

COMPARATIVE STUDY OF DISTILLERY WASTEWATER TREATMENT BY DIRECT UV IRRADIATION, UV/H₂O₂ AND UV-PHOTO-FENTON PROCESSES

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Received 13 October 2024

Accepted 29 June 2025

DOI: 10.59957/jctm.v60.i6.2025.15

ABSTRACT

The disposal of distillery stillage wastewater poses significant environmental challenges due to its high organic load and complex composition. Among the commonly used treatment methods are advanced oxidation processes (AOP) such as direct UV irradiation, UV/H₂O₂ and UV-Photo-Fenton. This study presents a comparative analysis of three methods: direct UV irradiation, UV/H₂O₂ and UV-Photo-Fenton processes, for the treatment of distillery wastewater. The effectiveness of each process was assessed by evaluating the reduction of chemical oxygen demand (COD) and biological oxygen demand (BOD₅). Direct UV irradiation alone showed limited efficacy in degrading the complex organic molecules present in the wastewater. The UV/H₂O₂ process, involving the photolysis of hydrogen peroxide, demonstrated a moderate improvement in pollutant reduction. However, the most effective treatment was achieved with the UV-Photo-Fenton process, which combines hydrogen peroxide, ferrous ions, and UV light. This method exhibited the highest removal efficiencies, achieving significant reductions in COD (96.36 %) and BOD₅ (94.60 %). The enhanced performance is attributed to the synergistic generation of hydroxyl radicals through Fenton chemistry and UV irradiation, leading to more effective degradation of recalcitrant compounds. The study concludes that while each AOP has specific advantages, the UV-Photo-Fenton process stands out as the most promising treatment option for distillery stillage wastewater, offering an efficient and sustainable solution for mitigating the environmental impact of distillery operations.

Keywords: advanced oxidation processes; bioethanol wastewater; hydroxyl radicals; organic pollutant degradation; ultraviolet irradiation.

INTRODUCTION

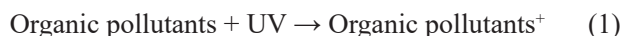
According to United Nations Environment Programme (UNEP), industries globally discharge around 300 to 400 million tons of various wastes such as heavy metals, solvents, toxic sludge compounds into water sources annually [1]. It is estimated that about 70 % of industrial wastewater in developing countries is untreated and directly discharged into water bodies, leading to significant environmental pollution. Industrial pollutants are a major source of water quality degradation [2]. This wastewater can severely impact ecosystems and

public health. Contaminants such as toxic chemicals and heavy metals (e.g., cadmium, lead, mercury, chromium), organic pollutants (e.g., PCBs, dioxins), and nutrients-rich chemicals (e.g., nitrogen, phosphorus) can cause severe ecological damage. Moreover, exposure to toxic chemicals can affect human health by contaminating natural resources and poses serious threats to ecosystems and wildlife. The treatment of recalcitrant wastewater is crucial to mitigate environmental contamination. Proper management of wastewater treatment byproducts is essential to prevent the dissemination of antibiotic resistance genes and bacteria into the environment.

Ultraviolet (UV) technology has emerged as a pivotal method for wastewater treatment, particularly in disinfection processes aimed at removing pathogens and organic contaminants [3]. The efficacy of UV disinfection is attributed to its ability to inactivate a wide range of microorganisms without the formation of harmful disinfection by-products. This method provides significant advantage over traditional chemical methods such as chlorination and ozonation [4]. Various methods for the removal of contaminants from wastewater have been explored. Among these, advanced oxidation processes (AOPs) have shown promising results in improving the overall quality of treated wastewater, making it suitable for reuse applications. For instance, the application of UV-based AOPs has been reported to significantly reduce the concentration of persistent organic pollutants and pathogens in secondary treated municipal wastewater [5, 6]. These AOPs have emerged as efficient and promising technologies for treating wastewater containing toxic and recalcitrant organic compounds [7].

Recently, UV/H₂O₂ and UV-Photo-Fenton processes have also been extensively studied. UV/H₂O₂ system utilizes UV light to generate hydroxyl radicals (•OH) from adding hydrogen peroxide during the treatment to boost degradation efficiency [8]. Meanwhile UV-Photo-Fenton involves the use of UV light to assist in the Fenton reaction, which utilizes iron as catalyst and hydrogen peroxide as oxidant to generate •OH for oxidation [9, 10]. Studies have also demonstrated the effectiveness of Fenton's reagent in degrading toxic pollutants and recalcitrant organics present in industrial wastewater [11 - 13]. The optimal pH range for the Fenton process has been identified to be between 2 and 4, emphasizing the importance of maintaining operating conditions in wastewater treatment [14]. During the treatment of distillery stillage wastewater, each process - direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton - operates through different mechanism which involves the breakdown of contaminants to degrade organic pollutants using UV light energy. The direct UV irradiation uses the exposure of photons from UV light to break chemical bonds in pollutants. This process relies on the absorption of UV photons by chemical bonds within the pollutants, leading to their dissociation into simpler molecules [15]. The absorption of UV light causes the pollutant molecules to undergo changes in their electronic configuration.

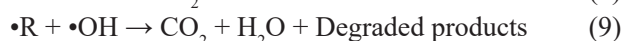
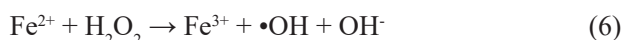
If the absorbed energy is sufficient, it would break the chemical bonds and forms smaller organic fragments. This mechanism is described by Eqs. (1, 2).



In UV/H₂O₂ process, the mechanism is based on the generation of highly •OH through the photolytic decomposition of hydrogen peroxide under UV irradiation. The •OH are strong oxidants capable of attacking and breaking down a wide range of organic pollutants into simpler and non-toxic products like CO₂ and H₂O. This process significantly enhances the degradation efficiency compared to direct UV treatment alone due to the high reactivity of •OH with organic pollutant molecules (RH) [16]. The reaction of •OH and RH break down the pollutants into smaller compounds which are easier to degrade and mineralize. Reaction mechanism of UV/H₂O₂ process is summarized in Eqs. (3 - 5).



UV-Photo-Fenton process is AOPs that combines UV light and the addition of hydrogen peroxide and iron ions (Fe²⁺) to generate •OH. The mechanism of this process is based on the synergistic effect of Fenton (H₂O₂ and Fe²⁺) and UV irradiation to promote more powerful treatment process [17]. Initially, the classical Fenton reaction occurs to produce •OH and Fe³⁺. Under UV light, the Fe³⁺ ions are reduced back to Fe²⁺ and enhanced the production of •OH. This cyclical regeneration of Fe²⁺ and continuous production of •OH make the UV-Photo-Fenton process achieve more powerful oxidant for degrading more complex organic pollutants in wastewater. This mechanism is summarized by Eqs. (6 - 9).



This study aims to evaluate and compare the

effectiveness of three methods - direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton - in the treatment of distillery stillage wastewater. The most efficient method between those three processes for reducing key pollutants, such as COD and BOD, to provide an environmentally sustainable solution for managing industrial wastewater is also investigated. By providing a comprehensive comparison, this research contributes valuable insights into the selection of appropriate methods for effective wastewater treatment, aiding to reduction of the environmental footprint of distillery operations.

EXPERIMENTAL

Materials

The distillery stillage wastewater used was obtained from a local bioethanol industry. Samples used in this study were collected from a point prior to the discharge of water into the primary wastewater treatment system and were stored in a refrigerator at 4°C. The wastewater samples were characterized first to determine the initial pollutant concentration as shown in Table 1. Reagents used in this study were ferrous sulfate heptahydrate (FeSO₄×7H₂O, Merck), hydrogen peroxide solution (H₂O₂ 30 % wt., PT PIP Indonesia), sodium hydroxide (NaOH, Merck), and distilled water.

Experimental procedure of direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton

All experiments were conducted in a laboratory-scale batch photochemical reactor using 500 mL of wastewater in a 1 L borosilicate glass beaker placed on a magnetic stirrer set at 200 rpm to ensure homogeneous mixing (Fig. 1). Ultraviolet radiation was supplied by a UV lamp (Hunta Ltd., λ = 395 nm) with an intensity of approximately 10 W positioned vertically 10 cm above the reactor.

Three treatment processes were evaluated: direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton. For the direct UV process, the wastewater was exposed to UV light alone for 60 min at a stirring speed of 200 rpm. In the UV/H₂O₂ process, hydrogen peroxide was added to a concentration of 100 mg L⁻¹. For the UV-Photo-Fenton process, FeSO₄·7H₂O was added to supply 50 mg L⁻¹ of Fe²⁺, followed by 100 mg L⁻¹ of H₂O₂. After each experiment, the pH was neutralized using NaOH

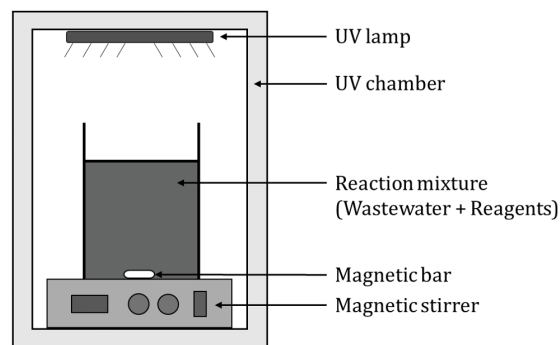


Fig. 1. Schematic diagram of photochemical reactor.

Table 1. Characteristics of raw distillery stillage wastewater used in this study.

Parameter	Value
pH	4.0
COD, mg L ⁻¹	66440
BOD ₅ , mg L ⁻¹	39800
Phenol, mg L ⁻¹	212.14
ORP, mV	330
Color	Dark brown

to terminate the reaction and precipitate excess iron. Samples were withdrawn at regular intervals (0, 15, 30, 45, and 160 mins) and measured for pH and oxidation reduction potential (ORP) value, then filtered using Whatman No. 42 filter paper, and analysed for COD and BOD₅ according to Standard Methods (APHA 5220 D and APHA 5120 B). The performance of each treatment process was evaluated based on the removal efficiency of COD and BOD₅. The percentage removal of COD and BOD₅ was calculated using Eq. (10):

$$\text{Removal efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (10)$$

where C₀ is the initial COD or BOD₅ concentration (mg L⁻¹), and C_t is the concentration at a given reaction time t, min. In addition, the degradation kinetics of COD were analysed by assuming a pseudo-first-order reaction model, which is commonly applied in advanced oxidation processes under conditions where oxidant concentration is in excess [18, 19]. The apparent first-

order rate constant (k_{app}) was determined using the linearized form of the first-order kinetic equation (Eq. (11)):

$$\ln \left(\frac{C_0}{C_t} \right) = k_{app} \cdot t \quad (11)$$

A plot of $\ln(C_0/C_t)$ versus time was constructed for each process, and the rate constant (k_{app}) was obtained from the slope of the linear regression line. The coefficient of determination (R^2) was also calculated to assess the goodness of fit of the kinetic model to compare the degradation rates and treatment efficiency of the direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes.

RESULTS AND DISCUSSION

Effects of different processes on pH profile

pH plays a critical role during the application of AOPs for distillery stillage wastewater treatment. Each process depends on pH for optimal generation of oxidants or reactive species. Thus, the pH profile is essential to be investigated for achieving optimal performance during the treatment. Table 2 and Fig. 2 show the profile of pH during treatment of distillery stillage wastewater using three processes: direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton over a 60 min reaction time. After the addition of oxidants to the raw wastewater (initial pH = 4.0), different changes in pH were observed on each process. In the direct UV irradiation process, no chemical additives were introduced, so the pH remained stable at approximately 4.0, reflecting the natural acidity of the untreated wastewater. Meanwhile in the UV/H₂O₂ process, the pH increased to 4.62 immediately after the addition of hydrogen peroxide. This increase can be attributed to the basic nature of hydrogen peroxide in solution, which can slightly elevate pH, especially in weakly buffered systems like distillery wastewater. In

contrast, the UV-Photo-Fenton process, which involves the addition of both ferric ion (Fe²⁺ from FeSO₄·7H₂O) and hydrogen peroxide, resulted in a pH decrease to 3.0. This significant acidification is due to the Fenton reaction, where Fe²⁺ catalyses the decomposition of H₂O₂ to generate •OH and releasing protons in the process. The release of Fe³⁺ hydrolysis products and acidic iron complexes further contributes to the drop in pH [20].

During 60 min treatment, direct UV irradiation and UV/H₂O₂ process maintains a relatively stable pH throughout the reaction. The pH stability indicated that both processes does not alter the pH, as there is minimal production of acidic and/or basic intermediates during the degradation of pollutants. This is consistent with findings that show limited pH change when using both processes [21, 22]. UV light alone does not produce reactive species that would alter pH levels, while the breakdown of hydrogen peroxide in UV/H₂O₂ process does not significantly impact the overall acidity of the solution. Meanwhile in UV-Photo-Fenton system, there is continuous decrease in pH by the end of 60 min reaction, which relates to the characteristics of the

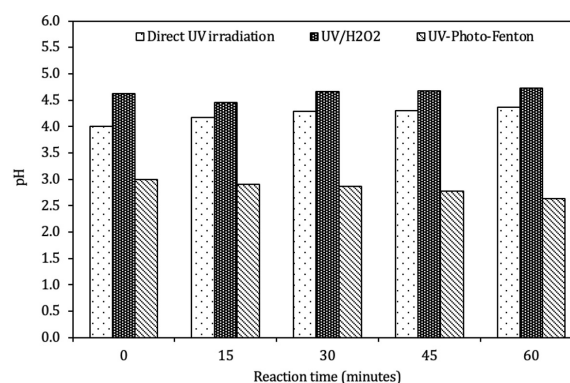


Fig. 2. Effect of direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes on pH profile during distillery stillage wastewater treatment.

Table 2. pH profile of each process during 60 min reaction.

System	Reaction time, min				
	0	15	30	45	60
Direct UV irradiation	4.00	4.17	4.29	4.30	4.37
UV/H ₂ O ₂	4.62	4.45	4.66	4.68	4.73
UV-Photo-Fenton	3.00	2.90	2.86	2.77	2.63

photo-Fenton reaction, where the interaction between H_2O_2 , Fe^{2+} , and UV light produces $\bullet OH$ and acidic intermediates from the degradation of high molecular weight organic compounds and leads to the formation of short-chain carboxylic acids and other intermediates, leading to a drop in pH levels. This result is in accordance with previous studies where acidification is noted as a byproduct of the Fe-catalysed reactions [23]. This decrease also suggests that the system maintains strong oxidative conditions throughout the reaction period, thus enhances the oxidation efficiency. Overall, the declining pH trend in the UV-Photo-Fenton system shows that it was the most reactive and effective process for organic pollutant breakdown.

Effects of different processes on ORP profile

Oxidation reduction potential (ORP) is often used as an indicator for monitoring the degradation process of wastewater treatment. ORP correlates with the overall oxidative capacity in the system. This parameter measures the tendency of a chemical species to acquire electrons and then be reduced, to provide insight about the effectiveness of the treatment process. Higher ORP values generally correspond with improved degradation of pollutants. Table 3 and Fig. 3 shows the profile of ORP during 60 min of distillery stillage wastewater treatment by using direct UV irradiation, UV/ H_2O_2 , and UV-Photo-Fenton. The initial ORP of raw wastewater was measured at 330 mV (Table 1), which changed differently after the oxidants was introduced depending on the treatment process applied. In the direct UV irradiation process and UV/ H_2O_2 process, the ORP values dropped to 149 mV and 307 mV, respectively. This decline is attributed to the rapid consumption of oxidizing agents (H_2O_2) by the reducing components of distillery stillage wastewater, such as sugars, phenolics, and volatile acids [24]. Results clearly show that the trend ORP values for each treatment process is following this order: direct UV irradiation

$< UV/H_2O_2 < UV$ -Photo-Fenton. The ORP values of direct UV irradiation process remains relatively low and stable around 149-164 mV. This suggests that the oxidative power of the process is limited since there are no additional oxidants are introduced, and the generation of reactive species is minimal. Meanwhile for UV/ H_2O_2 process, the ORP values are increasing and stabilizing in the range of 270-307 mV. The increase of ORP values is due to the presence of hydrogen peroxide, which is then decomposed by UV light into $\bullet OH$, creating a stronger oxidizing environment in the solution [25]. The highest ORP value is achieved by UV-Photo-Fenton system (around 531-553 mV). This condition is obtained due to the generation of large number of $\bullet OH$ and followed by other oxidizing species. The presence of Fe^{2+} in UV-Photo-Fenton system catalyses the decomposition of H_2O_2 into $\bullet OH$ radicals, while UV light promotes the photoreduction of Fe^{3+} back to Fe^{2+} , effectively sustaining the radical generation cycle. The high ORP values indicate a strong oxidative environment which corresponds to high efficiency in degrading complex

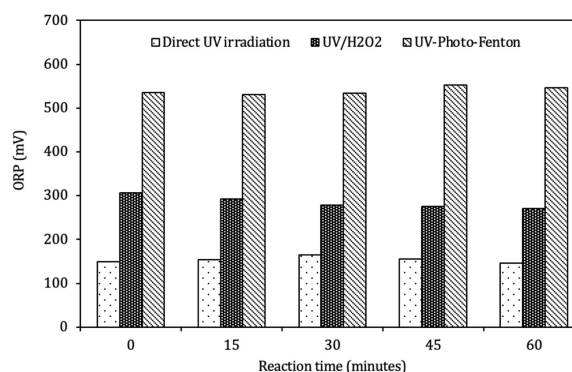


Fig. 3. Effect of direct UV irradiation, UV/ H_2O_2 , and UV-Photo-Fenton processes on ORP profile during distillery stillage wastewater treatment.

Table 3. ORP profile of each process during 60 min reaction.

System	Reaction time, min				
	0	15	30	45	60
Direct UV irradiation	149	154	164	155	146
UV/ H_2O_2	307	292	279	275	270
UV-Photo-Fenton	535	531	534	553	546

organic pollutants in wastewater [26]. The constant ORP values over time reflects a sustained production of reactive oxidizing species in the system. This result is consistent with the reaction mechanisms of each process, where the UV-Photo-Fenton provides the most robust oxidative condition, facilitating the rapid degradation of contaminants in the wastewater [27].

COD and BOD₅ degradation performance

To investigate the efficiency of direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes in treating distillery stillage wastewater, the COD and BOD₅ removal were measured and compared as presented in Table 4 and Fig. 4. The removal efficiencies of COD and BOD₅ varied significantly among the three processes. The results demonstrated that COD could be effectively degraded within approximately 60 min of reaction time. Direct UV achieved limited degradation, with only 59.95 % COD and 58.67 % BOD₅ removal. While direct UV irradiation is moderately effective for treatment, the incorporation of AOPs such as UV/H₂O₂ and UV-Photo-Fenton significantly enhances the degradation of organic pollutants in stillage wastewater. The UV/H₂O₂ process improved degradation performance, achieving 85.88 % COD and 84.30 % BOD₅ removal due to the formation of hydroxyl radicals via UV-induced decomposition of hydrogen peroxide. The UV-Photo-Fenton process yielded the highest degradation efficiency, with COD and BOD₅ removals of 96.36 % and 94.60 %, respectively. Together, these processes show an increasing efficiency in the order of direct UV irradiation < UV/H₂O₂ < UV-Photo-Fenton.

Raw stillage wastewater contains high concentration of COD (66400 mg L⁻¹). Thus, the abundant presence of this organic matter in the wastewater can hinder the direct UV disinfection process, decreasing the treatment efficiency [28]. The combination of UV irradiation with

hydrogen peroxide (UV/H₂O₂) utilizes the principle of generating •OH through the photolysis of hydrogen peroxide under UV light, which significantly increases the oxidation potential. Previous studies reported that the addition of hydrogen peroxide enhances the removal rates of various organic compounds and achieves higher degradation efficiencies for recalcitrant contaminants, which are typically challenging to degrade using conventional chemical wastewater treatment methods [29, 30]. Meanwhile UV-Photo-Fenton method leverages the photochemical reactions like UV/H₂O₂ process by involving ferrous ions to generate •OH. The presence of Fe(II) facilitates the Fenton reaction, which is effective in breaking down complex organic molecules into simpler, less harmful compounds. During this process, a continuous generation of •OH is also enhanced by UV irradiation, which also helps in regenerating the iron catalyst for sustained degradation of pollutants [31]. From Table 2 and Fig. 4, UV-Photo-Fenton process provides the most effective method for distillery wastewater treatment with the highest removal efficiency of COD (96.36 %) and BOD₅ (94.60 %) compared to

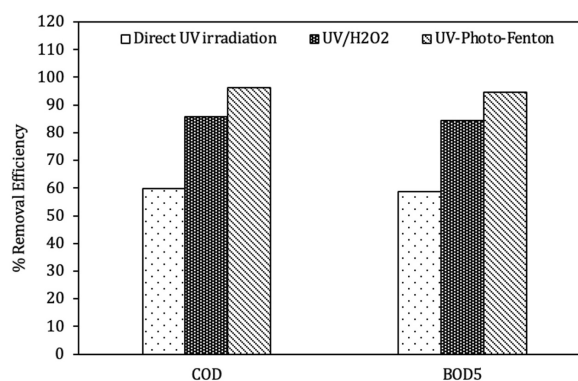


Fig. 4. Comparison of direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes on COD and BOD₅ removal efficiency.

Table 4. COD and BOD₅ removal of each process.

System	COD			BOD ₅		
	Raw, mg L ⁻¹	Final, mg L ⁻¹	Removal, %	Raw, mg L ⁻¹	Final, mg L ⁻¹	Removal, %
Direct UV irradiation	66400	26610	59.95	39800	16450	58.67
UV/H ₂ O ₂	66400	9380	85.88	39800	6250	84.30
UV-Photo-Fenton	66400	2420	96.36	39800	2150	94.60

direct UV irradiation and UV/H₂O₂. This method has demonstrated potential in enhancing the degradation of organic pollutants in wastewater.

Effects of different processes on COD profile and kinetic analysis

The COD degradation performance of three processes was evaluated as shown in Fig. 5. The COD removal significantly differ among the treatment methods. Direct UV irradiation resulted in a relatively slow and gradual COD reduction, with only 59.95 % removal within 60 min. The introduction of hydrogen peroxide in the UV/H₂O₂ process improved COD degradation, achieving approximately 85 % removal, due to the generation of •OH through photolysis of H₂O₂. While the most effective process was UV-Photo-Fenton, which achieved high COD removal (96.36 %) within the same period. This performance is attributed to the synergistic effect of Fe²⁺ and H₂O₂ under UV irradiation, which sustains production of •OH via both Fenton and photo-assisted redox cycles [32].

To quantitatively evaluate the degradation kinetics, the data were fitted to a pseudo-first order model. In this study, the apparent reaction rate constants were estimated by applying a first-order kinetic model based on initial and final COD concentrations over a fixed treatment time. This approach is commonly adopted in the kinetic analysis of AOPs, particularly when one reactant, typically the oxidizing agent or catalyst, is present in excess amount, and the degradation rate is primarily limited by the pollutant concentration. Under

such conditions, the rate law simplifies to a pseudo-first-order form, allowing linearization and estimation of an apparent rate constant (k_{app}) using Eq. (10), with result as presented in Fig. 6. The linear plots of $\ln(C_0/C_t)$ versus time for all processes exhibited good correlation, confirming the first-order kinetic assumption. The value of (k_{app}) were shown in Table 5, which following trend as followed: UV-Photo-Fenton > UV/H₂O₂ > direct UV irradiation. These values clearly indicate that the UV-Photo-Fenton process proceeds at a significantly higher rate than the other two methods. The excellent linear fit for UV-Photo-Fenton ($R^2 > 0.99$) highlights the stability and efficiency of the catalytic cycle, where Fe²⁺ continuously reacts with H₂O₂ to produce •OH, and UV light facilitates the regeneration of Fe²⁺ from Fe³⁺. This cyclic mechanism sustains radical availability, leading to a faster and more complete organic pollutants degradation. The kinetic data, supported by the observed COD trends, confirm that UV-Photo-Fenton offers the most rapid and efficient degradation pathway for high-strength wastewater like distillery stillage from bioethanol industry.

Table 5. Apparent first-order rate constants and correlation coefficients for COD degradation using different processes.

System	k_{app}, min^{-1}	R^2
Direct UV irradiation	0.0153	0.9592
UV/H ₂ O ₂	0.0320	0.9573
UV-Photo-Fenton	0.0558	0.9983

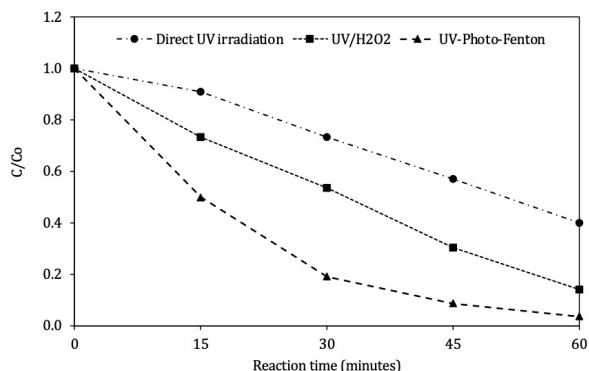


Fig. 5. COD profile of direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes during 60 min reaction.

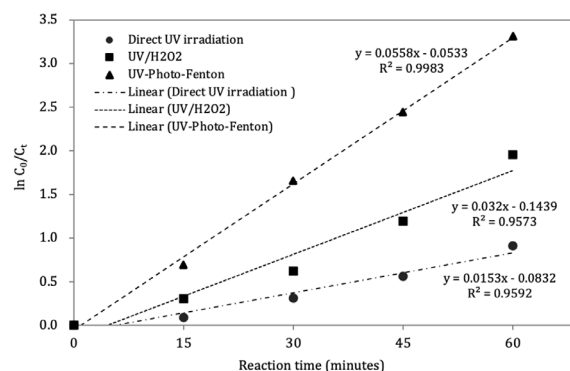


Fig. 6. First order kinetic plots for COD degradation of distillery wastewater treatment using direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton processes.

CONCLUSIONS

The comparative study of distillery stillage wastewater treatment using various methods: direct UV irradiation, UV/H₂O₂, and UV-Photo-Fenton has been conducted. It is demonstrated that the UV-Photo-Fenton process is the most effective method among the three. While direct UV irradiation and UV/H₂O₂ processes provided some level of COD and BOD₅ reduction, their overall efficiency was limited, particularly in dealing with the complex organic compounds present in distillery stillage. The UV-Photo-Fenton process achieved the highest reductions in COD (96.36 %) and BOD₅ (94.60 %) under natural acidic conditions (initial pH 4.0) without pH adjustment, indicating a more thorough breakdown of the organic matter in the wastewater. Furthermore, the kinetic evaluation based on first-order assumptions further confirmed that the UV-Photo-Fenton process had the highest apparent rate constant with the highest R² value, showing faster and more effective degradation of recalcitrant organics. These results suggest that UV-Photo-Fenton process holds significant potential to provide a robust and scalable solution for the sustainable management of wastewater in distillery industries.

Acknowledgements

This research was funded by the Directorate of Higher Education, Research, and Technology, Ministry of Education, Culture, Research, and Technology (DRTPM Kemendikbud Ristek) Indonesia as well as Institute of Research and Community Service (LPPM) Universitas Ahmad Dahlan under the grant number 107/E5/PG.02.00. PL/2024; 0609.12/LL5-INT/AL.04/2024; and 031/PTM/LPPM-UAD/VI/2024 fiscal year 2024. Authors would like to thank Ikhlasil Amal for his assistance in the laboratory work associated with this study.

Authors' contributions

K.A.W.: Experiments, Investigation; D.C.H.: Conceptualization, Methodology, Writing - original draft; M.S.: Supervision, Validation; M.K.B.: Conceptualization, Validation, Writing – review and editing.

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