

## Efficiency Assessment of $\text{FeSO}_4$ Coagulant on Yarn Dyeing Industrial Effluent

Md. Abu Sayed<sup>1</sup> and M.G. Mostafa<sup>2</sup>

<sup>1,2</sup>*Institute of Environmental Science University of Rajshahi, Rajshahi 6205, Bangladesh*

**Abstract:** The yarn dyeing industry consumes large quantities of water and chemicals for the wet processing of yarn. The chemicals used are very diverse in chemical composition, ranging from inorganic and organic compounds to polymers. The major environmental problem of colorant manufacturing is the removal of dyes from effluents. The study aimed to investigate the efficiency of  $\text{FeSO}_4$  coagulants in removing color, chemical oxygen demand (COD), and turbidity from yarn dyeing industrial effluents. It optimized the parameters, including pH, coagulant dose, temperature, and contact time. The result showed that the pH and temperature were optimized at 11.0 and 35°C, respectively. The color, COD, and turbidity removal achieved were 92.35%, 82.44%, and 82.73%, respectively. The results indicated that  $\text{FeSO}_4$  showed the potentiality in removing yarn dyeing industrial effluents that reduced the values of color, COD, and turbidity within the Department of Environment, Bangladesh (DoE-BD) standards for discharging yarn-dyed wastewater.

**Keywords:** *Coagulation; COD; Color removal efficiency; Effluents; wastewater.*

**Introduction:** Yarn-dyed industrial effluent is one of recalcitrant and highly toxic wastewater [1]. Yarn-dyed wastewater is classified as a hazardous waste due to its high content of non-biodegradable organic dye molecules. The unused substances from the processes are discharged as wastewater that is high in color, COD, BOD, pH, temperature, turbidity, toxic concentrated organic compounds, and heavy metals [2]. The direct discharge of this wastewater into the water bodies like lakes, rivers, etc. pollutes the water reservoirs and affects the flora and fauna. Azo dyes are widely used as colorants because of their range of brilliant shades, different usage methods, and comparatively low cost [3]. The dyes, azo dyes in particular are harmful to humans and other living organisms owing to their toxicity, carcinogenicity, and mutagenicity [4]. The overwhelming majority of synthetic dyes currently used in the industry exhibit considerable structural diversity and due to high molecular weight and complex structures, they show very low biodegradability [5]. As wastewater discharge from a yarn dyeing industry does not meet the DoE-BD standard limit. Therefore, the disposal of yarn dye wastewater is an issue in densely populated areas like the Sirajganj District of Bangladesh. Hence, the wastewater must be treated before discharging into a surface water body.

Several methods have been reported for removing color from dye wastewater, although no technology used today has universal application. The most common techniques for the treatment of textile effluents are the Fenton process [6], coagulation [7], nano-particle adsorption [8], biodegradation [9], electrochemical degradation [10], adsorption process [11,12], ozonation, oxidation, chemical precipitation, ion-exchange, reverse osmosis, ultra-filtration, and advanced oxidation processes have been known to decolorize the textile effluents [13]. But coagulation is a common and widely used process due to its high efficiency and low cost, and it has been proved effective in dye removal, especially dissolved dyes in wastewater. The colloids are destabilized by coagulation techniques. Coagulation means the agglomeration of suspended particles by adding a chemical substance. It involves the reduction of surface charges and the formation of complex hydrous oxide. The most important surface phenomena are an accumulation of electrically charged particle surfaces [14]. In the coagulation process, electrolytic compounds such as ferrous sulfate in wastewater give hydrolyzable metallic ions that can eliminate the surface electrical charges of the colloids. A negatively charged colloid (dye molecule) is surrounded by the opposite charges called counter ions that come from coagulants [15]. The opposite charges will in turn be surrounded by charges opposite to them forming an electric double layer. When the concentration of ions is low the thickness of a double layer is larger. When concentration is increased colloid charge will be neutralized causing double-layer separation. In this way, the colloids can be settled. Normally the colloids (dyes) are negatively charged and coagulants are usually inorganic and

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**Corresponding author details:** M.G. Mostafa

E-mail address: [mghostafa@ru.ac.bd](mailto:mghostafa@ru.ac.bd)

Tel: +8801556336488

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organic cationic (positive) charged in the water. The study selected a common iron salt, i.e., ferrous sulfate as a coagulant in treating the yarn dyeing industrial effluents due to its low cost and high treatment efficiency.

Bangladesh is getting industrialized for the previous two decades but very few are using Effluent Treatment Plant (ETP) based on biological processes [16]. The process requires a huge amount of capital for its initial setup along with foreign technology. As a result, almost all the industries are releasing their effluent to the nearest water reservoirs as well as to the environment without considering any mitigative measures. Hence, considering the present needs, the study aimed at assessing the potentiality of the coagulant  $\text{FeSO}_4$  in reducing the toxicity of untreated yarn dyeing effluents. This study aimed to evaluate the efficiency of the  $\text{FeSO}_4$  coagulant in terms of color, COD, and turbidity removal for the yarn dyeing effluents.

## Materials and Methods

**Sample Collection:** The yarn-dyed industrial effluent was collected in a five-liter acid-washed plastic container from Sirajganj district of Bangladesh when the industries were in operational condition. Before sampling, the container was washed with diluted acid and double-distilled water, and just before actual sampling, the container was rinsed with effluents to be sampled. Dissolved Oxygen (DO), pH, and electrical conductivity (EC) were also measured by portable Water and Soil Analysis kit. A few mL of concentrated  $\text{HNO}_3$  and HCl was added to the effluent to prevent the growth of microbial bacteria and sealed to prevent air oxidation.

**Characterization of the Raw Effluent:** The collected samples were immediately brought to the laboratory and analyzed for some physicochemical parameters including pH, EC, turbidity, TSS, TDS, COD, BOD, and heavy metals. TDS and TSS of the effluents were measured by the gravimetric oven drying method at  $105^\circ\text{C}$ . BOD of the effluents was determined by incubating the sample at  $20^\circ\text{C}$  for 5 days followed by titration. COD was measured by the closed reflux method. The above-mentioned parameters were also measured after treatment with  $\text{FeSO}_4$  coagulant, following the same procedures. All above parameters were measured as suggested by American Public Health Association [17] and performed at room temperature. The analysis results of the effluent samples were evaluated following the norms prescribed by the DoE.

**Optimization of Coagulation Parameters:** The dyeing effluent samples were treated with  $\text{FeSO}_4$  coagulant (Analytical Reagent Grade) under optimized conditions. The coagulation experiments were conducted using a jar test apparatus containing six jars to optimize the process parameters (dose of coagulant, pH, temperature, time). Each of the six jars was filled with 0.5L of effluent and dosed with a coagulant. Different doses of  $\text{FeSO}_4$  coagulant (100-1200 mg/L) were added to the effluent samples. The experiments were conducted without adjusting the pH. Then, the influence of pH for color and COD removal were studied at an optimized coagulant dose. The examined pH range was between 3.0 and 13.0 and the pH was adjusted using 0.1 M HCl and 0.1 M NaOH.

After coagulant addition, the sample was stirred rapidly at 180 rpm for 90 seconds to ensure complete dispersion of the chemicals, followed by slow mixing (45 rpm) for 30 min to aid in the formation of flocs, and finally, the mixed solution was allowed to settle for 30 min. These procedures were performed several times so that the optimum coagulant dose, pH, temperature, and reaction time could be calculated for the best color and COD removal as well as turbidity decrease [18].

The characteristic wavelength for dye wastewater was determined by scanning the visible range using a UV-VIS spectrophotometer (SHIMADZU UV-mini1240). The maximum absorbance wavelengths ( $\lambda_{\text{max}}$ ) for the wastewaters were found at 530 nm. The supernatant was collected from the top of the beaker for the analysis [19], and the absorbance was measured. Percentage color removal was calculated by comparing the absorbance values of the wastewater before and after treatment using the following equation:

$$\% \text{ of color removal} = \frac{A_i - A_f}{A_i} \times 100 \quad (1)$$

Where  $A_i$  and  $A_f$  are the absorbance of the solution before treatment and after treatment of the effluent, respectively.

## Results and Discussion

**Physicochemical Parameters:** The physicochemical characteristics of the raw effluent are presented in Table 1. The EC value of the analysis sample was almost four times higher than the DoE standard indicating a large number of ionic elements present that increased the pollution level in the discharged effluents. The value of all the parameters in Table 1 exceeded the DoE standard indicated that the wastewater was highly polluted. Due to the high 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 [7]. The analysis results were compared with some literature values illustrating that the concentration of EC, TDS, and TSS was higher compared to the literature values indicating severe pollution (Table 2). Concerning, BOD and COD, the results showed a medium level of pollution compared to other literature values. Therefore, it is imperative to improve the treatment method to reduce the impact of the effluents on the environment. Nevertheless, we sought alternatives for optimization and improvement of the treatment through coagulation with FeSO<sub>4</sub> coagulant agents [20].

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

Parameters	Results	DoE standard
pH	11.30	6-9
Electric conductivity, $\mu\text{S cm}^{-1}$	4580	1200
Turbidity, NTU	32.44	-
Total Dissolved Solids (TDS), mg/L	2837	2100
Total Suspended Solids (TSS), mg/L	575	150
DO, mg/L	2.5	-
COD, mg/L	1054	200
BOD, mg/L	324	50

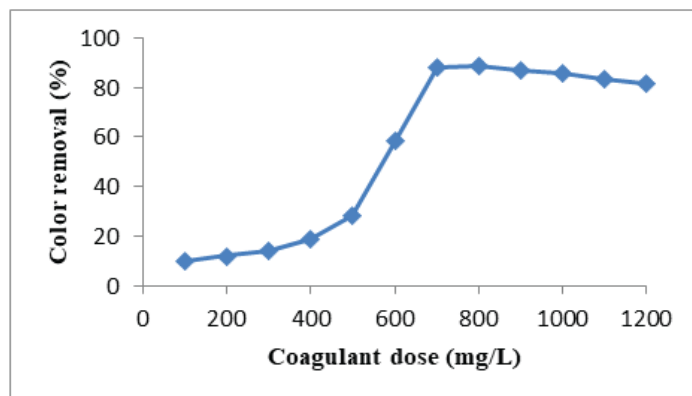
**Table 2:** Physicochemical characteristics of the dyeing wastewater from other researchers.

Physicochemical parameters							Reference
pH	Electric conductivity, $\mu\text{S cm}^{-1}$	Turbidity, NTU	Total Dissolved Solids (TDS), mg/L	Total Suspended Solids, mg/L	COD, mg/L	BOD, mg/L	
12.0	-	31.24	6620	9760	1638	-	[16]
10.21-11.53	-	-	250-2200	35-1200	1067-2430	163-645	[21]
8.2-11.4	3652-4380	-	-	306-442	1634-2020	547-687	[22]
8.66	-	81.5	242220	7116	3080	970	[23]
6.95-11.8	-	-	2900-3100	15-8000	150-30,000	80-6000	[24]
5.5-10.5	-	-	1500-6000	100-5000	150-10 <sup>4</sup>	100-4000	[25]
6-10	-	-	1800-6000	100-5000	150-10 <sup>4</sup>	100-4000	[26]
6.70-8.10	4150-5640	-	2937.5-3778	233-401	512-784	117-272	[27]
11.30	4580	32.44	2837	575	1054	324	This study

## Color Removal Efficiency

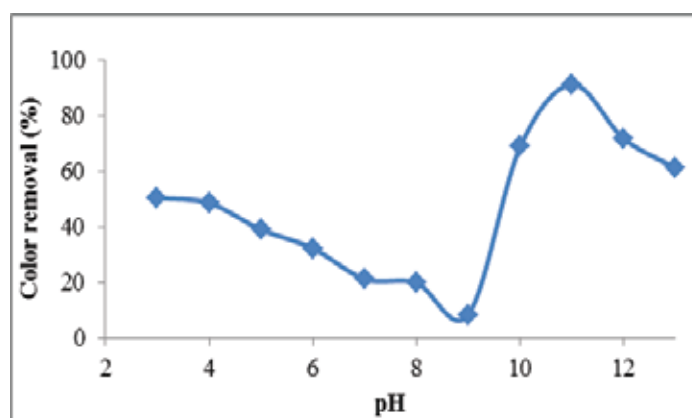
**Influence of Coagulant Dose:** Coagulation dose is one of the most important factors to consider in determining the optimum condition for the performance of coagulants. Essentially, a small or over-dosing would result in poor performance in flocculation. Therefore, it is important to determine the optimum dose to minimize the dosing cost and also the treatment performance. The effects of coagulant dose on the removal of color illustrated that the color

removal efficiency was increased with increasing the dose until it reached the maximum at a dose of 700 mg/L and then slowly decrease with the dose amount. The maximum removal achieved was about 88% (Fig. 1). The color removal efficiency of  $\text{FeSO}_4$  coagulant was increased gradually with dose due to high positive charged and polymeric effects of  $\text{FeSO}_4$  for dye removal [28]. Furthermore, the high concentrations (>700 mg/L) of the coagulant may confer positive charges on the particle surface (a positive zeta potential), thus redispersing the particles, resulting in a decrease in color removal [29]. A similar result was observed where  $\text{FeSO}_4$  gave more than 90% color removal efficiency at a coagulant dose of 1600 mg/L [30].



**Fig. 1:** Effect of coagulant dose on the color removal for yarn dyeing effluents (pH 11.30; contact time 30 min; temperature 30°C).

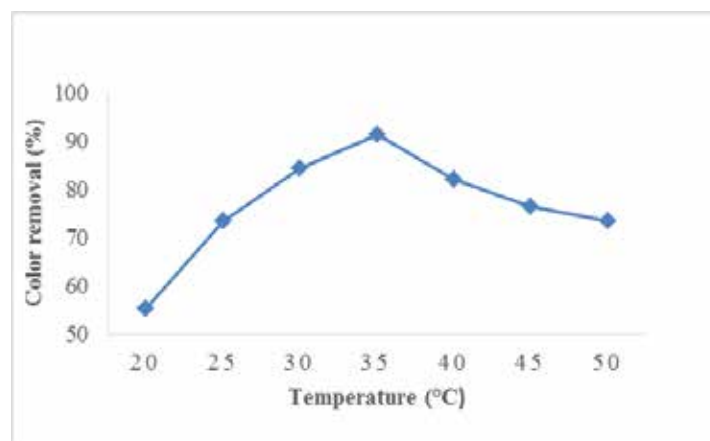
**Influence of pH:** One of the most important variables in the coagulation process using inorganic salts is pH, as the inorganic coagulant is converted into different ionic species as the pH value changes, thus influencing the coagulation [31]. Each coagulant is only effective in decolorization within a specific pH range which depends strongly on the nature of the wastewater to be treated. To examine the effect, the sample was adjusted to the desired pH (from 3 to 13) for each experiment by using 0.1 M NaOH (or HCl) solutions keeping other parameters fixed. Fig. 2 shows a decrease in the percentage of removal with an increase in pH. It is because the Fe salt was hydrolyzed to form a monomeric Fe hydrolyzed species. Hence, soluble  $\text{Fe}^{2+}$  and  $\text{Fe}(\text{OH})^+$  cations play an important role in destabilizing the negatively charged dye particles via charge neutralization [14]. However, at higher pH ranging from 9 to 11, the color removal percentage sharply increased and then decreased with increasing pH. It may be due to the formation of  $\text{Fe}^{2+}$ ,  $\text{Fe}(\text{OH})^+$ , and neutral  $\text{Fe}(\text{OH})_2$  species. These species are having large surface areas capable of adsorbing the maximum soluble dyes, and the color removal takes place by charge neutralization and sweeps flocculation [32-34]. The maximum color removal efficiency of  $\text{FeSO}_4$  was about 91% at a pH of 11.0. A similar study reported that about 98.5 % color was removed at pH 12.0 using  $\text{FeSO}_4$  [35], which supported the present findings. At another pH, the complexes of formed hydrolysis products caused the decrease of the removal efficiency [30].



**Fig. 2:** Effect of pH on color removal for optimum dose of coagulant (coagulant dose 700 mg/L  $\text{FeSO}_4$ , contact time 30 min and temperature 30°C).

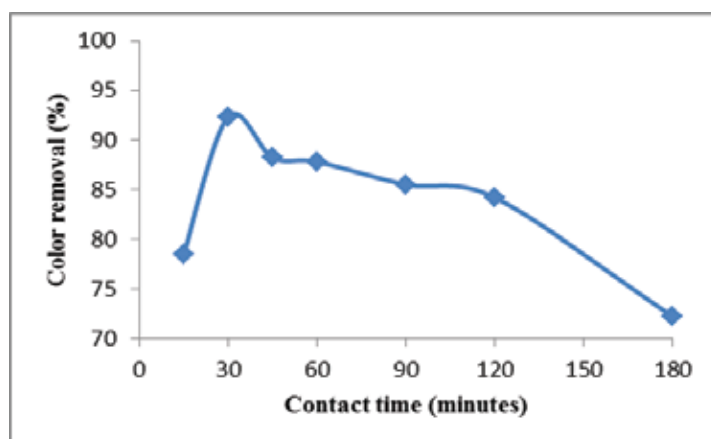


**Influence of Temperature:** Temperature affects the solubility of the metal hydroxide precipitate and the rate of formation of the metal hydrolysis products. Low temperature affects coagulation processes by altering coagulant solubility, increasing water viscosity, and retarding the kinetics of hydrolysis reactions. The temperature effects study results showed that the color removal percentage was increased with increasing temperature until it reached the maximum at 35°C. The color removal percentage in Fig. 3 shows an increase from 55% to 91% at a temperature ranging from 20 to 35°C and then decreased slowly with temperature. This is because coagulation with hydrolyzing metals is less efficient at lower temperatures and has a pronounced detrimental effect on flocculation kinetics [36]. A report showed that a decrease in temperature (0-24°C) impairs the flocs strength and virtually the flocs formation efficiency. It results in a decrease in the aggregation rate and bad settling [37]. The increased color removal performance with temperature may be a consequence of the improved kinetics, as occurs in most chemical reactions [31]. As the temperature increase (30 to 35°C), the viscosity of the raw water decreases, and brown movement becomes fierce gradually as a result of hydrolysis of Fe(II) ion and increased competition for bonding by the macromolecules and this accelerates the coagulation processes [34,38]. The reasons behind decreased performance above 35°C were: Floc breakage increases and floc recovery/re-formation decreases at a higher temperature. Warmer temperatures generally produce bigger flocs that break more easily and re-form less well, suggesting a weaker floc settlement. A study showed that the floc size was reduced at high temperature (above 35°C) [39].



**Fig. 3:** Effect of temperature on color removal for optimum dose of coagulant, and pH (coagulant dose 700 mg/L FeSO<sub>4</sub>, pH 11.0 and contact time 30 min).

**Influence of Contact Time:** Contact time plays a vital role in flocs formation and growth in the flocculation process. The effect of contact time on coagulation of dyeing effluents was studied using a time range from 15 min to 180 min and kept other parameters constant. The maximum color removal efficiency achieved was at 30 min of contact time. The color removal efficiency significantly dropped from 92% to about 72% with increasing reaction time from 30 min to 180 min. This was because a longer mixing time led to an increase in flocs breakage and reduced the size of the flocs formed. The trends illustrated in Fig. 4 showed that the longer or shorter contact time would result in the poor performance of FeSO<sub>4</sub> for binding and bridging. In a short period, the collisions between the flocculants and colloids are not efficient to precipitate suspended solids in the wastewater. The small sizes of flocs are not capable of settling down and thus, indirectly cause the sample to be turbid again [40]. Fig. 4 showed a lower percentage of reductions at longer contact time (i.e. 180 minutes). A similar observation was reported by Klimiuk that supported the present findings [41].

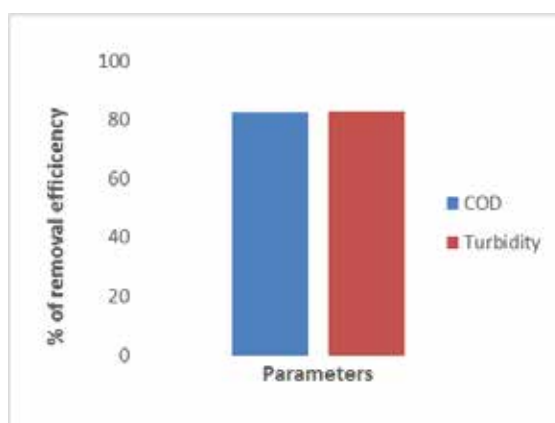


**Fig. 4:** Effect of contact time on color removal for optimum dose of coagulant, pH and temperature (coagulant dose 700 mg/L FeSO<sub>4</sub>, pH 11.0 and temperature 35°C).

**COD and Turbidity Removal:** COD is the amount of oxygen needed to oxidize the organic matter present in wastewater and turbidity related to color but, it has more to do with the loss of transparency due to the effect of suspended particles and colloidal material. Table 3 illustrates that the turbidity was 32.44 NTU, and COD was 1054 mg/L, found in the raw effluents, which was higher than the DoE standard limits, indicating a higher level of pollution. After treatment with FeSO<sub>4</sub> at optimized conditions, the reduction of COD was 82.44%, and that of turbidity was 82.73% (Fig. 5). The % removal of COD from dyeing effluents was observed almost similar as 75.2% [35], 78.6% [42], and 70% [43], treated with FeSO<sub>4</sub> coagulant. Therefore, FeSO<sub>4</sub> is an efficient coagulant for dyeing effluent treatment.

**Table 3:** COD and turbidity of the effluent before and after the treatment.

Parameters	Before treatment	After treatment	DoE std
Turbidity, NTU	32.44	5.60	-
COD, mg/L	1054	185	200



**Fig. 5:** Percentage removal of COD and turbidity from effluents after treatment with FeSO<sub>4</sub> coagulant.

**Sludge Management:** The treatment of wastewater brought about the amassing of toxins into sludge. Therefore, sludge becomes unstable, putrescible, and pathogenic and must be carefully handled the sludge for sustainable management. This study suggests that the sludge produced from the effluent treatment could be used directly in the brick-making process taking appropriate safety measures for the workers. Reports showed that about 6.66 % sludge was added to the bricks-making industries [44, 45]. Several incineration techniques were used to reduce the volume

and destruction of hazard elements in sludge. Further, the study observed that the oven-dried sludge was powdered and mixed with clay to make ceramic products and blocks as construction materials to quarantine the toxic elements in the sludge. In this way, large amounts of sludge could be used, thus minimizing the toxicity of the sludge in the environment.

**Conclusion:** The study investigated the characteristics of yarn-dyed wastewater and the efficiency of  $\text{FeSO}_4$  coagulant in the color removal, COD, and turbidity at optimized conditions. The study explored the following conclusive findings:

1) The textile wastewater was highly alkaline, and all parameters like EC, turbidity, color, and COD were found higher than the DoE standard guidelines for the effluent discharged.

2) The highest removal of color, COD, and turbidity was found to be 92.35%, 82.44%, and 82.73%, respectively, using a dose of 700 mg/l  $\text{FeSO}_4$  at a pH 11.0 and temperature of 35°C within 30 min contact time.

The results indicated that the coagulant  $\text{FeSO}_4$  showed potentiality in color, COD, and turbidity removal efficiencies. Finally, the treated effluent satisfied the DoE standards for discharging yarn-dried wastewater into surface waters.

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