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Fenton's oxidation of para-chlorophenol with zero-valent iron

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ABSTRACT

Para-chlorophenol (para-cp), a chlorophenol derivative, is potentially a pollutant that represents an important environmental problem, because of its toxic effects on the environment. In this study, the removal of para-cp from synthetic wastewater using a Fenton-like (Fe°/H_2O_2) system has been investigated. A Fenton-like oxidation process was performed on the synthetic wastewater and the optimum conditions were determined and calculated. According to the results of this experiment, removal efficiency under the optimum conditions of $[Fe^{\circ}] = 0.0035$ M, $[H_2O_2] = 0.0014$ M, and $[pH] = 4.0$ was 93.89% in 5 min. Increasing chloride ions from 0 to 4.6 mg/l and decreasing UV_{215} absorbance from 1 to 0.4 indicated that the generated intermediates' toxicity was reduced and the degradability was enhanced.

Keywords: Para-chlorophenol; Fenton-like process; Wastewater; Fe°/H_2O_2; Hydroxyl radical

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1. Introduction

Para-chlorophenol (para-cp) is a toxic, colorless, and somewhat acidic substance [1]. Para-cp is widely used as a solvent for the extraction of sulfur and nitrogen from coal, as an intermediate for the synthesis of insecticides, preservatives, antiseptics, disinfectants, dyes, and drugs, or as a solvent in the refinement of oils. Moreover, this compound is used in the pesticide industries and is used for the characterization of the wastewater discharged from these industries by para-cp concentrations varying from 500 to 4,000 $mg\,l^{-1}$ [2,3]. This compound is a toxic organic contaminant for humans and aquatic life causing oxygen demand in receiving waters and is among the most prevalent forms of organic chemical pollutants in industrial wastewaters [4,5].

The chemical and physical properties of para-cp are summarized in Table 1. Para-cp is listed among the 65 high-priority pollutants by the US Environmental Protection Agency because of its toxicity. It causes an unpleasant taste and odor in the drinking water and at concentrations higher than 1 $mg\,l^{-1}$, the pollutant is highly toxic to living organisms. Para-cp has been reported to be present in the effluent of sewage treatment plants (STPs), which indicates its poor biodegradability in the municipal sewage and STPs. Para-cp is very resistant to conventional chemical and biological treatments. Due to the stability of the C–Cl bond in the ring and its recalcitrant nature, para-cp is difficult to remove from the environment by conventional pollutant treatment technologies, such as

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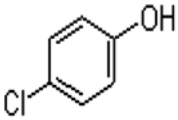
Table 1
Physical and chemical properties of para-cp

Formula	$C_6H_5N_2ClO$
Mol. mass	128.56 g/mol
Appearance	Solid, white to slightly yellow
Molecular structure	
Alternative name	Para- chlorophenol, 4-chloro hydroxybenzene, 4- chlorophenol
Melting point	218.5℃
Density	1.265 g/cm^3

biological treatment [6–8]. Para-cp was, therefore, chosen as a model compound in this study because of its high toxicity, its resistance to conventional chemical and biological treatments, and the serious risks it poses to the environment. Therefore, it is essential that new wastewater treatment technologies be used in order to reduce the negative impact on the water bodies and to facilitate recycling and reuse of wastewater [9,10].

Treatment methods used for the removal of para-cp from process or waste streams include biological treatment, incineration, adsorption over activated carbon, air stripping, etc. Due to para-cp's high toxicity for micro-organisms, filtration of wastewaters containing this pollutant is difficult by biological treatment. Also incineration, adsorption, or air stripping are not proper processes because of the emission of other hazardous compounds during the incineration process and post-treatment is required for adsorption or air stripping processes [2,11]. Among the various methods available, the advanced oxidation processes (AOPs) are among the new technologies used to remove organic contaminants and have been successful in degrading most of the organic compounds present in polluted waters [12–14]. The reason for the use of AOPs is the inability of biological processes in the removal and treatment of toxic organic pollutants and the production of hydroxyl radicals ($^\bullet$OH) which demonstrate great efficacy in breaking down organic matter and are strong and relatively non-selective oxidants that react with most organic compounds [15]. $^\bullet$OH is effective in degrading organic pollutants and is capable of mineralizing them ultimately to CO_2 and H_2O [15,16]. Fenton oxidation, one of the AOPs that involves the generation of the highly reactive hydroxyl radical ($^\bullet$OH) having stronger oxidation potential (2.80 V) than ozone (2.07 V), is of current interest in the treatment of various industrial wastewater components including aromatic amines and a wide variety of dyes as well as many other substances, e.g. pesticides and surfactants [2,13,14]. Therefore, the Fenton oxidation process has been widely used to treat a variety of wastes such as those associated with the textile and chemical industries. The advantages of the Fenton reagent are as follows: it is fast, inexpensive, and capable of achieving complete degradation (mineralization) of pollutants to less harmful byproducts, no energy input is necessary to activate hydrogen peroxide, its use is high efficient, the process of destroying the contaminants by it is rather simple (eventually leaving no residues), it is stable in treating a wide range of substances, and it is not necessary to use special equipment. However, the disadvantages of the Fenton reagent are the production of a substantial amount of $Fe(OH)_3$ precipitate and additional water pollution caused by the homogeneous catalyst that is added as an iron salt, which cannot be retained in the process [17]. To solve these problems, oxidation of para-cp in an aqueous medium using the Fenton reagent in the presence of iron powder has been studied. A Fenton reagent in the presence of iron powder (Fe°/H_2O_2) is an AOP that uses iron powder (Fe°) in reaction with H_2O_2, at an optimum pH, to produce $^\bullet$OH which oxidizes organic matter. The advantages of this process include the following: it is of low-cost and has effectiveness; iron powder can be used as a discard material; it is easy to operate; it allows the retention of the catalyst in the process; it is highly capable of degrading contaminants; and it is easily accessible. Therefore, it could be used to replace iron salts as a catalyst in the Fenton reaction [18,19]. The Fe°/H_2O_2 treatment process not only leads to oxidation but also to coagulation by the formation of $Fe(OH)_3$. In a Fe°/H_2O_2 process, chemical coagulation acts as a refining step after Fe°/H_2O_2 oxidation [20]. The reaction between $^\bullet$OH and para-cp is presented in Fig. 1. The objectives of this project were: (1) to study the oxidation of para-cp in an aqueous medium using the Fenton reagent in the presence of iron powder, (2) to evaluate the effects of pH, hydrogen peroxide, and Fe° concentrations, and (3) to study the produced intermediate products of Fenton oxidation of para-cp with zero valent iron.

2. Materials and methods

2.1. Reagent

For all experiments, the synthetic wastewater contained a concentration of 10 mg/l of para-cp. The para-cp (Formula: $C_6H_5N_2ClO$ and molecular

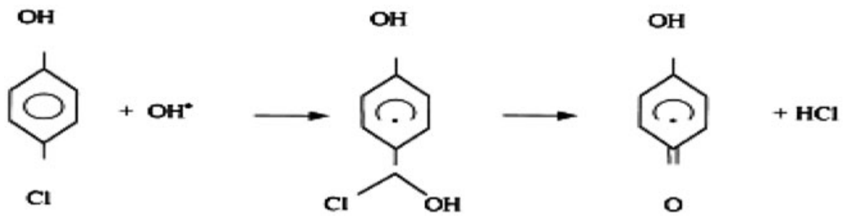


Fig. 1. Schematic representation of the reaction between •OH and para-cp [21].

weight = 128.56 g mol^{-1}), iron powder (95%) at a particle size of 70–100 μm, hydrogen peroxide with the technical grade (30% w/w and density of 1.13 kg l^{-1}), sulfuric acid (96%), and NaOH (98%) for pH adjustment were purchased from the Merck Company. All of the materials used in bench-scale tests were analytical grade and all the solutions were prepared with distilled water.

2.2. Analytical methods

This study was performed to determine the effects of different parameters, such as Fe° and H_2O_2 dosages, and the pH, in the removal of para-cp by a Fenton-like process. All experiments were done using the Standard Method. The pH measurement was performed using a pH meter (Model Jenway 3305). Initial H_2O_2 and iron concentrations in the solution were determined by iodometric titration and phenanthroline methods, respectively [22,23]. The UV absorbance was determined to be 215 nm. Additionally, the chloride ion was analyzed according to the 4500-B method [23]. Each experiment was conducted three times.

2.3. Fenton-like experiments

Batch experiments were performed in a cylindrical glass reactor with a magnetic stirrer using a constant speed of 180 rpm. An aqueous solution of the para-cp was prepared (10 mg/l) and subjected to a Fenton-like process. Then, the removal efficiency of para-cp by the Fenton-like process was determined at optimum values of pH, Fe°, and H_2O_2 dosages and contact time. The experiments were performed in three steps. In the first step, the initial concentrations of Fe° (0.002 M) and H_2O_2 (0.002 M) were kept constant (from run to run) to determine the optimum pH of the solution. In the second step, having the pH fixed at the optimum level, which was determined during the first step, and with the same iron powder concentration (0.002 M), the optimum level for H_2O_2 was measured and determined. Finally, in the third step, the optimum concentration for the iron powder was determined, while using pH and H_2O_2 optimum levels. After each step, the samples were centrifuged at 3,000 rpm (for 3 min) and the para-cp residual amount was determined by the 5530D method mentioned in the Standard Method at 500 nm, using a spectrophotometer [22]. All the experiments were performed at a temperature of 20°C.

3. Results and discussion

3.1. The effect of solution pH

Fig. 2 shows the results of the determination of the optimum pH in the para-cp removal by the Fenton-like process. To determine the optimum pH, pH was changed from 1 to 7 and the dosages of H_2O_2 and Fe° were controlled at 0.002 and 0.002 M, respectively. It was observed that the para-cp removal efficiency was the highest at a pH of 4.0. At this pH, the Fenton-like process was most effective, while at pHs greater and less than 4, a rapid decrease in para-cp removal was observed ($p < 0.05$). Increased removal efficiency at a pH of 4.0 might be due to the production of ferric ion

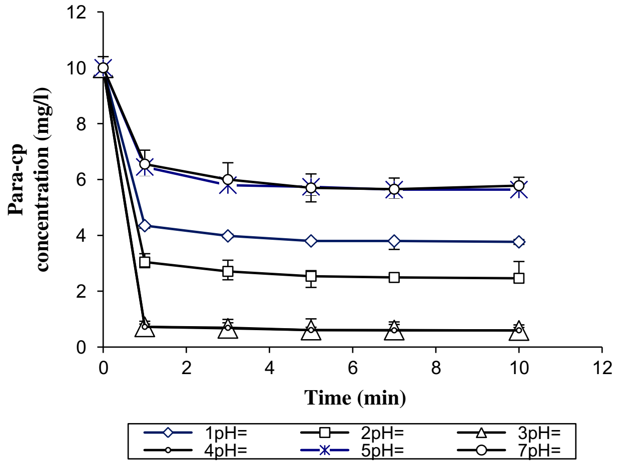


Fig. 2. The effect of pH on the removal of para-cp at different times by Fenton-like oxidation (H_2O_2 = 0.002 M and Fe° = 0.002 M).

and $^\bullet OH$ according to Eqs. 1 and 2. Moreover, at this pH, the ratio of the dissolved Fe^{2+} increases [24].

According to Hoon Hang (2008), the substantial decrease in the efficiency of para-cp removal (at $pH < 4$) happens because at this pH, the high level of H^+ ions may inhibit the generation of $^\bullet OH$ (due to the scavenging effect of $^\bullet OH$ by H^+) as shown in Equation 1 and $FeOH^{2+}$ is being the dominant form in the solution at $pH < 4$ which reacts more slowly with H_2O_2 [25].

$$^\bullet OH + H^+ + e^- \rightarrow H_2O \tag{1}$$

At pH values higher than 4.0, the loss of reactivity is due to the precipitation of Fe^{3+} as hydrous oxyhydroxide ($Fe_2O_3{\cdot}nH_2O$) and at alkaline solutions, H_2O_2 is unstable and decomposes to give O_2 and H_2O [24].

In a study similar to the present one, Zhouet al used a Fenton-like process to remove para-cp from wastewater. In both experiments, 4CP was quickly and completely degraded. At initial pHs of 3 and 4, the degradation was completed within 8 and 30 min, respectively. At a pH of 5, the removal rates dropped to 6.5% for 4CP. In another experiment, at a pH of 6.5 and a reaction time of 6 h, only 3.8 percent of 4CP could be removed. Although 4CP removal levels seem almost the same at pHs of 5 and 6.5, the involved processes seem to be different. At a pH of 5, similar to lower pHs, the solution color changed from colorless to green and finally to brown, while at a pH of 6.5, the solution remained colorless throughout the process. It seems as if no iron dissolution occurred at a pH of 6.5. Ferous measurements were 3.48 and 0.22 mg/l, respectively. The degradation of H_2O_2 was also different in these experiments. At a pH of 6.5 and a reaction time of 6 h, 12.5% of the H_2O_2 was degraded, while at a pH of 5 and a reaction time of 45 min, 70.4% of the H_2O_2 was degraded [26]. Hongyun et al. made use of a Fenton-like process to completely mineralize sulfathiazole. Varying levels of pH (3.5, 5.5, 7.0, and 9.0) were used to examine the pH effect. As the pH increased, the removal efficiency decreased. The k value for each pH level was 0.0341, 0.0169, 0.0069, and 0.004 min^{-1}, respectively. More than 95 and 84% of sulfathiazole were removed at pH 7.0 and pH 9.0, respectively, despite the fact that at higher pHs, the degradation rate was lower [27].

3.2. Effect of H_2O_2

To investigate the optimum concentration of hydrogen peroxide for para-cp removal from the solution, H_2O_2 was changed from 0 to 0.0016 M (pH was controlled at 4.0 and the Fe° dosage was 0.002 M). The results of the para-cp removal by the Fenton-like process, for the determination of optimum H_2O_2 concentration, are presented in Fig. 3. Para-cp removal efficiency was highest at 0.0014 M of H_2O_2 and a substantial decrease in the efficiency of para-cp removal was observed at H_2O_2 amounts less than 0.0014 M. There was no significant difference between the H_2O_2 dosages of 0.0014 and 0.0016 M ($p < 0.05$). This inhibitory effect at the higher dosages of H_2O_2 could be explained by the following: In the absence of organic substrates, H_2O_2 acts as a scavenger of the $^\bullet OH$ to produce the perhydroxyl radical ($HO_2^\bullet$) according to reaction 2 [28].

$$H_2O_2 + {}^\bullet OH \rightarrow H_2O + HO_2^\bullet \tag{2}$$

The decrease of para-cp removal efficiency, with reduction of H_2O_2 concentration from 0.0014 to 0 M, could be due to partial oxidation of Fe^{2+} in H_2O_2 concentrations being less than the optimum values [29]. Celalettin et al. used a Fenton-like process to decolorize azo dyes. A range of 5–30 mg/l was selected in the optimization study. Up to a dosage of 20 mg/l, the decolorization efficacy of both dyes increased. At dosages higher than this, the efficacy decreases. This can be explained by the scavenging behavior of excess H_2O_2 for RO 127. The increase in the efficacy for RY 145 was insignificant. For both dyes, an optimum concentration of 20 mg/l was selected [30]. Hongyun et al. used a Fenton-like process to mineralize sulfathiazole. At higher concentrations of H_2O_2, more sulfathiazole was degraded, such that at two different concentrations of H_2O_2 in the range 0.002–0.8 M, the removal rates were 0.0014 and 0.0579 min^{-1},

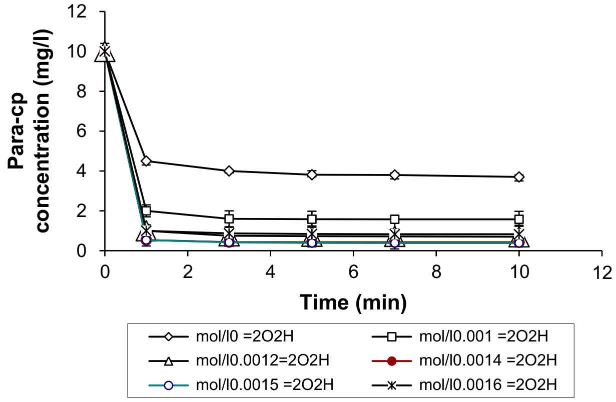


Fig. 3. The effect of H_2O_2 on the removal of para-cp at different reaction times by Fenton-like oxidation (Fe° = 0.0002 M and pH = 4. 0).

respectively (iron dosage 3 g/l; pH = 3. 5). The H_2O_2 concentration was fixed at 0.39 M at a pH of 3. 5. The K value increased from 0.0073 to 0.0603 min^{-1} as the iron dosage increased from 1 to 5 g/l [27].

3. Effect of Fe

Fig. 4 presents the results of the determination of the optimum Fe$^\circ$ concentration in the removal of para-cp by a Fenton-like process. To investigate the optimum concentration of Fe$^\circ$ for the removal of para-cp from solutions, the dosage of Fe$^\circ$ was varied from 0.0009 to 0.0071 M (During this stage, pH was controlled at 4. 0 and the H_2O_2 dosage was 0.0014 M). As presented in Fig. 4, increasing the Fe$^\circ$ has a great effect on the removal efficiency of para-cp. Experiments show that in the range of 0.0009–0.0035 M Fe$^\circ$, para-cp removal efficiency significantly increases with an increase in the dosage of Fe$^\circ$ (the para-cp removal efficiency by the Fenton-like process was 70.5 and 93.89% for 0.0009 and 0.0035 M, respectively) ($p < 0.05$). The highest para-cp removal efficiency (93.89%) was achieved at an Fe$^\circ$ dosage of 0.0035 M and there was no significant difference between the dosages of 0.0035 and 0.0071 M (a slight increase from 93.89 to 93.9% was observed). This can be explained by the fact that in overdoses of Fe^{2+} ions (formed from Fe$^\circ$ in the Fe$^\circ$/H_2O_2 process) OH radicals may be scavenged by participating in reactions with Fe^{2+} as in reaction 3 (the formation of orange-brown iron precipitate (Fe $(OH)_3$flocs)) [29]. According to Eqs. 4 and 5, it must be noted that Fe(II) regenerated through the reaction of Fe(III) with H_2O_2 that leads to an increase in the removal efficiency. Fanaet al used a Fenton-like process in the removal of sulfasalazine. As the iron concentration increased from 0.10 to 0.35 mM, the degradation ratio of sulfasalazine increased from 9.1 to 85.3% in 20 min. At iron levels higher than those mentioned, the degradation ratio decreased quickly. The smallest sulfasalazine residue could be observed at an iron concentration of 0.20 mM in the Fenton-like reaction and at a reaction time of 30 min. As already known, the Fenton process degradation efficacy depends on the amount of hydroxyl radical produced [31].

$$Fe^{2+} + {}^{\bullet}OH \rightarrow OH^- + Fe^{3+} \tag{3}$$

$$Fe^{3+} + H_2O_2 \rightarrow H^+ + FeOOH^{2+} \tag{4}$$

$$FeOOH^{2+} \leftrightarrow OH_2^{\bullet} + Fe^{2+} \tag{5}$$

3.4. A Study of the produced intermediate products

To study the degradability enhancement of para-cp by the Fenton-like process, chloride ion and UV_{215} absorbance analyses were performed on some samples. As presented in Fig. 5, UV_{215} absorbance decreased from 1 to 0.4, as the oxidation time increased. This may be due to the generation of non-phenolic intermediates and the reduction of phenolic compounds in the solution. In addition, as shown in Fig. 6, as oxidation time increases from 0 to 5 min, chloride ion is increased from 0 to 4.6 mg/l. It demonstrates that the generated intermediate products have a less chlorinated nature. An increase in the amount of chloride ions and a reduction in the UV_{215} absorbance indicated that the toxicity of the generated intermediates is reduced and degradability is enhanced [32,33].

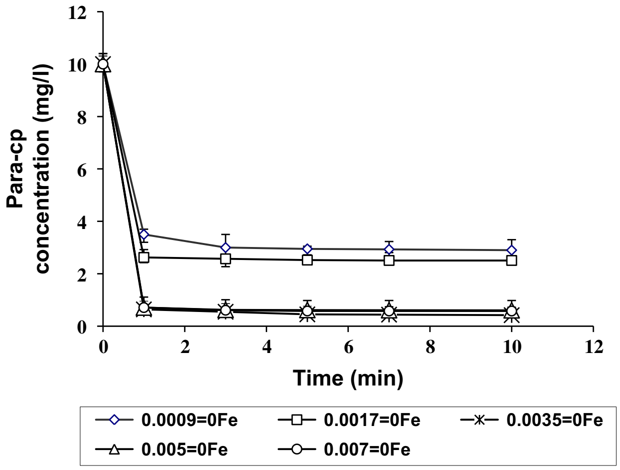


Fig. 4. The effect of Fe$^\bullet$ on the removal of para-cp at different times by Fenton-like oxidation ($H_2O_2 = 0.0014$ M, pH = 4. 0).

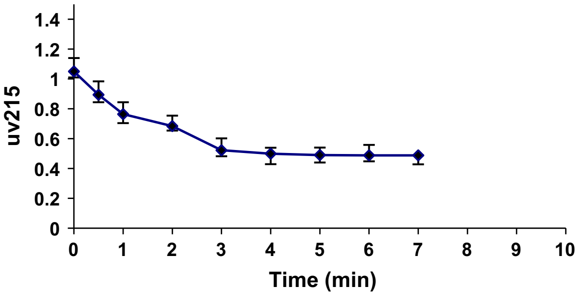


Fig. 5. UV215 absorbance with total phenolic compounds at the various reaction times (pH = 4, $H_2O_2 = 0.0014$ M, Fe$^\circ$ = 0.0035 M).

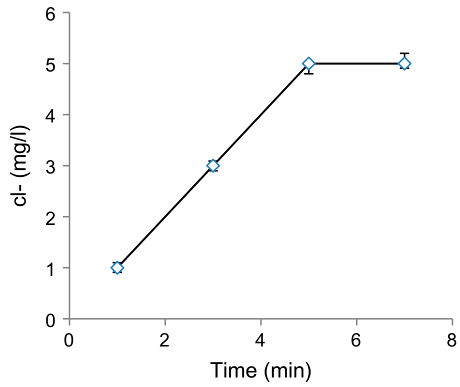


Fig. 6. Chloride ion production in Fenton-like oxidation at different reaction times.

4. Conclusion

The effectiveness of the Fe°/H_2O_2 for the degradation of the para-cp in aqueous solutions was studied. The results confirm that Fe° can be used as a heterogeneous catalyst for the activation of hydrogen peroxide in the Fenton process. The Fe°/H_2O_2 process was found to be highly efficient in the degradation of wastewater containing 10 mg/l para-cp. These results indicate that a Fenton reagent in the presence of iron powder (Fe°/H_2O_2) has obvious advantages in comparison with the Fe^{2+}/H_2O_2 reaction including: this process is fairly cost-effective (since the iron powder can be obtained as discard material); it is easy to operate; it permits the retention of the catalyst in the process and, as a result, the catalyst (iron powder) can be used repeatedly; and it provides great possibilities for the application of the Fenton reagent. Based on the results obtained, the optimal conditions for the Fenton-like process were determined to be [Fe°] = 0.0035 M, $[H_2O_2]$ = 0.0014 M, and pH = 4.0. The Fenton-like process showed a 93.89% removal efficiency under the selected conditions.

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