

The Treatment of High Concentration Dyeing Wastewater with Pulsed Current Electrocoagulation

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Abstract

In this study, a small pulsed current electrocoagulation device was used to treat high concentration dyeing wastewater from a specific dyeing mill, and the effects of the electrode materials, reaction time, voltage, pH value, and aeration on the results of the treatment were examined. The results showed that under the following operational conditions: electrode materials were iron electrodes, time period was 15 min, voltage was 120 V, and initial pH was approximately 6, the removal rates of the COD, ammonia nitrogen, and color were 79.45%, 23.89%, and 87.50%, respectively. On this basis, a pulsed current electrocoagulation device, with a handling capacity of 0.5 m³/h, was used to conduct a pilot plant test for a period of one month. The results showed that the effluent quality (COD 1217.4 mg/L and NH₄⁺-N 358.2 mg/L on average) of the high-concentration dyeing wastewater, whose COD and NH₄⁺-N concentrations were 5328 mg/L, 595 mg/L, respectively after the treatment of a pulsed current electrocoagulation reactor, was superior to the effluent quality (COD 1400 mg/L and NH₄⁺-N 450 mg/L) of the mill's actual pre-treatment system (flocculation-anaerobic treatment-acidification), and fully reached the influent requirements of the subsequent aerobic treatment. The results of this study showed that pulsed current electrocoagulation reactors may be effectively used for the pre-treatment of high concentration dyeing wastewater due to the observed advantages, such as good treatment effects, small investment, and economical space occupation.

Keywords: pulsed electrocoagulant, high concentration dye wastewater, pre-treatment, investment

1. Introduction

According to the incomplete statistics, China produces nearly 100,000 tons of dye each year. By calculating the ratio of each ton of dye producing 7 tons of wastewater (Yang, Jia, & Yang, 2006), it can be estimated that China produces nearly 700,000 tons of dyeing wastewater. As a result, the wastewater emissions, and the total amounts of pollutants of the dyeing industry, rank second and fourth among the industrial departments nationwide, and the dyeing industry has become one of the major water pollution industries in China.

High concentration dyeing wastewater has drawn attention internationally, due to its complicated water quality, high concentrations of COD, and poor bio-degradability. Many treatment methods have been proposed (Lu, Liu, Yang, & Chen, 2009; Marmanis, Dermentzis, Christoforidis, Ouzounis, & Moutzakis, 2015; Srinivasan & Viraraghavan, 2010). The purification techniques can be classified into three types: physical methods, chemical methods, and biological methods. However, traditional methods such as physical adherence, chemical oxidation, and biological oxidation have not proven to be sufficient (Hao, Kim, & Chiang, 1999; Sakalis, Mpoulmpasakos, Nickel, Fytianos, & Voulgaropoulos, 2005). The main method which has currently been adopted is a combined treatment technique which includes physico-chemical, chemical, and biological methods. Since the results of the pre-treatment of a combined treatment technique directly determine the influent conditions of the biochemical process, the pre-treatment is especially important in the whole course of the treatment. The pre-treatment methods of high concentration dyeing wastewater mainly include the following methods: physical (coagulant sedimentation and coagulant gas floating), chemical (advanced oxidation and electrochemical processes), and biological (anaerobic oxidation). Among these methods, the electrochemical process, due to its advantages of versatility, high energy efficiency, high degree of automation, low cost, and economic space occupation, has become the focus of research studies in recent years (Dermentzis, Marmanis, Valsamidou, Christoforidis, & Ouzounis, 2011; Gutiérrez

& Crespi, 1999; Lorimer, Mason, Plattes, Phull, & Walton, 2001). Electrocoagulation (EC), also referred to as electric flocculation, which is an electrochemical process which coagulates colloidal substances in water by using the flocculation of hydroxides of aluminum or iron, which are generated by a solution of aluminum or iron soluble electrodes, to purify the water (Ofir, Oren, & Adin, 2007), is one of electrochemical oxidation methods.

The pulsed current electrocoagulation process has such advantages as low energy consumption, and anodes which are not susceptible to deactivation, when compared with regular electronic flocculation processes. Additionally, while removing a portion of the COD, the biochemicalization performance of the wastewater may be enhanced at the same time. Therefore, significant improvements in the biochemical treatment efficiency of the organic wastewater can be achieved. The aforementioned advantages of the pulsed current electrocoagulation process in the treatment of dyeing wastewater have drawn extensive attention. Among the many studies which have been conducted, some have focused on the conditions for the use of the pulsed current electrocoagulation process, and have explored its effects in the treatment of dye wastewater (Chen et al., 2011; Khandegar & Saroha, 2013; Lin & Peng, 1996; Manenti et al., 2015; Moreno-Casillas et al., 2007). Other studies have combined the pulsed current electrocoagulation with other processes. All of the previous studies have achieved certain results. For example, Bayramoglu et al. used iron and aluminum as electrodes for the study of the treatment of textile wastewater by electrocoagulation. The results showed that when the solution was acidic, the treatment results of using aluminum electrodes to remove COD and turbidity exceeded those of the iron electrodes. However, when the solution was neutral or slightly alkaline, the treatment results of the iron electrodes exceeded those of the aluminum electrodes (Bayramoglu, Kobya, Can, & Sozbir, 2004). Also, Murat Eyvaz et al. explored the effects of electrocoagulation on dyes, as well as the removal rate of TOC, and adopted direct and alternate pulsed currents to treat dispersed dyes. These experiments showed that the alternate pulsed currents outperformed the direct currents, in terms of both the removal rate and energy consumption (Eyvaz, Kirlaroglu, Aktas, & Yuksel, 2009). Yee et al. studied the degradation of tetrachloroethylene and 2, 4-dichlorophenol, by coupling the discharge-produced plasma by high voltage pulse and aerobic biological process. The results of this research study showed that a front-end electro discharge treatment could effectively dechlorinate the wastewater, and the removal rate reached over 55%, which was conducive to subsequent biochemical treatments (Yee, Chauhan, Yankelevich, Bystritskii, & Wood, 1998).

In the current research study, using a high concentration dyeing wastewater of a specific printing/dyeing mill as the object of study, the treatment effects of a pulsed current electrocoagulation on high concentration dyeing wastewater were explored. Also, the effects of various conditional parameters of the treatment effects were examined, and the best gained parameters were used to conduct a pilot plant test over a one-month period.

2. Experiments

2.1 Source and Quality of the Wastewater

The wastewater in this study was obtained from the mixed wastewater of cloth and yarn washing, from a plant in Foshan, Guangdong Province, China. Its source is as shown in Figure 1.

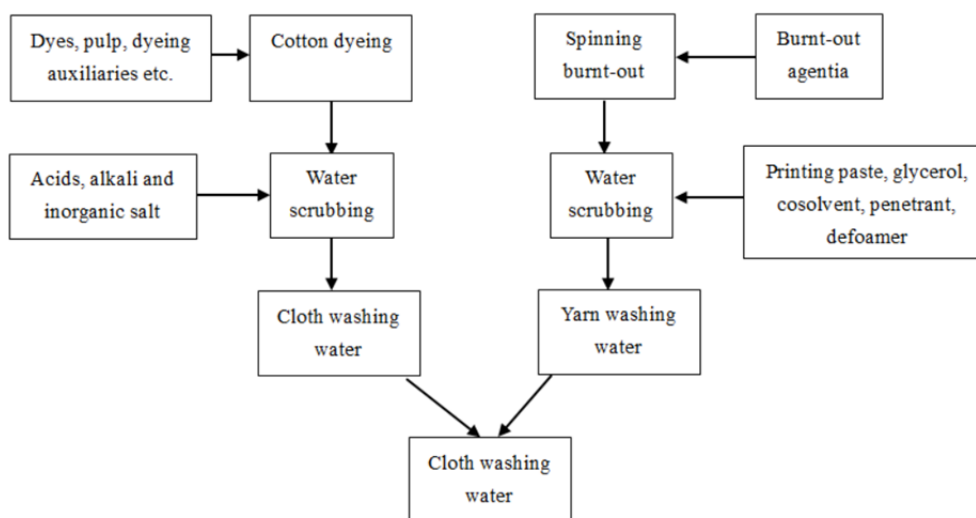


Figure 1. Source of the wastewater for the experiment

The cloth washing wastewater was produced when cotton cloths were washed after dyeing, and mainly included dyes, pulp, dyeing auxiliaries and fiber impurities, oiling agents, acids, alkali, inorganic salt, and so on. The concentrations of COD were 1500 to 3000 mg/L, and the ammonia nitrogen was approximately 150 mg/L, with a pH between 8 and 11. The yarn washing wastewater was produced by the mechanical and continuous reduction clearing of the yarn after going through a burnt-out process. In the burnt discharge, burnt-out agents such as acid-proof printing paste, glycerin, cosolvent, acid-proof penetrant, and acid-proof defoaming agents were used (in this particular plant, the parts of the textiles with printing were heated after the dilute vitriolization, and the water was evaporated to become concentrated sulphuric acid, in order for the cotton fibers to be carbonized. The cellulose fibers were hydrolyzed to form the printing. Therefore, the wastewater contained a large amount of refractory organic pollutants, with COD levels at approximately 10000 mg/L, ammonia nitrogen between 400 and 700 mg/L, and a pH of approximately 4. After the two were mixed, the COD was approximately between 4000 and 6000 mg/L, ammonia nitrogen approximately 300 to 600 mg/L, and pH approximately 6.

Due to the fact that the wastewater had high concentrations of COD and ammonia nitrogen, it would have been difficult for the effluent to meet the standards if the wastewater was simply directly discharged into the comprehensive sewage treatment system. Therefore, the plant separately pre-treated the wastewater beforehand. Fig. 2 illustrates the actual process of the treatment of the high concentration dyeing wastewater in the plant. From this figure, it can be seen that the method of pre-treatment was mainly a materialization-anaerobic-level 3 acidification process. A large amount of chemical reagents and PAM were used in the operation process, which produced a large amount of sludge. Therefore, a level-2 sludge thickener was set up in the sewage treatment station. Following the high concentration dyeing wastewater being treated by the system, the effluent pH was approximately 7, COD approximately 1400 mg/L, and ammonia nitrogen approximately 450 mg/L.

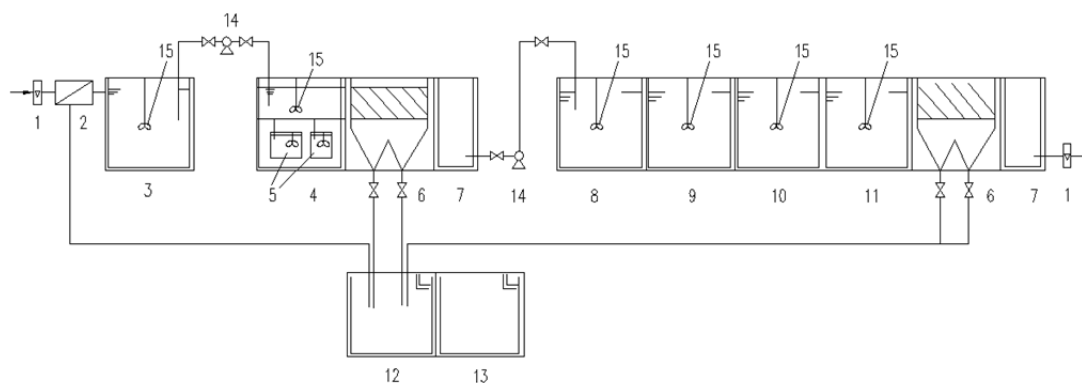


Figure 2. Process flow diagram of the pretreatment of textile wastewater with high concentrations

1.Flow meter;2.Grille;3.First deposition and adjusting pool;4.Physical and chemical reaction pool;5.Dosing tank;6.Sedimentation tank;7. Water reservoir;8.Anaerobic tank;9.Primary acidification pool;10.Secondary acidification pool;11.Tertiary acidification pool;12.Sludge concentration tank;13.Sludge concentration tank 2#;14. Water pump;15.Stirrer

2.2 Testing Apparatuses

2.2.1 Laboratory Testing Apparatus

In this test, a high-voltage pulsed power supply was adopted, obtained from the Guangdong Wstation Environmental Protection Technology Co., Ltd., which could provide a pulsed DC power supply. The electrodes were made by the author himself. Fig. 3 shows the structural diagram.

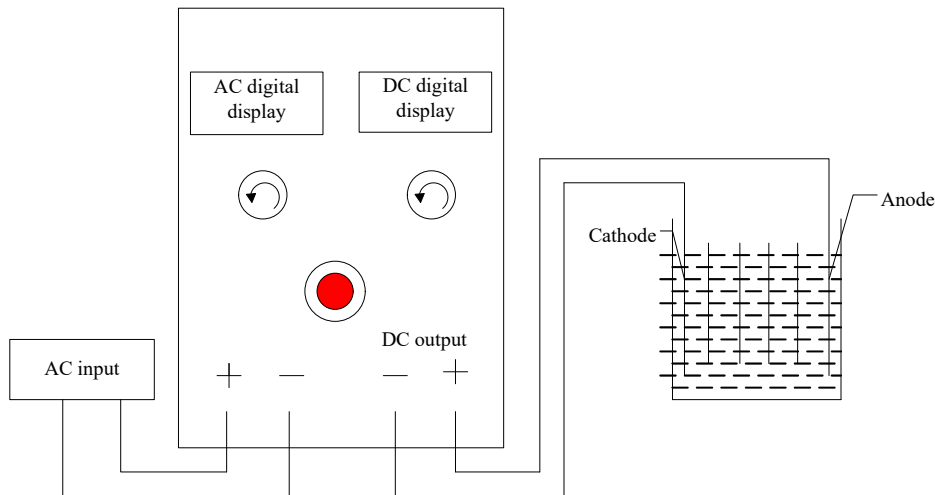


Figure 3. Device for the lab test

The device had the following characteristics:

Power supply: AC input, voltage 220 V; DC output, voltage 110 to 200 V, and electric current 0 to 20 A.

Electrode plate: The electrode plates were fixed by a fixed support, with a round center made of UVPC. The support could be detached to replace the electrode plates. There were six plates (Length×width×thickness=100 mm×80 mm×2 mm; Distance between the plates: 12 mm).

2.2.2 Pilot Plant Test Equipment

A high-voltage pulsed current electrocoagulation reactor, with a handling capacity of 0.5 m³/h (model EC-VF-A-Q/WJS 1-2012, made by Guangdong Wstation Environmental Protection Technology Co., Ltd.), was adopted for the pilot scale test. This reactor mainly consisted of two parts: a main engine realizing the electrocoagulation treatment of the wastewater (1.00 m x 0.65 m x 1.30 m in size), and a system control cabinet for the real-time control and adjustment of the treatment process conditions and functions. The method of the plate installation in the main engine was parallel, with an equidistant arrangement of 15 iron plates, the size of each plate being 500 mm in length x 240 mm in width x 6 mm in thickness, and the plate distance was set at 10 mm. The system's power supply was a AC380V x 3 phase 5 wire style, and the DC power output was: 0 - 500 V, 0 - 400 A (MAX). The power supply was operated by a rated voltage or a rated current, and the output had an automatic positive/negative switch function, with the ability of the time being automatically adjusted. Fig. 4 illustrates a section of the equipment.

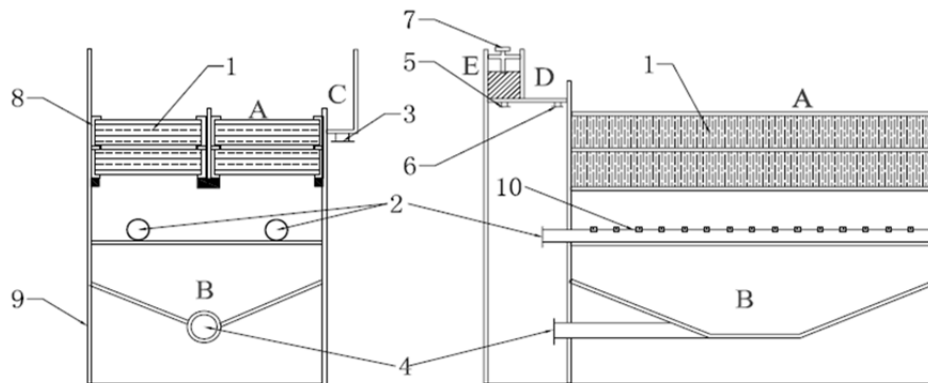


Figure 4. Profiles of the pulse electro-coagulation reactor

1. Plate; 2. Aerator pipe; 3. Water inlet; 4. Slag discharge; 5. Product water outlet; 6. Scum outlet; 7. Effluent level regulator; 8. Plate fixing device; 9. Equipment shell; 10. Aeration cap.

The five areas include the following: A. reaction area; B. sedimentation area; C. water distribution area; D. scum collection area; E. outlet area.

2.3 Tests and Analysis Method

2.3.1 Steps of the Laboratory Tests

Some impurities were attached to the electrodes after each of the tests. Therefore, in order to guarantee that all of the experiments were conducted under the same conditions, electrode activation was conducted before each experiment. This activation step used sand paper to polish the surface of the electrodes, and then the electrodes were soaked in a 1M H₂SO₄ solution for several minutes. Following the activation, 2.5 L of dyeing wastewater was added to the reaction tank, and an electrolysis experiment was conducted after the parameters were changed. Then, at the end of the reaction, the reactor was kept stationary for approximately 20 minutes. The solution became layered following the scum of the reactor floating up to the surface, and the flocculate settling. The clear liquid in the middle was then taken for filtration, and the COD, ammonia nitrogen, and color were measured.

2.3.2 Steps of the Pilot Plant Tests

Prior to the water being introduced, a sulfuric acid solution and slaked lime water were added in the acid and alkali boxes, respectively. Then, the system was turned on, the influent pH was adjusted, and parameters such as voltage and current were set up. At this point, the inlet valve was opened, and the flow was regulated so that the wastewater took its natural course from the water box at the higher level, to the water distribution area of the equipment. The lift pump was then started (starting of the pump was controlled by a water level gauge), the current was flocculated, and the positive and negative conversion time was set up, which started the reaction. The operating system was debugged with reference to the results of laboratory testing. After the system was stabilized, 500 mL of electrocoagulation effluent was taken every two hours. Following a stationary period of 20 minutes, the clear liquid in the middle was extracted for the measurement of various indexes of water quality, thereby acquiring the treatment effects of the device in continuous operation.

2.3.3 Analysis Method

The water quality indexes in this study were monitored according to the standard method provided in the Monitoring and Analysis Methods of Water and Wastewater, 4th edition (State Bureau of Environmental Protection, 2002), State Bureau of Environmental Protection, 2002.

3. Results and Discussions

3.1 Effects of the Electrode Materials on the Treatment Results

In this study's experiment, 2.5 L of wastewater were taken, and the effects of three electrode materials (aluminum, iron, and stainless steel) on treatment results, under the conditions of a voltage 120 V, and an electrocoagulation time of 15 minutes, were examined. The results are shown in Figure 5.

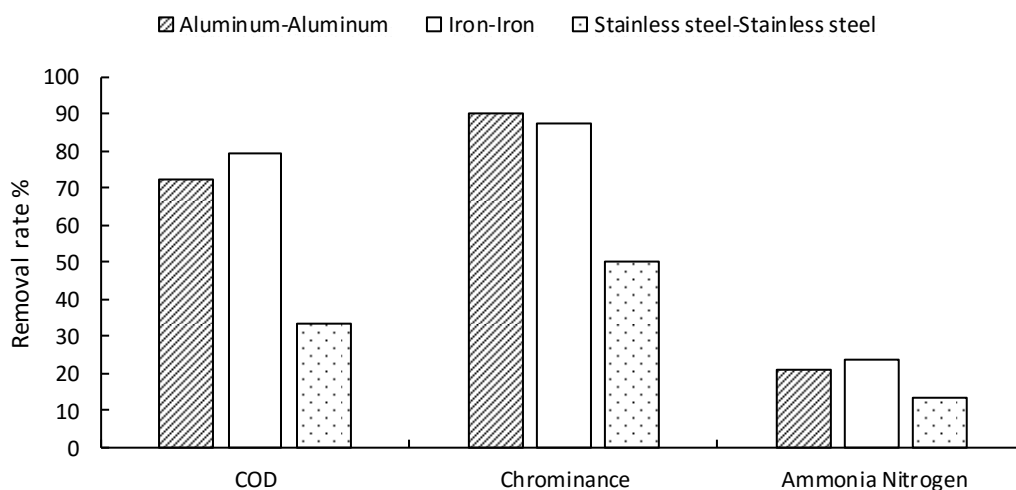


Figure 5. Effects of electrode materials on dyeing wastewater treatment

In Figure 5. The following can be seen: under the same conditions, the iron electrodes out-performed the other two electrodes in terms of COD and ammonia-nitrogen removal rates. Generally speaking, the wastewater had three effects in the electrolytic process. These included an electrolytic oxidation-reduction, electrolytic

flocculation, and electrolytic flotation (Mohan, Balasubramanian, & Basha, 2007). The flotation and flocculation effects of the iron electrode out-performed those of the other two electrodes. The results of a large amount of international studies regarding electrocoagulation mechanisms have determined that when an iron electrode reaction is adopted, the mechanisms of pollutant removal by the electrocoagulation consumption iron electrodes mainly include the following: complexation, electrostatic adherence, chemical regulation, and sedimentation and flotation of the floc surfaces of $\text{Fe}(\text{OH})_2$ and $\text{Fe}(\text{OH})_3$ (Edwards, Fendley, & Lunsford, 2006; Patrick, Jean-Francois, & Guy, 2007). Iron electrodes were adopted for testing in the subsequent reactions.

3.2 Effects of Reaction Times on the Treatment Results

The effects of reaction times on the treatment results were examined at a voltage of 120 V, as shown in Figure 6. It can be seen in Fig. 6 that the removal of COD by the electrocoagulation equipment was completed in a short time. The COD removal rate after 15 minutes of reaction time reached 78.42%. With the increase of time, the removal rate increased only slightly. The color removal rate was found to be the highest at 15 minutes. However, with the increase of time, the color basically remained unchanged. The removal rate of the ammonia nitrogen was determined to be the highest at 12 minutes, and subsequent reaction had only a slight fluctuation.

These results are probably due to the fact that in the period when the reaction starts the effects of flocculation, the oxidation and flotation are obvious. With the proceeding of the reaction, the pollutant concentration in the wastewater drops, while the degradation-resistant secondary pollutants from the electro-oxidation are produced. Also, with the proceeding of the reaction, insoluble substances attached to the electrode surfaces are generated in the reactor, which tends to enclose the places near the anodes. If the electrolysis time is too lengthy, overly abundant amounts of insoluble substances will also be produced which wrap up the colloidal particles. Then, these particles will lose absorption activity, which increases the resistance of electrodes, and reduces the flocculation performance, slowing the oxidation. The oxidation capacity of the electrodes after 15 minutes will be inadequate to oxidize the secondary pollutants with certain colors. Through the above analysis, and considering the anode passivation and electricity consumption, it was determined in this study that the electrolysis time should be controlled to within 15 minutes.

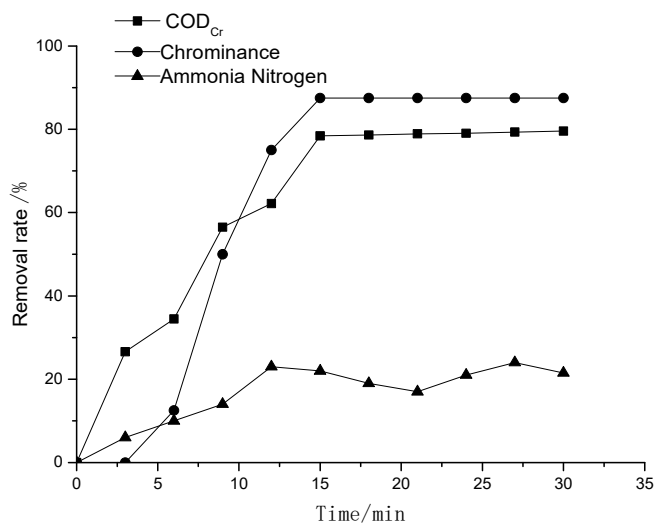


Figure 6. Effects of time on removal rates

3.3 Effects of Electrolytic Voltage on the Treatment Results

In this experimental study, 2.5 L of wastewater were taken to observe the effects of the iron electrodes' voltage on the treatment, under the condition of 15 minutes of electrocoagulation. The results are shown in Fig. 7. It can be seen in Fig. 7 that the COD removal rate when the voltage rate was 110 V was relatively low. When the voltage increased to 120 V, the removal rate was higher, and with the increase of voltage, the removal rate increased slightly. The color removal rate basically remained unchanged. The removal rate of ammonia nitrogen increased slightly with the increase of the voltage.

In summary, there was found to be a certain linear relationship between the current density in the course of the electrocoagulation and the cell voltage. With the increase in the voltage, the current density increased accordingly (Wang, Zhang, Chen, Ren, & Pei, 2002). When the voltage was set at 120 V, and the initial current was 5A, the

water produced more hydroxides of iron and bubbles than when the voltage was set at 110 V (current 2A). The electrochemical action was enhanced, COD and ammonia nitrogen removal rates increased, coloration substances were basically oxidized at 110 V, and the coloration substances in the secondary pollutants produced could barely be removed by the flocculation, flotation, and oxidation. After the voltage was increased, an oxygen evolution reaction appeared, and the direct and indirect electro-oxidation of the organic pollutants was inhibited. The main pollutants were very susceptible to the electrochemical reaction, and were removed when voltage was 120 V. However, the increase of the removal rate of the COD and ammonia nitrogen was not obvious. Therefore, considering the removal effects of voltage on the three pollutants and energy consumption, 120 V was chosen in this study as the best voltage for treating the dyeing wastewater.

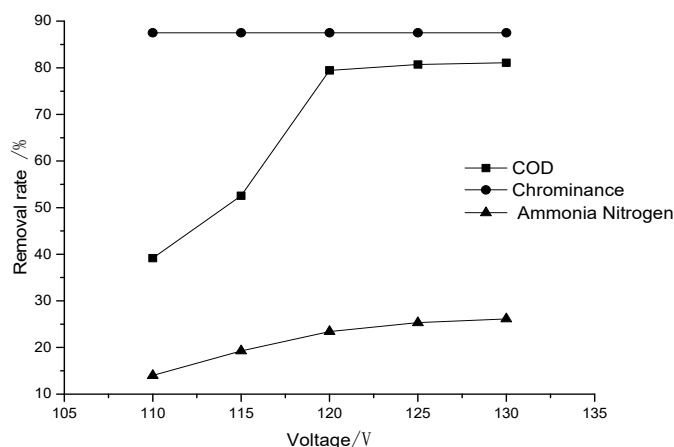


Figure 7. Effects of voltage on the removal rates

3.4 Effect of the Initial pH on the Treatment Results

In this experimental study, 2.5 L of wastewater were extracted to observe the effects of pH on the treatment, under the condition of 120 V voltage and 15 minutes of electrocoagulation. Fig. 8 illustrates the test results.

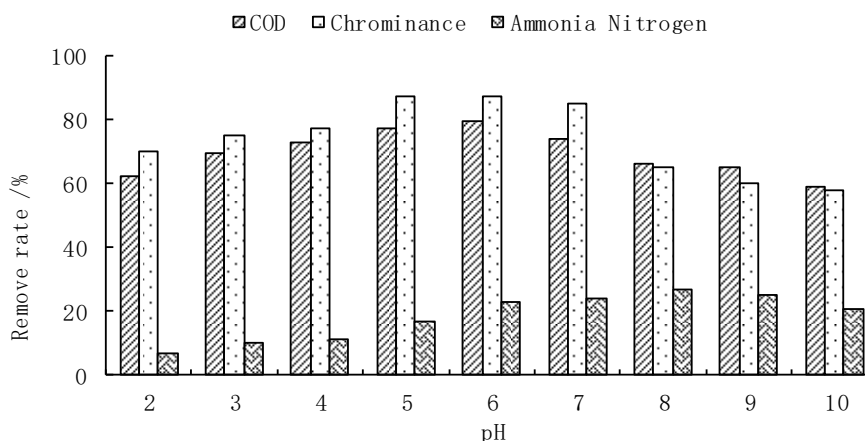


Figure 8. Effects of the initial pH on the removal rates

It can be seen in Figure 8 that too large or too small pH values caused reductions in the COD removal and color removal rates, and the removal results were the most effective when the pH is between 5 and 7. The removal rate of ammonia nitrogen increased with the increase of pH, and then dropped, and the removal effect was the best when the pH was between 6 and 9.

The results mentioned above were due to the fact that, under the condition of being overly acid, the iron plates reacted with the H^+ in water, and existed in the form of iron ions, thereby losing the flocculation effects. When the pH value was too high, the plates easily passivated, and the Fe^{2+} and Fe^{3+} sedimented in the form of hydroxides, and was unable to produce polynuclear poly iron carbonyl complex ions for charge neutralization and adherence fiber bridges. Therefore, when the pH was controlled at between 5 and 7, the removal effect of color and COD was

improved. With comprehensive consideration of the COD, color, and ammonia nitrogen removal effects, as well as the cost, pH regulators were not added in this study's tests. Also, the appropriate pH was controlled by adjusting the mixing ratio of the cloth and yarn washing wastewater.

3.5 Effects of Other Factors on the Treatment Results

3.5.1 Effects of the Aeration Methods on the Removal Rates

With the voltage set at 120 V, the effects of the aeration methods on the removal rates were observed, with the iron electrodes reacting for 15 minutes with the different aeration methods. The results are shown in Fig. 9.

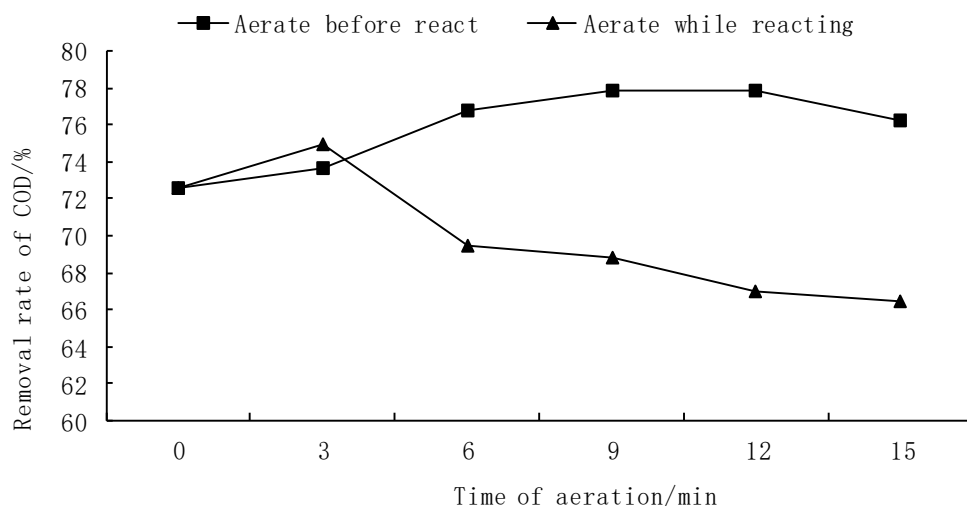


Figure 9. Effects of aeration times on the removal rates

It can be seen in Fig. 9 that when the aeration was completed before the reaction, and with the increase of the aeration time, the COD removal rate increased, and then basically remained unchanged after 9 minutes. When the aeration time was 3 minutes in the reaction, the removal rate obviously rose, and when the aeration time increased to 6 minutes, the removal rate dropped substantially. At the end of the test, the amount of flocculating constituents was obviously reduced when compared with no aeration. Therefore, it was determined that aeration should be conducted for a certain period prior to the reaction, and then the electrocoagulation should be conducted, in order to improve the rates of the pollutant removal.

3.5.2 Stability Test of the Flocculating Constituents

In this study, a water sample which had been treated under the most ideal conditions was collected, and placed in a stationary position for 10 minutes. Following this stationary period, it was observed that a layer of compact sediment was produced at the bottom of the wastewater. For the purpose of testing the stability of the sediment, the scum was removed from the wastewater sample which had not been moved, and then the wastewater was stirred for a certain time before being left undisturbed for one hour. At this point, clear liquid was taken from the sample water for testing. The COD had been changed from 1,107 mg/L to 1,098 mg/L, or basically no changes were observed. It was thereby concluded in this study that, following the treatment, the sediments did not emit any pollutants.

3.6 Pilot Plant Tests of the Pre-Treatment of High Concentration Dyeing Wastewater by a Pulsed Current Electrocoagulation Reactor

A pulsed current electrocoagulation reactor with a handling capacity of 0.5 m³/h (model EC-VF-A-Q/WJS 1-2012) was adopted as the equipment for the pilot plant tests in this study. Fig. 4 illustrates its structure.

The rated voltage was 120 V, current changes were between 3 and 8A with the change of water quality, and the wastewater was treated continuously for 32 days. It should be noted that the yarn and cloth washing wastewater of the plant were basically discharged every other day, and sometimes only cloth washing wastewater or only yarn washing wastewater was discharged, due to the different order conditions. Therefore, the concentration levels changed for the COD and ammonia nitrogen in the inlet water. Each day of the testing period, the COD and ammonia nitrogen levels in the outlet water were measured three times, with three groups for each time, and the average values were taken. Figs. 10 and 11 illustrate the results of the testing.

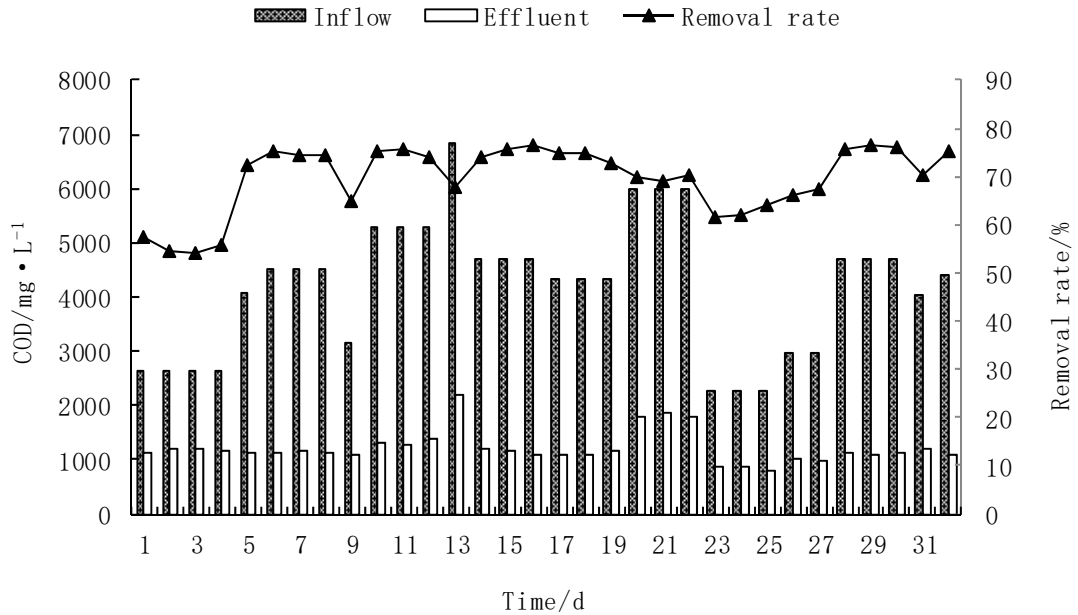


Figure 10. Results of the pilot-scale experiment for COD levels

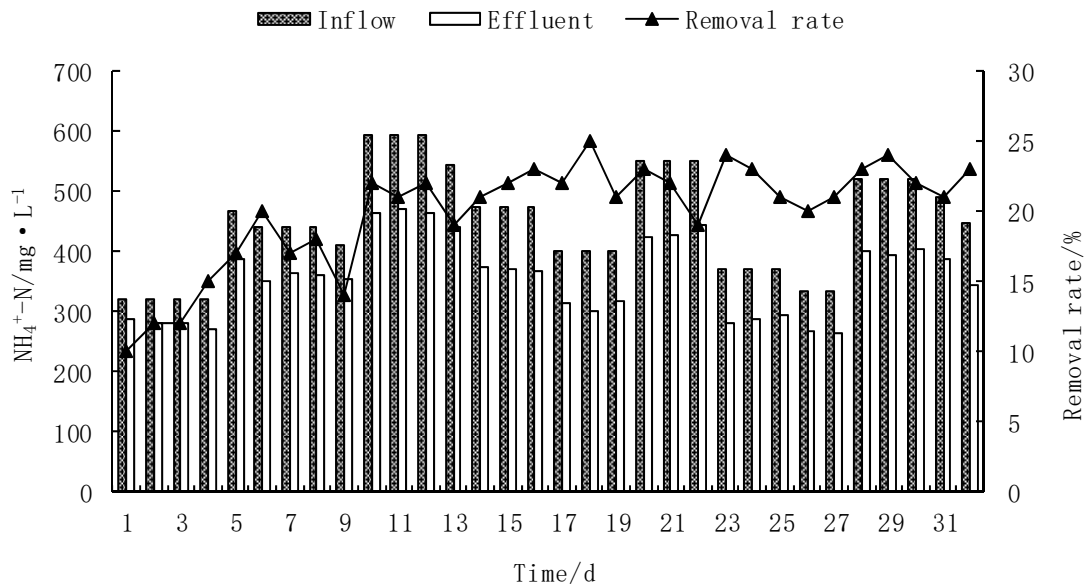


Figure 11. Results of the pilot-scale experiment for NH⁴⁺-N levels

As can be seen in Figs. 10 and 11, the initial period of treatment was a debugging period, with the COD removal rate was only approximately 55%, and the ammonia nitrogen removal rate at approximately 15%. After five days of operation, the COD removal rate fluctuated between 65 and 75%, and the ammonia nitrogen removal rate fluctuated at approximately 20%. The removal rate in the 9th and 13th day declined about 5%, that's because after several days' reaction, the plates passivated, put the plates in again after washed, it can be seen that the removal rate returned to normal. Therefore, it was found that the pilot plant tests varied from the best results of the laboratory tests. This was due to the fact that in laboratory tests, the following applied: various conditions could be well grasped, the same batch of wastewater was treated, and the plates were cleaned after each test.

4. Conclusions

(1) The most effective operational conditions which were concluded from the factor effect tests in this study were as follows: the electrode materials were iron electrodes; time was 15 minutes; voltage was 120 V; and the initial pH

was approximately 6. The wastewater which was treated under these conditions had a COD concentration of 5,328 mg/L, ammonia nitrogen concentration of 595 mg/L, and color of 400. The removal rates of the COD, ammonia nitrogen, and color were 79.45%, 23.89%, and 87.50%, respectively. It was determined that when appropriate aeration was completed before the reaction, the results of the treatment improved.

(2) The sediments which formed after treating the high concentration dyeing wastewater by pulsed current electrocoagulation were relatively stable. They were without reverse solution, which was conducive to later sludge treatment.

(3) The quality of the effluent after the treatment of high concentration dyeing wastewater through the pulsed current electrocoagulation reactor (on average COD 1217.4 mg/L, NH_4^+ -N 358.2 mg/L) was found to be superior to that of the effluent quality of the actual pre-treatment system of the plant in this study (COD 1400 mg/L, NH_4^+ -N 450 mg/L), completely meeting the influent requirements of the subsequent aerobic treatment system, and featured such advantages as a small investment, and economical space occupation.

Acknowledgments

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