



Enhanced Electro-Degradation of Direct Black 38 Dye Using Copper Oxide-Modified Graphite Electrode and CaCl_2 as Supporting Electrolyte

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Abstract

The dyes used in the textile industry generate substantial waste, posing significant environmental hazards. Because textile industries discharge large amounts of effluents, it is essential to develop effective methods to reduce the presence of reactive textile dyes. This work evaluated the removal of Direct Black 38 (DB-38) using the bare graphite electrode (GE) modified by copper oxide to form a graphite/Cu/Cu-oxide (GE/Cu/Cu-oxide). The electrode was prepared by electrodeposition. Electro-degradation of 50 ppm DB-38 dye was conducted at different applied potentials, reaction times, and CaCl_2 concentrations as supporting electrolytes. The X-ray diffraction patterns showed the highest peak at $2\theta = 36.55$, corresponding to graphite/ Cu_2O , and at $2\theta = 38.73$, corresponding to graphite/CuO. The GE/Cu/Cu-oxide electrode is eleven times more reactive than bare graphite. The GE/Cu/Cu-oxide electrode achieved a 99.08% electro-degradation efficiency of DB-38, with a reduction of 97.50% in COD, and 40.60% in TDS after 60 minutes, when 0.03 M CaCl_2 was used as the supporting electrolyte at a potential 12 V. Thus, the proposed process demonstrated promising results for dye treatment in various aqueous matrices, utilizing low-cost, readily available electrodes that have not been previously applied for this purpose.

1. Introduction

The textile industry generates large amounts, approximately 1000 m³ of wastewater per day, and if not properly managed, this waste can cause serious environmental harm [1]. Textile dyes can be classified into azo dyes, nitroso, triphenylmethane, acridine, quinoline, and indophenol [2]. Textile dye waste is difficult to degrade because it contains complex compounds such as heavy metals, synthetic dyes, and insoluble solid particles. One example is Direct Black 38 (DB-38), an azo-based dye that is both environmentally hazardous and carcinogenic [3].

Several methods are available for managing textile dye waste, including adsorption [4], photo-degradation [5], and electrochemistry [6, 7, 8]. The adsorption method is less effective for textile waste treatment

because the adsorbent still contains accumulated textile dyes [9]. The main drawback of the photo-degradation method is its slow reaction rate and high operational cost [5]. One approach to treating residual textile dyes is through electrochemical methods, particularly electrocoagulation. This method is relatively fast; however, it generates large amounts of surface flocs, leading to additional waste. This drawback can be reduced by using the electro-degradation method, which minimizes floc formation while achieving high color removal efficiency [8].

The electro-degradation process typically employs inert electrodes, such as graphite, due to their high electrical conductivity. However, graphite has relatively low electrical conductivity. The unmodified graphite electrodes exhibit lower electrochemical performance due to their inherently low specific surface area and

limited electroactive sites, which results in slower electron transfer kinetics and lower sensitivity [10]. This limitation can be overcome by modifying graphite electrodes with metals through electrodeposition. Incorporating copper oxide enhances the electrode's conductivity and accelerates electrode degradation [11]. Moreover, metals such as copper are inexpensive and promising anode materials, offering good stability and excellent electrochemical performance [12].

During the electro-degradation process, factors such as the type of electrolyte, the electrode material, and the duration of treatment significantly influence the efficiency of color removal [13]. Not all electrolytes are suitable for electro-degradation. One effective strong electrolyte is CaCl_2 , which has a higher degree of dissociation than many other electrolytes, thereby enhancing electrical conductivity during the process. Additionally, the presence of chloride ions helps accelerate color degradation [14].

Based on this background, the present study aims to degrade the textile dye DB-38 through electro-degradation. The method employs graphite electrodes modified with copper oxide and utilizes CaCl_2 as a supporting electrolyte. This approach is expected to provide a more efficient and environmentally friendly solution for managing residual textile dyes.

2. Experimental

2.1. Materials

Graphite electrode (15 cm, TRR-HP), DB-38 (Sigma-Aldrich, 45%), $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Merck, 99%), HCl (Merck, 37%), acetone (Merck, 99.5%), KCl (Merck, 99.5%), $\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$ (Merck, 99%), and CaCl_2 (Merck, 97%).

The instruments used in this study included standard laboratory glassware, a Bolin platinum electrode, a BK-1501T DC power supply, a Shimadzu 2450 double-beam UV-Vis spectrophotometer, an X'Pert Pro PANalytical X-ray diffractometer (XRD), a Nabertherm furnace, an eDAQ EChem ED410 voltammeter, and a PL-700PDS bench-top pH meter.

2.2. Preparation of Modified Electrode with Copper Oxide

The GE/Cu/Cu-oxide was prepared by the electrodeposition method. The GE was placed at the cathode, while the platinum electrode was at the anode. The GE was immersed in a 0.2 M $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ solution, followed by electrodeposition at 2.5 V for 1 hour (Figure 1 shows the schematic of the process) to form copper-coated graphite (GE/Cu). After deposition, the electrode was rinsed with distilled water and soaked in an HCl:acetone solution (1:1) for 15 minutes. The GE/Cu/Cu-oxide electrode was then formed by heating in a furnace at 400°C for 2 hours. Characterization was performed using XRD. Electrochemical characterization by cyclic voltammetry was conducted at a scan rate of 50 mV/s in 0.1 M $\text{K}_4[\text{Fe}(\text{CN})_6]$ containing 0.1 M KCl, using Ag/AgCl as the reference electrode, Pt as the counter electrode, and GE/Cu/Cu-oxide as the working electrode.

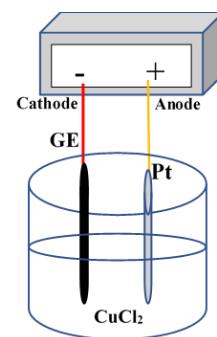


Figure 1. The schematic of the electrodeposition process

2.3. Electro-degradation of DB-38

The electro-degradation of 175 mL of 50 ppm DB-38 dye was performed using 0.025 M CaCl_2 , with variations in treatment time and applied potential (6, 9, and 12 V). The initial pH of the solution was measured as 5.6 to maintain a uniform starting point for all degradation tests. The distance between the anode and cathode was maintained at 2 cm. A schematic of the process using GE/Cu/Cu-oxide electrodes is shown in Figure 2. Samples were collected every 15 minutes, stored in vial bottles, and analyzed with a UV-Vis spectrophotometer. The best potential was identified based on the lowest absorbance value at the end of the process. Time variations were examined to monitor the electro-degradation reaction under different potentials and electrolyte concentrations.

The concentration of the supporting electrolyte, CaCl_2 , was evaluated through a parametric study at the selected potential conditions to identify the concentration that yielded the highest degradation efficiency. Electrolyte concentrations of 0.010, 0.015, 0.020, 0.025, and 0.030 M were tested. The resulting solutions were analyzed using a UV-Vis spectrophotometer at 579.5 nm, and the optimal concentration was determined as the lowest absorbance value at the end of the process. The electro-degradation efficiency of DB-38 dye during electro-oxidation was calculated using Equation (1).

$$\% \text{Efficiency} = \frac{A_{in} - A_{fn}}{A_{in}} \times 100\% \quad (1)$$

Where, A_{in} and A_{fn} represent the absorbance of the solution of DB-38 before and after treatment, respectively. These values were measured at the characteristic maximum absorption wavelength of 597.5 nm using a UV-Vis spectrophotometer.

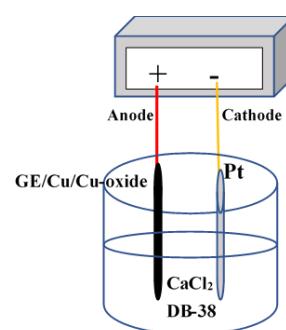


Figure 2. The schematic of the electro-degradation process

3. Results and Discussion

3.1. Modification of Graphite Electrode by Copper Oxide (GE/Cu/Cu-oxide)

Graphite was modified by electrodeposition to precipitate copper metal using direct current. Following electrodeposition, the presence of Cu on the graphite surface was indicated by the formation of a reddish copper layer at the electrode tip. The result of copper deposition on the graphite electrode is called the GE/Cu, presented in Figure 3(B). This electrodeposition treatment enhances the current strength used in the electro-degradation process by increasing the system's current density. Electrodeposition was carried out at an applied potential of 2.5 V, providing a stable current, and resulting in significant copper deposition. During the process, Cu^{2+} ions were reduced to Cu at the cathode, as evidenced by the formation of a copper precipitate layer.

After electrodeposition, the GE/Cu/Cu-oxide electrode was obtained through high-temperature heating. The physical appearance of the electrode is presented in Figure 3. The graphite electrode coated with copper exhibited a high-quality deposit, characterized by uniform surface morphology and strong adhesion to the substrate, in accordance with the criteria reported in the literature [11].

XRD was used to characterize the composition of the solid material. Measurements employed Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$) with 2θ scanning from 5° to 90° . Figure 4 presents the XRD patterns of the graphite electrode composition. The diffraction peak intensities vary among the graphite samples. In the XRD pattern, the GE sample shows a dominant peak at $2\theta = 26.42$, with an additional peak at $2\theta = 77.39$, which are characteristic of graphite according to the Crystallography Open Database (COD ID: 1200018). The main peak exhibits a high intensity of 17,334.57 and a d-spacing of 3.37352 \AA .

For the GE/Cu/Cu-oxide sample, additional diffraction peaks appear at $2\theta = 44.57, 50.38$, and 74.12 , indicating the presence of Cu species on the graphite surface. Peaks at $2\theta = 36.55, 42.38, 61.42$, and 77.56 correspond to Cu_2O (COD ID: 1000063), while peaks at $2\theta = 38.73, 54.67, 66.45$, and 68.04 are assigned to CuO (COD ID: 1011194). The observed peak shifts may result from variations in lattice parameters or crystal density. Overall, the XRD results confirm successful modification of the GE with copper oxide.

In the voltammetry characterization, data were obtained as voltammogram plots that illustrate the electrochemical reactivity of GE and GE/Cu/Cu-oxide. The applied potential allowed identification of the current response characteristics for each electrode. The voltammogram profiles of GE and GE/Cu/Cu-oxide are shown in Figure 5. Figure 5 shows an oxidation peak current of 7.35 mA in the blue line, whereas the red line shows a significantly higher oxidation peak current of

84.79 mA. This indicates that the reactivity of the GE/Cu/Cu-oxide electrode is greater than that of the GE. Based on the Randles–Sevcik equation, the peak current is directly proportional to the electroactive surface area. The higher the peak current, the larger the electroactive area [15]. The GE/Cu/Cu-oxide electrode provides a larger electroactive surface area, with more active sites available for the redox reaction.

In addition, the high peak current, as in GE/Cu/Cu-oxide, indicates strong electro-catalytic activity, which lowers the charge transfer resistance and accelerates the electron transfer kinetics compared to the GE surface [16]. The enhanced peak current demonstrates that copper oxide modification effectively increases the sensitivity of the graphite electrode during the electro-degradation process. This improvement in electrode reactivity is expected to enhance efficiency and reduce the time required for DB-38 electro-degradation.



Figure 3. Electrode physics (A) bare graphite (GE), (B) graphite-copper coated (GE/Cu), and (c) Graphite modified copper oxide (GE/Cu/Cu-oxide)

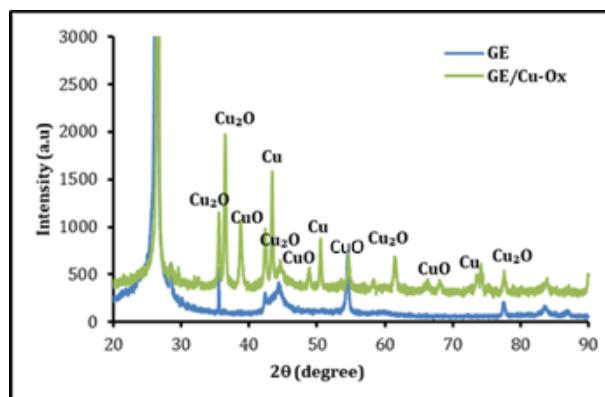


Figure 4. XRD characterization results of graphite composition

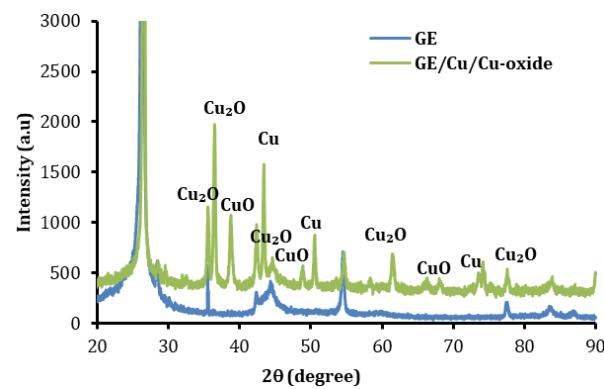


Figure 5. Cyclic voltammogram of $\text{K}_4[\text{Fe}(\text{CN})_6]$ in KCl 0.1 M solution at GE and GE/Cu/Cu-oxide at a scan rate of 50 mV/s

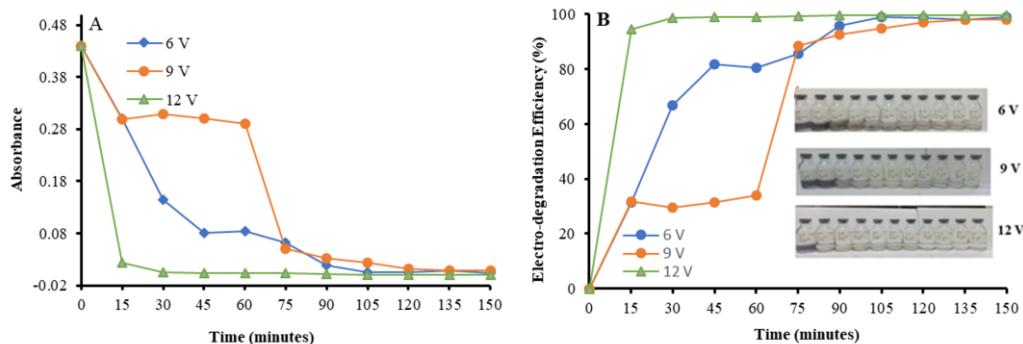


Figure 6. (A) Absorbance and (B) electro-degradation efficiency of DB-38 at the various potentials and times

3.2. Electro-degradation of DB-38 at the Various Potentials, Times, and Concentrations of CaCl_2 as a Supporting Electrolyte

Potential variations of 6, 9, and 12 V were applied to evaluate their effect on electro-degradation efficiency. Each applied potential produced different efficiency values, with higher potentials resulting in greater degradation efficiency. The samples used were simulated waste. The waste preparation was carried out by preparing a DB-38 dye solution at 50 ppm. The solution was prepared by dissolving DB-38 powder in Aquadest. The pH value of the DB-38 solution was measured before and after electro-degradation. The pH before and after electro-degradation were 5.65 and 7.28, respectively. The electro-degradation of 175 mL of 50 ppm DB-38 dye was performed using 0.025 M CaCl_2 .

The initial absorbance of the DB-38 solution, measured prior to electro-degradation, was 0.439. During the process, the GE/Cu/Cu-oxide electrode served as the anode and the platinum electrode as the cathode, both connected to a Direct Current (DC) power supply to maintain a stable current. Increasing the applied potential increased the current through the system, thereby accelerating the electro-degradation reaction and producing more pronounced color fading in the dye solution. Since the applied potential represents the energy driving the redox reaction, higher potentials accelerate the reaction, leading to faster degradation of the initially dark-colored DB-38 solution into a clearer state.

The absorbance measurements at different applied potentials over time showed significant variations, as presented in Figure 6. The highest electro-degradation efficiency was achieved with a final absorbance value of 0.001 at an applied potential of 12 V. This result can be attributed to the complex equilibrium established between the DB-38 dye molecules and the copper oxide on the electrode surface [11].

At an applied potential of 9 V, the treatment efficiency shows little change during the initial period (≤ 60 s). This behaviour is primarily attributed to mass transport limitations and the induction time required for the radical steady-state concentration. Specifically, at the onset of 9 V, the rapid initiation of the oxygen evolution reaction competes for active sites, and a stable diffusion layer must be established before the flux of pollutants to the anode matches the high kinetic capacity of the

electrode. On the other hand, during the first 60 seconds, the concentration of the target pollutant at the electrode interface may be high, but the overall bulk treatment efficiency remains low until a steady-state flux is established [17].

Time variations were also investigated in relation to the electro-degradation process. At different applied potentials, the highest electro-degradation efficiency was found to be 120 minutes, as this duration produced the lowest and relatively stable absorbance value. Beyond 120 minutes, no significant decrease in absorbance was observed. This indicates that most of the DB-38 dye had already degraded, and the reduced concentration of dye molecules limited further interactions between the solution and the electrode surface. At 135 and 150 minutes, the absorbance values for each potential variation fluctuated, showing slight increases and decreases. This behavior may be attributed to the fact that these durations exceeded the optimal time for DB-38 degradation, leading to instability in the absorbance values after most of the dye had already been degraded. As illustrated in Figure 6, the electrochemical degradation of DB 38 achieved near-total efficiency at 12 V for 60 minutes. This configuration was selected after evaluating its cost-effectiveness and energy requirements.

In varying electrolyte concentration, CaCl_2 acted as a supporting electrolyte, facilitating electron transfer during the electro-degradation process and thereby influencing current flow in the solution. The reaction time was fixed at 120 minutes for all concentration variations to determine the final absorbance values. Different CaCl_2 concentrations yielded different final absorbance values. At concentrations of 0.010, 0.015, 0.020, 0.025, and 0.030 M, the final absorbance values were 0.010, 0.009, 0.005, 0.004, and 0.001, respectively. The electrolyte concentration strongly influenced the electro-degradation efficiency. The comparison of electro-degradation efficiency over time at different supporting electrolyte concentrations is presented in Figure 7.

The concentration of the supporting electrolyte influences absorbance following electro-degradation, as depicted in Figure 7A. To better illustrate this effect, the ordinate scale is expanded to 0.00–0.04. Utilizing a potential of 12 V and a supporting electrolyte concentration of 0.03 M resulted in the minimum absorbance during the electro-degradation of DB-38.

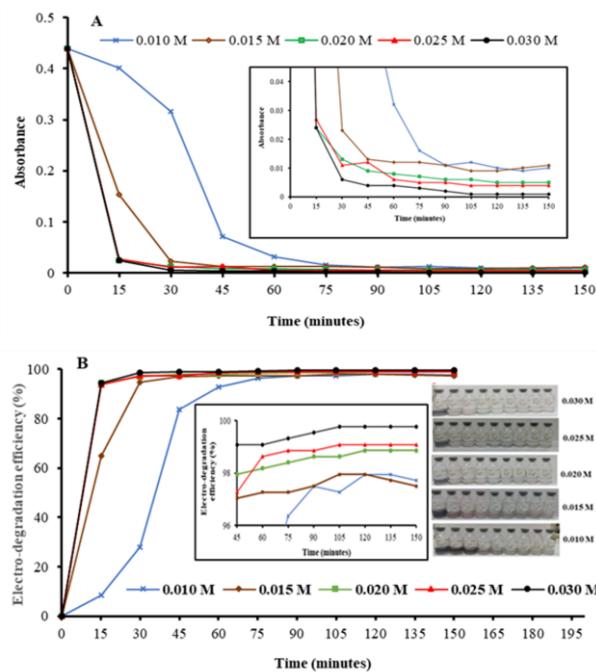


Figure 7. (A) Absorbance and (B) Electro-degradation efficiency of DB-38 at varied supporting electrolyte (CaCl_2) concentrations and times

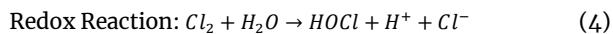
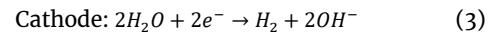
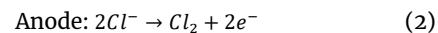
Figure 7(B) shows the electro-degradation efficiency of DB-38 at varied supporting electrolyte (CaCl_2) concentrations and times. Figure 7(B) shows a magnified view of the curve scale, specifically highlighting the range between 96% and 100% on the ordinate and from 45 to 150 minutes on the abscissa. At 0.010 M CaCl_2 , high efficiency was achieved after 45 minutes, whereas at 0.015 M, similar efficiency was reached in just 30 minutes. The 0.020 M and 0.025 M concentrations showed no significant difference, with both achieving high efficiency within 15 minutes.

Based on the trend observed between 30 and 60 minutes, the best concentration is 0.030 M, as the removal efficiency does not fully stabilize between 15 and 30 minutes. The best performance was observed at 0.030 M, yielding a degradation efficiency of 99.08% after 45 minutes. These results indicate that 0.030 M CaCl_2 is the best concentration, enhancing electrical conductivity and reducing system resistance to maximize electro-degradation efficiency [18]. The type and concentration of ions present in the solution significantly influence its electrical conductivity. An increase in electrolyte concentration enhances electro-degradation efficiency and accelerates the degradation of DB-38 dye.

The use of CaCl_2 as a supporting electrolyte is based on the ionic strength and oxidation of Cl^- ions. The divalent calcium cation (Ca^{2+}) provides a higher ionic strength at lower molar concentrations compared to monovalent like as NaCl or KCl [14, 19]. The effect of Ca^{2+} , a bivalent cation, is more marked than that of Na^+ in the elimination of organic matter [18].

The CaCl_2 is a strong electrolyte that dissociates completely into calcium ions (Ca^{2+}) and chloride ions (Cl^-). The chloride ions play a crucial role in the electro-degradation process, as they can be oxidized at the anode

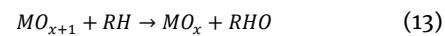
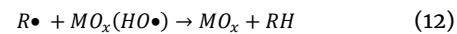
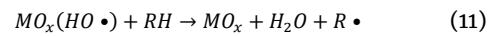
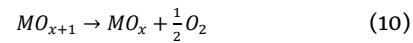
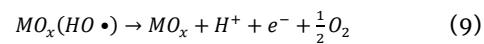
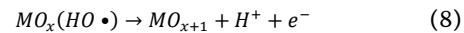
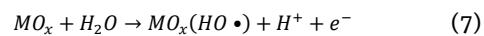
to produce chlorine gas (Cl_2). The generated chlorine then reacts with hydroxyl ions (OH^-) produced from water hydrolysis at the cathode, forming hypochlorite ions (OCl^-). These active chlorine species act as strong oxidizing agents, thereby accelerating the degradation of dye molecules and enhancing the overall color removal efficiency [20]. Hypochlorite (OCl^-) is the key oxidizing species that contributes to the degradation of DB-38 textile dye. The electrochemical reactions occurring in the presence of active chlorine from the CaCl_2 supporting electrolyte are described by Equations 2-5.



Hypochlorite ions (OCl^-) promote pollutant degradation by acting as strong oxidizing agents [20]. The reaction between hypochlorite ions and dye molecules in wastewater is described in Equation (6).



The electrodegradation reactions of pollutants are summarized in Equations (7-13). In this study, copper oxide (denoted as MO_x) acts as the active metal oxide species. When in contact with water, MO_x forms hydroxyl radicals bound to the metal oxide surface, $\text{MO}_x(\text{HO}\cdot)$ (Equation 7). This radical species can then be converted to a higher oxidation state, MO_{x+1} (Equation 8), with simultaneous oxygen evolution (Equation 9). Two MO_{x+1} species may also combine to release oxygen (Equation 10). Both $\text{MO}_x(\text{HO}\cdot)$ and MO_{x+1} are highly reactive oxidizing agents that play a key role in the oxidation of organic pollutants, as shown in Equations (11-13). Essentially, the $\text{R}\cdot$ in Equation (11) is the starting point for the fragmentation of the organic molecule (RH) into smaller, less toxic intermediates until complete degradation is achieved. The organic radical $\text{R}\cdot$ generated in Equation (11) is an unstable intermediate that typically participates in a series of fast propagation and termination steps. These processes predominantly occur at the anode surface and involve the generation of active metal oxide species [13].



The electro-degradation reaction effectively degrades the DB-38 dye, transforming the solution from an intense black color to a clear state. The degradation products are simpler, less toxic compounds, making the process environmentally safe.

3.3. Electro-degradation DB-38 at Various Electrodes

Overall, the best operational conditions were applied to the electro-degradation of DB-38, with GE serving as the control electrode. The electro-degradation efficiency of DB-38 dye during electro-oxidation was determined using Equation (1). These efficiency values allow for a direct comparison of electro-degradation performance between GE and GE/Cu/Cu-oxide. The comparison of electro-degradation efficiencies is presented in Figure 7.

Based on Figure 8, the electro-degradation of DB-38 using GE/Cu/Cu-oxide achieved a high efficiency of 99.77% within 60 minutes. In contrast, GE required 60 minutes to achieve a lower degradation efficiency of 92.48%. This indicates that GE/Cu/Cu-oxide is significantly more effective and faster at degrading DB-38 than GE. The color degradation profiles using GE are presented in Figure 8(A), while the results obtained with GE/Cu/Cu-oxide under optimum conditions are shown in Figure 8(B).

Based on the efficiency values obtained, the GE/Cu/Cu-oxide electrode demonstrates clear advantages, reducing the electrode degradation time while achieving high degradation efficiency. The presence of copper oxide enhances the electrode's ability to oxidize chloride ions into chlorine gas, as shown in reaction equation (1). This process accelerates subsequent reactions (4) and (5), leading to a rapid increase in the electro-degradation efficiency of the DB-38 dye. The results indicate that the GE/Cu/Cu-oxide electrode is highly effective for the degradation of textile dyes. Based on experimental observation, the durability and reusability of the modified electrode were 5 times higher under the same conditions. Overall, the modified graphite electrode offers the dual benefits of shortened degradation time and high efficiency, with the electro-degradation rate strongly influenced by the catalytic activity of copper metal.

3.4. The Effect of Electro-degradation by GE/Cu/Cu-oxide on pH, COD, and TDS

The samples, consisting of DB-38, were characterized using water quality parameters relevant to textile waste. This aims to compare the initial state with the state after electro-degradation. The characterization results provide information on whether the sample is suitable for disposal into the environment after treatment. The results of the textile wastewater sample characterization before and after electro-degradation are shown in Table 1.

Table 1. Characterization results of wastewater samples before and after electro-degradation

Parameter	Electro-degradation of wastewater samples		Description
	Before	After	
pH	5.65	7.28	Increased
COD	137.6 mg/L	3.4 mg/L	Decreased by 97.5%
TDS	151 mg/L	90 mg/L	Decreased by 40.6%

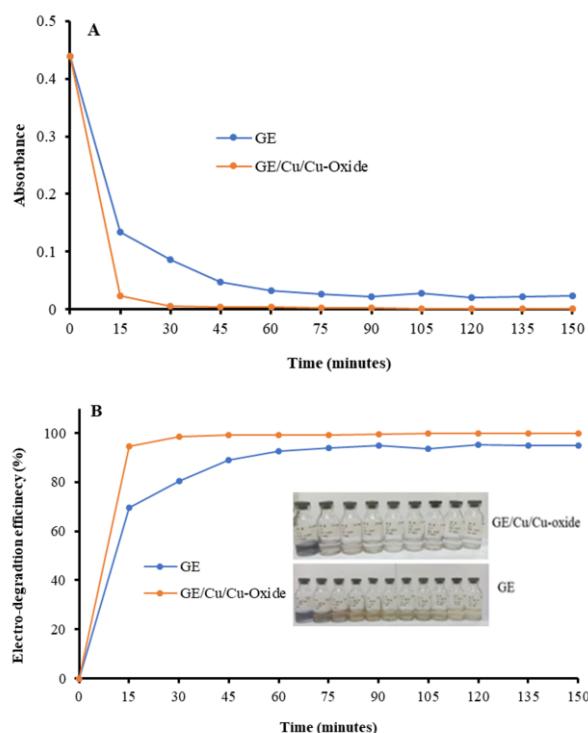


Figure 8. (A) Absorbance and (B) Electro-degradation of DB-38 using GE and GE/Cu/Cu-oxide electrodes

pH is an important parameter for assessing water quality. In the DB-38 textile dye wastewater sample, the initial pH before treatment was 5.65. After the electro-degradation process, the pH increased to 7.28. According to the Regional Regulation of the Special Region of Yogyakarta Number 7 of 2016 (pH 6–9), the treated wastewater meets the required pH standard [21]. The pH value of the sample prior to electro-degradation was below the threshold specified in textile wastewater quality standards. Following electro-degradation, the pH increased to 7.28, which falls within the permissible range. This indicates that the treatment process applied in this study successfully improved the pH from an initially unsafe level to an environmentally safe level.

Chemical Oxygen Demand (COD) is a key parameter for assessing water quality, as it reflects the amount of oxygen required to oxidize organic substances in wastewater. The COD of the untreated DB-38 dye solution was 137.6 mg/L, which, although still within the textile wastewater discharge limit, is close to the maximum allowable value of 150 mg/L. This indicates the need for further treatment to ensure environmental safety. After the electro-degradation process, the COD decreased to 3.4 mg/L, corresponding to a removal efficiency of 97.5%. This reduction demonstrates the effectiveness of electro-degradation in removing organic pollutants and confirms that the treated wastewater meets quality standards. The decrease in COD is attributed to the degradation of dye molecules into simpler compounds such as H_2O , CO_2 , and short-chain organic products that no longer absorb in the visible region. The difference in COD values indicates that the untreated sample contained a higher concentration of organic chromophoric compounds, while the treated sample exhibited improved water quality [22].

Total Dissolved Solids (TDS) is an important parameter for assessing water quality, representing the concentration of dissolved substances in a solution, including organic and inorganic matter such as minerals, salts, metals, and ions. In this study, the TDS value of the sample before electro-degradation was 151 mg/L, which decreased to 90 mg/L after treatment, corresponding to a 40.6% reduction. The higher TDS value observed in the untreated sample is associated with greater turbidity compared to the treated sample. Since TDS is closely related to water clarity, it can be stated that an increase in TDS generally leads to higher turbidity levels, while a decrease in TDS indicates improved water quality [22]. Based on the characterization of the samples before and after electro-degradation, the process effectively reduced both COD and TDS, key parameters that influence overall water quality.

4. Conclusion

Based on this research, copper oxide can be successfully deposited onto the surface of graphite electrodes, enhancing their sensitivity and catalytic activity. The resulting GE/Cu/Cu-oxide demonstrated high performance in the electro-degradation of DB-38, achieving an efficiency of 99.08%. The best operating conditions were identified as a reaction duration of 60 minutes, an applied potential of 12 V, and a CaCl_2 electrolyte concentration of 0.030 M. Furthermore, the electro-degradation process significantly reduces COD and TDS values, thus complying with permitted environmental quality standards.

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