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# Decolourization of Synthetic Wastewater Related to the Textile Industry Using Photo-Fenton

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**Article**

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**ABSTRACT**

*The artisanal textile industry plays a vital role in the economy by providing a wide variety of products. However, this sector generates large quantities of wastewater containing a diversity of organic compounds, including dyes that, when released into the environment, contribute significantly to water pollution. In this research, samples of synthetic wastewater containing green aniline were subjected to decolourization using the photo-Fenton process. Synthetic textile wastewater was prepared at a concentration of 40 mg/L, and a calibration curve was established. During the treatment, ferrous sulfate (at concentrations of 50 and 150 mg/L) and $H_2O_2$ (at concentrations of 0.5 and 1.0 g/L) were added to 1 L of the synthetic effluent in a stirring medium (at 1000 rpm) and exposed to solar radiation for 1, 2, or 3 hours, while maintaining a pH of 3. To quantify the degree of decolourization, absorbance was measured at the beginning and end of the treatment using a colourimeter. The optimal conditions for $Fe^{2+}$ concentration, H2O2 concentration, and treatment time, with an initial concentration of 40 mg/L, were found to be 150 mg/L, 1.0 g/L, and 3 hours, respectively. Under these conditions, the percentage of decolourization reached 97.546%. The study demonstrated that the concentration of $Fe^{2+}$ and treatment time, as well as the interaction between $Fe^{2+}$ concentration, $H_2O_2$ concentration, and treatment time, significantly influenced the degree of decolourization. Conversely, the concentration of $H_2O_2$ and other interactions did not exhibit significant influence.*

**KEYWORDS**

*decolorization, synthetic wastewater, textile industry, photo-Fenton*

## INTRODUCTION

The global textile industry has experienced exponential growth in recent decades, emerging as one of the fastest-expanding manufacturing sectors [1]. This surge is primarily fueled by a sustained increase in demand for textile goods, driven by factors such as demographic growth and consumption patterns. While this growth brings evident economic and social benefits, it also leads to significant environmental damage that cannot be ignored [2].

Moreover, as one of the most water and chemical-intensive sectors, the textile industry produces dyes, which account for approximately 8% to 20% of the total contaminant load, along with auxiliary chemicals, all dissolved in large volumes of water [3–5]. This poses a severe threat to delicate aquatic ecosystems globally, as both dyes and chemical additives exhibit notable stability and persistence in the environment [6].

On the other hand, wastewater from textile dyes poses significant challenges in decomposition due to its high biochemical oxygen demand (BOD), substantial concentration of dissolved solids, and stable chemical structure [7]. Specifically, nitrogen dyes, such as aniline, possess a complex aromatic structure, limiting the effectiveness of conventional biological treatment methods [8]. Thus, the urgent need to explore and develop treatment alternatives capable of bleaching textile wastewater is emphasized, as also highlighted in various studies [9,10].

Given this context, new technological alternatives, such as advanced oxidation processes (AOP), are imperative, as these unconventional methods are highly effective in degrading toxic or persistent pollutants, thanks to the generation of hydroxyl radicals (•OH) [11]. The efficiency of AOPs can be enhanced with suitable catalysts and the implementation of hybrid processes, such as UV/photocatalysis, UV-Fenton, and photo-Fenton [12]. These strategies offer promising prospects to enhance the efficiency of wastewater treatments, particularly in the textile industry.

The photo-Fenton method involves the activation of hydrogen peroxide in the presence of iron and ultraviolet light, generating highly reactive hydroxyl radicals. Its efficacy has been demonstrated in treating textile wastewater, showcasing its ability to decompose resistant dyes and persistent organic compounds [13]. The key lies in its efficiency in triggering advanced oxidation reactions, achieving a more comprehensive degradation of contaminants, which can further be optimized through careful selection of catalysts and manipulation of reaction conditions [14].

In the specific context of the textile industry, photo-Fenton emerges as a promising tool to address the complexity of dyes and chemicals present in effluents, as its application not only aims for efficient decolourization but also contributes to reducing the total pollutant load, thereby mitigating the environmental impact associated with textile waste [15]. This approach offers an innovative and sustainable solution to improve the efficiency of wastewater treatments, aligning with the necessity for more environmentally responsible practices. In this manner, the following research aims to achieve the decolourization of wastewater from the artisanal textile industry through the photo-Fenton method, based on its capacity, thereby contributing to the evolving understanding and development of sustainable solutions in wastewater treatment.

## MATERIALS AND METHODS

In the experimental setup and fading test procedure section (Figure 1), the following equipment was utilized: a colourimeter, Elettronica Veneta brand, model EVS-BIO-05/EV; a datalogger, Elettronica

Veneta brand, model EV2010/EV; an analytical balance, Sartorius brand, model A2214, with a capacity of 220 g and a precision of ± 0.1 mg; a pH meter, SI Analytics brand, model HandyLab 600; a stopwatch; a magnetic stirrer with a digital heating plate, JKI brand, model MI0102005; and a Lenovo personal computer.

Initially, the preparation of synthetic textile wastewater was conducted by dissolving 40 mg of green aniline in 1 L of water to achieve a concentration of 40 mg/L. The solution was vigorously shaken until homogenized. It is worth noting that this concentration was determined based on various studies, considering the availability of different types of aniline in the market. For instance, [16], [17], and [18] investigated low levels ranging from 1.83 to 30 mg/L, while [18], [19], and [20] examined higher concentrations ranging from 150 to 280 mg/L. The results of these studies showed inconsistency regarding the optimal initial concentration for achieving greater degradation.

Subsequently, to determine the concentrations of $Fe^{2+}$, $H_2O_2$, and treatment time required for decolourization of the residual water, the sample beaker was placed on a magnetic stirrer, exposed to solar rays, and stirred at 1000 rpm. Distilled water was added until the volume reached 1 L (refer to Figure 1).

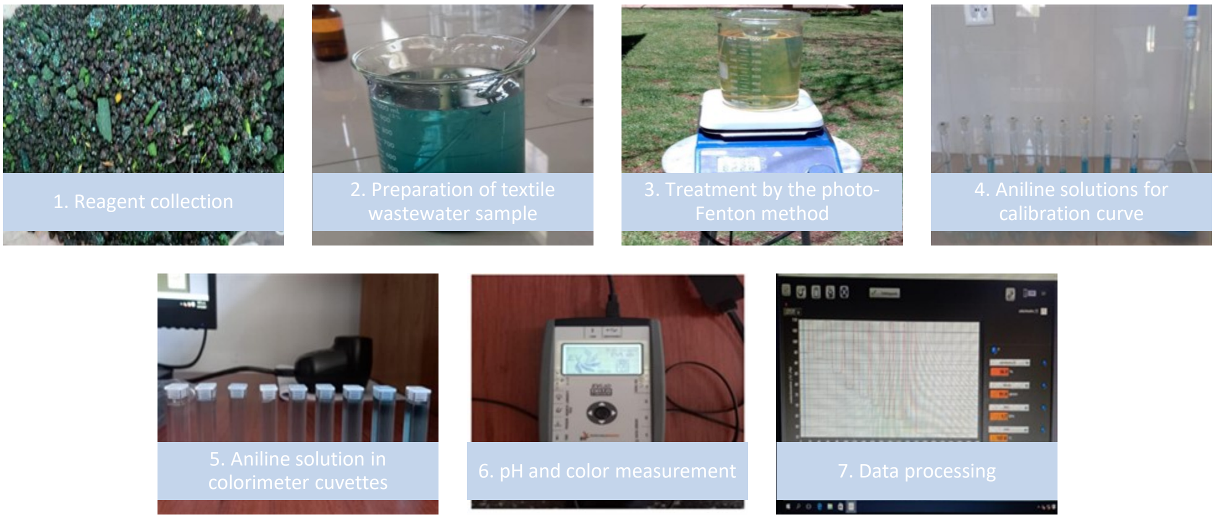


Figure 1. Experimental setup and decolourization testing procedure

The pH level maintained throughout the experiment was 3, adjusted using drops of $H_2SO_4$. Subsequently, ferrous sulfate (at concentrations of 50 and 150 mg/L) was weighed, and the volume of the $H_2O_2$ solution (at concentrations of 0.5 and 1.0 g/L) was measured and added to the beaker according to the experimental design matrix. Samples were then collected after 1, 2, or 3 hours to measure the absorbance using a colourimeter for each sample, as outlined in Table 1.

Table 1. Analytical techniques for collecting specific data

| Variables or parameter | Specific techniques |
|---|---|
| Dye concentration | Spectroscopy |
| pH | Potentiometry |
| Weight | Gravimetry |

Finally, to determine the concentration of aniline after the photo-Fenton treatment, it was necessary to establish a calibration curve. To accomplish this, solutions at various concentrations (0, 4, 8, 12, 16, 20, 24, 28, 32, and 36 mg/L) were prepared from the aniline matrix solution in 100 mL vials. This involved measuring 0, 10, 20, 30, 40, 50, 60, 70, 80, and 90 mL, respectively, to fill the vials to 100 mL. Absorbance readings were then taken using a colourimeter for all solutions, including distilled water.

## RESULTS AND DISCUSSION

In this research, a randomized block factorial design was employed due to the consideration of three study variables. Two variables were assessed at two levels each, while one variable had three levels, resulting in a 2×2×3 design as depicted in Table 2.

Table 2. Design matrix

| N° | Fe2+ concentration (mg/L) | $H_2O_2$ concentration (g/L) | Treatment time (h) |
|---|---|---|---|
| 1 | 50 | 0.5 | 1 |
| 2 | 50 | 0.5 | 2 |
| 3 | 50 | 0.5 | 3 |
| 4 | 50 | 1.0 | 1 |
| 5 | 50 | 1.0 | 2 |
| 6 | 50 | 1.0 | 3 |
| 7 | 150 | 0.5 | 1 |
| 8 | 150 | 0.5 | 2 |
| 9 | 150 | 0.5 | 3 |
| 10 | 150 | 1.0 | 1 |
| 11 | 150 | 1.0 | 2 |
| 12 | 150 | 1.0 | 3 |

For the hypothesis test, an analysis of variance (ANOVA) was conducted to establish the relationship between the concentrations of $Fe^{2+}$, $H_2O_2$, and treatment time, as presented in Tables 5, 6, and 7, respectively.

Table 5. Normality tests

| | | Kolmogorov-Smirnova | | | Shapiro-Wilk | | |
|---|---|---|---|---|---|---|---|
| | | Statistic | gl | Sig. | Statistic | gl | Sig. |
| Decolourization of textile wastewater (%) | $Fe^{2+}$ concentration | | | | | | |
| | 50 mg/L | 0.185 | 12 | 0.200* | 0.883 | 12 | 0.095 |
| | 150 mg/L | 0.206 | 12 | 0.168 | 0.862 | 12 | 0.052 |
| | Concentration $H_2O_2$ | | | | | | |
| | 0.5 g/L | 0.160 | 12 | 0.200* | 0.929 | 12 | 0.372 |
| | 1.0 g/L | 0.165 | 12 | 0.200* | 0.931 | 12 | 0.389 |
| | Treatment time | | | | | | |
| | 1 h | 0.174 | 8 | 0.200* | 0.937 | 8 | 0.581 |
| | 2 h | 0.171 | 8 | 0.200* | 0.889 | 8 | 0.231 |
| | 3 h | 0.248 | 8 | 0.160 | 0.867 | 8 | 0.140 |

Since the asymptotic significance for the concentration of $Fe^{2+}$, $H_2O_2$, and treatment time is greater than 0.05, we cannot reject the null hypothesis (indicating that interaction effects are not present). Therefore, we conclude that the independent variables analyzed follow a normal distribution.

Table 6. Homogeneity of variance test

| | | Decolourization of textile wastewater | | | |
|---|---|---|---|---|---|
| | | Based on the mean | It is based on the median | It is based on the median and with adjusted gl | It is based on the trimmed trimmed mean |
| Concentration $Fe^{2+}$ | Statistic Levene's | 0.008 | 0.007 | 0.007 | 0.008 |
| | df1 | 1 | 1 | 1 | 1 |
| | df2 | 14 | 14 | 13.145 | 14 |
| | Sig. | 0.931 | 0.936 | 0.936 | 0.932 |
| Concentration $H_2O_2$ | Statistic Levene's | 0.008 | 0.007 | 0,007 | 0.008 |
| | df1 | 1 | 1 | 1 | 1 |
| | df2 | 14 | 14 | 13,145 | 14 |
| | Sig. | 0.931 | 0.936 | 0.936 | 0.932 |
| Treatment time | Statistic Levene's | 0.008 | 0.007 | 0.007 | 0.008 |
| | df1 | 1 | 1 | 1 | 1 |
| | df2 | 14 | 14 | 13.145 | 14 |
| | Sig. | 0.931 | 0.936 | 0.936 | 0.932 |

For the analysis of homogeneity of variances, the asymptotic significance for the concentrations of $Fe^{2+}$, $H_2O_2$, and treatment time is greater than 0.05. Therefore, it is concluded that the independent variables analyzed exhibit homogeneity of variances.

Table 7. Analysis of variance

| Origin | Sum of squares | gl | Quadratic mean | F | Sig. |
|---|---|---|---|---|---|
| Corrected model | 14723.084[a] | 11 | 1338.462 | 265.30 | 0.000 |
| Intersection | 90547.575 | 1 | 90547.575 | 17947.87 | 0.000 |
| Concentration $Fe^{2+}$ | 810.263 | 1 | 810.263 | 160.61 | 0.000 |
| Concentration $H_2O_2$ | 16.733 | 1 | 16.733 | 3.32 | 0.094 |
| Treatment time | 13805.174 | 2 | 6902.587 | 1368.20 | 0.000 |
| Concentración de $Fe^{2+}$ × Concentration $H_2O_2$ | 11.147 | 1 | 11.147 | 2.21 | 0.163 |
| Concentration $Fe^{2+}$ × Treatment time | 6.074 | 2 | 3.037 | 0.60 | 0.563 |
| Concentration $H_2O_2$ × Treatment time | 14.480 | 2 | 7.240 | 1.43 | 0.276 |
| Concentration $Fe^{2+}$ × Concentration $H_2O_2$ × Treatment time | 59.212 | 2 | 29.606 | 5.87 | 0.017 |
| Error | 60.540 | 12 | 5.045 | | |
| Total | 105331.199 | 24 | | | |
| Total corrected | 14783.624 | 23 | | | |

a. $R^2$ =0.996 (Adjusted $R^2$ =0.992)

The asymptotic significance is less than 0.05 for the variables concentration of $Fe^{2+}$ and treatment time. Consequently, the interaction of $Fe^{2+}$ concentration × $H_2O_2$ concentration × treatment time exhibits significant differences, indicating their influential role in the decolourization process of artisanal textile wastewater using the photo-Fenton method. Conversely, since the significance is greater than 0.05, the variable concentration of $H_2O_2$ and the interaction of $Fe^{2+}$ concentration × $H_2O_2$ concentration, $Fe^{2+}$ concentration × treatment time, and $H_2O_2$ concentration × treatment time do not influence the decolourization of artisanal textile wastewater through the photo-Fenton process.

**Calibration curve**

To ascertain the final concentration of green aniline in the various experiments, a calibration curve was plotted (Figure 2). This curve correlates the measured absorbance obtained from the colourimeter with the initial concentration of aniline.

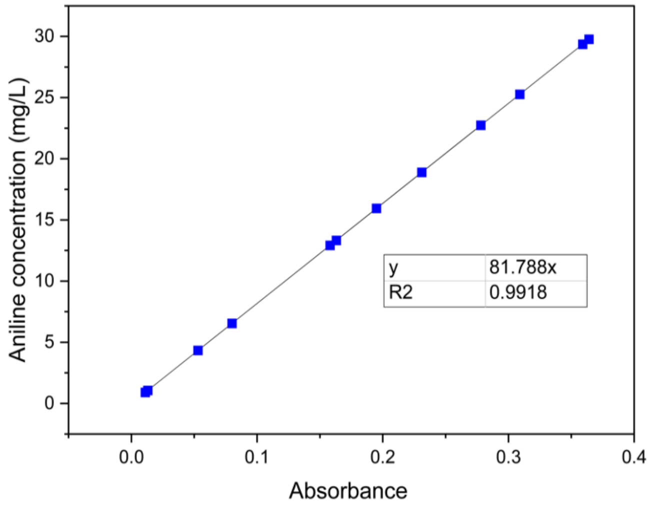


Figure 2. Calibration curve

The calibration curve plays a crucial role in determining the concentration of green aniline in experiments, as it establishes a quantitative relationship between the absorbance measured in the colourimeter and the known concentration of the aniline. Without this curve, assigning concentration values to absorbance readings would be impossible, making it difficult or even impossible to determine the concentration of aniline in the samples.

**Aniline concentration after photo-Fenton treatment**

Table 3 illustrates that when the initial concentration of the pollutant is relatively low, a higher degradation occurs, attributed to the increased production of •OH radicals. Conversely, in situations with higher concentrations, inhibition of the $H_2O_2$ reaction may occur, resulting in lower degradation, as indicated by [17]. Furthermore, the concentration of $Fe^{2+}$ (photocatalyst) significantly influences the degradation rate of the dye (pollutant). It is evident that at a concentration of 150 mg/L, the decolourization surpasses that obtained at 50 mg/L. However, it is emphasized that determining the optimal condition experimentally is crucial due to the variability in wastewater and contaminant characteristics.

Nevertheless, it is important to note that excessively high levels of $Fe^{2+}$ can hinder aniline degradation, as observed by [17]. Additionally, [1] mentions that dilute $Fe^{2+}$ may suppress aniline degradation. Moreover, [2] points out that $H_2O_2$ accelerates the corrosion of Fe(O) and generates nanoscale iron hydroxides. Consequently, aniline is initially degraded with OH, followed by the removal of degradation products by iron hydroxides through adsorption and coprecipitation. On a different note, [3] utilized an iron-carbon internal circulation microelectrolysis reactor to almost completely degrade aniline. They investigated the effects and optimization of reaction time, initial pH, aeration rate, and Fe/C ratio on the aniline removal rate, achieving a reduction to 0.26 mg/L under optimal conditions, with a Fe/C ratio of 1:2.

Table 3. Final concentration depending on the concentration of Fe2+ and H2O2 and treatment time

| N° | $Fe^{2+}$ concentration (mg/L) | $H_2O_2$ concentration (g/L) | Treatment time (h) | Absorbance | | Aniline concentration (mg/L) | |
|---|---|---|---|---|---|---|---|
| | | | | I | II | I | II |
| 1 | 50 | 0.5 | 1 | 0.359 | 0.347 | 29.362 | 28.380 |
| 2 | 50 | 0.5 | 2 | 0.195 | 0.231 | 15.949 | 18.893 |
| 3 | 50 | 0.5 | 3 | 0.080 | 0.095 | 6.543 | 7.770 |
| 4 | 50 | 1.0 | 1 | 0.364 | 0.377 | 29.771 | 30.834 |
| 5 | 50 | 1.0 | 2 | 0.231 | 0.213 | 18.893 | 17.421 |
| 6 | 50 | 1.0 | 3 | 0.053 | 0.060 | 4.335 | 4.907 |
| 7 | 150 | 0.5 | 1 | 0.309 | 0.321 | 25.272 | 26.254 |
| 8 | 150 | 0.5 | 2 | 0.163 | 0.179 | 13.331 | 14.640 |
| 9 | 150 | 0.5 | 3 | 0.013 | 0.021 | 1.063 | 1.718 |
| 10 | 150 | 1.0 | 1 | 0.278 | 0.289 | 22.737 | 23.637 |
| 11 | 150 | 1.0 | 2 | 0.158 | 0.168 | 12.923 | 13.740 |
| 12 | 150 | 1.0 | 3 | 0.011 | 0.013 | 0.900 | 1.063 |

**Percentage of decolourization after photo-Fenton treatment**

Similarly, concentration evaluations of the wastewater decolourization percentage estimates were conducted and are presented in Table 4. Thus $Fe^{2+}$ concentrations of 50 and 150 mg/L yielded decolourization rates of 55.612% ± 7.347% and 67.234% ± 7.204%, respectively. It is noteworthy that these concentrations align with similar results obtained by [22] and [23], who investigated $Fe^{2+}$ levels of 100 and 224 mg/L in their respective studies. This concentration directly correlates with the characteristics and concentration of the pollutant, influencing the generation of •OH radicals and, consequently, the efficiency of the process.

Regarding the treatment duration of 1, 2, and 3 hours, the decolourization rates were 32.422% ± 2.649%, 60.691% ± 2.157%, and 91.1567% ± 2.401%, respectively.

Table 4. Percentage of decolourization

| N° | $Fe^{2+}$ concentration (mg/L) | $H_2O_2$ concentration (g/L) | Treatment time (h) | Textile residual water decolourization (%) | | |
|---|---|---|---|---|---|---|
| | | | | I | II | Average |
| 1 | 50 | 0.5 | 1 | 26.595 | 29.049 | 27.822 |
| 2 | 50 | 0.5 | 2 | 60.128 | 52.767 | 56.448 |
| 3 | 50 | 0.5 | 3 | 83.642 | 80.575 | 82.109 |

| N° | $Fe^{2+}$ concentration (mg/L) | $H_2O_2$ concentration (g/L) | Treatment time (h) | Textile residual water decolourization (%) | | |
|---|---|---|---|---|---|---|
| | | | | I | II | Average |
| 4 | 50 | 1.0 | 1 | 25.573 | 22.915 | 24.244 |
| 5 | 50 | 1.0 | 2 | 52.767 | 56.448 | 54.608 |
| 6 | 50 | 1.0 | 3 | 89.163 | 87.732 | 88.447 |
| 7 | 150 | 0.5 | 1 | 36.819 | 34.365 | 35.592 |
| 8 | 150 | 0.5 | 2 | 66.671 | 63.400 | 65.036 |
| 9 | 150 | 0.5 | 3 | 97.342 | 95.706 | 96.524 |
| 10 | 150 | 1.0 | 1 | 43.157 | 40.908 | 42.033 |
| 11 | 150 | 1.0 | 2 | 67.694 | 65.649 | 66.671 |
| 12 | 150 | 1.0 | 3 | 97.751 | 97.342 | 97.546 |

During the investigation, the optimal conditions for $Fe^{2+}$ concentration, $H_2O_2$ concentration, and treatment time were determined to be 150 mg/L, 1.0 g/L, and 3 hours, respectively. Under these conditions, the decolourization of the textile wastewater reached a remarkable 97.546%. This result is comparable to that achieved by [23], who attained a 95% decolourization rate. However, it should be noted that in their study, they utilized higher levels of the variables and a different initial concentration of aniline.

Additionally, three levels were considered, based on the observations of Gürses et al. [4]. In their study on optimizing the photo-Fenton process for wastewater decolourization, they noted that varying the evaluation times allowed for a comprehensive understanding of the decolourization kinetics. The authors concluded that interactions among the factors influencing process efficiency can be evaluated with a small number of experiments, eliminating the need for high costs associated with experimentation and result analysis.

The conducted research exhibits unique characteristics. To begin with, the materials and equipment utilized were low-cost and readily available for all experimental procedures, ensuring accessibility to the researchers at all times. Furthermore, it is noted that generally, when the initial concentration of the contaminant is relatively low, degradation is more pronounced due to the higher production of -OH radicals. Conversely, at higher concentrations, the reaction of $H_2O_2$ in the redox-active centres may be inhibited, as highlighted by [17]. Additionally, a significant amount of ultraviolet (UV) light can be absorbed by the pollutant molecules rather than the catalyst, potentially diminishing efficiency and catalytic activity, as indicated by the same authors.

The photo-Fenton (photocatalytic) process necessitates a level of energy supplied by UV light, typically ranging from wavelengths of 100 to 400 nm, for various photocatalytic chemical reactions. Hence, in studies, artificial UV light is often employed, although there is a growing trend towards utilizing solar radiation as a natural source. Several researchers underscore the importance of leveraging solar radiation as a UV energy source, including [24–26].

During the experimental phase of the thesis, tests were conducted on sunny days to ensure the availability of UV light. However, due to the energy from other wavelengths contained in solar rays, there was a slight increase in temperature during treatment. It is noteworthy that this factor positively influenced the decolourization of the dye.

## CONCLUSION

The application of the photo-Fenton process for decolorizing synthetic wastewater related to the artisanal textile industry has emerged as a promising and efficient strategy. Through this research, a deeper understanding of the reaction mechanisms involved in this advanced oxidation process, as well as the influence of key factors, has been achieved. It has been established that the concentration of $Fe^{2+}$, treatment time, and the interaction of $Fe^{2+}$ concentration × $H_2O_2$ concentration × treatment time significantly impact the degree of decolourization. Conversely, the concentration of $H_2O_2$ and other interactions do not exert a significant influence on the process. Optimal conditions were found to be 150 mg/L for $Fe^{2+}$ concentration, 1.0 g/L for $H_2O_2$ concentration, and 3 hours for treatment time. Moreover, the highest percentage of decolourization of the residual water achieved was 97.546% for the sample with an initial concentration of 40 mg/L. However, it should be noted that this percentage may vary depending on factors such as the initial concentration, type of dye, or contaminant present in the sample. Lastly, the initial concentration level of the dye was found to be directly related to the concentration of $Fe^{2+}$ and $H_2O_2$, treatment time, and the degree of decolourization of the wastewater.

*Author Contributions*

Conceptualization – Huaman K, Lozano A; methodology – Huaman K, Lozano A and Navarro N; formal analysis – Huaman K, Lozano A, Navarro N and Ortega K; investigation – Huaman K, Lozano A, Navarro N and Ortega K; resources – Huaman K; writing-original draft preparation – Lozano A, Ortega K; writing-review and editing – Huaman K and Navarro N, Lozano A, Ortega K; visualization – Lozano A, Ortega K; supervision – Huaman K. All authors have read and agreed to the published version of the manuscript.

*Conflicts of Interest*

The authors declare no conflict of interest.

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