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Removal of Reactive Red 141 and Disperse Red 13 Dyes from Aqueous Solutions Using Different Coagulants: An Optimization and Comparison Study

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ABSTRACT

This study investigated the performance of different coagulants for the removal of different dye types from synthetic dye solutions. The ability to use each of the following: aluminium sulphate ($Al_2(SO4)_3$, aluminium chloride ($AlCl_3$), and ferric chloride ($FeCl_3$) as chemical coagulants were examined for removing reactive red 141 (RR 141) dye and disperse red 13 (DR 13) from dye solution. Coagulation studies determined the optimum pH, mixing time, coagulant dosages, and initial dye concentrations. The maximum efficiency for removing RR 141 was 65.7% by aluminium chloride at the operation condition of pH 8, mixing time 10 min, and dye concentration of 100 mg/L. In contrast, under the same conditions, ferric chloride could remove more than 98% of DR 13. Since the disperse dye type has better colour removal, the maximum volume of sludge was 0.3 kg/m^3 which was produced when $FeCl_3$ was used as a coagulant. The results demonstrated that coagulation is a promising technology for dye removal, especially for dispersed dyes as it has some characteristics such as colloidal dispersion and very low water solubility.

Keywords: *Textile wastewater, Chemical coagulation, Reactive dye, Disperse dye.*

Farklı Koagülanlar Kullanılarak Sulu Çözeltilerden Reaktif Kırmızı 141 ve Dispers Red 13 Boyalarının Giderimi: Bir Optimizasyon ve Karşılaştırma Çalışması

ÖZ

Bu çalışmada, farklı boya türlerinin sentetik boya çözeltilerinden gideriminde çeşitli koagülanların performansı araştırılmıştır. Boyarmadde olarak Reaktif kırmızı 141 (RR 141) ve dispers kırmızı 13 (DR 13) seçilmiş, koagülan olarak da alüminyum sülfat, alüminyum klorür ve demir klorür kullanılmıştır. Boya gideriminde pH, karıştırma süresi, koagülan dozajı ve başlangıç boya konsantrasyonlarının etkisi incelenmiştir. RR141 boyasının maksimum giderim (%65.7) koşulları pH 8, 10 dakika karıştırma süresi ve 100 mg/l başlangıç boya konsantrasyonu altında alüminyum klorür ile elde edilirken, demir klorür ise aynı deney şartları altında DR 13 boyasını 98% oranında gidermiştir. Dispers boya tipinde daha iyi renk giderimi elde edildiğinden, olduğundan, demir klorür kullanıldığında açığa çıkan çamur miktarı da fazla olmuştur (~0.3 kg/m3). Elde edilen sonuçlar, koagülasyonun, kolloidal dispersiyon ve çok düşük suda

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çözünürlük gibi bazı özelliklere sahip olması nedeniyle, özellikle dispers boyalar için boya giderimi için umut verici bir teknoloji olduğunu göstermiştir.

Anahtar Kelimeler: *Tekstil atıksuları, Kimyasal koagülasyon, Reaktif boya, Dispers boya.*

I.INTRODUCTION

Water usage varies according to different sectors of the textile industry, for example, coloring process is the sector that consumes water with more than 100 L/kg of processed fabric. In addition, the composition of the wastewater in connection to seasonality, which can greatly vary from one industry to another [1]. In order to produce textiles, a high concentration of various organic dyes, additives, and salts is needed. This results in wastewater that is typically warm and has high turbidity, chemical oxygen demand, suspended solids, pH ranging from 2 to 12, and suspended solids [2], [3] The discharge of textile wastewaters alters the dynamics of the aquatic ecosystem, reduces light penetration, and has a detrimental aesthetic impact on water supplies [4], [5]. Dyes and some by-products such as aromatic amines can be mutagenic and carcinogenic [6], [7]. Dye in wastewater is harmful, even in small amounts, as its release into aquatic habitats can have negative aesthetic and health consequences on living things [8].

Numerous methods for treating textile wastewater, including electrochemical [8]–[10], biological [11]–[13], adsorption[14]–[16], coagulation[17], [18], and advanced oxidation processes[19]–[21], have been reported in the literature. Biological remedies take a long time, especially for bacteria to adapt, and need reactors with a high volume. In addition, the organisms are sensitive to pH, temperature, and oxygen, especially in aerobic environments and when exposed to harmful substances. In addition, some colours are also not biodegradable [17]. Moreover, the adsorption process is constrained by the adsorbent's capacity and needs a prefiltration step to remove suspended solids (SSs). Regenerating the adsorbent is also a difficult problem. Although the AOPs can remove the majority of pollutants, their high cost, numerous operating challenges, and complexity as technology may prevent their widespread use [22].

Because of its high efficiency and low cost, small equipment space and less sludge production, the coagulation-flocculation method is frequently employed in the treatment of textile wastewater [23]. The coagulation process involves adding chemicals to wastewater to change the physical properties of the dissolved and suspended solids and stimulate their removal through sedimentation [24]. By adding substances referred to as coagulants, colloidal particles are destabilized throughout the coagulation process. Double-layer compression is first encouraged by an increase in ionic strength, and subsequently, the particle surface is neutralized by the adsorption of counter anions [25]. The direct addition of a coagulant and modification of solution pH are characteristics of the coagulation process. The majority of the colloids have negative charges and are stable in water solutions [26]. To stabilize the water solution, some cations are needed to interact with colloids and neutralize their charges. The more efficient and often utilized coagulants in water purification are hydrolyzing metal salts [27]. The choice of a coagulant is crucial for the removal of contaminants during the coagulation/flocculation process. Coagulants can be divided into a wide variety of inorganic and organic types. Iron and aluminium salts, for example, were frequently used in the treatment of textile effluent as inorganic coagulants [28]. The present study aimed at evaluating and comparing the efficiency of three chemical coagulants ($AlCl_3$, $FeCl_3$ and $Al_2(SO_4)_3$) for different dye solution. A coagulation process was conducted under different experimental conditions to assess the treatability of textile wastewater by the coagulation method. In this study, the influence of pH, coagulant type and concentration, dye concentration and type on the coagulant capacity of removing colour and absorbance has been evaluated.

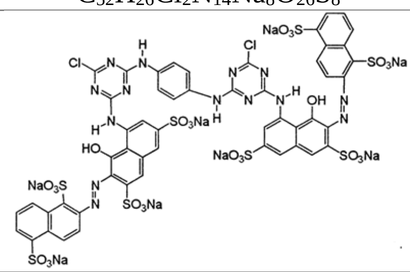
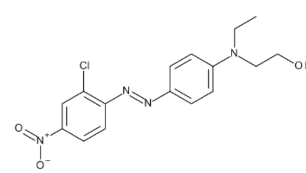
II.MATERIAL AND METHODS

Within the scope of the experimental study, the pre-treatment efficiencies of reactive red 141 (RR141) and disperse red 13 (DR13) dyes, which are used in textile factories, will be investigated with the chemical coagulation process with various configurations. The effect of parameters such as pH, mixing time, and coagulant dosage on chemical coagulation treatment efficiency will be investigated, and the removal efficiency will be monitored at different dye concentrations under optimum conditions. For this purpose, the effect of the chemical coagulation process on the pre-treatment of dyestuffs in textile waters will be figured out by conducting laboratory-scale experiments. In addition, chemical coagulation, and purification comparisons of reactive red and disperse red dye types will be observed. Sludge formation is important for the coagulation process because disposal requires extra costs. Therefore, sludge formation will be measured for each coagulant after the experiments.

A. MATERIALS

All chemicals, including H_2SO_4 (Sigma-Aldrich), NaOH (Sigma-Aldrich), $Al_2(SO_4)_3$ (Sigma-Aldrich), $AlCl_3$(Sigma-Aldrich), $FeCl_3$ (Sigma-Aldrich), Reactive 141 (Dystar), Disperse Red 13 (Dystar) were used as received. Doubly deionized water (Millipore Milli-Q system, resistivity ≥18.2 MΩ cm) was used for the preparation of all solutions. The cholesterics of reactive 141 and disperse are shows in Table 1.

Table 1. *Dyes properties.*

Dye name	**Reactive Red 141**	**Disperse Red 13**
Molecular formula	$C_{52}H_{26}Cl_2N_{14}Na_8O_{26}S_8$	$C_{16}H_{17}ClN_4O_3$
Molecular structure		
CAS	61931–52–0	3180-81-2
Molecular wight *(g/mol)*	1774.15	348.78
Melting point	121-123 *°C*	122-129 *°C*
Wavelength absorbance *(λmax)*	544 *nm*	524 *nm*

B. DYE ANALYSIS

The dyes used in this study was RR141 and DR 13. The absorbance was measured with spectrophotometer (UV spectrophotometer light wave, Hach DR5000) at maximum absorption wavelengths, Reactive red 141, λmax = 544 nm, and Disperse red 13, λmax = 524 nm (see Figure 1). The samples were filtered through 0.2 μm membranes while measuring absorbance. The efficiency of dye removal was expressed as the percentage ratio of decolorized dye concentration to that of initial one.

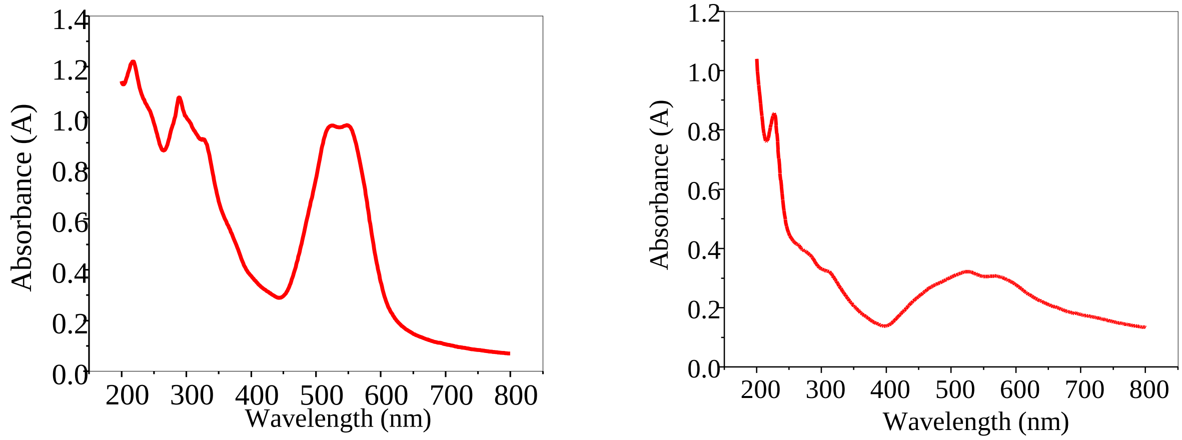


Figure 1. *UV–visible spectra and standard curve of the* ***(a)*** *Reactive Red 141* ***(b)*** *Disperse Red 13.*

C. COAGULATION STUDIES

Coagulation studies were performed in a conventional model of jar-test apparatus, equipped with four 100 mL beakers. The calculated quantity of coagulant $Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$, were added to the synthetic dye solution, and the pH was adjusted to the required value using H_2SO_4, and NaOH. The mixture was stirred at the rate and during the time according to experiment. After mixing, the samples filtered to measure the quantity of sludge formed. The investigated parameter was pH, mixing time, coagulant dosage, dye construction and type are described below.

D. pH

pH significantly affects the coagulation process as the charge of coagulant and dissolved ions as well as solubility is high pH dependent. To investigate the pH influence on the treatment of dye solution by coagulants, the pH, in range of 4-8, was used while the other parameters was fixed (RR 141 concentration 100 mg/L, mixing time 10 min, and investigated coagulants dosage ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$) 200 mg/L). The pH was adjusted using 0.1 N H_2SO_4 and 1 N NaOH solutions.

E. MIXING TIME

Since the equilibrium between floc formation and breakage is reached at a constant shear applied during a "characteristic time," the time of mixing affects the destabilization of colloid and the downstream aggregation of particles. To study the influence of mixing time on the removal of dye form dye solution, mixing time of (5, 10, 15, 20, 25) min where selected. The other parameters were fixed as fellow: RR 141 concentration 100 mg/L, coagulant dosage ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$) of 200 mg/L, the optimum pH was set because of optimum value reached in the previous section ($Al_2(SO_4)_3$= 8, $AlCl_3$=8. $FeCl_3$=5).

F. COAGULANT DOSAGE

The optimum coagulant dose for treating wastewater by coagulation is often chosen based on how well different coagulant dosages work at optimum pH level [29]. As a result, the coagulation processes at different coagulants doses were also examined at optimum pH. Coagulants dosages of (200, 400, 600, 800, 1000) mg/L at optimum pH of ($Al_2(SO_4)_3$= 8, $AlCl_3$=8. $FeCl_3$=5), and mixing time of ($AlCl_3$=10 min, $FeCl_3$= 10 min was determined as $Al_2(SO_4)_3$=20 min) were investigated. The dye concentration (RR141 was fixed at 100 mg/L.

G. INITIAL DYE CONCENTRATION

The effect of initial dye concentration was investigated under optimum pH, mixing time and coagulant dosage. RR 14 concentration of (100, 200, 300, 400, 500) mg/L at optimal pH conditions ($AlCl_3$=8, $FeCl_3$=5, $Al_2(SO_4)_3$=8), mixing time conditions ($AlCl_3$=10min, $FeCl_3$=10 min, $Al_2(SO_4)_3$=20 min), and coagulant dosage conditions (200 mg/L) were studied.

H. DYE TYPE

The effect of dye type (Reactive and Disperse) was investigated at the same optimum conditions (pH, mixing time, coagulant dosage, and type) determined in above procedure. The investigated parameters are shows in Table 2.

Table 2. *Investigated parameter under optimum conditions for RR141 and DR13.*

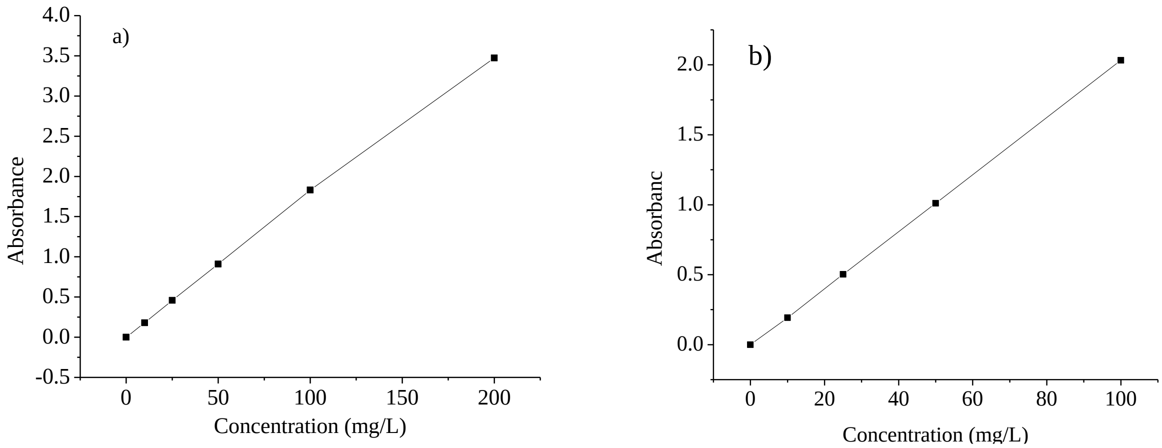
	Coagulant		
	$AlCl_3$	$FeCl_3$	$Al_2(SO_4)_3$
pH	8	5	8
Time *(min)*	10	10	20
Coagulant Dosage *(mg/L)*	200	200	200
Dye dosage *(mg/L)*	100	100	100

İ. DYE REMOVAL

Initially, the original dye solution was prepared with a concentration of 1000 mg/L of Reactive Blue 19 dye, and standard solutions were made from this solution, at concentrations of 0, 10, 25, 50 and 100 mg/L. Using a spectrophotometer, the calibration curve was prepared at λmax = 544 nm for RR141, λmax = 524 nm for DR13. Using the equation obtained from the calibration curve (Figure 2), the unknown dye concentration was determined where the dye removal efficiency was found using eq. (1).

$$\text{Dye removal} = \frac{C_0 - C}{C_0} * 100 \qquad (1)$$

Where C_0 and C are the initial dye concentration and dye concentration of dye after treatment.



(a) ***(b)***

Figure 2. *Calibration curve for* ***(a)*** *RR141,* ***(b)*** *DR 13.*

III.RESULTS AND DISCUSSION

A. EFFECT OF THE INITIAL PH ON THE PERFORMANCE OF THE COAGULANTS

To study the effect of initial pH on the treatment of synthetic dye solution, five initial pH were tested (4,5,6,7,8) the other parameters were kept fixed as fellow: RR 141 concentration was 100 mg/L and the mixing speed and time were 60 rpm and 10 min, and coagulant ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$) dosage was 200 mg/L. Figure 3 present the effect of pH on dye RR141 removal using three different coagulants ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$). According to Figure 3, the best dye RR141 removal efficiency for the investigated coagulants $Al_2(SO_4)_3$, $AlCl_3$, and $FeCl_3$ was 51.41%, 65.68%, and 65.75% at pH values of 8, 5, and 8, respectively. Similar results was obtained by Hussein & Jasim (2019) where the optimum pH for aluminum salts ($Al_2(SO_4)_3$, $AlCl_3$) where ferric chloride showed high dye removal efficiency at pH 5. As it is clear with ferric chloride that the best value of pH for RR141 was five which might be due to strong precipitation at acidic pH. As the precipitation increased, solution will more clearly give very low value of percentage removal. However, the percentage removal of RR141 with alum was maximum at basic condition. This means that when applying ferric chloride as coagulant favourable pH is acidic [31]. However, for alum the favourable pH is basic [32], [33].

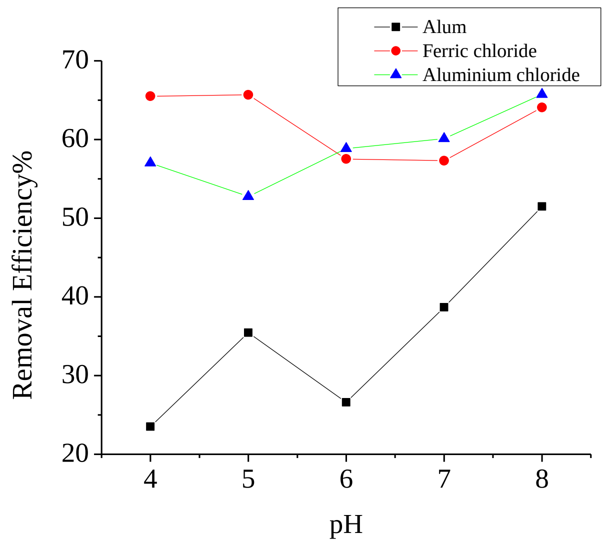


Figure 3. *Effect of pH on dye RR141 removal using three coagulants ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$).*

B. EFFECT OF MIXING TIME

After obtaining the optimum pH for the investigated coagulants, the optimum time for RR141 removal was evaluated at five different mixing time (5,10,15,20,25) min at pH ($Al_2(SO_4)_3$= 8, $AlCl_3$=8. $FeCl_3$=5), RR 141 concentration 100 mg/L, and coagulant dosage ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$) of 200 mg/L. Figure 4 shows the effect of mixing time on RR141 removal. The results obtained show that the best time for RR141 removal using Alum was 20 min mixing time as the removal efficiency reached 56.89%. Regarding aluminium chloride and ferric chloride, the highest RR141 removal efficiency was obtained at 10 mixing time where the removal efficiency was 65.74% and 65,68%, respectively. The Figures 4 shows similar pattern for all investigated coagulants, the removal efficiency increased gradually until reached the highest dye removal point. Increasing mixing time after these points diversly affect the dye removal. Further mixing time disrupt the produced flocs resulting on destabilization of the colloid and the downstream aggregation of particles [34].

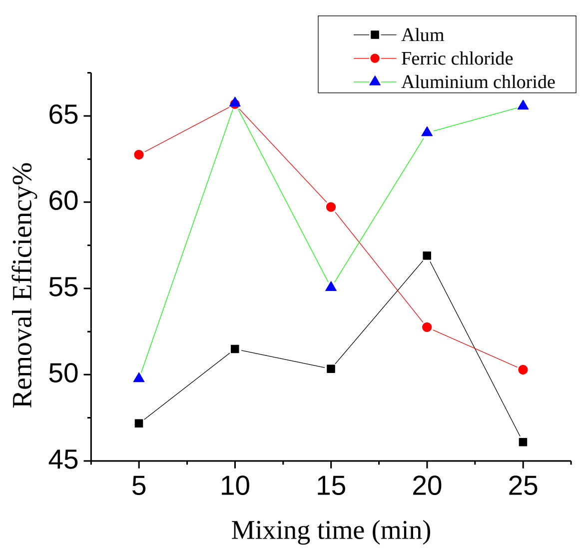


Figure 4. *Effect of mixing time on RR141 removal using three coagulants ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$).*

C. EFFECT OF THE COAGULANT CONCENTRATION

After determining the optimum pH and mixing time, the optimum coagulant dosage was tested using five dosages concentration (200,400,600,800,1000) mg/L, at optimal pH of ($Al_2(SO_4)_3$= 8, $AlCl_3$=8. $FeCl_3$=5), and mixing time of ($AlCl_3$=10 min, $FeCl_3$= 10 min was determined as $Al_2(SO_4)_3$=20 min) were investigated in this section. The dye concentration (RR141 was fixed at 100 mg/L. According to Figure 5, the optimum dosage for all investigated was 200 mg/L which resulted in removal efficiency of 56.89%, 65.68%, and 65.74 for ($Al_2(SO_4)_3$, $FeCl_3$, and $AlCl_3$, respectively. After reaching to optimum coagulant dosage, increasing of coagulants dosage resulted in decreasing in RR141 removal efficiency decreased. This decrease is due to destabilization with the increase of positive ions resulting from excess coagulants[29].

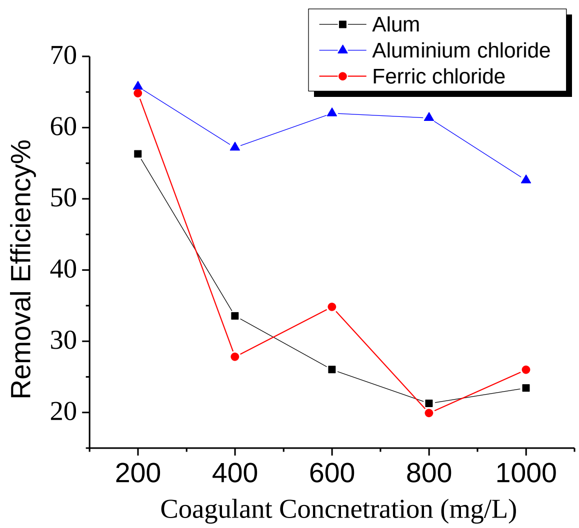


Figure 5. *Effect of coagulant concentration on RR141 removal using three coagulants ($Al_2(SO_4)_3$, $AlCl_3$, $FeCl_3$).*

D. EFFECT OF THE DYE CONCENTRATION

The effect of dye solution on the performance of coagulants for dye removal was tested by different concentrations (100, 200,300,400,500) mg/L at pH conditions of pH ($AlCl_3$=8, $FeCl_3$=5, $Al_2(SO_4)_3$=8), mixing time ($AlCl_3$=10min, $FeCl_3$=10 min, $Al_2(SO_4)_3$=20 min), and coagulant dosage (200 mg/L). As shows by Figure 6, the removal efficiency of RR141 was decreased as initial dye concentration increased. The dye removal efficiency decreased from 56.89% to 26.31% when RR141 concentration increased from 100 mg/L to 500 mg/L using $Al_2(SO_4)_3$ as coagulant. In terms of $FeCl_3$, the removal efficiency dropped by two-fold to reach 27.11% when the RR141 concentration was 500 mg/L. Regarding to $AlCl_3$, when RR141 concentration increased five times (100 to 500 mg/L), the removal efficiency dropped by 20% to reach around 40%. Aluminium chloride was found more effective than alum and ferric chloride when the RR141 was increased. The similar trend was observed by [35].This decrease because of the accumulation of dye on the surface of investigated coagulants resulting in electrostatic repulsion between dye molecule of the same charge.

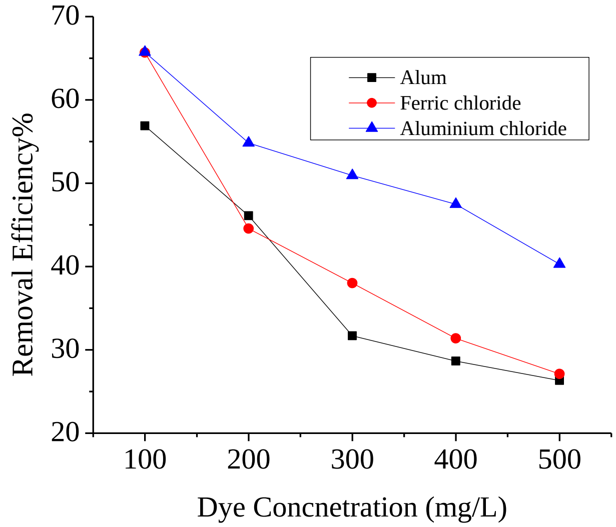


Figure 6. *Effect of RR141 concentration on performance of coagulants.*

E. EFFECT OF DYE TYPE

To investigate the effect of dye type on the performance of coagulant for dye removal, the same optimum conditions achieved for RR141 in the previous section, was applied for DR 13. Figure 7 presents the comparison between RR141, and DR 13 removal effeminises using coagulants. The results showed that disperse dyes are highly removed by coagulation process. Ferric chloride could remove more than 98% of DR13 while at same conditions the removal efficiency of RR 131 was 65.68%. As disperse dyes have some characteristics such as colloidal dispersion and very low water solubility, the removal of such dyes by coagulation is easier than reactive dyes which has high water solubility[36].

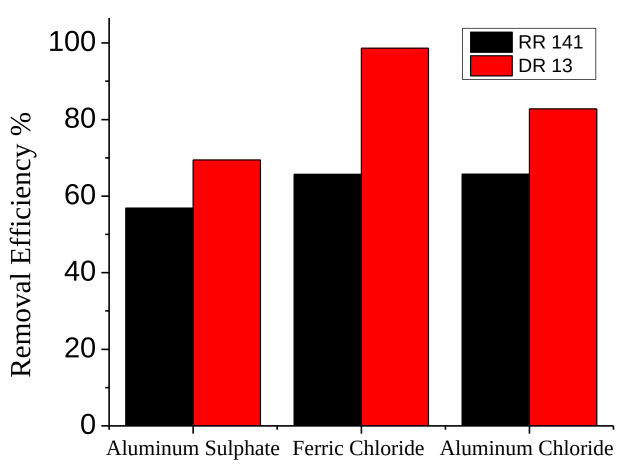


Figure 7. *Comparison between RR141 and DR 13 removal by coagulants.*

F. SLUDGE PRODUCTION RESULTS

Generally, depending on the dye type and concentration, coagulant concentration, and operating conditions, certain amount of sludge is producing during the coagulation treatment [37], where a direct relation between sludge production and removal efficiency take place. In this study, As the dye was the only pollutant, the amount of produced sludge was equal to the sum of the amount of dye removed from the prepared dye solution and the amount of coagulant thrown into the solution. As shows in Table 3, the maximum volume of produced sludge was calculated as mg produced from treated one liter. Alum produced less sludge than ferric chloride and aluminium chloride. As the the highest removal efficiency of DR13 was obtained using $FeCl_3$, the maximum sludge volume was produced (298.64 mg from one treated liter).

Table 3. *The Amount of Sludge Formed by investigated coagulants*.

	$AlCl_3$	$FeCl_3$	$Al_2(SO_4)_3$
RR141 Sludge (mg for treated L)	265.74	265.68	256.89
DR 13 Sludge (kg for treated L)	282.76	298.64	269.43

IV.CONCLUSION

The present study compared the efficiency of $Al_2(SO_4)_3$, $AlCl_3$, and $FeCl_3$ as chemical coagulants in dye removal from different dye solutions. It was verified that pH, mixing time, coagulant dosage, and dye concentration affect the removal efficiency. Each coagulant has different optimum pH as follows; Alum, ferric chloride, and aluminum chloride at 8,5, and 8, respectively, which means that dye removal by coagulants is highly pH dependent. The performance of ferric chloride was higher than other coagulants in terms of RR 141 and DR 13 which reached 65.68% and 98,64%, respectively. Both, an increase in initial dye concentration and a further increase of coagulant dosage (above optimum dosage) resulted in a decrease in removal efficiency. The volume of sludge produced because of the coagulant used follows the sequence as alum less than aluminum chloride and aluminum chloride less than ferric chloride. The results showed that coagulation is a promising technology for dye removal especially for disperse dyes as it has some characteristics such as colloidal dispersion and very low water solubility.

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