



RESEARCH ARTICLE

PERFORMANCE COMPARISON OF UV, UV/H₂O₂, UV/Fe²⁺, H₂O₂/Fe²⁺, UV/H₂O₂/Fe²⁺ PROCESSES IN THE REMOVAL OF COD AND COLOR FROM TEXTILE WASTEWATER

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Received Date: 29.10.2020

Accepted Date: 11.12.2020

ABSTRACT

In this study, the removal of chemical oxygen demand (COD) and color from textile industry wastewater were investigated in comparison with UV, UV/H₂O₂, UV/Fe²⁺, H₂O₂/Fe²⁺, UV/H₂O₂/Fe²⁺ processes. Initial pH, hydrogen peroxide dosage, ferrous iron dosage, and UV radiation were selected as variables. A maximum of 95.65% COD and 98.52% color removal were achieved by the photo-Fenton method. Another effective method was Fenton process by which 83.76% COD and 80.44% color removal efficiencies were obtained. It was concluded that the Fe²⁺/H₂O₂ process (Fenton) with UV light (photo-Fenton) can provide higher removal efficiencies in shorter process times in the treatment of textile wastewater.

Keywords: *Textile wastewater, COD, Color, Photo-Fenton, UV/H₂O₂/Fe²⁺*

1. INTRODUCTION

The textile industry generally creates large volumes of wastewater containing highly concentrated organic dyes, inorganic ions such as chlorides and sulfates, soaps, oils, solvents, salts, and many other soluble compounds. The complex structure of these wastewaters can prevent their biodegradation. For this reason, they must be treated with appropriate methods to avoid environmental risks before being introduced into the receiving environment [1,2]. The main source of COD and color parameters in textile wastewater is dyestuff and polyvinyl alcohol, and they are characterized by high concentrations of COD (150-10,000 mg/L), BOD (100-4000 mg/L), and color content (50-2500 Pt-Co). Also, these wastewaters are considered toxic and carcinogenic [3, 4]. BOD/COD ratios of textile wastewaters are mostly in the range of 0.1–0.25 and are resistant to biodegradation [5,6,7,8]. Physico-chemical processes such as coagulation/flocculation, precipitation, adsorption, ion exchange, membrane separation, and oxidation or biological methods have been applied in most cases. Because of the toxicity possibilities of textile wastewaters, biological methods are seen inefficient especially if the pollutants are present in high concentrations. In physicochemical methods, sludge formation occurs relatively more [4,5]. Recently, electrochemical Technologies such as Advanced Oxidation Processes (AOPs) have been considered as efficient and practical alternatives for the removal of pollutants from textile wastewaters [9]. By using the hydroxyl radicals ([•]OH) produced in advanced oxidation

processes, organic pollutants are converted into carbon dioxide and water quickly and efficiently [10]. Among these methods, especially $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ and $\text{UV}/\text{H}_2\text{O}_2/\text{Fe}^{2+}$ (photo-Fenton) processes attract attention. In the Fenton reaction, a highly reactive hydroxyl radical is formed by the H_2O_2 ferrous reaction (Eq. 1). In addition, the ferric iron formed can be oxidized by H_2O_2 to form a hydroperoxyl radical ($\bullet\text{O}_2\text{H}$) (Eq. 2)



Eq. 3 shows that $\text{Fe}(\text{OH})^{2+}$ complexes formed Fenton reactions can transform into hydroxyl radicals by UV light, also ferrous ions can be occurred, which is used in fenton process. This situation creates much less sewage sludge. Eq. 4 show that hydrogen peroxide can be convert to two hydroxyl radicals under exposure to UV light. Thus, the efficiency of fenton processes can be improved by UV radiation [11,12,13].



For removing several pollutants from synthetic or real textile wastewaters, advanced oxidation methods such as electro-Fenton (2), photo-Fenton (4), UV, $\text{UV}/\text{H}_2\text{O}_2$, $\text{UV}/\text{H}_2\text{O}_2/\text{Fe}^{2+}$ (5) ve peroxi-coagulation were used (6). Most of them were a comparison of one or two methods. In this study, COD and color removal from real textile wastewater by UV, $\text{UV}/\text{H}_2\text{O}_2$, UV/Fe^{2+} , $\text{H}_2\text{O}_2/\text{Fe}^{2+}$, and $\text{UV}/\text{H}_2\text{O}_2/\text{Fe}^{2+}$ methods were investigated in detail.

2. MATERIAL AND METHODS

2.1. Wastewater Characterization

The textile industry wastewater used in the experiments was supplied from the dyeing process effluent of an enterprise operating in the fields of printing services (including clothing) on fabrics and textile products in the organized industrial zone of Fatsa district of Ordu. Wastewater characterization is given in Table 1.

Table 1. Characteristics of untreated textile wastewater.

Analytical parameter	Value
pH	6.8-8.2
Total suspended solids (mg/L)	77.8
Turbidity (NTU)	872
Conductivity ($\mu\text{S}/\text{cm}$)	7960
COD (mg/L)	2962-4412
Color (Pt-Co)	1426-1779

2.2. Apparatus and Procedure

All experiments were carried out in a plexiglass column with an inner diameter of 7.8 cm, an outside diameter of 8.35 cm, and a height of 48.5 cm (Fig. 1). A 16W UV-C (254 nm) low-pressure mercury-vapor lamp mounted in a quartz tube was placed vertically in the center of the reactor. At the base of

the reactor, a 2 cm vertical space was left for the magnetic stirrer to rotate. The mixing of the wastewater in the reactor was provided by the magnetic stirrer at the bottom.



Figure 1. Experimental setup.

All experiments were carried out with 2.1 L wastewater volume at room temperature (22 ± 2 °C). Before each experiment, the wastewater was filtered through 2 coarse filter papers. H_2SO_4 and NaOH solutions were used for pH adjustments. The reactor was covered with aluminum foil to prevent light from coming out. With the help of the tap placed in the reactor, samples of treated wastewater were taken at certain time intervals. Final pH values were recorded after each experiment. The treated wastewater samples taken after the experiments were centrifuged at 5000 rpm for 10 minutes to remove the flocs. Thermoreactor (Spectroquant TR 4220) and photometer (Merck Spectroquant NOVA 60A) were used for COD analysis. This photometer was also used for color measurements. COD measurements were carried out according to the closed-reflux colorimetric method (5520-D). Color measurements were made according to the visual comparison method (2120-B) [14]. H_2SO_4 (98%), potassium dichromate ($K_2Cr_2O_7$), mercury sulfate ($HgSO_4$), silver sulfate (Ag_2SO_4), potassium hydrogen phthalate ($C_8H_5KO_4$) were used for COD analysis. Ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) was used as the Fe^{2+} source. H_2O_2 (35%) was used in Fenton experiments. All reagents used were analytical grade.

3. RESULTS AND DISCUSSION

3.1. UV Process

3.1.1. Effect of pH

The effect of UV was examined for a process time of 120 minutes. As can be seen from the Fig. 2 and Fig. 3, COD and color removal efficiencies obtained at the initial pH values of 4, 7, and 10 increased

with increases in the process time. While the maximum efficiencies achieved were 7.78%, 11.56%, and 8.61% for COD at pH 4, 7, and 10, they were 10.50%, 13.72%, and 11.02% for color. As can be seen from Fig. 2 and Fig. 3, pH 7 was optimal for both COD and color removal. By increasing the initial pH from 4 to 7, the efficiency for COD increased from 7.78% to 11.56% after 120 minutes, while for the color it was slightly increased from 10.50% to 13.72%. This indicates that initial pH is very effective in removing COD and color removal. On the other hand, UV radiation alone was not effective in COD and color removal from real textile wastewater. Concerning the UV effect, a similar result was obtained in the dye removal study by Bali (2004).

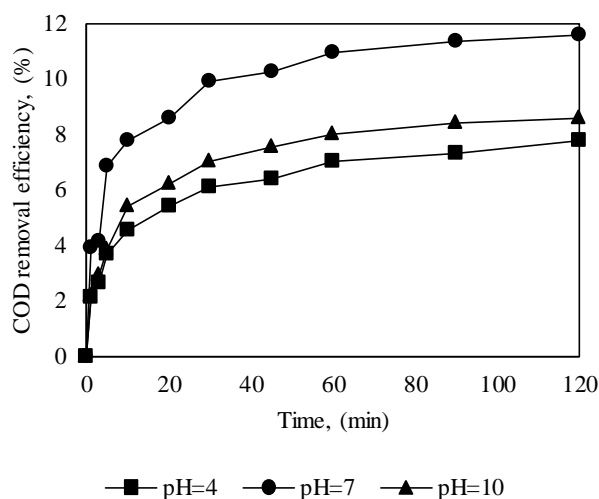


Figure 2. Effect of initial pH on COD removal efficiency in UV process.

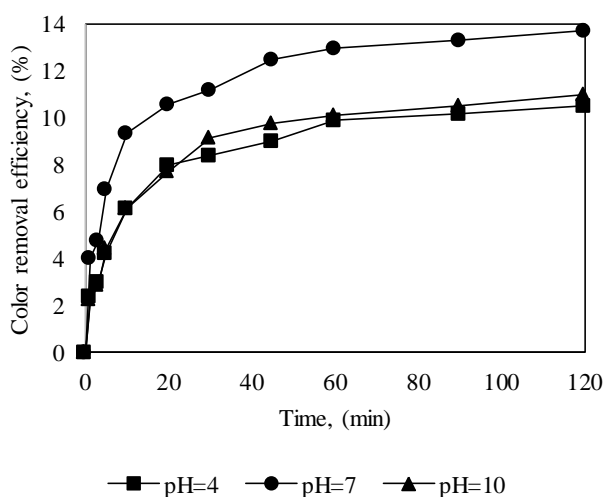


Figure 3. Effect of initial pH on Color removal efficiency in UV process.

3.2. UV/H₂O₂ Process

3.2.1. Effect of pH

The change over time of the effects of initial pH values varying between 4 and 9, in which H₂O₂ concentration was kept constant at 25 mM, on COD and color removal efficiencies is shown in Fig. 4 and Fig. 5. With the increase of pH from 4 to 7, COD and color removal efficiencies increased from 20.58 to 26.70%, and from 37.32 to 49.87%, respectively. Increasing pH from 7 to 9 decreased COD and color removal efficiencies from 26.70% to 25.49%, and from 49.87% to 44.90, respectively. Initial pH values of 7 and 9 reached 7.12 and 9.10, respectively, at the end of 120 minutes. No significant changes occurred in other pH values.

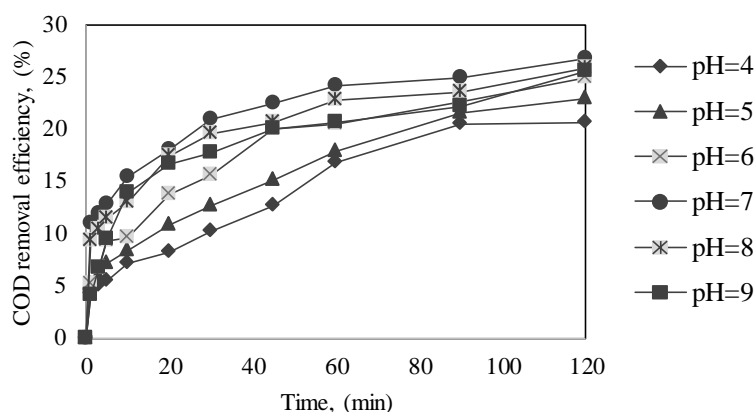


Figure 4. Effect of initial pH on COD removal efficiency in UV/H₂O₂ process.

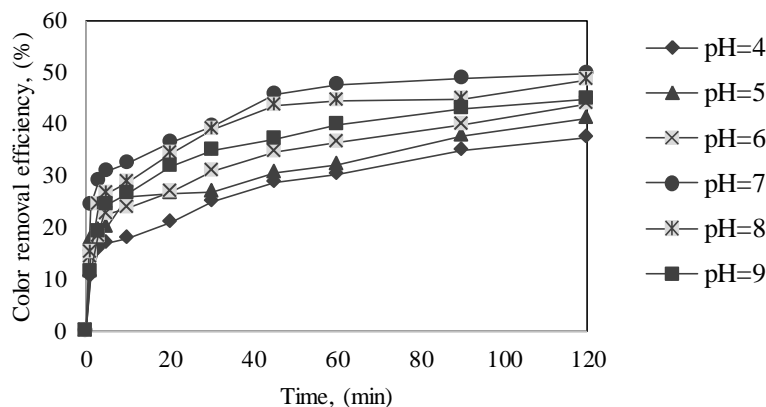


Figure 5. Effect of initial pH on Color removal efficiency in UV/H₂O₂ process.

3.2.2. Effect of H₂O₂

The change over time of the effects of H₂O₂ concentrations varying between 5 and 100 mM, in which pH was kept constant at 7, on COD and color removal efficiencies is shown in Fig. 6 and Fig. 7. With the increase of H₂O₂ concentration from 5 mM to 100 mM, COD and color removal efficiencies

reached 33.08% and 61.21%, respectively, which are the maximum values for both. As can be seen from Fig. 6 and Fig. 7, while the COD removal efficiencies were dependent on different H₂O₂ concentrations after 60 minutes, the observed dependence for color removal efficiencies was over the entire experiment period. The initial pH value of 7 varied between 7.07 and 7.40 for 5 and 100 mM H₂O₂ concentrations, respectively.

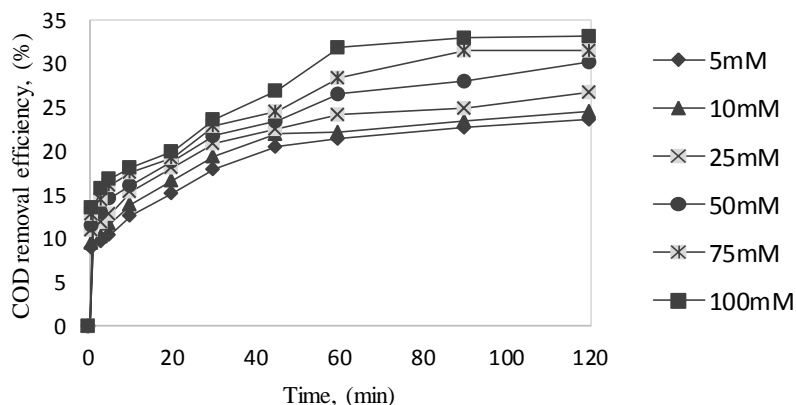


Figure 6. Effect of H₂O₂ concentration on COD removal efficiency in UV/H₂O₂ process.

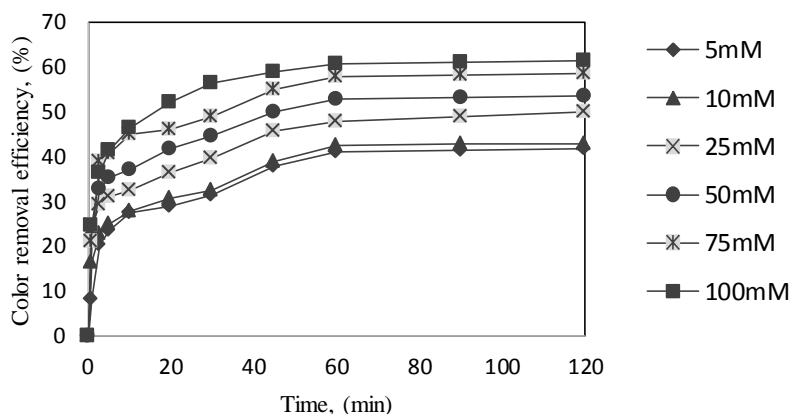


Figure 7. Effect of H₂O₂ concentration on Color removal efficiency in UV/H₂O₂ process.

3.3. UV/Fe²⁺ Process

3.3.1 Effect of pH

The change over time of the effects of initial pH values varying between 4 and 9, in which Fe²⁺ was kept constant in 2 mM, on both COD and color removal efficiency is shown in Fig. 8 and Fig. 9. Maximum of 44.02% COD and 55.42% color removal efficiencies were obtained at pH 4 at the end of 120 minutes, while COD removal of 43.21% and color removal of 54.86% were obtained in the first 60 minutes. By increasing the pH from 4 to 9, COD and color removal efficiencies decreased from 44.02% to 29.30% and from 55.42% to 41.09, respectively. Initial pH values between 4 and 9 changed between 4.07 and 9.05 at the end of 120 minutes.

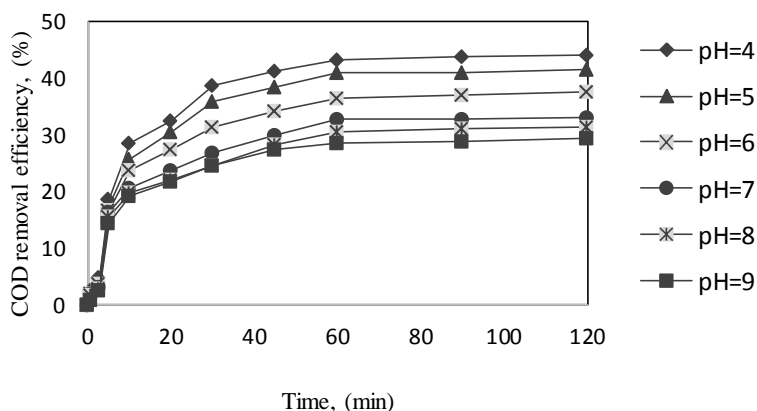


Figure 8. Effect of initial pH on COD removal efficiency in UV/Fe²⁺ process.

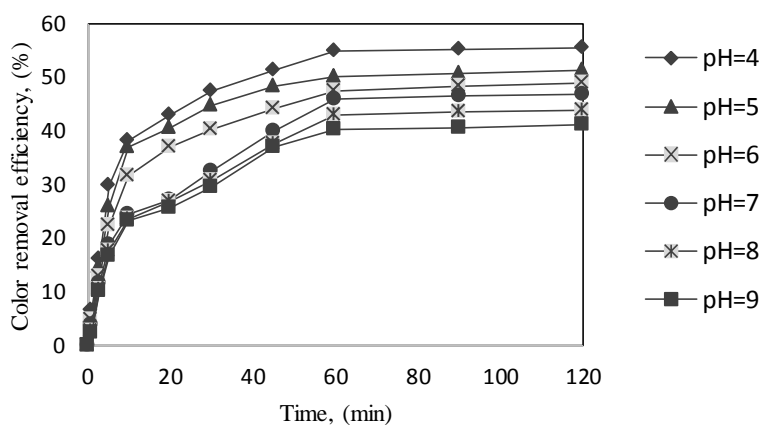


Figure 9. Effect of initial pH on Color removal efficiency in UV/Fe²⁺ process.

3.3.2. Effect of Fe²⁺

The change over time of the effects of initial Fe²⁺ concentrations varying between 0.25 and 3 mM, in which pH was kept constant at 4, on both COD and color removal efficiencies is shown in Fig. 10 and Fig. 11. A maximum COD of 19.62% and 33.74% color removals obtained at the 0.25 mM Fe²⁺ concentration increased to 49.89% and 59.44%, respectively, by increasing the Fe²⁺ concentration to 3 mM. The initial pH 4 value changed between 4.03 and 4.04 at the end of 120 minutes.

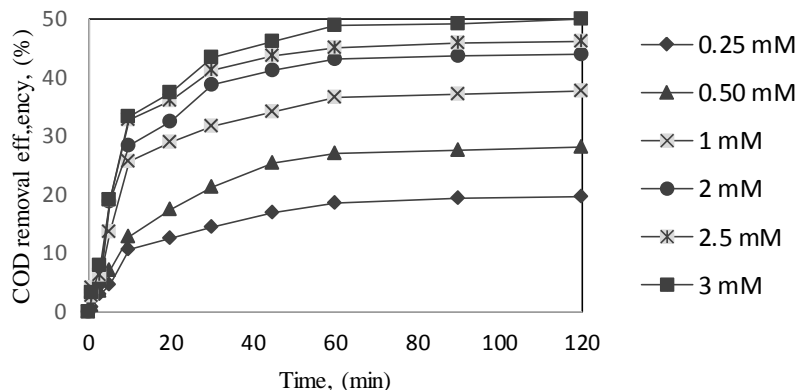


Figure 10. Effect of initial Fe^{2+} concentration COD removal efficiency in UV/ Fe^{2+} process.

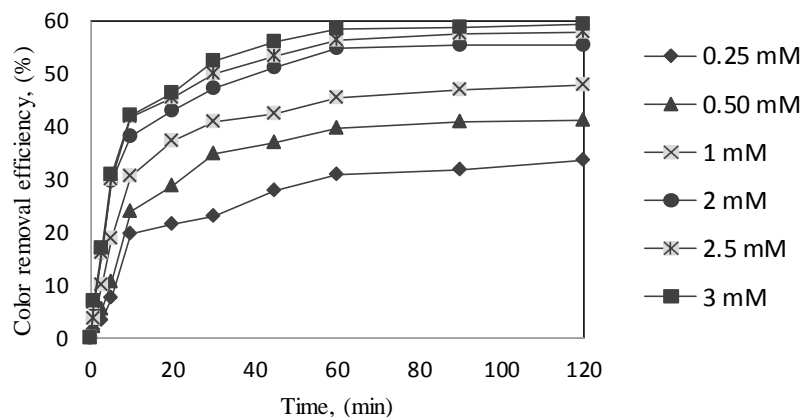


Figure 11. Effect of initial Fe^{2+} concentration Color removal efficiency in UV/ Fe^{2+} process.

3.4. $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ Process

3.4.1. Effect of pH

The effect of initial pH values varying between 2 and 4.5 on COD and color removal efficiencies by keeping the 60 mM H_2O_2 concentration and the 2 mM Fe^{2+} concentration constant ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio: 30) is shown in Fig. 12 and Fig. 13. The maximum COD of 83.76% and color of 80.44% were obtained in the first 30 minutes at pH 2. With increasing pH from 2 to 4.5, COD and color removal efficiencies decreased to 48.54% and 55.42%, respectively, after 30 minutes. Initial pH values varying between 2 and 4 varied between 2.09 and 4.56.

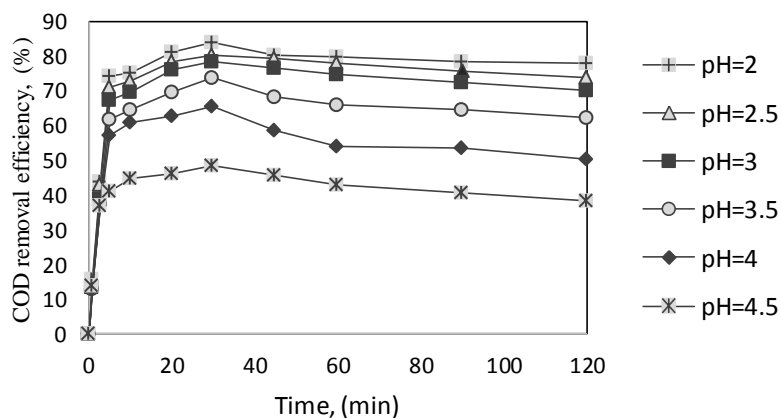


Figure 12. Effect of initial pH on COD removal efficiency in H_2O_2/Fe^{2+} process.

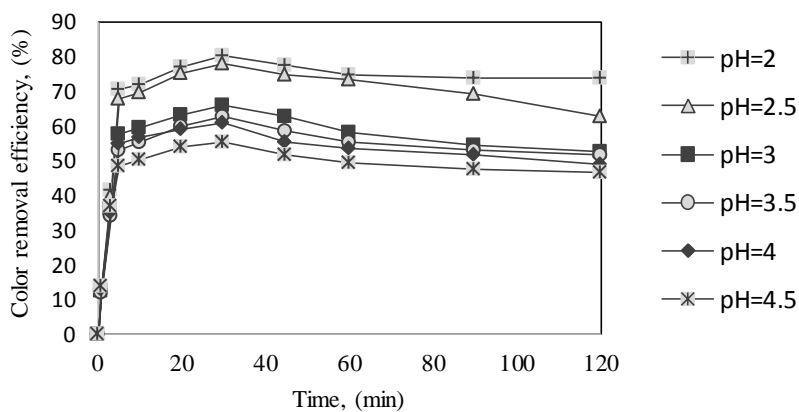


Figure 13. Effect of initial pH on Color removal efficiency in H_2O_2/Fe^{2+} process.

3.4.2. Effect of H_2O_2

The effects of H_2O_2 concentrations varying between 5 and 100 mM on COD and color removal efficiencies by keeping the pH 2 and 2 mM Fe^{2+} concentration constant are shown in Fig. 14 and Fig. 15. As can be seen from Fig. 14 and Fig. 15, COD removal of 83.76% and color removal of 80.44% was obtained at H_2O_2 concentration of 60 mM in the first 30 minutes. The efficiencies obtained with 50, 75 and 100 mM H_2O_2 in the first 30 minutes were 78.38%, 76.34%, and 72.71% for COD, while these values were 77.87%, 67.72%, and 59.25% for color removal. The results indicated that the H_2O_2/Fe^{2+} molar ratio should be at least 25 (50 mM $H_2O_2/2$ mM Fe^{2+}) in the conditions of this study. Initial pH values of 2 varied between 2.08 and 2.10 at the end of 120 minutes.

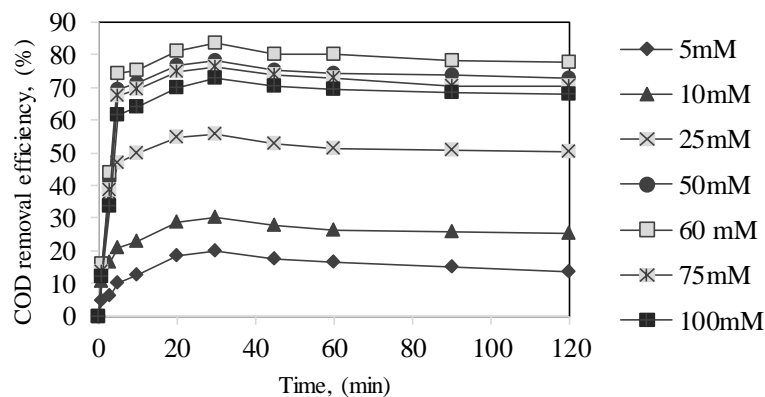


Figure 14. Effect of H_2O_2 concentration on COD removal efficiency in H_2O_2/Fe^{2+} process.

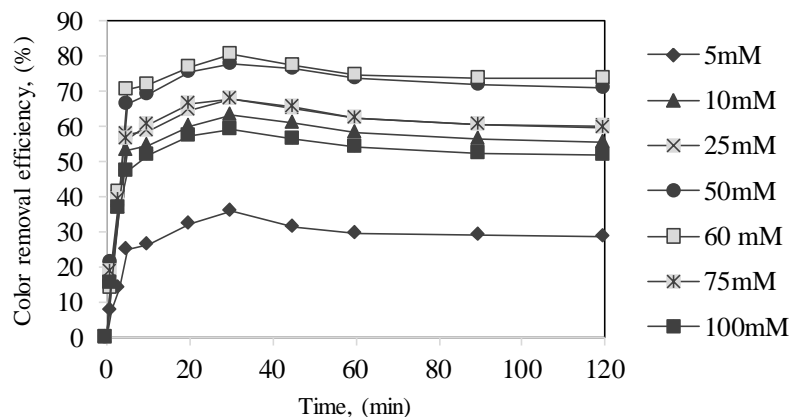


Figure 15. Effect of H_2O_2 concentration on Color removal efficiency in H_2O_2/Fe^{2+} process.

3.4.3. Effect of Fe^{2+}

The effects of Fe^{2+} concentrations varying between 0.25 and 3 mM, in which pH was kept at 2 and H_2O_2 was kept in 60 mM, on both COD and color removal efficiency are shown in Fig. 16 and Fig. 17. The maximum of 83.76% COD and 80.44% color removal efficiencies obtained at Fe^{2+} concentration of 2 mM decreased to 76.54 for COD at the end of 30 minutes when the Fe^{2+} concentration was increased to 3 mM and decreased to 67.81 for color. Initial pH 2 value varied between 2.03 and 2.02 at the end of 120 minutes.

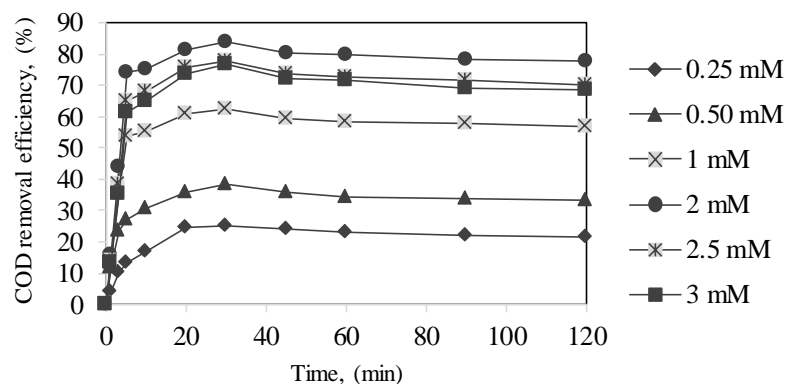


Figure 16. Effect of Fe^{2+} concentration on COD removal efficiency in $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ process.

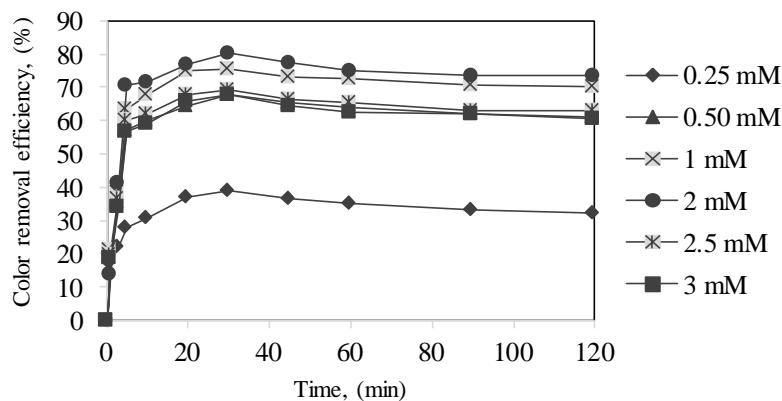


Figure 17. Effect of Fe^{2+} concentration on Color removal efficiency in $\text{H}_2\text{O}_2/\text{Fe}^{2+}$.

3.5. UV/ $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ Process

3.5.1. Effect of pH

The effect of initial pH values varying between 2 and 4.5, in which H_2O_2 was kept constant in 60 mM and Fe^{2+} was kept in 2 mM, on both COD and color removal efficiencies is shown in Fig. 18 and Fig. 19. COD of 49.86% and color of 53.21% removal efficiencies at pH 2 in the first 3 minutes, increased to 71.27% and 82.74%, respectively at the end of the 5 minutes. While the maximum values of 88.60% and 93.95%, of COD and color, are reached after 60 minutes, these values decreased to 79.57% and 83.42 when the pH increased from 2 to 4.5. Initial pH values between 2 and 4.5 varied between 2.08 and 4.53 in parallel with increasing pH values.

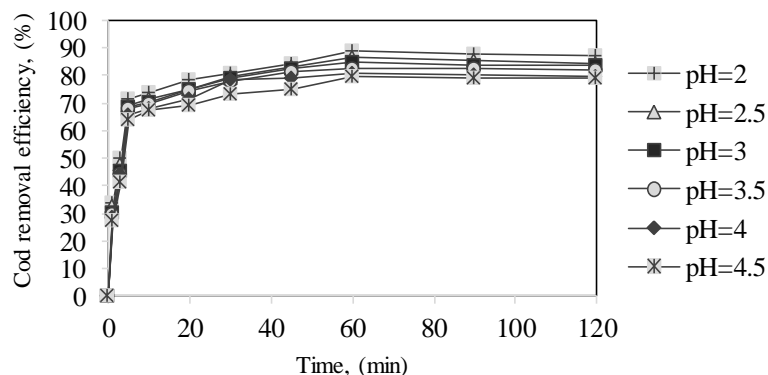


Figure 18. Effect initial pH on COD removal efficiency in UV/H₂O₂/Fe²⁺ process.

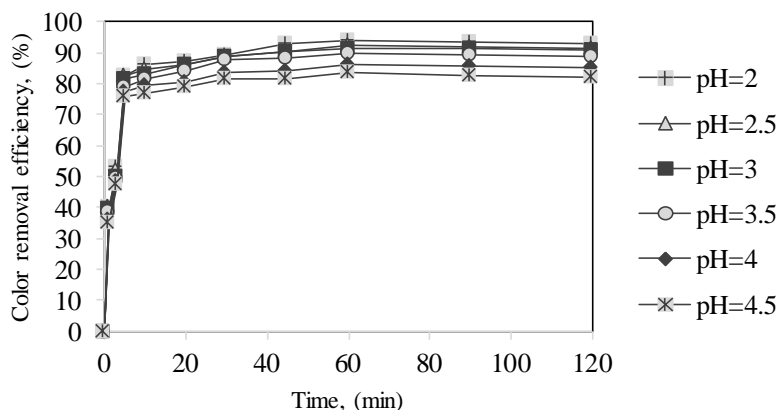


Figure 19. Effect initial pH on Color removal efficiency in UV/H₂O₂/Fe²⁺ process.

3.5.2. Effect of H₂O₂

The effects of H₂O₂ concentrations varying between 5 and 100 mM, in which pH was kept at 2 and Fe²⁺ was kept in 2 mM, on both COD and color removal efficiencies are shown in Fig. 20 and Fig. 21. By increasing the H₂O₂ concentration from 5 to 50 mM, COD removal efficiency increased from 42.29% to 90.30% in the first 60 minutes and color removal increased from 57.79% to 95.83% at the end of the same period time. With increasing the concentration to 100 mM, the efficiencies increased to 84.87% and 92.86% for COD and color, respectively. No significant differences were determined after the first 60 minutes between efficiencies achieved with H₂O₂ concentrations other than 5 and 10 mM. The initial pH of 2 varied between 2 and 2.08 for 5 and 100 mM H₂O₂ concentrations, respectively.

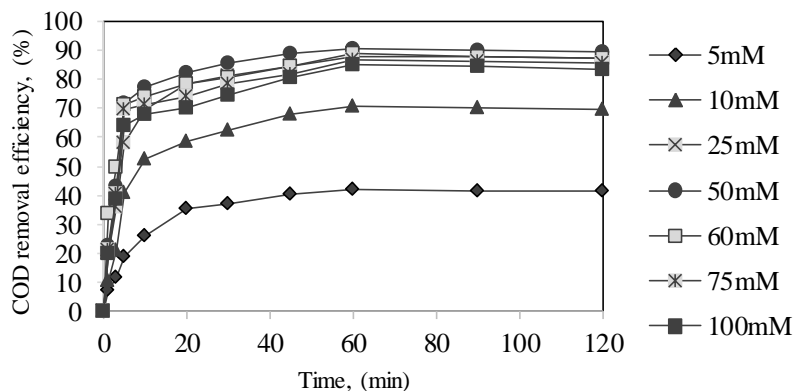


Figure 20. Effect of H_2O_2 concentration on COD removal efficiency in UV/ H_2O_2 / Fe^{2+} process.

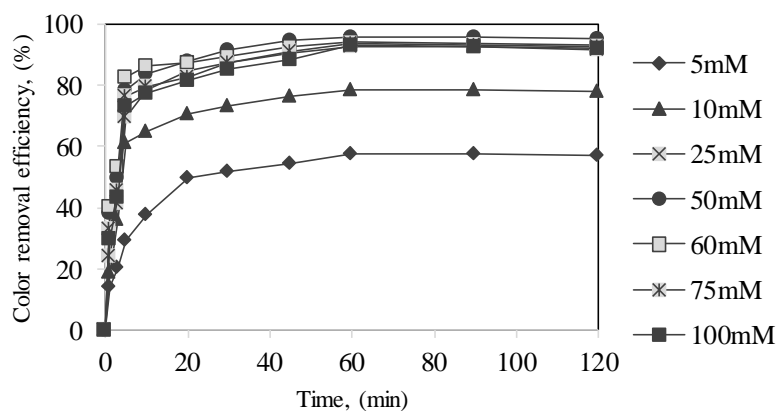


Figure 21. Effect H_2O_2 concentration on Color removal efficiency in UV/ H_2O_2 / Fe^{2+} process.

3.5.3. Effect of Fe^{2+}

The effects of Fe^{2+} concentrations varying between 0.25 and 3 mM, in which pH was kept at 2 and H_2O_2 was kept in 50 mM, on both COD and color removal efficiency are shown in Fig. 22 and Fig. 23. Maximum COD and color removal efficiencies of 95.65% and 98.52% were achieved in the first 60 minutes with 3 mM Fe^{2+} concentration. Fe^{2+} concentrations of 0.25 and 0.50 mM were not sufficient for COD removal. While relatively high Fe^{2+} concentrations were required for high COD removals, this was not determined for color removal. For example, the COD and color removal efficiencies of 95.65% and 98.52%, respectively, at a concentration of 3 mM Fe^{2+} at the end of 60 minutes were 59.97% and 82.36%, respectively, at 0.25 M Fe^{2+} concentration. The initial pH 2 changed between 2 and 2.03. By examining all experimental conditions, pH: 2, H_2O_2 : 50 mM, Fe^{2+} : 3 mM, and process time: 60 minutes were determined as optimum operating conditions.

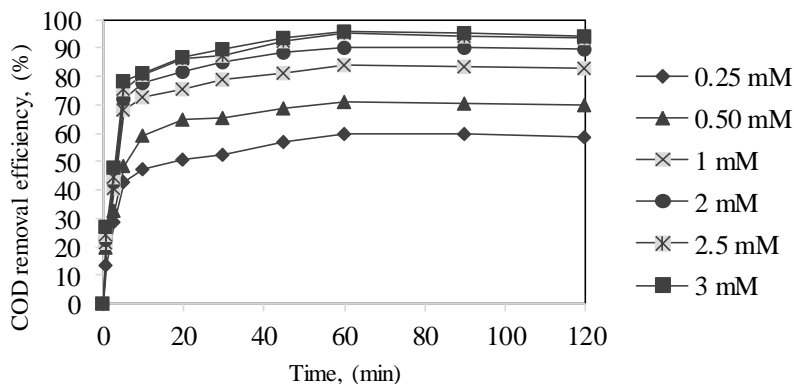


Figure 22. Effect Fe^{2+} concentration on COD removal efficiency in UV/ H_2O_2 / Fe^{2+} process.

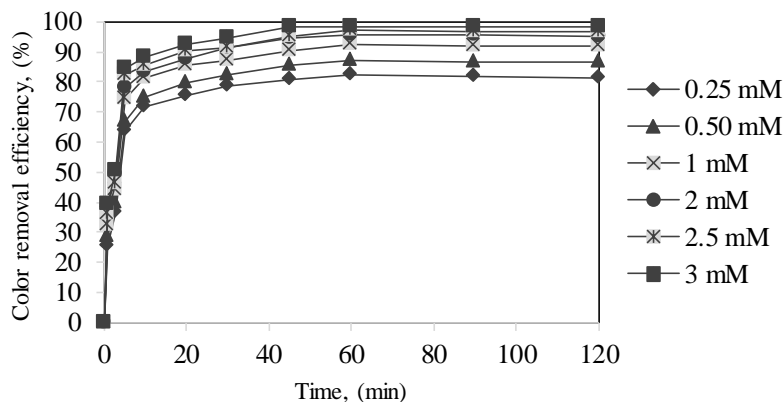


Figure 23. Effect Fe^{2+} concentration on Color removal efficiency in UV/ H_2O_2 / Fe^{2+} process.

3.6. Comparison of Processes

Under the optimum conditions determined for each process (UV: pH 7; UV/ H_2O_2 : pH 7 and 100 mM H_2O_2 ; UV/ Fe^{2+} : pH 4 and 3 mM Fe^{2+} ; H_2O_2 / Fe^{2+} : pH 2 and 60 mM H_2O_2 ; UV/ H_2O_2 / Fe^{2+} : pH 2, 50 mM H_2O_2 and 3 mM Fe^{2+}) obtained COD and color removal efficiencies are given in Fig. 24 and Fig. 25, respectively. As can be seen from Fig. 24 and Fig. 25, a maximum of 95.65% COD and 98.52% color removal efficiencies were obtained at the end of 60 minutes by the photo-Fenton method. It was understood that a combination of UV radiation with Fenton process caused an increase of 9.99% and 18.08%, respectively in 83.76% COD and 80.44% removal efficiencies obtained by Fenton process. In the H_2O_2 / Fe^{2+} process, the decreases in COD and color efficiency after 30 minutes indicated that no H_2O_2 could react and the remaining Fe^{2+} ions created color and COD. Also, the UV process was not effective alone. UV/ H_2O_2 and UV/ Fe^{2+} processes were relatively efficient only in color removal.

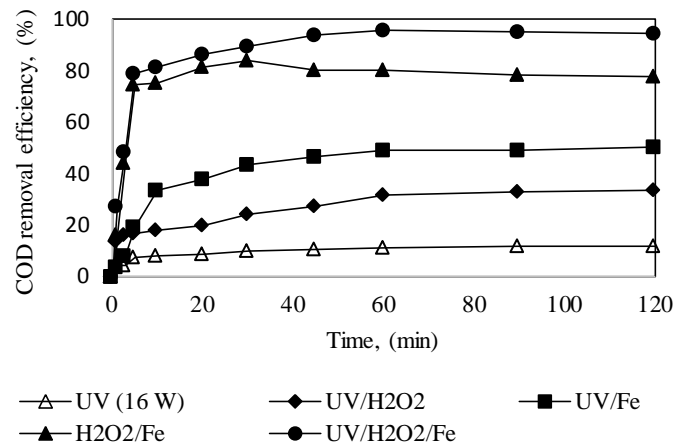


Figure 24. Comparison of UV, UV/H₂O₂, UV/Fe²⁺, H₂O₂/Fe²⁺, UV/H₂O₂/Fe²⁺ processes on COD removal efficiency.

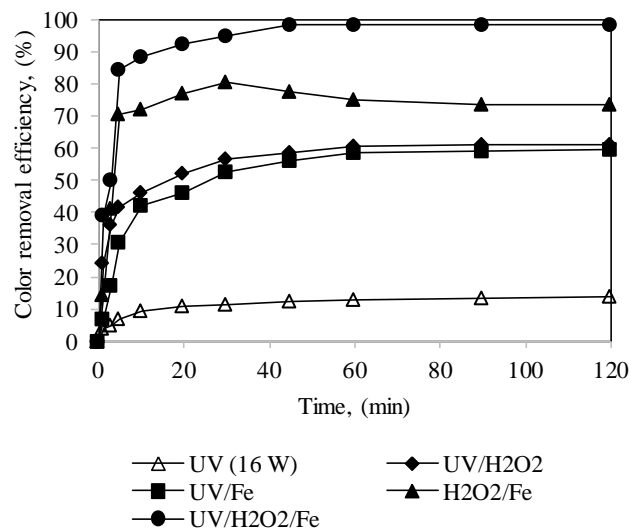


Figure 25. Comparison of UV, UV/H₂O₂, UV/Fe²⁺, H₂O₂/Fe²⁺, UV/H₂O₂/Fe²⁺ processes on Color removal efficiency.

4. CONCLUSION

- UV process alone was not effective in COD and color removal. Maximum of 11.56% COD and 13.72% color removal efficiencies were achieved at the optimum conditions.

- While UV/H₂O₂ process achieved a maximum of 33.08% COD and 61.21% color removal efficiency, a maximum of 49.89% COD and 59.44% color removal efficiency were achieved with the UV/ Fe²⁺ process.
- While the maximum COD and color removal was 83.76% and 80.44% in the first 30 minutes in the Fenton process, these values were reached in a short time like 10 minutes in the photo-Fenton process. For the photo-Fenton process, a maximum of 95.65% COD and 98.52% color removal efficiencies were achieved within 60 minutes.
- In UV, UV / H₂O₂, UV/Fe²⁺, H₂O₂/Fe²⁺, and UV/H₂O₂/Fe²⁺ processes, maximum COD and color removal efficiencies were achieved at pH values of pH 7, 7, 4, 2, and 2, respectively.
- It has been determined that the textile wastewater treated only with the photo-Fenton method met the COD and color discharge standards of Water Pollution and Control regulation in force in our country.

ACKNOWLEDGMENTS

This study was supported by the Scientific Research Project Fund of Ondokuzmayis University (Project No: PYO.MUH.1904.19.008). We would like to thank for the financial support.

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