

# Color removal from simulated dye water and actual textile wastewater using a composite coagulant prepared by polyferric chloride and polydimethyldiallylammonium chloride

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## Abstract

In this study, a new composite coagulant, which was prepared by polyferric chloride (PFC) premixed with polydimethyldiallylammonium chloride (PDMDAAC), was adopted to treat the simulated dye water and the actual textile wastewater. The effect of coagulant dosage and pH of dye water on the color removal was examined. And the color removal results treated by the composite coagulant (PFC–PDMDAAC) were compared with those treated by PFC, PDMDAAC and PFC followed by PDMDAAC (PFC/PDMDAAC), respectively. The following results are discovered. Synergy between PFC and PDMDAAC increases zeta potential of PFC–PDMDAAC and reduces the effect of pH on zeta potential and Fe(III) species of PFC–PDMDAAC. Treatment with PFC–PDMDAAC is very effective in removing disperse blue HGL (98%) and reactive blue STE (86%). And small changes in the color removal efficiency of PFC–PDMDAAC are observed in all pH range. The color removal efficiency for disperse blue HGL and reactive blue STE is higher than 96% and 98% in the pH range 7.5–10.5, respectively. Furthermore, PFC–PDMDAAC is shown to be more effective in removing dye than that of PFC/PDMDAAC. Finally, the treatment of the actual textile wastewater has shown that PFC–PDMDAAC is a more effective coagulant.

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**Keywords:** Color removal; Composite coagulant; Polyferric chloride; Polydimethyldiallylammonium chloride; Textile wastewater

## 1. Introduction

Textile industry generally discharges large volumes of wastewater. This wastewater, which has high pH, alkalinity and temperature, contains high concentration of organic matter, non-biodegradable matter, toxic substances, detergents and soaps, oil and grease, sulfide, and suspended/dissolved solids. Although biological treatment processes such as aerated lagoons are generally efficient in BOD and SS removal, they are ineffective for removing color from the wastewater because of the low biodegradability of many textile chemicals and dyes [1]. Therefore, textile wastewater has to be decolorized before being fed to the subsequent treatment units, such as the adsorption and the biological treatment.

The removal of color by coagulation is a widely employed treatment process [2–5]. Al(III) and Fe(III) coagulants are two

principal inorganic coagulants used in wastewater treatment. These hydrolysable cations are readily available as sulfate or chloride salts in both liquid and solid form. However, aluminum is suspected harmful to human and living organisms [6]. Thus, ferric ions are often the coagulant of choice to destabilize colloidal and suspend solids. The treatment of textile wastewater with ferrous sulfate, regulating pH in the range of 8.5–9.5 by lime, was proved to be very effective in removing color (70–90%) and COD (50–60%) from textile wastewater [7]. However, it was found that reactive dye solution was difficult to decolorize by inorganic coagulants which might be effective on decolorizing disperse dye solution [8], and ferric coagulants need a large amount of alkali to resist the pH depression that occurs during coagulation [9]. Therefore, research on novel coagulants has been particularly active in recent years [10]. Among these studies, the use of inorganic coagulants and organic coagulants together was proved to be effective to treat water and wastewater [11].

In this study, a new inorganic–organic composite coagulant, which was prepared by inorganic polyferric chloride (PFC)

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premixed with organic polydimethyldiallylammonium chloride (PDMDAAC), was used to treat the simulated dye water and actual textile wastewater. PFC is a relatively new polymeric coagulant which contains a range of pre-formed Fe(III) hydrolysis species of high positive charge and medium to large molecular weights [12]. PFC has been shown to be more efficient than the monomeric salt for removing turbidity, algae, color and natural organic matter, and it is less corrosive than  $\text{FeCl}_3$  [6]. PDMDAAC, which has high positive charge, is an organic coagulant. It has been reported that PDMDAAC not only effectively remove the turbidity but also reduce the formation of  $\text{CHCl}_3$  in water treatment [13]. And the efficiency of poyaluminum chloride in combination with PDMDAAC can be improved and the dosage of coagulant can be induced [14]. In principle, PFC–PDMDAAC should be more effective in removing color of textile wastewater than PFC or PDMDAAC; however, there are few reports on the use of PFC combined with PDMDAAC in water or wastewater treatments.

The aim of the present work was to test the ability of PFC–PDMDAAC as a coagulant to treat the simulated dye water and the actual textile wastewater. The effect of coagulant dosage and pH of dye water on the color removal was examined. And the color removal results treated by the composite coagulant (PFC–PDMDAAC) were compared with those treated by PFC, PDMDAAC and PFC followed by PDMDAAC (PFC/PDMDAAC), respectively.

## 2. Materials and methods

### 2.1. Properties of coagulants used for color removal

Three different coagulants including PFC, PDMDAAC and PFC–PDMDAAC were examined for their color removal efficiencies on the simulated dye water and the actual textile wastewater.

The PFC used in this study was prepared in our laboratory. Stock  $\text{FeCl}_3$  solution of 8.3% was prepared with  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (A.R.) in distilled water. Then, the  $\text{FeCl}_3$  solutions were slowly mixed with  $\text{Na}_2\text{CO}_3$  powder (C.P.) to obtain the PFC solution with desired  $[\text{OH}^-]/[\text{Fe}]$  ( $B$ ) value.  $\text{Na}_2\text{HPO}_4$  (A.R.) was added to the PFC solution as a stabilizer ( $[\text{Na}_2\text{HPO}_4]/[\text{Fe}]=0.04$ ). The target PFC had the following characteristics:  $W_{\text{Fe}} = 7.9\%$ ;  $B = 1.0$ ; and pH 0.83.

PDMDAAC was obtained from Bin Zhou Chemical Co., Shandong, China. Its intrinsic viscosity ( $\eta$ ), which is proportional to the molecular weight of PDMDAAC, was 1.02 dL/g.

PFC–PDMDAAC was prepared by PFC and PDMDAAC. A measured amount of PDMDAAC was injected into the stock solution of PFC with strong stirring to prepare PFC–PDMDAAC. The percentage of PDMDAAC by weight in the flocculants ( $W_p$ ) was 7.0%. The properties of PFC–PDMDAAC do not change in 3 months.

### 2.2. Wastewater characteristics

The simulated dye water was studied in order to determine the effectiveness of coagulants in removing various types of

dyes. Among the commercial textile dyes, disperse and reactive dyes are of great environmental concern because of their widespread applications. Therefore, disperse blue HGL (D.B. HGL) and reactive blue STE (R.B. STE), which were obtained from Jinan No. 2 Textile Dyeing Mill, China, were chosen to investigate the effectiveness of PFC–PDMDAAC. The simulated dye water was prepared by adding 0.1 g of D.B. HGL or R.B. STE to 1 L of distilled water. The characteristic wavelength for each simulated dye water was determined by running a scan of the dye on a spectrophotometer (UV-2450). The maximum absorbance wavelength ( $\lambda_{\text{max}}$ ) was used for all absorbance readings. Percentage of color removal was calculated by comparing the absorbance values for the waste after treatment to the absorbance value for the original dye waste. Distilled water served as a reference. The maximum absorbencies for D.B. HGL at  $\lambda_{\text{max}} = 532$  nm and R.B. STE at  $\lambda_{\text{max}} = 593$  nm were 1.526 and 1.342, respectively.

The actual textile wastewater to be tested was obtained from the Binzhou textile company in Shandong Province, China. The wastewater colored dark blue, which was caused by two main dyes—disperse blue and active blue, and contained high concentrations of organic matter including dyes, additive, surfactant and others. Since the actual textile wastewater contained different kinds of dyes, the traditional method of applying the single maximum absorbance was not appropriate for the actual wastewater. According to the absorbance curve of the actual textile wastewater, three wavelengths (330, 480, and 594 nm) were chosen to determine color content of the actual textile wastewater. Color content was determined by taking the sum of absorbencies measured the absorbance at 330, 480 and 594 nm. The characteristics of the wastewater were: COD = 4860 mg/L, Abs (sum) =  $\text{Abs}_{(330)} + \text{Abs}_{(480)} + \text{Abs}_{(594)} = 3.756$ , pH 10.34.

### 2.3. Coagulation test procedures

All coagulation experiments were conducted in 1.0 L plexi-glass beakers using a conventional Jar-test apparatus, the DC-506 Laboratory Stirrer. Five hundred milliliters suspensions were dosaged with different coagulants. The solutions were stirred rapidly at 120 rpm for 3 min during coagulation addition, and followed by slow stirring at 40 rpm for 12 min and sedimentation for 12 min. After sedimentation, supernatant samples were taken from a point of 3 cm below the surface of the test water sample for analysis.

### 2.4. Determination of Fe(III) speciation in coagulants

The Fe(III) species distribution in coagulants was measured by a timed complexation spectroscopy method involving reactions of Fe with Ferron (8-hydroxy-7-iodoquinoline-5-sulphonic acid). A full description of this method can be found elsewhere [15,16]. A brief description is provided as follows. Visible light absorbance is measured as a function of time at a wavelength of 600 nm to quantify the amount of Fe complex formed. The reactions of Fe(III) monomeric species with ferron are complete within 1 min (denoted as  $\text{Fe}_a$ ), reaction over the next 3 h is Fe(III) polymeric species ( $\text{Fe}_b$ ), any Fe(III) that

remains unreactive after 3 h is considered to be precipitated ( $\text{Fe}_c$ ).

### 2.5. Determination of zeta potential

The malvern Zetasizer 3000 was used to measure Zeta potential of PFC and PFC–PDMDAAC. Experiments were performed at coagulant dosage of 0.01 mol/L as Fe. And pH of the coagulant solutions was adjusted by adding HCl (1 mol/L) or NaOH (1 mol/L). After 5 min of gentle stirring, the samples were analyzed and the data were recorded.

## 3. Results

### 3.1. Determination of optimal dosage for chemical coagulation of synthetic dye wastewater

The optimal dosages of coagulants for D.B. HGL and R.B. STE were determined by varying the coagulant dosage at pH 7.

Dye removal for D.B. HGL as a function of coagulant dosage was shown in Fig. 1A. Treatment of synthetic D.B. HGL wastewater by PDMDAAC yielded an optimum color removal of 74% at a dosage of 18 mg/L. PFC can remove 88% of D.B. HGL at a dosage of 30 mg/L, but its color removal efficiency was lowest among all coagulants when dosage was lower than 25 mg/L. The advantages of PFC–PDMDAAC treatment for synthetic D.B. HGL wastewater were clearly evident in Fig. 1A. The color removal efficiency of PFC–PDMDAAC can reach 95% with a dosage of only 16 mg/L. And above 16 mg/L, there was a slight increase in removal efficiency of D.B. HGL. Treatment of D.B. HGL with PFC–PDMDAAC yielded an optimum color removal of 98% at a dosage of 30 mg/L.

Fig. 1B was a plot of the color removal efficiency of synthetic R.B. STE wastewater versus the dosage of coagulation. When the dosage of coagulant was lower than 45 mg/L, the efficiency of PDMDAAC was the highest among all coagulation. However, the color removal efficiency of PDMDAAC was only 68% at the dosage of 45 mg/L. When the dosage was higher than 45 mg/L, the efficiency of PFC–PDMDAAC was the highest among all coagulation and it can reach 86%.

Based on the results mentioned above, PFC–PDMDAAC shows two main advantages: the color removal efficiency of D.B. HGL and R.B. STE is improved compared to use of PFC and PDMDAAC alone; and the optimal dosage of PFC–PDMDAAC for D.B. HGL is reduced compared to PFC.

### 3.2. Determination of optimal pH for chemical coagulation of synthetic dye wastewater

The subsequent experiments were designed to find the optimal pH of synthetic D.B. HGL and R.B. STE wastewater that allowed for maximum dye removal. Destabilizations of synthetic D.B. HGL and R.B. STE wastewater were examined at various pH conditions (3, 4.5, 6, 7.5, 9, 10.5), respectively. For synthetic D.B. HGL wastewater, the dosage of PFC and PFC–PDMDAAC were 30 and 16 mg/L, respectively. For synthetic R.B. STE

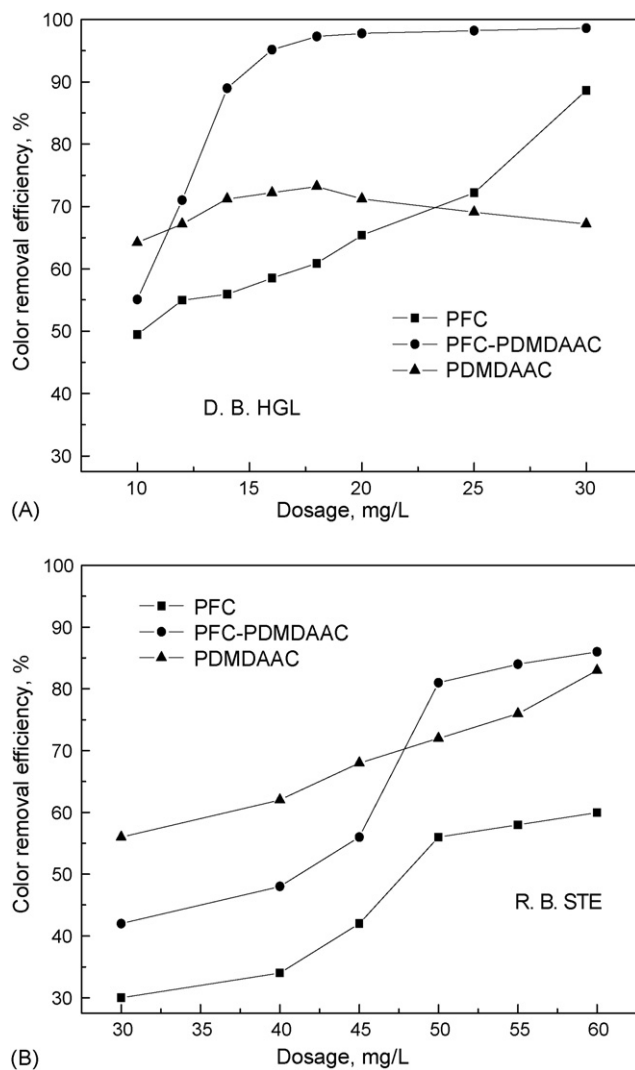


Fig. 1. Decoloration of dye solution according to coagulants dosage.

wastewater, the dosage of PFC and PFC–PDMDAAC were 60 mg/L.

The maximum removals of PFC for synthetic D.B. HGL and R.B. STE wastewater were 98% and 77% at pH 6 (Fig. 2), respectively. And the difference of color removal efficiencies of PFC was significant at different pH. For example, the color removal efficiencies for synthetic D.B. HGL and R.B. STE wastewater were 80% and 49% at pH 10.5, respectively.

Small changes in the color removal efficiency of PFC–PDMDAAC for synthetic D.B. HGL wastewater were observed in all pH range. All of the color removal efficiency for synthetic D.B. HGL wastewater was higher than 96%. For synthetic R.B. STE wastewater, the color removal efficiency increased from 89% to 99% as the pH value increased from 3.0 to 7.5, and the efficiency decreased appreciably as the pH is higher than 7.5. However, the color removal efficiency for synthetic R.B. STE wastewater was still higher than 98% from 7.5 to 10.5.

The results of this study (Fig. 2) indicate that the high original pH of the actual textile wastewater would be advantageous using

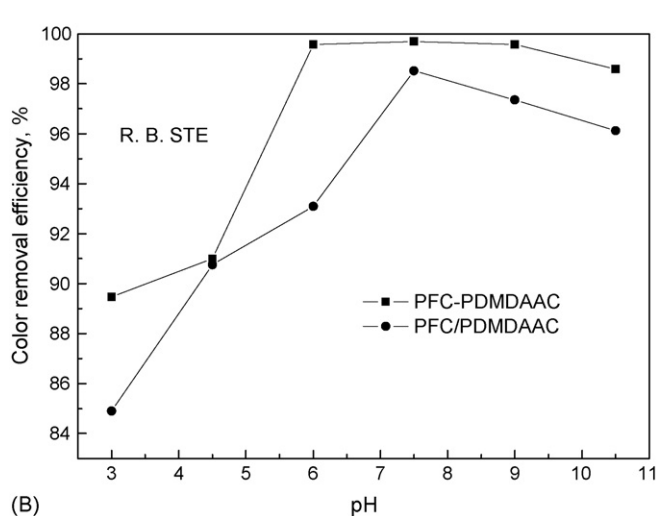
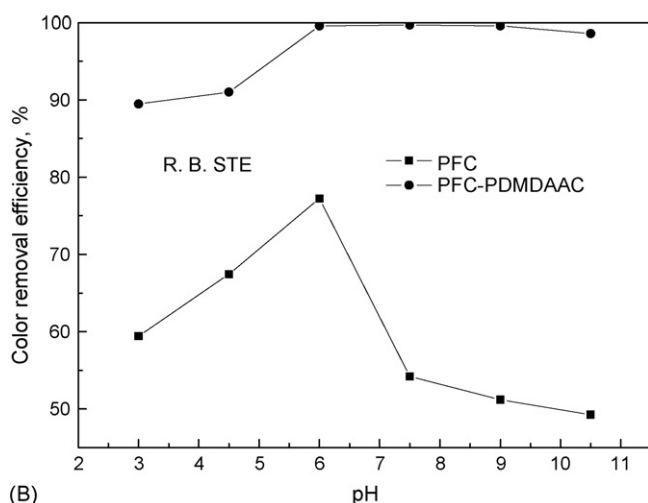
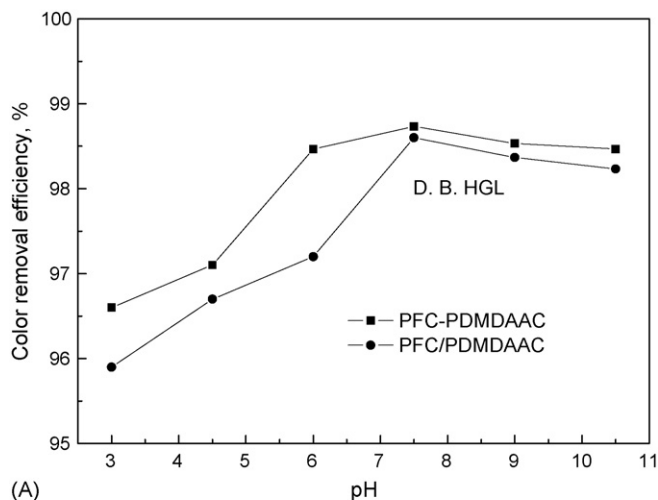
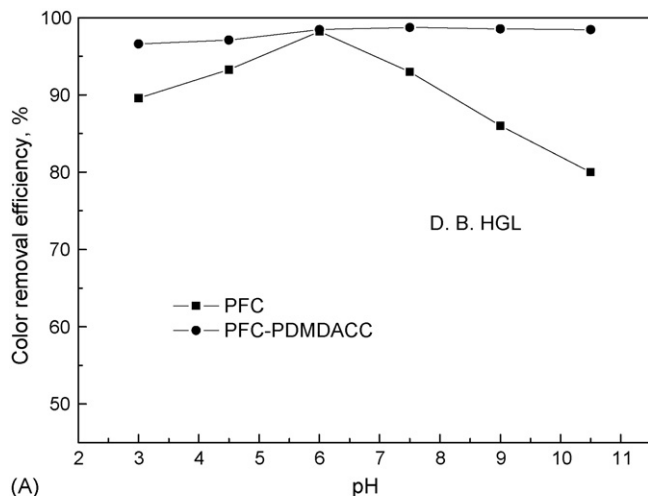


Fig. 2. Decoloration of dye solution according to solution pH.

Fig. 3. Decoloration of dye solution according to addition method of coagulant.

PFC–PDMDAAC, and alkali, which is necessary to maintain pH value for chemical decolorization treatment [5], is not needed for PFC–PDMDAAC.

### 3.3. Effect of the addition method of PFC with PDMDAAC on synthetic dye wastewater

In generally, the method of using inorganic–organic composite coagulants is that inorganic coagulant and organic coagulant are added to wastewater, respectively [10]. The color removal efficiency of PFC–PDMDAAC (PFC premixed with PDMDAAC) was compared with that PFC/PDMDAAC (PFC was added first, and then PDMDAAC) in this study. The results are shown in Fig. 3. It can be seen that the effective pH range of PFC–PDMDAAC was similar to that of PFC/PDMDAAC, but the color removal efficiency of the former was slightly higher at the same pH. This indicates that there are no significant differences of PFC–PDMDAAC with PFC/PDMDAAC in treating stimulate dye water, but for actual treatments, PFC–PDMDAAC would be more convenient than PFC/PDMDAAC.

### 3.4. Treatment of the actual textile wastewater

A similar test has been carried out on the treatment of actual textile wastewater from the Binzhou textile company, Shandong, China. Fig. 4 shows the relationship of color removal efficiency with coagulants dosage of PFC, PDMDAAC, PFC–PDMDAAC. As displayed by the results, PFC–PDMDAAC was more efficient in removing color than PFC and PDMDAAC. The removal efficiency of PFC–PDMDAAC at the dosage of 150 mg/L was 88.9%. While at the same dosage, the color removal efficiencies of PFC and PDMDAAC approached 52.7% and 43.1%, respectively.

## 4. Discussion

The performance of color removal by chemical coagulation depends on the characteristic of both dyes and coagulants. Disperse dye with low solubility tend to be adsorbed by  $\text{Fe}(\text{OH})_x$  particles [8]. While reactive dyes, which are characterized by azo bands ( $\text{N}=\text{N}$ ) and have  $-\text{SO}_3^-$ ,  $-\text{COO}^-$ ,  $-\text{OH}$  groups, have high solubility, they are not prone to be adsorbed by  $\text{Fe}(\text{OH})_x$  particles [17]. Therefore, PFC can remove D.B. HGL efficiently



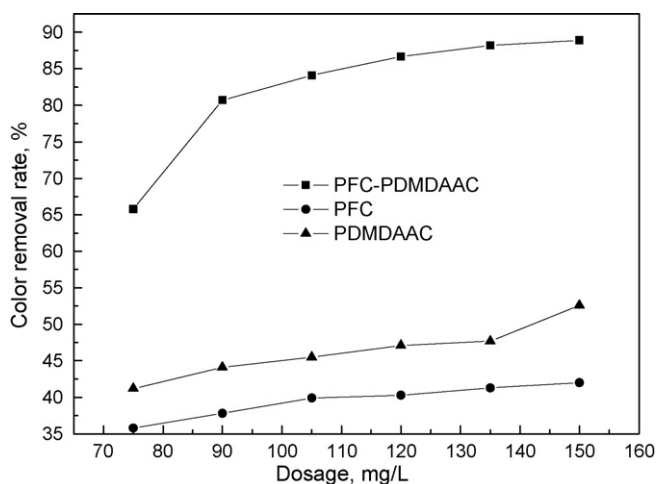


Fig. 4. Color removal of textile industry wastewater using coagulants.

(88%), but not R.B. STE (60%). PDMDAAC with high positive charge can remove dye by charge neutralization. Moreover, PDMDAAC can react with reactive dyes by  $-\text{NH}_2$  group to produce the insoluble compound [18]. Therefore, the color removal efficiency of R.B. STE by PDMDAAC is higher than that of D.B. HGL (as shown in Fig. 1b). PFC–PDMDAAC, which is composed by PFC and PDMDAAC, has the advantages both of PFC and PDMDAAC, so it can remove both D.B. HGL and R.B. STE efficiently (Fig. 1).

To investigate the reason of high efficiency and the dye removal mechanism of PFC–PDMDAAC, the variation of zeta potential and Fe(III) species of PFC–PDMDAAC hydrolysis products as a function of pH was measured, and the results was compared with that of PFC. In this series of experiments, the pH value was varied by HCl or NaOH solution. It need to be pointed out that the pH value in zeta experiments was same as the pH of PFC and PFC–PDMDAAC solution (0.01 mol/L as Fe), while the pH value in Fe(III) species experiments was that of the diluted water before coagulants were added. In this study, the working solution was the distilled water. The data obtained were summarized in Figs. 5–7.

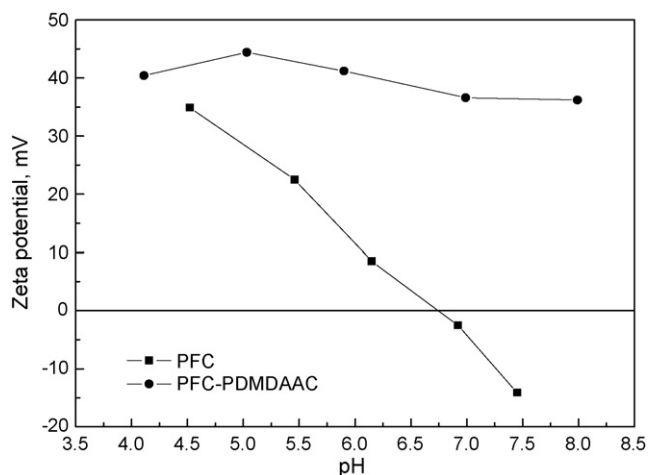


Fig. 5. Variations of the zeta potential with respect to pH for PFC–PDMDAAC and PFC.

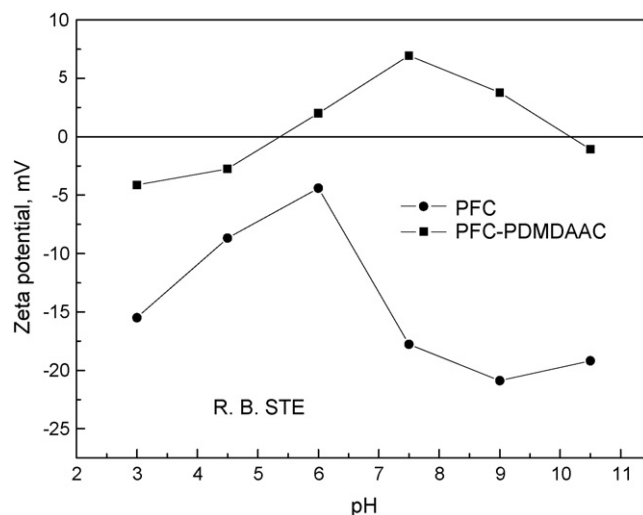


Fig. 6. Variations of the zeta potential with respect to pH for R.B. STE treated by PFC–PDMDAAC and PFC.

Zeta potential is a controlling parameter of the charge neutralization of coagulant and can usually be used to interpret the trend of coagulation efficiency. The zeta potential of PFC decreased from 34.9 to  $-14.1$  mV as the pH was increased from 4.5 to 7.5 (Fig. 5), while the zeta potential of PFC–PDMDAAC was practically constant in a wide range of pH (from 4 to 8). And the positive zeta potential of PFC–PDMDAAC was higher than that of PFC at the same pH. The dye in water has a negative charge on their surfaces [8], so the higher the positive charge on coagulant surfaces, the higher the color removal efficiency. The difference of the zeta potential between PFC and PFC–PDMDAAC may be one reason that PFC–PDMDAAC can remove dye efficiently in wider pH range than PFC.

The variation of zeta potential with respect to pH for R.B. STE treated by PFC–PDMDAAC and PFC is shown in Fig. 6. The dosage of PFC and PFC–PDMDAAC were 60 mg/L. It can be seen that Zeta potential of R.B. STE treated by PFC–PDMDAAC was more near zero potential than that of PFC at the same pH. This indicates that R.B. STE treated by PFC–PDMDAAC is less stable than that of PFC due to high positive zeta potential of PFC–PDMDAAC. Zeta potential of R.B. STE was affected by pH. For R.B. STE treated by PFC–PDMDAAC, an increase in pH resulted in a decrease in negative zeta potential value and an increase in positive zeta potential at  $\text{pH} < 7.5$ , while at  $\text{pH} > 7.5$ , positive zeta potential decreased. For R.B. STE treated by PFC, an increase in pH resulted in a decrease in negative zeta potential value at  $\text{pH} < 6.0$ , while at  $\text{pH} > 6.0$ , positive zeta potential decreased. Compared with Fig. 2, the variation of zeta potential according to solution pH is similar with the variation of color removal efficiency according to solution pH. This means that “charge neutralization” mechanism can be considered as one driving force for color removal.

The coagulation activity largely depends on the Fe(III) hydrolysis species for ferric salts coagulants. The Fe(III) species in PFC–PDMDAAC and PFC solution, measured and calculated by the Ferron method, were shown in Fig. 7. Previous studies has shown that ferron reagent can not react with PDM-

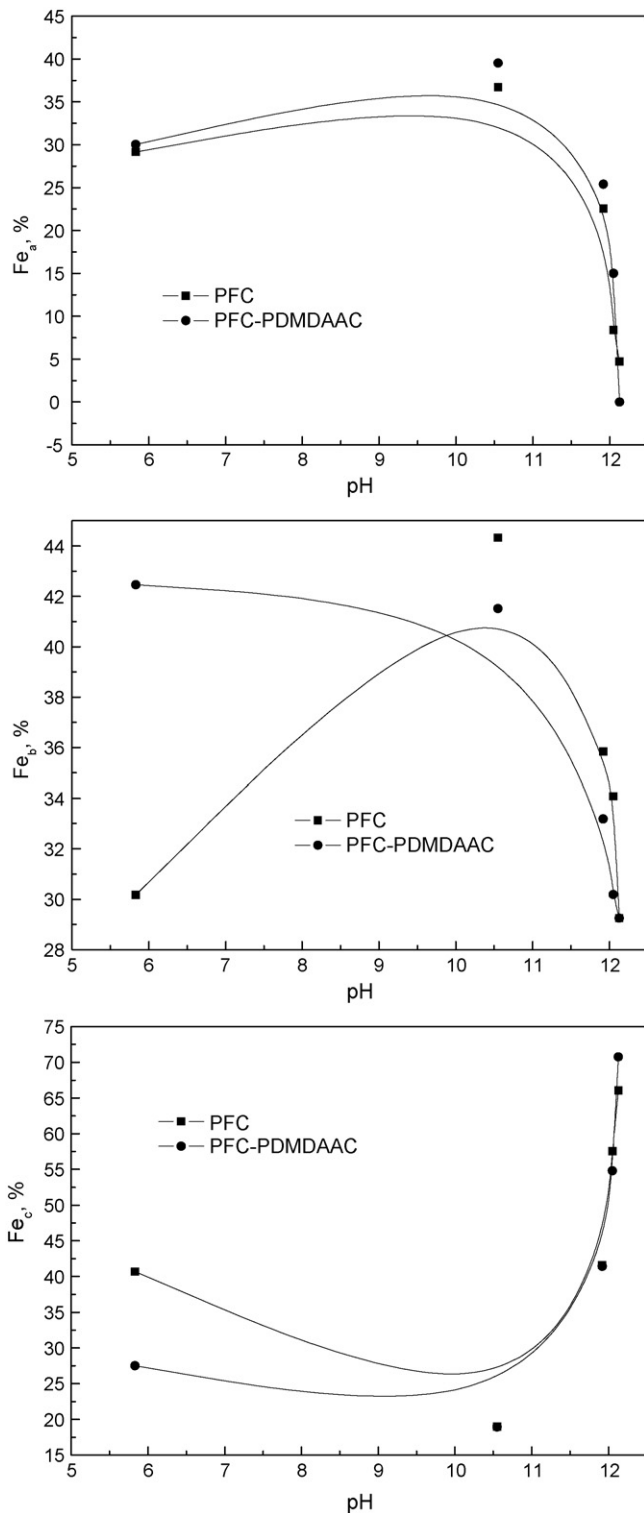


Fig. 7. Distribution of Fe(III) species in PFC and PFC-PDMDAAC.

DAAC [14], so the data, obtained by the ferron method, was only denoted the distribution of Fe(III) hydrolysis species. The variety of the content of Fe<sub>a</sub> and Fe<sub>c</sub> versus the pH of working solution observed for PFC and PFC-PDMDAAC were similar, but for PFC-PDMDAAC, the content of Fe<sub>a</sub> was higher than that for PFC, and Fe<sub>c</sub> was lower than that for PFC at the same pH (Fig. 7). The Fe<sub>b</sub> of PFC increased with the increase of pH

when pH was lower than 10.55 and then decreased rapidly with the increase of pH when pH was higher than 10.55, while the Fe<sub>b</sub> of PFC-PDMDAAC decreased with pH increasing during tested pH range. And the content of Fe<sub>b</sub> for PFC-PDMDAAC was higher than that for PFC under the condition of pH < 10.55. It was concluded from these results that the changes of Fe(III) species in PFC-PDMDAAC is smaller than that in PFC.

The above results suggest that shifting the pH of coagulation or working solution leads to the change of surface charge and Fe(III) species of PFC or PFC-PDMDAAC, which must results in the variation of color removal efficiency. For PFC, although the content of Fe<sub>b</sub>, which is known as the most active coagulating component in PFC [19], is not highest in the low pH range, the zeta potential is highest. Therefore the color removal efficiency is not the highest under this condition, but the dyes can be remove efficiently because of the charge neutralization of Fe(III) hydroxide which has high positive potential. Increasing solution pH will enhance the hydrolysis of iron salts and decrease the positive potential on the surface of Fe(III) hydroxide. However, in the neutral pH, although the charge neutralization of Fe(III) hydroxide in PFC is lower than that in the low pH, the dye can be removed through adsorption by Fe<sub>b</sub>. Therefore, the dye can be removed by charge neutralization and adsorption of Fe(III) hydroxide. In the high pH range, the negatively charged Fe(OH)<sub>3</sub> or the Fe(OH)<sub>4</sub><sup>-</sup> forms from iron salt before it could coagulate the dyes. The increase in the negative charges on the PFC surface increases the repulsion force between PFC and dyes. Thus the dyes cannot be removed efficiently because that the ability for PFC to neutralize and adsorb the negative charge on dyes became small. The mechanism of color removal of PFC-PDMDAAC is not same as that of PFC. D.B. HGL loses the stability by charge neutralization, because there is enough positive charge on PFC-PDMDAAC to neutralize the negative charge on dyes in all pH range. In addition, PDMDAAC with high molecular weight can bridge D.B. HGL, which has been lost their high negative charge density. Therefore, the color removal efficiency of D.B. HGL by PFC-PDMDAAC is higher than that by PFC. For R.B. STE, PDMDAAC can react with it [18], and then product can be coagulated by charge neutralize. Thus, PFC-PDMDAAC can remove R.B. STE efficiently. Moreover, the changing of zeta potential and the content of Fe<sub>b</sub> with pH for PDMDAAC is smaller than that for PFC, so the optimal pH range of PFC-PDMDAAC is wider than that of PFC.

## 5. Conclusions

The main conclusions from this work are:

- 1 The efficiency of PFC-PDMDAAC for treating dyes solution and textile wastewater is higher than that of PFC, PDMDAAC and PFC/PDMDAAC;
- 2 Synergy between PFC and PDMDAAC can increase zeta potential of PFC-PDMDAAC and reduce the effect of pH on zeta potential and Fe(III) species of PFC-PDMDAAC;
- 3 Mechanism of color removal by PFC and PFC-PDMDAAC is different. The charge neutralization and adsorption are main function of color removal by PFC; for PFC-PDMDAAC, D.B.

HGL lose the stability by charge neutralize, and then PDM-DAAC with high molecular weight can bridge D.B. HGL, while R.B. STE react with PDMDAAC to produce the insoluble compound which can be coagulated by charge neutralize of PFC–PDMDAAC.

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