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Research Article

Enhance Removal Pollutant from *Batik* Industrial Wastewater via Photo- Fenton Process: Efficiency and Kinetic Study

Oki Setiawan[1]*, Siti Adriani[1], Haposan Vincentius Manalu[1], Aninditiya Amalia[2], Ifti Luthviana Dewi[1], Insan Kamil[1], Darti Purnama Sari[1], Popi Sasniati[1]

[1]*Mechanical Engineering, Politeknik Negeri Batam, Batam, Indonesia.*
[2]*Chemical Engineering, Universitas Muhammadiyah Gresik, Gresik, Indonesia.*

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Abstract

The present research evaluates the efficiency of the Photo-Fenton-like process under UV irradiation by varying ratio of hydrogen peroxide (H_2O_2) to Chemical Oxygen Demand (COD) and H_2O_2 to ferrous ion (Fe^{2+}) using wastewater from a *batik* manufacturer in Gresik. Initial experiments demonstrated that varying the ratio of H_2O_2/COD contributes to Chemical Oxygen Demand (COD) removal, while maintaining a constant H_2O_2/Fe^{2+} ratio. The result reveals a significant effect on Chemical Oxygen Demand (COD) removal achieving 64.58% efficiency which indicates that highly reactive OH^* radicals successfully generated in photo-Fenton-like process. Further optimization by increasing the ratio H_2O_2/COD = 15 (g/g) and H_2O_2/Fe^{2+} = 20 (g/g) resulting on maximum percentage of Chemical Oxygen Demand (COD) removal of 92.47% proving the massive production of OH^* radicals. The kinetics reaction model which describes the Chemical Oxygen Demand (COD) degradation rate showed the BMG kinetic model, with parameters of 1/m = 0.9352, 1/b = 0.7159, and a coefficient of determination (R^2) of 0.9986, indicating an excellent fit and high predictive accuracy of the kinetic model for this process.

Keywords: Wastewater; Photo-Fenton; Kinetic Study; Advanced Oxidation Process; Chemical Oxygen Demand

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1. Introduction

One of Indonesia's most renowned traditional crafts, recognized both nationally and internationally, is the textile industry particularly its signature product, *batik*. *Batik* is a treasured cultural heritage of Indonesia, highly respected and upheld by communities across the archipelago and around the world. The batik industry contributes significantly to national economic growth, especially through the involvement of numerous micro, small, and medium enterprises (MSMEs) in its production processes [1]. The batik industry is spread across 38 regions in Java, with key production centers in Solo, Pekalongan, Tegal, Lasem, and Banyumas. Most *batik* entrepreneurs operate on a small scale from home and typically lack proper wastewater treatment facilities [2].

The recognition of batik as a cultural heritage by United Nations Educational, Scientific, and Cultural Organization (UNESCO) has spurred the growth of batik production in various regions, each developing unique and distinctive motifs. While this expansion boosts the economy, it also has environmental consequences particularly in the form of liquid waste generated during production. Wastewater from batik-making mainly arises during soaking, boiling, and rinsing stages. In many cases, this wastewater is discharged directly into rivers or nearby water bodies without adequate treatment, causing serious environmental and health issues. Batik industry wastewater is typically high in volume and

* Corresponding Author.
Email: oki@polibatam.ac.id (O. Setiawan)

contains significant pollutants such as dyes, starch, aluminium sulphate, and wax. It often has high pH, elevated Chemical Oxygen Demand (COD), and Total Suspended Solids (TSS), along with intense coloration. The synthetic and complex molecular structure of the dyes makes them stable and difficult to degrade naturally, posing a severe threat to aquatic ecosystems [2]. Proper treatment of *batik* wastewater is essential, and producers are obligated to manage it responsibly using appropriate treatment technologies to ensure safe disposal. In general, for the sake of human health and environmental sustainability, wastewater discharge must be regulated in accordance with standards set by relevant authorities [3].

One promising treatment method is the Advanced Oxidation Processes (AOPs), which are used to treat batik industry wastewater. AOPs are classified into two main categories: photochemical and non-photochemical methods. Photochemical AOPs utilize ultraviolet (UV) light in techniques such as hydrogen peroxide (H_2O_2)/UV and photocatalytic oxidation. Non-photochemical methods include ozonation with elevated pH, combinations like O3/H_2O_2, ozone/catalyst, and the Fenton system. Both AOP approaches produce highly reactive radicals such as hydroxyl radicals ($OH^\bullet$), superoxide radicals ($O_2^\bullet$), and sulfate radicals ($SO_4^\bullet$) that break down pollutants in wastewater into simpler compounds like water (H_2O) and carbon dioxide (CO_2) [4].

A hybrid process combining ultrasonic (US), UV light, ferrous ions (Fe^{2+}), and H_2O_2 can significantly boost hydroxyl radical generation, regenerate consumed Fe^{2+}, and effectively accelerate pollutant degradation in wastewater. The chemical reactions involved are shown in equations (1) through (8) [5]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3}+ + HO^{\cdot} + OH^{-} \quad (1)$$
$$HO^{\cdot} + HO_2 \rightarrow H_2O + O_2 \quad (2)$$
$$Fe(OH)_2+ + hv \rightarrow Fe^{2+} + HO^{\bullet} \quad (3)$$
$$Fe^{3+} + H_2O_2 \leftrightarrow Fe - (OOH)^{2+} + H^{+} \quad (4)$$
$$Fe - (OOH)^{2+} + H^{+} \rightarrow Fe^{2+} + HO_2 \quad (5)$$
$$Fe^{2+} + HO \rightarrow Fe^{3+} + OH^{-} \quad (6)$$
$$Fe^{3+} + H \rightarrow Fe^{2+} + H^{+} \quad (7)$$
$$H_2O_2 + hv \rightarrow 2HO^{\cdot} \quad (8)$$

A study evaluating the Fenton process at pH of 3 for treating textile wastewater at four concentration levels found that a H_2O_2 to Fe ratio of 1:25 and a reaction time of 30 minutes resulted in excellent outcomes. The treatment achieved a 92% reduction in color, 89% reduction in COD, and 94% reduction in turbidity, showing that acidic pH, optimal reaction time, and proper reagent ratios significantly improve treatment efficiency [6].

Another study focused on removing pollutants from hand-drawn batik wastewater using a combination of biological and advanced oxidation processes. The research evaluated the effectiveness of integrating bio-equalization and electrocatalytic technologies. An 18 m^3 bio-equalization tank was inoculated with 10% (v/v) activated sludge and equipped with four evenly distributed inlet pipes. It was connected to a 1.2 m^3 electrocatalytic reactor fitted with a Ti/$RuIrO_2$ anode and stainless-steel cathode. The treated wastewater met local discharge standards with a 48-hour hydraulic retention time (HRT) in the bio-equalization tank and 90-minute electrolysis duration [2].

Ultraviolet (UV) energy, sourced from UV lamps or sunlight, can enhance treatment efficiency when combined with oxidizers to accelerate hydroxyl radical formation. UV light aids pollutant degradation via two primary mechanisms: direct photolysis and hydroxyl radical generation from H_2O_2 decomposition. In direct photolysis, UV light breaks down pollutant molecules directly. In the second mechanism, UV reacts with H_2O_2 to produce reactive hydroxyl radicals that degrade pollutants [7].

Previous research has aimed to reduce COD levels in batik industry wastewater. The novelty of this current study lies in its use of photo-Fenton treatment with UV B light. The goal is to reduce the initial COD level of 11,404.8 mg O_2/L to meet wastewater discharge standards. This method was chosen for its high efficiency in breaking down complex pollutants into simpler compounds like water and carbon dioxide. Its advantages include effective removal of color and COD, fast reaction times, and cost-effectiveness for small-scale industries. Moreover, this technology offers an environmentally friendly solution that supports the sustainability of Indonesia's *batik* industry.

This study introduces a novel approach in the treatment of batik industrial wastewater by employing the Photo-Fenton process under UV B irradiation. Unlike conventional methods commonly applied in batik wastewater treatment such as aerobic anaerobic biological processes, classical Fenton oxidation, or Photo-Fenton driven by UV C light this research specifically utilizes UV B as the irradiation source. This innovation not only demonstrates high pollutant degradation efficiency, but also provides a more cost effective and potentially scalable alternative compared to UV C systems. Furthermore, the study contributes scientific significance by investigating the kinetics of COD reduction, offering insights into the degradation mechanism and reaction rates. These findings may serve as a foundation for optimizing process design and advancing sustainable wastewater treatment technologies for the *batik* industry.

2. Materials and Method

2.1 Materials

This study utilized wastewater samples obtained from a batik industry as the primary raw material. The chemicals used included sulfuric acid (H_2SO_4) with a purity of 95–97% (Merck), distilled water (aquadest), ferrous sulfate ($FeSO_4$) (Merck), hydrogen peroxide (H_2O_2) 30% (Sundopex), Ferrous Ammonium Sulfate (FAS) (Pudak), potassium dichromate ($K_2Cr_2O_7$) (Merck), ferroin indicator (Merck), mercury(II) sulfate ($HgSO_4$) (Merck), silver sulfate ($AgSO_4$) (Merck), and nitric acid (HNO_3) (Merck).

2.2 Methods

The experimental procedure began with the initial characterization of the wastewater, including measurement of Chemical Oxygen Demand (COD) and pH, to determine the baseline parameters of the batik industrial effluent used in this study. A 1000 mL sample of the characterized wastewater was transferred into a beaker. The sample was then adjusted to the desired pH and mixed with predetermined concentrations of Hydrogen Peroxide (H_2O_2) 30% and ferrous ions (Fe^{2+}). The prepared sample was placed in a reaction box and irradiated using a UV lamp, positioned horizontally above the reactor. The UV lamp served as the UV light source for the Photo Fenton process ($UV/Fe^{2+}/H_2O_2$). The initial COD analysis was performed according to the Indonesian National Standard (SNI 06-6968.73.2009). The COD test aimed to evaluate the degradation efficiency of the wastewater treatment process. The analysis was conducted using the closed reflux method, where potassium dichromate ($K_2Cr_2O_7$) acted as the oxidizing agent, sulfuric acid (H_2SO_4) with silver sulfate (Ag_2SO_4) served as a catalyst, and mercury (II) sulfate ($HgSO_4$) was added to suppress chloride interference. The sample was heated in a heating block at 150 °C for 2 hours. During this step, organic compounds in the sample were oxidized by potassium dichromate. After the reflux process, the excess dichromate was titrated using a standard solution of ferro ammonium sulfate (FAS). The amount of dichromate consumed correlates directly with the oxygen demand, and thus the COD concentration is calculated and expressed in mg/L O_2.

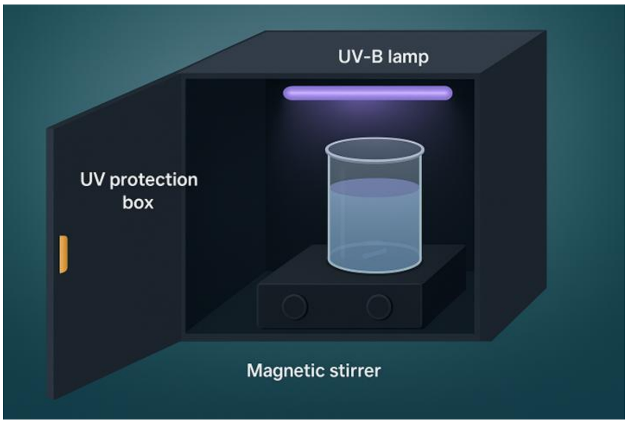


Figure 1. Apparatus setup for *batik* wastewater treatment using the photo-fenton method.

2.3 Analysis

The Chemical Oxygen Demand (COD) concentration was calculated using the following equation:

$$COD(mg/L) = \frac{(A-B)(N)(8000)}{mL.sample} \quad (9)$$

Where, A is volume of solution required to titrate the blank (mL), B is volume of Ferrous Ammonium Sulfate (FAS) solution required to titrate the sample (mL), and N is concentration of the Ferrous Ammonium Sulfate (FAS) solution (N). To ensure the quality of the analysis results, all samples were stored in a refrigerator, and testing was carried out within a maximum of one week from the time of sample collection.

The percentage of COD removal from the wastewater was determined using the following equation:

$$\%\ COD\ Romoval = \left(\frac{COD_0 - COD_t}{COD_0}\right) \quad (10)$$

Where, COD_0 is COD concentration at the initial time (t = 0), and COD_t is COD concentration at a given time t.

3. Results and Discussion

Determining the pH value is crucial in the research of a photo Fenton process to remove pollutants in the degradation process of the Fenton reaction. Producing OH* radicals depend von pH value as proven by several studies [5] and [8]. Acidic conditions in the Fenton reaction are necessary to generate OH* radicals. In this experiment, the solution condition is at a pH value of 3, as it obtains the optimal performance because the condition of H_2O_2 is stable. At higher pH values > 4, the formation of radical hydroxyl (OH*) is restrained [9]. The ratio of H_2O_2/COD (g/g), was varied which is 6, 8, and 10 and constant ratio of H_2O_2/Fe^{2+} (g/g) at 7 and in the presence of UV lamp in 60 minutes.

3.1 Effect of UV/Fe^{2+}/H_2O_2 Ratio

This research studied the effect of enhancing the ratio of H_2O_2/COD by evaluating the COD reduction in photo-Fenton-like process with a constant ratio of H_2O_2/Fe^{2+} at 7 (g/g). Research showed that OH* radical cannot be spontaneously generated only using H_2O_2 [10]. Thus, Fenton-like reaction is required to produce hydroxyl radicals. Figure 2 demonstrates the percentage of COD reduction of contaminants with a varied ratio of H_2O_2/COD. It can be seen that as the ratio of H_2O_2/COD increases, the percentage of COD removal is also increasing. This result indicates that the pollutant concentration has dropped. It proves that the Fenton reaction successfully generates OH* radicals.

This research unveils that higher dose H_2O_2 leads to faster removal of water pollution because OH* radicals formed faster at the high dose H_2O_2 [9], as illustrated in the graph. This result proves that H_2O_2 has significant role on COD removal as it can produce OH* radicals to remove the pollutants. At the ratio 10, the reduction is 52.45% in 10 minutes, whereas at the smallest ratio, the removal is only at 9.36%. Pamula *et al.* [11] also found that increasing the dose H_2O_2 shorten the time required to remove wastewater. According to the result, the percentage of COD removal increases significantly from 27.90% to 50.93 % in 60 minutes when the H_2O_2/COD ratios 6 and 8, because of more formation of hydroxyl radicals as the concentration of H_2O_2 rises. The most significant degradation occurred at ratio 10, reaching 64.58% [12]. The result was achieved using Fenton-like process in acidic condition and UV radiation. Asaithambi *et al.* [7] reported that COD removal reached optimal efficiency of 84.40%. it was reported that combining UV and Fe^{2+}/H_2O_2 process generated more hydroxyl radicals to degrade the contaminants compared to Fe^{2+}/H_2O_2 alone, which achieved only 40.50% removal. This result proves that the presence of UV irradiation is the most effective methods on removing color pollutants as it utilizes the combination of H_2O_2 and Fe^{2+} in Fenton-like reaction.

The COD removal is rising up evenly, without much change at the ratio 10 in an hour because of the low concentration of Fe^{2+} compared to H_2O_2, resulting the formation of H_2O and O_2 [13]. Thus, further study need to be done to increase the COD removal percentage by enhancing concentrate of Fe^{2+}. Figure 3 depicts The percentage of COD removal with various ratio of H_2O_2/COD and H_2O_2/Fe^{2+}, such as H_2O_2/COD = 10 (g/g) and H_2O_2/Fe^{2+} = 7 (g/g), H_2O_2/COD = 11 (g/g) and H_2O_2/Fe^{2+} = 12 (g/g), H_2O_2/COD = 15 (g/g) and H_2O_2/Fe^{2+} = 20 (g/g) at pH value = 3. Figure 2 clearly shown that by increasing the ratio of H_2O_2/COD and H_2O_2/Fe^{2+} gives noticeable impact on reducing the contaminants as the percentage COD removal rapidly risen from 64.57% to 90.60% at H_2O_2/COD = 10 (g/g) and H_2O_2/Fe^{2+} = 7 (g/g), H_2O_2/COD = 11 (g/g) and H_2O_2/Fe^{2+} = 12 (g/g), respectively. Increasing the ratio of H_2O_2/Fe^{2+} verify that Fe^{2+} can improve on generating OH* radicals with maximum efficiency on COD removal. The ratio H_2O_2/COD = 15 (g/g) and H_2O_2/Fe^{2+} = 20 (g/g) obtained as the highest COD reduction of 92.47%, although further increasing the ratio less than 2%. This result indicates that the ratio reaches its limit.

3.2 Kinetic Studies

The kinetics of pollutant degradation in *batik* industry wastewater can be quantitatively

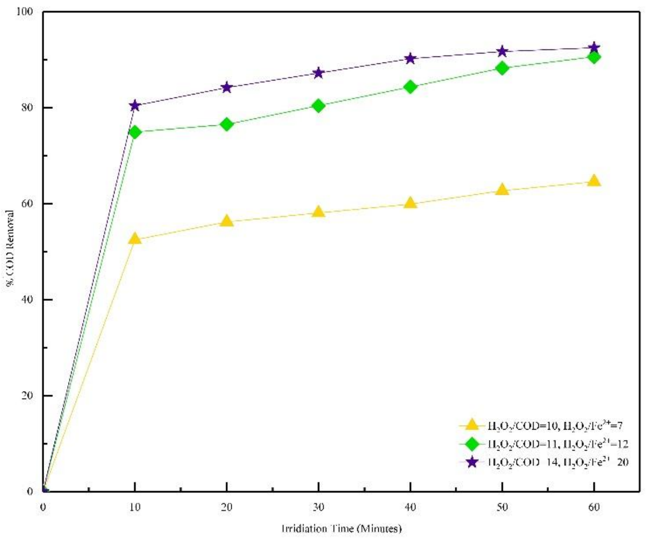


Figure 2. The percentage of COD removal with ratio H_2O_2/Fe^{2+} = 7 (g/g) and pH of 3.

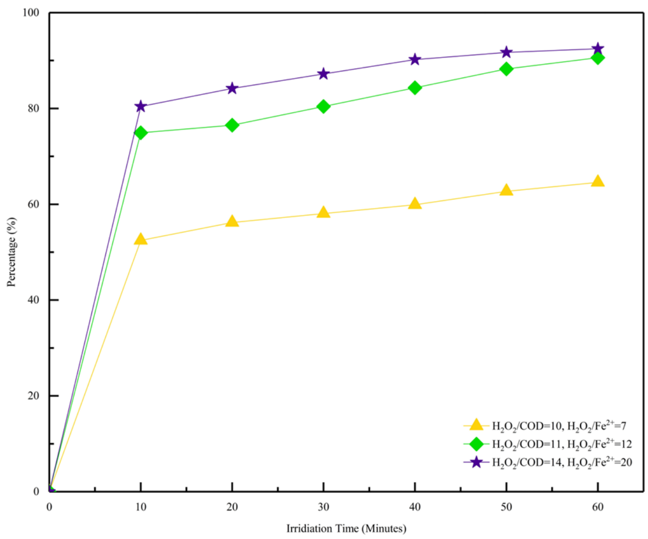


Figure 3. The percentage of COD removal at pH of 3 under different H_2O_2/COD and H_2O_2/Fe^{2+} ratios and Y axis % COD Removal

described using reaction kinetics models. In this research, three kinetic models were employed: Pseudo 1st order, Pseudo 2nd order, and the Behnajady Modirshahla Ghanbery (BMG) model. Pseudo 1st order model is assumed when the degradation rate is primarily dependent on the initial pollutant concentration, while the oxidant concentration (H_2O_2/Fe^{2+}) is in excess or considered constant. This assumption has been successfully applied in UV/H_2O_2 and fenton process for industrial wastewater and dye degradation [12]. Pseudo 2nd order model is used when both pollutant concentration and oxidant concentration significantly influence the degradation rate. In photo-feton systems, increasing the H_2O_2/COD and H_2O_2/Fe^{2+} ratio enhances the generation of OH radicals, leading to higher COD removal efficiencies.

Linear regression analysis was conducted for each model, and the results are presented in Table 2. The parameters k_1 and k_2 represent the kinetic rate constants for the 1st and 2nd order models, respectively, while the BMG model is expressed in terms of the inverse constants $1/m$ and $1/b$. Here, $1/m$ indicates the reaction rate, and $1/b$ reflects the maximum achievable oxidation capacity [14]. The kinetic evaluation of the Fenton reaction process on a laboratory scale can serve as a scientific basis for reactor design, process optimization, and scale-up strategies. In this study, the reduction of Chemical Oxygen Demand (COD) in batik industry wastewater was analyzed to determine the most suitable kinetic model. Linear regression based on the equations of Pseudo 1st order, Pseudo 2nd order, and BMG models was applied to assess the COD removal efficiency. The corresponding kinetic parameters (k_1, k_2, $1/m$, and $1/b$) derived from the regression analysis are summarized in Table 2.

Table 1. Kinetic model equations used to analyze Photo Fenton degradation *batik* wastewater.

Kinetic Model	Equation
1st-order	$\ln\frac{C_0}{C_t} = k_1.t$
2nd-order	$\frac{1}{C_t} - \frac{1}{C_0} = k_2.t$
BMG	$\frac{t}{1-(C_t/C_0)} = m + b.t$

Table 2 presents the rate constant values obtained for various H_2O_2/COD and H_2O_2/Fe^{2+} ratios. Figures 1a, 1b, and 1c show the linearized representations of the pseudo 1st order, pseudo 2nd order, and Behnajady Modirshahla Ghanbery (BMG) kinetic models, respectively. In general, both the 1st and 2nd order kinetic models adequately describe the rate of Chemical Oxygen Demand (COD) reduction in *batik* wastewater [15]. This is evidenced by the R^2 values, which show no significant differences between the two models, with average R^2 values of 0.8014 and 0.9124, respectively.

A study by previous researcher [16] which investigated the degradation of printing ink effluent and pre-treated industrial wastewater using UV/H_2O_2, found that a pseudo 1st order kinetic model provided the best fit, achieving up to 90% conversion. In contrast, the present study identified the BMG model as providing the best correlation, with R^2 values consistently greater than 0.9 or closest to 1. This aligns with findings from previous researches [14,15,17], all of whom reported that the BMG model most accurately represents the degradation kinetics in advanced oxidation processes. Among the various experimental ratios examined, increasing the concentration of H_2O_2 had a significant effect on COD reduction, likely due to the increased generation of hydroxyl radicals (OH*). According to researcher [18], hydroxyl radicals are extremely short-lived, with a typical lifespan of approximately 10^{-9} seconds. This corresponds with the second-order rate constants (k_2) in Table 1, which range from 10^{-5} to 10^{-7}, consistent with previously reported data. The short lifetime of OH* radicals also pose challenges in their detection and quantification. The BMG model was found to be the most suitable for describing the reaction kinetics of pollutant degradation in *batik* wastewater. Specifically, the parameters $1/m$ = 0.9352 and $1/b$ = 0.7159 indicate that a higher $1/m$ value corresponds to a faster initial degradation

Table 2. Kinetic results for Photo Fenton degradation *batik* wastewater.

H_2O_2/COD ratio	H_2O_2/Fe^{2+} ratio	1st Order		2nd Order		BMG		
		k_1, min^{-1}	R^2	k_2, L.mg^{-1}.min^{-1}	R^2	$1/m$ (min^{-1})	$1/b$	R^2
6	7	0.0055	0.9847	5.654E-07	0.9914	0.3229	0.0171	0.7854
8	7	0.0119	0.7988	1.517E-06	0.8770	0.5213	0.1129	0.9798
10	7	0.0173	0.6515	2.664E-06	0.7669	0.6493	0.3137	0.9956
11	12	0.0394	0.8170	1.409E-05	0.9531	0.9133	0.4077	0.9945
15	20	0.0431	0.7551	1.796E-05	0.9738	0.9352	0.7159	0.9986

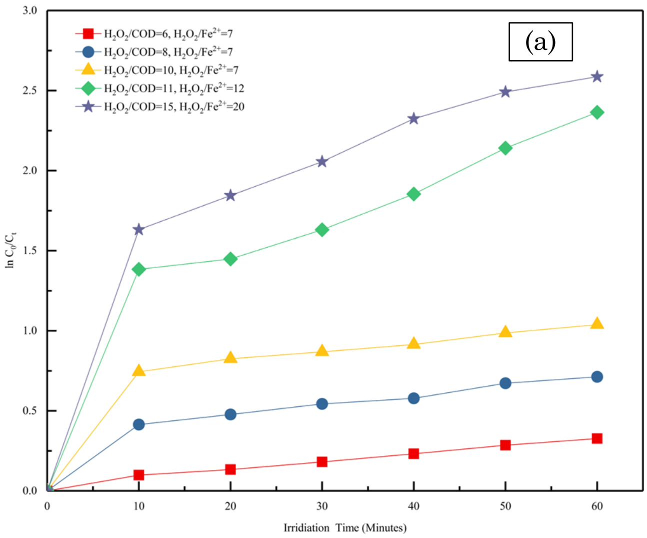
rate, while a higher $1/b$ value reflects a greater maximum oxidation capacity [19]. These findings demonstrate that kinetic modeling can effectively predict the degradation performance of pollutants in photo Fenton processes. This conclusion is further supported by the work of researcher [20], who showed that non phenolic aromatic compounds in Fenton oxidation are attacked by hydroxyl radicals (OH*), with acetaminophen (ACTP) yielding p-Hydroquinone (p-HQ) and p-Benzoquinone (p-BQ) as intermediates. The reaction rate constant for OH* in this context was reported as 7.1×10^9 $L.mol^{-1}.s^{-1}$.

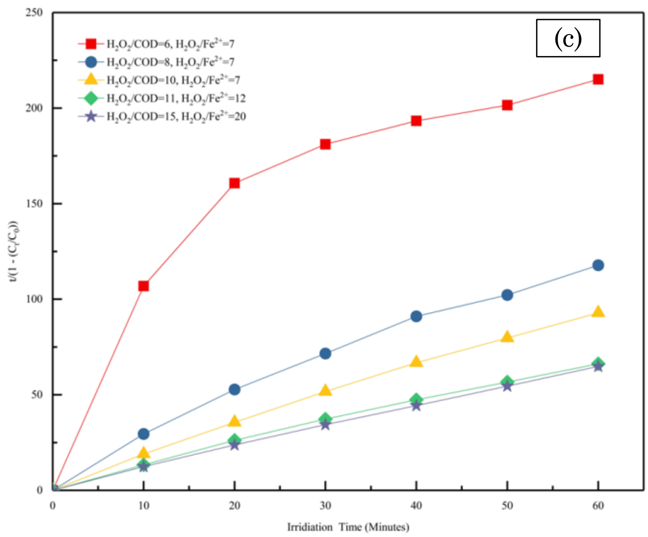
4. Conclusion

Based on research conducted to evaluate the effectiveness of the photo-Fenton method in degrading pollutants in *batik* industry wastewater, the optimal conditions were found at pH of 3, with an H_2O_2/COD ratio of 15 (g/g), and an H_2O_2/Fe^{2+} ratio of 20 (g/g). Under these conditions, the Chemical Oxygen Demand (COD) removal efficiency reached 92.47%. The reaction kinetics model describing the COD degradation rate followed the BMG kinetic model, with parameters of $1/m$ = 0.9352, $1/b$ = 0.7159, and a coefficient of determination (R^2) of 0.9986, indicating a very high correlation and strong model fit. The objectives stated in the introduction have been fully achieved. This study confirmed the high efficiency of the Photo-Fenton process under UV B irradiation, identified the optimum operating conditions, and established the most suitable kinetic model to describe COD degradation. These findings validate the research goals and demonstrate the potential of UV B–driven Photo-Fenton as an effective and scalable treatment technology for *batik* industrial wastewater.

CRedit Author Statement

Author Contributions: Oki Setiawan: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing Review & Editing, Supervision, Project Administration; Siti Adriani: Formal Analysis, Data Curation, Writing Original Draft Preparation, Visualization, Software; Haposan Vincentius Manalu: Validation, Writing Review & Editing, Data Curation; Aninditiya Amalia: Methodology, Investigation, Resources, Validation; Ifti Luthviana Dewi: Writing Review & Editing, Validation; Insan Kamil: Investigation, Resources, Writing Review & Editing, Validation; Darti Purnama Sari: Investigation, Resources, Writing Review & Editing, Validation; Popi Sasniati: Investigation, Resources, Writing Review & Editing, Validation. All authors have read and agreed to the published version of the manuscript.





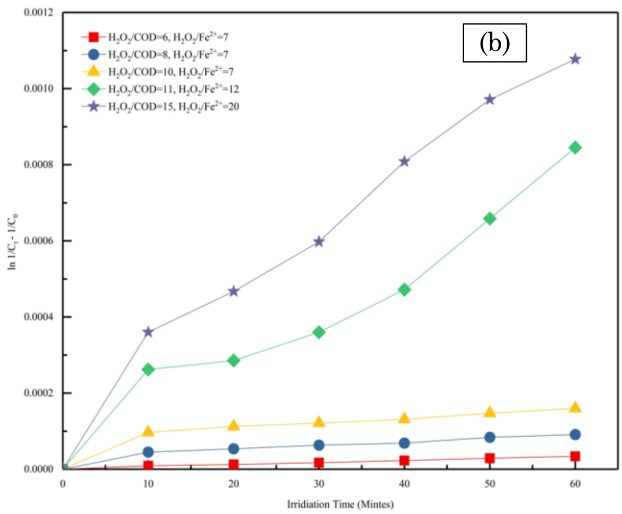


Figure 4. The kinetic study Chemical Oxygen Demand removal a 1st order, b. 2nd order, c. BMG.

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