**Indonesian Journal of Environmental Management and Sustainability**
***e*-ISSN:2598-6279 *p*-ISSN:2598-6260**

**Research Paper**

# Recalcitrant Industrial Wastewater Treatment Using Fenton and Photo-Fenton Oxidation: A Comparison Study

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**Abstract**

The growth rate in agro-industrial sectors has both positive and negative effects on technological, social, and economic development. Agro-industry production generates substantial volumes of wastewater, primarily from the aqueous discharges of its manufacturing processes. Some of this wastewater contains harmful pollutants that endanger human life, health, and the sustainability of the environment and ecosystem. For example, wastewater from the bioethanol industry contains high concentrations of organic pollutants and recalcitrant compounds, with COD and BOD values exceeding 50,000 mg/L and 30,000 mg/L, respectively. The Fenton process is an oxidation method that generates hydroxyl radicals through the reaction between $H_2O_2$ and $Fe^{2+}$ ions. These hydroxyl radicals are highly effective at breaking down recalcitrant compounds. In this study, a comparative analysis of recalcitrant wastewater treatment using Fenton and photo-Fenton oxidation processes was conducted. The effects of dilution factors, or initial concentrations of recalcitrant wastewater (1:25, 1:50, and 1:75), were examined. Higher dilution ratios enhanced the degradation of COD and BOD levels in wastewater, with the optimal dilution factor for both processes being 1:75. Under optimal conditions, the removal efficiencies for COD, BOD, potassium, and phenol were in the range of 72.29-99.99%. The photo-Fenton process demonstrated higher removal efficiency compared to the Fenton process. The conclusion from this study suggests that the photo-Fenton process could be successfully employed as an advanced treatment method for effectively breaking down recalcitrant wastewater. These findings could be useful for adapting these processes to field-scale applications.

**Keywords**

Fenton, Organic, Photo-Fenton, Recalcitrant, Wastewater

Received: 3 May 2024, Accepted: 10 August 2024
`https://doi.org/10.26554/ijems.2024.8.3.100-109`

## 1. INTRODUCTION

One of the primary causes of life on Earth is the amount of liquid water on the planet's surface. Water represents 71% of the earth's surface and is almost everywhere. However, development and careless human activities have contaminated freshwater supplies, leading to a scarcity of clean water. It is concerning that the industrialized world discards approximately 80% of its wastewater into the environment untreated despite its high volume. Numerous pollutants may be present, such as pathogens, fat, oils, grease, nutrients, and inorganic materials (Dutta et al., 2021; Hakika et al., 2022). All of these substances have adverse effects on human health and the environment, including eutrophication, the development of anaerobic conditions in water bodies, and mutagenic, teratogenic, and carcinogenic impacts on human health (Aziz et al., 2016; Zhu et al., 2015). Expanding contamination of organic and inorganic pollutants by resources is one of the most critical environmental concerns of our time (Behrouzeh et al., 2022).

Industrial wastewater with recalcitrant contaminants must be treated efficiently to remove these pollutants. Components of recalcitrant contaminants in the context of wastewater degradation are resistant to biodegradation and deterioration through the conventional biological degradation process (Ahmed et al., 2022). This pollutant is frequently an organic compound that is difficult to destroy and are able to endure in the environment, causing harm to the human body and ecosystem. Recalcitrant wastewater is generally originated from a variety of sources, including paper mills, tanneries, textile industries, bioethanol, petrochemical treatment, as well as industrial operations, pharmaceutical, agricultural practices, and human activities like cleaning and washing (Wang et al., 2022; Kakavandi and Ahmadi, 2019). The sectors have the potential to produce wastewater that contains significant amounts of organic matter, heavy metals, and other contaminants that are challenging to eliminate utilizing conventional biological treatment methods (Rao and Shrivastava, 2020). Additionally, the high levels of Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) in this effluent surpass pollution control limits, consequently posing significant harm to the environment. Conventional treatment methods have been reported for treating wastewater containing recalcitrant component, including adsorption (Ribeiro et al., 2020; Ahile et al., 2020), coagulation-flocculation (García and Hodaifa, 2017; Yang et al., 2021), and filtration (Ghazzaf et al., 2022). However, these conventional methods have limitations when dealing with recalcitrant pollutants (Biragova et al., 2020; Singh et al., 2022).

Sustainable Development Goals' target 6.3 is: 'By 2030, improve water quality by reducing pollution, eliminating dumping and minimizing release of hazardous chemicals and materials, halving the proportion of untreated wastewater and substantially increasing recycling and safe reuse globally'. Thus, wastewater treatment method is necessary to minimize the quantity of organic and inorganic pollutants in the wastewater before it is released into the environment. The researchers have developed many strategies, such as membrane filtration, activated carbon, electrochemical oxidation to reduce the amount of dissolved oxygen in the atmosphere (Galvão et al., 2006; Palaniandy et al., 2016). Several wastewater treatments should include degradation and mineralization of organic pollutants to an efficient, renewable, cost-effective, and to a less difficult process for the treatment of recalcitrant compounds (Ali et al., 2013; Hasan et al., 2012).

The advanced oxidation-degradation process (AOP) has been shown to be an effective method to reduce recalcitrant activity because it produces highly reactive radicals that can break down complex organic structures with a strong resistance to chemical, and physical conventional treatment methods. AOP are the most effective method of removing organic and inorganic pollutants from wastewater because persist after biological treatment (Alcalá-Delgado et al., 2018). Among these, the Fenton and photo-Fenton process is particularly appealing due to its strong ability to produce hydroxyl radicals and oxidize a wide range of organic pollutants. This reaction utilizes high oxidation oxidative potential from $H_2O_2$ when iron (Fe) is used as a catalyst in an acidic environment. The chemical reaction involved in Fenton processes are shown in Equations (1)–(6) (Hakika et al., 2019).

$$\mathrm{Fe}^{2} + \mathrm{H_2O_2} \rightarrow Fe^{3+} + \cdot\mathrm{OH} + \mathrm{OH}^{-} \tag{1}$$

$$\mathrm{OH}^{-} + \mathrm{H_2O_2} \rightarrow \mathrm{HO_2} + \mathrm{H_2O} \tag{2}$$

$$\mathrm{Fe}^{2+} + \cdot\mathrm{OH} \rightarrow \mathrm{Fe}^{3+} + OH^{-} \tag{3}$$

$$\mathrm{Fe}^{3+} + \mathrm{H_2O} \rightarrow \mathrm{Fe}^{2} + \mathrm{O_2} + H^{+} \tag{4}$$

$$\cdot\mathrm{OH} + \cdot\mathrm{OH} \rightarrow \mathrm{H_2O_2} \tag{5}$$

$$\text{Organic Pollutant} + \cdot\mathrm{OH} \rightarrow \text{Degraded Product} \tag{6}$$

Fenton and photo-Fenton reactions produce extremely potent and highly oxidizing radicals enable the destruction of a wide variety of organic compounds (Tavares et al., 2022). Photo-Fenton reaction which includes the interaction between hydrogen peroxide and ion iron (II), is combined with the strong oxidation hydroxyl radicals produced by UV radiation in this procedure. According to the results, organic matter, especially that which is resistant to biological degradation, may be preserved using this method. Photo-Fenton reaction combines UV radiation with Fenton's reaction ($Fe^2+/H_2O_2$) to procedure highly reactive hydroxyl radicals that can break down complex organic pollutants (Wang et al., 2023).

These methods have been shown to effectively treat a wide variety of wastewater types. The application of Fenton and photo-Fenton processes is promising for treating diverse and variable waste streams, offering low cost and short reaction times while also eliminating the need for large technological devices. Due to the potential of these AOPs in degrading persistent organic pollutants, a comparative study of Fenton and photo-Fenton processes is required to be investigated. Studies have demonstrated that both Fenton and photo-Fenton processes offer promising treatment options for some non-complex wastewaters (Al-Balushi et al., 2023; Ikrari et al., 2022). Nevertheless, only a few studies have explored the treatment of actual recalcitrant wastewater using these methods. Comparing the Fenton and photo-Fenton processes for treating recalcitrant wastewater would highlight the significance of these AOPs for environmental remediation. This study aims to evaluate and compare the effectiveness of the Fenton and photo-Fenton methods for treating recalcitrant wastewater. One crucial factor that may affect the effectiveness of these processes is the initial concentration of the wastewater. Increased initial concentration can lead to a greater demand for oxidizing agents, potentially negatively impacting overall process effectiveness. Therefore, the impact of the dilution factor on the performance and degradation of organic pollutants was also examined.

## 2. EXPERIMENTAL SECTION

### 2.1 Materials

Recalcitrant wastewater is obtained from a distillery unit of industry producing bioethanol derived from sugar fermentation in Yogyakarta, Indonesia. Physico-chemical properties

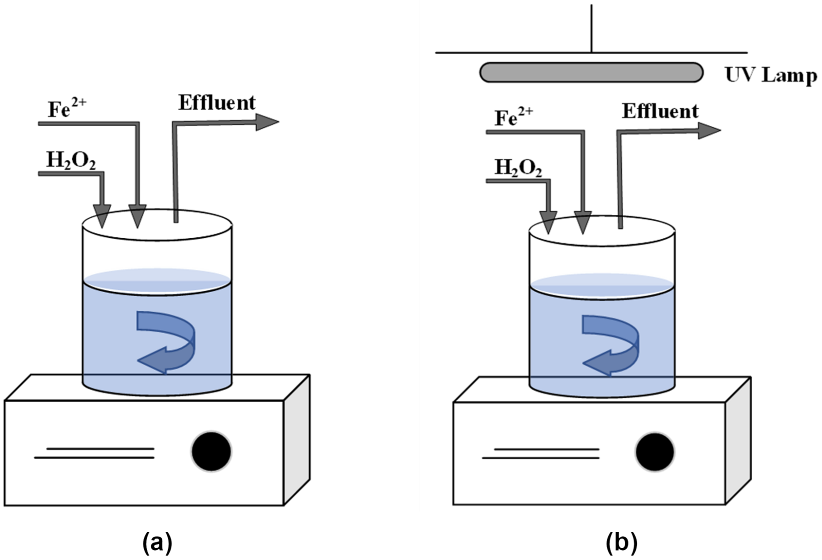


**Figure 1.** Experimental Apparatus Treatment of Recalcitrant Wastewater Using: (a) Fenton and (b) Photo-Fenton Method

of recalcitrant wastewater is shown in Table 1. Chemical reagents used in this study included hydrogen peroxide 30% ($H_2O_2$ 30% w/w, Merck), ferrous sulfate heptahydrate ($FeSO_4.7H_2O$, Merck), natrium hydroxide (NaOH, Merck) and distilled water.

**Table 1.** Physico-Chemical Properties of Recalcitrant Wastewater

| Parameter | Unit | Value |
|---|---|---|
| COD | mg/L | 66,500 |
| BOD | mg/L | 39,500 |
| pH | - | 4.00 |
| Potassium | mg/L | 1.77 |
| Phenol | mg/L | 3.13 |
| Color | - | Dark Brown |

### 2.2 Fenton and Photo-Fenton Treatment

Fenton and photo-Fenton experiments were carried out in a laboratory scale reactor with a 1 L capacity at the Laboratory of Bioprocess and Environment, Universitas Ahmad Dahlan. Both the Fenton and photo-Fenton methods were conducted using a batch reactor. For the photo-Fenton treatment, the reactor was equipped with an external UV lamp system installed at the top of the reactor. A magnetic stirrer was used to ensure thorough mixing of the reagents and solution. Three dilution factors of raw wastewater (1:25; 1:50; 1:75) were used in this experiment. A schematic diagram of the experimental apparatus is shown in Figure 1.

For the Fenton method, the reactor was first filled with 500 mL recalcitrant wastewater solution, which was diluted to 1:25, 1:50, and 1:75 with distilled water. Then, Fenton reagents and were added to the sample with constant dosage ($H_2O_2 = 3.3$ g/L and $FeSO_4.7H_2O_2 = 0.6$ g Fe/L) (Biddinika et al., 2024). Subsequently, the photo-Fenton experiment was carried out according to the same protocol as the Fenton process, with the solution then being irradiated with a UV lamp for 30 minutes. The solution for both methods was constantly stirred for 60 minutes at ambient temperature to complete the reaction. At 15-minute intervals, supernatants of the solution were withdrawn for the analysis of pH, Total Dissolved Solids (TDS), and Oxidation-Reduction Potential (ORP). After the reaction concluded, NaOH was used to adjust the pH of the solution to 7, thereby stopping the Fenton reaction and precipitating iron oxides. Once precipitation was completed, the liquid was separated, and analyses for COD, BOD, potassium, and phenol were performed.

### 2.3 Analytical Method

The pH, TDS, and ORP values were measured using a Lutron YK-2001PHA. Standard methods were employed to measure the concentrations of COD (APHA Method 5220), BOD (APHA Method 5120), potassium (APHA Method 3500-K), and phenol (APHA Method 5530) in the samples. The removal efficiency of each pollutant (COD, BOD, potassium, phenol) in recalcitrant wastewater was calculated using Equation (7) as follows:

$$\%\text{Pollutants Removal} = \frac{P_0 - P}{P_0} \times 100\% \qquad (7)$$

Where $P_0$ : concentration of pollutants (COD, BOD, potassium, phenol) (mg/L) $P$ : final concentration of pollutants (COD, BOD, potassium, phenol) (mg/L)

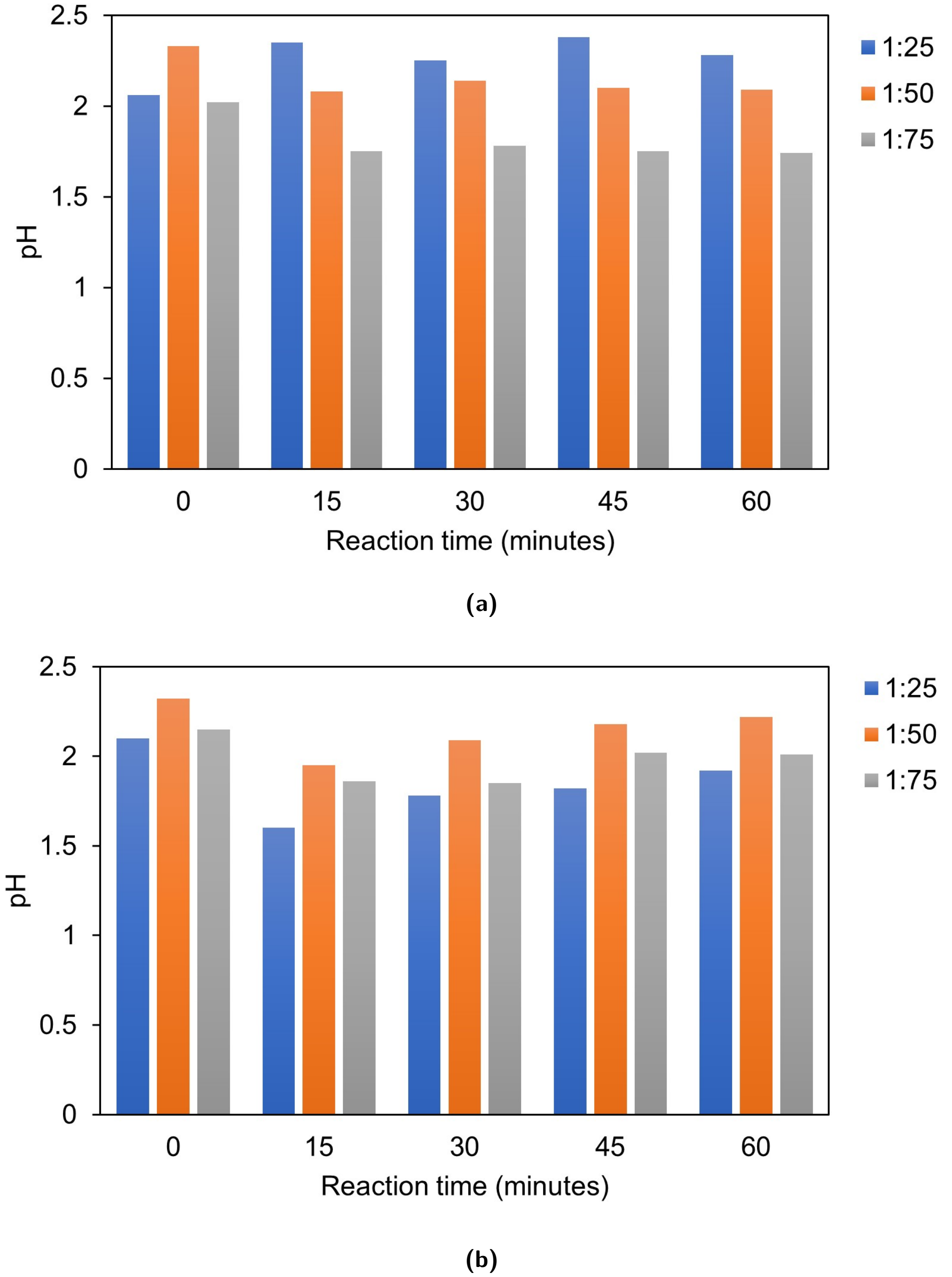


**Figure 2.** Effect of Dilution factor of Bioethanol pH Profile During: (a) Fenton and (b) Photo-Fenton

## 3. RESULT AND DISCUSSION

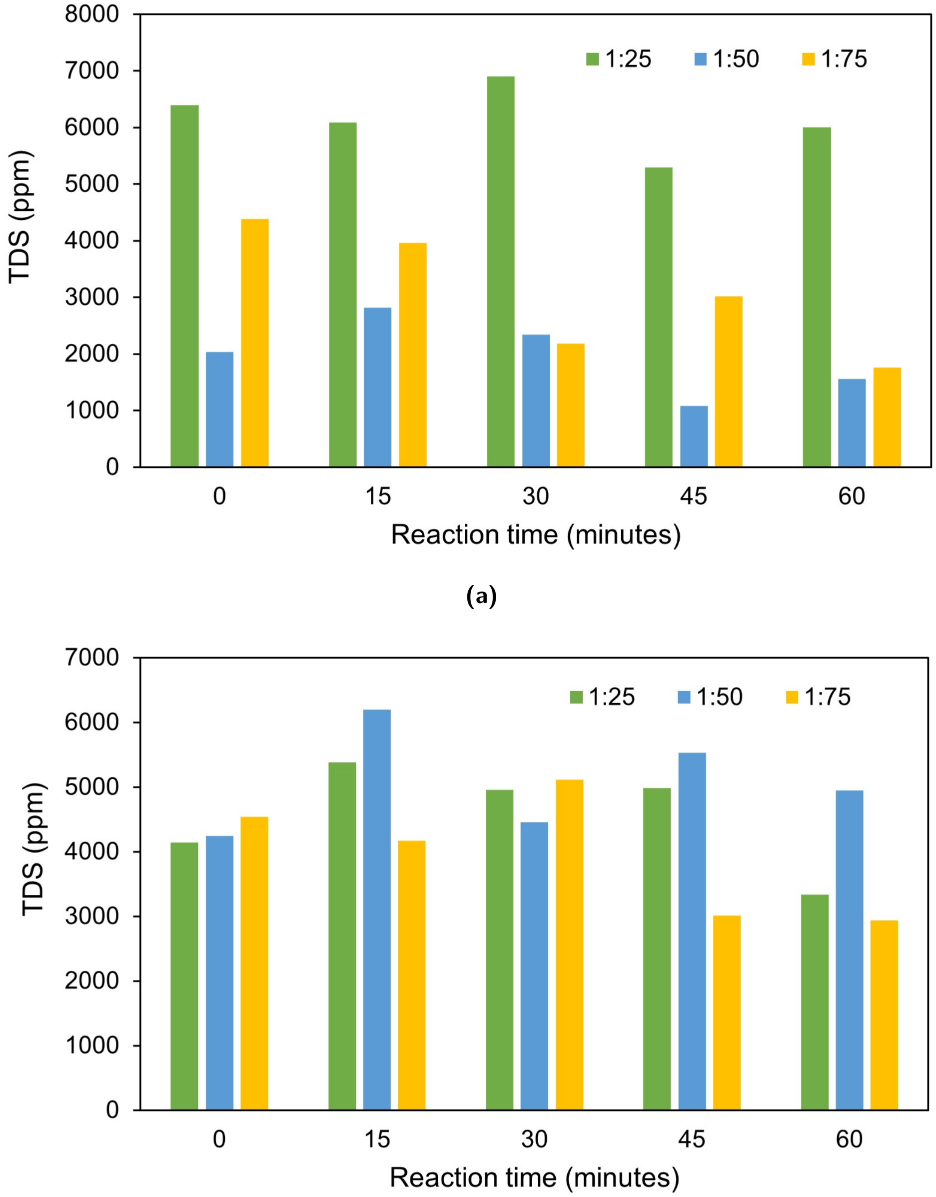
### 3.1 Effect of Dilution Factor of Wastewater During Fenton and Photo-Fenton Method

Three different dilution factors of recalcitrant wastewater (1:25, 1:50, 1:75) were experimentally investigated in this study. The dilution factor significantly affects the performance of Fenton and photo-Fenton treatments, as evidenced by the parameters discussed below.

### 3.1.1 pH Profile

Figure 2 (a) and (b) shows the pH profile during Fenton and photo-Fenton treatment, respectively.

pH is a crucial parameter in the Fenton and photo-Fenton processes. The pH during each treatment ranged from 1.5 to 2.5 under all conditions. Previous studies have indicated that the Fenton reaction proceeds most rapidly at pH values between 2 and 3, enhancing the yield of hydrogen peroxide. Within this range, hydrogen peroxide remains in its most



(a)

(b)

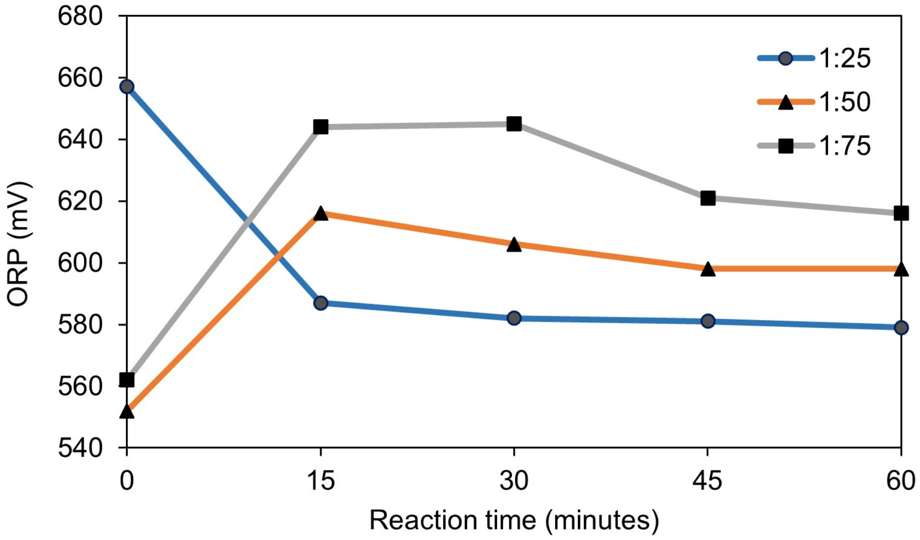
**Figure 3.** Effect of Dilution Factor on TDS Profile During: : (a) Fenton and (b) Photo-Fenton

stable condition (Bolobajev et al., 2014; De La Obra et al., 2017; Hakika et al., 2022).

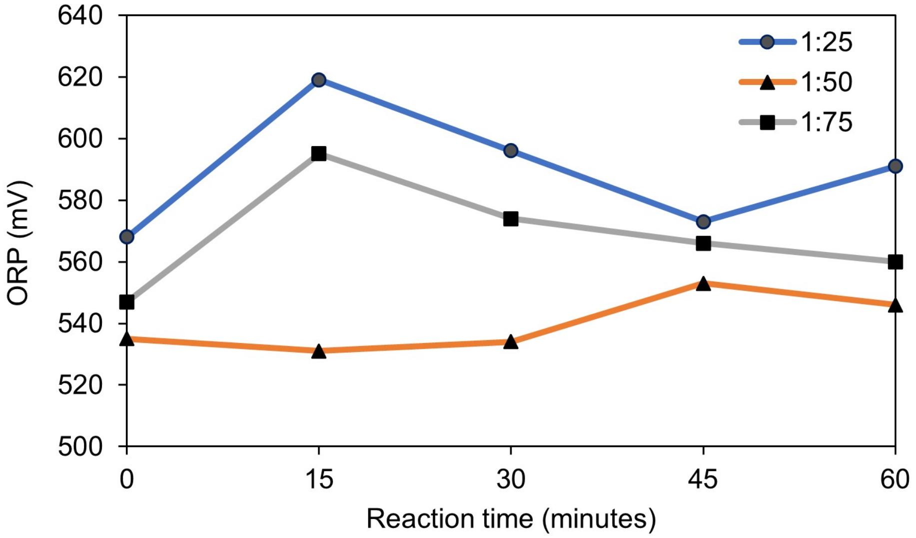
### 3.1.2 TDS Profile

The effect of dilution factor during Fenton and photo-Fenton treatment on the TDS profile is presented in Figure 3 (a) and (b).

TDS is a critical parameter in assessing water quality, representing the total concentration of inorganic and organic substances dissolved in water. Results show that lower TDS levels were obtained by increasing the dilution factor of wastewater. Higher TDS levels can negatively impact the efficiency of the Fenton and photo-Fenton processes by reducing the effectiveness of the reaction between iron ions and hydrogen peroxide. This condition decreases the availability of radicals and affects the pH range of the solution, which is critical for the Fenton and photo-Fenton reactions (Al-Balushi et al., 2023; Carbajo et al., 2021). Thus, the optimum dilution factor was found to be 1:75.



**(a)**



**(b)**

**Figure 4.** Effect of Dilution Factor on ORP Profile During: : (a) Fenton and (b) Photo-Fenton

### 3.2 ORP Profile

Figure 4 (a) and (b) presented the profile of ORP during Fenton and photo-Fenton reaction under three different dilution factors of wastewater. It is shown that dilution factor has a big impact on performance of Fenton and photo-Fenton treatment based on ORP profiles.

ORP is influenced by the formation of hydroxyl radicals, which are responsible for the degradation of organic pollutants. Additionally, ORP is affected by the presence of inorganic carbon and the hydrolytic speciation of iron ions, both of which are influenced by the pH value. ORP measurement is a key parameter in evaluating the redox activity and oxidizing capability of the Fenton system, as an increase in ORP values leads to a significant improvement in the oxidation process (Arbter et al., 2022; Vitale et al., 2016). Figure 4 shows that during Fenton treatment, the highest ORP was obtained with a dilution factor of 1:75, followed by 1:50 and 1:25. However, with a dilution factor of 1:25, the ORP profile decreased significantly within the first 15 minutes of the reaction. Therefore, a dilution factor

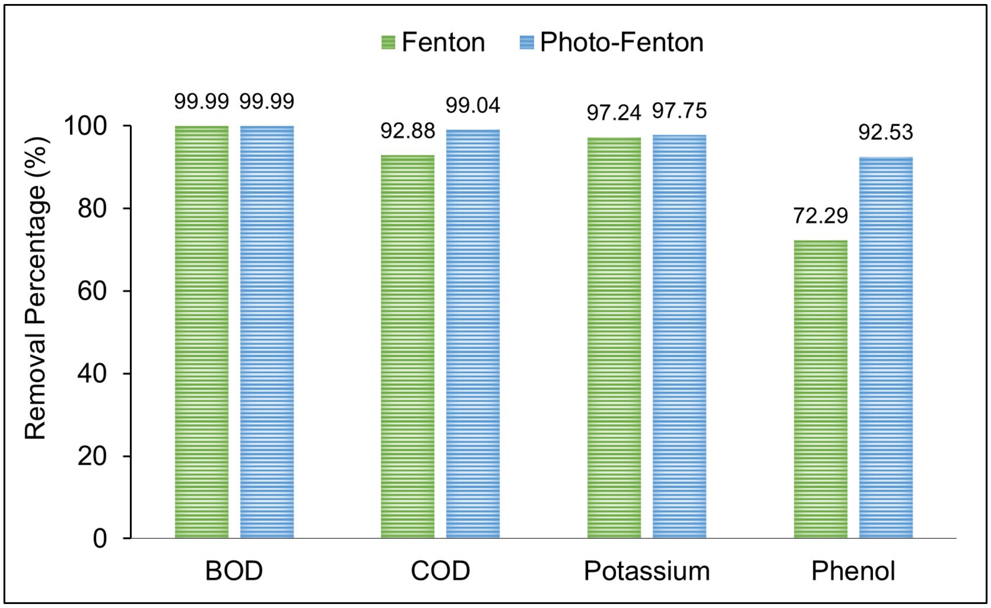


**Figure 5.** Effect of Fenton and Photo-Fenton Treatment on Pollutants Removal Percentage

**Table 2.** Percent Removal of Pollutants in Wastewater Using Fenton Treatment

| Parameter | This study | Guerreiro et al. (2016) | Tara et al. (2019) | Rao and Shrivastava (2020) | Pimentel Prates et al. (2023) |
|---|---|---|---|---|---|
| COD | 92.88% | 69.20% | 79.50% | 87.00% | 87.00% |
| BOD | 99.99% | 45.70% | 77.00% | 79.00% | n.a. |
| Potassium | 97.24% | n.a. | n.a. | n.a. | n.a. |
| Phenol | 72.29% | n.a. | n.a. | n.a. | n.a. |

*n.a.: not available

**Table 3.** Percent Removal of Pollutants in Wastewater Using Photo-Fenton Treatment

| Parameter | This Study | Buthiyappan and Raman (2019) | Villegas et al. (2016) |
|---|---|---|---|
| COD | 99.04% | 91.00% | 94.00% |
| BOD | 99.99% | n.a. | n.a. |
| Potassium | 97.75% | n.a. | n.a. |
| Phenol | 92.53% | n.a. | 66.50% |

*n.a.: not available

of 1:75 is recommended as the optimum condition based on this parameter.

### 3.3 Organic Pollutant Removal Efficiency of Fenton and Photo-Fenton Method

The removal efficiency of pollutants in recalcitrant wastewater was evaluated after Fenton and photo-Fenton treatment, using a dilution factor of 1:75 as the optimum condition. Figure 5 shows that both Fenton and photo-Fenton processes effectively reduced organic and recalcitrant compounds in terms of COD, BOD, potassium, and phenol in wastewater. The removal efficiency of the Fenton and photo-Fenton methods for treating recalcitrant wastewater reached over 72%.

When comparing the removal efficiency of wastewater treatment using Fenton and photo-Fenton methods, significant differences were observed. For COD removal, photo-Fenton treatment achieved higher efficiency (99.04%) compared to Fenton treatment (92.88%). Similarly, for phenol removal, the photo-Fenton process reached 95.53%, much higher than the 72.29% achieved by the Fenton treatment. The photo-Fenton process incorporates UV light, which enhances the degradation efficiency of organic compounds (Dogan et al., 2021; Ribeiro et al., 2021). Additionally, during the photo-Fenton process, hydrogen peroxide acts as an electron acceptor that reacts with conduction band electrons, as shown in Equations (8) and (9) (Park et al., 2006).

$$H_2O_2 + e^- \rightarrow \cdot OH + OH^- \tag{8}$$

$$H_2O_2 + hv \rightarrow 2 \cdot OH \tag{9}$$

This condition leads to the generation of more hydroxyl radicals, which are essential for the degradation of organic pollutants in wastewater. Overall, the removal percentage of each pollutant using photo-Fenton treatment exceeded 92%, demonstrating higher efficiency compared to the Fenton treatments.

### 3.4 Comparison of Organic Pollutants Percent removal

The efficiency of removing organic pollutants through the Fenton and photo-Fenton processes between the results achieved in this study with findings reported in other relevant studies has been extensively analyzed and compared as shown in Tables 2 and 3, respectively.

The variations in percent removal across different studies can be attributed to several factors, including the concentration of contaminants and the operating conditions employed. Tables 2 and 3 indicate that, compared to Fenton processes, photo-Fenton processes achieve better efficiency in degrading organic pollutants from wastewater in terms of COD, with almost complete (more than 90%) removal of COD. The percent removal of organic pollutants achieved in this study is consistent with other results reported in the literature. This suggests that the photo-Fenton process is a promising and highly efficient method for treating recalcitrant compounds. Moreover, this study also examined the degradation of other toxic pollutants, such as potassium and phenol, demonstrating that the photo-Fenton method is effective for treating specific pollutants in industrial wastewater.

## 4. CONCLUSIONS

The treatment of recalcitrant wastewater was studied by comparing the Fenton and photo-Fenton methods. It was concluded that the dilution factor of the wastewater significantly influenced the oxidation rate in both processes. The optimum conditions for recalcitrant wastewater treatment using both methods were found at a dilution factor of 1:75. Under the optimal conditions of the Fenton process, the removal efficiency of BOD, COD, potassium, and phenol reached 99.99%, 92.88%, 97.24%, and 72.29%, respectively. Meanwhile, for the photo-Fenton process, a removal percentage greater than 92% was achieved for all pollutants (BOD, COD, potassium, and phenol). Based on the results of this study, the photo-Fenton process exhibits higher removal efficiency compared to the Fenton process. Therefore, it can be concluded that the photo-Fenton process presents a promising alternative treatment method for recalcitrant wastewater. This method also offers the advantage of a relatively short reaction time, which can enhance the overall efficiency of treating recalcitrant wastewater.

## 5. ACKNOWLEDGEMENT

Authors gratefully acknowledge the financial support provided by the Hibah Riset Nasional Muhammadiyah Batch VII 2024 under Grant No. 0258.266/I.3/D/2024.

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