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Investigating coagulation behavior of chitosan with different Al species dual-coagulants in dye wastewater treatment

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ABSTRACT

In this study, chitosan (CS) was used as a coagulant aid with different Al species ($Al_a/Al_b/Al_c$) to treat disperse yellow (DY) wastewater. The effects of coagulant dosages and initial pH on color removal, floc structure and regrowth were investigated. Results showed that the efficiency could be significantly improved with the addition of CS, due to the strong interaction between flocculants and Al species–dye aggregates. CS with the protonation of amino groups could combine with the azo bond and methyl of DY wastewater. Moreover, larger flocs would be generated as CS was added, especially at lower Al dosages. Based on the experimental results of this research, the initial solution pH had an impact on the hydrolysates of Al species and charge of CS, and further influence the color removal efficiency and floc properties. The color removal efficiency was relatively large at pH 6.0–7.0. The bigger flocs were formed under alkaline condition for Al_a and its dual-coagulant, and under acidic condition for Al_b/Al_c and the corresponding dual-coagulants, respectively. In coagulated water, the values of strength and regrowth also increased as CS was dosed, due to the introduction of bridging and charge neutralization.
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1. Introduction

Regulations concerning the administering of wastewaters have become more and more severe [1], and dye wastewater produced by textile, paper, leather and food industries should be concerned because of the color and the toxicity. For recent decades, more than 100,000 types of dyes are available commercially [2,3], most of them are toxic in the environment and make a threat to the ecosystem. Generally, some organisms of dyes, with synthetic origin and complex aromatic molecular structure [4], are decolorized by the destruction of dye molecules and the separation of dyes in wastewater treatment [5]. The destruction methods include chemical oxidation, photo-catalysis and biodegradation, whereas they are complex and require extensive energy to break down the dye molecules [2,6]. In addition, conventional processes are usually applied to separate the dyes, such as adsorption, coagulation/flocculation and membrane filtration, of which coagulation is the most widely used due to its simple operation and low cost [3,7,8]. Therefore, coagulants including inorganic coagulants, synthetic polymer flocculants and inorganic–organic dual-coagulants play important roles in dye molecule removal, and the efficiency of coagulation is affected by many factors such as the coagulant dosage and initial pH, etc.

In coagulation process, coagulants which achieve better flocs characteristics (in terms of size, strength and settling ability) and coagulation efficiency get more attention [9]. In addition, Al salt coagulants have been widely used, which include three species (Al_a, Al_b, and Al_c) according to a series of hydrolysis reactions [10]. However, inorganic coagulants have many drawbacks when they are dosed, such as larger dosage and residual metal. Therefore, it is desirable to develop a high efficient and non-toxic organic coagulant aid, which could reduce the dosage of coagulants. Chitosan (CS), as a natural, no secondary pollution and renewable bio-flocculant, has attracted more attention in dye wastewater treatment, which is produced from crustacean shells [11,12]. The molecular weight affects the solubility of CS and coagulation performance, and the solubility decreases with the increase of molecular weight [13]. Previous studies were more focused on the coagulation performance of CS as coagulant [14], and the application of CS with polyaluminum chloride in water treatment [13], but the study of three Al species-CS dual coagulants is insufficient. The amino groups of CS molecule will enhance its activity and charge neutralization for Al species–dye aggregates [15], and long chain has high affinity to adsorb dye molecules [16]. Therefore, considering the advantages of CS and operating cost [17,18], the comparative study of coagulation mechanisms and interactions between

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Table 1
The physicochemical properties of DY water sample.

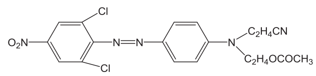
Type	Chemical structure	Chromophore	Wavelength (nm)	Zeta potential (mV)	pH
Monoazo		–N=N–	465	−23.77 to −25.44	8.21 ± 0.20

Table 2
The physicochemical properties of CS.

Type	Deacetylation degree	Molecular weight	Viscosity (100 rpm at 25 °C)
CS	90 ± 5%	590–630 kDa	57.1 ± 2.3 mPa s

three Al species-CS dual coagulants under different conditions was discussed.

The aim of this study is to investigate the effect of different dosages and pH on coagulation performance, in accordance with color removal efficiency and zeta potential, and floc properties which related to floc size, strength, recoverability, regrowth rate and structure. In general, these studies provide theoretical guidance for practical applications.

2. Materials and methods

2.1. Dyeing wastewater preparations

The Disperse Yellow S-2RFL (DY) wastewater, with negative charges in aqueous solutions, was prepared by dissolving dye particles in tap water to make the concentration of 100 mg/L. The physicochemical properties of water sample were showed in Table 1.

2.2. Coagulants

Three Al species coagulants were used in this study. The concentration of Al_a stock solution was 1.0 g/L by dissolving $AlCl_3 \cdot 6H_2O$. To get Al_b, the amount of NaOH was gradually added to $AlCl_3 \cdot 6H_2O$ solutions with the basicity of 2.4 [19]. The synthesis method of Al_c is similar to that of Al_b, but the synthesis temperature is higher [19]. The proportion of different Al species is related to the Al-ferron complexation timed spectrophotometry method [20]. Therefore, the content of Al_b and Al_c could be reached to 95.47% and 83.69%, respectively. CS was supplied by Sinopharm Chemical Reagent Co., Ltd. In order to facilitate the preparation of the CS solutions, CS diluted with 1% (HCl w/w) solution to make the concentration of 1.0 g/L. The property parameters of CS are listed in Table 2.

2.3. Coagulation–flocculation experiments

Before coagulation, the initial solution pH was controlled with 0.1 M HCl and 0.1 M NaOH. The experiments on dye coagulation were conducted on a jar test apparatus at room temperature (ZR4-6, Zhongrun Water Co. Ltd., China). A coagulation procedure was the same for the changes of dosages and pH, which was set as follows: the mixtures were stirred at 200 rpm for 1.5 min and 40 rpm for 15 min, followed by sedimentation for 20 min. In addition, coagulants were dosed in succession during the rapid stirring at 200 rpm, and an interval between different Al species and CS was 30 s. The supernatant was obtained for the measurement of absorbance (TU-1810 UV/VIS spectrophotometer, Puxi Co. Ltd., China) and zeta potential (Zetasizer Nano ZS, Malvern, UK). Moreover, color removal efficiency was calculated by absorbance values according to the following equation [21]:

$$\text{Color removal efficiency (\%)} = (A_0 - A)/A_0 \times 100\% \quad (1)$$

In this equation, A_0 and A were absorbance values of raw water sample and treated water sample, respectively.

2.4. Image captures of flocs

In this study, an optical microscope (Olympus, Japan), which equipped with a camera and connected to a PC, was used to observe floc characteristics. The solution after coagulation process containing flocs was taken by plastic head dropper, and dripped a little on a glass slide, and then covered with a cover slip. The images were captured at magnifications of 100×.

2.5. PDA measurements

The flocs in real-time were monitored by Photometric Dispersion Analyzer 2000 (PDA 2000, Malvern, UK) [22,23]. In order to investigate the effects of floc breakage and regrowth, a part of program was similar to coagulation without the stage of sedimentation. The stirring rate of 200 rpm for 5 min was introduced after the stage of slow stirring, and then another 40 rpm was reintroduced for 20 min to regenerate flocs.

The flowchart of PDA 2000 during coagulation was showed in Fig. 1. The ratio value (FI), which determined by the time-weighted average of steady state ratio value, was strongly related to floc size [24]. In addition, FI values also increased as flocs grew. The time-weighted ratio variance (TWV) of the steady state ratio value signified the distribution range of floc size, and bigger TWV illustrated that flocs have opener and more porous structure. They were calculated as follows:

$$FI = \sum_{i=1}^{N} (\text{ratio}_i \cdot \text{time}_i) / \sum_{i=1}^{N} \text{time}_i \quad (2)$$

$$TWV = \sqrt{\sum_{i=1}^{N} \left[(\text{ratio}_i - R)^2 \cdot \text{time}_i \right] / \sum_{i=1}^{N} \text{time}_i / R} \times 100\% \quad (3)$$

Floc breakage factor (B) and recovery factor (R) were well established parameters for describing floc strength ability to resist shear force, and recoverability to make broken flocs regenerate, and they can be calculated respectively as follows [25,26]:

$$B = R_2/R_1 \times 100 \quad (4)$$

$$R = (R_3 - R_2)/(R_1 - R_2) \times 100 \quad (5)$$

In the above formulas, R_1, R_2 and R_3 are the floc ratio of the first steady stage, after floc breakage phase and new steady stage after regrowth, respectively.

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