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Removal of 2,4-dichlorophenol from aqueous solution using ultrasonic/H_2O_2

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ABSTRACT

The effect of ultrasound/hydrogen peroxide on the removal efficiency of 2,4-dichlorophenol (2,4-DCP) from aqueous solutions was investigated. The effects of solution pH, sonication time, H_2O_2 concentration, and *tert*-butanol (*t*-BuOH) concentration were also examined on process efficiency in 2,4-DCP degradation. Ultrasonic treatment at 20 kHz in combination with hydrogen peroxide resulted in increased removal efficiency of 2,4-DCP. The removal efficiency was improved with increasing sonication time, and the maximum efficiency was obtained at pH of 3. *t*-BuOH decreased the removal efficiency of 2,4-DCP under optimum conditions by quenching hydroxyl radicals. According to the obtained results, 74.6% chemical oxygen demand removal was achieved with US/H_2O_2 process after 90 min of reaction time at pH of 3 and H_2O_2 concentration of 0.1 mol/L. The decomposition kinetic data fitted well with the pseudo-first-order kinetic model with a rate constant of 0.025 min^{-1}.

Keywords: Ultrasonic process; Hydrogen peroxide; 2,4-dichlorophenol

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1. Introduction

Chlorophenol and other phenol-based compounds are widely used in various formulations such as preservatives, herbicides, pesticides, and solvents. These compounds are present in industrial wastewater (i.e., plastic, petrochemical, and pulp and paper wastewater). Many phenolic compounds are identified as possible carcinogens; long-term exposure to these compounds has been associated with the risk of liver and kidney cancers as well as neurological defects [1,2]. Chlorophenols are easily absorbed by dermal contact, respiration, and digestion. They accumulate primarily in the liver and kidneys. Even in small quantities they can be stored in muscles, adipose tissue, and brain [3,4].

According to the United States Environmental Protection Agency (USEPA) Clean Water Act, 2,4-dichlorophenol (2,4-DCP), as one of the most common chlorophenol compounds, has been listed as a priority toxic substance, which can be found in contaminated waters at various concentrations (in the range 150–200 mg/L) [5]. 2,4-DCP is characterized by its high toxicity to living organisms. Even the small amount of 2,4-DCP if released into the environment can pose serious consequences [6].

Due to the adverse effects on human health and the high level of toxicity to aquatic life, it is necessary to treat the effluent containing these compounds prior to discharge into water bodies.

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Several physical and chemical methods including ion exchange, adsorption, chemical oxidation, chemical coagulation, and biological degradation have been applied for treating chlorophenolic wastewater [4–11]. However, these methods are sometimes limited due to their high cost, increased sludge production, low efficiency, and the lengthy time required to achieve the desired efficiency [11]. Moreover, due to their aromatic structure, phenolic compounds have toxic effects on microorganisms, which play a significant role in the biological treatment of wastewater. Therefore, these compounds are resistant to biodegradation even at low concentrations [12].

Recently advanced oxidation processes (AOPs) have been considered for the treatment of hazardous pollutants due to their high efficiency and ease of use. AOPs have been defined as those oxidation processes, which are primarily based on the generation of the hydroxyl radical, and are successfully used to decompose many toxic pollutants in aqueous solutions, without producing additional hazardous by-products or sludge [13]. AOPs generally use strong oxidants like hydrogen peroxide (H_2O_2) or ozone (O_3), and irradiation sources like UV light or ultrasounds, separately or in combination [4,12].

Recently, several studies have investigated developing ultrasonic (US) processes alone or in combination with oxidizing agents for the degradation of 2,4-DCP [14,15]. Phenolic compound removal using ultrasound is based on oxidative degradation by free radical attack; mainly, the hydroxyl radical acts as a strong oxidant (2.87 V) [16]. Drijvers et al. [12] investigated the effects of ultrasound/H_2O_2/CuO processes on the degradation of phenol. Zhou et al. [15] investigated the degradation of 2,4-DCP using ultrasound/Fenton.

The coupling of ultrasound with hydrogen peroxide (US/H_2O_2) can enhance the efficiency of generated free radicals and, consequently, can result in a higher degradation rate. Hydrogen peroxide (H_2O_2) is a powerful oxidant that can react directly or indirectly with organic materials in aqueous solutions. Oxidation in indirect reactions is associated with free radicals formed from H_2O_2 decomposition. Previous studies have shown that in the presence of H_2O_2, increased level of hydroxyl radical generation increases the efficiency of the US process [16,17]. However, to my knowledge there have been no studies done on ultrasound enhancement for the degradation of chlorophenol compounds using an US/H_2O_2 system.

2. Objectives

This study investigated the effects of an ultrasound/hydrogen peroxide system as a new technology of AOP on 2,4-DCP removal from aqueous solutions. Moreover, the effects of some operation parameters such as solution pH, H_2O_2 concentration, and *tert*-butanol concentrations on the process efficiency were investigated. The efficiency of US/H_2O_2 in chemical oxygen demand (COD) removal was also studied under optimal conditions.

3. Materials and methods

3.1. Chemicals

In this study 2,4-DCP was chosen as the target pollutant. All chemicals used including 2,4-DCP (≥99.5%), ammonium hydroxide (0.5 N), potassium ferrocyanide, 4-amino anti-Perrin (30%), hydrogen peroxide solution (30%), phosphate buffer, sulfuric acid, and sodium hydroxide were commercially available and purchased from Merck, Germany. Aqueous solutions were prepared using ultrapure water (conductivity 0.056 µS/cm) obtained from Millipore MilliQ (Bedford, MA, USA).

3.2. Pilot study

Experiments were carried out in a 5-L stainless steel tank, equipped with a digital US device (model LUC-405, Daihan Labtech Co., Ltd., Korea), at a constant power of 350 W with a frequency of 40 kHz. Mixing equipment and other measuring devices (pH and temperature) were also embedded in the reactor at 90 min. Fig. 1 shows the pilot used in this study.

3.3. Batch experiments

Experiments were carried out in the initial 2,4-DCP concentration of 0.3 mol/L with neutral pH. The effects of other parameters such as pH, H_2O_2 concentration, and *tert*-butanol concentrations as an $OH^\bullet$ scavenger were also investigated. A stock solution of 2,4-DCP (1,000 mg/L) was prepared. Samples with different concentrations of 2,4-DCP were obtained by diluting the stock solution in distilled water. The experiments were carried out as expressed in Table 1.

3.4. COD measurement

COD tests were performed according to Standard Methods 410. Each test was carried out under optimal conditions [18].

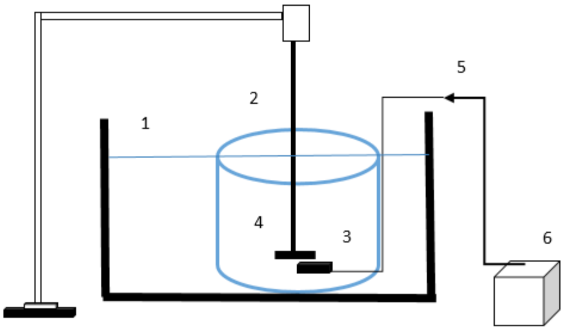

Fig. 1. Schematic of pilot study: 1 – ultrasonic chamber, 2 – reactor, 3 – mixer, 4 – mixer holder, 5 – air hose, and 6 – air pump.

Table 1
Experimental stages

	Parameters	Range
1	pH	3, 7, and 10
2	H_2O_2 concentration, mol/L	0.04, 0.05, 0.08, 0.09, and 0.1
3	*tert*-Butanol concentration, mol/L	0.04
4	COD removal, mg/L	–

3.5. Analysis

The 2,4-DCP concentration at regular certain reaction intervals was analyzed using UV–Vis spectroscopy at the wavelength of 500 nm according to the Standard Methods for the examination of water and wastewater [16]. The DCP removal efficiency was defined as follows (Eq. (1)):

$$R\% = \frac{(C_0 - C_t)}{C_0} 100 \quad (1)$$

where C_0 is the initial concentration of the DCP (mol/L); C_t is the instant concentration of the DCP (mol/L); and $R\%$ is the percentage of DCP removal.

4. Results and discussion

4.1. Effect of initial pH value on US/H_2O_2 process

Fig. 2 shows the effect of the initial pH value on the removal efficiency of 2,4-DCP. The experiment was conducted at initial 2,4-DCP concentration of 0.3 mol/L and an initial H_2O_2 concentration of 0.004 mol/L. The pH value was adjusted to 3, 7, and 10. As shown in Fig. 2, the 2,4-DCP removal efficiency increased with the increase in sonication time and the decrease in pH level. The solution pH has a direct impact on hydroxyl radical generation and thereby can affect the oxidation process. After 90 min of reaction, in 0.004 mol/L of H_2O_2, the removal efficiency of 2,4-DCP was 25.8% and 13.67% at pH values of 3 and 10, respectively (data not shown). Due to low efficiency at 0.004 mol/L of H_2O_2, experiments were repeated at 0.08 mol/L H_2O_2. According to obtained results, the process efficiency increased to 73.24% and 55.4% at pH of 3 and 10, respectively. Therefore, the maximum removal efficiency was obtained at pH of 3. This finding is consistent with previous studies [17]. Generally, the US/H_2O_2 process efficiency is related to the pH level due to the effect of pH on hydroxyl radical regeneration. Under acidic conditions, superoxide radical ($O_2^{\bullet-}$) reacts with hydrogen ions and produces hydroperoxyl radicals ($HO_2^{\bullet}$). As a result, hydroperoxyl radicals can produce hydrogen peroxide and then can be converted to hydroxyl radicals according to the following Eqs. (2)–(5):

$$O_2^{\bullet-} + H^+ \rightarrow HO_2^{\bullet} \quad (2)$$

$$HO_2^{\bullet} + HO_2^{\bullet} \rightarrow H_2O_2 + O_2 \quad (3)$$

$$H_2O_2 + e^- \rightarrow HO^{\bullet} + OH^- \quad (4)$$

$$H_2O_2 + O_2^{\bullet-} \rightarrow HO^{\bullet} + OH^- + O_2 \quad (5)$$

On the other hand, at higher pH values, the degradation rate of 2,4-DCP decreases, which can be attributed to the rapid decomposition of hydroxyl [19,20].

4.2. Effect of H_2O_2 concentration on US/H_2O_2 process

The effect of H_2O_2 level on the removal efficiency of 2,4-DCP is shown in Fig. 3. The experiments were conducted at initial 2,4-DCP concentration of 0.3 mol/L and optimum pH of 3. The concentrations of hydrogen peroxide were set at 0.004, 0.05, 0.08, 0.09, and 0.1 mol/L. Results showed that the removal efficiency increased with the increase of H_2O_2 concentration. After 90 min of reaction time, the removal efficiency increased to 92.5% at the initial H_2O_2 concentration of 0.1 mol/L. However, the removal efficiency decreased slightly with further increases in the hydrogen peroxide concentration (according to the pre-test results, data not shown). The removal efficiency at the higher H_2O_2 concentrations can be explained as follows: at low levels of H_2O_2, further increases of H_2O_2 concentration increase the possibility of collision between US waves and H_2O_2, therefore, promoting hydroxyl radical generation by H_2O_2. This phenomenon results in a higher degradation rate of 2,4-DCP. However, when the H_2O_2 concentration is too high, this reaction can result in generating hydrogen peroxide inhibitors and consequently results in decreased removal efficiency [21]. Therefore, to achieve the maximum removal efficiency of 2,4-DCP by US/H_2O_2, the hydrogen peroxide concentration should be kept at a constant optimum level. After that, 0.1 mol/L was used as the optimal condition for the next steps of the experiment.

4.3. Effect of tert-butanol on US/H_2O_2 process

Fig. 4 shows the removal efficiency of 2,4-DCP under the optimum conditions (reaction time = 90 min, pH = 3, H_2O_2 concentration = 0.1 mol/L, and initial 2,4-DCP concentration = 0.3 mol/L) in the presence of *tert*-butanol as a radical scavenger. *tert*-Butanol is a strong hydroxyl radical scavenger,

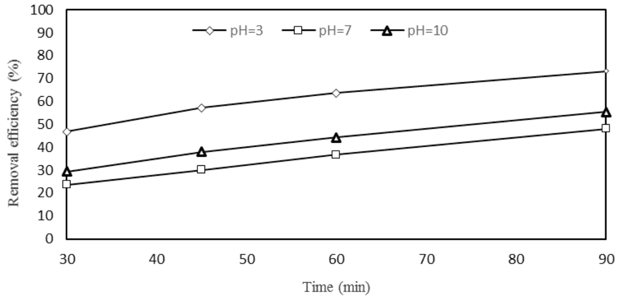

Fig. 2. Effect of pH on US/H_2O_2 process efficiency in 2,4-DCP removal (H_2O_2 = 0.08 mol/L and 2,4-DCP = 0.3 mol/L).

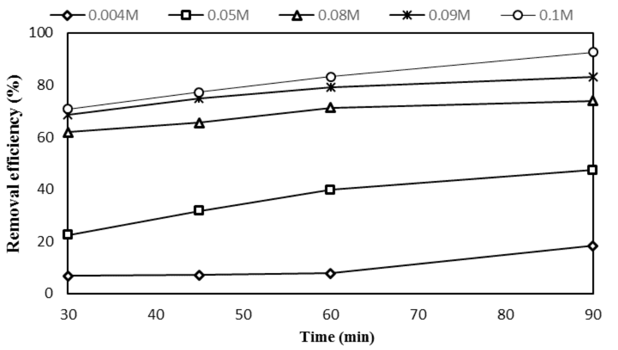

Fig. 3. Effect of H_2O_2 concentration on US/H_2O_2 process efficiency in 2,4-DCP removal (pH = 3 and 2,4-DCP = 0.3 mol/L).

which reacts with free hydroxyl radicals prior to the reaction of 2,4-DCP and OH$^\bullet$ [18]. Due to the strong attractive effect of *tert*-butanol, it is considered to be a very suitable indicator for radical reactions [22,23]. Following the reaction, intermediate substances are generated, which can hinder the OH$^\bullet$ generation and radical chain reactions.

As results show, in the presence of *tert*-butanol (0.04 mol/L), the removal efficiency of 2,4-DCP was decreased from 92.5% to 69.7%. This decrease is attributed to the presence of *tert*-butanol as a radical scavenger, which decreases the concentration of hydroxyl radicals, and consequently results in lower removal efficiency. These results are comparable with the results of Asgari et al. [12] on the removal of phenol using a catalyst ozonation process.

4.4. Effect of US and H_2O_2 stand-alone and in combination

In this part, the effect of US and H_2O_2 alone as well as US/H_2O_2 on 2,4-DCP removal efficiency was studied under optimal conditions obtained from the previous steps (pH = 3, H_2O_2 = 0.1 mol/L, 2,4-DCP = 0.3 mol/L, and reaction time = 90 min). As can be seen from Fig. 5, US and H_2O_2 stand-alone have limited efficiency on 2,4-DCP removal with US and hydrogen peroxide alone showing 14.5% and 22.87% of removal efficiency, respectively; while in combination, the US/H_2O_2 showed higher removal efficiency. A comparison between the efficiency of US and H_2O_2 separately in 2,4-DCP removal revealed the benefits of the combined US/H_2O_2 process in the pollutant mineralization. This indicated that ultrasound can activate hydrogen peroxide and produce more active hydroxyl radicals compared with US and H_2O_2 alone. Previous literature has investigated the effect of US and H_2O_2 alone and in combination with other agents for removing phenolic compounds. In a study on pentachlorophenol removal using microwave/H_2O_2, it was found that microwave and H_2O_2 have little efficiency in pentachlorophenol mineralization [24], which is consistent with our findings. Seid-Mohammadi et al. [9] reported similar results in the use of persulfate and ultraviolet alone and in combined conditions (UV/persulfate) for removing phenol at high concentrations from saline wastewaters.

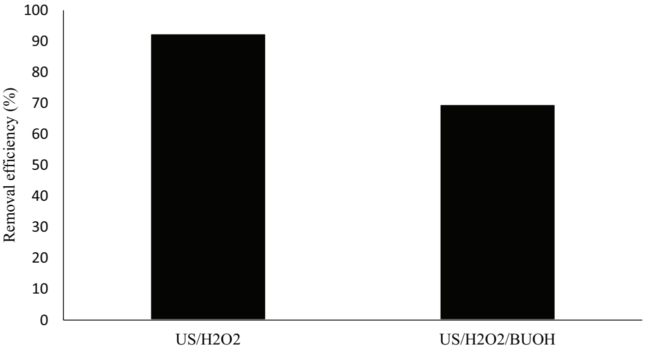

Fig. 4. Effect of *tert*-butanol on US/H_2O_2 process under optimal conditions (reaction time = 90 min, pH = 3, H_2O_2 concentration = 0.1 mol/L, and initial 2,4-DCP concentration = 0.3 mol/L).

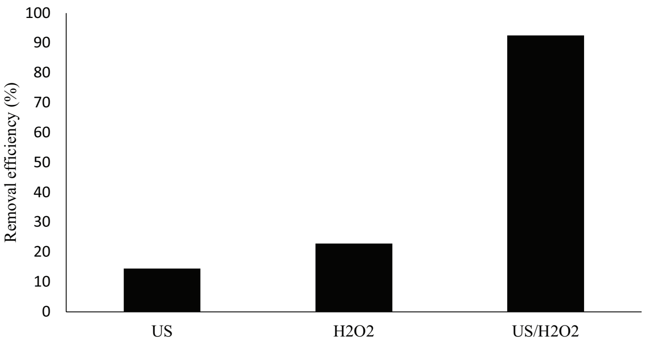

Fig. 5. Effect of US alone, H_2O_2 alone, and US/H_2O_2 on the oxidation of 24-DCP (optimal conditions obtained from previous steps: pH = 3, H_2O_2 concentration = 0.1 mol/L, and 2,4-DCP = 0.3 mol/L).

4.5. Reaction kinetic of 2,4-DCP degradation

Fig. 6 shows the reaction kinetic of 2,4-DCP degradation in the US/H_2O_2 process. Chemical kinetics is used to study the rate of chemical reactions. In the pseudo-first-order kinetic model, the half-life is independent of the initial concentration of reactants; whereas in the other orders of the reaction, half-life varies with initial concentration. In these reaction orders, the number of effective collisions increases with increasing concentrations of an oxidizing agent and thus increases the reaction rate. Results indicated that the 2,4-DCP degradation in the US/H_2O_2 system under optimum conditions approximated a pseudo-first-order kinetic model with a reaction constant of 0.025 min^{-1}, which is consistent with the findings of previous studies [25,26].

4.6. Effect of US/H_2O_2 process on COD removal

The effect of US/H_2O_2 on COD removal, produced by 2,4-DCP, is shown in Fig. 7. The experiment was conducted under optimum conditions (pH = 3, H_2O_2 = 0.1 mol/L, 2,4-DCP = 0.3 mol/L, and reaction time = 90 min). COD residual was measured during various reaction times (15, 30, 60, and 90 min). Results showed that COD concentrations decreased over time with 74.6% COD removal after 90 min of reaction. This result indicated that the US/H_2O_2 process has high efficiency in 2,4-DCP mineralization. A comparison of the efficiency of 2,4-DCP removal and COD degradation using the

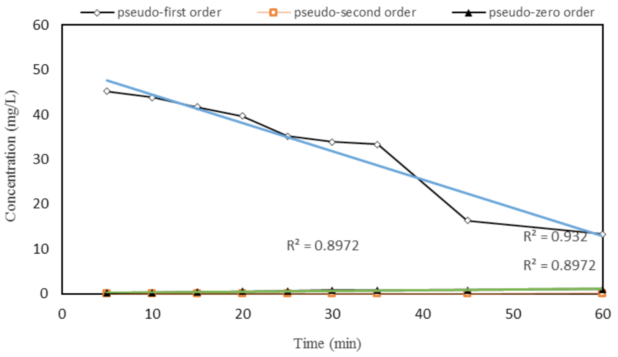

Fig. 6. Reaction kinetic of 2,4-DCP decomposition in US/H_2O_2 process.

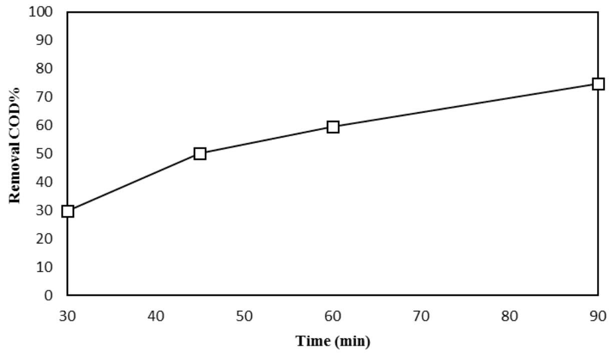

Fig. 7. Effect of US/H_2O_2 process on COD removal (pH = 3 and DCP = 0.3 mol/L).

US/H_2O_2 process reveals lower efficiency in COD mineralization. This can be attributed to the production of organic intermediates during the oxidation process, which may need more time degradation by the US/H_2O_2 process. The produced intermediates can also reduce the process efficiency in removing COD [12].

5. Conclusions

The effectiveness of 2,4-DCP degradation using an US process alone and in combination with different levels of H_2O_2 at different pH and reaction times was investigated. Synthetic water containing 0.3 mol/L of 2,4-DCP was used in the experiments. The following conclusions can be made from this study:

- With the initial H_2O_2 concentration of 0.08 mol/L, pH 3 was found to achieve the highest 2,4-DCP degradation (74%, after 90 min reaction) compared with other pH levels.
- The efficiency of US/H_2O_2 was increased with increasing H_2O_2 concentration. The maximum removal efficiency of 2,4-DCP (92.5%) was obtained at 0.1 mol/L of H_2O_2.
- In the presence of *tert*-butanol, the removal efficiency of 2,4-DCP was decreased due to the radical scavenging effects of *tert*-butanol.
- Degradation of 2,4-DCP by US/H_2O_2 system under optimum conditions approximated a pseudo-first-order kinetic model with a reaction constant of 0.025 min^{-1}.
- Results show that 74.6% COD removal can be achieved by US/H_2O_2 process after 90 min of reaction at pH of 3 with initial H_2O_2 of 0.1 mol/L.

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