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ORIGINAL RESEARCH ARTICLE

Plasma electrolysis as a sustainable strategy for Remazol Red RB-133 degradation in azo dye wastewater

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ABSTRACT

This study investigates the degradation of Remazol Red RB-133 in batik wastewater using plasma electrolysis, an advanced oxidation process (AOP) that generates highly reactive hydroxyl radicals (●OH). The plasma system, operated at 60 °C with air injection, achieved rapid degradation 86.4% within 5 minutes and up to 99% after 60 minutes exceeding the performance of non-plasma techniques such as electrocoagulation. Degradation kinetics were characterized through UV-Vis spectroscopy and LC-MS/MS, revealing the progressive breakdown of azo chromophores and aromatic rings into low-molecular-weight, less toxic intermediates, which were subsequently mineralized into CO_2 and H_2O, as indicated by significant degradation in Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC). Mass spectral analysis confirmed the formation and subsequent transformation of intermediate compounds, including carboxylic acids and inorganic ions such as SO_4^{2-}, NO_3^-, and NH_4^+. The degradation mechanism followed a radical-based pathway comprising initiation, propagation, and termination stages. These findings demonstrate the high efficiency and environmental sustainability of plasma electrolysis for treating dye-laden wastewater and provide insights into the mechanistic pathway of azo dye mineralization, contributing to the advancement of water treatment technologies aligned with SDG 6.

Keywords: plasma electrolysis; azo dye; Remazol Red RB-133; hydroxyl radical; wastewater treatment; COD; TOC; LC-MS/MS; UV-Vis spectroscopy

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1. Introduction

Remazol Red RB-133 is an anionic azo dye commonly found in textile wastewater. Its complex aromatic structure, high solubility, and resistance to natural degradation render it highly persistent and toxic to the environment[1]. Conventional wastewater treatment plants predominantly rely on biological degradation processes, either aerobic or anaerobic, which have shown limited efficacy in removing such recalcitrant compounds[2,3]. Attempts to improve treatment performance by integrating physical and chemical techniques such as coagulation, filtration, adsorption, or chlorination often lead to increased operational costs without substantially enhancing dye removal efficiency[4]. Plasma electrolysis, classified under Advanced Oxidation Processes (AOPs), has been explored as a promising approach for degrading persistent organic pollutants including azo

dyes[5]. Prior studies indicate that this method can achieve up to 95% decolorization efficiency[6]. The process generates reactive oxygen species, particularly hydroxyl radicals (●OH), which non-selectively oxidize organic molecules, transforming them into intermediate compounds and ultimately mineralizing them into CO_2, H_2O, and inorganic ions[7]. **Figure 1** illustrated the molecular formula of Remazol Red RB-133 is $C_{27}H_{18}ClN_7Na_4O_{15}S_5$ with a molecular weight of 968.18 g/mol, underscoring its structural complexity and resistance to conventional biodegradation[8].

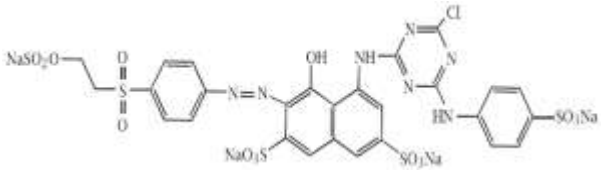

Figure 1. Remazol Red RB-133 molecular structure.

The persistent nature of Remazol Red RB-133 in batik wastewater is a significant environmental, as chemical oxygen demand (COD) values have been reported as high as 13,800 mg/L, far exceeding the regulatory limits in Indonesia (PerMenLH No. 51/1995, which sets maximum COD at 150 mg/L and color concentration at 15 mg/L). Accordingly, plasma electrolysis offers an environmentally sustainable approach that contributes to achieving SDG 6, which emphasizes universal access to safe water and the responsible management of water resources. The efficiency of plasma generation and pollutant degradation is influenced by key process variables such as input power, Na_2CO_3 electrolyte concentration, air injection, and time operation which can enhance the production of ●OH radicals.

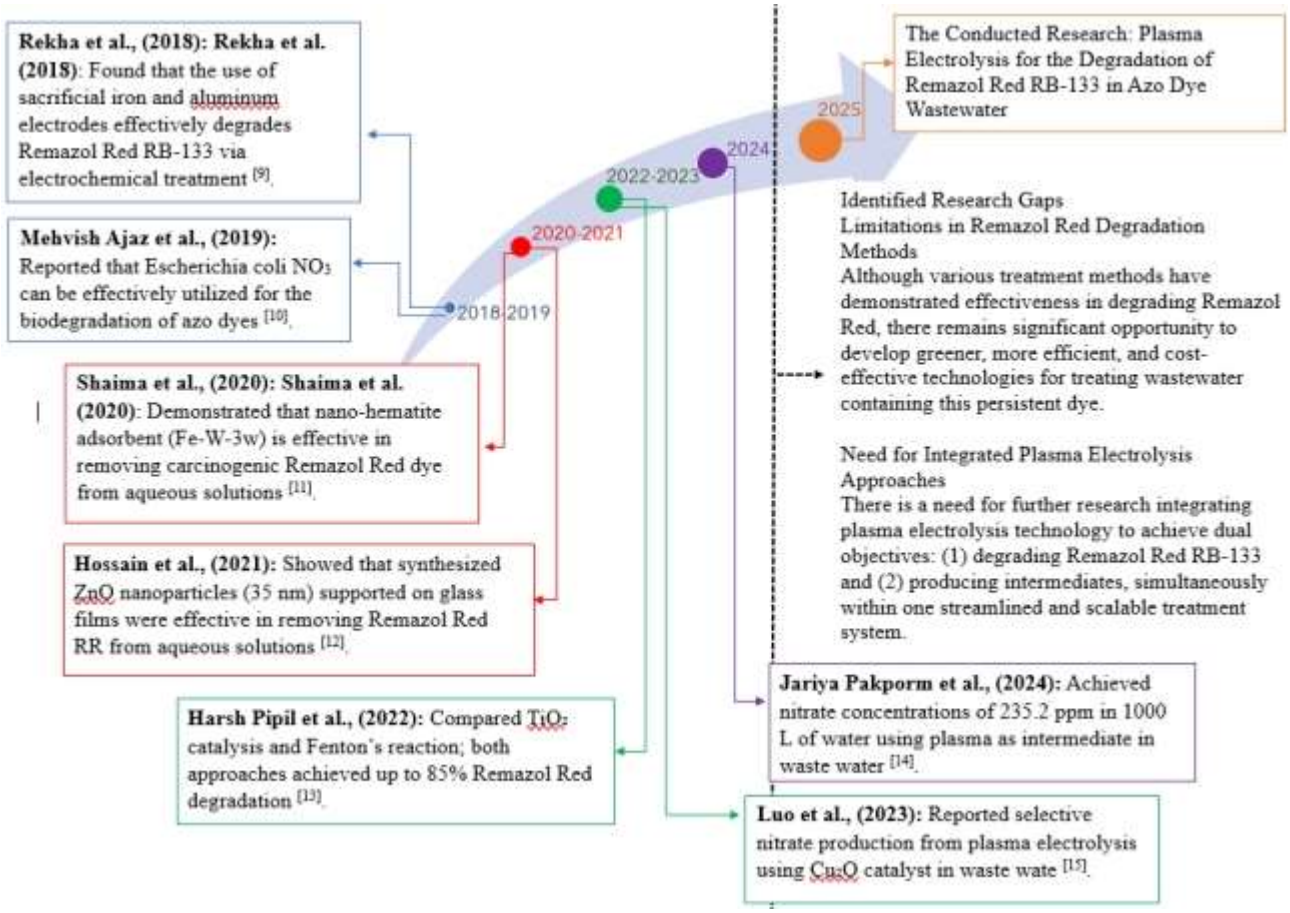

Figure 2. State of the art and research gap of the research.

Shown in **Figure 2**, although plasma electrolysis effectively decolorized Remazol Red RB-133, the formation of aromatic intermediate compounds suggests incomplete mineralization. This highlights the need

for extended treatment or combined methods to achieve full degradation and detoxification of the wastewater. The identification of intermediates also contributes to understanding the degradation pathway of azo dyes under plasma-based AOPs. This study aims to investigate the performance of Remazol Red RB-133 degradation and identify the resulting intermediate compounds formed during the plasma electrolysis process. The novelty of this study lies in addressing the high ecological and health risks posed by persistent pollutants such as Remazol Red RB-133, as well as the limitations of conventional treatment technologies. These challenges underscore the urgent need for innovative, efficient, and environmentally sustainable approaches. This study presents an innovative utilization of plasma electrolysis technology for the degradation of Remazol Red RB-133 in textile wastewater, with the objective of advancing sustainable approaches to industrial effluent treatment and contributing to the development of environmentally responsible wastewater management strategies.

2. Materials and methods

2.1. Material

Sodium Carbonate (Na_2CO_3) with Merck number 1.06392.1000, white crystalline solid with a molecular weight of 142.04 g/mol (anhydrous), used as a salt in the electrolyte solution. Remazol Red RB-133 ($C_{27}H_{18}ClN_7Na_5O_{18}S_6$) with Merck Number 1.12221.0025 is a synthetic textile dye in the form of a red powder with a molecular weight of 968.18 g/mol. It is commonly used as a model pollutant in studies on azo dye degradation. Distilled water (aquadest) was used as the solvent.

2.2. Methods

2.2.1. Plasma electrolysis experimental design

The schematic diagram of the experimental setup is presented in **Figure 3**.

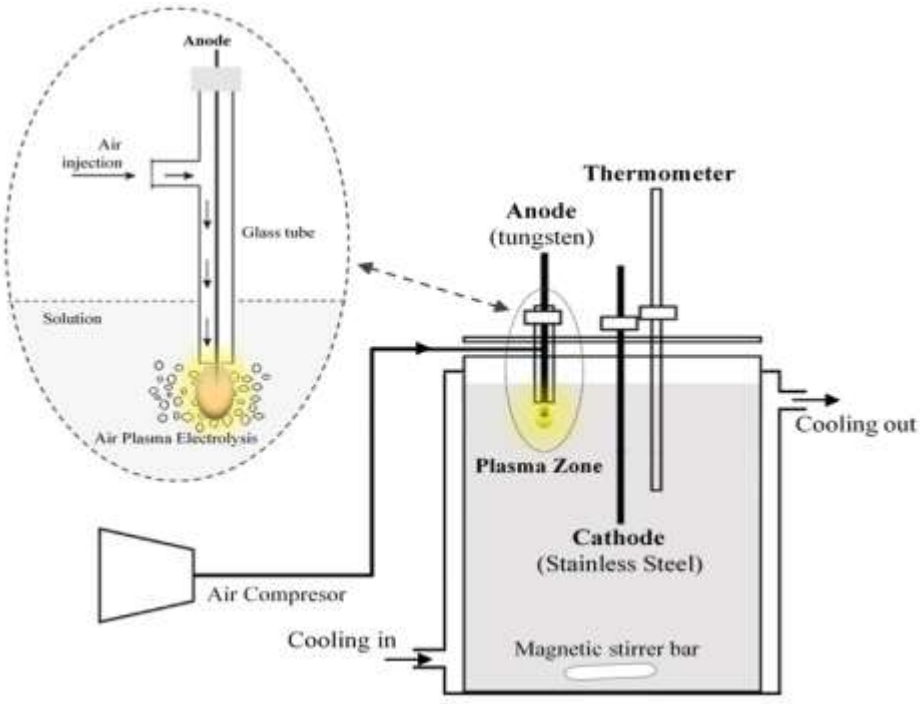

Figure 3. Plasma electrolysis reactor.

Based on **Figure 3**, the system consists of a cylindrical plasma electrolysis reactor constructed from borosilicate glass (Pyrex) with a total volume capacity of 1.2 liters. The reactor is equipped with two electrodes: a tungsten rod (Ø 2 mm, Alfa Aesar) serving as the anode, and a stainlesssteel rod (grade 316, Ø 5 mm, McMaster) as the cathode. The anode was encased in a glass sheath with 5 mm of its length exposed and

immersed in the electrolyte solution. Both electrodes were connected to a programmable DC power supply (GWINSTEK GPR-1000HD), with adjustable output ranging from 0 to 1000 V and a maximum current of 5 A. The electrolyte used in all experimental runs was a 0.02 M solution of Na_2CO_3 with Merck number 1.06392.1000. Temperature monitoring was performed using a digital thermometer (KIRAY 300, France) with ±1°C accuracy. Electrical parameters were measured using a digital multimeter (UNI-T 61E), while voltage readings were cross-validated with a benchtop voltmeter (HIOKI 3239-50) at 700V. A flow meter (OMEGA FL-3400) was used to monitor air injection flow rates at 0.08 L/men during plasma generation. The setup also included a diode bridge rectifier, a ceramic capacitor (100 µF, Vishay), and insulated wiring to ensure electrical safety and signal stability[16].

2.2.2. Procedure

In this investigation, Remazol Red RB-133 ($C_{27}H_{18}ClN_7Na_5O_{18}S_6$), a representative synthetic azo dye with a molecular weight of 968.18 g/mol (Merck catalogue no. 1.12221.0025), was employed as the target contaminant at an initial concentration of 200 ppm to evaluate the effectiveness of the plasma electrolysis treatment process. The degradation process was monitored by measuring changes in dye concentration using a UV-Visible spectrophotometer (Shimadzu UV-1800, Japan). The degradation efficiency was calculated based on the relative concentration decline over time using the equation[17]:

$$Degradation\ Remazol\ Red\ RB-133 = \frac{C_0 - C_t}{C_0}\ x\ 100\% \quad (1)$$

To investigate the transformation products generated during plasma electrolysis, the treated samples were analyzed using Liquid Chromatography–Mass Spectrometry (LC-MS/MS). Chromatographic separation was carried out on an ACQUITY UPLC® H-Class system (Waters, USA), which was fitted with an HSS C18 column (1.8 µm; 2.1 × 100 mm) and maintained at 50 °C. A gradient elution method was applied, in which mobile phase A consisted of water with 5 mM ammonium formate and mobile phase B comprised acetonitrile containing 0.05% formic acid, delivered at a flow rate of 0.2 mL/min. Prior to injection, samples were filtered through a 0.2 µm syringe filter, and an injection volume of 5 µL was used. Mass detection was performed using a Xevo G2-S QTof mass spectrometer (Waters, USA), operated in positive electrospray ionization (ESI) mode, with a scanning range of 50–1300 m/z. Additional instrumental settings included a source temperature of 100 °C, desolvation temperature of 350 °C, gas flow rate of 793 L/h, and a collision energy ramp of 25–50 V[18].

In addition, mineralization efficiency was assessed through Total Organic Carbon (TOC) analysis, which was conducted using a Shimadzu TOC-L CPH Analyzer (Japan). This analysis was carried out via high-temperature catalytic combustion, followed by detection using a non-dispersive infrared (NDIR) sensor. The TOC values were used to quantitatively determine the extent of organic carbon oxidation and served as an indicator of the completeness of organic compound degradation.

3. Result and discussion

The treatment of batik wastewater using plasma electrolysis was performed at a controlled temperature of 60 °C, with an air injection flow rate maintained at 0.8 L/min over a 60-minute operational period. The degradation process occurred most rapidly during the initial phase, with a plateau observed after approximately 15 minutes. Compared to treatments without air injection, the introduction of air significantly enhanced the degradation efficiency of Remazol Red. Within just 5 minutes, the degradation efficiency reached 86.42% at an air flow rate of 0.08 L/min, representing a 55.4% increase relative to the process without air injection. At 30minutes, the degradation reached 97.5% for an initial dye concentration of 200ppm, indicating superior performance compared to several previous studies. During the first 10 minutes, the presence of injected air facilitated the generation of hydroxyl radicals (●OH), which play a critical role in the oxidative breakdown of Remazol Red. Although degradation continued between the 10 and 15minute, the rate of increase diminished

due to the reduced concentration of the dye. After 20minutes, the degradation rate stabilized, likely due to the limited availability of dye molecules in the solution. Excess ●OH radicals, in the absence of sufficient dye molecules, tend to recombine into hydrogen peroxide (H_2O_2), a species with a lower oxidative potential[19]. Moreover, ●OH radicals also react with intermediate degradation products, contributing to their further breakdown and eventual mineralization, as reflected by the decreasing Total Organic Carbon (TOC) values.The plasma electrolysis process generates highly reactive ●OH radicals with a high oxidation potential (2.81V). Additionally, glow-discharge plasma emits ultraviolet (UV) radiation and shock waves, which synergistically enhance the degradation efficiency. The degradation process follows a radical mechanism consisting of three main stages: initiation, propagation, and termination, wherein reactive species such as hydroxyl radicals (●OH) are generated, react with target compounds, and eventually stabilize or recombine[20].

$$\text{Initiation} \quad : H_2O_2 + hv \rightarrow 2^{\bullet}OH \tag{2}$$

$$\text{Propagation} \quad : H_2O_2 + {}^{\bullet}OH \rightarrow H_2O + HO_2{}^{\bullet} \tag{3}$$

$$HO_2{}^{\bullet} + H_2O_2 \rightarrow H_2O + O_2 + {}^{\bullet}OH \tag{4}$$

$$\text{Termination} \quad : {}^{\bullet}OH + {}^{\bullet}OH \rightarrow H_2O_2 \tag{5}$$

$${}^{\bullet}OH + HO_2{}^{\bullet} \rightarrow H_2O + O_2 \tag{6}$$

$$HO_2{}^{\bullet} + HO_2{}^{\bullet} \rightarrow H_2O_2 + O_2 \tag{7}$$

This is in line with previous research Zhang et al. (2024), the pH of the solution significantly influences the degradation pathway by modulating the reactivity and stability of hydroxyl radicals (●OH), which are known to be highly pH-dependent. At acidic to neutral pH (typically pH < 7), ●OH is more stable and remains in its radical form, thereby playing a dominant role in non-selective oxidation of organic compounds through hydrogen abstraction or electron transfer. In contrast, at higher pH (alkaline conditions), the reactivity of ●OH may be suppressed due to its rapid recombination or conversion into less reactive species such as superoxide ($O_2^{-}\bullet$) or hydroperoxide anion (HO_2^{-}), altering the degradation pathway. Moreover, under alkaline conditions, other radicals such as $O_2^{-}\bullet$ may become more prominent, which follow different reaction mechanisms. Consequently, the dominant degradation mechanism may shift from hydroxyl radical-based oxidation at low pH to other radical or non-radical mechanisms at high pH, thus affecting the efficiency, selectivity, and intermediate products of the overall degradation process[21].

In addition to color removal, the effectiveness of this process is also demonstrated by reductions of COD and TOC, as shown in **Figure 4**.

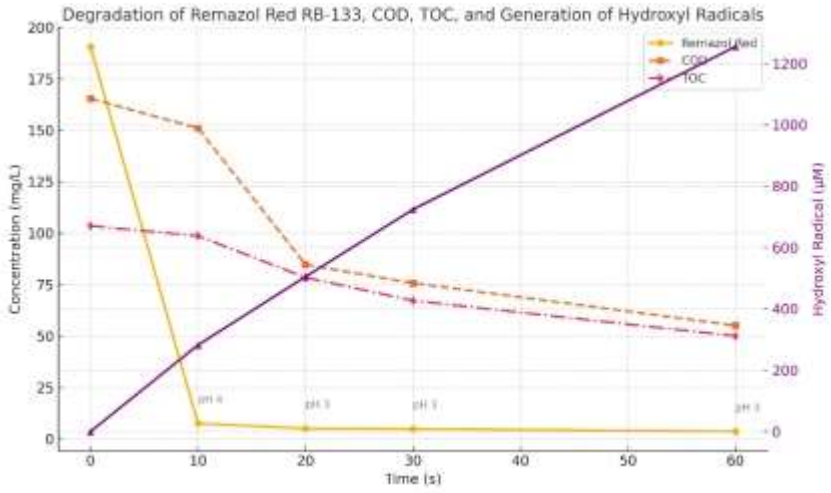

Figure 4. Degradation of Remazol Red RB-133, COD, TOC and Generating of Hydroxyl Radicals.

Figure 4 illustrated that Remazol Red degradation proceeded rapidly during the first 10 minutes, after which the rate slowed due to the significantly reduced residual dye concentration. Nonetheless, the ongoing plasma electrolysis continued to generate ●OH radicals capable of oxidizing soluble intermediate compounds, as evidenced by the continuous decline in COD levels[22]. The decreasing TOC further indicates that mineralization proceeded effectively. COD and TOC serve as indicators of the amount of oxygen required to oxidize organic matter in water samples into CO_2 and H_2O. These values are critical for assessing organic pollution levels in water and must comply with environmental quality standards. The degradation process was highly effective, with Remazol Red concentrations decreasing by 97.5% at 30 minutes and 98.99% at 60 minutes. The findings of this study are consistent with those reported by Sukreni et al. (2019), who achieved approximately 86% degradation of Remazol Red RB-133 within the first 5 minutes of plasma electrolysis[23]. Moreover, the results align with the work of Farawan et al., (2019), which demonstrated up to 99% degradation of the same dye after 30 minutes of treatment. COD decreased by 54.3% and 67.03%, while TOC decreased by 35.2% and 51.7% at 30 and 60minutes, respectively[24]. The concurrent decrease in COD, TOC, and pH suggests complete oxidation of organic compounds into CO_2 and H_2O. Dissolved carbon was likely converted into inorganic carbon species such as carbonate in the aqueous phase or released as gaseous CO_2, depending on the pH. pH is a crucial factor in wastewater treatment. Several studies have shown that degradation rates improve at lower pH values due to the increased of ●OH and H_2O_2[25]. Additionally, ●OH radicals exhibit higher oxidation potentials at acidic pH (2.70 V at pH 3) compared to alkaline conditions (2.34 V at pH 9).

Although significant decolorization of Remazol Red occurred during the degradation process, the extent of color removal was not directly proportional to the reduction in COD and TOC[26]. This indicates that the azo chromophore group (-N=N-) responsible for the red color was successfully degraded, while lower-molecular-weight intermediates were still present in the Na_2CO_3 solution.

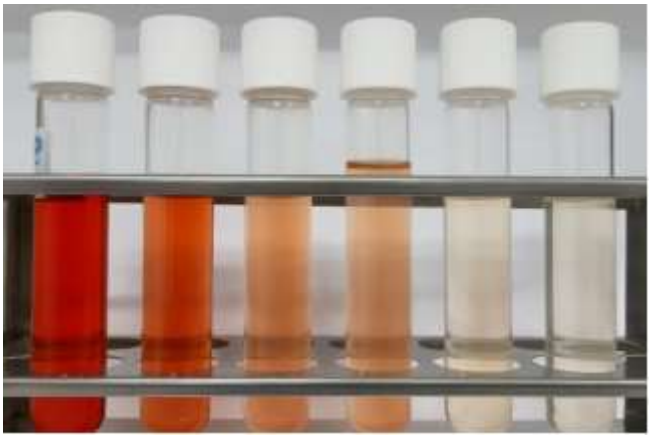

Figure 5. Visual discoloration of the Remazol Red RB-133 solution observed at sequential time intervals: (a) 0 minutes, (b) 5 minutes, (c) 10 minutes, (d) 20 minutes, (e) 30 minutes, and (f) 60 minutes.

The visual transformation of color during the plasma electrolysis process is presented in **Figure 5**, where the initial red hue of the Remazol Red RB-133 solution gradually fades, with the solution appearing nearly transparent by the 30th minute. The observed discoloration is primarily attributed to the cleavage of the azo bond (-N=N-) within the chromophoric group, leading to the formation of colorless intermediate compounds[23]. Subsequent degradation pathways involve the breakdown of aromatic rings and other cyclic and aliphatic structures, producing various intermediate species[27]. These intermediates are further oxidized into low-molecular-weight compounds such as carboxylic acids, which are ultimately mineralized into carbon dioxide (CO_2). The diminishing absorbance in the visible region of the UV-Vis spectrum confirms the continuous decrease in the concentration of Remazol Red RB-133 over time.

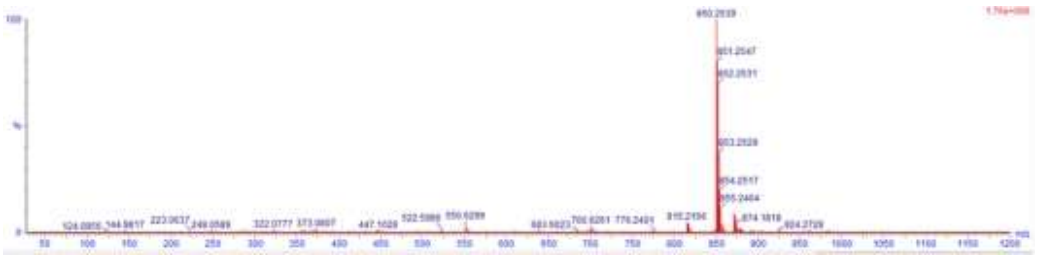

Figure 6. Mass spectral profile of Remazol Red RB-133 at the initial stage (t = 0 minutes).

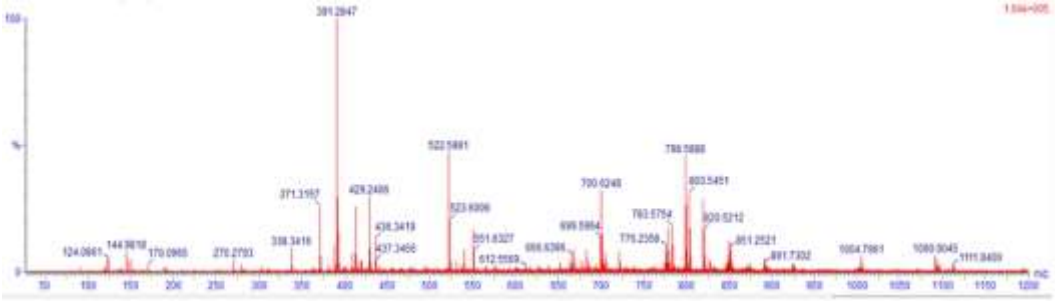

Figure 7. Mass spectrometric analysis of Remazol Red RB-133 at t = 5 minutes.

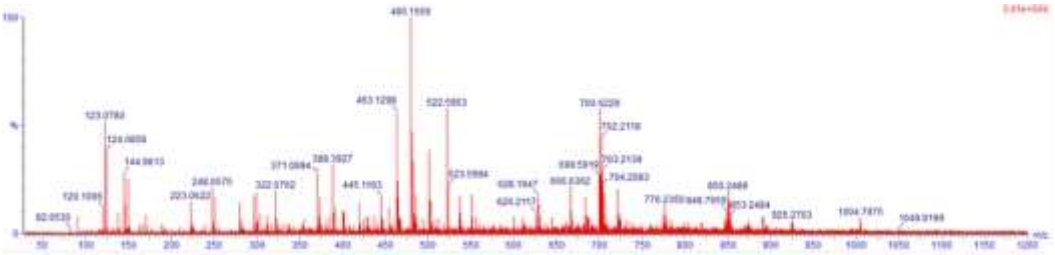

Figure 8. Mass spectrum illustrating degradation products of Remazol Red RB-133 at the 10-minute.

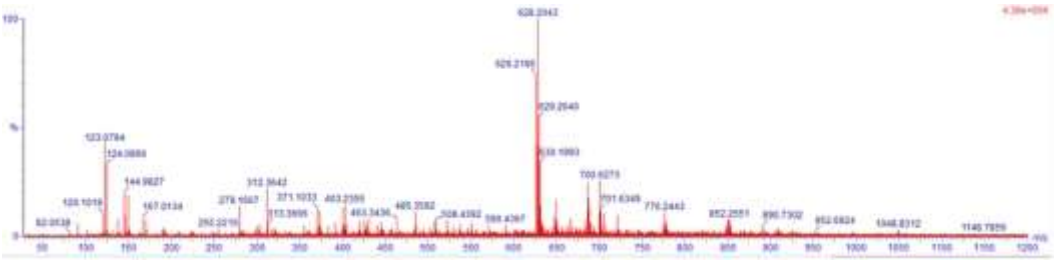

Figure 9. Mass fragmentation pattern of Remazol Red RB-133 observed at 20 minutes.

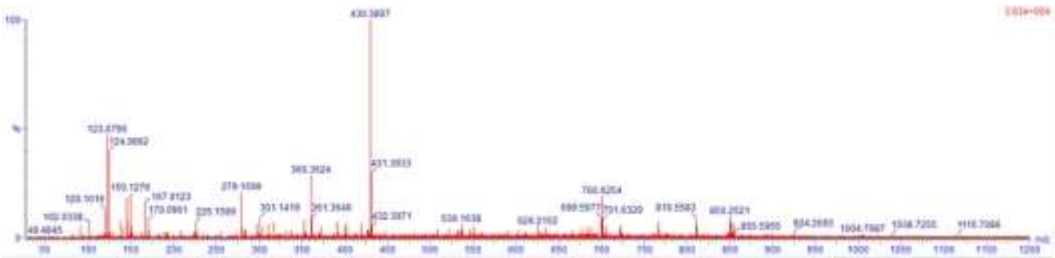

Figure 10. Mass spectral profile of Remazol Red RB-133 following 30 minutes of degradation.

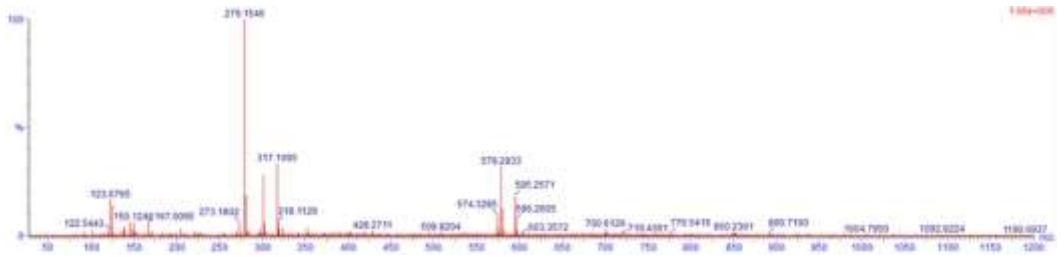

Figure 11. Mass spectrum indicating degradation products of Remazol Red RB-133 at 60-minute.

Figure 6 presents the chromatogram of the Remazol Red solution at the initial condition (t = 0 minutes), exhibiting a dominant peak with the highest intensity at a retention time (Rt) of 17.45 minutes. This peak corresponds to the parent compound, Remazol Red. The degradation of Remazol Red RB-133 was monitored through UV-Visible (UV-Vis) spectroscopy by observing changes in the absorbance spectrum over time. The dye exhibits a characteristic absorbance peak in the visible region, typically centered around λ_max ≈ 530–550 nm, which corresponds to the azo (-N=N-) chromophore group responsible for the compound's intense red coloration. As the plasma electrolysis process progresses, the intensity of this peak gradually diminishes, as illustrated in **Figures 7-11**. Progressive Degradation (t = 5 to 30minutes), as the plasma electrolysis process proceeds: The absorbance intensity at λ_max decreases steadily, suggesting the breakdown of the azo bond and the gradual loss of conjugated π-electron systems. New absorbance peaks emerge in the lower UV region (200–350nm), typically associated with smaller aromatic or aliphatic intermediate compounds. This spectral shift indicates that the parent dye is being converted into intermediate species with lower molecular weights and different electronic structures. By the 60minute, the peak at λ_max is significantly diminished or disappears entirely, indicating near-complete decolorization and degradation of Remazol Red. The remaining spectral features in the UV region suggest the presence of non-colored, low-toxicity by-products such as carboxylic acids and short-chain organic fragments[28].

This decline in intensity signifies the progressive degradation of the parent dye compound. The emergence of new peaks at lower retention times indicates the formation of intermediate degradation products, which are presumed to possess lower molecular weights, higher polarity, and reduced toxicity compared to the original Remazol Red RB-133 molecule. These intermediates are indicative of cleavage reactions occurring during the degradation process. The elemental composition of azo dyes particularly sulfur, chlorine, and nitrogencontributes to the formation of water-soluble ionic species during degradation). Sulfur is oxidized into sulfate ions (SO_4^{2-}), although the rate of sulfate formation is generally slower than the rate of decolorization. Nitrogen-containing moieties, on the other hand, are transformed into ammonium (NH_4^+), nitrate (NO_3^-), and eventually molecular nitrogen (N_2). Ammonium ions undergo further oxidation to nitrate, followed by the release of nitrogen gas, completing the nitrogen mineralization pathway[29].

4. Conclusion

This study demonstrates the effectiveness of plasma electrolysis as a sustainable and high-performance technology for the degradation of Remazol Red RB-133 in textile wastewater. The process exhibited rapid and efficient dye removal, achieving over 97% degradation within 30 minutes and 99% at 60 minutes under optimized conditions with air injection. Spectroscopic and chromatographic analyses confirmed the cleavage of the azo bond and the stepwise formation of low-molecular-weight intermediates, which were ultimately mineralized into inorganic end products such as CO_2, NO_3^-, and SO_4^{2-}. This study acknowledges several limitations that warrant consideration. First, the identification of reactive oxygen species (ROS) such as hydroxyl radicals (●OH), superoxide anions ($O_2^{\bullet-}$), and ozone (O_3) involved in the plasma-assisted oxidation process was not directly confirmed using Electron Spin Resonance (ESR) spectroscopy due to the unavailability of suitable instrumentation. Consequently, the presence and role of specific radical species were

inferred indirectly through theoretical references and degradation behavior. Second, the potential contribution of nanobubbles to reactive species generation was not experimentally evaluated. These limitations suggest that conclusions regarding the underlying degradation mechanisms should be interpreted with caution. The progressive decrease in COD and TOC values supports the occurrence of complete mineralization beyond mere decolorization. Compared to conventional treatment methods, such as electrocoagulation, plasma electrolysis offers superior degradation efficiency, reduced processing time, and minimal secondary pollution. The study not only validates plasma electrolysis as a viable method for industrial wastewater treatment but also provides mechanistic insights into the degradation pathway of azo dyes. These results affirm the potential of plasma-based AOPs to address environmental challenges related to persistent organic pollutants and support their broader implementation in sustainable water management strategies.

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Author contributions

Conceptualization and Supervision: Harianingsih, Nur Qudus; Data Curation: Nabila Khoirunisa, Kristian Saputra; Manuscript Preparation and Writing: Catur Rini Widyastuti; Review and Editing: Nuni Widiarti.

Conflict of interest

The authors declare no conflict of interest.

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