



# Degradation of phenol and 4-chlorophenol from aqueous solution by Fenton, photo-Fenton, sono-Fenton, and sono-photo-Fenton methods

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## Abstract

In this study, the effects of Fenton process (FP), photo-Fenton process (P-FP), sono-Fenton process (S-FP), and sono-photo-Fenton process (S-P-FP) for the degradation of phenol and 4 chlorophenol (4-CP) from aqueous solution were evaluated. The effects of reaction time, initial concentration, initial pH and Fenton reagent dosages on degradation were investigated. UV-A, UV-B, and UV-C light were used as ultraviolet (UV) light source. In the study with the addition of ultrasound (US), ultrasound with a frequency of 40 kHz and a power of 180 watts was used. While the phenol degradation efficiency was 95% in FP, it reached 99% in P-FP, S-FP and S-P-FP. The 4-CP degradation efficiency was 55.3% in FP, 64.1% in P-FP with UVA, 70% in S-FP and 99% in S-P-FP. This study is especially important in terms of revealing the synergistic effect of different processes in an integrated reactor.

**Keywords** Phenol · 4-Chlorophenol · Fenton · Photo Fenton · Sono Foto Fenton

## Introduction

Phenols are one of the most common pollutants that must be treated carefully before being discharged into receiving waters. Phenol and its compounds are of environmental concern due to their acute toxicity, carcinogenicity, and persistent effects. Phenols have been recognized as priority pollutants by the US Environmental Protection Agency (EPA) and the European Union. The EPA requires that the phenol level of wastewater be reduced to less than 1 mg/L [1].

Phenolic compounds such as phenol and 4-chlorophenol (4-CP) are used in numerous industries, such as tanning, refineries, manufacturing dyes, pharmaceutical, petrochemical industries, papermaking, pesticides, antimicrobial production, and industrial wood preservatives [2, 3]. These are categorized as highly toxic organic pollutants for the environment and humans [4, 5].

Phenol and phenolic compounds cause various health risks due to their acute toxicity and biologically resistant nature [3, 6]. Phenol is defined as one of the priority pollutants due to its ecotoxicological effects [7]. Therefore, it is critical to remove phenol and its derivatives from wastewater before wastewater discharge [8]. 4-CP, on the other hand, is difficult to decompose by conventional biological treatment due to the presence of benzene ring and chlorinated atoms [9]. Common technologies used to remove phenol and 4-CP include; photocatalytic degradation [10], adsorption/biosorption [1, 11, 12], ozonation [13, 14], biological treatment [15, 16], membrane [17], advanced oxidation method [3, 18–21], and ion exchange [22].

Advanced oxidation processes (AOPs) are methods used to remove organic pollutants that are difficult to decompose with conventional processes [23]. The degradation of toxic and biologically resistant compounds with AOPs occurs effectively [24]. Fenton oxidation stands out as a promising method for the degradation of phenol from industrial wastewater. It has been stated that the yield is higher, especially when the phenol concentration is below 100 mg/L [25]. In the Fenton oxidation process, hydroxyl radicals ( $\cdot\text{OH}$ ) are produced from hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) catalyzed by Fe(II) (Eq. 2).  $\cdot\text{OH}$  is a strong oxidant for effective removal of organic pollutants [26]. Mineralization of

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complex chemicals occurs with  $\cdot\text{OH}$ , which has a high oxidative power [27].

The degree of degradation of organic pollutants by Fenton processes accelerates as the production of  $\cdot\text{OH}$  with UV–VIS radiation (photo-Fenton processes) will increase (Eq. 3) [28].

Ultrasound, another AOPs method, is effectively used in the oxidation of organic compounds in water, either alone or in combination with other methods [29]. The ultrasound process is explained by two mechanisms. The first mechanism is pyrolysis in the cavitation bubble, which is expected to be the main reaction for the degradation of polar organic compounds. The second mechanism is the generation of reactive radicals [30].

Although several studies were carried out for the degradation of phenol and 4CP using individual Fenton systems, phenol degradation by comparison between the Fenton-like systems has not been addressed deeply in the literature. Hence, the aim of this study is to evaluate the degradation of phenol and 4-CP in aqueous solutions using different AOPs, including  $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ ,  $\text{Fe}^{2+}/\text{H}_2\text{O}_2/\text{UV}$ ,  $\text{Fe}^{2+}/\text{H}_2\text{O}_2/\text{US}$ , and  $\text{Fe}^{2+}/\text{H}_2\text{O}_2/\text{UV/US}$ . Within the scope of the study, the effects of pH,  $\text{H}_2\text{O}_2$  and iron dose, phenol and 4-CP concentration and reaction time were examined separately. In the optimum conditions obtained, the synergistic effects of the processes were determined, as well as the phenol and 4-CP degradation efficiencies of each process. This study is especially important in terms of demonstrating the synergistic effect of different AOPs on phenol and 4-CP degradation in an integrated reactor.

## Experimental

### Reagents

1000 mg/L phenol stock solution (Merck Company) was prepared in deionized water. Solutions of different concentrations in mg/L were prepared by diluting the stock solution with distilled water.  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (purity 99%) and  $\text{H}_2\text{O}_2$  (purity 35%) stock solution were used as Fenton's reagent. The pH was adjusted using 0.1 N NaOH and 0.1 N  $\text{H}_2\text{SO}_4$ . When the pH reached the desired value, determined amounts of  $\text{Fe}^{2+}$  (as  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) and  $\text{H}_2\text{O}_2$  were added to the sample. It was accepted that the reaction time started with  $\text{H}_2\text{O}_2$  dosing. Experiments were carried out in 250 ml flasks with 100 ml liquid volume. Tubular, compact single-ended low-pressure mercury vapor lamps emitting UV radiation were used. UV-A (365 nm), UV-B (302 nm), UV-C (256 nm) light were used as UV light (Philips brand). Ultrasound studies were conducted with an ultrasound device with a frequency of 40 kHz and a power of 180 watts. pH measurements were made with Adwa AD8000 brand device. All chemicals used in the study were of

analytical standard. The values of phenol and 4-CP solutions were analyzed by spectrophotometer (Merck Spectroquant Pharo 300) for assessing the remaining phenol compound using a direct photometric method (Method 5530) according to Standard Methods [31]. Experiments were repeated three times and mean values are presented. The standard deviation ( $\leq 3\%$ ) and error bars are indicated in the figures. Phenol and 4-CP degradation is calculated by Eq. 1.

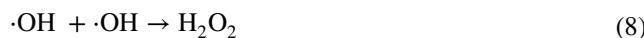
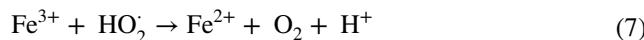
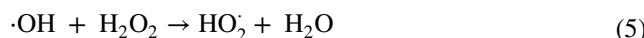
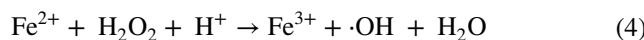
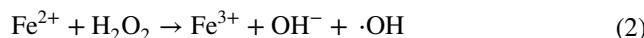
$$\text{Degradation (\%)} = \left( \frac{C_0 - C_e}{C_0} \right) \times 100 \quad (1)$$

where  $C_0$  and  $C_e$  represent initial and equilibrium concentrations of phenol (mg/L), respectively.

### General procedure

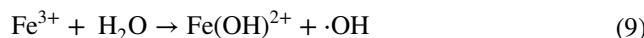
#### Fenton process (FP)

Fenton process is carried out using Fe ion and  $\text{H}_2\text{O}_2$  reagent together.  $\cdot\text{OH}$  is produced by the reaction that takes place. Equation (2) is considered the core of Fenton's chemistry. However, other reactions (Eqs. 4–8) must also be considered in order to understand the whole process [32].



#### Photo-Fenton process (P-FP)

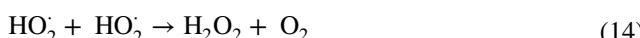
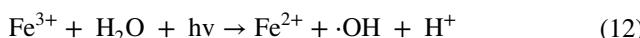
The process in which  $\text{H}_2\text{O}_2/\text{UV}$  and Fe ions are used together is called photo-Fenton type oxidation. As seen in Eqs. 9 and 10 in acidic environment,  $\text{Fe(OH)}_2^+$  complex is formed [27]. Exposure of this complex to UV rays leads to the production of more  $\cdot\text{OH}$  ions (Eq. 11) [33]:



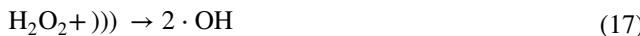


### Sono-Photo-Fenton process (S-P-FP)

S-P-FP is the method in which ultrasonic (US), ultraviolet radiation (UV), and Fenton are used together. In this process,  $\cdot\text{OH}$  production in the aqueous system increases with the degradation rate of pollutants and  $\text{Fe}^{2+}$  regeneration [34]. In S-P-FP, the amount of iron ions in the treated water is low. This is of economic importance. In addition to Eqs. 6 and 8, 12–19 describe S-P-FP [35].



By US,



## Results and discussion

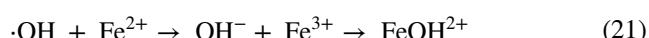
Various parameters such as pH value, catalyst dosage,  $\text{H}_2\text{O}_2$  concentration and phenol concentration were investigated to explore the optimal conditions of phenol mineralization and to investigate the role of each parameter in fenton-like oxidation. Under the optimum conditions obtained, P-FP oxidation took place under three different UV lamps (UV-A, UV-B, UV-C). Then, US was applied to optimum conditions. Finally, S-P-FP was performed by using Fenton UV and US together to determine the synergistic effect.

### The effect of $\text{H}_2\text{O}_2$

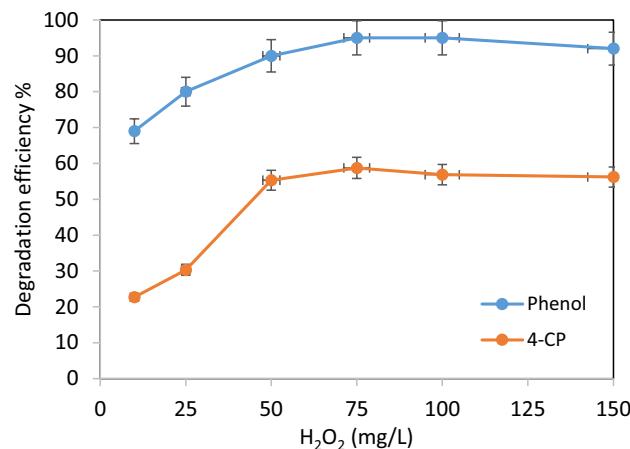
The initial concentration of  $\text{H}_2\text{O}_2$  is an effective and important parameter in overall degradation efficiency for

Fenton-like reactions [36]. The study was carried out under constant conditions of pH 3, time ( $t$ ) 30 min, phenol and 4-CP concentration 100 mg/L and  $\text{Fe}^{2+}$  amount 50 mg/L and 10 mg/L, respectively, to determine the effect of  $\text{H}_2\text{O}_2$  on phenol and 4-CP degradation. In the study, different concentrations of  $\text{H}_2\text{O}_2$  (5, 10, 25, 50, 75, 100 mg/L) were used and the results are given in Fig. 1. Degradation increased with increasing  $\text{H}_2\text{O}_2$  concentration, but the improvement was not evident above 50 mg/L. Degradation for phenol and 4-CP was determined as 69% and 22.6% for 10 mg/L  $\text{H}_2\text{O}_2$ , 90% and 55.3% for 50 mg/L, and 95% and 56.8% for 100 mg/L, respectively.

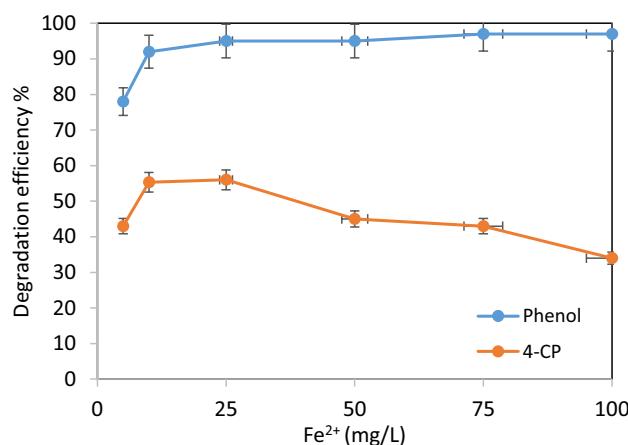
With the increase in  $\text{H}_2\text{O}_2$  concentration, the formation of  $\cdot\text{OH}$  increased and thus the degradation of phenol and 4-CP also increased (Eqs. 20–21). In low amounts of  $\text{H}_2\text{O}_2$ , the rate of phenol degradation was very slow due to insufficient  $\cdot\text{OH}$  production.



However, the continued increase in  $\text{H}_2\text{O}_2$  concentration creates hydroperoxyl radicals with less oxidizing ability as a result of the reaction of hydroxyl radicals and additional  $\text{H}_2\text{O}_2$  molecules (Eq. 22) [37]. Therefore, the degradation does not continue to increase at the same rate despite increasing  $\text{H}_2\text{O}_2$ .



**Fig. 1** Effect of  $\text{H}_2\text{O}_2$  on the degradation. Conditions: concentration 100 mg/L, pH 3,  $\text{Fe}^{2+}$  50 mg/L (for phenol) and 10 mg/L (for 4-CP), time 30 min

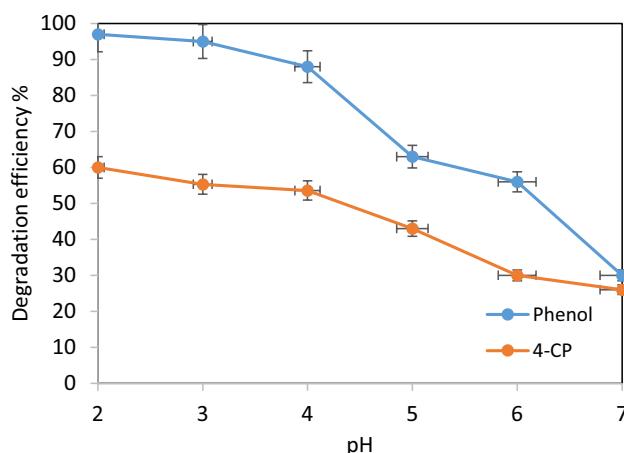


**Fig. 2** Effect of  $\text{Fe}^{2+}$  on the degradation. Conditions: concentration 100 mg/L, pH 3,  $\text{H}_2\text{O}_2$  100 mg/L (for phenol) and 50 mg/L (for 4-CP), time 30 min

### The effect of $\text{Fe}^{2+}$

In the study, different dosages of  $\text{Fe}^{2+}$  (5, 10, 25, 50, 75, 100 mg/L) were used to determine the effect of the amount of  $\text{Fe}^{2+}$  on the degradation of Phenol and 4-CP. Other conditions were applied as; pH 3, time ( $t$ ) 30 min, phenol and 4-CP concentration 100 mg/L and  $\text{H}_2\text{O}_2$  amount 100 mg/L for phenol and 50 mg/L for 4-CP, which was the optimum amount determined in the previous step. The results are given in Fig. 2.

As can be seen in Fig. 2, the degradation rate increased with the increase in  $\text{Fe}^{2+}$  amount. With the use of 5 mg/L  $\text{Fe}^{2+}$ , the phenol degradation was 78%, and the 4-CP degradation was 43%. When the amount of  $\text{Fe}^{2+}$  was increased to 10 mg/L, the phenol and 4-CP degradation was determined as 92% and 55.3%, respectively. Although the amount of  $\text{Fe}^{2+}$  increased in phenol degradation, no significant change was observed after 10 mg/L. The efficiency for 50 mg/L  $\text{Fe}^{2+}$  was 95%, while it was 97% for 100 mg/L  $\text{Fe}^{2+}$ . The degradation for 50 mg/L and 100 mg/L  $\text{Fe}^{2+}$  in 4-CP was 43% and 34%, respectively. While an efficiency of over 90% was obtained in phenol degradation with FP, it was observed that the change in the degradation rate was not significantly dependent on the amount of reagent. The degradation efficiency of 4-CP with FP was lower than with phenol. 4-CP degradation decreased with increasing  $\text{Fe}^{2+}$  amount. At high  $\text{Fe}^{2+}$  dose, the production of  $\cdot\text{OH}$ , usually resulting from the breakdown of  $\text{H}_2\text{O}_2$ , is so high that most of the  $\cdot\text{OH}$  is spent via side reactions before being used for phenol degradation (Eqs. 19–20) [38]. As seen in 4-CP degradation, this causes a decrease in efficiency.



**Fig. 3** Effect of pH on the degradation. Conditions: concentration 100 mg/L,  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L (for phenol), and  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L (for 4-CP), time 30 min

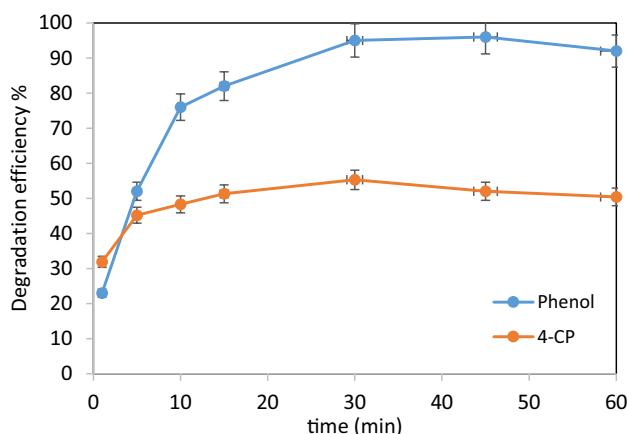
### Effect of initial pH

The solution pH is very important in the phenol degradation process, as it affects the decomposition rate of  $\text{H}_2\text{O}_2$  and the surface charge of the catalyst [15]. Therefore, pH values varying in the range of 2–7 were investigated to determine the effect of pH on phenol degradation efficiency. The results are shown in Fig. 3. Other conditions were held constant against changing pH (for Phenol;  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L, and for 4-CP;  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L;  $t$  30 min, concentration 100 mg/L).

As seen in Fig. 3, the degradation for phenol and 4-CP at different pH values were determined as 95% and 55.3% at pH 3, 63% and 43% at pH 5, and 30% and 26% at pH 7, respectively. Degradation decreased significantly with increasing pH. The optimal pH range of the Fenton reaction is usually around 3 [39]. High acidity in oxidation ( $\text{pH}=3–5$ ) increases free radical formation and organic material oxidation. The acidity of the solution significantly affects the rate of formation of the  $\cdot\text{OH}$  radical [40]. At higher pH,  $\text{H}_2\text{O}_2$  rapidly decomposes into  $\text{H}_2\text{O}$  (Eq. 5) and molecular oxygen (Eq. 23) [41]. Also, at high pH, degradation efficiency decreases due to the precipitation of iron [42].



When the pH is greater than 3,  $\text{Fe}^{3+}$  starts to precipitate as  $\text{Fe}(\text{OH})_3$  and breaks down  $\text{H}_2\text{O}_2$  into  $\text{O}_2$  and  $\text{H}_2\text{O}$  preferably [43]. Also, above pH 5, ferryl ions ( $\text{FeO}_2^+$ ) are formed. Ferril ions are more selective and weaker oxidants than  $\cdot\text{OH}$  [44].



**Fig. 4** Effect of reaction time on the degradation. Conditions: concentration 100 mg/L pH 3,  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L (for phenol), and  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L (for 4-CP)

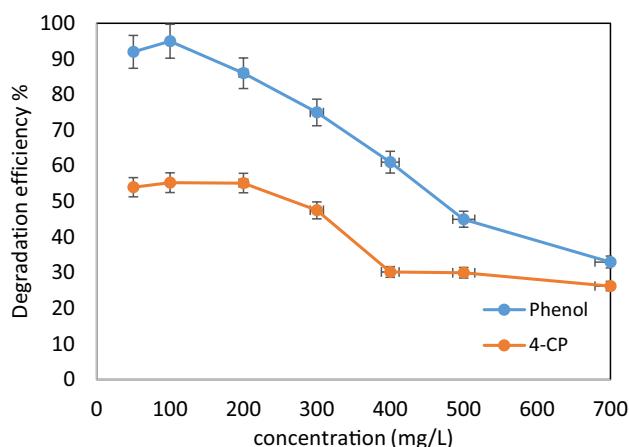
### The effect of reaction time

To determine the effect of reaction time on degradation, degradation rate were investigated at 1, 5, 10, 15, 30, 45, 60 min and under optimum conditions (for Phenol;  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L, and for 4-CP;  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L; t 30 min, pH 3; concentration 100 mg/L). The results are given in Fig. 4.

In Fig. 4, it is seen that rapid degradation takes place from the first minute of the reaction. The degradation efficiency of phenol was higher than 4-CP. Since there was no significant change in efficiency after 30 min of the reaction, the optimum reaction time was determined as 30 min. The phenol and 4-CP degradation efficiencies were determined as 52%, 45.2% at the 5th minute, and 95%, 55.3% at the 30th minute, while it was 92% and 50.4% at the 60th minute, respectively. At the beginning of the reaction, Eqs. (2) and (4) occurred rapidly and the formed  $\cdot\text{OH}$  was used in the decomposition. In the following time, since no additional Fenton reagent was added to the system, the degradation slowed down (Eqs. 5–8). A short degradation period indicates easily degradable organic substances, and a long period indicates the presence of hard-to-degrade organic substances [45, 46]. In this study, the short reaction time showed that phenol and 4-CP are organic substances that degrade easily in fenton-like processes.

### Effect of concentration

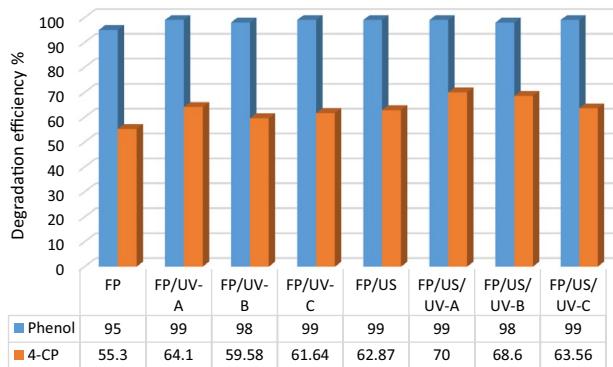
In the study, the previously determined optimum conditions were kept constant and the concentration was determined as 50, 100, 200, 300, 400, 500, 600, 700 mg/L. As the initial concentration of phenol and 4-CP increased, the degradation efficiencies decreased. The degradation efficiency for phenol and 4-CP was determined as 95%, 55.3% for the



**Fig. 5** Effect of concentration on the degradation. Conditions: pH 3,  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L (for phenol), and  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L (for 4-CP), time 30 min

concentration 100 mg/L, 75% and 47.5% for 300 mg/L, and 33% and 26% for 700 mg/L, respectively (Fig. 5).

In AOPs, high concentrations of organic pollutants prevent free radicals from oxidizing partial oxidation products [47]. Therefore, in order to prevent the deactivation of free radicals in oxidation processes, the pollutant/catalyst ratio should be adjusted to an appropriate value. Otherwise, the localization of free radical formation at the site of the active catalyst stops the free radical reaction in oxidation [48]. This causes a decrease in the degradation efficiency inversely proportional to the increasing concentration. In addition, while the phenol concentration increases, the rate of degradation decreases due to the presence of a constant amount of  $\cdot\text{OH}$  [49].



**Fig. 6** Effect of different AOPs on the degradation. Conditions: concentration 100 mg/L, pH 3,  $\text{Fe}^{2+}$  50 mg/L,  $\text{H}_2\text{O}_2$  100 mg/L (for phenol), and  $\text{Fe}^{2+}$  10 mg/L,  $\text{H}_2\text{O}_2$  50 mg/L (for 4-CP), time 30 min

## Synergistic effects (comparative study in FP, P-FP, S-FP, S-P-FP)

In order to determine the effect of different UV light sources on phenol and 4-CP degradation, UVA, UVB, and UVC lights were applied to the optimum conditions determined in the Fenton study. Degradation efficiencies obtained for each light source are given in Fig. 6. Different studies have been conducted on the degradation of acidic phenol solutions under UV light using  $\text{Fe}^{2+}$  [50–52]. In this study, while the phenol degradation efficiency was 95% in FP, it increased up to 98–99% in P-FP. The 4-CP degradation efficiency was 55.3% in FP, the highest value in P-FP was 64.1% with UVA light source and the lowest value was 59.58% with UVB light source.

As seen in Fig. 6, the photo-Fenton process with the addition of UV increased the phenol and 4-CP degradation efficiencies. This is because photolysis of  $\text{H}_2\text{O}_2$  in the presence of UV light produces  $\cdot\text{OH}$  (Eq. 24) and  $\text{Fe}^{2+}$  produces small amounts of  $\text{H}_2\text{O}_2$  [53].



S-FP study was performed with the addition of US at optimum Fenton conditions. The synergistic effects of US, UV and Fenton reagents were investigated with the S-P-FP study. The phenol and 4-CP degradation efficiencies obtained with S-FP were 99% and 62.87%, respectively. In the S-P-FP study, the degradation efficiency increased significantly, especially for 4-CP. Among all processes, the highest 4-CP degradation efficiency of 70% was achieved in S-P-FP where UVA light was used. Phenol degradation was high (98–99%) for all processes. The high efficiency obtained in the S-P-FP process was due to the generation of additional hydroxyl radicals by ultrasound according to Eqs. 17–19 [37].

In S-P-FP, two stages are described in terms of  $\cdot\text{OH}$  formation. The first stage is the formation of  $\cdot\text{OH}$  (due to the reaction of the iron ion with  $\text{H}_2\text{O}_2$ ) and the second stage is the photochemical reaction of an iron ion with water [35].

## Conclusion

The aim of this study is to investigate the degradation of phenol and 4-CP in aqueous solutions using different AOPs such as FP, P-FP, S-FP, and S-P-FP. Within the scope of the study, the effects of pH,  $\text{H}_2\text{O}_2$  and iron dose, phenol and 4-CP concentration, different UV light sources and reaction time were examined separately. In the optimum conditions obtained, the synergistic effects of the processes were determined, as

well as the phenol and 4-CP degradation efficiencies of each process. Optimum conditions obtained in the study were; pH 3, t 30 min, concentration 100 mg/L,  $\text{Fe}^{2+}$  50 mg/L for fenol and 10 mg/L for 4-CP and  $\text{H}_2\text{O}_2$  amount as 100 mg/L for phenol and 50 mg/L for 4-CP, respectively. The efficiency of phenol degradation for all processes was higher than the 4-CP degradation. Phenol degradation was 95% with FP, 98–99% with P-FP, 99% with S-FP and S-P-FP. 4-CP degradation was determined as 55.3% with FP and 64.1, 59.58, and 61.64% with P-FP with UVA, UVB, UVC, respectively. While it reached 62.87% with S-FP and 70, 68.6, and 63.56% with S-P-FP with UVA, UVB, UVC, respectively. The highest 4-CP degradation was 70% under UVA light in S-P-FP. The study showed that Fenton-like processes can be used as an efficient and reliable method for phenol and 4-CP degradation from aqueous solutions.

## Declarations

**Conflict of interest** The author declares that there are no financial or commercial conflicts of interest that could have appeared to influence the work stated in this paper.

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