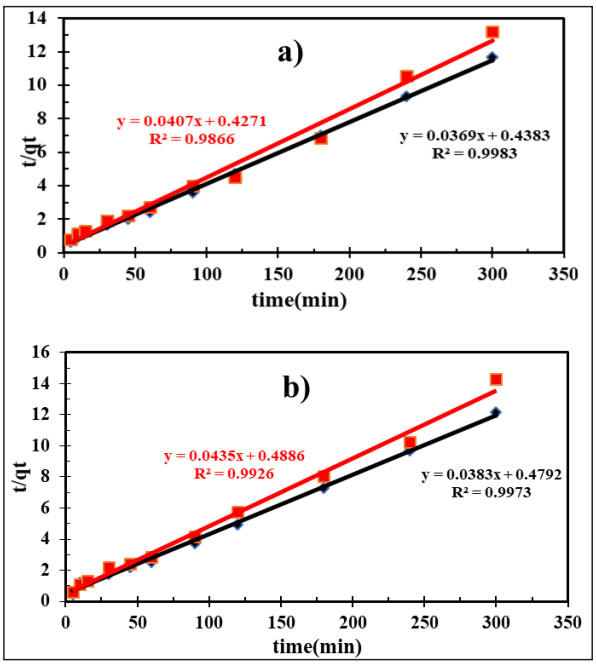


**Figure 11.** Pseudo -2nd order kinetic plots of adsorption for textile dyeing effluents a) Ef1, and b) Ef2 onto IONPs

**Table 2.** Kinetic parameters calculated by pseudo-first-order and pseudo-second-order equations for dyes adsorbed on IONPs

| Parameters | Pseudo-first-order kinetics | | Pseudo-second-order kinetics | |
|---|---|---|---|---|
| | Ef1 | Ef2 | Ef1 | Ef2 |
| Rate constant | 0.0534 | 0.0462 | $3.1210^{-3}$ | $3.0610^{-3}$ |
| $q_e$ cal. (mg/g) | 25.68 | 19.74 | 27.70 | 26.11 |
| $q_e$ exp. (mg/g) | 24.86 | 23.85 | 25.68 | 19.74 |
| $R^2$ | 0.9779 | 0.9642 | 0.9983 | 0.9973 |

### *3.5 Comparative studies*

It has been reported by different researchers that the percentages of color removal were in the range of 6 to 74.5, using various inorganic coagulants such as $MgCl_2$, $FeSO_4$, lime, PAC, and ferric sulfate as shown in Table 3, even though the dosage was high. Alum removed about 74% color but the dosage was too high. In the present study, $FeSO_4$ coagulant proved effective as it removes 75.3%, and 76.0% color from real textile effluents Ef1 and Ef2, respectively. It indicates that different coagulant was suitable for the treatment of various types of the textile dyeing effluents.

Table 4 compares the maximum dye adsorption capacity of IONPs and other adsorbents published in the literature. In comparison, it is clear that the maximum adsorption capacity of IONPs is superior to most of other adsorbents. Although the adsorption capacity of Palladium nanoparticles AC and alumina nanoparticles is also high, the use of synthetic IONPs is much more economical than other adsorbents due to their large surface area, economic feasibility, and small diffusion resistance (Afkhami et al., 2010). Moreover, the adsorbed dye can be easily desorbed to free the adsorbent, which could be further used to treat wastewater. This suggests that the adsorption property of IONPs gives the material great potential for applications in color and COD removal from wastewater.

**Table 3.** Comparison of ferrous sulfate with different coagulants in color removal from textile effluents

| Coagulant | Dyes / Effluents | Dose (mg/L) | % of color removal | Reference |
|---|---|---|---|---|
| $MgCl_2$ | Effluents | 2000 | 62 | ( Mohamed et al., 2014) |
| $FeSO_4$ | textile wastewater | 200 | 6 | (Patabandige et al., 2020) |
| Lime | Textile effluent | 700 | 53.9 | (Zorpas et al., 2012) |
| PAC | Textile wastewater | 30 | 44.5 | (Bazrafshan et al., 2015) |
| $Fe_2(SO_4)_3$ | Textile wastewater | 70000 | 57.9 | (Patel and Vashi, 2010) |
| Alum | Dyeing wastewater | 5000 | 74 | (Kumar et al., 2008) |
| Alum | Textile wastewater | 70000 | 74.5 | (Patel and Vashi, 2010) |
| $FeSO_4$ | Dyeing effluent (Ef1) | 700 | 75.3 | This study |
| $FeSO_4$ | Dyeing Effluent (Ef2) | 700 | 76.0 | This study |

**Table 4.** Comparison of iron oxide nanoparticle and other adsorbents for dye removal from the dyeing effluents

| Adsorbent | Dyes | Maximum adsorption capacity, (mg/g) | References |
|---|---|---|---|
| Palladium nanoparticles AC | Congo red | 126.58 | (Ahmadi et al., 2015) |
| $TiO_2$/GMAC | Reactive Black 5 | 56 | (Belayachi et al., 2015) |
| Bentonite clay | Reactive Black 5 | 29.38 | (Amin et al., 2015) |
| Kaolin | Procion brilliant red | 3.51 | (Rahman et al., 2013) |
| Sepiolite | Reactive Yellow 138:1 | 3.23 | (Rahman et al., 2015) |
| Alumina nanoparticle | Color black G | 263.16 | (Bhargavi et al., 2015) |
| Sawdust Carbon | Methylene blue | 12.49 - 51.4 | (Salleh et al., 2011) |
| Ficus Carica Bast AC | Methylene blue | 30-45 | (Pathania et al., 2017) |
| Magnetite nanoparticle | Mixture of dyes (Ef1) | 208.3 | This study |
| Magnetite nanoparticle | Mixture of dyes (Ef2) | 39.2 | This study |

### *3.6 Economic feasibility study*

The study selected the best coagulant and adsorbent in terms of color and COD removal efficiency and compared with other treatment methods in terms of cost-effectiveness, as shown in Table 5. The economic feasibility study mainly focused on the total costs of chemical reagents. The main chemicals used in the treatment processes were $FeSO_4$ for the C-F process and synthesized $Fe_3O_4$ nanoparticles for adsorption. The quantity of coagulant and adsorbent required for 1 $m^3$ was calculated from the quantity of coagulant and adsorbent required for 1 L of effluent. The operating cost was calculated based on the recent market price in December, 2023. In comparison with other research, the present study achieved the highest percentage of color removal with the lowest cost.

**Table 5.** Comparison of the treatment cost with others

| Sample | Coagulant + adsorbent | Amount ($Kg/m^3$) | % of color/COD removal | Cost ($/$m^3$) | Reference |
|---|---|---|---|---|---|
| Effluent | $Fe_2(SO_4)_3$ + Activated carbon | 70 + 9 | 71.4 | 86.5 | (Patel and Vashi, 2010) |
| Textile effluents | $Al_2(SO_4)_3$ | 0.6 | 93.12 | 0.75 | (Couto Junior et al., 2013) |
| Effluent | $Al_2(SO_4)_3$ + Activated carbon | 1 + 1 | 48.20 | 1.960 | (Mukherjee, 2014) |
| Ef1 | $FeSO_4$ + IONPs | 0.6 + 2.5 | 99.0 | 0.45 | This study |
| Ef2 | $FeSO_4$ + IONPs | 0.6 + 2.5 | 99.5 | 0.45 | This study |

## 4. Conclusion

The study used FeSO4 as a coagulant and synthesized IONPs as adsorbents to determine the effectiveness of these materials for removing color from dyeing effluents. The IONPs were synthesized by a simple co-precipitation method controlled by the pH and temperature of the reaction media. The X-RD analysis results confirmed that the synthesized iron-oxide particles were found to be within the nano-size range (10–20 µm). The optimal conditions for coagulation-flocculation (C-F) treatment were found at pH 11.0, coagulant dose 700 mg/L, reaction time 30 min, mixing speed 45 rpm, and temperature 30 °C, at which the color removal efficiency was about 75 %. The study showed that a combination of coagulation and adsorption on IONPs for the discoloration of textile effluents was more efficient than the coagulation-flocculation process alone. In the combination process, more than 99.5% color removal was achieved for both effluents. The SEM images showed the rough and porous surface of virgin IONPs having a slightly hazy and smooth surface for the adsorbed IONPs. The EDS analysis confirmed the presence of various metal ions on the IONPs surface. Finally, it can be concluded that the combination of chemical

coagulation and adsorption processes has the potential to remove color from textile wastewater.

**Conflict of Interest:**

The authors ensured that there was no direct or indirect involvement of human, animal, or any biological elements that have been tested that may arise any conflict of interest in this research. Moreover, the authors declare that they have no conflicts of interest in the subject matter or materials discussed in the manuscript.

**Acknowledgements**

One of the authors would like to thank the University Grants Commission, Bangladesh, for granting the UGC fellowship.

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