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Removal of Turbidity and Congo Red Dye from Aqueous Solution by Coagulation-Flocculation Process

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Keywords:
Aluminum sulfate; Coagulation; Decolorization; Ferric sulfate; Red dye; Turbidity.

Highlights:

- *One of the most important methods of treating polluted water is chemical coagulation due to its low cost, availability of materials, and ease of application.*
- *The coagulant $Fe_2(SO_4)_3$ performance was better than (alum) in removing turbidity and color.*
- *Sedimentation time, mixing speed, and coagulant dose significantly affect the removal efficiency.*

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Abstract: Water pollution is currently one of the most serious problems facing humanity. In this regard, making this natural resource usable and unpolluted is of great social and economic importance. However, eliminating organic pollutants has received special attention to purify water waste. Chemical coagulation procedures are used by determining the optimal speed and dose of coagulants required to remove turbidity and dye from an aqueous solution while fixing pH and temperature to optimal values based on previous research. Aluminum sulfate (alum) and ferrous sulfate $Fe_2(SO_4)_3$ were used. Alum was found less efficient than $Fe_2(SO_4)_3$. It has been found that reducing turbidity was more effective than removing pigment. Maximum turbidity (NTU) and decolorization of (88% and 83%), respectively, were achieved with an optimum dose of 30 mg/L at pH 11, a settling time of 50 min, and a stirring speed of 120 rpm. Maximum turbidity (NTU) and color removal of (84% and 81%), respectively, were achieved at an optimum dose of 30 mg/L at pH 8, a settling time of 50 minutes, and a stirring speed of 120 rpm. On the other hand, physical and chemical technologies can be used as a rapid and cost-effective treatment method in terms of the availability of materials used compared to other treatment processes.

إزالة العكارة والصبغة الحمراء من المحلول المائي بعملية التخثر والتلبد

وديس يعقوب عذيب، علي جويد جعيل
قسم الهندسة المدنية/ كلية هندسة / جامعة واسط / واسط – العراق.

الخلاصة

يعد تلوث المياه حاليًا أحد أخطر المشاكل التي تواجه البشرية. وفي هذا الصدد، فإن جعل هذا المورد الطبيعي صالحًا للاستخدام وغير ملوث له أهمية اجتماعية واقتصادية كبيرة. ومع ذلك، فقد حظي التخلص من الملوثات العضوية باهتمام خاص لتنقية مخلفات المياه، حيث يتم استخدام إجراءات التخثر الكيميائي من خلال تحديد السرعة والجرعة المثلى لمواد التخثر اللازمة لإزالة العكارة والصبغة من المحلول المائي مع تثبيت الرقم الهيدروجيني ودرجة الحرارة إلى القيم المثلى بناءً على الأبحاث السابقة. تم استخدام كبريتات الألومنيوم (الشب) وكبريتات الحديدوز . لقد وجد أن الشبة أقل كفاءة من كبريتات الحديد. لقد وجد أن تقليل التعكر أكثر فعالية من إزالة الاصباغ. تم تحقيق الحد الأقصى من التعكر وإزالة اللون (٨٨٪ و ٨٣٪) على التوالي مع الجرعة المثلى البالغة ٣٠ مجم / لتر لكبريتات الحديد عند درجة الحموضة ١١، وزمن الترسيب ٥٠ دقيقة، وسرعة التحريك ١٢٠ دورة في الدقيقة. الحد الأقصى للتعكر وإزالة اللون (٨٤٪ و ٨١٪) على التوالي عند الجرعة المثالية البالغة ٣٠ مجم/لتر للشبه عند درجة حموضة ٨، وزمن استقرار قدره ٥٠ دقيقة، وسرعة مزج تبلغ ١٢٠ دورة في الدقيقة. ومن ناحية أخرى يمكن استخدام التكنولوجيا الفيزيائية والكيميائية كوسيلة معالجة سريعة وفعالة من حيث التكلفة وتوفر المواد المستخدمة مقارنة بعمليات المعالجة الأخرى.

الكلمات الدالة: التخثر، الصبغة الحمراء، العكارة، كبريتات الألومنيوم، كبريتات الحديديك، إزالة اللون.

1.INTRODUCTION

Effluents from the have recently become a major environmental issue. The textile industries' use of synthetic chemical dyes in various textile-manufacturing processes, such as dyeing, bleaching, and finishing, has eliminated huge amounts of colored industrial waste [1]. Over 10,000 professional dyes are utilized, and approximately 7 × 10^5 tons of artificial dyes are produced globally yearly [2]. Wastewater has a high concentration of suspended particles, variable pH, darker color, increased chemical oxygen demand and total organic carbon [3]. Wastewater, which is of high turbidity, contains high-density solid particles with strong color. Even at very low concentrations (less than 1 ppm for some dyes), these dyes cause a tint in water, are highly visible and undesirable, and negatively affect bodies of water such as lakes and rivers [4]. Since most dyes are toxic and biologically resistant, conventional biological methods for treating effluents are ineffective [3]. Therefore, untreated wastewater is highly dangerous and toxic to humans and animals due to the carcinogenic chemicals. Considering that the wastewater from textile dyeing works harms to the aquatic environment, it is critical to develop an ecologically friendly and energy-efficient technology for treating wastewater before it is released into the aquatic environment. Over the last decade, several traditional methods for treating contaminated water and numerous combinations of chemical, physical, and bio-oxidation processes have been developed [5]. However, these processes produce many chemical contaminants, increasing the total burden on the processing facility [5]. Recently, the cavity has garnered considerable attention as an advanced oxidation process (AOP) for wastewater cleanup [6]. Cavitation is the formation, growth, and eventual collapse of microbubbles or cavities in a brief period in many places in the reactor, resulting in of releasing a large amount of energy [7]. Ultra-sonication has been researched for effluent remediation over the last two decades [8]. However, because of high maintenance costs and low energy efficiency, it has yet to find industrial applications [6]. Due to the difficulties of decomposition and damage to human health, dyes have been designated serious industrial pollutants. It is critical to develop methods to remove these pollutants [9]. Coagulation is one of the most important procedures required to treat pollution. Water is required in all purification methods. Removing turbidity cause colloidal particles and grains, water mud, organic matter, depigmentation, COD, and other contaminants. The roof is made from manufacturing waste such as fabrics [10]. Chemical coagulation is done by adding coagulants and auxiliaries and is the most common method for removing contaminants. Sintering materials include alum, iron sulfate, PAC, iron chloride and others [11]. Chemical coagulation can be generated by electrostatic instability. When a chemical reagent is applied, it causes interactions between dye molecules and water [12]. Coagulation does not remove dye since it does not rely on the partial breakdown of dye components. Toxic and harmful intermediates are formed. Furthermore, due to its ease of usage and low cost, this method may be applied in large-scale operations [13]. Several studies used a chemical coagulation to remove colors and turbidity from aqueous solutions. Kim et al. [13], used ferric chloride ($FeCl_3$) for decolorization as a coagulant. Water is used to make dye solutions. The solution was mixed for 2 minutes at 250 rpm under quick mixing conditions, then for 15 minutes at 40 rpm following rapid mixing, then for 30 minutes to settle. Under ideal circumstances, the maximum removal efficiency for distributed blue and yellow pigments was 97.7% and 99.6%, respectively. To scatter blue, pH 6, 0.93 mM ferric chloride is used, and to scatter yellow, pH 5, 0.74 mM ferric chloride is used. The maximum removal efficiency for reactive blue and reactive yellow was 60.9% and 71.3%, respectively, below ideal coagulation conditions of pH 7- and 1.85-mM

ferric chloride for reactive blue. For reactive yellow, pH 6- and 2.78-mM ferric chloride were used. Asilian et al. [14] used coagulation to remove color and COD from wastewater. Experiments were conducted on samples containing 100 mg/L of acrylic water base color and 400 mg/L of acrylic water base color. To eliminate color, coagulation/flocculation techniques using ferrous sulfate, alum, lime, and poly electrolytes were used. Treatment with alum and ferrous sulfate alone was effective (>99%) in removing color. The experiment was conducted carefully to determine the optimal alum and $FeSO_4$ concentrations, pH, and temperature. Zhou et al. [15] used aluminum sulfate in the coagulation process for decolorization and COD removal of wastewater, and the results showed that the optimal coagulant dose increased with initial pH. Therefore, the appropriate initial pH should be higher than 7 to ensure effective removal of colorants. Aluminum and Salts [16] proved that turbidity removal was affected by pH, coagulant dosage, and initial turbidity. They found that the highest turbidity removal fell within 82-99.4% for initial turbidity of 10-1000 NTU at pH of 5-7 and coagulant dose of 10-20 mg/L. Abdulmajeed and Oleiwi [17] revealed that the coagulant dose, pH, and settling time were the most significant factors in turbidity reduction. They observed that turbidity could be reduced from 92 to 2.1 NTU at a pH of 6, a coagulant dose of 80 mg/L, and a settling time of 120 minutes. The present research aims to increase the efficacy of removing suspended particles and color, determine the appropriate doses under ideal conditions, and treat large volumes of polluted water.

2.EXPERIMENTAL PROGRAM

2.1. Apparatus and Procedures

2.1.1.Preparation and Description of Congo Red (CR)

In this research, the anionic pigment Congo red was used. Congo red has twice the molecular intricacy of azobenzene [18]. It is extensively used in manufacturing paper, plastics, leather, and most importantly fabrics [19]. Multiple studies have disclosed that, like many azo dyes, Congo red is hazardous, necessitating developing a feasible and effective way to neutralize this pigment in waste [20]. The major physicochemical properties of this hue are shown in Table 1 [21].

2.1.2.Chemicals

The chemicals used in this research were $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$ as coagulants for coagulation and sedimentation processes. All chemicals were of industrial quality and were used without further purification. 1M HCl or sodium hydroxide (NaOH) solution was used to change the pH. Table 2 shows the molecular weight and origin of the chemicals used in the present experiments.

Table 1 The Congo Red's Primary Physical and Chemical Features [21].

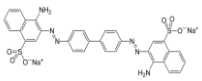
Name	Characteristics Summary	Chemical Structure
The Name of The Chemical	(CR)The Congo Red	
Appearance	Dark Red Powder	
The Structure of Molecules	$C_{32}H_{22}N_6\ Na_2\ O_6\ S_2$	
P Ka	> 4	
Λ max	A wavelength of 500 nm	
T°	> 360°	
Water	The Solubility is Quite High.	
Interests	Textile	

Table 2 Chemical Materials Used in the Present Experiments.

Chemical Compound	Molecular Weight (g/mol)	Country of Origin
Sodium Hydroxide (NaOH)	39.99	England
Hydrochloric Acid (HCl)	36.5	England
Iron (III) sulfate	399.88	Romil
Alum	190.45	BDH

2.1.3.Analytical Analysis

The pH, color, and turbidity of synthetic tissue fluids effluent samples were measured. WTW 340i/05281018 multimeter, made in Germany (a digital pH meter), was used to determine the medium pH. An instrument called a turbidimeter (COLE PALMER-USA/08391.45/12,106,698) was used to measure turbidity. Colorimetry was performed using a spectrophotometer (Spectro UV-Vis double bean PC UVD-3000, Germany). All the equipment used is in Civil Engineering Department/ Wasit University/ Environmental Laboratory. The proportion of removal effectiveness was determined using Eq. (1).

$$CR, NTU\ Removal\ (\%) = \left(\frac{C_o - C_f}{C_o}\right) * 100 \quad (1)$$

Where C_o denotes the starting concentration (in mg/L), and C_f denotes the final concentration (in mg/L). Color and turbidity (NTU) were assessed according to accepted practices. A 0.45 µm diameter membrane (47 mm) (NO. 5) filter was used to isolate the colorimetric supernatants from each beaker before determining the colorimetric and NTU at the maximum wavelength. The above algorithm was used to determine how well NTU and dye were removed [19].

- Removal with ferrous sulfate.

Equation (2) lists the fundamental formulae that apply to the aggregation of ferrous sulfate [22]:

$$Fe_2(SO_4)_3 + 3Ca(HCO_3)_2 \rightarrow 2Fe(OH)_3 + 3CaSO_4 + 6CO_2 \quad (2)$$

To maintain the ideal pH for aqueous solution treatment, variable $Fe_2(SO_4)_3$ content (10, 20 ,30, and 40 mg/L) was stirred using three rapid

mixing speeds (100, 110, and 120 rpm) for one minute. Then, it was slowly mixed at a rate of (40) rpm for 20 minutes. A jar test device (A.S.U, Pird, Phips) located in the Department of Civil Engineering / Wasit University laboratories, consisting of four graduated cylinders with a capacity of 1000 ml, was used to add the dye solution and oxidizing materials necessary for the experiment. The sedimentation times (30, 40, and 50 minutes) were used. $Fe_2(SO_4)_3$ concentrations of 30 mg/L were necessary for efficient NTU removal and color reduction.

- Removal with aluminum sulfate.

Aluminum ion, AL^{+3}, acts very similarly to Fe^{+3}. Equation (3) shows that Al (OH) $_3$ precipitates when aluminum sulfate is added to wastewater or water [22]:

$$Al_2(SO_4)_3 + 6\,CO_3 \rightarrow 2\,Al(OH)_3 + 3\,SO_4^{-2} + 6\,CO_2 \quad (3)$$

For aqueous solution treatment, similar to the $Fe_2(SO_4)_3$ experiment, the following conditions were applied: different aluminum sulfate concentrations (10, 20, 30, and 40 mg/L), three rapid mixing speeds (100, 110, and 120 rpm) for one minute, then at a slow speed of (40) rpm for 20 minutes. A jar test device (A.S.U, Pird, Phips) located in the laboratories of the Department of Civil Engineering / Wasit University laboratories, consisting of four graduated cylinders with a capacity of 1000 ml, was used to add the dye solution and oxidizing materials necessary for the experiment. Also, sedimentation times of (30, 40, and 50 minutes) were used, and pH at the optimum condition of 8 was used. An Alum concentration of 30 mg/L and fast mixing at 120 rpm for 1 minute were required for a successful NTU removal color concentration.

2.1.4.Production and Treatment of The Aqueous Solution

Sample solutions were prepared from 10.4 mg/L of dye concentration mixed with 1 liter of deionized water to achieve the best color intensities (10, 20, 30, and 40 mg/L). It is important to stress that under typical circumstances, the company's wastewater effluent includes about 10 mg/L [23].

2.2.Experiments with Coagulation and Flocculation

As shown in Fig. 1, a standard test jar setup was utilized for the coagulation and flocculation processes. Four (1-liter) beakers were filled with industrial effluent and transferred to jars. Using a Thermo-Fisher portable pH meter, samples were mixed in jars immediately at 120 rpm. A coagulant made of inorganic elements $Fe_2(SO_4)_3$ or (Alum) was added throughout the mixing procedure, and fast mixing continued for 1 minute at (100, 110, and 120 rpm). Then it settled for (30, 40, and 50 minutes) after 20 minutes of gradual mixing at 40 rpm. Following sedimentation, samples were collected for each beaker using a pipette 3-4 cm below the surface of the wastewater treatment. Temperatures ranging from 27 to 30° C were used for all test. The entire quantity of the finished dye solution was measured using a spectrophotometer after the coagulation-flocculation process was completed. A turbidity meter was used to estimate the NTU values.

Fig. 1 Congo Red Color and Turbidity Reduction Using Coagulants in a Jar System.

3.RESULTS AND DISCUSSIONS

3.1.Results

The results are listed in Tables 3 and 4, and samples of these results are shown graphically. Table 3 depicts the efficiency of removing turbidity and color from an industrial solution simulating textile water using an alum coagulant under optimal conditions using a coagulant dose of (10-40 mg/L), a variable sedimentation time of (30-50 minutes), and three mixing speeds (100, 110, and 120 rpm) with pH fixed at 8, which is similar Köse and Biroğul [24] results. The best removal rates were obtained at a dose of 30 mg/L and a time of 50 minutes. The rates for turbidity and color were 84% and 81%, respectively. These removal rates agree with [25, 26]. Table 4 depicts the efficiency of removing turbidity and color from an industrial solution simulating textile water using an alum coagulant under optimal conditions using a coagulant dose of (10-40 mg/L), a variable sedimentation time of (30-50 minutes), and three mixing speeds (100, 110, and 120rpm) with pH fixed at 11. The best removal rates were obtained at a dose of 30 mg/L and a time of 50 minutes. The rates for turbidity and color were 88% and 83%, respectively. The results agree with [27].

Table 3 Experimental Results of the Alum.

		Alum Removal Efficiency (%) pH (8), fast mixing 120 rpm											
		30 min				40 min				50 min			
		Alum Dose (mg/L)											
		10	0	30	40	10	20	30	40	10	20	30	40
Removal%	NTU	68	6	81	77	71	78	83	80	73	80	84	83
Alum Dose, (mg/L)	Color	60	3	78	72	61	75	79	73	63	79	81	74

Table 4 Experimental Results of $Fe_2(SO_4)_3$.

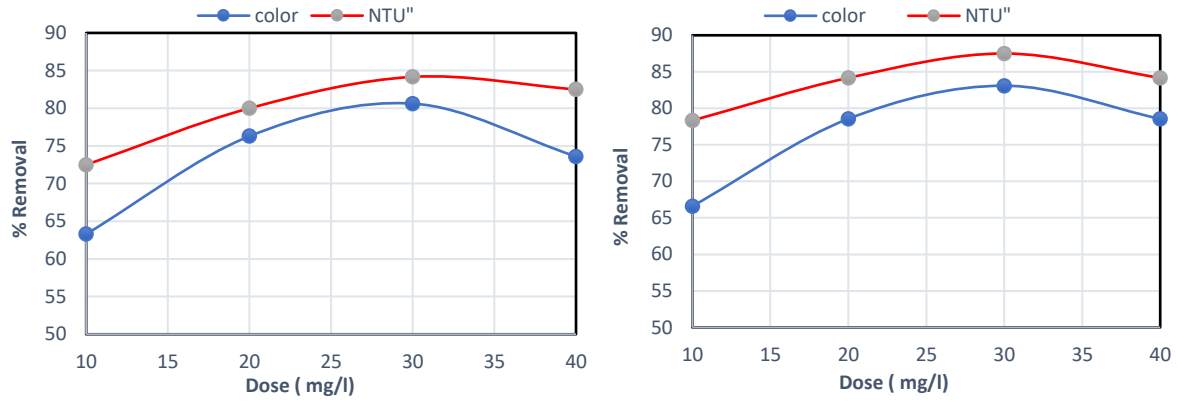
		$Fe_2(SO_4)_3$ Removal Efficiency (%) pH (11), fast mixing 120 rpm											
		30 min				40 min				50 min			
		Fe_2SO_4 Dose (mg/L)											
		10	20	30	40	10	20	30	40	10	20	30	40
Removal%	NTU	74	82	84	83	77	83	87	84	78	84	88	84
Alum Dose, (mg/L)	Color	62	74	81	73	65	77	82	76	67	79	83	79

3.2.Efficiency of $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$

Figure 2 shows the effect of the removal ratio upon adding alum and $Fe_2(SO_4)_3$ generated at pH (8 and 11), respectively, rapid mixing at 120 rpm, and sedimentation time of 50 min on the decolorization and turbidity efficacy. Figure 2 (a) depicts that decolorization and turbidity increased with the amount of alum added and when the alum dose was greater than 30 mg/L. The rate of decolorization decreased gradually. When the $Fe_2(SO_4)_3$ dose was higher than 30 mg/L, the decolorization decreased slowly and became constant, as shown in Fig. 2 (b). The effect of different alum and $Fe_2(SO_4)_3$ doses at pH (8 and 11), rapid mixing at 120 rpm, and settling time of 50 min on NTU removal efficacy was investigated. Eliminating of NTU increased with high alum and $Fe_2(SO_4)_3$ amounts. However, it is essential to note that a clear Congo red staining was associated with a high yield at very low anticoagulant doses. This behavior could be because the Congo red, i.e., negative charges, and the coagulant, i.e., positive charges, have a charge-attractive connection, enhancing the appearance of the block. Following the initial dosage, a lump figuration was noticed. Furthermore, the decreased output was linked to instability produced by excess coagulant. When plentiful, they cancel all positively charged particles, causing repulsive forces. As a result, the water will be extremely charged with poor coagulation and purity. Coagulation procedure for efficient color and NTU removal utilizing alum coagulant added in varied quantities of (10, 20, 30, and 40 mg/L), setting the pH value at 8, and quick mixing at different speeds of (100, 110, and 120 rpm) as a function of time, with sedimentation times of (30, 40, and 50 minutes) as shown in Fig. 3 (a, b). In addition, when the coagulant dosage increased, the clearance rate increased until it reached its maximum value. Following the ideal dosage, the elimination process began to slow. The decolorization efficiency and NTU increased with sedimentation duration, with the highest removal at 50 minutes and 30 mg/L being 84% and 81% for turbidity and color, respectively. These results are similar to [15]. Coagulation process for effective color and NTU removal using $Fe_2(SO_4)_3$ coagulant added in various amounts of (10, 20, 30, and 40 mg/L), pH adjusted to 11, and rapid mixing at various speeds of (100,110 and 120 rpm). Sedimentation periods of 30, 40, and 50 minutes are given as a function of time. Fig. 4 (a, b) shows an example. Furthermore, when the coagulant dose increased, so did the clearance rate until it reached its maximal amount. Similar behavior was reported in [28].

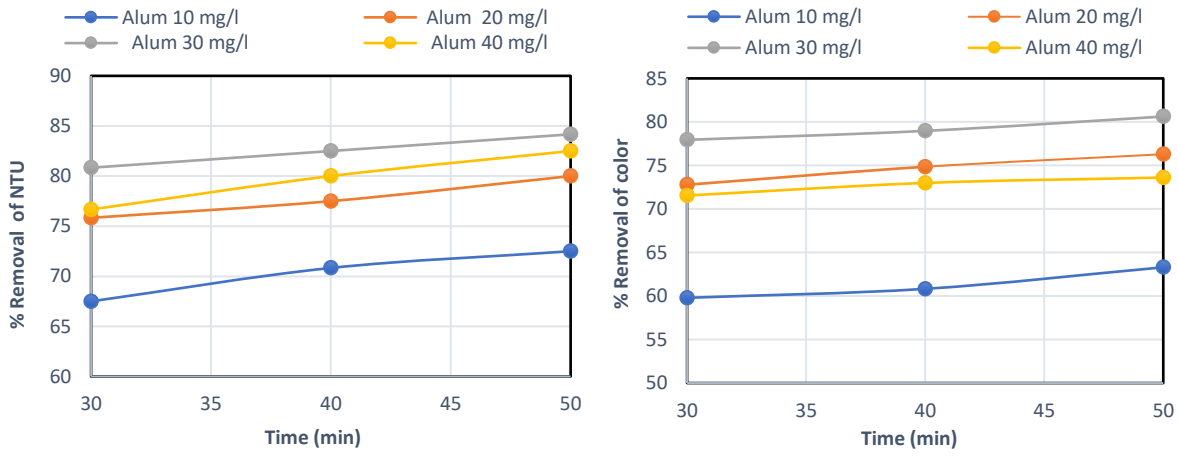
The elimination process began to slow after the appropriate dosage. The effectiveness of decolorization and NTU increased with sedimentation time, with the maximum removal at 50 minutes and 30 mg/L was 88% and 83% for turbidity and color, respectively.

Figure 5 (a, b) shows a comparison of the coagulant effect of in the mixing speed range (100, 110, and 120 rpm) as a function of settling time for (30, 40, and 50 minutes) of mixing time to evaluate the effect of mixing rate. The duration depends on the decolorization efficiency at pH 11 for $Fe_2(SO_4)_3$ coagulation and pH 8 for alum coagulation. With a deposition time of 30 min, raising the spin rate above 100 rpm became negligible. Likewise, mixing at 120 rpm resulted in a 2% increase in removal. More than 10 minutes, the settling time was insignificant. At 120 rpm and 50 min of deposition, the highest decolorization efficiency by alum and $Fe_2(SO_4)_3$ were 81% and 83%, respectively. In addition, the maximum turbidity removal efficiency was 84% and 88%, respectively, at 120 rpm and 50 minutes. Therefore, 120 rpm was the best speed for both coagulants. When all results were examined, $Fe_2(SO_4)_3$ outperformed alum. The color removal efficiency initially increased with the coagulant dose. However, after achieving its maximum, the color removal efficiency and NTU stabilized with increasing coagulant dose, indicating saturation. With pH 11, the color and NTU removal efficacy changed dramatically. The highest decolorization and NTU efficiencies were found for $Fe_2(SO_4)_3$ at pH 11, mixing at 120 rpm, and concentration time of 50 minutes which agrees with [27]. The highest decolorization and NTU efficiency were found at pH 8, mixing at 120 rpm, and concentration time of 50 minutes, which is similar to [24–26] results, which is also accurate for the semi-material, as shown in Fig. 6.



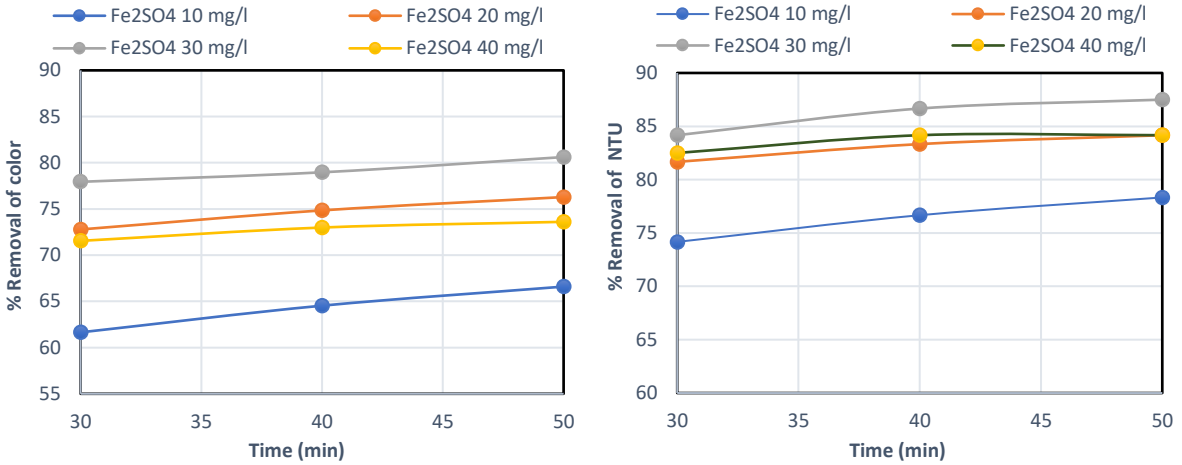
(a) (b)

Fig. 2 Effect of Dosing on Decolorization Efficiency and NTU Removal Efficiency (a) Alum and (b) $Fe_2(SO_4)_3$ at pH (8 and 11), Speed 120 rpm and Sedimentation Time 50 min.



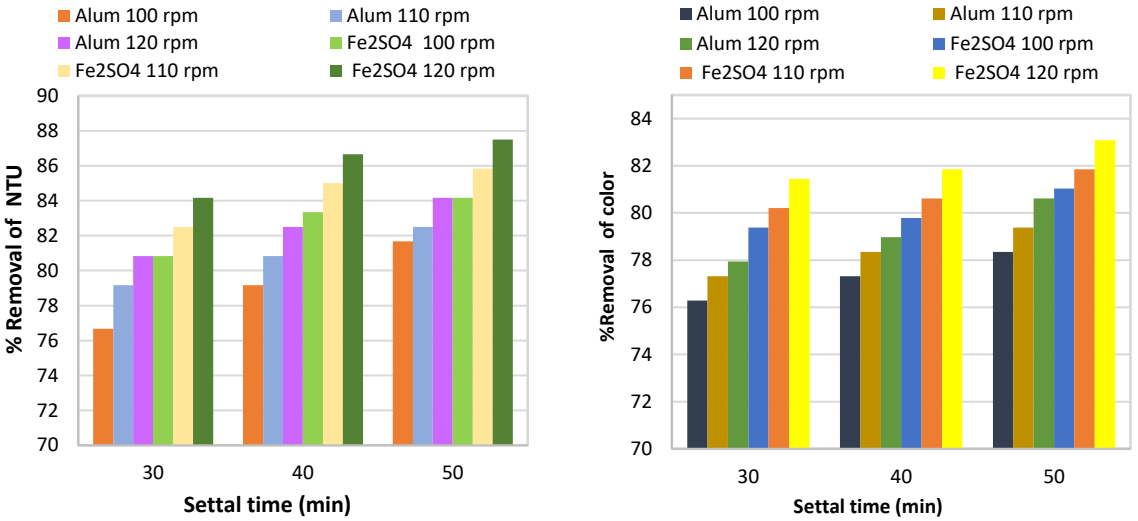
(a) (b)

Fig. 3 Effect of Alum Dosage on (a) NTU Removal Efficiency and (b) Decolorization Efficiency.



(a) (b)

Fig. 4 Effect of $Fe_2(SO_4)_3$ Doses on the Profiles of (a) Color Removal Efficiency, (b) NTU Removal Efficiency.



(a) (b)

Fig. 5 Comparison Effect of Fast Mixing on (a) NTU Efficiency and (b) Decolorization Efficiency.

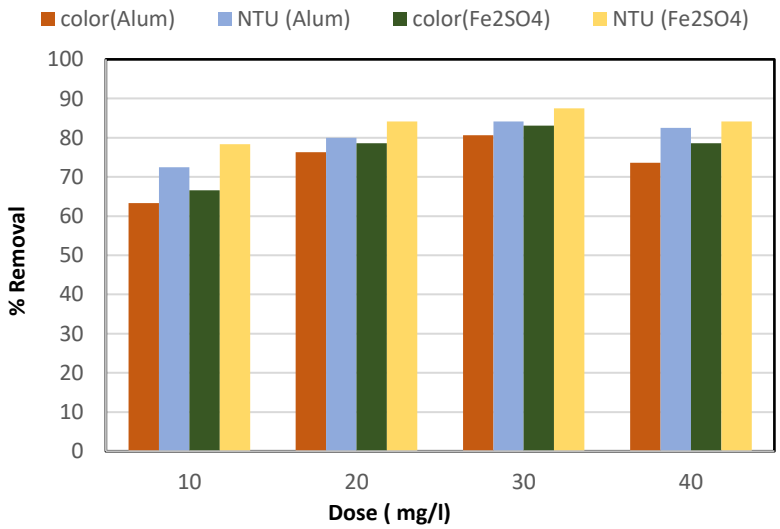


Fig.6 Comparison of Decolorization Efficiency and NTU Efficiency (%) at 120 rpm, 50 min, Alum = 30 mg/L, $Fe_2(SO_4)_3$ = 30 mg/L.

4.CONCLUSIONS

The main conclusions of the present study could be summarized as the efficiency of coagulants in color and turbidity reduction followed the order ($Fe_2(SO_4)_3$ > *ALUM)*. Also, it was found that the maximum turbidity and color removal efficiency using $Fe_2(SO_4)_3$ were 88% and 83%, respectively. While using the alum, the maximum turbidity and color removal efficiency were 84% and 81%, respectively. A direct relation was found between the removal efficiency, sedimentation time, and mixing speed, with the optimal speed of (120 rpm) and sedimentation time of (50 minutes) for both coagulants utilized during treatment. While an inverse relationship was found with the coagulant dosage. When the coagulant dose surpasses the ideal limit stated, the clearance rate drops dramatically. The results of this study showed that the coagulation and flocculation treatment technology, which uses $Fe_2(SO_4)_3$ and alum as active agents, is a fast and effective method due to its low cost and convenience for disposing of colored effluents while preserving the ecosystem and living organisms. One of the most important recommendations is to study the effect of temperature on the efficiency of aqueous solution treatment using other coagulants and doses. It is also suggested to study the efficiency of removing other pollutants, such as COD, BOD, and TSS.

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