Removal of COD and Color Concentration in Batik Wastewater Using Electrofenton

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Abstract

The effluent discharged from the batik textile industry often contains azo compounds and organic contaminants. One method employed to address these pollutants is the Electrofenton (EF) technique. This study aims to assess the effectiveness of electrofenton in reducing both color content and Chemical Oxygen Demand (COD) in batik dyeing wastewater. The Electrofenton process was conducted in batch mode, varying electrode distances at 4 cm and 6 cm, H_2O_2 : FeSO₄ molarity ratios of (10:0.05) and (10:0.1), and electric voltage strengths at 6 volts and 8 volts. The research revealed that employing an electric voltage of 8 volts, an electrode distance of 4 cm, a contact time of 120 minutes, and a Fenton dosage molarity of 10:0.05 M resulted in a remarkable removal efficiency of 97.8% with a final concentration of 103 mg/L for color content. Additionally, for COD, employing the same parameters led to removal effectiveness of 90.1%, with a final concentration of 730 PtCo. The impact of Fenton dosage on COD removal was statistically analyzed using the t-test. The computed t-value of 3.444 surpassed the tabulated t-value of 2.776, indicating a significant influence of Fenton dosage on COD removal.

Keywords: Batik, Electrofenton, Dye, Fenton.

Abstrak

Limbah cair industri tekstil batik seringkali mengandung senyawa azo dan kontaminan organik. Salah satu metode yang digunakan untuk mengatasi polutan ini adalah teknik Electrofenton (EF). Penelitian ini bertujuan untuk mengkaji efektivitas elektrofenton dalam menurunkan kadar warna dan Chemical Oxygen Demand (COD) pada limbah pencelupan batik. Proses Electrofenton dilakukan secara batch, variasi jarak elektroda 4 cm dan 6 cm, rasio molaritas H2O2:FeSO4 (10:0.05) dan (10:0.1), serta kuat tegangan listrik 6 volt dan 8 volt. Penelitian mengungkapkan bahwa penggunaan tegangan listrik 8 volt, jarak elektroda 4 cm, waktu kontak 120 menit, dan molaritas dosis Fenton 10:0.05M menghasilkan efisiensi

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penghilangan yang luar biasa sebesar 97,8% dengan konsentrasi akhir sebesar 103 mg/L untuk kandungan warna. Selain itu, untuk COD, penggunaan parameter yang sama menghasilkan efektivitas penyisihan sebesar 90.1%, dengan konsentrasi akhir 730 PtCo. Dampak dosis Fenton terhadap penurunan COD dianalisis secara statistik menggunakan ujit. Nilai t yang dihitung sebesar 3,444 melampaui nilai t yang ditabulasi sebesar 2,776, menunjukkan pengaruh yang signifikan dari dosis Fenton terhadap penyisihan COD.

Kata Kunci: Batik, Elektrofenton, Dye, Fenton.

1. Introduction

In Indonesia, the batik textile sector has experienced consistent expansion owing to the rising popularity of its distinctive fashion trend The involvement of numerous micro, small, and medium enterprises (MSMEs) in the production of the Batik industry contributes to the expansion of economic growth (Utami et al., 2023). According to current study, we have witnessed a substantial increase in the number of small and medium-sized batik enterprises (SMEs), with a recorded count of approximately 6,120 units with a workforce of 37,093 people and capable of achieving a production value of around 407.5 billion rupiahs per month or the equivalent of 4.89 trillion rupiahs per year(Subekti, Hafiar, & Komariah, 2020).

Wastewater in the batik industry is generated through various processes such as coloring, washing, and rinsing batik fabric. The utilization of synthetic dyes results in the production of wastewater that contains high concentrations of pollutant substances (Erlinda Ningsih, Yustia Wulandari Mirzayanti, Achmad Chusnun Ni'am, Dita Aulia Fajrin & Imami, 2022), (Kusworo et al., 2022). Due to the dye-based nature of wastewater, any potential treatment method should be capable of effectively removing color, Chemical Oxygen Demand (COD), and Total Suspended Solids (TSS) (Can-Güven, 2021). Numerous physicochemical methods, such as chemical coagulation-precipitation, adsorption, electrocoagulation, flocculation, and advanced oxidation, have been developed for Batik wastewater treatments.

Electrochemical oxidation is widely recognized as an effective and practical technology due to its environmental compatibility, versatility, energy efficiency, and safety(Can-Güven, 2021). Electrocoagulation (EC) involves the use of electrical current to dissolve sacrificial metal electrodes like iron (Fe) or aluminium (Al), resulting in the production of coagulants or destabilization agents (Erlinda Ningsih, Yustia Wulandari Mirzayanti, Achmad Chusnun Ni'am, Dita Aulia Fajrin & Imami, 2022)

The Electrofenton (EF) process employs various electrolytic reactors, including the bubble reactor, filter press reactor, double-electrode electrochemical cell, three-electrode electrochemical cell, and double compartment cell. In this process, different cathodes are examined and used as the working electrode, while various anodes are utilized as the counter electrode (Nidheesh & Gandhimathi, 2012). Correspondingly, the present study aimed to investigate the COD and color removal from batik medium enterprises wastewater by electro-Fenton. The optimum values of variations in plate spacing, voltage magnitude, and molarity of

 $\mathrm{H}_{2}\mathrm{O}_{2}\mathrm{:}FeSO_{4}$ were determined. EF processes were evaluated in terms of pollutant removal efficiency.

2. Method

2.1 Location and Time

Batik industry wastewater used in this study was obtained from batik industries located in Kampung Batik Jetis, Sidoarjo Regency, East Java Province. Preparation of wastewater sample, tools, materials, and turbidity (SNI 19-7117.6-2005) analysis was conducted at the Quality and Engineering Laboratory of the Environmental Engineering Department, Institut Teknologi Adhi Tama Surabaya. Analysis of COD (SNI 6989.2.2019), color (SNI 6989.80.2011), and ammonia (SNI 19-7117.6-2005) in wastewater was carried out at the Surya Sembada Water Supply Company (PDAM) Surabaya. This research was carried out from August 2022 to January 2023.

2.2 Tools and Materials

The following chemical reagents were used: ferrous-sulfate heptahydrate (analytical grade, 99%), hydrogen peroxide (analytical grade, 30%), sodium hydroxide (chemical purity grade, 95%), sulfuric acid (analytical grade, 95–98%), and potassium dichromate (analytical grade, 99.8%). Distilled water was used in cleaning and preparing the reagents during the experiments. Iron and aluminium electrodes with lengths and widths of 7 cm and 5 cm were bought, and a direct current (DC) power supply 3005DK was used.

2.3 Preparation of Batik Industry Wastewater

Batik industry at Kampung Batik Jetis, Sidoarjo doesn't have process yet their wastewater. One hundred liters of batik wastewater samples were directly taken from batik industry at Kampung Batik Jetis, Sidoarjo. Initial characterization tests were performed on wastewater samples obtained which included pH (Indonesia National Standard) (SNI) 06-6989.11-2004), temperature (SNI 06-6989.23-2005), turbidity (SNI 06-6989.25-2005), COD (SNI 6989.2.2019), color (SNI 6989.80.2011), and ammonia (SNI 19-7117.6-2005).

2.4 Implementation of EF Process

The experiments were conducted in a batch reactor containing a 3000 ml sample of the batik wastewater, as illustrated in Fig. 1. The set-up consisted of an electrochemical cell, 2-iron and 2 aluminium electrode rods, electrode connectors, a magnetic stirrer, and a DC power source. The electrodes were cleaned using distilled water to remove impurities and dried overnight at 50°C the oven. The experiment was conducted in an open reactor at room temperature (24 ± 2 °C). The iron electrodes were spaced 60 mm and 40 mm apart to allow effective oxidation and arranged in parallel connection to the DC power source at 6 V and 8 V. 30% analytical grade hydrogen peroxide was added to the cell containing a prepared sample of the collected batik wastewater. Before the EF process, the pH of the cell was adjusted to 3.5 ± 0.5 using 1M H₂SO₄ or 1M NaOH. To improve the generation of hydroxyl radicals throughout

the electrochemical cell. The solution was stirred continuously at 300 rpm using a magnetic stirrer. 0.1M and 0.05M of ferrous-sulfate heptahydrate catalyst were added to the cell to initiate the EF reaction.



Figure 1. Electrofenton Reactor Illustration

2.5 Analytical Tests

In this study, hydrogen-peroxide concentration was operated at reaction times of 30, 60, 90, and 120 min to investigate optimal removal of COD and color demand using the EF process (all experiments were carried out in duplicates to avoid error). At the end of each EF treatment, COD and color tests were carried out using Indonesian standards. The data collected from the COD and color tests were then statistically analyzed using SPSS. Regression linear analysis was used to compare the removal efficiency of the EF process in terms of COD and color (at a 95% confidence interval). The removal efficiency of the EF process was evaluated using the removal efficiency equation

3. Result and Discussion

3.1 Characteristics of Batik Wastewater

The characteristics of batik wastewater were initially examined through testing, and the obtained results are listed in Table 1.

No.	Parameter	Result	Indonesia Standard
1	COD	7411 mg/L	150 mg/L
2	Color	4474 PtCo	-

Table 1. Initial characteristics of batik wastewater

3.2 Effect of Plate Spacing

3.2.1 Effect of Plate Spacing on COD Removal from Batik Wastewater

Treatment based on the variation of plate spacing was aimed to discover whether there was a significant effect caused by the two plate spacing used, which were 4 cm and 6 cm. This experiment was conducted to know the COD removal of batik wastewater. The result of COD removal is shown in Figure 2.



Figure 2 (a). Plate Spacing Variation with a Voltage of 6 V and Fenton Dose of 10:0.05 (b). Plate Spacing Variation with 6 V Voltage and a 10:0.1 Fenton Dose (c). Plate Spacing Variation with 8 V Voltage and Fenton Dose 10:0.05. (d). Plate Spacing Variation with a Voltage of 8 V and Fenton Dose 10:0.1.

As shown in Figure 2, the highest COD decrease is indicated by a molarity ratio of 10:0.05 M with a contact time of 120 minutes, which is 90.1% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm. The lowest COD decrease was shown at the H_2O_2 :FeSO₄ molarity ratio of 10:0.1 M over a 30 minutes, which was 77.6% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm. In this study, the efficiency of COD concentration separation decreased as the electrode distance was reduced. The reduced COD efficiency increases from a contact time of 30 minutes to 120 minutes. This is because the longer the contact time on this electro-Fenton process will cause the Fe²⁺ ion to rise; the more significant the concentration of Fe²⁺, the more increased the oxidation against pollutants shown with the more substantial reduction of COD in waste (Sholeh et al., 2015). This is due to the increasing number of hydroxyl radicals. (Tamas, 2017). The decrease in COD can be seen as the closer electrode distance, the more powerful current, and the more Fe²⁺ production needed in the Fenton reaction (Ratni dan Priyadi, 2019). The distance between electrodes affects the electron transfer speed between anodes that receive electrons, with the cathode as the place where the reduction process occurs (Ratni dan priyadi, 2019). The decrease in

processing efficiency occurs when the electrode gets farther or increases the value of the electrical barrier is also getting more prominent so that the electric current that flows gets smaller and causes the formation of coagulants is also decreased (Mustikaayu and Noor. 2022); but if the distance between the electrodes is too close will cause the amount of the coagulation to increase. Still, the system will be disturbed due to the short connection between the electrodes (Saputra and Hanum, 2017).

3.2.2 Effect of Plate Spacing on Color Removal from Batik Wastewater

Treatment based on the variation of plate spacing was aimed to discover whether there was a significant effect caused by the two plate spacing used, which were 4 cm and 6 cm. This experiment was conducted to know the color removal of batik wastewater. The result of color removal is shown in Figure 3.



Figure 3 (a). Plate Spacing Variation with a Voltage of 6 V and a Fenton Dose of 10:0.05(b). Plate Spacing Variation with a 6 V Voltage and a 10:0.1 Fenton Dose (c). Plate Spacing Variation with an 8 V Voltage and a Fenton Dose 10:0.05. (d). Plate Spacing Variation with a Voltage of 8 V and a Fenton Dose 10:0.1.

As depicted in Figure 3, the most significant reduction in color efficiency, at 97.8%, was observed when the electrical voltage was set at 8 volts, with an electrode spacing of 6 cm, a duration of 120 minutes, and a Fenton dose of 10:0.1 M. This decrease in color can be attributed to adsorption processes, where molecular substances exit the waste solution and adhere to the surface of solid matter (coagulants) during the electrocoagulation process (Setianingrum, et al, 2016). The function of adsorption is to separate the aromatic and organic compounds of the ocean. (Setianingrum, et al, 2016). The smaller the electrode's distance, the higher the color dispersion because the ionisation reaction between the electrodes will be faster, so the decrease of color occurs faster. The electrode works well in forming the coagulation in the early minutes of observation of the electrocoagulation process. In the following minutes, no significant increase is achieved because the dispersion of color substance has reached a figure > 90%.

3.3 Effect of Voltage Magnitude

3.3.1 Effect of Voltage Magnitude on COD Removal from Batik Wastewater

Treatment based on the variation of voltage magnitude was aimed to discover whether there was a significant effect caused by the two voltage magnitudes used, which were 6 and 8 volts. This experiment was conducted to know the COD removal of batik wastewater. The result of COD removal is shown in Figure 4.



Figure 4 (a). Voltage Magnitude Variation with a Plate Spacing of 4 cm and a Fenton Dose of 10:0.05(b).
Voltage Magnitude Variation with a Plate Spacing of 4 cm and a 10:0.1 Fenton Dose (c).
Voltage Magnitude Variation with a Plate Spacing 6 cm Voltage and a Fenton Dose 10:0.05.
(d). Voltage Magnitude Variation with a Plate Spacing of 6 cm and a Fenton Dose of 10:0.1.

The highest COD decrease is shown by a molarity ratio of 10:0.05M with a contact time of 120 minutes, which is 90.1% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm as depicted in Figure 4. The lowest COD decrease was shown at the H₂O₂:FeSO₄ molarity ratio of 10:0.1 M over a 30 minutes, which was 77.6% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm. The higher the voltage, the more Fe²⁺ is produced in the Fenton reaction. According to the law of ohm, the greater the voltage, the stronger the current generated. (Rahmawati, et al, 2020). On the electrodes form hydrogen gas and hydroxide ions that can affect the reduction of COD levels in wastewater. The appearance of gas bubbles during the process occurs so that foam appears on the wastewater surface, and after the process on the cathode plate, there are white spots as a sign of the resulting hydrogen gas on the cathode plate (Ni'am, Caroline, & Afandi, 2018). In addition, on the used cathode plates will appear a brown-colored stain appears that indicates the presence of metal ions from the wastewater that reduced its metal and attached to the cathode plate. The decrease in COD occurs due to the formation of flocks around the electrode plate formed by a positive coagulant ion compound. Molecules in the dye waste form flocks and colloidal particles in the waste bind other particles on the waste, such as colloid Fe(OH)₂, which are charged positively because they bind H⁺ ions. This electrofenton process with combined Fe⁺ ions can increase fenton reagents' performance in lowering COD.

3.3.2 Effect of Voltage Magnitude on Color Removal from Batik Wastewater

Treatment based on the variation of voltage magnitude was aimed to discover whether there was a significant effect caused by the two voltage magnitudes used, which were 6 volt and 8 volt. This experiment was conducted to know the color removal of batik wastewater. The result of color removal is shown in Figure 5.

The highest color decrease is shown by a molarity ratio of 10:0.1 M with a contact time of 120 minutes, which is 97,8% of the initial color with a voltage magnitude of 8 volts and a plate spacing of 6 cm. The lowest color decrease was shown at the H₂O₂:FeSO₄ molarity ratio of 10:0.1 M over 30 minutes, 93,8% of the initial color with an voltage magnitude of 6 volts and a plate spacing of 4 cm (Figure 5). The adsorption process causes reduced color. The adsorption process serves to dissolve aromatic and soluble compounds. (Setianingrum et al., 2016). The greater electrical voltage causes the solubility of the anode to be higher so that the number of hydroxocation complexes will increase and cause the existing dye material to form a larger cluster. (Setianingrum et al, 2016). With the formation of larger flocks, the amount of mud produced will be significant, so the dye material is removed more and more (Setianingrum et al, 2016).



Figure 5 (a). Voltage Magnitude Variation with a Plate Spacing 4 cm and a Fenton Dose of 10:0.05(b).
Voltage Magnitude Variation with a Plate Spacing 4 cm and a 10:0.1 Fenton Dose (c). Voltage Magnitude Variation with a Plate Spacing 6 cm Voltage and a fenton dose of 10:0.05. (d).
Voltage Magnitude Variation with a Plate Spacing 6 cm and a Fenton Dose of 10:0.1.

3.4 Effect of H2O2:FeSO4 Molarity

3.4.1 Effect of H₂O₂ : FeSO₄ Molarity on COD Removal from Batik Wastewater

Treatment based on the variation of H_2O_2 : FeSO₄ molarity was aimed to discover whether there was a significant effect caused by the two H_2O_2 : FeSO₄ molarity used, which were 10: 0.05M and 10: 0.1 M. This experiment was conducted to know COD removal of batik wastewater. The result of COD removal is shown in Figure 6.

Figure 6 denoted that the highest COD decrease is shown by a molarity ratio of 10:0.05M with a contact time of 120 minutes, which is 90.1% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm. The lowest COD decrease was shown at the H_2O_2 : FeSO₄ molarity ratio of 10:0.1 M over a 30 minutes, which was 77.6% of the initial COD with a voltage magnitude of 8 volts and a plate spacing of 4 cm. The longer the contact time used in this electrofenton process will produce more and more OH radicals, so it can oxidise organic materials into more straightforward elements and improve processing efficiency (Tamas, 2017). There was a more OH-forming reaction on the H+ ion, increasing the electrofentone process's pH so that in the 120 minutes, the pH increased from 4 to an average of 6.9. The oxidation

process of the organic molecules of the Fenton reagent has a process that means that the organic molecule is rapidly decomposed (approximately 30 minutes) due to the formation rate of OH radicals from the H_2O_2 and Fe^{2+} reactions (Babuponnusami and Muthukumar, 2014). As the organic molecule breaks down rapidly, the suspended solid will also be degraded due to the formation of iron ions $Fe(OH)_3$ which will bind the decomposed suspended solids (X) to the waste causing the COD in the waste also decreases (Tamas, 2017).



Figure 6 (a). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing of 4 cm and a Voltage Magnitude 6 volts. (b). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing of 4 cm and a Voltage Magnitude 8 volt. (c). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 6 cm Voltage and a Voltage Magnitude 6 volt. (d). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 6 cm and a Voltage Magnitude 8 volts.

3.4.2 Effect of H₂O₂ : FeSO₄ Molarity on Color Removal from Batik Wastewater

Treatment based on the H_2O_2 :FeSO₄ molarity magnitude was aimed to discover whether there was a significant effect cause by the two H_2O_2 : FeSO₄ molarity used, which were 10 : 0.05M and 10 : 0.1 M. This experiment was conducted to know color removal of batik wastewater. The result color removal is show in Figure 7.



Figure 7 (a). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 4 cm and a Voltage Magnitude 6 volt.
(b). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 4 cm and a Voltage Magnitude 8 volt.
(c). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 6 cm Voltage and a Voltage Magnitude 6 volt.
(d). H₂O₂:FeSO₄ Molarity Variation with a Plate Spacing 6 cm and a Voltage Magnitude 8 volt.

As shown in Figure 7 that the highest color decrease is shown by a molarity ratio of 10:0.1 M with a contact time of 120 minutes, which is 97,8% of the initial color with a voltage magnitude of 8 volts and an plate spacing of 6 cm. The lowest color decrease was shown at the H₂O₂: FeSO₄ molarity ratio of 10:0.1 M over a 30 minutes period, which was 93.8% of the initial color with an voltage magnitude of 6 volts and an plate spacing of 4 cm. There is the formation of OH reactions that are also more than H+ ions making the pH in the fenton process increased, so that in the 120 minutes the pH is obtained from 4 to 6.9. The coloring substance used in this waste is azo coloring. The substance azo has a special bond, the bond of nitrogen (-N=N-). This decrease in color absorption values indicates that the hydroxyl radicals formed from the fenton reagent will attack the azo group bond on the coloring molecule (Tunç, et al, 2012). Color degradation is more efficient in acidic media due to the better sensitivity of the punctified form to the oxidation process. Color loss is visually significant for all organic compounds according to the load obtained from electrolysis. The addition of the OH-electrolyte

to the azo bond leads to rapid breakdown. The rapid clearance of the solution has to do with the bond (-N=N-) reinforcing this suggestion. The results of a decrease in azo color absorption indicate that hydroxyl radicals formed from fenton reagents will attack the azo group bond on color molecules.

3.5 Statistical Analysis

The results in the study analyzed the main parameters of COD and color will be tested for significance in the statistical test. The statistical test aims to determine the influence between each variable in the research. Significance test in study using Dual Linear Regression Analysis with SPSS software. The Dual Linear Regression Test aims to determine the influence of electrical voltage variations, electrode distances, and fenton doses in reducing COD and color levels after electrofenton treatment. The statistical test uses a double linear regression test with a 95% confidence rate. Significant influence in statistical tests is shown by t count tests larger than t tables. The following will describe the statistical test results on each variable:

Parameter	R Value	R square	T table	T counts	Sig.				
COD									
Fenton dose molarity (X1)	0.893	0.798	2.776	3.444	0.026				
Plate spacing (X2)	0.893	0.798	2.776	1.852	0.138				
Voltage magnitude (X3)	0.893	0.798	2.776	0.697	0.524				
Color									
Fenton dose molarity (X1)	0.544	0.296	2.776	0.065	0.951				
Plate spacing (X2)	0.544	0.296	2.776	0.848	0.444				
Voltage magnitude (X3)	0.544	0.296	2.776	0,978	0,383				

Table 2. Stastitical Results

The result hypothesis COD shows that the sig value of 0.026 > 0.05 indicates an influence of fenton doses molarity on the decrease in COD.

4. Conclusion

The study shows that variation plate spacing, voltage magnitude and H_2O_2 : FeSO₄ molarity affects COD, color and turbidity removal from batik wastewater. While maximum removal COD is 90.1% with variation molarity ratio of 10:0.05, contact time of 120 minutes, voltage magnitude of 8 volts and a plate spacing of 4 cm, while maximum removal color is 97.8% with a variation molarity ratio of 10:0.05, contact time of 120 minutes, voltage magnitude of 6 volts and an plate spacing of 4 cm, while maximum removal turbidity is 99.7% with variation molarity ratio of 10:0.1, contact time of 120 minutes, voltage magnitude of 8 volts and an plate spacing of 4 cm, while maximum removal turbidity is 99.7% with variation molarity ratio of 10:0.1, contact time of 120 minutes, voltage magnitude of 8 volts and an plate spacing of 6 cm.

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