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Original Article

Optimization of Dye Waste Degradation Through Electrolysis with PbO_2/Pb from Used Batteries

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Abstract *- Chemical laboratories have a risk of danger due to the use of chemicals, including liquid waste that can pollute the environment. Laboratory liquid waste contains hazardous organic and inorganic compounds. One of the laboratory liquid wastes is dye waste. Waste treatment is important to reduce the negative impacts and hazards to health and the environment. One effective method for degrading dye waste is Electrodecolorization, an electrochemical process for degrading dyes using electric current. This method is able to degrade dye waste without producing new waste. This study aims to determine the optimization of the degradation of dye waste from the laboratory by electrolysis using PbO_2/Pb electrodes from used batteries. This study was conducted by electrolyzing dye waste with variations in working potential, variations in electrolysis time, and variations in electrode distance. The absorbance of dye waste before and after electrolysis was measured using a UV-Vis Spectrophotometer at a wavelength of 330 nm. The results showed that the optimization of dye waste degradation occurred at a working potential of 6 V, electrolysis time of 90 minutes, and electrode distance of 1 cm.*

Keywords *- Degradation, Dye Waste, Electrodecolorization, Used Batteries, Electrolysis.*

1. Introduction

Chemical laboratory activities have the potential to pose hazards to users due to the risk of exposure to chemicals in the form of gases, liquids, concentrated suspensions, or powders [1]. Laboratory hazards include the toxic levels found in various chemicals [2]. Continuous laboratory activities involving testing will lead to an increase in the amount of liquid waste generated, classified as Hazardous and Toxic Materials (HTMs) [3].

Waste management must be carried out in accordance with applicable procedures to minimize the risk of accidents in the laboratory [4]. Laboratory waste includes dye-containing waste [5]. Water pollution caused by dyes is one of the most significant environmental problems, with around 80% of wastewater being discharged directly into the environment without prior treatment. This situation can exacerbate the impact of pollution on aquatic ecosystems [6]. Some types of dyes are toxic and have carcinogenic and mutagenic effects on aquatic life and humans [5].

Numerous studies have been conducted on the removal of color and organic compounds from industrial textile wastewater (Dye Waste) [7]. One of the efforts to reduce the harmful impact of dye waste in laboratories is by applying an alternative treatment method using electrolysis. The treatment of colored wastewater through this process is known as the Electrodecolorization method [8]. Electrodecolorization is a more effective, efficient, and economical method for degrading dyes [9].

Degradation is the process of breaking down polymer chains into smaller molecules [10]. The degradation of complex compounds into shorter and simpler molecular chains eliminates the toxic, carcinogenic, and mutagenic properties of the waste. This chemical structural change not only reduces environmental hazards but is also marked by the disappearance of color in the waste, serving as a visual indicator of the degradation process's success. The Electrodecolorization method is highly efficient and capable of reducing dye pollutants without generating new waste. One of the critical factors influencing the success of the electrolysis process is the selection of electrode materials [11]. The use of PbO_2/Pb from used car batteries as the anode can be a solution to the issue of the unavailability of ideal anodes, those with perfect stability and activity, which remains a significant challenge in the electrodecolorization of wastewater containing organic substances [9].

Previous Electrodecolorization studies predominantly employed commercial PbO_2-based or mixed-metal oxide electrodes and typically required external electrolytes, such

as Na_2SO_4, to improve solution conductivity. These approaches increase operational costs and contribute additional chemical waste. Furthermore, most investigations were conducted using pure synthetic dye solutions, which do not represent the complex and low-conductivity characteristics of real laboratory wastewater [9], [12]. There is a need for a low-cost electrochemical system that operates without added electrolytes and utilizes waste-derived electrodes while still achieving high decolorization efficiency in real wastewater samples.

This study addresses these gaps by investigating the electrochemical degradation of dye-containing laboratory wastewater using PbO_2/Pb electrodes reclaimed from spent lead–acid batteries. Repurposing these waste-derived electrodes provides a cost-effective alternative while simultaneously offering an environmentally responsible approach to managing discarded batteries. The Electrodecolorization process is performed without the addition of Na_2SO_4 or any supporting electrolyte, relying solely on the intrinsic conductivity of the wastewater. This strategy aims to demonstrate a practical and more sustainable treatment method for real laboratory dye waste.

2. Methods

2.1. Type and Design Research

This research is an experimental study that involves optimization tests for the degradation of laboratory dye waste through electrolysis using used car batteries.

2.2. Tools and Materials

2.2.1. Tools

The tools used include a stirring rod, a suction bulb, a spray bottle, a vial bottle, a Buchner funnel, a PbO2/Pb electrode from a used car battery, a beaker glass, a watch glass, a clamp, a cuvette, a Buchner flask, a volumetric pipette, a dropper, a vacuum pump, power supply, UV-Vis spectrophotometer, stand, and stopwatch.

2.2.2. Materials

The materials used include distilled water (Aquadest), filter paper, laboratory dye waste, and solid Na_2SO_4.

2.3. Work Procedure

2.3.1. Tools

The tools used include a stirring rod, a suction bulb, a spray bottle, a vial bottle, a Buchner funnel, a PbO2/Pb electrode from a used car battery, a beaker glass, a watch glass, clamp, cuvette, Buchner flask, volumetric pipette, dropper, vacuum pump, power supply, UV-Vis spectrophotometer, stand, and stopwatch.

2.3.2. Materials

The materials used include distilled water (aquadest), filter paper, laboratory dye waste, and solid Na_2SO_4

2.3. Work Procedure

2.3.1. Sample Filtration

The laboratory dye waste is separated from impurities or sediments by vacuum Buchner filtration using filter paper as the medium.

2.3.2. Determination of Maximum Wavelength

The maximum wavelength is determined by measuring the absorbance of laboratory dye waste in the range of 250 to 700 nm. The measurement is carried out by scanning the wavelength using a UV-Vis spectrophotometer. The wavelength with the highest absorbance in the visible region is identified as the maximum wavelength.

2.3.3. Variation of Applied Voltage

A total of 100 mL of laboratory dye waste is placed into a beaker glass. Electrodecolorization is performed at 2 V for 15 minutes. The absorbance of the dye solution before and after electrodecolorization is measured using a UV-Vis spectrophotometer at the maximum wavelength. This step is repeated with variations of applied voltage at 3, 4, 5, and 6 V. The percentage of decolorization is determined for each variation.

2.3.4. Variation of Electrolysis Time

A total of 100 mL of laboratory dye waste is placed into a beaker glass. Electrodecolorization is carried out at the optimum working voltage for 15 minutes. The absorbance of the solution before and after electrodecolorization is measured using a UV-Vis spectrophotometer at the maximum wavelength. This step is repeated with time variations of 30, 45, 60, 75, 90, 105, and 120 minutes. The percentage of decolorization is calculated for each time variation.

2.3.5. Variation of Electrode Distance

A total of 100 mL of laboratory dye waste is placed into a beaker glass. Electrodecolorization is conducted using the optimum working voltage and optimum electrolysis time with an electrode distance of 1 cm.

The absorbance of the solution before and after electrodecolorization is measured using a UV-Vis spectrophotometer at the maximum wavelength. This step is repeated with electrode distances of 2 and 3 cm. The percentage of decolorization is calculated for each distance variation.

2.3.6. Analysis Method

Quantitative analysis in the equation (1) is carried out by measuring the absorbance before and after electrolysis, followed by calculating the decrease in color intensity (percentage of decolorization).

$$Percentage\ of\ decolorization = \frac{initial\ absorbance - final\ absorbance}{initial\ absorbance} \times 100\% \quad (1)$$

3. Results and Discussion

The research began by determining the maximum wavelength. This was based on the absorbance measurement of the waste using a UV-Vis spectrophotometer within the wavelength range of 250–700 nm. Figure 1 shows the highest absorption peak of 1,734 at a wavelength of 330 nm. The maximum wavelength (λ max) is essential in decolorization analysis because it indicates the point at which the dye absorbs light most effectively. The amount of light absorbed by the dye waste results in significant changes in absorbance values.

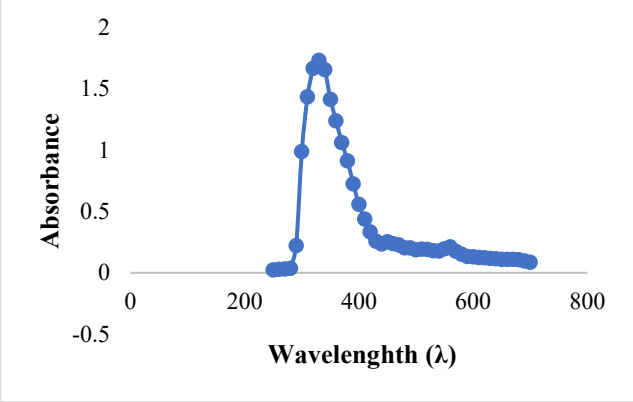

Fig. 1 Relationship between Wavelength and Absorbance

The wavelength range of 250–380 nm represents the region of dissolved organic matter [13], which is defined as a collection of organic molecules dissolved in water that cannot be filtered using a specific pore-sized filter. These molecules vary in size and structure and are capable of passing through filters with a pore size of 0,45 µm [14]. The wavelength range of 380–750 nm represents the turbidity region [13], where the transparency of a liquid decreases due to the presence of insoluble substances known as cloudy or turbid water. As is known, turbidity is one of the indicators that the water is not clean or safe for consumption [15].

The sample testing was first conducted by comparing the use of an additional electrolyte, Na_2SO_4, with a condition without added Na_2SO_4 under the same time, distance, and voltage conditions. The results showed that the absorbance value of the laboratory dye waste without the addition of the electrolyte (Na_2SO_4) was lower than that of the waste with the addition of Na_2SO_4. This indicates that the electrolysis process can occur without the need for external electrolytes, making the process more economical by reducing the cost of additional chemicals [16]. Electrodecolorization was carried out using lead (Pb) as the cathode and lead dioxide (PbO_2) as the anode. The decolorization process proceeds more rapidly due to a tandem mechanism that breaks down the structure of the dye molecules. Decolorization occurs through two reaction pathways which is conventional redox reactions and reactions involving radicals. In this process, a portion of the water undergoes radical reactions, while the rest is involved in redox reactions. The involvement of radicals is based on the formation of hydroxyl radicals (•OH) during electrolysis. These hydroxyl radicals attack dye molecules and break them down into simpler compounds. The formation of hydroxyl radicals (•OH) takes place on the surface of the PbO_2 electrode as a result of the electrical energy supplied by the power source. The PbO_2 electrode acts as an effective medium for generating hydroxyl radicals, which then attack the dye compounds. This triggers a chain reaction that accelerates the degradation of the dye molecules [17], as described in the following reactions [18].

$$PbO_2 + H_2O \rightarrow PbO_2\,[\bullet OH] + H^+ + e^-$$
$$R\bullet\,[\bullet OH] \rightarrow \bullet R + H^+ + e^-$$
$$\bullet R + H^+ + e^- \rightarrow CO_2 + H_2O + \text{byproducts}$$

Based on the reactions above, R represents the organic substrate of the dye compound, consisting of elements such as C, H, O, and others. The oxidation products at the anode are estimated to be H_2O and CO_2 if the process occurs completely, along with the formation of short-chain carbon compounds that are no longer able to absorb radiation in the visible light wavelength range [18].

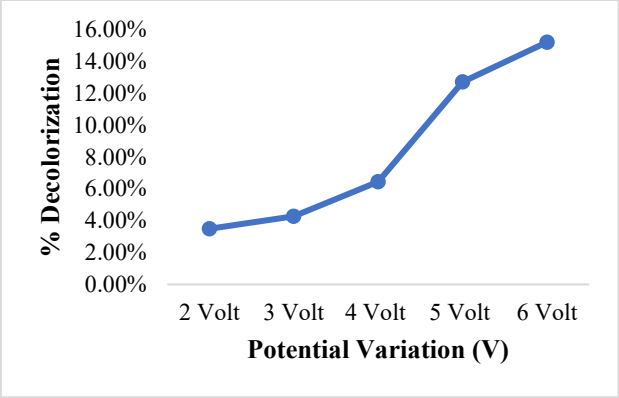

Fig. 2 Relationship between Applied Voltage Variation in Electrodecolorization and Decolorization Percentage

The applied voltage was tested by conducting electrodecolorization at 2, 3, 4, 5, and 6 V for 15 minutes with an electrode distance of 2 cm. Absorbance was then measured at a wavelength of 330 nm to determine the percentage of decolorization. Figure 2 shows that the optimum working potential was achieved at 6 V, with a decolorization percentage of 15,19%. An increase in applied voltage directly affects the efficiency of decolorization through variations in electrochemical mechanisms that occur at each voltage level. At low potentials (2–4 V), the dominant reaction is the direct oxidation of dye molecules on the electrode surface, where electrons are directly transferred from the dye molecules to the electrode. However, the production of free radicals such as hydroxyl radicals (•OH) remains minimal due to the low electric current, thus limiting

decolorization efficiency (Bergaoui et al., 2006). At higher potentials (5–6 V), the electric current increases, enhancing the production of hydroxyl radicals (•OH). This accelerates dye degradation through an indirect oxidation mechanism, where free radicals randomly attack dye molecules, thereby maximizing decolorization efficiency [19].

Conductivity tests were conducted on both the sample and distilled water. The conductivity of the sample was approximately 147 µS/cm, which is higher than that of distilled water (<1 µS/cm), but still considered low to effectively support electrochemical operations at high voltages. Low conductivity results in high solution resistance (R) and heat release. According to Ohm's Law (V = I/R), this high resistance limits the electric current generated [20]. The inability of the power supply to reach voltages above 6 V indicates that internal resistance within the system caused either by electrode passivation or low solution conductivity is restricting current flow, even when the voltage is increased. Electrode passivation occurs when a non-conductive layer forms on the surface of the PbO_2/Pb electrode. This layer obstructs electron and ion transfer, reduces the active surface area of the electrode, and increases resistance. The conductivity of approximately 147 µS/cm suggests that the sample contains ions; however, the concentration is not sufficient to support a high current flow at voltages above 6 V.

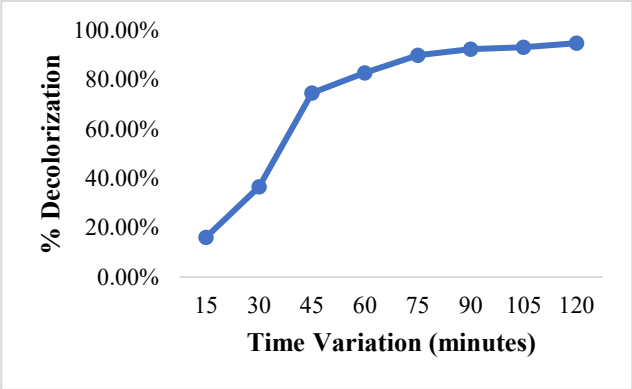

Fig. 3 Relationship between Electrodecolorization Time Variation and Percentage of Decolorization

Optimization of electrolysis time was carried out by performing electrodecolorization at durations of 15, 30, 45, 60, 75, 90, 105, and 120 minutes using the optimum working voltage and an electrode distance of 2 cm. Absorbance was then measured at a wavelength of 330 nm to determine the percentage of decolorization. Figure 3 shows that the percentage of decolorization increases with longer electrolysis time. This indicates that the longer the electrolysis process, the more dye molecules are degraded. Based on the results, the optimum electrolysis time was determined to be 90 minutes, with a decolorization percentage of 92,30%. Although the reaction continues slightly beyond 90 minutes, the additional effect is minimal (<1%) and does not justify the extra time, energy consumption, or potential risks. Therefore, the 90-minute mark is selected as the optimum electrolysis time.

The decrease in decolorization efficiency may indicate the occurrence of side reactions that produce reactive intermediate compounds, which absorb light at specific wavelengths [21]. This is possibly due to prolonged electrolysis duration and electrode corrosion, which can trigger the re-adsorption of dye molecules onto the electrode surface. Meanwhile, fluctuations or spikes in decolorization values may result from imbalances in electrochemical reactions or disturbances during the initial stages of electrolysis, when the number of oxidizing radicals generated is still insufficient to effectively break down the molecular structure of the dye compounds.

This performance aligns with previously reported findings on PbO_2-based electrochemical oxidation, where high decolorization efficiencies are typically obtained through advanced oxidation on the PbO_2 surface. However, many earlier studies relied on synthetic dye solutions and required additional electrolytes such as Na_2SO_4 to increase conductivity [22, 23]. In contrast, the present work demonstrates that comparable efficiency can be achieved using real laboratory wastewater without introducing supporting electrolytes.

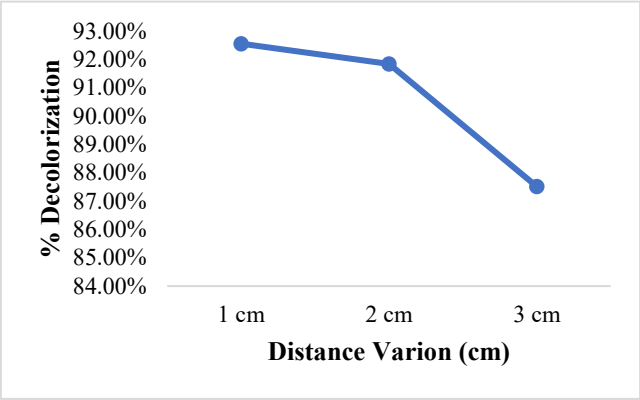

Fig. 4 Relationship between Electrode Distance Variation in Electrodecolorization and Percentage of Decolorization

Optimization of electrode distance was conducted by performing electrodecolorization at electrode gaps of 1, 2, and 3 cm using the optimum working voltage (6 V) and electrolysis time (90 minutes). Absorbance was measured at a wavelength of 330 nm to determine the percentage of decolorization. Figure 4 shows that the optimum distance was 1 cm, achieving a decolorization percentage of 92,55%. Based on the graph showing the relationship between decolorization percentage and electrode distance variation, it is evident that electrode spacing significantly affects the efficiency of the electrolysis process. The greater the

distance between electrodes, the lower the decolorization efficiency. This decline occurs because an increased electrode gap leads to higher solution resistance, resulting in decreased electric current flow [21]. Consequently, the formation of oxidizing radicals is reduced, and the degradation of dye compounds becomes less effective. In addition, an excessively wide electrode gap lowers mass transfer efficiency and increases the risk of forming unwanted by-products due to incomplete reactions. The spacing also affects the interaction time between the electrodes and the solution, enough to allow the reactions to occur efficiently without causing short circuits or excessive current resistance.

Based on the findings, the optimum electrode distance is 1 cm, as it produces a higher electric current compared to the 2 cm and 3 cm distances. The increased current accelerates the formation of hydroxyl radicals (•OH), which play a crucial role in breaking down dye compounds, making the decolorization process faster and more efficient. This spacing maintains a balance between electric current strength, even current distribution, and optimal contact time between the electrodes and the solution. These conditions support the maximal generation of oxidizing radicals, allowing the dye degradation process to proceed more completely.

A key novelty of this study is the use of PbO_2/Pb electrodes fabricated from components taken from used batteries. This recycling-based approach decreases reliance on commercially manufactured electrodes and reduces hazardous battery waste, providing a low-cost and more sustainable alternative. The recovered PbO_2 and Pb materials were able to function effectively as anode and cathode components during the electrolysis process [25]. Another important outcome of this work is that high decolorization efficiency was achieved without the addition of Na_2SO_4 or other supporting electrolytes. Operating the system using only the inherent conductivity of the laboratory wastewater simplifies the procedure and reduces chemical consumption, which is advantageous for small-scale laboratory treatment systems [11]. The decolorization is understood to proceed through a combination of direct anodic oxidation at the PbO_2 surface and indirect oxidation by hydroxyl radicals (•OH) generated during electrolysis. This mechanism is consistent with established electrochemical oxidation pathways reported for PbO_2 anodes. The high oxygen evolution overpotential characteristic of PbO_2 enables more energy to be directed toward pollutant oxidation, promoting effective degradation under moderate operating voltages [26, 27].

Potential limitations of the system include the possibility of surface passivation during prolonged operation, a challenge that has been noted in various PbO_2-based electrochemical systems. Further studies may evaluate reaction intermediates and explore improvements in the pretreatment of battery-derived materials to enhance long-term electrode durability PbO_2. Overall, the findings indicate that electrodes fabricated from spent lead-acid batteries can be applied as a cost-effective and environmentally conscious option for the treatment of dye-containing laboratory wastewater, particularly in contexts where commercial electrodes or supporting electrolytes are limited.

4. Conclusion

Based on the optimization study of laboratory dye waste degradation using the electrolysis method with electrodes from used car batteries, the results showed that the maximum wavelength was observed at 330 nm; the optimum working potential was 6 V with a decolorization percentage of 15,19%; the optimum electrolysis time was 90 minutes with a decolorization percentage of 92,30%; and the optimum electrode distance was 1 cm with a decolorization percentage of 92,55%. The degradation results indicated that the sample became clearer with lower light absorbance, suggesting that the waste was effectively degraded while maintaining a stable pH. For future research, optimization can be conducted more easily with the assistance of software such as Response Surface Methodology (RSM).

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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