SAKARYA ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ DERGİSİ

Sakarya University Journal of Science
SAUJS

ISSN 1301-4048 e-ISSN 2147-835X Period Bimonthly Founded 1997 Publisher Sakarya University
http://www.saujs.sakarya.edu.tr/

Title: Application of Classical Fenton Process and Advanced Photo Electro Fenton Process for the Degradation of COD from Wood Processing Wastewater: A Comparative Study

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Recieved: 2022-09-09 00:00:00

Accepted: 2023-02-20 00:00:00

Article Type: Research Article

Volume: 27
Issue: 3
Month: June
Year: 2023
Pages: 512-522

How to cite
Murat SOLAK; (2023), Application of Classical Fenton Process and Advanced Photo Electro Fenton Process for the Degradation of COD from Wood Processing Wastewater: A Comparative Study. Sakarya University Journal of Science, 27(3), 512-522, DOI: 10.16984/saufenbilder.1173306
Access link
https://dergipark.org.tr/en/pub/saufenbilder/issue/78131/1173306

Sakarya University Journal of Science 27(3), 512-522, 2023

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Application of Classical Fenton Process and Advanced Photo Electro Fenton Process for the Degradation of COD from Wood Processing Wastewater: A Comparative Study

Murat SOLAK*[1]

Abstract

In this study, Chemical Oxygen Demand (COD) removal efficiency from wood processing wastewaters by Fenton Process (FP) and Photo Electro Fenton Process (PEFP) were examined. Important operating parameters such as pH, Fe^{+2} concentration/(amper for PEFP), H_2O_2 concentration and reaction time were optimized. Optimum operation conditions of the FP were pH 3.5, 1.4 gr/L Fe^{2+} concentration and 50 gr/L H_2O_2 concentration and 150 min. reaction time while they were pH 3.00, 9.99 mA/cm^2 current density and 70 gr/L H_2O_2 concentration and 150 min reaction time in PEFP. At the optimum conditions, COD removal efficiency of FP and PEFP was 91% and 99%, respectively. Sludge production of FP was 20% higher than PEFP at the optimum conditions.

Keywords: Wood processing wastewater, Fenton process, photo electro Fenton process

1. INTRODUCTION

Depending on rapid developments in organic material demand due to the increasing demands in home usages, the number of the wood processing industries has increased in recent years. The amount of wastewater generated by this sector, which uses high amounts of water in production processes, has also increased. In the process of manufacturing wood products, trees brought to the factory as billets are peeled from their shells. It is then processed by immersing in water at high temperatures. The wood is held in water at high temperature for about one day. This process is the source of wastewater. Finally, the logs are converted into smaller wood products by various processes. This wastewater generated in production processes has high organic pollutant as COD, BOD and colour. In the treatment of these kind of wastewaters conventional methods such as coagulation-flocculation are not enough. Therefore, it would be more appropriate to apply advanced treatment techniques as oxidation [1], membrane filtration [2], biological processes [3] to such wastewaters. The Fenton oxidation process is one of the processes where the organic pollutants are effectivley removed from wastewaters at low pH values with Fe(II) reagent and H_2O_2 oxidant [4]. The formation of the

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homogeneous hydroxyl radicals (OH) is the main reaction for the Fenton processes and hydroxyl radicals play important role in this process, oxidizing the organic pollutants into CO_2, H_2O and inorganic ions by dehydrogenating or hydroxylating reaction Eq. (5) or Eq. (6), respectively. Fenton reactions are given in Equation 1 to 7 [5].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\cdot} \quad (1)$$

Fe (II) is oxidized to Fe (III) by another reaction of hydroxyl radicals in the environment.

$$OH^{\cdot} + Fe^{2+} \rightarrow OH^- + Fe^{3+} \quad (2)$$

Fe^{3+} decomposes H_2O_2 with a mechanism that involves hydroxyl and hydroperoxyl radicals.

$$Fe^{+3} + H_2O_2 \leftrightarrow Fe - OOH^{2+} + H^+ \quad (3)$$

$$Fe - OOH^{2+} \rightarrow HO_2^{\cdot} + Fe^{2+} \quad (4)$$

$$Fe^{2+} + HO_2^{\cdot} \rightarrow Fe^{+3} + HO_2^- \quad (5)$$

$$Fe^{+3} + HO_2^{\cdot} \rightarrow Fe^{2+} + H^+ + O_2 \quad (6)$$

$$OH^{\cdot} + H_2O_2 \rightarrow H_2O + HO_2^{\cdot} \quad (7)$$

Photo Electro Fenton process is an advanced Fenton process which is used in a wide scope of wastewaters as landfill leachate [6], coking industry [7], dairy industry [8], dye production industry [9]. The difference of the Fenton process from the electro-Fenton process is the method of addition of Fe(II) to the wastewater. In the Fenton process, Fe(II) is added externally, but in Electro-Fenton process Fe(II) ions are produced by electrolysis. In addition, to enhance the effectiveness of electro Fenton process, and to oxidize the organic materials effectively a UV lamp can be added to this process. Photo Electro Fenton process, is the wastewater treatment technique that UV and electro Fenton processes are used together [10], [11]. The Photo Electro Fenton process takes place in two ways. i) production of hydroxyl radicals by photo reduction of $Fe(OH)^{2+}$ (Eq. (8)) and (ii) production of Fe (II) by photolysis of Fe (III) (Eq.(9)).

$$Fe(OH) \xrightarrow{2+h\upsilon(k_4)} Fe^{2+} + \ {}^{\cdot}OH \quad (8)$$

$$R(CO_2) - Fe(III) + h\upsilon \rightarrow R(\ {}^{\cdot}CO_2) + Fe(II) \rightarrow \ {}^{\cdot}R + CO_2 \quad (9)$$

In the present study, the performance of Fenton process (FP) and photo electro-Fenton (PEF) process for the removal of high COD from wood processing wastewater was compared. The effects of operating parameters such as pH, Fe^{2+} concentration/current, H_2O_2 concentration and reaction time on COD removal were investigated.

2. MATERIALS AND METHODS

2.1 Wastewater samples

Characterization of wood processing wastewater is given in Table 1. Raw wastewater is supplied from a wood processing company that produces wood products in Düzce, Türkiye.

Table 1 Raw wastewater characterization

Parameter	Value/Concentration
pH	5.52 ± 0,21
Conductivity	4.16 ± 0,1 mS/cm
COD	3850 ± 150 mg/L
SS	1500 ± 120 mg/L
TDS	2.07 ± 0.02 g/L
Colour	
460nm	12 m^{-1}
525nm	15 m^{-1}
620nm	17 m^{-1}

2.2 FP experiments

Fenton process experiments were performed with a volume of 400 mL wood processing wastewater by Jar Tests (Fig. 1). $FeSO_4.7H_2O$ (Ferrous sulfate - heptahydrate) as a Fe^{2+} source and H_2O_2 (hydrogen peroxide, 50% W/W) as an oxidant were used in the experiments. Since Fenton process is efficient at low pH, pH was adjusted by adding H_2SO_4, $FeSO_4.7H_2O$ was initially added to the pH-adjusted sample, and H_2O_2 was added in the second step. After the addition of $FeSO_4.7H_2O$ and H_2O_2, the samples were mixed at 150 rpm with rapid mixing for 5 minutes and at 50 rpm with slow mixing for 45 minutes. When the determined reaction times were completed, the pH of the wastewater sample was increased to 7.5-8.0. Then the pollutants in the wastewater were allowed to settle for 1 hour. The supernatants were filtered with using 0.45 μm filter papers and soluble COD and colour analysis were conducted. pH adjustment was done by using 1 N H_2SO_4 and 1 N NaOH solutions.

Figure 1 Jar test

2.3 PEFP experiments

PEFP experiments were performed with a volume of 750 mL of wood processing wastewater by the PEF reactor (Fig. 2). Reactor dimensions were as 20 cm x 10 cm x 10 cm (HxLxB). 40 W (254 nm) UV lamp was used for the UV light source in PEFP. 10 iron electrodes with dimensions of 1 cm x 20 cm were used for the electro Fenton process in the reactor (Fig. 3). 10 mL H_2O_2 oxidant was added as soon as the electrolysis process was started in the reactor. Then the oxidant was added to the reactor with an interval of 10 min. 3A-30V DC Power Supply was used to give an electricity to the electrodes.

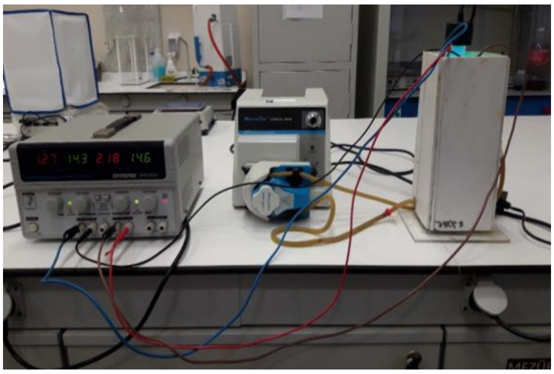


Figure 2 PEFP experimental design

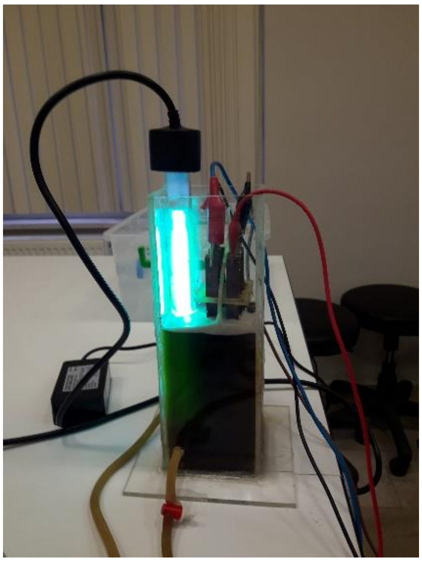


Figure 3 PEFP reactor

2.4 Analytical methods

In experimental studies, Standard Method (5220 D) was used for the determination of COD concentrations in inlet and outlet water samples. pH measurements were performed by electrometric method with Hanna HI 2211 pH meter, conductivity and TDS measurements were performed by electrometric method with Hach Conductivity-TDS meter [12].

3. RESULTS AND DISCUSSION

3.1 Effect of pH on FP and amper on PEFP

pH is one of the most important parameters affecting the efficiency of Fenton processes. Especially, the wastewater medium should be acidic for removing organic contaminants such as COD, BOD and color [13]. Because, at higher pH values, free iron species ferric oxyhydroxide decreases due to precipitation [14], while efficiency of the electro Fenton decreases under pH 3 through complex formation of H_2O_2 and Fe^{3+} [15].

In the experimental conditions, where Fe^{2+} concentration was 0.8 g/L, H_2O_2 concentration was 40 g/L and reaction time was 75 minutes, optimum pH was determined as 3.0, COD removal efficiency was 59% and sludge volume was 3.45 kg/m^3 in Fenton process (Fig. 4.). A similar result for the effect of pH was observed in a study by Çetinkaya et al. In PEFP, in the conditions of 1.25 A of current, 40 g/L of H_2O_2, 75 min of reaction time, optimum pH was determined as 3.5, COD removal efficiency was determined as 74% and sludge volume was 0.48 kg/m^3. Also, low pH value was effective on removal of COD by Fenton processes. Sludge volume was decreased while pH increased in all Fenton processes in the study. Similar results were observed in the study of Su et al. 2019 [16]. In FP, sludge volume was 14 times larger, and COD removal efficiency was approximately 15 % less than PEFP, in optimum pH values.

EF processes have been preferred over other electrochemical and oxidation other methods due to the high mineralization rates and treatment efficiency of organic pollutants. This high efficiency is partly due to the continuous regeneration of Fe^{2+} at the cathode. EF process thus avoids Fe^{3+} accumulation in the medium and minimizes the production of iron sludge [17, 18] At the same time another reason why the sludge volume is less than the classical Fenton process is that ultraviolet light can increase the catalytic capacity of the catalysts, increasing the degradation efficiency of organic pollutants and reducing iron sludge production [19, 18].

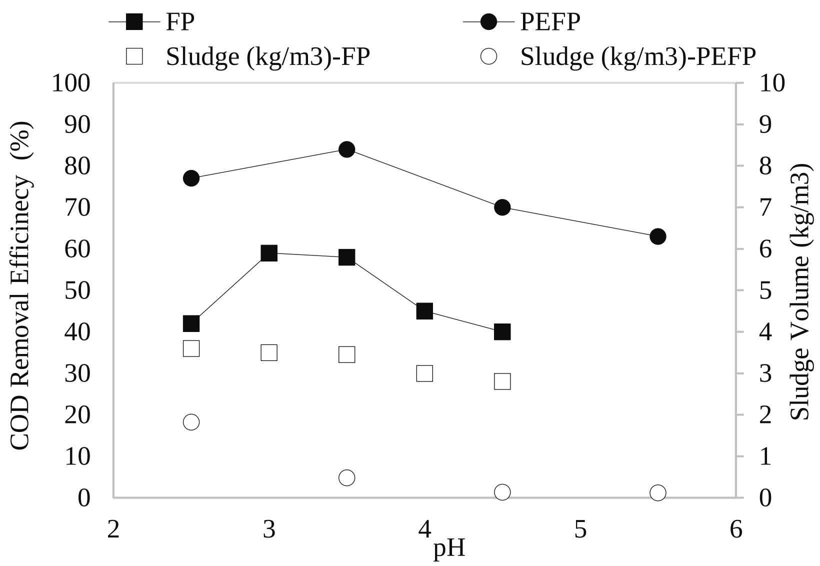


Figure 4 Effect of pH parameter on FP and PEFP
(FP: Fe^{2+} =0.8 g/L, H_2O_2 = 40 g/L, Reaction Time= 75 min),
(PEFP: I=1.25A-8.33 mA/cm^2, H_2O_2 = 40 g/L, Electrolysis Time= 75 min)

3.2 Effect of Fe^{2+} concentration on FP and amper on PEFP

Ferrous ion is an important parameter which influences the Fenton processes. Because while the concentration of Fe^{2+} increases, more hydroxyl radicals produces in the solution [20]. In the study same trends were observed. At the conditions of pH 3.5, H_2O_2 40 g/L, reaction time 75 min, optimum Fe^{+2} concentration was determined as 1.4 g/L, COD removal efficiency was determined as 75%, and sludge volume was 4.5 kg/m^3, in Fenton process. At the conditions of pH 3, 40 g/L of H_2O_2, 75 min of reaction time, optimum current was determined as 1.5 A-9.99 mA/cm^2, COD removal efficiency was determined as 88% and sludge volume was 0.88 kg/m^3, in PEFP (Fig. 5.). At the optimum conditions, sludge volume was 4 times larger, and COD removal efficiency was approximately 13% less than PEFP. Increasing of the Fe^{+2} concentration from 0.6 to 1.4 g/L, COD removal efficiency was enhanced from 28% to 75%. When current increased from 1 to 2 A, the COD removal efficiency was increased from 73% to 89% in PEFP.

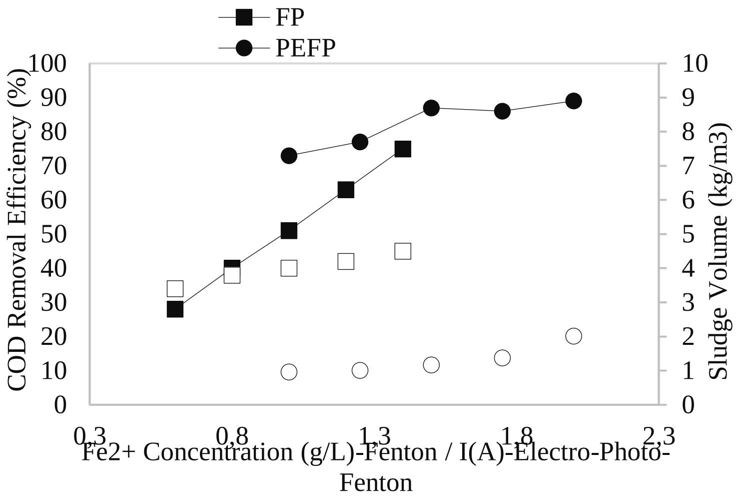


Figure 5 Effect of Fe^{2+} Concentration on FP and Effect of Amper on PEFP
(FP: pH 3.5, H_2O_2 = 40 g/L, Reaction Time= 75 min)
(PEFP: pH 3, H_2O_2 = 40 g/L, Electrolysis Time= 75 min
(1A-6.66mA/cm^2, 1.25A-8.33 mA/cm^2, 1.5A-9.99 mA/cm^2, 1.75A-11.66 mA/cm^2, 2A-13.32 mA/cm^2)

3.3 Effect of H_2O_2 concentration on FP and PEFP

H_2O_2 is a main parameter affecting the degradation efficiency of organic pollutants. Because of that in order to determine the maximum removal efficiency of organic pollutants, the optimal H_2O_2 concentration should be examined. Generally, the removal efficiency of organic pollutants increases with the increasing of H_2O_2 concentration [21, 22]. However, H_2O_2 has to be given in an optimal dose, otherwise, the excessive H_2O_2 not only increases the operating costs, but also increases the scavenging effect of OH by H_2O_2 [23, 24], which has a negative effect on the degradation of organic pollutants. At the conditions of pH 3.5, 1.4 g/L of Fe^{+2}, 75 min of reaction time, optimum H_2O_2 concentration was determined as 50 g/L, COD removal efficiency was determined as 82%, and sludge volume was 3.3 kg/m^3, in FP. At the conditions of pH 3, 1.5 A of current, 75 min of reaction time, optimum H_2O_2 concentration was determined as 70 g/L, COD removal efficiency was determined as 95% and sludge volume was 2.96 kg/m^3, in PEFP (Fig. 6.). In optimum conditions, sludge volume of FP was 1.1 times larger, and COD removal efficiency was approximately 13% less than PEFP.

Increasing of H_2O_2 concentration from 30 to 60 g/L, COD removal efficiency was enhanced from 36% to 82% in FP. Increasing of H_2O_2 concentration from 30 to 70 g/L, COD removal efficiency was enhanced from 79% to 95% in PEFP.

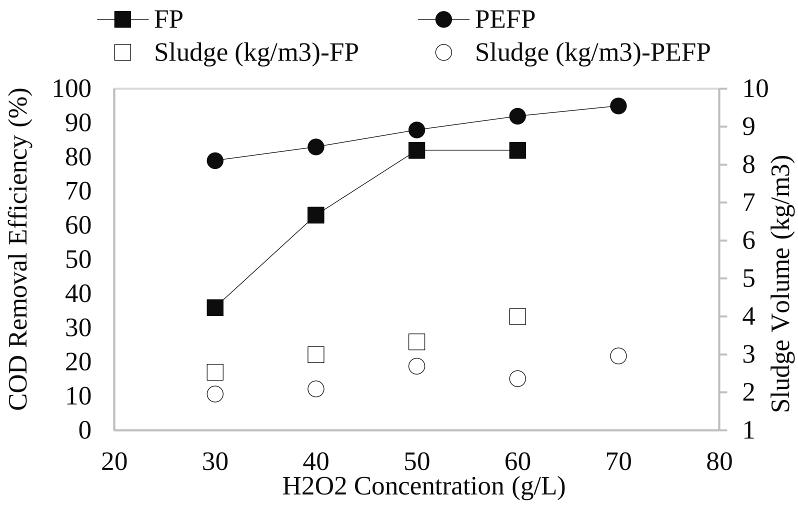


Figure 6 Effect of H_2O_2 concentration on FP and PEFP
(FP: pH 3.5, Fe^{2+} =1.4 g/L, Reaction Time= 75 min)
(PEFP: pH 3, I=1.5A-9.99 mA/cm^2, Electrolysis Time= 75 min)

3.4 Effect of reaction time on FP and PEFP

In Fenton processes, the reaction time has to be optimized for effective COD removal. While iron ions are added at the beginning of the reaction in the Fenton process, the reaction begins with the dissolution of the iron electrodes in the electrochemical Fenton process. For this reason, reaction time and optimization of the electrochemical Fenton process is more important than the classical Fenton process. At the conditions of pH 3.5, 1.4 g/L of Fe^{+2}, 50 g/L of H_2O_2, optimum reaction time was determined as 150 min, COD removal efficiency was determined as 91 %, and sludge volume was 10 kg/m^3, in FP. In the conditions of 3 of pH, 1.5 A of current, 75 min of reaction time, optimum H_2O_2 concentration was determined as 1.5 A, COD removal efficiency was determined as 99 % and sludge volume was 8.3 kg/m^3, in PEFP (Fig. 7.). At the optimum conditions, sludge volume of FP was 1.1 times higher, and COD removal efficiency was approximately 13% less than PEFP. In a study, COD removal efficiency was determined as 97% in an initial concentration of 2000 mg/L from landfill leachate wastewater by photo electro Fenton process [6].

Fenton reaction is seen in Eq. 10. Fe^{2+} oxidizes to Fe^{3+} to formation of hydroxyl radicals [10].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\cdot} \qquad (10)$$

This reaction is propagated by ferrous ion regeneration, which is mainly due to the reduction of the produced ferric species with hydrogen peroxide.

$$H_2O_2 + Fe^{3+} \rightarrow Fe^{2+} + {}^{\cdot}H_2O + H^+ \qquad (11)$$

The rate constant of Eq. (1) varies between 63 and 76 L mol^{-1}s^{-1} whereas the rate constant of Eq. (2) is in the order of 0.01–0.02 L mol^{-1}s^{-1} [25-26-11]. This means that the consumption of ferrous ions is more rapid than the regeneration. This results in the formation of large amount of ferric hydroxide sludge during the process, which occurs additional sludge disposal problems [27, 28, 11]. In the study FP was produced more sludge than PEFP. It is thought that sufficient iron dissolution in the electrochemical Fenton process and also the support of this process with UV causes less sludge formation compared to the FP.

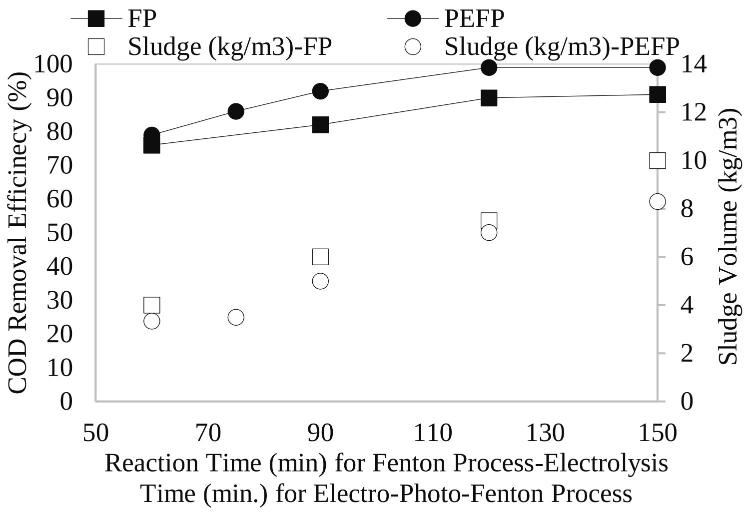
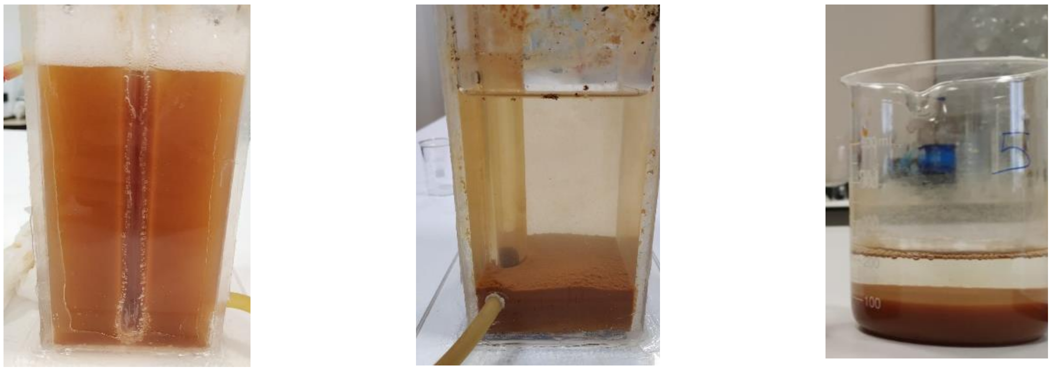


Figure 7 Effect of reaction time on FP and PEFP
(FP: pH 3.5, Fe^{2+} =1.2 g/L, H_2O_2 = 50 g/L)
(PEFP: pH 3, I=1.5A-9.99 mA/cm^2, H_2O_2 = 70 g/L)

Raw wood processing wastewater, sludge occurred in PEFP after neutralization (after 60 min) and sludge occurred in FP after neutralization (after 60 min) was given in Fig. 8a, 8b and 8c., respectively. The obtained sludge of Fenton processes is seen as a reddish-brown color.



a) b) c)

Figure 8 a) Raw wood processing wastewater, b) Sludge occurred in PEFP after neutralization (after 60 min), c) Sludge occurred in FP after neutralization (after 60 min)

In the neutralization phase ferric hydroxide occurs as the main chemical in Fenton sludge. In pH 3.7-7.0 ferrihydrite occurs and in high pH values as >7.0 the Fenton sludge was transformed to goethite. The chemical compounds formed in the precipitate can be explained in Eq. 10-11 [16]. Since the precipitation process was carried out at the pH of 7.5-8 range in the study, it is thought that the fenton sludge obtained may be goethite.

i) Schwertmannite

$$Fe_8O_8(OH)_{8-2x}(SO_4)_x + 2XOH^- \rightarrow 8FeOOH + xSO_4^{2-} \quad (10)$$

ii) Ferrihydrite

$$Fe_5HO_8.4H_2O \rightarrow 5FeOOH + 2H_2O \quad (11)$$

3.5 Reaction kinetics of FP and PEFP

The oxidation of organic pollutants by Fenton procesess are commonly considered as a single-stage process that conforms to pseudo first-order kinetics [29]. In this study, all of the Fenton processes showed the same trend. R^2 values of 0, 1. and 2. order reaction kinetics of FP and PEFP processes are given in Table 2 and Equations are given in Figure

9. It was determined that FP and PEFP were compatible with the 1st order kinetic.

Table 2 R^2 values of the kinetic models

Process	0.	1st	2nd
FP	0.80	0.95	0.72
PEFP	0.78	0.96	0.95

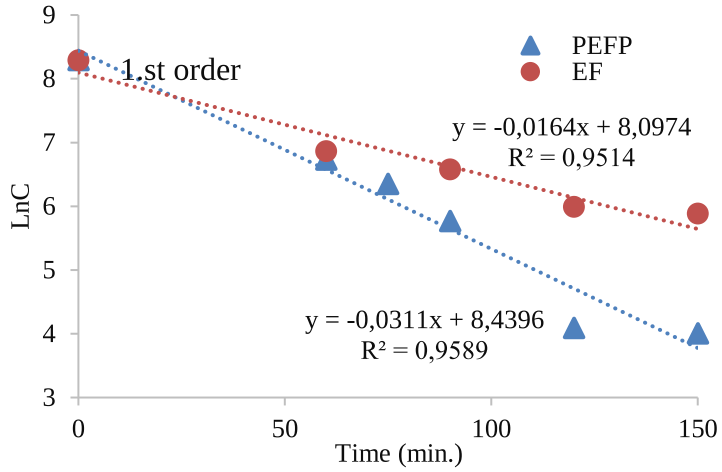


Figure 9 1st order kinetic model of EF and PEFP

4. CONCLUSION

Fenton and Photo Electro Fenton Processes have high removal efficiency in the removal of COD from wood processing industry. In acidic medium conditions all Fenton processes are effective. Optimum values for FP were determined as 1.4 mg/L of Fe concentration, 50 g/L of H_2O_2 concentration and 150 min of reaction time; for PEFP were determined as 1.5 A of amper, 70 g/L of H_2O_2 concentration and 150 min of reaction time. At the optimum conditions, Fenton process and electro-photo-Fenton process discharge COD concentration was 360 mg/L and 40 mg/L, respectively. And sludge production of Fenton process was 20% greater than PEFP.

PEFP can be an effective approach improving the wastewater quality for recovery and/or reuse of the wood processing wastewater due to its higher COD removal efficiency and lower discharge COD concentration and sludge volume when compared to FP.

Acknowledgments
The author would like to acknowledge the reviewers and editors of Sakarya University Journal of Science.

Funding
The author has no received any financial support for the research, authorship or publication of this study.

Authors' Contribution
The first author contributed 100%.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics
The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic

publication environment other than Sakarya University Journal of Science.

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