

## Evaluation of X-3B Dye Removal and COD Reduction from Dyeing Wastewater Using Iron Wastes and Coagulation Process



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### ABSTRACT

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#### Keywords:

*COD, coagulation, decolorization, dyeing wastewater, zero valent iron*

This study was conducted to investigate the ability of zero valent iron (ZVI) available in iron solid wastes with coagulation processes to reduce (COD) content and remove the color from dyeing wastewater, and the mechanism of iron particles process for color degradation and COD reduction was inquired. (X-3B) dye was synthesized for dyeing wastewater. Using 440 L/hr of air flow and mixing at 500 rpm for 20 minutes, then 200 rpm for 10 minutes, while using 70g/L of iron shreds, (ZVI) showed that (COD) the content was reduced by 33% and color by 45% had been eliminated.  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , and PAC were used in the coagulation process to determine which was best compatible with the ZVI process. The results showed that (PAC) tests with optimal dosages of 1200 mg/L had peak (COD) and color removal efficiency. (PAC) provided a 97% reduction in (COD) and a 98% elimination of color. The ideal mixing speed was 200 rpm of quick mixing followed by 40 rpm of gentle mixing. The findings showed that a 90-minute settling time produced the smallest volume of settled sludge.

## 1. INTRODUCTION

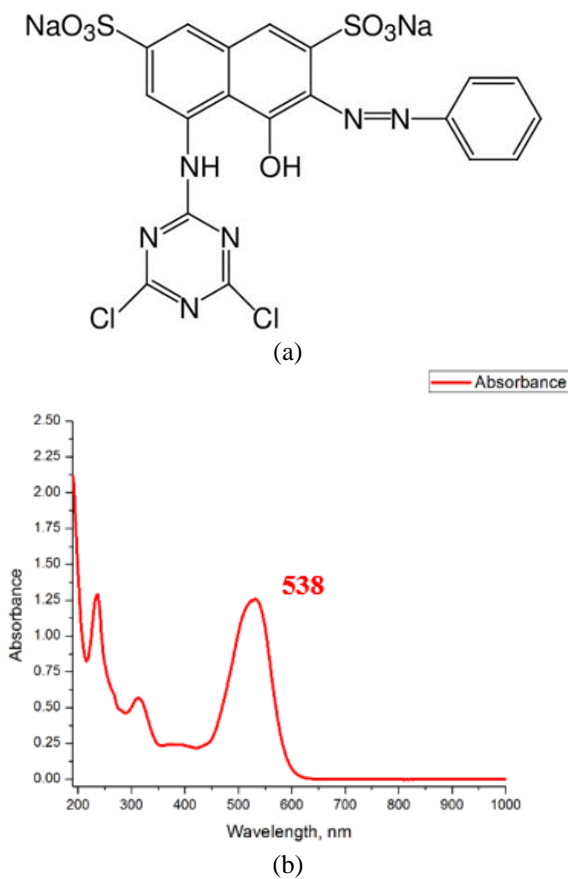
With large percentages of non - fixed (approximately 20%) colors, textile effluent serves as one of the main manufacturing water contamination sources in industrialized regions. Due to its widespread use, bio-resistance to traditional aerobic treating wastewater, and hazardous aromatic compounds, dye is a serious problem [1]. Decolorization is one of the most difficult processes in treating wastewater [2]. The adsorption process and biological treatment are currently the most extensively utilized methods for treating dyeing wastewater, [3-5]. Activated carbon has a large specific surface area and is porous, making it a typical dye adsorbent. Microbial activity-based biological therapy has the potential to be inexpensive and energy-efficient. Despite this, there are drawbacks to each of these methods. Activated carbon adsorption, for instance, only converts colors from their liquid form to their solid state. The biological process is challenging to activate and manage [6]. Sahinkaya et al. [7] discussed the biological treatment that coupled with nanofiltration to improve the wastewater disposed from of denim textile industry for reuse. The results obtained indicate that (COD) and color removals were about 84% and 75% respectively. Although the treatment is quite efficient, the treated wastewater doesn't meet the reuse indication. In recent years, zero-valent iron has been shown to be useful in reducing hazardous compounds in water [8, 9]. A good degrading agent, zero-valent iron is affordable, environmentally safe, and simple to make. Also, industrial filing iron waste can be used to add two electrons to a variety of environmental pollutants by converting it into zero-valent iron [10]. It has been demonstrated to be effective in reducing

nitro-aromatic chemicals, insecticides, nitrate, and ions of metal as Cr (VI) [11-14], among other chlorinated solvents [15] studied the capability of zero-valent iron to remove arsenic compounds for the groundwater. Their results showed that more than 98% of arsenate could be removed steadily with a hydraulic resident time of two hours at last, and the effluent meets the drinking water standard. When  $\text{Fe}^0$  reacts with water or  $\text{H}^+$ , hydrogen atoms can be produced, resulting in Azo bond splitting ( $-\text{N}=\text{N}$ ), which is detrimental to the chromosphere Azo dye group and conjugated system [16]. The use of particles of iron results in sludge with a low iron concentration and has the added benefit of making it simple to recycle used iron powder using magnetism [17]. When treating wastewater with coagulation and the zero-valent-iron (ZVI) technique [18] investigated the effects of dose and pH on COD elimination. Their findings demonstrated that for the advanced treatment of coking wastewater, (ZVI) was superior to coagulation. Azo dyes are complex compounds that could be susceptible to breakdown by (ZVI) [19-21]. Using iron scrap particle electrolysis as the main target parameter in this study, we intend to determine whether electrolysis can be applied to colored solutions for COD and color as the primary parameters. In order to determine the effect of airflow and pH on the removal efficiency, pertinent factors need to be understood as they relate to the discoloration mechanism of the dye. It queries to find the adequate coagulant for the coagulation process that can familiarize with iron scrap particles electrolysis process to give good results with low cost. The novelty in this research is the use of a disposed solid waste materials in wastewater treatment field as a well-known zero valent iron process.

## 2. METHODOLOGY

### 2.1 Colored aqueous solution

X-3B dye was studied in this experimental work. Figures 1-a and 1-b depict the chemical composition and maximum wavelength curve of the dye. The chemical composition was presented to reveal the chemical structure of the dye, furthermore, it will enable the researcher to understand the mechanism of removal and how iron scraps particle could break the bonds of chemicals that consist the dye composition. The maximum wavelength is important to analyze the dye concentration before and after the removal. The dye was dissolved in distilled water at a concentration of 1000 mg/L for each dye in order to create a synthetic solution of the dye effluent.



**Figure 1.** (a) The chemical structure of X-3B (b) The wavelength curve of X-3B

### 2.2 Preparation of iron scraps particles

Iron scraps particles were collected from industrial sectors in Ramadi. After steeping and washing with sodium hydroxide solution NaOH (1%) for 10 minutes, distilled water was used to rinse the iron scrap particles, subsequently was dried in oven at 105°C. HCl (5%) hydrochloric acid solution was then added, steeped and washed for another 20 minutes, and then rinsed with distilled water once more and dried in oven at 105°C.

#### Coagulants

Three different coagulants were used in this study namely; Ferric Chloride  $FeCl_3 \cdot 6H_2O$ , Ferrous Sulfate  $FeSO_4 \cdot 7H_2O$ , and Aluminum poly chloride (PAC), to select the suitable one

with optimum removal efficiency after treatment with iron scrap particles. (COD) removal and color removal efficacy of wastewater was compared in order to choose the typical coagulant with iron scraps electrolysis in this paper.

#### Jar experiment tests

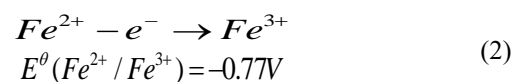
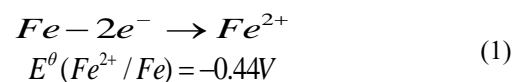
Jar-test equipment was used for coagulation experiments. In this experiment, a dyed solution containing One liter of a 1000 mg/L concentration was used. After adding 70g of prepared iron scrap particles and 200 rpm for 10 minutes, each beaker was continuously aerated for 20 minutes. After one minute of rapid mixing at 500 rpm, different doses of coagulants were added to the jars, followed by 10 minutes of slow mixing at 200 rpm. During sedimentation, time was recorded for 0, 10, 30, and 60 minutes, and COD and color were measured in the supernatant. These experiments included three groups of experiments in different varied parameters; first finding the optimum coagulant that can be most familiar to the iron scraps particles in coagulation process and also investigating the optimum dose of each coagulant used and recording the optimal settling time. Following the results of the first group of jar tests, the second group investigated the pH impact on (COD) and color removal using the optimum dose of coagulant and settling time. The last group was represented by examining the effect of the mixing velocity and mixing time on coagulation process and also increasing the settling time to 90 minutes to study its effect. It was implemented for two coagulants that have been tested in the first step. The rapid mixing in coagulation process was changed in different mixing velocities at (200, 220, 240, 260, 280, and 300) rpm for two minutes of mixing time, and the slow mixing was carried out at (40 rpm) for 20 minutes.

## 3. RESULTS AND DISCUSSION

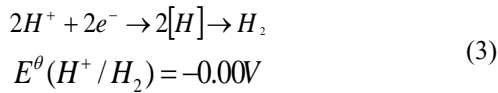
### 3.1 Removal by ZVI

The findings indicated that roughly 40% of the content of COD and 28% of the color can be removed by iron particles. The investigations demonstrated that iron hydroxides produced during the hydrolysis process by ZVI particles have an impact on the pH level of the dye. After the iron scraps were hydrolyzed, the pH of the dye was raised from 7.9 to 9.2. Iron scrap particles cause the wastewater to become alkaline after mixing and aeration because ferrous ions that dissolve from their surface collide with hydroxyl ions in the solution, precipitating ferrous hydroxide on the surface of the iron scrap particles to obstruct the process [22]. The dye molecule is decreased when the iron particle comes into touch with the electrolyte solution used for dyeing. The transitional product is created when the dye molecule reacts with acidic  $H^+$  after receiving electrons from the iron. The terminal products are created when this product obtains electrons and reacts with  $H^+$  once more [23]. The following are examples of the reactions:

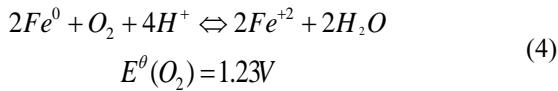
#### Anode Reaction:



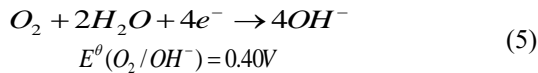
**Cathode Reaction:**



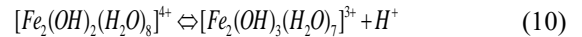
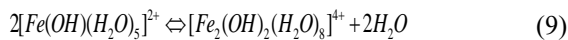
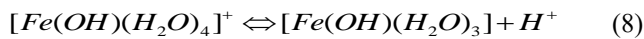
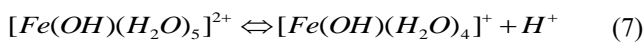
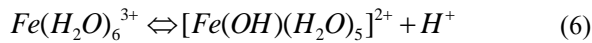
**Oxidation:**



**Neutral to alkaline:**



The oxidation and precipitation of the Fe<sup>2+</sup> released from the anodes results in the production of ferrous and ferric hydroxides, which lead to coagulation in the treatment system. Radicals and other oxidants created during electrolysis have the ability to oxidize organic pollutants. Other removal techniques include precipitation and tangling in the ferrous and ferric hydroxide floc. As a gelatinous suspension, the produced ferrous and ferric hydroxides remain in the aqueous stream and can remove contaminants from wastewater by coagulation or electrostatic attraction [24]. As a result, iron electrolysis can result in the production of nascent Fe<sup>2+</sup>. With the help of the oxygen in the solution, it can be transformed into Fe<sup>3+</sup>, yielding Fe(H<sub>2</sub>O)<sub>6</sub><sup>3+</sup>, which can subsequently be transformed into additional hydroxyl irons like Fe(OH)<sub>2</sub><sup>2+</sup> or Fe(OH)<sub>3</sub><sup>+</sup>. They can mix various organic chemicals to create floccules, which causes these organic chemicals to precipitate from wastewater [16]. The following is the primary response:



The efficacy of the dye degradation may be impacted by the initial pH values since the reduction potential for Fe<sup>+2</sup>/Fe increases as the H<sup>+</sup> concentration rises.

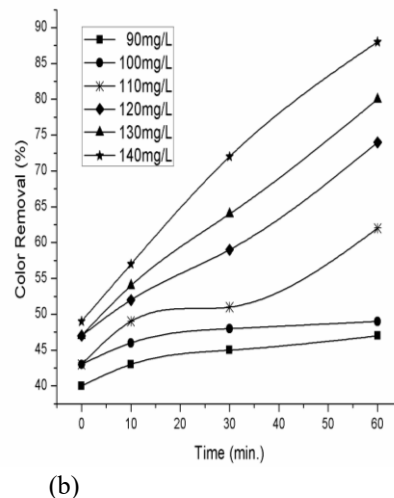
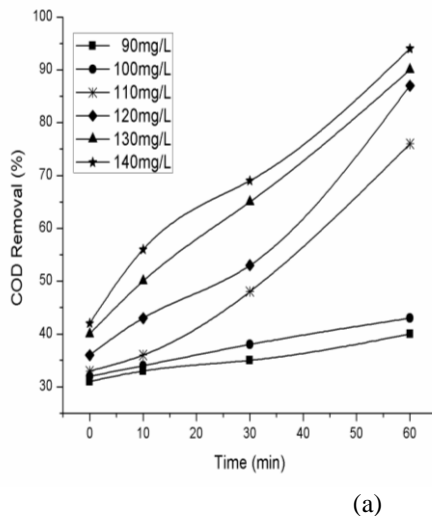
**3.2 Coagulation process**

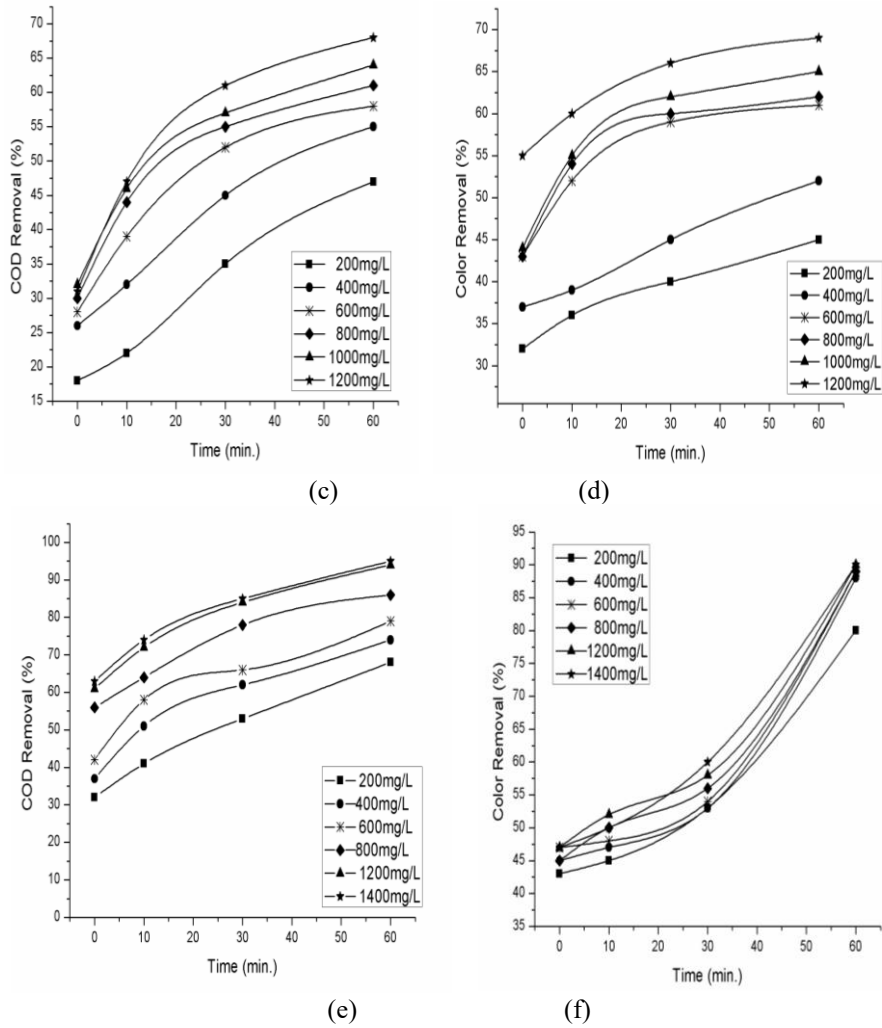
**Coagulants dosage**

Figure 2 shows that optimum dose of FeCl<sub>3</sub>.6H<sub>2</sub>O for (COD) and color removal is 140mg/L at settling time of 60 minutes. (COD) percent reduction was 94%, while color percent removal was 88%. Flocs were slightly recognized and pH of the supernatant was 3.5, and volume of settled sludge was higher than that in the other FeSO<sub>4</sub>.7H<sub>2</sub>O. About 68% and 69% of (COD) content and color were removed by ferrous sulfate with 1200mg/L dosage at settling time of 60 minutes and pH after treatment was 5.3. After settling, the flocs were not visible and the settled sludge was not clear in the experiments of the studied dye solution. The figure elucidates that about 94% of (COD) content and 90% of color were removed with an optimum dose of 1200 mg/L of PAC at settling time of 60 minutes. Settling sludge was higher than that in other coagulants and flocs formation was visible in this experiment while pH here was 5.2.

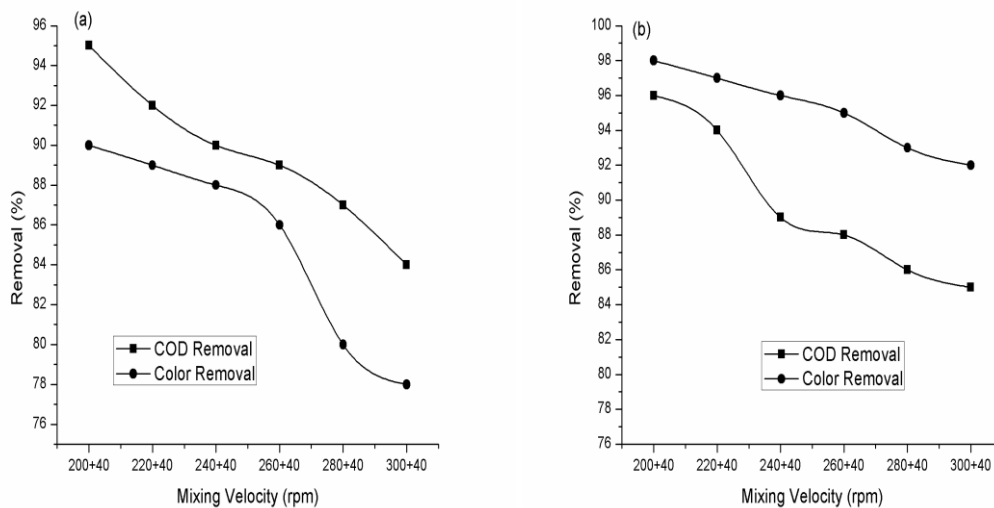
**Studying the effect of PH**

The pH effect studying is an important factor in the dye removal to know its effect on the reduction efficiency of (COD) content and color degradation or removal. These experiments were applied for the artificial dyeing wastewater after treating by the iron particulates. The optimum doses and settling time concluded in coagulants studying of this research were used for pH adjustment. X-3B's pH was changed after the iron process from 7.9 to 9.2. [23] discovered that pH had a significant impact on how quickly iron degraded colored effluent. They discovered that as the acidity rose, the rate of deterioration rose quickly. FeCl<sub>3</sub>.6H<sub>2</sub>O tests using X-3B found that 4 was the ideal pH; the findings reveal that 78% of the (COD) content was removed, and color removal efficiency was 58%. pH effect by FeSO<sub>4</sub>.7H<sub>2</sub>O experiments elucidated that 80% of (COD) value and 71% of pigment was eliminated in pH 8. PAC experiments of pH adjustment show that pH of 9 represent the optimum, giving a removal efficiency of 97% for (COD) and 87% for color and these results represented in pH 6.





**Figure 2.** COD and Color removal versus settling time at different doses of the coagulant (a, b)  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (c, d)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (e, f) PAC



**Figure 3.** COD and color removal efficiency by at different mixing velocities (a)  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (b) PAC

**Effect of the mixing velocity**

The coagulants tested in these experiments were Ferric Chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) with its optimum dose of (140 mg/L) and Ploy Ferric Chloride (PAC) with its optimum dose of (1200 mg/L), while the settling time was 60 minutes. Selection of these two coagulants was based on their effectiveness and

workability with iron hydrolysis to give a good reduction efficiency of (COD) and color removal, and because they were less sensitive to pH and can be used over a pH range from 4 to 9, besides, both of these coagulants leave a low settling sludge after a specified settling time that can be recognized as compared as to  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ . Mixing time and rapid mixing

velocity conditions in chemical coagulation process was changed to find the most effective and efficient velocity. For treatment with iron wastes, no parameters were changed and its effect was studied in the bench scale experiments. Figure 3 shows that increasing the rapid mixing velocity in FeCl<sub>3</sub>.6H<sub>2</sub>O experiments conduce to decrease the removal efficiency in (COD) and color. The optimum reduction in (COD) was 95% and color removal was 90% achieved in 200 rpm of rapid mixing with 40 rpm of slow mix. The experiments of PAC show that (COD) reduction efficiency and color removal was 98% and 96% respectively, under the same mixing conditions used in FeCl<sub>3</sub>.6H<sub>2</sub>O.

#### 4. CONCLUSIONS

Iron waste particles hydrolysis was investigated with coagulation process for treatment of a synthetic dyeing wastewater for X-3B. With 1000 mg/L as an initial aqueous solution, it is possible to eliminate 45% of the color and 33% of the (COD) value using 70g/L of iron scraps particles in a hydrolysis process with thorough blending velocity of about 500rpm continued for twenty minutes put up with 200rpm until 10 minutes under a 440 L/hr airflow. Iron increases pH of the synthetic wastewater after treatment from 7.9 to 9.2. In coagulation process, PAC represented the most familiar in its results after iron scraps electrolysis giving high removal efficiency in (COD) and color, and less effect to pH and can be used over a pH range from acidic to basic conditions. The ideal PAC concentration was 1200 mg/L, while the ideal mixing speed was 200 rpm of quick mixing and 40 rpm of gradual mixing. When the least amount of settling sludge had appeared after 90 minutes of settling, the best removal efficiencies were achieved. From the results obtained in this study it can say that iron wastes particles are efficient to reduce (COD) content and eliminate the color in dye wastewaters with a low cost and easy processes and also it can reduce the dosages of the chemical coagulants when these chemicals are used without iron scrap to get, somewhat, the same removals.

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