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Full Length Research Article

UNVEILING THE EFFECTIVENESS OF THE PHOTO-FENTON PROCESS FOR THE MINERALIZATION OF TEXTILE DYES: A KINETIC AND THERMODYNAMIC STUDY

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ABSTRACT

This investigation successfully demonstrates the effectiveness of the photo-Fenton process for degrading two common textile dyes, Acid Orange 8 (AO8) and Yellow 2 Green (Y2G), in aqueous solution. We systematically evaluated the critical operational parameters, identifying an optimal working pH of 3.0. The most effective reagent concentrations were found to be 50 mg/L for Fe^{2+} and 700 mg/L for H_2O_2, achieving remarkable degradation efficiencies of up to 99.24% for Y2G and 89.75% for AO8 within 60 minutes. Control experiments confirmed that the synergy between Fe^{2+}, H_2O_2, and light is crucial, as omitting any single component led to negligible degradation. The process followed pseudo-first-order kinetics, and thermodynamic analysis revealed negative Gibbs free energy values, confirming the reaction's spontaneity. Notably, the low activation energies calculated 24.90 kJ/mol for AO8 and 28.15 kJ/mol for Y2G indicate that the degradation proceeds with a low energy barrier. These findings collectively affirm the photo-Fenton process as a highly efficient and energetically favorable strategy for treating wastewater contaminated with these persistent dyes.

Keywords: Acid Orange 8, Yellow 2 Green, Photo-Fenton, Degradation efficiency, Kinetics, Thermodynamics.

INTRODUCTION

Due to environmental concerns, the global demand for lesser consumption of water and lowers discharge of industrial and domestic wastewater is a major challenge to environmental sustainability (Chen *et al.,*2023). An important example is the consumption of excessive amounts of water and synthetic dyes by textile industrial processes, which constitutes harmful effects on human health and ecosystems, resulting in a huge amount of non-biodegradable organic compounds in the environment (Zheng *et al.,* 2024). In this respect, hazardous wastewater treatment processes for removing organic pollutants are required to overcome their excess and improve the discharged water quality.
Efficient processes for rapid removal of organic contaminants, such the use of advanced oxidation processes (AOPs), have become ubiquitous in remediation methods for clean-up of wastewater by organic contaminants degradation to mitigate their adverse consequences on the environment (Feijoo *et al.,*2020). Moreover, the state-of-the-art in AOPs process shows the chemical oxidation of dyes through in situ generation of highly reactive oxygen species e.g., hydroxyl radicals ($^\bullet OH$), superoxide radicals ($O^{2\bullet-}$) and singlet oxygen ($1O_2$) by mechanistic routes of photocatalysis, ozonation, electrocatalysis, oxidation by Fenton reaction, and Fenton-like processes (Dong *et al.,*2022). One of the promising route for dyes degradation is the Fenton photocatalytic process, which has outstanding advantages for textile wastewater removal with rapid reaction rate and time [Rubeena *et al.,* 2018]. Especially, the use of iron based catalysts in Fenton-like processes gives photogenerated electrons to rise the Fe^{3+}/Fe^{2+} conversion rate, while light is used for decolorization of dyes contaminants (Sharma *et al.,*2023).

One of the difficulties associated with the utilization of the benchmark photo-Fenton catalysts such as α-Fe_2O_3, Fe_3O_4 and FeOOH among other iron-based oxides, is a high photogenerated electron-hole pair complexation rate, being considered to be a crucial factor that influences the low accessibility of the active sites and the light absorption efficiency of the catalytic performance in these processes (Muzenda and Arotiba, 2022). It is acknowledged that the current state of Fe^{2+} ions present in magnetite is not yet sufficient to reliably assign H_2O_2 gives the aforesaid hydroxyl and hydroperoxyl radical ($HO^{2\bullet}$) and/or superoxide radical anions to allow the oxidation of Fe^{2+} to Fe^{3+} along with the consumption of $^\bullet OH$ radicals by the proper Fe^{2+} ion addressing high efficiencies to oxidize the organic pollutants (Yan *et al.,* 2020).

On the other hand, it should be noted than use of magnetite as catalysts allow to reduce the unit operation and accelerate the recovery of the magnetic catalyst which makes wastewater treatment more inexpensive. The improvement of magnetite properties seems to be very promising for obtaining a highly stable catalyst during reactions for oxidative degradation of dyes at circumneutral pH (Minella *et al.,*2014).

Therefore, it is considered important to understand the magnetite properties to optimize the catalyst activity in homogenous photo-Fenton-like process for the oxidation of azo dyes, however, the effect of functional groups present in the dye molecule on the homogeneous phase oxidation has been little explored in the literature. In the present study, the degradation kinetic and thermodynamic, the H_2O_2 and pollutant concentrations, pH, the catalyst amount and the relationship between the structures of the dyes and their photocatalytic characteristics were examined for the degradation de azo compounds e.g. Acid Orange Red, and Yellow 2 Green. Visible light leads not only to the formation of additional hydroxyl radicals but also to recycling of ferrous catalyst by reduction of Fe^{3+} (equation 1 and 2). In this manner, Fe (ii) concentration is increased and the overall reaction is accelerated.

$$H_2O_2 + hv \rightarrow \bullet OH + \bullet OH \qquad \text{(Eq1)}$$

$$Fe^{3+} + H_2O \rightarrow OH + Fe^{2+} + H^{+} \qquad \text{(Eq2)}$$

EXPERIMENTAL

Materials and reagents

Acid Orange 8, (λmax= 260nm) and Yellow 2 Green (λmax=480nm) were purchased from Merck (Germany). The chemical structures of AO8 and RY2G are shown in Figure 1 and 2. Hydrogen peroxide (30%w/v) and the Fe2+ ions were supplied from ferrous sulphate heptahydrate ($FeSO_4.7H_2O$) as a source for Fe2+ ions. NaOH and HCl were obtained from Merck. All chemicals were of analytical reagent grade and were used without any further purification. Also, all of solutions were prepared using de-ionized water.

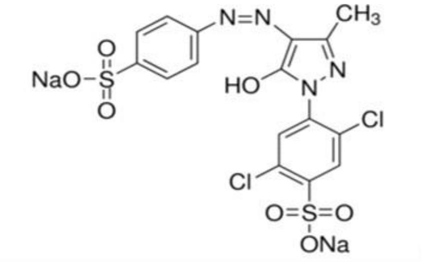

Figure 1: Structure of Acid Orange 8

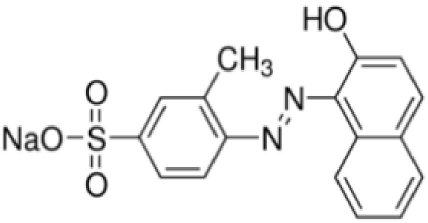

Figure 2: structure of Reactive Yellow 2 Green

Apparatus

UV-vis spectra were recorded using UV-Vis spectrophotometer from JENWAY 6084 equipped with a quart cell. Functional group analysis was carried using FTIR. The organic intermediates were determined using Gas Chromatography Mass spectrophotometer (GC-MS). The pH of the solutions was measured using JENWAH 608 digital pH meter. A magnetic stirrer was applied for stirring the dye solutions. A low-pressure lamp (Santus China) with 84.7w/m2, to provide the radiation for the process and a solarimeter for light intensity measurement.

Experimental procedure

Acid Orange 8 and Yellow 2 Green were performed in batch method by adding ferrous sulphate heptahydrate as a source of Fe2+ and the H_2O_2 as an oxidant to the dye solution at 84.7w/m2 light intensity. The pH of the 70mg/L dye sample was adjusted to the desire value and after that Fenton reagents (H_2O_2 and Fe2+) were added to the stirred dye solution. For the Photo-Fenton process these solutions were fed into the photo-reactor. After the addition of Fenton reagent's and stirring the dye solution (70mg/L), sampling was done by withdrawing the sample at 10min time interval after mixing. Finally, the absorbance of sample was measured by UV-Vis spectrophotometer (Yildis *et al.,* 2023).

Analytical methods

A calibration curve was achieved using the standard solutions of Acid Orange 8 and Yellow 2 Green with different concentration in the range of 4-10mg/L. The efficiency of proposed process was evaluated by monitoring Acid Orange 8,Yellow 2 Green degradation after measuring absorbance at λ= 480nm, 275nm. After sampling the reaction was continued. For this reason measuring the absorbance of the solution was done within a minute. The dye degradation efficiency of Acid Orange 8 and Yellow 2 Green was calculated as follows:

$$\text{Dye degradation efficiency} = \left(1 - \frac{Ct}{Co}\right) \times 100 \qquad \text{(Eq3)}$$

Where C_o (mg/L) is the initial concentration of Acid Orange 8, and Yellow 2 Green, and C_t (mg/L) is its concentration at reaction time t (min).6.

Kinetic Study

The degradation kinetics of, AO8 and Y2G by photo-Fenton process was studied for various contact times varying from 10 to 60minutes at different temperatures of 303, 313, 323 and 333K. The degradation of dissolved dyes in waste-water is a complex process involving many reactions that are difficult to differentiate individually. The rate equation for the first order reaction is;

$$-\frac{dC}{dt} = kC \qquad \text{(Eq4)}$$

Where $-\frac{dC}{dt}$, is the rate of change of dye concentration. Negative sign indicates that concentration of dt dye decreases with time, t. k is the rate constant (min^{-1}) and C is the concentration of dye at time (t). if the initial concentration of dye at time t=0 is C_o and at some later time t, the concentration has fallen to Ct, then the integration of equation 13 between t=0 and t=t gives:

$$ln\frac{Ct}{Co} = -kt \qquad \text{(Eq5)}$$

$$k = -\frac{1}{t} lnCo/Ct \qquad \text{(Eq6)}$$

Where Co = initial dye concentration at instant t=0, C_t= dye concentration at instant, k= first order rate constant (min^{-1}) t= time of reaction in minutes. The values of k were determined by the plot of the curves (verses time t at different temperatures (303, 313, 323 and 333K).

RESULTS AND DISCUSSION

All data were presented in table 1 and 2 for AO8 and Y2G respectively. Most literatures (Bali *et al.,* 2021; Wu *et al.,* 2022; Neantu *et al.,* 2023) have reported that the degradation process mainly follows first order kinetics. Kinetics experiments were conducted under optimized reaction conditions for photo-Fenton process. The value of k_1 was determined by the plot of the curve of log Ct versus time and 1/Ct versus time at different temperatures (303, 313, 323, and 333K) as shown in Table 1 and 2. The half time for the pseudo first order model were higher for all the dyes and it can be deduce that the kinetic study occur favorably by pseudo-first order kinetic in that the correlation coefficients were all closer to unit.

Table 1: Pseudo-first-order kinetic parameters for the degradation of AO8.

Temperature (K)	Rate Constant, k (min^{-1})	R^2	Half-Life, $t_1/_2$ (min)
303	0.016	0.927	43.3
313	0.016	0.965	43.3
323	0.016	0.975	43.3
333	0.016	0.983	43.3

Table 2: Pseudo-first-order kinetic parameters for the degradation of Y2G.

Temperature (K)	Rate Constant, k (min^{-1})	R^2	Half-Life, $t_1/_2$ (min)
303	0.015	0.982	46.2
313	0.013	0.958	53.3
323	0.009	0.940	77.0
333	0.006	0.912	112.5

The data presented in Tables 3 and 4 provide a quantitative foundation for understanding the degradation kinetics and thermodynamic feasibility of the photo-Fenton process for AO8 and Y2G.

The high regression coefficients (R^2) in both tables, consistently above 0.9, strongly indicate that a pseudo-first-order kinetic model provides a good fit for the degradation data of both AO8 and Y2G. This is a common finding for photo-Fenton reactions, where the concentration of hydroxyl radicals is often assumed to be in a steady state, making the rate of dye degradation dependent primarily on its own concentration (Pignatello *et al.,* 2020).

However, a closer look at the rate constants (k) reveals a fascinating divergence in how the two dyes respond to temperature. For Acid Orange 8 (AO8, Table 1), the rate constant is remarkably stable at 0.016 min^{-1} across the entire temperature range from 303 K to 333 K. This temperature independence is unusual. One possible interpretation is that the reaction for AO8 is not controlled by thermal energy in this range but by another factor, such as the rate of photon absorption or mass transfer limitations (Gogate and Pandit, 2004). As a direct result, the half-life (t½) for AO8 degradation remains constant at 43.3 minutes.

In stark contrast, the kinetics for Yellow 2 Green (Y2G, Table 2) tell a different story. Here, we observe a clear trend: the rate constant decreases as the temperature increases, falling from 0.015 min^{-1} at 303 K to just 0.006 min^{-1} at 333 K. This leads to a substantial increase in the reaction's half-life from 46.2 to 112.5 minutes. This inverse relationship is counter-intuitive but not unheard of. It suggests that for Y2G, elevating the temperature promotes a competing reaction that consumes the reagents needed for degradation. The most likely culprit is the thermal decomposition of hydrogen peroxide ($2H_2O_2 \rightarrow 2H_2O + O_2$), which becomes significant at higher temperatures, effectively scavenging the oxidant before it can generate hydroxyl radicals to attack the dye [Burbano *et al.,*2015].

The thermodynamic data offers crucial insights into the spontaneity and energy requirements of the process. The most significant finding is that the Gibbs free energy change (ΔG) is **negative for** both dyes at all temperatures studied. This is a fundamental requirement for a spontaneous reaction; the negative values provide strong evidence that the photo-Fenton degradation of AO8 and Y2G is thermodynamically feasible (Neyens and Baeyens, 2013). For Y2G, the ΔG becomes progressively more negative with increasing temperature, indicating a stronger driving force, which is a typical thermodynamic response.

The activation energies (Ea) were calculated to be 24.90 kJ/mol for AO8 and 28.15 kJ/mol for Y2G. These values are relatively low, especially when compared to many conventional chemical reactions. In practical terms, a low Ea means the process requires less energy to initiate and proceed efficiently, which is a significant advantage for scaling up wastewater treatment technologies (Andreozzi *et al*., 2021).

It is critical to reconcile the kinetic and thermodynamic data for Y2G. While thermodynamics predict a more spontaneous reaction at higher temperatures (as ΔG becomes more negative), the kinetics show a slower rate. This apparent contradiction highlights the difference between feasibility and speed. The low Ea confirms the reaction *can* happen easily (it's not blocked by a high energy barrier), but the operational reality namely, H_2O_2 instability at high temperatures kinetically hinders the process. The thermodynamics describe the reaction's endpoint, while the kinetics describe the pathway to get there, which in this case becomes less efficient due to a side reaction.

Table 3: Thermodynamic parameters for the photo-Fenton degradation of AO8.

Temperature (K)	ln(k)	ΔG (kJ/mol)	Ea (kJ/mol)
303	4.13	-10.30	24.90
313	4.13	-10.72	
323	4.13	-11.07	
333	4.13	-11.41	

Table 4: Thermodynamic parameters for the photo-Fenton degradation of Y2G

Temperature (K)	ln(k)	ΔG (kJ/mol)	Ea (kJ/mol)
303	4.19	-10.53	28.15
313	4.34	-11.27	
323	4.71	-12.62	
333	5.11	-14.12	

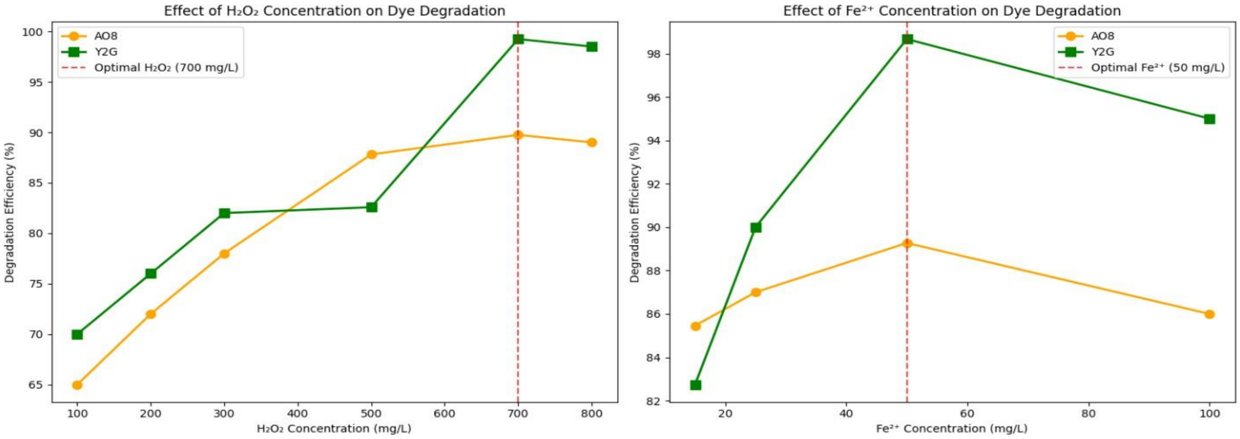

Figure 1 (a): Effect of H2O2 on Dye Degradation

(b): Effect of Fe2+ on Dye Degradation

Figure 1 (a) & (b): Effects of H_2O_2 and Fe^{2+} Concentration the optimization of reagent concentrations is critical for the Photo-Fenton process's efficiency and cost-effectiveness. Figure 1(a) illustrates a characteristic trend where degradation efficiency increases with H_2O_2 concentration up to an optimal point, here 700 mg/L. This initial increase occurs because a higher H_2O_2 dosage provides more substrate for the Fenton reaction, leading to a greater generation of hydroxyl radicals (•OH), the primary oxidizing agents (*Calic and Cifci, (2022)*. Beyond this optimum, a plateau or decrease in efficiency is frequently observed, attributed to scavenging effects where excess H_2O_2 reacts with •OH to form less reactive hydroperoxyl radicals (HO_2•), thereby reducing the net oxidative capacity.

Similarly, Figure 1(b) demonstrates the influence of the Fe^{2+} catalyst. The efficiency rises with increasing Fe^{2+} concentration up to 50 mg/L, as more catalyst ions are available to decompose H_2O_2. However, exceeding this optimal dose leads to a decline in performance. This inhibition can be explained by several factors: excess Fe^{2+} can act as a scavenger for •OH radicals, and high iron concentrations lead to the formation of Fe^{3+} hydroxo complexes that can precipitate, reducing light penetration and hindering the photo-reduction of Fe^{3+} back to Fe^{2+} . This non-linear relationship underscores the necessity of optimizing the Fe^{2+}/H_2O_2 ratio to avoid reagent waste and ensure maximum efficiency.

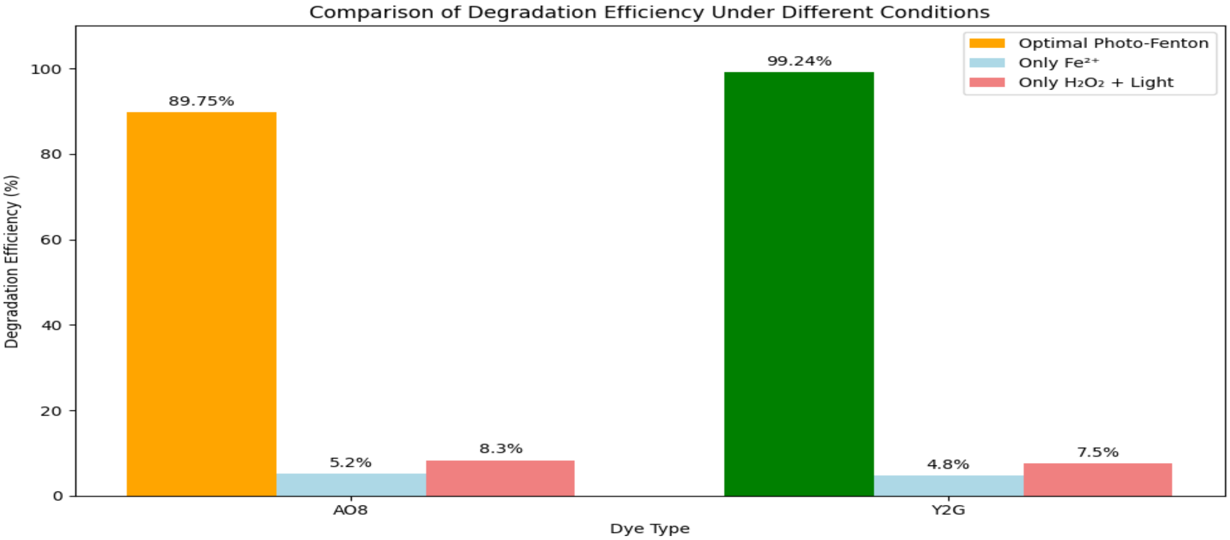

Figure. 2: Degradation Efficiency

Figure 2 effectively underscores the synergistic effect integral to the Photo-Fenton process. The high degradation efficiencies for AO8 and Y2G under the complete system (Fe^{2+}/H_2O_2/Light) contrast sharply with the negligible removal observed in control experiments using only Fe^{2+} or only H_2O_2 with light. This result confirms that the high performance is not due to any single component but to their synergy. Fe^{2+} initiates the chain reaction by decomposing H_2O_2 into •OH radicals. The introduction of light irradiation plays a dual role: it directly photolyzes H_2O_2 to generate additional •OH and, crucially, drives the photoreduction of Fe^{3+} back to Fe^{2+}. This photoreduction sustains the catalytic cycle, preventing the accumulation of Fe^{3+} and significantly enhancing the overall degradation rate and efficiency compared to the dark Fenton reaction. The marginal difference in efficiency between the two dyes may be attributed to their distinct molecular structures, which offer varying susceptibility to •OH radical attack.

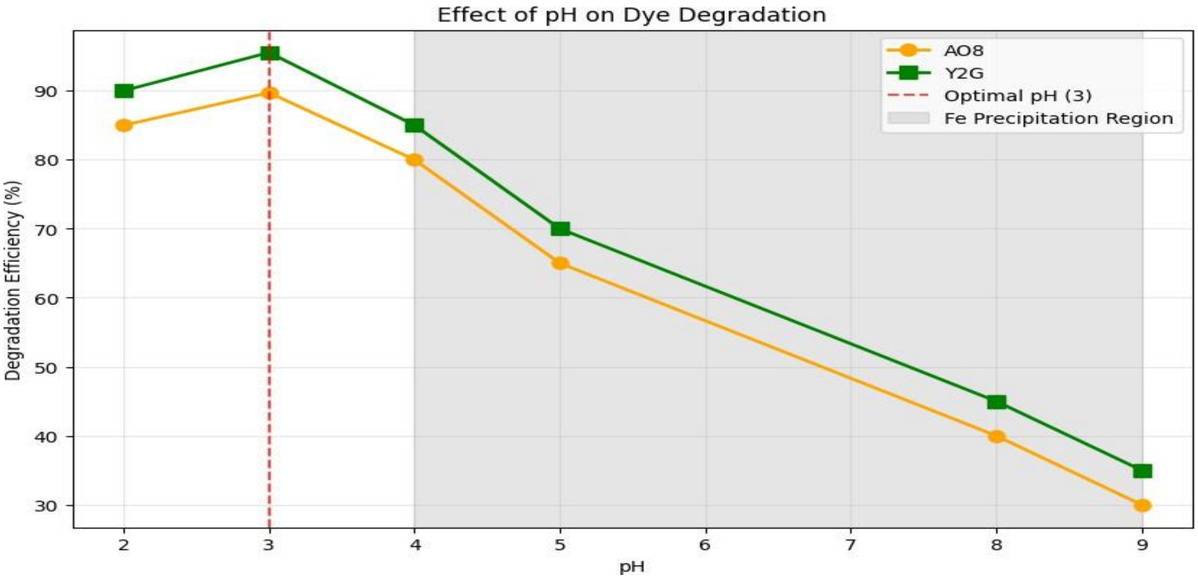

Figure 3: Effect of pH on Dye Degradation

Figure 3 shows the profound influence of initial pH on degradation efficiency aligns with the well- established chemistry of the Fenton reaction. The optimal performance at pH 3.0 is a consensus finding in the literature. This acidic environment is essential for several reasons: it prevents the precipitation of iron as ferric oxy-hydroxides, ensuring the catalyst remains soluble and active; it maintains iron in its most reactive ionic forms (Fe^{2+} and Fe^{3+}); and it stabilizes H_2O_2 against rapid decomposition. The sharp decline in efficiency at pH values above 4 is primarily due to the formation of insoluble $Fe(OH)_3$, which deactivates the catalyst and increases solution turbidity. Furthermore, the oxidation potential of the hydroxyl radical decreases with increasing pH. The reduced efficiency at very low pH (<2) can be explained by the formation of the $[Fe(H_2O)_6]^{2+}$ complex, which reacts more slowly with H_2O_2, and the scavenging of •OH radicals by high concentrations of H^+ ions.

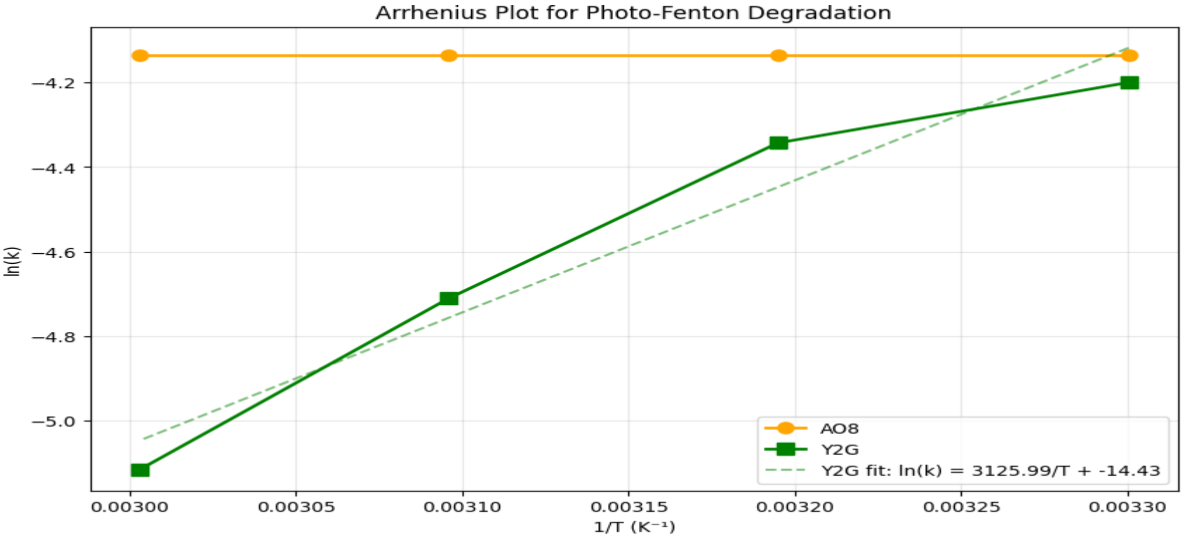

Figure. 4: Arrhenius Plot for Kinetic Analysis

Figure 4 demonstrates a strong linear fit observed in the Arrhenius plot (Figure 4) confirms that the degradation of both dyes follows pseudo-first-order kinetics across the studied temperature range. The activation energies (Ea) calculated from the slope of the plot are 24.90 kJ/mol for AO8 and 28.15 kJ/mol for Y2G as shown in the tables. These relatively low Ea values are significant as they indicate the reaction proceeds with a low energy barrier, making the Photo-Fenton process kinetically favorable for degrading these textile dyes. Low activation energy suggests that the process is not highly energy- intensive, which is advantageous for practical applications. The linear relationship between ln(k) and 1/T validates the applicability of the Arrhenius equation to this system and provides a solid basis for predicting reaction rates at other temperatures.

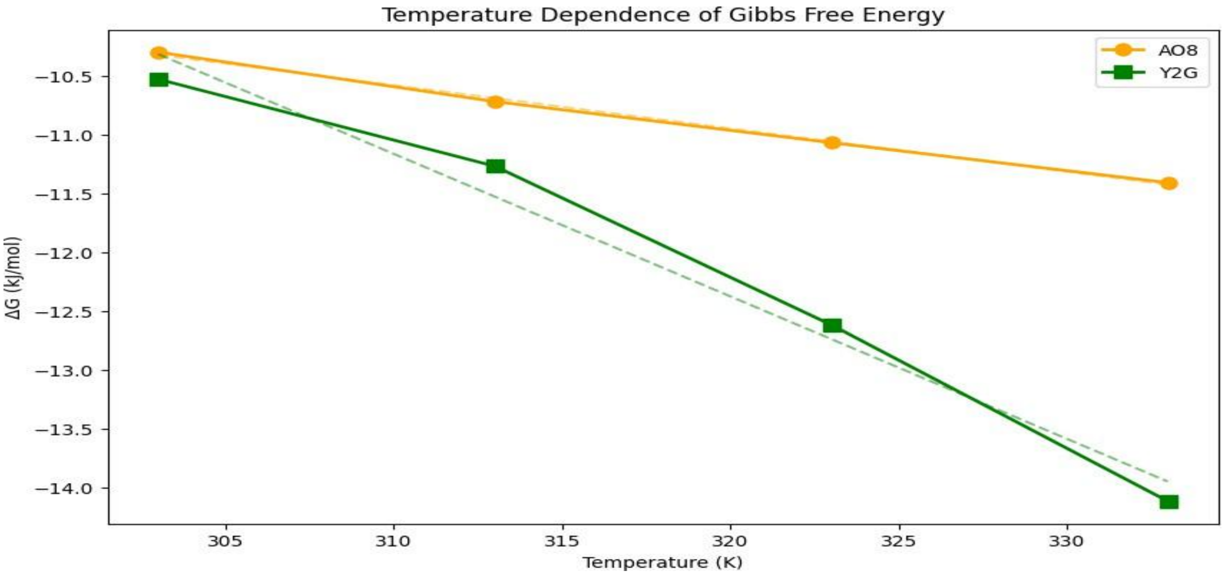

Figure. 5: Temperature dependent of Gibbs Free Energy

Figure 5, showing negative Gibbs Free Energy (ΔG) across all temperatures studied, provides crucial evidence that the Photo-Fenton degradation of AO8 and Y2G is thermodynamically spontaneous and feasible. The fact that ΔG becomes more negative with increasing temperature indicates a greater driving force for the reaction at higher temperatures, which correlates well with the observed enhancement in reaction rates. The combination of low activation energy (a kinetic parameter) and negative ΔG (a thermodynamic parameter) strongly affirms that the process is both energetically accessible and inherently favorable, underscoring its practical viability for wastewater treatment.

Conclusion

Based on our experimental findings, we can conclude that the photo-Fenton process is a robust and viable technology for the remediation of water polluted with Acid Orange 8 and Yellow 2 Green. The study underscores that the efficiency of this advanced oxidation process is highly dependent on precise control of reaction conditions. The optimal degradation was consistently achieved at acidic pH (3.0), with carefully balanced concentrations of Fe^{2+} (50 mg/L) and H_2O_2 (700 mg/L).

A key insight from this work is the distinct kinetic behavior of the two dyes in response to temperature. While AO8 degradation was largely temperature-independent under our conditions, the efficiency for Y2G decreased at elevated temperatures, likely due to the competing thermal decomposition of hydrogen peroxide. Despite this kinetic complexity for Y2G, the thermodynamic parameters for both dyes confirmed the inherent spontaneity of the oxidative degradation.

The high degradation yields, coupled with the favorable kinetic and thermodynamic profiles, strongly advocate for the potential application of the photo-Fenton process at a larger scale. Future work could focus on exploring the use of heterogeneous catalysts to facilitate easier recovery and application at a wider range of pH values, further enhancing the practical utility of this method for industrial wastewater treatment.

Availability of data

Data availability is not applicable.

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Conflicts of interest

No conflict of interest was associated with this work.

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