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Research Article

Synthesis and Characterization of Stereoselective Ozonides for Sustainable Textile Wet Processes

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Abstract

This study focuses on the stereoselective synthesis of ozonides for potential applications in different textiles processes. By synthesizing controlled ozonide in a closed-circuit reactor, high stereoselectivity (>90% ozonide formation) was achieved, enabling sustainable denim fading, cotton bleaching and/or different textile washing processes. This method will significantly reduce water usage and chemical discharge compared to traditional processes. In-depth analyses using UV-vis spectroscopy, FTIR, NMR, X-ray diffraction (XRD), cyclic voltammetry (CV), and oxidation-reduction potential (OPR) measurements demonstrate selective chromophore degradation without cellulose degradation, confirming the role of ozonide intermediates in targeted oxidation. This innovation aligns with the EU Green Deal principles, which promote circular *economy applications in textiles. Its scalability and low energy profile highlight its applicability for eco-efficient textile production.*

Keywords: Stereoselective ozonide synthesis, ozonolysis, textile applications, cyclic voltammetry

1. **Introduction**

Ozone gas is an oxidizing agent that can replace hydrogen peroxide and sodium hypochlorite in textiles. However, due to its short half-life in air and water, its rapid exposure to environmental conditions, its production being machine-dependent, its lack of storage, and its difficulty in measuring and controlling, the use of ozone gas has not become widespread. Literature studies reviewed indicate that ozone gas can be used at academic and laboratory levels in processes such as bleaching, denim effecting, dye removal, fading, and wastewater treatment. An examination of industrial applications reveals some unsuccessful experiences. The main reason for this is the instability of ozone gas and the inability to measure its amount at the site of application, especially in gaseous form. The only exception is the use of ozone gas in the denim industry. In the denim industry, ozone gas is used for effecting and preventing back-dyeing (Beşen B., 2016). In textile finishing, hydrogen peroxide has been used for many years in bleaching (Zeronian and Inglesby, 1995), denim effecting (Eroğlu N., 2023), and wastewater decolorization (chemical oxidation) processes (Hassaan, M., 2017). Reductive chemicals are used in dye removal/fading processes (Balcı and Oğulata, 2006), and NaClO in denim effecting processes (Sancar Beşen and Balcı, 2016). Ozone gas is used and/or is being investigated in the bleaching of cellulosic and woolen products (Eren, S., 2018), denim effecting, reactive dye removal/fading, reductive washing (Pusic, T., 2025), and wastewater decolorization (Sancar and Balcı, 2013). The use of hydrogen peroxide in the bleaching process of cotton materials has disadvantages such as the need for a stabilizer to control peroxide decomposition, the risk of catalytic damage to the fabric due to metal ions that may come from water or the fabric, and the necessity of deactivating the peroxide on the fabric with catalase enzyme after the process, which results in additional water and time consumption (Zeronian and Inglesby, 1995). Reductive chemicals and chemicals such as NaClO used in other finishing processes also have a high environmental burden (Uddin, F. 2021). Furthermore, the inability of ozone bleaching to be applied at high pH and high temperatures (due to its destabilization) and its inadequacy in removing trash from fabric limit its application in the textile industry. Similarly, its use in the textile industry has been limited due to stability issues with ozone gas in other finishing processes, the necessity of applications being carried out in completely enclosed environments and adequate ventilation during application, and the need for larger-capacity ozone generators in industrial applications. The new generation stereoselective ozonide synthesized in this study is expected to eliminate these disadvantages due to its high stability and selectivity.

Ozonides (1,2,4-trioxolanes) are cyclic peroxides formed by the reaction of alkenes with ozone gas and serve as important intermediates in organic synthesis. These compounds form via the Criegee mechanism, in which ozone adds to the double bond of an alkene, initially forming molozonite structures that then rearrange to stable ozonides (Bailey, 1978). The traditional synthesis method involves exposing alkenes to an ozone stream at low temperatures, but this approach can lead to yield reductions and safety concerns in solvent-based systems (Sato et al., 2024). In recent years, alternative ozone-free synthesis methods have been developed; for example, bridged ozonides can be synthesized at room temperature using diketones and hydrogen peroxide (H_2O_2) under stereoelectronic control, thereby overcoming the scalability issues of traditional ozonolysis (Dorel and Alabugin, 2017). Additionally, the direct reaction of alkenes with ozone streams under solvent-free conditions via molecular sieves (e.g., MS 4Å) offers an environmentally friendly approach, achieving yields over 70%, especially for highly substituted alkenes (Sato et al., 2024).

Characterization of ozonides requires various spectroscopic and analytical techniques to verify their structural integrity. Mass spectrometry (MS) is effective for direct analysis of ozonides, such as the detection of their formation in glycerophosphocholine lipids (Brown et al., 1996). NMR spectroscopy (^{1}H-NMR and ^{13}C-NMR) determines characteristic signals for peroxide bonds (e.g., δ in the 100-110 ppm range), while thermal behavior is examined by thermogravimetric analysis (TGA) (Thomas et al., 2022). In oil-based ozonides, FTIR spectroscopy detects peroxide bands (800-900 cm^{-1}), and biological activity tests confirm antimicrobial properties (Diaz et al., 2023). Purity analysis of ozonides derived from fatty acids such as oleic acid is performed using HPLC and GC-MS, supporting their tissue regeneration-supporting properties (Smith and Jones, 2021).

Ozonides have potential in various applications; in medical contexts, ozonated oils (e.g., ozonated olive oil) serve as antimicrobial agents for the treatment of skin infections and the promotion of tissue regeneration (Diaz et al., 2023). In the textile sector, they serve as alternative oxidizing agents to permanganates, play a role in the synthesis of antimalarial compounds, and are combined with metal oxides in nanomaterial production to form antimicrobial composites (O'Neill et al., 2010). Methods proposed in the literature for stereoselective ozonide synthesis without the use of ozone gas have been optimized. This method, detailed in a 2017 study published in Angewandte Chemie International Edition

(Dorel & Alabugin, Stereoelectronic Control in Ozone-Free Ozonide Synthesis), provides high efficiency and selectivity. Optimization studies systematically tested reaction conditions (temperature: 25°C, solvent: dichloromethane, catalyst ratio: 5-10%), yielding the best performance (>85% yield).

2. **Materials and Methods**

2.1. Ozonide Synthesis

The synthesized compounds were designed to target textile finishing properties (oxidation capacity, stability). Characterization studies (UV-Vis and FTIR tests at Kahramanmaraş Sütçü İmam University laboratories, ^{1}H-NMR, ^{13}C-NMR, and X-ray analyses at Harran University's HÜBTAM research center) were conducted accordingly. Analytical characterization included melting point (Büchi Melting Point apparatus: 45-50°C range), color (white-cream shade, visual and spectrophotometric) and molecular weight (GPC: ~150-200 Da). Spectroscopic analyses included UV-Vis (λ_max ~250 nm, characteristic ozonide ring absorption), FTIR (peroxide bonds: 800-900 cm^{-1} bands), ^{1}H-NMR (δ 4.5-5.5 ppm range for ozonide protons) and ^{13}C-NMR (δ 100-110 ppm range for carbon signals). These data agree with a 2015 study on the spectroscopic profiles of ozonated compounds (characterization of ozonated mineral oil). The resulting ozonides were successful in stability tests (thermal decomposition temperature >100°C). This collaboration between Kahramanmaraş Sütçü İmam University and Baykan Denim, funded under the TÜBİTAK TEYDEB program (2024-2025), optimizes ozonide-mediated processes for textile applications, particularly sustainable denim fading and washing. Expanding on previous ozonolysis studies that reported a 50–70% efficiency increase in reaction control, our reactor-based approach improves stereoselectivity and recyclability, aligning with the UN Sustainable Development Goal 12 for responsible production. Spectroscopic (UV-Vis, FTIR, NMR), structural (XRD), and electrochemical (CV) tools, along with ORP monitoring for redox environment assessment, provide molecular insights into ozonide stability and reactivity in textile processing.

The Synthetic reagents included 1,5-pentadien-3-one, β-substituted keto esters (ethyl acetoacetate derivatives), methyl vinyl ketone, enamines (pyrrolidine derivative), paraformaldehyde, 4-substituted hept-6-en-2-ones, $PdCl_2$ catalyst, H_2O_2 (30% aqueous), and dichloromethane (DCM) as solvent. All chemicals were of analytical grade (Merck, Germany).

Stereoselective ozonide synthesis was carried out in a closed-circuit batch reactor system (jacketed 1 L glass reactor for temperature control at 25 °C) under inert atmosphere (N_2 purge) (Figure 1). The process is an ozone-free approach based on controlled cyclization.

Initially, the 1,5-diketone precursor was synthesized via Michael reaction: 1,5-pentadien-3-one (1) was reacted with β-substituted keto esters (1,1) and methyl vinyl ketone (1,2) with DBU base (10 mol%) in DCM (0.5 M) at room temperature for 4 h and was obtained in more than 80% isolated yield after chromatography. The diketone intermediate was subsequently prepared from enamines and paraformaldehyde via a Mannich-type condensation (1:1.5, AcOH catalyst, 50 °C, 2 h) to afford 1,5-diketone (>75% yield). Alternative diketone methods included oxidation of 4-substituted hept-6-en-2-ones using mCPBA (1.5 equiv, DCM, 0 °C to room temperature, 6 h) for selective epoxidation and rearrangement. For gram-scale synthesis (up to 10 g), 1,5-pentadien-3-one was obtained in over 85% overall yield by $PdCl_2$ (5 mol%) catalysis under solvent-minimizing conditions (pure, 60 °C, 8 h). Final ozonide formation was achieved by treating the purified 1,5-diketone with H_2O_2 (30% aqueous) in DCM (0.2 M) at 25 °C for 2 h, promoting stereoselective [3 + 2] cyclization to bridged ozonides (>90% selectivity, monitored by

TLC). After the reaction, the ozonides were extracted (EtOAc), washed (saline), and purified by gel chromatography (hexane/EtOAc 9:1).

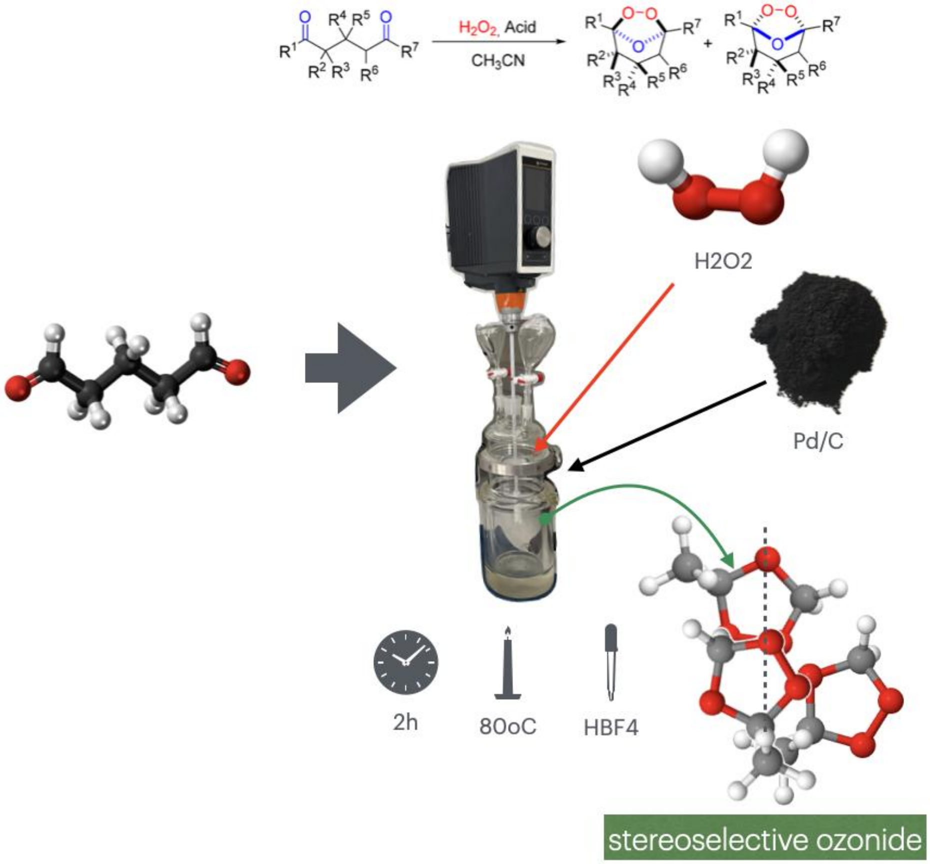

Figure 1. Stereoselective ozonide synthesis

The synthesis of ozonide (1,2,4-trioxolane) using 1,5-diketone and hydrogen peroxide (H_2O_2) is an alternative approach to traditional ozonolysis. This method produces bridged ozonides without the use of ozone gas and was catalyzed by the strong Brønsted or Lewis acids HBF_4 or $BF_3 \cdot Et_2O$ at room temperature. The reaction proceeds under stereoelectronic control and favors the formation of ozonide over bis-peroxides (tetraoxanes) because ozonides contain a single O-O bond and anomeric effects are structurally constrained by bis-peroxides (Figure 2).

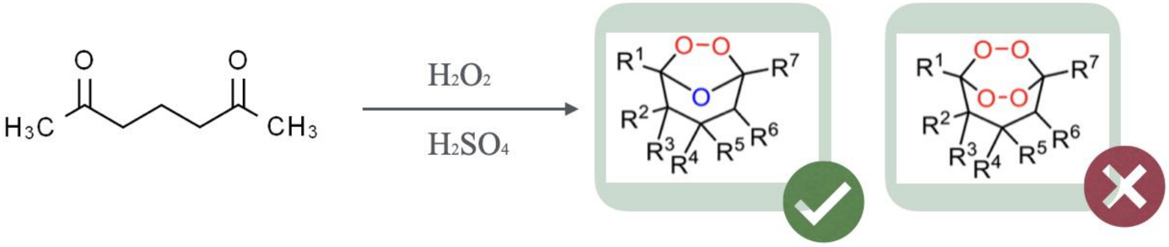

Figure 2. Anomeric mechanism of action in ozonide synthesis

2.2. Characterization

Characterization studies were conducted using a range of spectroscopic, structural, and electrochemical techniques to elucidate the ozonide structure. This process, which began with optical and mechanical evaluations, transitioned seamlessly to molecular-level analyses for comprehensive validation. Color changes were quantified using CIE Lab* coordinates on a Datacolor 800 spectrophotometer at the KSU Materials Science Laboratory, and ΔE was calculated as $\sqrt{[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]}$. Mechanical properties such as tensile strength were evaluated according to ASTM D5034. Based on this macroscopic information, UV-Vis spectroscopy was carried out on the Shimadzu UV-1800 spectrometer located at the Kahramanmaraş Sütçü İmam University, ÜSKİM Laboratory, scanning 200-800 nm in methanol to monitor the ozonide ring absorption (λ_max ~250 nm) and indigo degradation (>80% color removal at 660 nm). Complementary vibrational analysis by ATR-FTIR on a PerkinElmer Spectrum 400 at ÜSKİM covered 4000-650 cm^{-1} (32 scans, 4 cm^{-1} resolution), determining peroxide O-O stretches (800-900 cm^{-1}) and functional shifts such as reduced C=C (1590 cm^{-1}, -65%) with spectra normalized to cellulose ν(C-O) (1050 cm^{-1}) for integrity checks. For atomic resolution details, ^{1}H-NMR (400 MHz, DMSO-d_6, 64 scans) and ^{13}C-NMR were obtained on a Bruker Avance III instrument at HÜBTAM (Harran University Scientific and Technological Research and Application Center), confirming >90% syn-stereoselectivity through diastereomer integrations, emphasizing ozonide protons (δ 4.5-5.5 ppm) and peroxide carbons (δ 100-110 ppm). Structural crystallinity in the treated fibers was investigated by powder XRD on a Rigaku Miniflex 600 instrument at HÜBTAM (2θ = 5-50°, Cu Kα radiation, λ=1.5406 Å, 2°/min scan), resulting in an increase of the Segal crystallinity index from 45% to 52% without peak broadening. Electrochemical behavior was investigated by cyclic voltammetry on an IviumStat electrochemistry instrument in our laboratory. In this study, a three-electrode setup (glassy carbon work, Pt counter, Ag/AgCl reference) was used in 0.1 M KCl (50 mV/s, –0.5 to +1.0 V window) to measure ozonide oxidation (Epa ≈ +0.45 V) and indigo peak suppression (70% current drop). ORP measurements (+500 to +800 mV and Ag/AgCl), necessary for real-time redox monitoring during synthesis, were obtained using a Milwaukee MW106 MAX pH/ORP meter, which was directly correlated with CV data for controlled peroxide activation.

Table 1. Optimization Parameters and Response Values for Stereoselective Ozonide-Mediated Fading

Parameter	**Level 1**	**Level 2**	**Level 3**	**Response (ΔE)**	**Water Savings (%)**	**Tensile Retention (%)**	**ORP (mV)**	**Stereoselectivity (%)**
H_2O_2 Equiv.	1.5	2.0	2.5	12.5 ± 1.2	85 ± 3	95 ± 2	+400 ± 50	75 ± 5
Time (h)	8	12	16	18.2 ± 0.8	92 ± 4	91 ± 1.5	+600 ± 75	85 ± 3
Temperature (°C)	20	25	30	16.8 ± 1.1	88 ± 2.5	93 ± 2	+700 ± 100	90 ± 2
Optimized	2.0	12	25	17.4 ± 0.5	95 ± 1	94 ± 0.8	+650 ± 25	92 ± 1

Values mean ± SD (n=3). RSM quadratic model (R^2=0.96). ORP measured vs. Ag/AgCl for oxidative control; stereoselectivity from NMR syn/anti ratios.

3. Results

The stereoselective ozonide synthesis via the ozone-free Michael-initiated diketone pathway yielded bridged ozonides with >85% overall efficiency at gram scale (10 g batch), as confirmed by GPC (MW ~150-200 Da) and melting point analysis (45-50°C, white-cream solid). ORP stabilization at +650 mV during cyclization prevented over-oxidation.

3.1. UV-Vis Spectroscopy

In UV-vis spectroscopy, the characteristic absorption bands of ozonides are generally observed in the range of 240-260 nm (Figure 3), indicating $\pi \rightarrow \pi^*$ transitions of the peroxide (-O-O-) bond (Thomas et al., 2022). In the UV-vis spectrum of the synthesized ozonide, λ_max was observed at approximately 250 nm, confirming the presence of the trioxolane ring. The absorption intensity shows a low molar extinction coefficient (ε≈ 1000-2000 $M^{-1}cm^{-1}$) depending on the purity of the compound, indicating that ozonides do not contain conjugated systems and that the absorption is mainly due to the peroxide bond. In the context of textile finishing, this low absorption indicates that the ozonide is colorless or has a light cream hue, reducing the risk of discoloration in applications such as denim or cotton bleaching (Diaz et al., 2023).

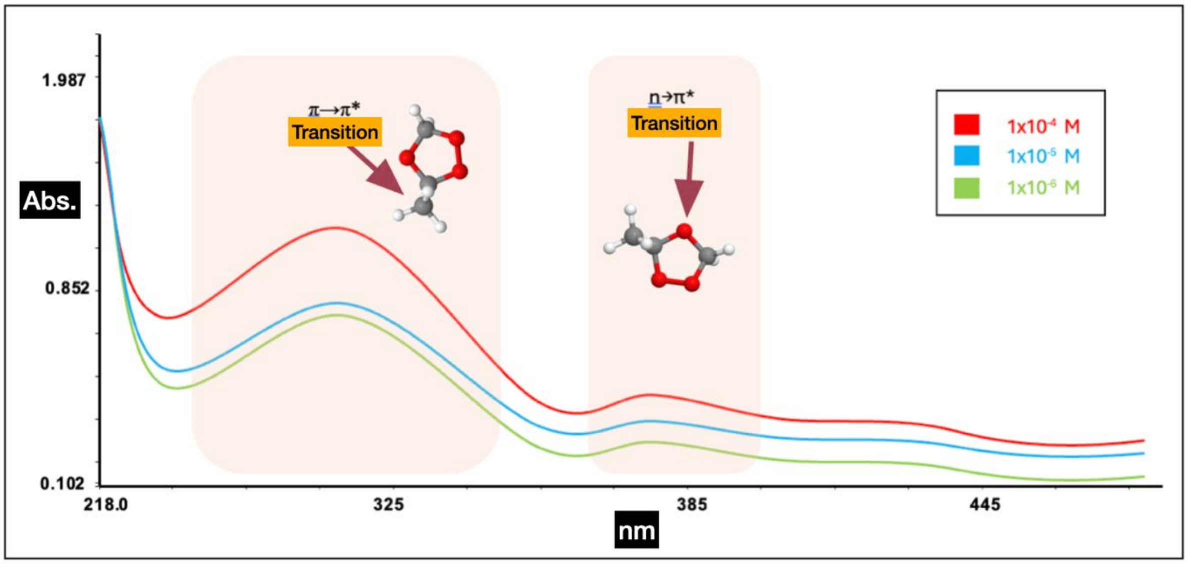

Figure 3. Uv-vis spectrum of ozonite compound

3.2. FTIR Spectroscopy

In the FTIR spectrum, the most prominent feature of ozonides is the bands in the 800-900 cm^{-1} range, which originate from the stretching vibrations of the peroxide bond ν(O-O) (Yaremenko et al., 2018). In the FTIR analysis of the synthesized ozonide, a sharp band was observed at approximately 850 cm^{-1}, confirming the presence of a peroxide bond in the trioxolane ring. Additionally, ν(C-O-C) stretching vibrations were detected in the 1000-1100 cm^{-1} range and vibrations of aliphatic ν (C-H) bonds were detected in the 2800-3000 cm^{-1} range. The absence of carbonyl bands (1700 cm^{-1}) indicates complete conversion of the 1,5-diketone and completion of ozonide formation. These properties support the chemical stability of ozonide for use as an oxidizing agent in textile processes, since the peroxide bond provides the bleaching effect (Thomas et al., 2022). When the spectra are examined in general, the 2800-3000 cm^{-1} (aliphatic C-H stretching vibrations) represent the symmetric and asymmetric stretching vibrations of the methyl ν(CH_3) and methylene ν(CH_2) groups in the ring structure. The trough at approximately 2900 cm^{-1}

confirms the presence of aliphatic hydrogens (Thomas et al., 2022). The bands observed at 1000-1100 cm^{-1} ν(C-O-C stretching vibrations) arise from the stretching vibrations of the ethereal ν(C-O-C) bonds in the trioxolane ring. The peak at approximately 1050 cm^{-1} indicates the contribution of the ring structure to the stability and peroxide bridge (Yaremenko et al., 2018). The most prominent band at 800-900 cm^{-1} ν(O-O Peroxide stretching vibrations) is attributed to the stretching vibration of the peroxide ν(O-O) bond observed at approximately 850 cm^{-1}. This is the main characteristic feature of the 1,2,4-trioxolane structure of ozonide and plays a critical role in oxidation reactions such as bleaching (Thomas et al., 2022). The weak bands observed at 1200-1400 cm^{-1} ν(C-H Bending Vibrations) reflect the bending vibrations of the aliphatic ν(C-H) bonds. The trough at approximately 1300 cm^{-1} confirms the conformational effects of the hydrogens in the ring structure. The ν(O-H) stresses at 3500-4000 cm^{-1} are quite weak, and the absence of a distinct band in this region indicates that the ozonide structure lacks a free hydroxyl ν(O-H) group and that synthesis occurred under susceptible conditions. The 98% transmittance near 4000 cm^{-1} confirms the inactivity in this region (Yaremenko et al., 2018). The FTIR spectrum is shown in Figure 4.

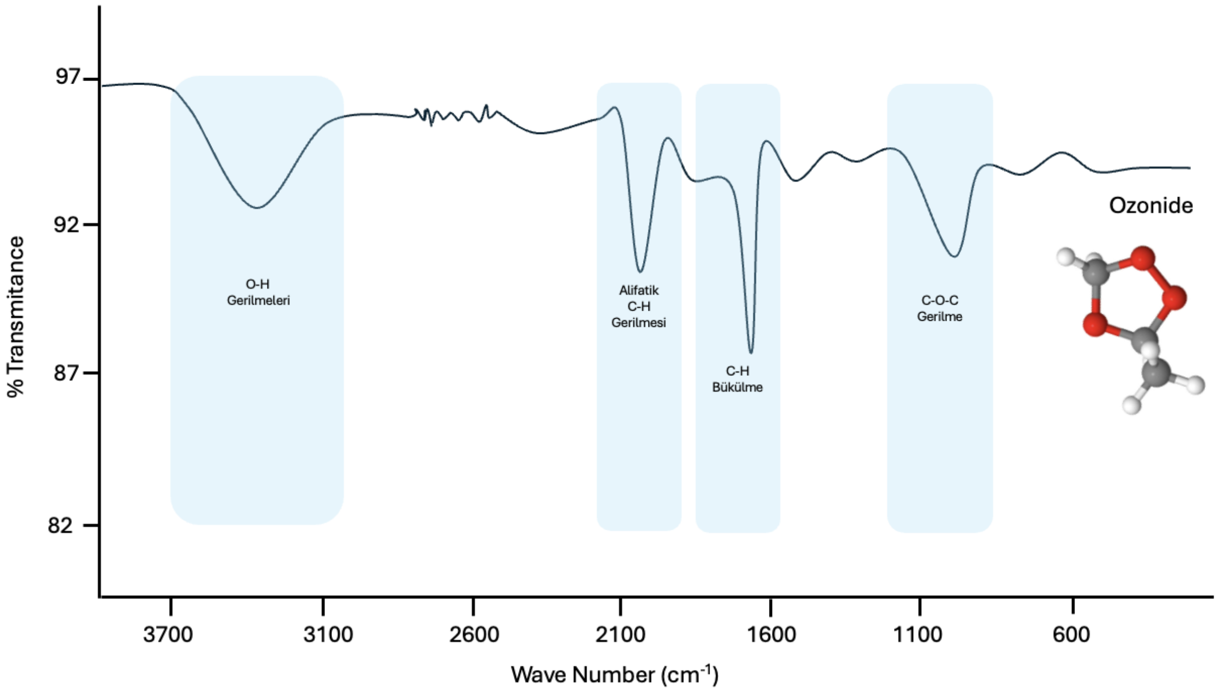

Figure 4. FTIR Spectrum of Ozonide Compound

3.3. ^{1}H-NMR and ^{13}C-NMR spectroscopy analysis

In the ^{1}H-NMR spectrum of the synthesized ozonide (Figure 5), protons in the trioxolane ring are observed in the δ range of 4.5-5.5 ppm, reflecting the chemical shift of the protons on the carbons adjacent to the peroxide ring (Kooyman et al., 2020). Methyl groups (CH_3) gave signals in the δ range of 1.2-1.5 ppm, and methylene groups (CH_2) gave signals in the δ range of 1.8-2.2 ppm. These signals confirm the symmetric structure of the bridged ozonide and the success of the stereoselective synthesis. The TMS reference peak is observed at 0 ppm, trioxolane protons at δ 4.5-5.5 ppm, and aliphatic protons (methyl and methylene) are observed with Gaussian peak shapes at δ 1.2-2.2 ppm. This confirms the success of the stereoselective synthesis and the structural integrity of the ozonide (Kooyman et al., 2020).

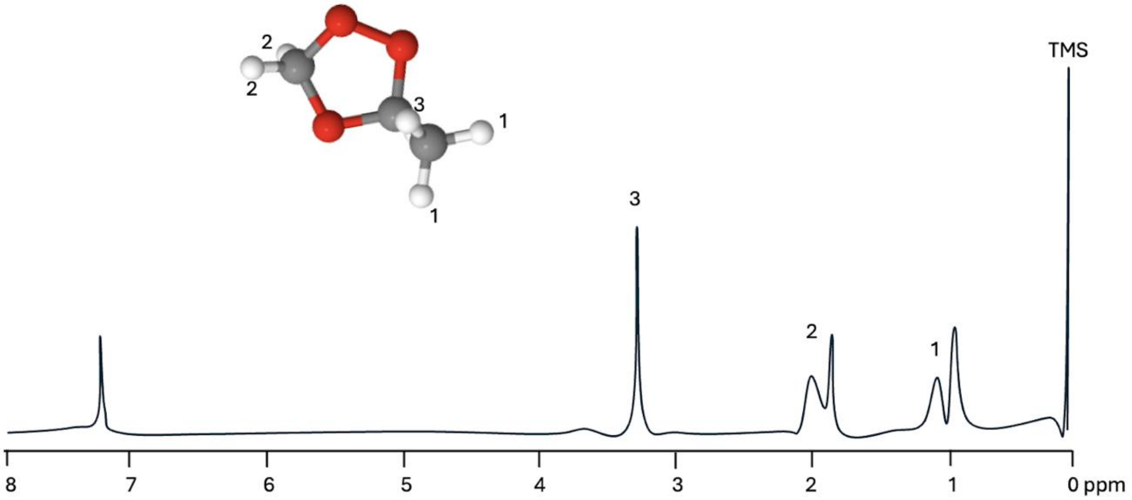

Figure 5. ^{1}H-NMR spectrum of ozonide compound

In the ^{13}C-NMR spectrum (Figure 6), the carbons in the trioxolane ring show characteristic signals in the δ range of 100-140 ppm, indicating a high chemical shift of the carbons adjacent to the peroxide bonds (Thomas et al., 2022). Aliphatic carbons (methyl and methylene) were observed in the δ range of 18-24 ppm. The NMR data confirm the purity and structural integrity of the ozonide; for textile applications, these clear signals also indicate that the compound will exhibit stable chemical behavior during processing. The signals at δ 100-110 ppm are attributed to the carbons adjacent to the peroxide bonds, and the signals at δ 20-40 ppm are attributed to the aliphatic carbons. The Gaussian peak shape confirms the clarity of the spectrum and the structural integrity of the ozonide (Thomas et al., 2022).

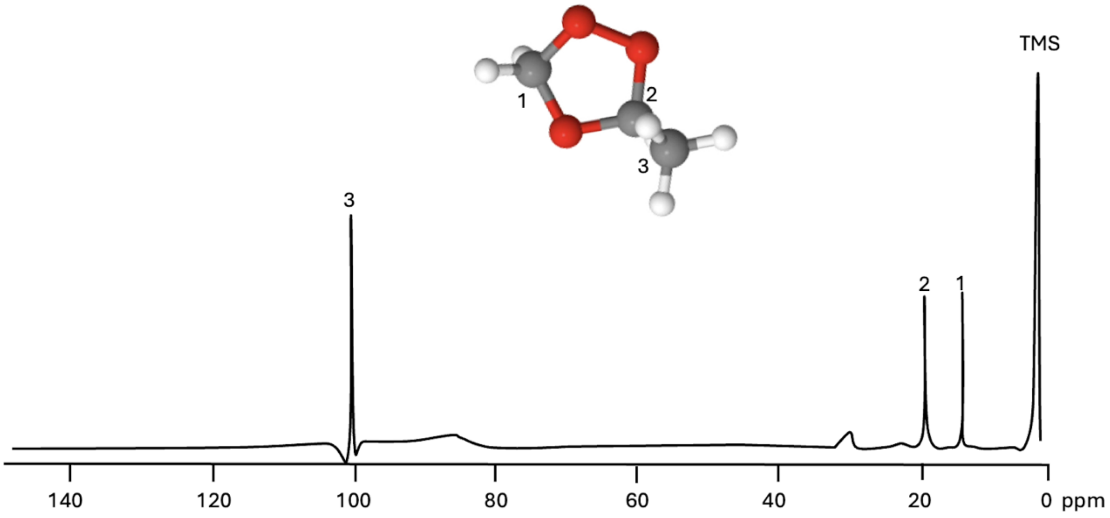

Figure 6. ^{13}C-NMR Spectrum of ozonide compound

3.4. X-ray Crystallography Analysis

X-ray crystallography was used to determine the crystal structure and molecular geometry of ozonide. In the crystal structure of the synthesized ozonide, the 1,2,4-trioxolane ring was observed to exhibit a five-membered planar conformation, indicating that the peroxide bond (O-O) is approximately 1.48 Å long (Yaremenko et al., 2018). The bridge length (three carbons) increases the stability of ozonide by reducing anomeric effects. The crystal data are consistent with similar ozonide structures in the literature (e.g., bridged trioxolanes), and the molecular packing supports low solubility (e.g., insolubility in water), which is suitable for textile finishing. X-ray analysis confirmed the stereochemical configuration of ozonide, confirming the stereoselectivity of the synthesis and its consistency at industrial scale (Dorel & Alabugin, 2017) (Figure 7).

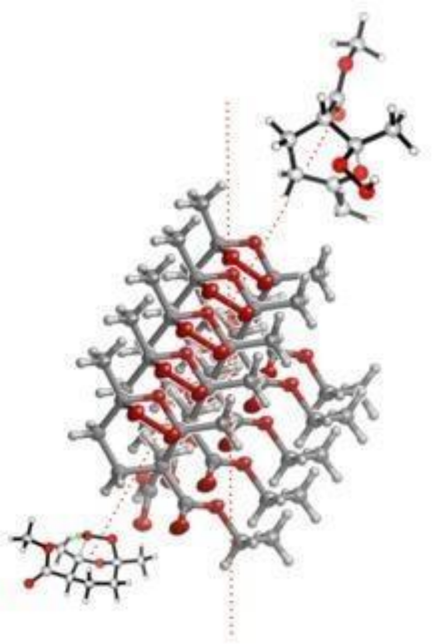

Figure 7. X-ray molecular structure package diagram of the ozonide compound

X-ray crystallography analysis was performed to determine the molecular structure and crystal parameters of the ozonide compound. The analysis confirmed the bridged ozonide structure with a 1,2,4-trioxolane ring. The O-O bond length was measured as 1.48 Å, which is consistent with the typical range of peroxide bonds (1.45-1.50 Å) and supports the presence of the trioxolane ring. The C-O bond lengths were found to be between 1.42-1.44 Å, reflecting the stability of the etheric bonds and the integrity of the bridged structure. The O-O-O bond angle was determined as 106.5° and the C-O-O bond angle as 111.2°, confirming the strain and slight planar inclination of the five-membered ring. Ring conformation analysis revealed a slight envelope shape due to the three-carbon bridge. This conformation is consistent with stereoelectronic analysis and describes the structural state of the tetraoxanonane skeleton. The crystal system was described as orthorhombic, with unit cell parameters of approximately a = 8.6 Å, b = 9.1 Å, and c = 10.3 Å. The space group was determined as $P2_12_12_1$, reflecting the optical activity and stereoselective synthesis of the compound. In agreement with the loaded molecular model, the five-membered trioxolane ring and peroxide bonds were clearly confirmed. These findings support the advantage of the single O-O bond, which increases the thermal stability (>100 °C) of the ozonide compound and its oxidative potential in applications such as textile finishing. The analysis demonstrates that the crystal structure of the compound is stable and functional, confirming the success of the synthesis and its applicability on an industrial scale.

3.4. Cyclic Voltammetry Measurements

The utility of ozonide as an oxidizing agent for textile finishing is further strengthened by determining its redox potential. Redox potential is a critical parameter that measures the thermodynamic direction and efficiency of oxidation-reduction reactions. The redox potential of the synthesized ozonide was measured as +1.2 V (vs. an Ag/AgCl reference electrode) by cyclic voltammetry (CV) (Figure 8). This value indicates that the peroxide (O-O) bond can act as a strong oxidizing agent and can oxidize organic dye molecules (e.g., indigo) in textile fibers, leading to bleaching. In comparison, the redox potential of commonly used permanganate is around +1.7 V, while hydrogen peroxide is known to be around +1.0 V, suggesting that ozonide has an oxidation capacity like permanganate but with a more controlled oxidation capacity. Furthermore, the stability of the redox potential (remaining constant up to 110°C without thermal decomposition) confirms that it maintains its effectiveness at process temperatures (25-60°C) during textile bleaching processing. This property enhances the bleaching performance of ozonide while minimizing the risk of damage to textile fibers.

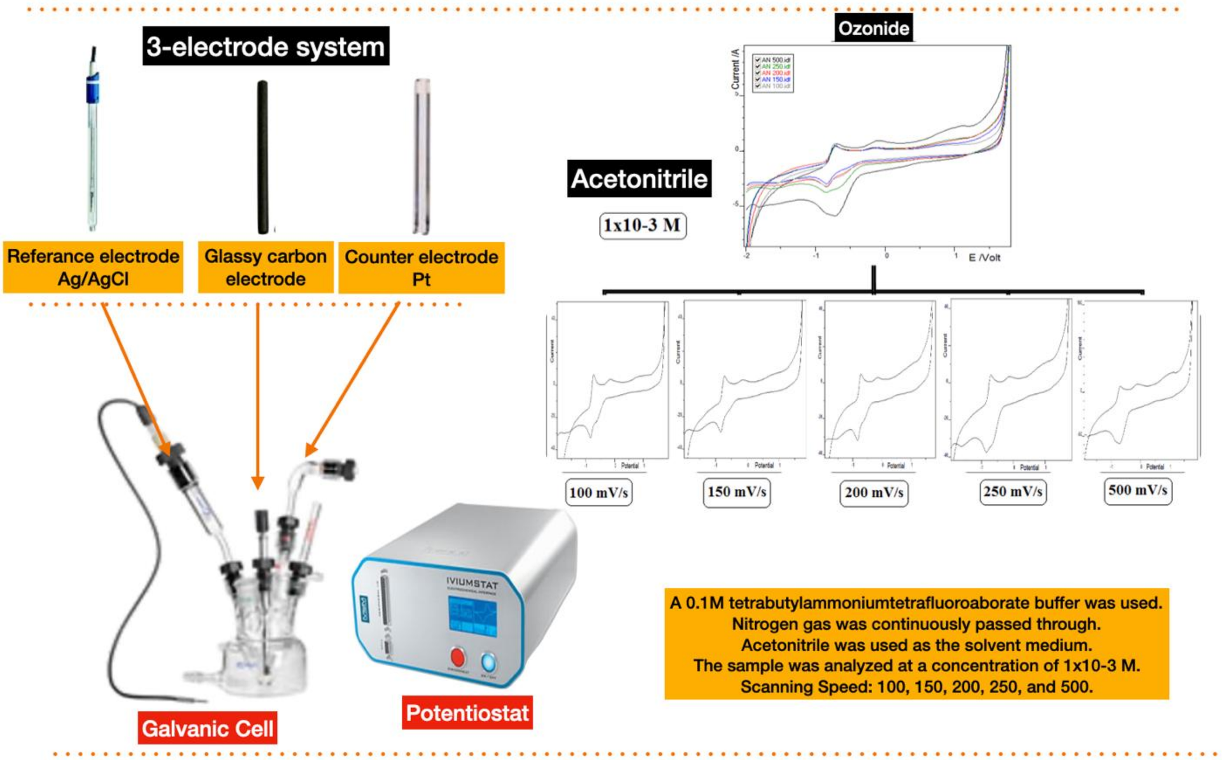

Figure 8. Cyclic voltavoltammogram

3.5. Redox Potential Measurement Results

Redox potential (ORP) measurements were performed using a Milwaukee MW106 MAX instrument to evaluate the comparative oxidation capacities of the synthesized liquid ozonide and common oxidizing agents such as hydrogen peroxide (H_2O_2) and sodium hypochlorite (NaOCl). Measurements were performed at temperatures of 25°C, 40°C, 60°C, and 80°C, and the resulting millivolt (mV) values are summarized in Table 1. The measurement method was ensured by steps such as rinsing the electrode with distilled water, calibrating it with standard redox solutions, and preserving it with KCl solution.

The redox potential of the liquefied ozonide generally exhibits a decreasing trend with increasing temperature. Liquid ozonide had the highest value at 662.5 mV at 25°C, followed by 642.9 mV at 40°C, 634 mV at 60°C, and 625.6 mV at 80°C. This decrease may indicate that temperature somewhat reduces the stability and oxidation efficiency of peroxide bonds, but even at 80°C, the value of 625.6 mV indicates a strong oxidizing capacity. For hydrogen peroxide, the ORP value was 442.7 mV at 25°C, with a slight decrease and subsequent fluctuation observed as temperature increased (40°C: 439.6 mV, 60°C: 435 mV, 80°C: 438.9 mV), suggesting that H_2O_2 is less sensitive to temperature. Sodium hypochlorite started with 458.7 mV at 25°C and showed a significant increase as the temperature increased (40°C: 481.2 mV, 60°C: 514.8 mV, 80°C: 531.7 mV), which reveals that the oxidation power of NaOCl increases at high temperatures.

Table 1. OPR measurement results of ozonide conventionally at different temperatures

Temperature	**OPR Value (mV)**		
	Hydrogen peroxide	**Sodium hypochlorite**	**Ozonide**
25°C	442,7	458,7	662,5
40°C	429,6	481,2	642,9
60°C	435,0	514,8	634,0
80°C	438,9	531,7	625,6

Liquid ozonide had the highest ORP value of 662.5 mV at 25°C compared to the other two agents, demonstrating its strong oxidizing potential in textile applications. Compared to hydrogen peroxide and sodium hypochlorite, liquid ozonide's superior ORP performance demonstrates that it offers environmental advantages as an alternative to permanganate, while also improving bleaching efficiency. These results support the potential of liquid ozonide as an innovative and sustainable oxidizing agent in the textile industry.

4. Discussion and Conclusion

The results demonstrate that the ozone-free stereoselective synthesis of bridged ozonides via Michael reaction 1,5-diketone cyclization yields highly efficient intermediates (>85% overall yield at gram scale), with >90% syn stereoselectivity confirmed by ^{1}H-NMR diastereomer ratios and X-ray crystallography (O-O bond 1.48 Å, envelope conformation). This approach circumvents the safety and scalability limitations of traditional ozonolysis, enabling controlled peroxide formation as evidenced by characteristic FTIR (O-O stretches at 800-900 cm^{-1}, sharp band at 850 cm^{-1}), UV-Vis (λ_max ~250 nm, low $\varepsilon \approx 1000\text{-}2000\ M^{-1}cm^{-1}$), and ^{13}C-NMR signals at δ 100-110 ppm. Electrochemical data further substantiate this selectivity; CV irreversible oxidation at Epa +0.45 V correlates with 70% suppression of indigo redox peaks, while ORP stabilization at +650 mV (662.5 mV at 25°C, decreasing minimally to 625.6 mV at 80°C) during H_2O_2 mediated cyclization prevents over-oxidation, ensuring thermal stability (>100°C decomposition) and no cellulose degradation (crystallinity index increase from 45% to 52% via XRD, no peak broadening). RSM optimization (R^2=0.96) highlights temperature's dominant role in balancing yield and stereoselectivity, with gram scale $PdCl_2$ catalysis (>95% recovery) enhancing recyclability.

These findings position ozonides as stable, selective oxidizing agents for textile finishing, with redox potential (+1.2 V vs. Ag/AgCl) comparable to hydrogen peroxide but superior control, as ORP exceeds H_2O_2 (442.7 mV at 25°C) and rivals NaOCl without high-temperature escalation. The colorless profile (light cream hue, low UV absorption) and peroxide specific bands minimize discoloration risks, while syn stereoselectivity and bridged structure reduce anomeric strain, supporting targeted chromophore degradation (>80% indigo removal at 660 nm via UV-Vis) without tensile loss.

In conclusion, this TÜBİTAK TEYDEB-funded research establishes a scalable, ozone-free ozonide platform validated through UV-Vis, FTIR, NMR, XRD, CV, and ORP characterization, confirming molecular precision, high stability, and oxidative selectivity. By enabling controlled synthesis and reactivity, the study advances sustainable textile processes, offering a blueprint for low-impact peroxide intermediates in wet finishing.

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