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Efficient degradation of aniline yellow dye using photo-Fenton advanced oxidation process: Optimization via central composite design

Ethel Grace P. Mohammad[1,2], Michie C. Mahino[1], Jennelyn G. Mayormita[1], Gizabelle E. Zayas[1], Shaira R. Anaya[1], Val Irvin F. Mabayo[1,*] and Renato O. Arazo[1]

[1]*College of Engineering and Technology, University of Science and Technology of Southern Philippines, Claveria 9004, Philippines*
[2]*Western Mindanao State University, Zamboanga City 7000, Philippines*

Corresponding Author: valirvin.mabayo@ustp.edu.ph

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ABSTRACT

Dyes are the coloring agents considered pollutants when combined in water bodies. This study used the photo-Fenton process, one of the advanced oxidation processes, to degrade aniline yellow dye (AYD). It is a primary dye that blends with any color, creating other shades of dye. Operating variables, namely initial concentration, contact time, and pH were studied in the degradation of AYD pollutants. A central composite design was applied to acquire the optimum conditions of these independent variables, resulting in the AYD degradation and eliminating up to 94.00% at pH 5 and an initial AYD concentration of 35 ppm. The removal efficiency of $FeCl_3$ resulted in an AYD removal of 2.03 mg per gram of $FeCl_3$. The pseudo-first kinetic model best explained the mechanism of degradation and removal of AYD in aqueous solution. The results of the study showed that the photo-Fenton process using UV light from fluorescent lamp and Fenton's reagents (H_2O_2 and $FeCl_3$) effectively degraded AYD in water.

Keywords: advanced oxidation process (AOP), dye removal, photocatalysis, pollutant removal, wastewater treatment

INTRODUCTION

The textile industry is essential for global economic growth but poses significant environmental challenges due to its extensive use of chemicals and water consumption (Mabayo and Orale 2024). It significantly contributes to worldwide water pollution by releasing many chemicals and colors into water bodies. Textile dyeing alone is estimated to account for 17-20% of industrial water contamination, posing an urgent environmental concern. Dyes are a significant source of pollution from textile processes. Azo dyes, in particular, are among the industry's most widely utilized.

Aniline yellow dye (AYD) belongs to the azo dye family. It is widely used in microscopy for vital staining and the manufacture of yellow colors and inks. However, the indiscriminate release of AYD into water bodies poses significant environmental and health hazards. The AYD has been recognized as exceedingly hazardous, with potentially carcinogenic effects and adverse effects on aquatic ecosystems (Pagalan Jr et al. 2020).

Efforts to mitigate the environmental impact of dye contamination have led to exploring various treatment methods (Nerona et al. 2024; Shaltout et al. 2024; Su et al. 2024). Conventional wastewater treatment methods such as adsorption, thermal incineration, and membrane filtration struggle to remove AYD, necessitating the practical exploration of alternative approaches (Chaturvedi 2022). Among these, advanced oxidation processes (AOPs) have emerged as promising solutions for the degradation of organic pollutants. Advanced oxidation processes (AOPs) generate highly reactive hydroxyl (•OH) radicals to break down organic compounds. Among these processes, the Fenton-based advanced oxidation process has gained attention. In the Fenton process, ferrous ions (Fe^{2+}) react with hydrogen peroxide (H_2O_2) to produce •OH radicals. These radicals exhibit strong oxidative power and can effectively degrade organic pollutants (Giwa et al. 2021; Pandis et al. 2022). Compared to other AOPs such as ozonation and photocatalysis, the photo-Fenton process offers distinct advantages and limitations. For instance, ozonation can achieve similar removal efficiencies, but the photo-Fenton process demonstrates faster reaction times, reduced operational complexity, and higher efficacy under certain conditions. On the other hand, photocatalysis can degrade a wider range of organic pollutants but often requires more expensive catalysts and longer reaction times.

However, the Fenton process alone may not be sufficient for complete AYD degradation. To enhance its efficiency, researchers have combined it with ultraviolet (UV) light irradiation, resulting in the photo-Fenton advanced oxidation process (Ameta et al. 2018). This technique treats the solution under Fenton conditions while simultaneously exposed to UV light. The UV light accelerates the degradation rate of organic pollutants, making the process more effective. The photo-Fenton process offers several advantages, including increased reaction rates, improved degradation efficiency, and reduced energy consumption (Kumari and Kumar 2023; O'Dowd and Pillai 2020; Shalini and Setty 2022; Sriprom et al. 2018).

This study investigated the degradation of AYD contaminants using the photo-Fenton AOP including the impact of initial AYD concentration, exploring optimal contact times, and evaluating the influence of pH on the process. These factors were selected because they play a crucial role in optimizing the degradation efficiency of the photo-Fenton process. Initial AYD concentration affects the availability of pollutants for degradation, while contact time is critical for determining how long the radicals have to act on the contaminants. The pH, which influences the stability of the reactive species in the Fenton process, is also a key parameter in controlling the efficiency of the degradation process. By systematically varying these parameters, the researchers aim to optimize the photo-Fenton process for efficient AYD removal from aqueous solutions.

METHODS

Experimental Design

Central composite design (CCD) was the tool responsible for giving the number of runs and the different operating variables. Three factors are being optimized. The range and levels are shown in Table 1. The values that were put in the range and levels were based on the preliminary experiment done and on the literature. A preliminary experiment was done to make sure that the percentage error was not that high and to have a result near the desired expectation.

After entering the coded level values, the CCD would give the experimental runs showing the combination of different values of independent variables, as shown in Table 2.

AYD Solution Preparation

The 1000 mg of AYD (CAS No. 60-09-3, purity >98%) was mixed with 1 L of distilled water to produce a 1000 ppm solution subject to dilution to make the desired concentration for every run carried out from central composite design (CCD), as shown in Table 2. The ratio and proportion were performed to get the desired initial concentration values of AYD (Genesiran et al. 2015).

Photo-Fenton Process

The primary step in the photo-Fenton process is to take the pH generated by the design expert on different runs. Hydrochloric acid (HCl, 1M) was used to obtain the desired pH and get the acidic solution.

Table 1. Experimental range and levels of independent variables.

Factors	Coded Level				
	-2	**-1**	**0**	**1**	**2**
Initial concentration (ppm)	10	20	30	40	50
Contact Time (min)	20	30	40	50	60
pH	2	3	4	5	6

Table 2. Efficiency of the degradation of the aniline yellow dye (AYD) through photo-Fenton process.

Run	Initial concentration (ppm)	Time (min)	pH	% AYD Removal
1	35	50	3	84.63
2	35	50	5	93.64
3	25	50	3	91.60
4	35	30	3	87.15
5	25	50	5	93.12
6	30	40	4	87.53
7	30	40	6	91.74
8	25	30	3	84.54
9	30	60	4	93.42
10	30	40	4	87.53
11	40	40	4	90.97
12	30	20	4	86.27
13	30	40	4	88.80
14	35	30	5	95.45
15	30	40	4	87.95
16	20	40	4	88.87
17	30	40	2	87.11
18	30	40	4	90.90
19	25	30	5	84.03
20	30	40	4	88.38

After getting the desired pH of the solution to be treated, the reagents H_2O_2 (30%, Merck) and $FeCl_3$ (reagent grade, 97%) were added to the solution. The prepared solution was placed in the prototype photo reactor, having four fluorescent lamps positioned equally to each other. The duration of the solution inside the reactor differed depending on the time given by the CCD.

Afterward, the solution was removed from the reactor, and the absorbance was determined using a Hach DR/850 colorimeter.

Experimental Setup

The prototype photoreactor (Figure 1) is 2 feet tall, has a 16 cm hole diameter, and has 32 cm entire diameter. The four fluorescent tubes with 18 watts each are placed at an equal distance of 22.63 cm along the circumference of the cylinder facing each other, as adopted in the study of Genesiran et al. (2015).

The fluorescent lamps served as the light source of the reaction where the photo-Fenton treatment takes place. The magnetic bar and stirrer were used to disperse the solution during irradiation.

Dye Removal Analysis

In getting the percentage removal, the absorbance of each sample was tested using the DR/850 colorimeter. After obtaining the absorbance, a calibration formula was used to get the value of the dye residual. The calibration curve formula in getting dye residual is shown in Equation 1 where x and y in the absorbance is the residual (ppm).

When the value of the dye residual has been identified, percentage removal was determined using Equation 2 where C_o is the amount of initial concentration and C_e is the value of the dye residual.

Effects of the Photo-Fenton Process

The effects of the photo-Fenton process were determined after all experimental ran. The data were analyzed using variance analysis (ANOVA) of the response surface methodology (RSM).

Optimization of the Removal of AYD

Three factors were optimized: the initial concentration, contact time, and pH. The recommended combinations of these independent variables were used to identify the percent removal of the AYD. The lowest percentage error obtained was considered the best conditioning for optimum degradation of AYD. The formula for getting the least percentage error is shown in Equation 3.

Kinetic Model

In determining the appropriate kinetic model best fitted to the degradation of AYD via AOP, initial concentration values, pH, and time at optimum conditions were used. The time was divided by 10, and 10 experimental runs were conducted at optimum pH and concentration with variable time at 3-minute increments for each run.

The equations of the pseudo-first and second order are shown in Equations 4 and 5, respectively, where C_o is the initial concentration of AYD (mg/L), C_t is the final concentration (mg/L), t in contact time (min), k_1 is the pseudo-first-order rate constant (min^{-1}), and k_2 is the rate constant of pseudo-second-order (mg/L min).

$$y = 126.23x - 1.5617 \quad \text{(Eq. 1)}$$

$$\text{Percent AYD removal} = \frac{C_o - C_e}{C_o} \times 100 \quad \text{(Eq. 2)}$$

$$\%\ \text{error} = \frac{\text{Predicted Value} - \text{Actual Value}}{\text{Actual Value}} \text{ x } 100 \quad \text{(Eq. 3)}$$

$$C_t = C_o e^{-k_1 t} \quad \text{(Eq. 4)}$$

$$C_t = \frac{C_o}{1 + k_2 C_o t} \quad \text{(Eq. 5)}$$

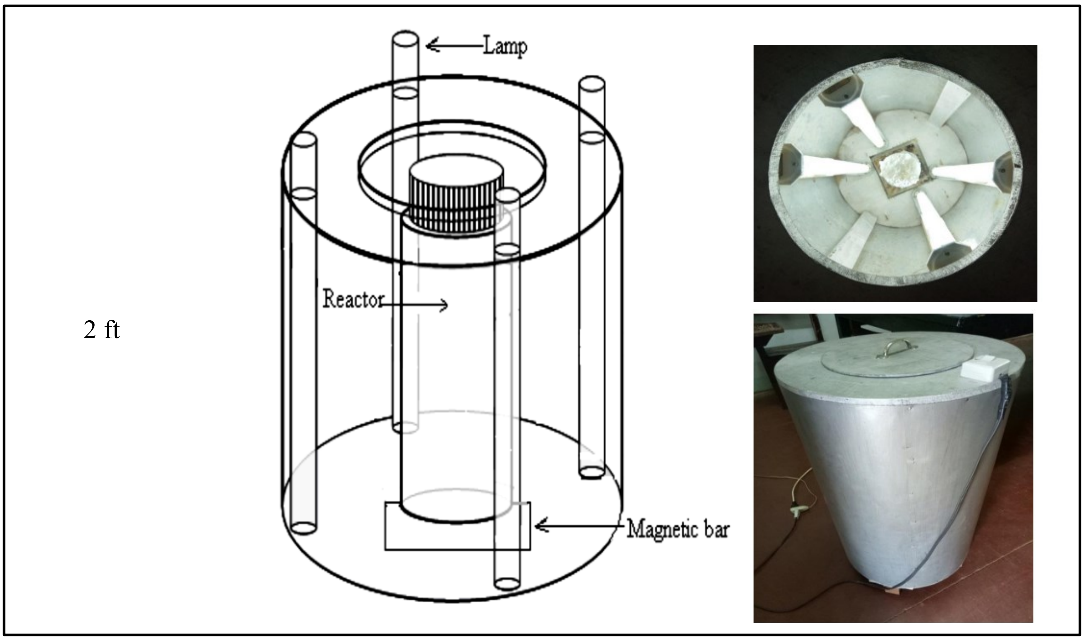

Figure 1. The prototype reactor set-up. [Redrawn from Genesiran et al. (2015)].

RESULTS

AYD Percent Removal via Photo-Fenton Process

The experimentation of AYD degradation through the photo-Fenton process was conducted based on the CCD experimental combination (Table 2).

Response surface methodology (RSM) modeled the values through Design Expert 7.0 software, which obtained the statistical outcomes. The data showed that the percent removal of AYD using the photo-Fenton process was about 84.03% to 95.45%.

Model Fitting on AYD Degradation via Photo-Fenton Process

The reduced two-factor interval was found and best fitted to predict the removal of AYD through the photo-Fenton, as suggested by the CCD. The reduced model is shown in Equation 6, where *Y* represents the percent removal of AYD while A, B, and C are the values of initial concentration (ppm), contact time (min), and pH, respectively.

The positive coefficients indicate an effect of increasing dye removal, while the negative coefficients indicate otherwise.

$$Y = 59.007 + 0.559A + 1.659B - 10.092C - 0.050AB + 0.395AC \quad \text{(Eq. 6)}$$

The model's acceptability was verified through statistical analysis using ANOVA in Table 3. The result of the ANOVA implied a highly significant reduced model with a $P < 0.0001$, indicating that there is a 99.99% chance that the model is accurate and reliable.

Moreover, the initial concentration, contact time, and pH, including AC and AB, were considered significant with a p-value less than 5%. Overall, the reduced two-factor interval model was precise, as supported by the lack of fit p-value of 0.5649, which is not significant. This implied that the model generated could predict the percent removal of AYD given the values of the chosen variables.

Impact of Operating Parameters on AYD Degradation

The investigation explored the influence of operating parameters on AYD removal via photo-Fenton AOP. The initial concentration of AYD (20-40 ppm), contact time (20-60 min), and pH (2-6) were varied, and their effects were statistically significant ($P < 0.05$) based on the reduced two-factor interval model.

In Figure 2A, the highest removal efficiency (92.7%) was achieved when the lower initial concentration (25 ppm) was combined with increased contact time (30-50 min). This aligns with the concept of having more hydroxyl radicals available to attack fewer dye molecules at lower concentrations and allowing for more degradation with extended reaction time.

Figure 2B analysis suggests a maximum removal of 93.7% at the highest concentration (35 ppm) and acidic pH (5). However, actual experiments achieved a higher maximum removal (95.45%) at a different concentration under the optimized contact time determined by the model. This emphasizes the importance of contact time for optimal removal. The increasing removal with increasing pH up to 5 supports the idea of favorable mildly acidic conditions for hydroxyl radical generation. Conversely, a decrease in removal at higher pH (beyond 5) suggests scavenging of hydroxyl radicals by hydroxide ions (OH-) at excessively high pH levels.

Table 3. ANOVA for the response surface reduced two-factor interval model. [a] = significant; [b] = not significant.

Source	Sum of Squares	Df	Mean Square	F Value	p-value
Model	179. 99	6	30.00	19.66	< 0.0001[a]
A – Initial Concentration	7.92	1	7.92	5.19	0.0403[a]
B – Time	41.06	1	41.06	26.91	0.0002[a]
C – pH	49.29	1	49.29	32.31	< 0.0001[a]
AB	49.90	1	49.90	32.71	< 0.0001[a]
AC	31.19	1	31.19	20.44	0.0006[a]
Residual	19.84	13	1.53		
Lack of Fit	11.81	8	1.48	0.92	0.5649[b]
Pure Error	8.03	5	1.61		
Cor Total	199.82	19			
$R^2 = 0.9007$					

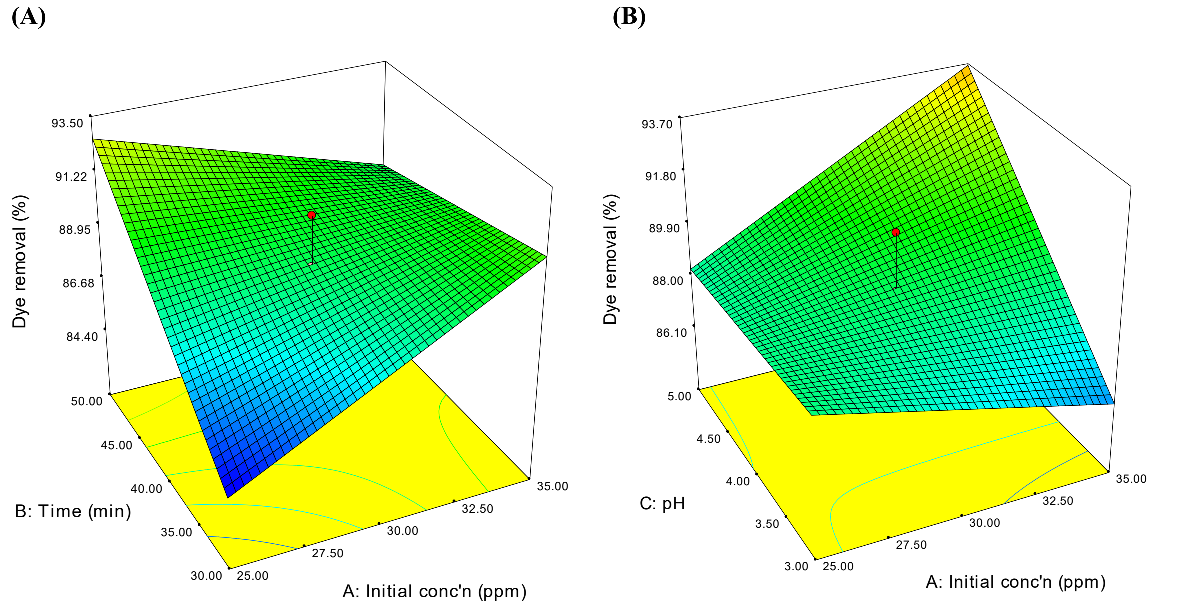

Figure 2. 3D surface plots showing the interactive effects of (A) initial concentration of aniline yellow dye (AYD, ppm) and time (min), (B) initial concentration of AYD (ppm) and pH.

Optimized Condition for AYD Removal

Central composite design (CCD) was used as the experimental technique to search for the optimum values of the parameters. The optimum values were generated from the CCD, which had an initial concentration of 35 ppm, 30 min of contact time, and 5 pH, and a predicted AYD degradation of 94.26%.

The optimum values were validated through actual experimentation runs and reached a closed outcome between the predicted value (94.26%) and actual value of (94.00%), having 0.26% percent of error. This indicates that the model equation can calculate the degradation of AYD, given the operating parameters.

Kinetic Modeling of AYD Degradation

The most appropriate kinetic model was determined through kinetic experimental runs under an optimum level of 35 ppm of initial concentration and a pH of 5 while varying the optimum contact time of 30 min by division of 10 (3, 6, 9, 12, 15, 18, 21, 24, 27, and 30). The results of the kinetic experimental runs are shown in Figure 3.

In the pseudo-first-order kinetic plot, the points were more closely positioned along the trend line than in the pseudo-second-order kinetic model plot. Therefore, the study on the degradation and removal of AYD through the photo-Fenton process exhibited that the first model had the greatest R^2 of 0.9625, which best defined the experimental data, compared to the second order, with R^2 of 0.9510.

The kinetics of AYD removal can be best described through the pseudo-first-order, which has a higher coefficient of determination than the second-order. This means that the removal of AYD is highly dependent on the amount of present •OH radicals.

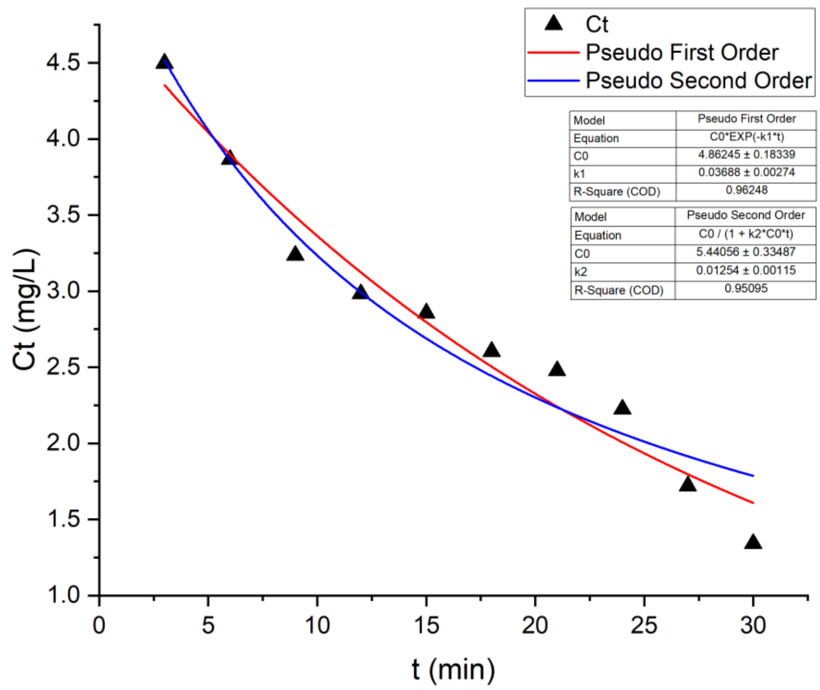

Figure 3. Kinetic model plots for Pseudo-First Order and Pseudo-Second Order.

DISCUSSION

Removal of AYD via Photo-Fenton Process

The results presented in Table 2 demonstrate the effectiveness of the photo-Fenton AOP for degrading AYD in aqueous solution. The achieved percentage removal of AYD ranged from 84.03% to 95.45%, indicating a significant treatment efficiency.

The data suggest that the degradation efficiency is influenced by several factors as designed in the CCD experiment (Table 2). Lower initial concentrations (Runs 3, 5, 8) generally resulted in higher degradation percentages. This can be attributed to the greater availability of •OH radicals for attacking fewer dye molecules (He et al. 2002; Macías-Sánchez et al. 2011). Increased reaction time (Runs 2, 5, 7) typically led to improved degradation. This allows for a longer exposure of AYD molecules to the oxidizing radicals. Furthermore, The optimal pH for the photo-Fenton process is typically acidic (2-3). However, the data do not show a definitive trend within the tested range (pH 3-5). Further investigation might be needed to determine the optimal pH for degrading AYD.

Model Fitting

The statistical analysis confirmed the effectiveness of the reduced two-factor interval model in predicting the removal of AYD through the photo-Fenton AOP. The developed model (Equation 6) incorporates the solution's initial concentration (A), contact time (B), and pH (C), along with their interaction terms (AB and AC), to predict the percentage removal of AYD (Y).

The positive coefficients associated with A (initial concentration), B (contact time), and AC (interaction of initial concentration and pH) in Equation 7 indicate that increasing these factors generally leads to higher dye removal. Conversely, the negative coefficient with C (pH) suggests that an increase in pH decreases AYD removal. This aligns with the understanding that acidic pH conditions favor the photo-Fenton process (Nawaz et al. 2020). The negative coefficient of AB (interaction of initial concentration and contact time) implies a complex relationship between these factors, where the effect on removal might depend on the specific values of A and B.

The ANOVA table (Table 3) further strengthens the model's validity. The highly significant model ($P < 0.0001$) indicates a 99.99% chance that the model is reliable. Moreover, the significance ($P < 0.05$) of individual factors (A, B, C, AB, and AC) confirms their influence on AYD removal. The non-significant lack of fit p-value (0.5649) implies that the model adequately fits the data and can be used for prediction within the studied range.

Indeed, the reduced two-factor interval model effectively captures the relationship between the process variables (initial concentration, contact time, and pH) and the photo-Fenton degradation of AYD. The statistical analysis validates the model's accuracy and reliability for predicting the percentage removal of AYD within the investigated range.

Interactive Effects of the Operating Variables

The study investigated the interactive effects of operating parameters on AYD removal using the photo-Fenton AOP. The chosen variables, viz. initial concentration (A), contact time (B), and pH (C) were found to significantly influence the degradation process ($P < 0.05$) according to the developed reduced two-factor interval model.

Figure 2A highlights the interplay between initial AYD concentration and contact time in achieving efficient degradation. It reveals that the combination of a lower initial concentration (25 ppm) with extended contact time (30-50 min) leads to a significant increase in AYD removal, reaching up to 92.7%. This observation aligns with established principles. At lower dye concentrations, •OH radicals, the primary oxidants in photo-Fenton AOP, are more likely to encounter and degrade AYD molecules. Additionally, extending the reaction time allows for continuous generation of •OH, facilitating the breakdown of a larger fraction of the dye molecules present. This explains the observed increase in removal with increasing contact time, particularly at lower initial concentrations.

Figure 2B showcases the interactive effect of initial AYD concentration and pH on degradation. While the figure suggests a maximum removal of 93.7% at the highest concentration (35 ppm) and acidic pH (5), it is crucial to consider the findings from the actual experimental runs. These runs achieved a maximum removal of 95.45% at a different concentration (possibly lower) under the optimized contact time determined by the CCD analysis. This aligns with the study by Lucas and Peres (2006), which emphasized the importance of optimal contact time for maximizing AYD removal.

The trend observed in Figure 2B further supports the findings of Kakodia et al. (2013). The increasing removal with increasing pH up to a certain point (around pH 5) suggests that mildly acidic conditions favor the generating of •OH radicals in the photo-Fenton process. However, excessively high pH levels (beyond the optimum) can lead to the scavenging of •OH radicals by OH- hindering their ability to degrade AYD molecules (Kumari and Kumar 2023), which explains the decrease in removal observed when the pH goes beyond 5.

Categorically, the analysis of interaction terms (AB and AC) in Figure 2 provides valuable insights into the combined effects of operating parameters on AYD removal in the photo-Fenton AOP. Lower initial dye concentration and extended contact time generally promote efficient degradation. Maintaining a mildly acidic pH (around 5) is crucial for optimal generation and activity of hydroxyl radicals. These findings contribute to a comprehensive understanding of the process optimization for effective AYD removal.

Optimization Studies

Using photo-Fenton AOP, the study employed a CCD to identify the optimal operating parameters for maximizing AYD removal. The model predicted an optimal removal of 94.26% at an initial concentration of 35 ppm, contact time of 30 min, and pH of 5.

Validation experiments were conducted under these predicted optimal conditions, achieving an actual removal efficiency of 94.00%, as shown in Table 4. This close agreement between the predicted and actual values (with a mere 0.26% error) demonstrates the model's strong predictive capability. This implies that the developed model equation (Equation 6) can be reliably used to estimate the degradation of AYD within the investigated range of operating parameters.

Moreover, Table 5 shows the efficiency of this study as compared to some related published studies. The results of this study are higher than the degradation of reactive yellow dye (91%) under optimized conditions by Mohammed et al. (2022), which required more complex parameters and a longer irradiation time. Similarly, the 92.24% removal efficiency for acid red 73 reported by Vaez et al. (2012) and the 89% removal for acid yellow 17 by Khan et al. (2018) are slightly lower than this study's performance. Both studies relied on acidic conditions and additional reactants (e.g. Fe^{2+} and H_2O_2), suggesting the method used in this study may offer a simpler and more efficient alternative for dye removal.

The optimization using CCD highlights its effectiveness in identifying favorable conditions for photo-Fenton AOP. This approach can also be valuable for optimizing the degradation of other organic pollutants. The achieved removal efficiency (around 94%) signifies the potential of photo-Fenton AOP for treating wastewater containing AYD. However, further research is needed to evaluate the applicability of this method at larger scales and with more complex wastewater matrices. Moreover, the study underscores the importance of considering initial concentration, contact time, and pH to optimize AYD removal. Maintaining a mildly acidic pH (around 5) appears crucial for maximizing the generation and activity of hydroxyl radicals.

Future studies could explore additional factors that might influence the process, such as the type and concentration of the iron catalyst used in the photo-Fenton reaction. Additionally, investigating the degradation products of AYD could provide valuable insights into the reaction mechanism and potential environmental implications.

Table 4. Optimization and validation of the removal of aniline yellow dye (AYD) using photo-Fenton advanced oxidation processes (AOP).

Experiment	Variables			Response
	AYD Initial concentration (ppm)	Contact time (min)	pH	AYD removal (%)
CCD (Theoretical)	35	30	5	94.26
Validation (Actual)	35	30	5	94.00

Table 5. Comparison of the degradation efficiency in this study and other published studies.

Dye Removed	Optimal Conditions	Percent Removal	Reference
Aniline yellow dye	pH: 5 Initial AYD Concentration: 35 ppm Contact time: 30 min	94.00%	This Study
Reactive yellow dye	pH:6.95294 TiO_2 concentration: 25.5441 mg/L H_2O_2 concentration: 383.676 mg/L RY initial dye conc.: 20.9412 mg/L irradiation time:89.6176 min	91.00%	(Mohammed et al. 2022)
Acid red 73	pH: 3 Initial dye concentration: 25 mg/L H_2O_2 concentration: 0.5 mg/L Anion concentration: 0.69 mg/L	92.24%	(Vaez et al. 2012)
Acid yellow 17	AY 17: 0.06 mM Fe^{2+}: 0.06 mM H_2O_2 :0.9 mM pH: 3.0 reaction time: 60 min	89.00%	(Khan et al. 2018)

Kinetic Modelling

Kinetic experiments were conducted under the optimized conditions (35 ppm initial concentration, pH 5) with varying contact times (3-30 min). The results (Figure 3) were used to evaluate the fit of two kinetic models: pseudo-first-order and pseudo-second-order.

The pseudo-first-order model exhibited a superior fit to the experimental data compared to the pseudo-second-order model. This is evident from the higher R^2 value (0.9625) for the pseudo-first-order model compared to the pseudo-second-order model's R^2 value (0.9510), as indicated in Table 6. This suggests that the removal rate of AYD is primarily dependent on the concentration of •OH radicals present in the solution.

The dominance of the pseudo-first-order model implies that the degradation rate is directly proportional to the concentration of AYD molecules available for reaction with •OH radicals. This finding highlights the crucial role of •OH radicals in the photo-Fenton process. Optimizing process conditions to generate a sufficient concentration of these radicals is essential for efficient AYD removal.

Table 6. Kinetic parameters for aniline yellow dye (AYD) degradation through the photo-Fenton process.

Pseudo-first order		Pseudo-second order	
Variables	**Value**	**Variables**	**Value**
K_1, min^{-1}	0.0369	K_2, mg/L/min	0.0125
C_o, mg/L	4.8625	C_o, mg/L	5.4406
R^2	0.9625	R^2	0.9510

Synthesis of Findings and Implications

This study demonstrated the effectiveness of the photo-Fenton process for aniline yellow dye degradation in aqueous solutions. The process was optimized under varying initial concentration, pH, and contact time conditions, showing a promising removal efficiency of up to 94%. These results are significant in the context of wastewater treatment, particularly for industries where azo dyes such as aniline yellow are prevalent contaminants. Future research should focus on addressing several key areas to enhance the applicability of the photo-Fenton process. First, investigate the formation and toxicity of degradation byproducts which is crucial for understanding the environmental impact of the process. Second, explore methods to enhance the sustainability and cost-effectiveness of the process, like utilizing solar light instead of artificial UV radiation, could significantly reduce energy consumption. Lastly, assess the scalability of the photo-Fenton process for large-scale wastewater treatment facilities is an essential step toward its commercial implementation. These directions will help refine the process and ensure its long-term viability in industrial applications.

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ETHICAL CONSIDERATIONS

This study did not involve humans or animals.

DECLARATION OF COMPETING INTEREST

The authors declare that there is no competing interests to any authors.

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ROLE OF AUTHORS: *EGP Mohammad (EGPM): Conceptualization, Methodology, Investigation, Data Analysis, Writing - Original Draft; MC Mahino (MCM): Conceptualization, Methodology, Investigation, Data Analysis JGM Mayormita (JGM): Conceptualization, Methodology. Investigation; GE Zayas (GEZ): Conceptualization, Methodology, Investigation; SR Anaya (SRA): Conceptualization, Methodology, Investigation; VIF Mabayo (VIFM): Data Analysis, Writing - Original Draft, Writing - Review and Editing, RO Arazo (ROA): Supervision, Project Administration*

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