Reaktor

p-ISSN 0852 – 0798
e-ISSN 2407 – 5973

Website: http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 26 No. 2, August Year 2026, pp. 1-8

Performance and Kinetics Evaluation of Pyrite-Activated Heterogeneous Fenton-Like Oxidation for COD Removal from Real Small-Scale Textile Effluent

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(Received: 20 February 2026; Accepted: 3 August 2026; Published: 15 August 2026)

Abstract

This study evaluated the performance of pyrite-activated heterogeneous Fenton-like oxidation in the treatment of real effluent generated from a local batik/textile industry. The influence of key operational parameters, including initial pH, hydrogen peroxide concentration, and pyrite dosage, was systematically investigated to determine the optimal conditions for COD removal. The results showed that acidic conditions significantly enhanced oxidation performance, with maximum COD removal achieved at pH 3. The optimum operating conditions were an H_2O_2 concentration of 10 g. L^{-1}, a pyrite dosage of 1 g. L^{-1}, and a reaction time of 6 h at room temperature, resulting in approximately 90% COD removal. Kinetic analysis demonstrated that the apparent pseudo-first-order model provided a better fit to the experimental COD degradation data than the pseudo-second-order model, yielding an apparent rate constant of 0.362 h^{-1} (R^2 = 0.996). Overall, this study demonstrates the potential applicability of natural pyrite as an effective and low-cost catalyst for treating real textile effluent. Further investigation into catalyst stability and reusability is recommended to enhance the economic feasibility of the process.

Keywords: COD removal; Fenton; heterogeneous; kinetic evaluation; pyrite

How to Cite This Article: Irawaty, W., Hartono, S.B., Wijaya, C.J., Gunarto, C., Puspitasari, N., Putro, J.N., Pardosi, G.N., and Hamidah, U., (2026) Performance and Kinetics Evaluation of Pyrite-Activated Heterogeneous Fenton-Like

Oxidation for COD Removal from Real Small-Scale Textile Effluent, Reaktor, 26 (2), 1–8, https://doi.org/10.14710/reaktor.82807

INTRODUCTION

The expansion of small- and medium-scale textile enterprises, including batik production units, has contributed substantially to local economic growth. However, this rapid development is accompanied by increasing environmental concerns, particularly related to wastewater discharge from dyeing and finishing processes.

Textile manufacturing involves multiple wet processing steps, including scouring, bleaching, dyeing, washing, and finishing, each generating effluents containing a complex mixture of chemicals. These wastewater streams may contain dyes, surfactants, alkaline agents, salts, heavy metals, and various organic additives. Synthetic dyes, especially those containing aromatic and azo structures, are known for their chemical stability and resistance to biodegradation (Yaseen and Scholz, 2019; Lellis et al., 2019). As a result, textile effluent is typically characterized by high chemical oxygen demand (COD), intense coloration, and variable alkalinity. The persistence of dye molecules in aquatic environments can reduce light penetration, inhibit photosynthesis, and disrupt aquatic ecosystems by decreasing dissolved oxygen levels (Hasan and Carr, 2018).

Conventional treatment methods such as biological processes and coagulation–flocculation often exhibit limited effectiveness in degrading recalcitrant dye compounds. Consequently, Advanced Oxidation Processes (AOPs) have been widely explored as promising alternatives for textile wastewater treatment. These processes rely on the generation of highly reactive oxidizing species capable of non-selectively degrading organic pollutants (Sun et al., 2023). Among various AOPs, Fenton-based oxidation has attracted considerable attention due to its operational simplicity, relatively low chemical cost, and strong oxidative capability associated with hydroxyl radicals (•OH), which possess a high redox potential ($E° = 2.80$ V) (Pignatello et al., 2006; Wang et al., 2021; Li et al., 2023).

Despite its effectiveness, the conventional homogeneous Fenton process suffers from several limitations, including narrow optimal pH range, catalyst loss, and generation of iron sludge requiring post-treatment handling (Shokri and Fard, 2022). To address these drawbacks, heterogeneous Fenton systems have been developed by employing solid iron-containing catalysts, allowing surface-mediated activation of hydrogen peroxide while reducing dissolved iron concentration in the treated effluent.

Various iron-based materials have been investigated as heterogeneous catalysts, including iron oxides, magnetite, zero-valent iron, and iron sulfides (Jiang et al., 2020; Zhang et al., 2021; Quynh et al., 2023; Zhu et al., 2023). Among these materials, natural pyrite (FeS_2) has emerged as an attractive candidate due to its abundance, low cost, and intrinsic iron content. Previous studies have demonstrated the effectiveness of pyrite in degrading model dye compounds such as rhodamine B, reactive orange, and methylene blue under Fenton-like conditions (Khataee et al., 2016; Fayazi, 2021; Javed, 2025). Nevertheless, most investigations have focused on synthetic dye solutions rather than real textile effluents, which typically contain more complex and variable compositions.

In addition, the catalytic performance of pyrite is strongly influenced by its mineral origin and physicochemical properties, leading to variations in degradation efficiency. Therefore, evaluation using real textile wastewater is essential to assess its practical applicability. Furthermore, although many studies report removal efficiencies, fewer investigations provide kinetic analysis to better understand the degradation behavior under optimized operating conditions. To date, limited studies have systematically linked operational optimization with kinetic modeling in real batik/textile effluent treated using natural pyrite as a heterogeneous Fenton-like catalyst.

Accordingly, the present study aims to evaluate the performance of pyrite-activated heterogeneous Fenton-like oxidation for the treatment of real textile effluent generated from small-scale batik production. Emphasis is placed on assessing the influence of operational parameters and examining the degradation kinetics based on COD removal. This work aims to contribute to a better understanding of catalyst performance in real wastewater systems and to support the development of cost-effective oxidation treatment for small-scale textile industries.

RESEARCH METHOD

Materials

All chemicals used in this study were of analytical grade and were used as received without further purification. Hydrogen peroxide (H_2O_2, 30%, CAS 7722-84-1), sulfuric acid (H_2SO_4, Fluka, 97%, CAS 7664-93-9), silver sulfate (Ag_2SO_4, Merck, CAS 10294-26-5), potassium dichromate ($K_2Cr_2O_7$, Merck, CAS 7778-50-9), mercury (II) sulfate ($HgSO_4$, Merck, CAS 7783-35-9), 1,10–phenanthroline ($C_{12}H_8N_2$, Merck, CAS 66-71-7), and ammonium iron (II) sulfate ($(NH_4)_2Fe(SO_4)_2$, Merck, CAS 7783-85-9) were employed to conduct the Fenton reactions. The wastewater used in this study was supplied by a local batik empowerment enterprise ‘Wisata Kampung Batik Okra Surabaya’, located at Kranggan VII, Bubutan, Surabaya City, East Java, Indonesia. The wastewater sample was real effluent produced during the wash-off stage and was collected from several production batches. The raw effluent had an initial COD of 1,833 mg L^{-1}, an initial pH of 4.0, and exhibited a dark-colored appearance. Natural pyrite

obtained from Bali (Indonesia) was ground and sieved to obtain a particle size of less than 0.105 mm prior to experimental use.

Method

Fenton oxidation experiments were conducted at a laboratory scale following the same pre-reaction procedure described in our previous work (Irawaty et al., 2023). A predetermined amount of pyrite and H_2O_2 solution were added to a reaction tube containing pH-adjusted water. The mixture was allowed to stand for 20 min. Subsequently, an equal volume of the effluent was introduced to initiate the oxidation process. After a certain time, the reaction was terminated, and the supernatant was collected for COD analysis (Lovibond, RD125). All reactions were performed at room temperature, except the COD analysis (150°C, SNI 6989.73:2009). The treatment performance under different operating conditions was evaluated in terms of COD removal efficiency, calculated as:

$$\text{COD removal (\%)} = \frac{COD_0 - COD_t}{COD_0} \times 100 \quad \ldots. (1)$$

where COD_0 and COD_t are the initial COD and the COD at reaction time t, respectively.

The COD degradation data obtained under the selected operating conditions were evaluated using apparent first-order and second-order kinetic models. The apparent rate constants were obtained from the slopes of the corresponding linear regressions. Model performance was compared using the coefficient of determination (R^2). The term "apparent" is used because COD represents the combined contribution of multiple organic constituents in the real textile effluent rather than the concentration of a single reacting compound. Since the residual H_2O_2 concentration was not monitored during the reaction, the kinetic fitting was used only to describe the observed COD decay and not to establish that H_2O_2 remained in excess.

RESULTS AND DISCUSSION

Effect of Pyrite Concentration

Figure 1 shows the effect of pyrite dosage on COD removal. As seen, the removal efficiency increased markedly as the pyrite concentration was increased from 0.2 to 1 g L^{-1}, improving from 38% to 90%. Further increases in catalyst loading resulted no noticeable improvement.

The initial increase in COD removal can be attributed to the greater availability of catalytic active sites in our system. With more pyrite present in the system, more surface area becomes available for hydrogen peroxide activation, leading to the formation of more hydroxyl radicals according to reactions (Wang et al. (2021):

$$FeS_2 + 15/2\ H_2O_2 \rightarrow Fe^{3+} + 2SO_4^{2-} + H^+ + 7H_2O \quad (2)$$

$$FeS_2 + 2H^+ \rightarrow Fe^{2+} + S + H_2S \quad (3)$$

$$H_2O_2 + Fe^{2+} + H^+ \rightarrow Fe^{3+} + \cdot OH + H_2O \quad (4)$$

Hydroxyl radicals are widely recognized as the key reactive species in the Fenton-based degradation system as they actively attack organic contaminants, leading to the formation of intermediate products that are eventually further converted into final end products.

$$\cdot OH + \text{dye/contaminants} \rightarrow H_2O + CO_2 + \text{mineral acids} \quad (5)$$

As a result, the degradation rate of organic compounds is increased by pyrite concentration that is reflected by higher COD removal percentages. However, beyond a certain catalyst concentration, in this study was 1 g/L and up, the improvement in COD removal became marginal and eventually approached a steady value. Several studies on heterogeneous Fenton-like systems have reported similar trends (Kumar et al., 2021; Chen et al., 2021; Kumar et al., 2020). This stagnation may be associated with several factors. First, the amount of hydrogen peroxide in the system becomes the limiting reagent when an excessive catalyst is present, meaning that additional pyrite does not translate into proportional increases in oxidation activity. Second, at higher catalyst dosage, particle aggregation and reduced effective surface exposure may occur, which can limit mass transfer between the oxidant and the catalyst surface. In addition, excessive catalyst concentration may reduce overall oxidation efficiency due to possible side reactions between Fe^{2+} and hydroxyl radicals, as reported in a previous study (Sun et al., 2021). Therefore, although increasing pyrite dosage enhances COD removal up to an optimal level (1 g L^{-1} in this study), further addition does not result in a significant improvement in process performance.

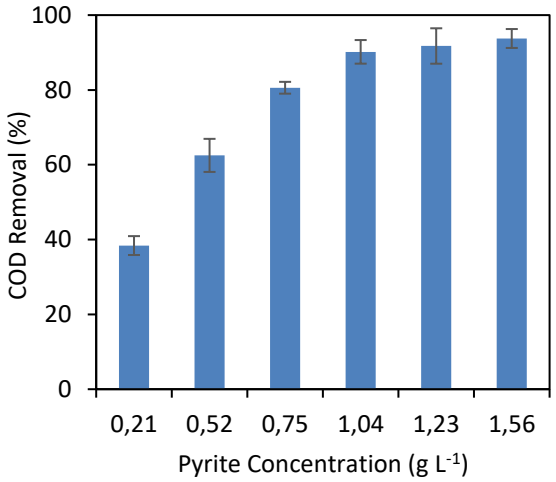

Figure 1. Effect of pyrite dosage on COD removal. Experimental conditions: initial pH: 3, reaction time: 6 h, H_2O_2 concentration: 10 g L^{-1}

Effect of pH

The influence of initial pH on COD removal was studied in the range from 2 to 6 as shown in Figure 2. The results clearly show that pH strongly affects the oxidation performance. COD removal increased from pH 2 to pH 3, indicating that mildly acidic conditions provided the most favorable condition. Interestingly, while pH 2 did not yield the maximum removal, it still outperformed the higher-pH conditions (pH 4-6),

resulting in the overall trend: pH 2 > pH 4 > pH 5 > pH 6. This behavior suggests that the system benefits from acidic conditions, whereas the oxidation efficiency gradually declines as the pH shifts toward neutral (Lin et al., 2022; Omar et al., 2024).

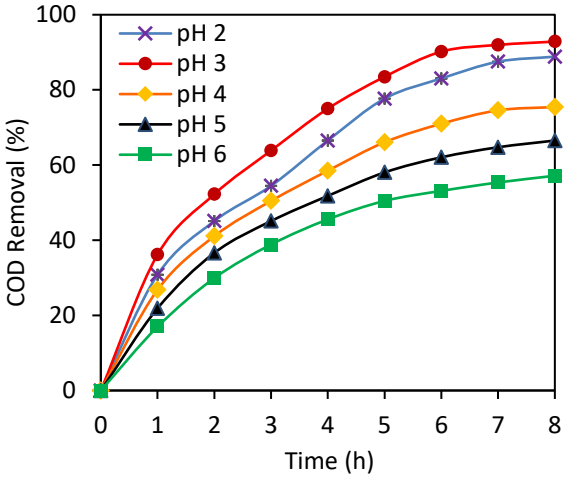

Figure 2. Effect of pH on COD removal. Experimental conditions: Pyrite dosage: 1 g L^{-1}, H_2O_2 concentration: 10 g L^{-1}

The pH dependence observed in the present study is consistent with previous reports on heterogeneous Fenton systems, in which maximum oxidation efficiency is generally achieved within the acidic range of pH 2.5-3.5 (Pignatello et al., 2006; Li et al., 2023). The highest COD removal obtained at pH 3 suggests that this condition provides the most favorable conditions for iron-mediated activation of H_2O_2 and subsequent hydroxyl radical generation. Although COD removal remained relatively high at pH 2, the oxidation efficiency was slightly lower than that at pH 3, which may be attributed to the excessive proton concentration under strongly acidic conditions. Such conditions can reduce the effective utilization of hydrogen peroxide and consequently limit hydroxyl radical production (Lin et al., 2022). As the pH increased from 4 to 6, the gradual decline in COD removal can be attributed to changes in iron speciation and the precipitation of iron (oxy)hydroxides, which decrease the availability of catalytically active iron species and suppress hydrogen peroxide activation (Chen et al., 2021; Omar et al., 2024).

Effect of H_2O_2 Concentration

The effect of hydrogen peroxide concentration is presented in Figure 3. The results indicate that increasing H_2O_2 concentration up to 10 g L^{-1} significantly improved the removal efficiency. However, the further addition of hydrogen peroxide did not provide substantial enhancement in COD reduction. In this study, the optimum COD removal efficiency was obtained at 10 g L^{-1}.

At lower dosages, the amount of hydrogen peroxide may be insufficient to generate adequate oxidizing species required for degrading organic compounds present in the effluent, resulting in lower COD removal. As the hydrogen peroxide concentration increases, more oxidizing species can be formed in the system, thereby enhancing the degradation of organic matter and improving removal efficiency. Similar trend was reported by others (Ganesan et al., 2024; Wang et al., 2021).

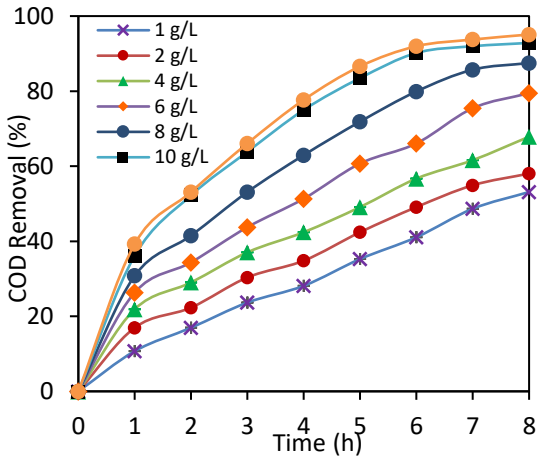

Figure 3. Effect of H_2O_2 concentration on COD removal. Experimental conditions: Pyrite dosage: 1 g L^{-1}, pH: 3

However, when the hydrogen peroxide dosage exceeds the optimal level, no further improvement in COD removal is observed. This behavior may be attributed to the excess oxidant, which can act as a scavenger of reactive species, leading to non-productive side reactions and thereby reducing the effective oxidation capacity of the system. In addition, surplus hydrogen peroxide may undergo self-decomposition reactions that do not directly contribute to organic degradation (Ayoub, 2022). Such effects have been widely reported in Fenton-based oxidation systems (Chen et al., 2021; Sun et al., 2021; Quynh et al., 2023).

A comparison between the present study and previously reported heterogeneous Fenton systems is summarized in Table 1. Although differences in catalyst type, wastewater composition, and operating conditions exist, the optimum acidic conditions observed in the present work are generally consistent with those reported in previous studies. Nevertheless, noticeable variations in hydrogen peroxide dosage and treatment performance can be observed. Wang et al. (2021) demonstrated rapid degradation of synthetic dye solutions using natural pyrite over a wide pH range (3-10) with a relatively low hydrogen peroxide dosage, whereas Ganesan et al. (2024) achieved 74% COD removal from synthetic textile wastewater at pH 3 using steel flakes as the iron source and 4.5 g L^{-1} H_2O_2. Similarly, Yong et al. (2012) reported 70% COD removal from textile wastewater using natural pyrite with an initial solution pH of 9, which gradually decreased during the oxidation process due to pyrite dissolution.

Table 1. Comparison of operating conditions and treatment performance of representative heterogeneous Fenton systems.

Waste water	Catalyst	Optimum pH	Optimum H_2O_2 (g L^{-1})	COD removal (%)	Ref.
Synthetic dye	Natural pyrite (1 g.L^{-1})	3 – 10	0.15	Degradation > 95	Wang et al. (2021)
Synthetic textile waste water	Steel flakes (1 g.L^{-1})	3	4.5	74	Ganesan et al. (2024)
Textile waste water	Natural pyrite (10 g. L^{-1})	9 (Initial pH)	0.34	70	Yong et al. (2012)
Batik effluent	Natural pyrite (1 g. L^{-1})	3	10	90	this study

Compared with these previous studies, the present work required a higher hydrogen peroxide dosage (10 g L^{-1}) to achieve the optimum COD removal of approximately 90%. This difference is likely associated with the greater complexity of the real batik effluent, which contains a heterogeneous mixture of dyes, surfactants, and other organic additives rather than single-component model pollutants or synthetic wastewater. Consequently, the wastewater exhibits a higher oxidant demand to achieve effective degradation of the overall organic load. Despite the higher hydrogen peroxide requirement, the results demonstrate that natural pyrite is an effective heterogeneous Fenton-like catalyst for treating real batik wastewater under practical operating conditions.

Kinetics Analysis of COD Removal

Kinetic analysis was performed to determine the overall kinetic model of the effluent degradation. The oxidation of organic matter in the Fenton-based system can be described by a bimolecular reaction involving COD and hydrogen peroxide. In its general form, the reaction rate may be expressed as:

$$\text{rate} = -\frac{d[COD[}{dt} = k\,[COD]\,[H_2O_2] \quad (6)$$

which represent an overall second-order reaction, first order with respect to COD and first order with respect to hydrogen peroxide.

The degradation of organic matter in Fenton systems is commonly described by a bimolecular rate expression involving both the organic substrate and hydrogen peroxide. Under certain operating conditions, when the variation in hydrogen peroxide concentration is relatively small over the reaction period, the rate equation may be approximated by an apparent first-order expression with respect to COD. This approximation has been widely adopted to describe the overall degradation behavior in Fenton and heterogeneous Fenton-like systems.

By incorporating the constant hydrogen peroxide concentration into the rate constant, a new apparent rate constant can be defined as:

$$k' = k\,[H_2O_2] \quad (7)$$

Substituting this expression into equation (6) yields:

$$rate = -\frac{d(COD)}{dt} = k'\,[COD] \quad (8)$$

which corresponds to a pseudo-first-order kinetic model with respect to COD. Integration of this equation leads to the linear form:

$$\ln\left(\frac{COD_0}{COD_t}\right) = k't \quad (9)$$

where COD_0 and COD_t represent the initial COD and the COD at time t, respectively, and k' is the pseudo-first-order rate constant (h^{-1}). The value of k was obtained from the slope of the linear regression.

In addition to the pseudo-first-order model, the degradation data were also evaluated using a pseudo-second-order kinetic approach. In this model, the reaction rate is assumed to be proportional to the square of the COD concentration, which can be expressed as:

$$rate = -\frac{d(COD)}{dt} = k''(COD)^2 \quad (10)$$

Integration of this equation gives the linear form:

$$\frac{1}{COD_t} = \frac{1}{COD_0} + k''t \quad (11)$$

where k'' is the pseudo-second order rate constant (L g^{-1} h^{-1}). Thus, a linear relationship between $1/COD_t$ and time would indicate pseudo-second-order behavior. The suitability of each model was assessed based on the correlation coefficient (R^2) from the corresponding linear fits.

Unlike the pseudo-first-order model, which is derived from the assumption of excess hydrogen peroxide in a bimolecular reaction system, the pseudo-second-order model represents an empirical approach that considers the possibility of concentration-dependent interactions during oxidation. In heterogeneous Fenton-like systems, such behavior may arise from surface-mediated reactions or complex radical–pollutant interactions. Therefore, a comparison of both models was performed solely to identify the kinetic expression that best represents the experimental COD degradation data. Kinetic modelling was conducted using experimental data collected at the optimum operating conditions, namely a pyrite dosage of 1 g L^{-1}, hydrogen peroxide concentration of 10 g L^{-1}, and initial pH of 3. The corresponding kinetic parameters obtained are presented in Table 2.

Table 2. Kinetic parameters for the effluent degradation at optimal conditions.

Kinetic model	Rate constant (k)	R^2
Pseudo-first-order	0.362 h^{-1}	0.996
Pseudo-second-order	0.0009 L g^{-1} h^{-1}	0.916

As shown in Table 2, the apparent first-order model exhibited a higher correlation coefficient (R^2 = 0.996) than the apparent second-order model (R^2 = 0.916), indicating that the first-order model provides a better empirical description of COD removal under the investigated conditions. The high linearity suggests that the rate of COD degradation was approximately proportional to the remaining organic load during the reaction period.

It should be noted, however, that this kinetic relationship should not be interpreted as direct evidence that hydrogen peroxide remained in excess throughout the reaction. The pseudo-first-order approximation is commonly adopted to describe Fenton oxidation systems, but confirmation of constant hydrogen peroxide concentration would require independent monitoring of residual H_2O_2 during the reaction. Since residual H_2O_2 was not measured in the present work, the apparent first-order model is interpreted as an empirical representation of COD decay rather than a mechanistic proof of excess oxidant.

Although the present study demonstrates the effectiveness of pyrite-activated Fenton-like oxidation for COD removal, dissolved iron concentration was not monitored during the reaction. Therefore, the relative contributions of heterogeneous surface catalysis and homogeneous Fenton reactions arising from iron dissolution could not be quantitatively distinguished. Previous studies have reported that pyrite can release Fe^{2+} under acidic conditions, allowing dissolved iron species to participate in homogeneous Fenton reactions in parallel with surface-mediated oxidation (Diao et al., 2016; Wang et al., 2021). Furthermore, the complexity of Fenton chemistry has been widely recognized, involving simultaneous heterogeneous and homogeneous reaction pathways depending on catalyst properties and operating conditions (Pignatello et al., 2006). Consequently, the oxidation system investigated in this work is more appropriately described as a pyrite-activated heterogeneous Fenton-like process rather than a purely heterogeneous mechanism. Future work should include iron leaching measurements to quantify the contribution of dissolved iron and further elucidate the dominant oxidation pathway.

CONCLUSION

This study evaluated the performance of batch-operated Fenton oxidation in the treatment of effluent from the local batik (textile) industry, with particular emphasis on identifying the key operational parameters influencing COD removal. The results demonstrated that acidic conditions were favorable, with maximum COD removal achieved at pH 3. The optimal operating parameters were H_2O_2 concentration of 10 g L^{-1}, pyrite dosage of 1 g L^{-1}, and a reaction time of 6 h performed at room temperature, resulting in approximately 90% COD removal. Kinetic evaluation demonstrated that the apparent pseudo-first-order model provided a better fit to the experimental COD degradation data than the pseudo-second-order model, with an apparent rate constant of 0.362 h^{-1}. Further investigations should include catalyst characterization, iron leaching analysis, and catalyst reusability to better correlate catalyst properties with oxidation performance and to further evaluate the practical applicability of the pyrite-activated heterogeneous Fenton-like process.

ACKNOWLEDGMENT

This work was supported by Research and Community Service Institute, Widya Mandala Surabaya Catholic University under Research Grant No. 193A/WM01.5/N/2025. The authors also thank Mr. Novi and Ms. Kiki for their technical support during the laboratory work and experimental data collection.

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