



University of Belgrade,
Technical Faculty in Bor

Chamber of Commerce
and Industry of Serbia

XVI International Mineral Processing & Recycling Conference



Proceedings



Editors:
Zoran ŠTIRBANOVIĆ
Milan TRUMIĆ

28-30 May 2025
Belgrade, Serbia





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ANODE NANOMATERIAL RECOVERED FROM SPENT BATTERIES FOR PEROXIDE-ASSISTED CRYSTAL VIOLET PHOTOCATALYTIC DEGRADATION

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ABSTRACT – This study explores the photocatalytic degradation of Crystal Violet (CV) using anode nanomaterials recovered from spent nickel-metal hydride (NiMH) batteries, activated by hydrogen peroxide (H₂O₂). The research focuses on the potential application of recycled perovskite-based materials, such as Lanthanum Cobalt Oxide (LaCoO₃), Nickel Oxide (NiO), and Cerium Dioxide (CeO₂), for the efficient removal of organic dyes from textile industry wastewater. Structural characterization via X-ray diffraction (XRD) confirmed the presence of crystalline phases with high crystallinity, essential for photocatalytic activity. Photocatalytic experiments were conducted under UV-A light ($\lambda = 395$ nm) at pH 5.5, demonstrating a degradation efficiency of 99.52% within 60 minutes. The reaction followed pseudo-first-order kinetics, with a significant increase in efficiency over time. These findings highlight the potential of using recycled battery materials as sustainable and effective photocatalysts for wastewater treatment, offering a promising solution to mitigate environmental contamination caused by synthetic dyes and other persistent organic pollutants.

Keywords: Anode Material, Perovskites, Crystal Violet, Photocatalysis.

INTRODUCTION

Textile industry wastewater is a significant source of environmental contamination due to its high concentration of organic pollutants, particularly synthetic dyes. These dyes are known for their chemical stability and resistance to biodegradation, making their removal from water bodies a major challenge. The uncontrolled discharge of such pollutants into aquatic ecosystems leads to severe consequences, including reduced water quality, disruption of aquatic life, and long-term ecological imbalances [1].

In 2020, it was estimated that approximately 200,000 tons of organic dyes were released into textile industry wastewater, posing a substantial environmental threat. The accumulation of these persistent contaminants not only affects aquatic organisms but

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also compromises human health through the contamination of drinking water sources. The widespread presence of dye pollutants in industrial effluents highlights the urgent need for effective and sustainable wastewater treatment methods to mitigate their adverse effects on ecosystems and environmental quality [2].

Traditional methods for dye removal from wastewater are often inefficient or economically unsustainable, driving the search for alternative approaches based on advanced oxidation processes (AOPs) [3]. Hydroxyl radicals generated during AOP exhibit strong oxidative properties, enabling them to degrade complex organic molecules into smaller, environmentally benign compounds such as water and carbon dioxide [4].

Photocatalytic degradation is one of the most promising solutions for breaking down organic pollutants, utilizing photocatalysts that, in the presence of light, generate reactive oxygen species capable of decomposing complex molecules into smaller, environmentally friendly products [5]. Special attention in recent years, has been given to the use of recycled materials, such as perovskite materials similar to the cathode materials that can be obtained from waste batteries [6, 7]. These materials can serve as a very efficient catalyst for dye degradation when activated by hydrogen peroxide.

This paper presents the efficiency of photocatalytic degradation of crystal violet using anode material from spent nickel-metal hydride (NiMH) batteries in combination with hydrogen peroxide. The aim is to examine the degradation kinetics, reaction mechanisms, and the potential application of this method in wastewater treatment.

EXPERIMENTAL

Sample Preparation

The examined sample originated from a discarded battery of an unidentified manufacturer. To ensure safe handling and prevent short circuits, the battery was first disassembled after fully discharging its six individual cells. The discharge process was carried out using a laboratory-made apparatus equipped with a 5.5 Ω resistive wire, enabling controlled discharging of the battery samples.

A hacksaw was used to remove the cell terminals, followed by a longitudinal cut to systematically separate the plastic casing, metal casing, anode, cathode, and separator. The anodic material was subjected to thermal treatment at 580°C for 10 minutes, facilitating its detachment from the current collector. Anodic material was finely ground using an agate mortar and pestle. To maintain sample integrity and prevent cross-contamination, meticulous cleaning of the equipment was performed between uses.

RESULTS AND DISCUSSION

X-ray Diffraction Analysis

XRD analysis was conducted via Rigaku MiniFlex 600 (Japan) to characterize the structural properties of the anode material derived from waste batteries and assess its suitability as a photocatalyst for the degradation of Crystal Violet. The diffraction patterns provide insights into the crystalline phases present, potential impurities, and any structural modifications resulting from material processing. The XRD pattern of anode material is shown in Figure 1.

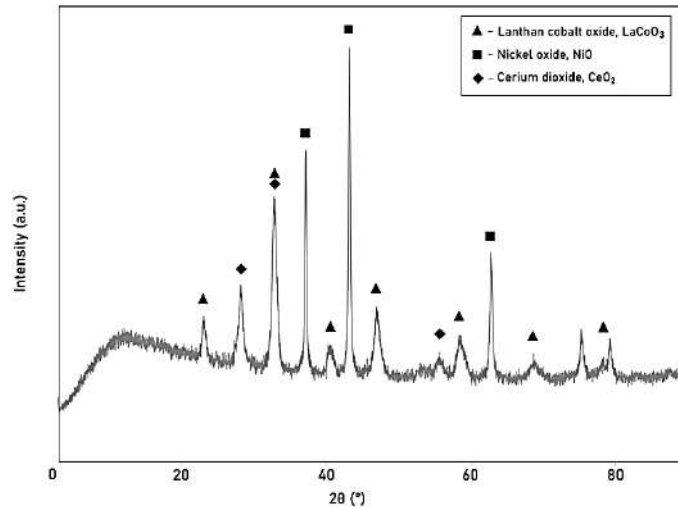


Figure 1 XRD analysis of anode material

The X-ray diffraction (XRD) pattern shown in Figure 1., reveals the crystalline phases present in the analyzed sample, confirming the presence of multiple components. The identified peaks correspond to Lanthanum Cobalt Oxide (LaCoO_3), Nickel Oxide (NiO), and Cerium Dioxide (CeO_2), as indicated by the reference diffraction patterns. The presence of LaCoO_3 suggests that the anode material retains its perovskite structure, which is crucial for its potential photocatalytic applications [6].

XRD pattern of analyzed material presented a high-intensity peak at 32.7° , which might be associated with planes (110) and/or (104), corresponding to hexagonal crystal symmetry with an $R3c$ space group, assuming angles $a=b=90^\circ$ and $\gamma = 120^\circ$, further allowing for calculation of crystal lattice constants [8].

The size of crystallites was assessed using the Scherrer equation: $D = 0.9\lambda / \beta \cos\theta$, where D denotes the crystallite size, λ represents the X-ray wavelength (1.5418 \AA), θ corresponds to the Bragg diffraction angle, and β signifies the full width at half maximum (FWHM) of the diffraction peak. To evaluate the crystallite size of the sample, the peak broadening of the (024) reflection of the LaCoO_3 phase at $2\theta = 46.9^\circ$ was analyzed [8].

Crystal lattice constants for the LaCoO_3 phase are determined as $a=b \approx 5.47 \text{ \AA}$ (hexagonal system) and $c \approx 13.39 \text{ \AA}$, while crystallite size calculated from the (024) peak is approximately 8.84 nm . The volume cell is determined to be $V \approx 346.8 \text{ \AA}^3$.

Based on characteristic peaks and confirmed planes (111) and (002), crystal lattice constants for NiO in the cubic system are determined as $a=b=c \approx 4.18 \text{ \AA}$, while the crystallite size calculated from the (002) peak is approximately 34.8 nm . The unit cell volume is determined to be $V \approx 73.1 \text{ \AA}^3$.

Crystal lattice constants for CeO_2 in the cubic system are determined as $a=b=c \approx 5.45 \text{ \AA}$, while the crystallite size calculated from the (111) peak is approximately 11.0 nm . The unit cell volume is determined to be $V \approx 161.9 \text{ \AA}^3$.

The strong and sharp diffraction peaks could indicate a high degree of crystallinity, suggesting that the material possesses well-ordered structural characteristics [9]. The presence of NiO and CeO₂ as additional phases might contribute to enhanced catalytic properties [10]. These structural insights are essential for understanding the material's reactivity and effectiveness in the photocatalytic degradation of CV when activated with hydrogen peroxide.

Photocatalytic Degradation and Kinetics

Degradation experiments were carried out using a previously prepared 20 ppm crystal violet solution to investigate photocatalytic properties, and accessed via CGOLDENWALL 721 UV-VIS spectrophotometer (China). The experiments were performed with exposure to UV-A light ($\lambda = 395$ nm), with the addition of peroxide in order to modify the pH. The collected data were analyzed using a pseudo-first-order model, expressed as [11]:

$$\ln\left(\frac{C_0}{C_t}\right) = kt \quad (1)$$

Here, C_0 and C_t denote the concentrations of the model compound at the initial time (zero) and at time t , respectively, while k represents the rate constant of the reaction. Figure 2(a) illustrates the degradation efficiency of the CV with the addition of H₂O₂. The relationship between $\ln(C_0/C_t)$ and the irradiation time t is presented in Figure 2(b), showing a linear trend. The rate constants for the reaction were determined by calculating the slopes of the $\ln(C_0/C_t)$ versus time plots.

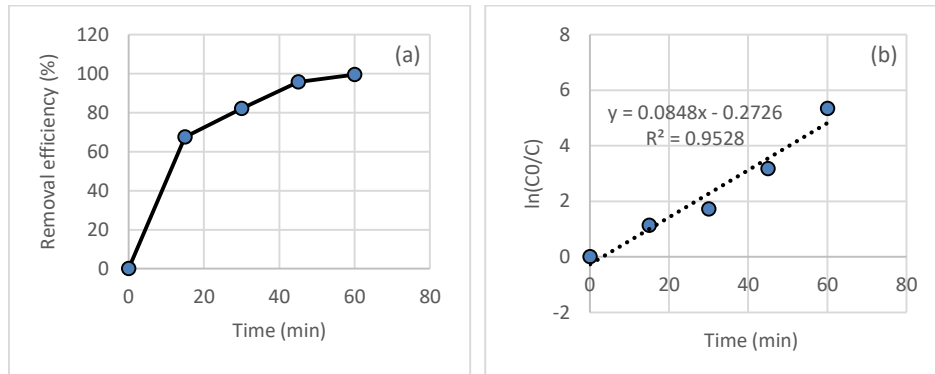


Figure 2 (a) Photocatalytic degradation efficiency of crystal violet, (b) Kinetic curve

The photocatalytic process shows a significant increase in efficiency over time. At 15 minutes, the degradation efficiency is 67.57%, reaching 99.52% at 60 minutes, indicating near-complete degradation of CV. CV degradation follows pseudo-first-order kinetics, which is typical for photocatalytic reactions.

In conclusion, the combination of this anode material and H₂O₂ demonstrates high efficiency in degrading CV, following pseudo-first-order kinetics. Most of the dye is degraded within the first 45 minutes, and near-complete removal is achieved within 60 minutes. This suggests that the anodic material is a promising candidate for the effective removal of CV from wastewater.

CONCLUSION

This study has shown that the combination of recycled anode material and H₂O₂ is highly effective in breaking down complex organic molecules, such as CV, into environmentally benign compounds. The use of recycled materials, such as perovskite-based anode nanomaterials, offers a sustainable and cost-effective approach to wastewater treatment. By repurposing waste from spent batteries, this method not only addresses the issue of environmental contamination caused by synthetic dyes but also contributes to the circular economy by reducing electronic waste. The findings suggest that these materials have significant potential for large-scale applications in industrial wastewater treatment, particularly in the textile industry, where dye pollution is a major concern.

In conclusion, this study highlights the feasibility of using recycled anode nanomaterials as efficient photocatalysts for the degradation of organic dyes. The combination of these materials with H₂O₂ under UV light provides a promising solution for the removal of persistent pollutants from wastewater, offering both environmental and economic benefits. Further research could explore the optimization of reaction conditions and the application of this method to other types of organic pollutants, paving the way for broader implementation in environmental remediation technologies.

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