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Research Article

Investigating the Removal of Toluene from Textile Industrial Wastewater Using Integrated Advanced Oxidation-Based Fenton and Ultrasound Process

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ABSTRACT

The manufactured wastewater of textiles contains perilous solvents like methyl isobutyl ketone, methanol, methyl ethyl ketone, xylene, and toluene. These hazardous volatile organic compounds, like as toluene, pose significant risks to human health and aquatic ecosystems. Advanced oxidation processes (AOPs) are promising for their degradation due to the generation of highly reactive hydroxyl radicals. The purpose of this study was to use a combined Fenton-ultrasound AOP to maximize the removal of toluene from both synthetic and actual textile effluent. The impact of H_2O_2 concentration (50–200 mg/L), Fe^{2+} dose (10–25 mg/L), beginning toluene concentration (10–70 mg/L), pH (3–7), and ultrasonic frequency (0 or 35 kHz) on removal efficiency over 30 min at 25 °C was assessed using batch tests. Headspace-gas chromatography was used to measure the amounts of toluene.100 mg/L H_2O_2, 10 mg/L Fe^{2+}, 10 mg/L toluene, and pH 3 were the ideal circumstances. With combined Fenton-ultrasound, the highest removal effectiveness was 89.7%, as opposed to 86.2% for Fenton alone and 6% for ultrasound alone. Due to interference from co-pollutants, including xylene and benzene, 72.3% clearance was attained for actual textile wastewater under these circumstances. For the remediation of toluene in industrial effluents, this integrated method exhibits exceptional efficiency and cost-effectiveness, providing a scalable option for the treatment of wastewater from refineries and textiles.

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1. Introduction

In recent years, with the increasing growth of various industries, many pollutants have entered the surface and underground waters. For this purpose, many studies have been used to remove environmental pollutants, including heavy metals, effluents containing drugs [1-3], chemical dyes, etc [4-6]. Aromatic hydrocarbons are frequently released into the natural environment through the incomplete burning of organic substances such as oil, petroleum derivatives, gas, coal, and wood. Aromatic hydrocarbons can be categorized based on their structure as either monocyclic or polycyclic hydrocarbons. Monocyclic aromatic hydrocarbons include notable compounds like Toluene, Benzene, Xylene, and Ethylbenzene, while polycyclic hydrocarbons consist of fused benzene rings. Polycyclic hydrocarbons are further divided into light and heavy categories, depending on the quantity of benzene rings within their structure. Those with up to four benzene rings are considered light, whereas those with more than four rings fall into the heavy polycyclic hydrocarbons group [7-10]. The solvents of volatile organic compounds were a significant part of some wastewater from textile manufacturing. Based on the Toxic Release Inventory data of the U.S. Environmental Protection Agency, methyl isobutyl ketone, methanol, methyl ethyl ketone, xylene, and toluene were perilous organic compounds discharged from textile facilities. Advanced oxidation processes (AOPs) stand out as highly effective techniques for eliminating organic contaminants. They leverage reactive species generated on-site to facilitate degradation. This method has proven successful in swiftly eliminating a wide range of pollutants, including dyes, pesticides, endocrine disruptors, and various emerging contaminants. AOPs offer a promising avenue for completely transforming the targeted compounds into less or non-toxic forms and eventually achieving complete mineralization [11-14]. The produced water, a byproduct of the petrochemical industry, accounts for the majority of the waste produced through the production process of petrochemicals. Significant levels of volatile organic chemicals with low molecular weight (VOCs), such as toluene, are found in the created water. These substances affect major biological issues such as the stratospheric ozone hole, water and air pollution, and global warming. They also disrupt aquatic ecosystems, which leads to damage to the aquatic life. They are extremely volatile, partially soluble in seawater, and can be decomposed in the aquatic environment. Additionally, they pose a concern to human health due to their teratogenic or carcinogenic activities, which result in mutagenic, acute, and chronic disorders. The maximum permissible concentration of toluene in drinking water was subsequently established by the WHO (World Health Organization) at 0.7 mg/L. For the removal of toluene from contaminated water, there are many conventional water treatment methods that work well, such as breaking down organic matter, evaporating water, exposing water to light, filtering water through membranes, sticking pollutants to activated carbon, and trapping contaminants in particles and silt [15-18]. Although their application is complicated and varies depending on the areas polluted by toluene, these conventional solutions have a number of drawbacks. Utilizing extremely reactive oxidants, including hydroxyl radicals, is referred to as using advanced oxidation processes.

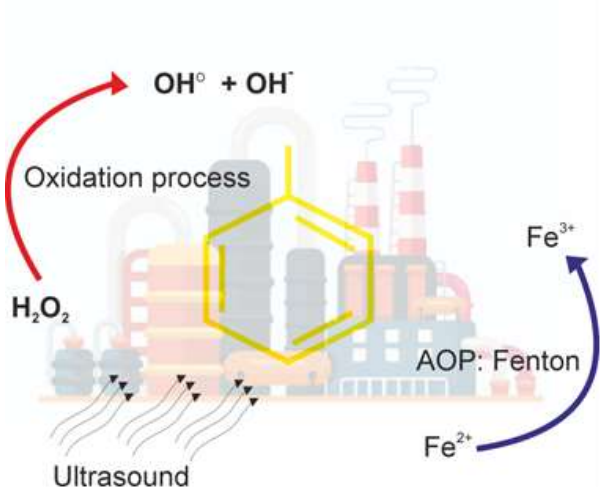
Under ideal conditions, sono-Fenton processes can remove up to 90% of recalcitrant organics and textile dyes from wastewater, including reactive yellow 81 and bisphenol A. However, their effectiveness for volatile organic compounds (VOCs) like toluene, which is common in textile effluents at low concentrations (10–50 mg/L), is still

unknown. In the past, sono-Fenton research has mostly concentrated on high-dosage regimens for BTEX mixtures as biological process pretreatments, frequently ignoring stand-alone applications in intricate textile matrices with interfering species like dyes and surfactants. In addition, low-frequency ultrasonic support has been studied for Fenton-like oxidation of textile sludge, although not particularly for toluene cleanup in low-reagent, cost-effective settings.

Using lower dosages (10 mg/L Fe^{2+} and 100 mg/L H_2O_2) to improve hydroxyl radical generation through ultrasound-induced cavitation and H_2O_2 sonolysis, this study optimizes a standalone sono-Fenton process for low-concentration toluene removal from synthetic and real textile wastewater. We show a 3.5% efficiency boost over traditional Fenton by thorough parametric evaluation and real effluent validation, offering a scalable, cost-effective option for on-site industrial treatment.

Examples of AOPs include UV radiation combined with ozone, ozonation with the addition of hydrogen peroxide (H_2O_2), and UV radiation combined with H_2O_2. The creation of two hydroxyl radicals is the result of photolytic cleavage in the latter technique, which was employed in the current investigation. Since wastewater has a high scavenging capacity and low UV transmittance, volatile organic chemical removal with this method is currently uncommon. However, both hospital and municipal wastewater have been the subject of various research [19]. Cavitation can be caused by using ultrasonic waves, which will increase the efficiency of wastewater removal. Cavitation causes the production of small bubbles in the liquid environment, increasing the production of free radicals such as H and OH in the liquid environment, which can be used in wastewater treatment. Lhotský et al. removed toluene from contaminated groundwater with a combination of aeration and UV/H_2O_2 [20]. Sampaio et al. studied the degradation of toluene from gas streams in a reactor with activated carbon by heterogeneous Fenton oxidation [21].

This study lies in the application of a combined advanced oxidation process (Fenton-ultrasound) to remove toluene from textile industrial wastewater. Unlike previous studies that focused on high concentrations of the pollutant, this study uses low doses of Fe^{2+} and H_2O_2 under acidic conditions and addresses the removal of toluene in synthetic wastewater and in real wastewater. This approach aims to minimize chemical consumption and sludge production while maintaining high VOC degradation efficiency to provide a scalable, cost-effective, and environmentally friendly method for industrial wastewater treatment.



Schematic 1. Remove toluene from the effluent using an integrated advanced oxidation-based Fenton and ultrasound process

2. Materials and Methods

2.1. Materials

Toluene, Fe (II), Phosphate buffer, sulfuric acid, sodium hydroxide, and H_2O_2 were all purchased from Sigma Chemical Company. The LUC-405 digital ultrasound device, made in Korea, and the Hanna digital pH meter were used. The concentration of toluene as a pollutant was measured by applying Agilent 7890 A, Palo Alto, headspace-gas chromatography (CA, USA). This apparatus was equipped with an FID (flame ionization detector), and a capillary column of HP-5 (0.25 µm thickness, length of 30 m, 0.32 mm internal diameter).

2.2. Method

In this section, the effect of changes in the parameters of hydrogen peroxide concentration, iron concentration, pH, and pollutant concentration on pollutant removal percentage and the graphs of pollutant removal percentage changes according to the change of the above parameters in the pollutant removal process have been investigated.

Every experiment was carried out in a glass reactor with a water bath to maintain a regulated temperature of 25°C. Toluene (Sigma Chemical Company) was dissolved in deionized water to produce synthetic wastewater samples. For the Fenton process, Fe^{2+} (Sigma Chemical Company) and hydrogen peroxide (H_2O_2, Merck Company)

3. Result and Discussion

3.1. The effect of H_2O_2

The experiment involved testing H_2O_2 at four different concentrations. As depicted in Figure 1, an enlarge in the H_2O_2 concentration led to an increase in the percentage removal of toluene. However, increasing the concentration beyond 100 mg/L decreased the efficiency of the process. At this stage, the highest percentage of toluene removal (82.9%) was achieved at the optimal.

Concentration of 100 mg/L H_2O_2 (Table 1). Under these conditions, increasing the concentration of the oxidizing agent up to a certain threshold increased the reaction rate. However, beyond that threshold, it had a reverse effect on organic matter removal. This can be attributed to the high concentration of H_2O_2 acting as a scavenger for free radicals, thereby reducing the concentration of free radicals.

Table 1. The effect of H_2O_2 concentration on the removal of toluene

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	Initial Toluene (mg/L)	pH	Reminded Toluene (mg/L)	Toluene Removal percent	Time (min)	Ultrasonic (kHz)	Temp. (ºC)
1	50	15	10	3	7.66	74.45	30	0	25
2	100	15	10	3	5.12	82.92	30	0	25
3	150	15	10	3	5.92	80.26	30	0	25
4	200	15	10	3	7.07	76.42	30	0	25

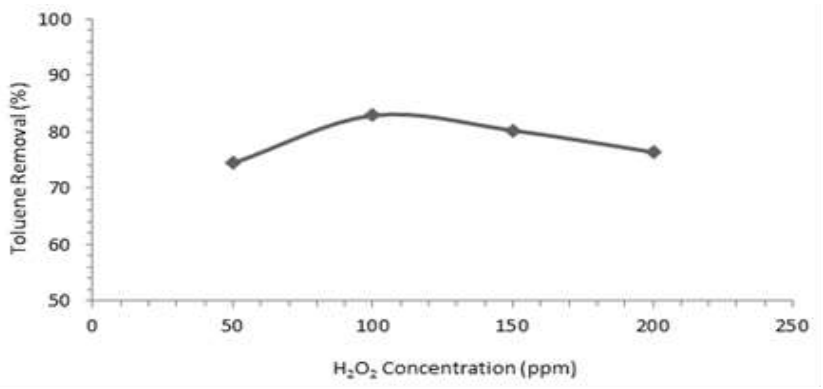


Fig. 1. The efficacy of H_2O_2 concentration on the removal of toluene.

3.2. Effect of iron concentration

According to Figure 2, at first, in the presence of iron and hydrogen peroxide, hydroxyl radical is formed according to the following equation, which plays an important role in the degradation of organic matter (Eq. 1). By increasing iron, the amount of hydroxyl radical increases, and therefore the percentage of toluene removal increases. By further increasing the concentration of iron, according to equation (2), it consumes the hydroxyl radical and causes a decrease in the concentration of hydroxyl radical. Therefore, to create a balance between the production and degradation of hydroxyl radicals, the concentration of iron should be optimal. Due to the lack of a significant difference in the percentage of toluene removal and also saving in the consumption of raw materials, preventing iron deposition in the reaction medium, and avoiding process design to remove it, the lowest concentration of 10 mg/L was considered as the optimal concentration despite the lower percentage of removal (Table 2).

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

$$Fe^{2+} + OH^{o} \longrightarrow Fe^{3+} + OH^{-} \quad (2)$$

Table 2. The efficacy of iron concentration on the removal of toluene

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	Initial Toluene (mg/L)	pH	Reminded Toluene (mg/L)	Toluene Removal percent	Time (min)	Ultrasonic (kHz)	Temp (ºC)
1	100	10	10	3	5.69	81.01	30	0	25
2	100	15	10	3	5.08	83.05	30	0	25
3	100	20	10	3	4.58	84.72	30	0	25
4	100	25	10	3	5.07	83.1	30	0	25

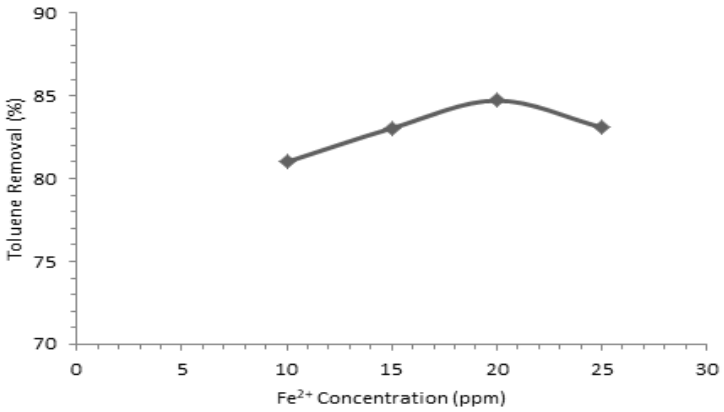


Fig. 2. The effect of Fe2+concentration on toluene removal

3.3. Effect of pH

The results presented in Figure 3 indicate that the removal of organic matter is much higher in acidic pH than in alkaline and neutral conditions. The results of Table 3 indicate that the pH of the reaction medium has a very significant effect on the reaction rate and degradation of organic matter. The catalytic activity of the Fenton reaction is maximum at pH 2.8 to 3, which decreases sharply with increasing or decreasing pH. Therefore, sufficient production of hydroxyl radicals in acidic conditions increase the oxidation of organic matter. At pH higher than three, iron forms iron (III) hydroxide precipitate, and water is broken down into oxygen and water. Additionally, the formation of iron complexes at high pH leads to a decrease in the concentration of iron ions. Furthermore, at pH values above 5, weaker oxidizing agents such as ferryl ions are formed, which are much more selective than hydroxyl radicals.

Table 3. The effect of pH on the removal of toluene

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	Initial Toluene (mg/L)	pH	Reminded Toluene (mg/L)	Toluene Removal percent	Time (min)	Ultrasonic (kHz)	Temp (ºC)
1	100	10	10	3	4.77	84.1	30	0	25
2	100	10	10	5	6.10	76.65	30	0	25
3	100	10	10	7	10.44	65.2	30	0	25

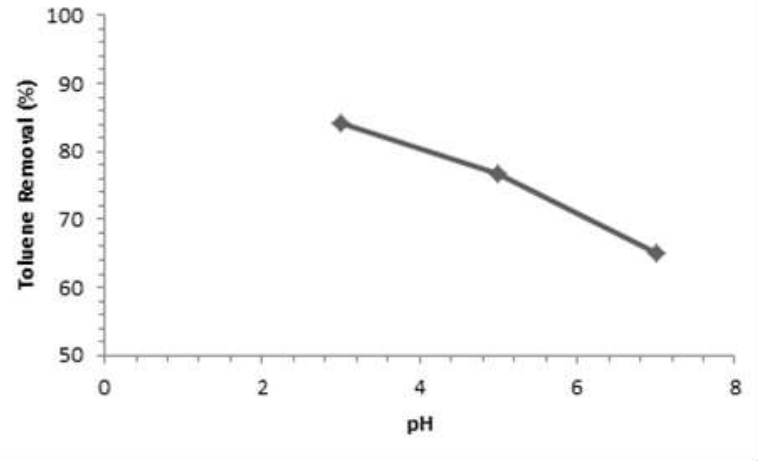


Fig. 3. The effect of pH on toluene removal

3.4. Effect of pollutant concentration

As shown in Figure 4 and Table 4, the percentage of pollutant removal has an inverse relationship with its initial concentration. This is due to the constant amount of available hydroxyl free radicals, regardless of the pollutant concentration. Therefore, as the pollutant concentration increases, the percentage of pollutant removal decreases.

Table 4. The effect of pollutant concentration on removal

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	pH	Initial Toluene (mg/L)	Reminded Toluene (mg/L)	Tolueneremoval percent	Time (min)	Ultrasonic (kHz)	Temp (ºC)
1	100	10	3	10	1.38	86.2	30	0	25
2	100	10	3	30	4.77	84.1	30	0	25
3	100	10	3	50	9.95	80.1	30	0	25
4	100	10	3	70	20.09	71.3	30	0	25

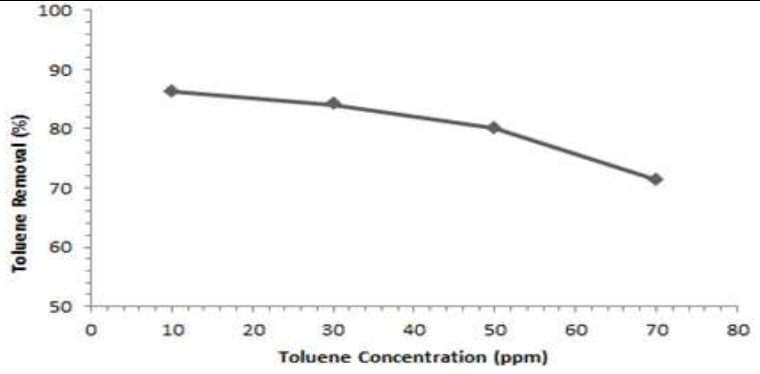


Fig. 4. The effect of pollutant concentration on toluene removal

3.5. Effect of ultrasonic

Figure 5 shows a comparison between the combined Fenton/ultrasonic process with the Fenton process alone, and the ultrasonic process alone. As can be seen, the removal percentage in the combined process is 3.5% higher than the Fenton process alone, and this is due to the sonolysis of H_2O_2 molecules and the production of free radicals. For the ultrasonic process alone, the pollutant removal efficiency is 6% (Table 5**)**. In the oxidation process by ultrasound, the ultrasonic process plays two roles: reactant and catalyst. As a reactant, ultrasound is responsible for the sonolytic decomposition of organic molecules, and as a catalyst, it causes the sonolysis of hydrogen peroxide oxidant and the production of free radicals.

Table 5. The effect of ultrasonic on toluene removal

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	pH	Initial Toluene (mg/L)	Reminded Toluene (mg/L)	Toluene removal percent	Time (min)	Ultrasonic (kHz)	Temp (ºC)
1	100	10	3	10	1.39	86.2 ± 0.5	30	0	25
2	100	10	3	10	1.03	89.7± 0.6	30	35	25
3	0	0	3	10	9.4	6 ± 0.3	30	35	25

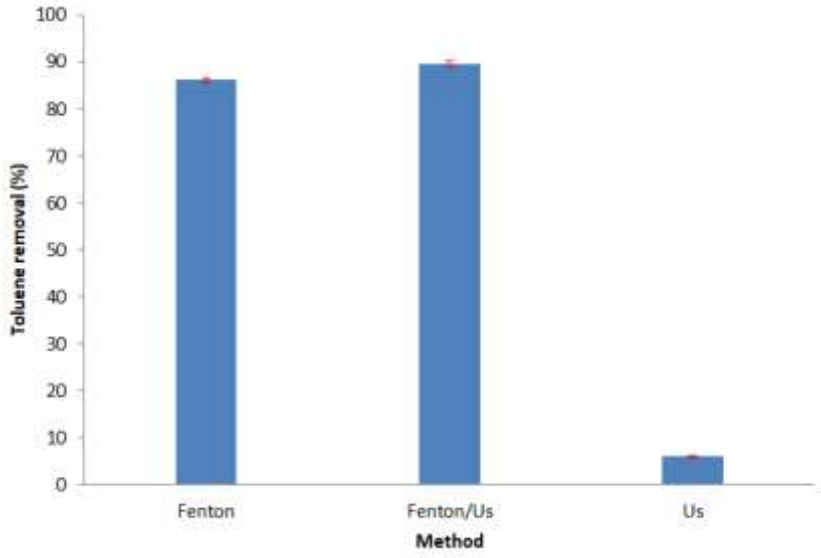


Fig. 5. The effect of ultrasonic on toluene removal (Error bars represent standard deviations from triplicate experiments (n=3)).

3.6. The effect of combined Fenton-ultrasound and ultrasonic process on the removal of refinery effluent

As shown in Table 6, the removal percentage of toluene from real wastewater in the combined Fenton and ultrasonic process was 72.3%, which was 17.5% lower than the removal percentage of toluene from synthetic wastewater in the same process. The reason for this is due to the presence of interfering compounds such as benzene, xylene, and ethylbenzene in the refinery wastewater. Ultrasound waves alone had no effect on the real wastewater and the removal percentage of toluene was zero.

The Fenton reaction (Eq. 1) and ultrasound-induced cavitation produce hydroxyl radicals (OH·), which attack toluene and start its oxidation to intermediates like cresols or benzaldehyde, which are then mineralized to CO_2 and H_2O. The Fenton process uses ultrasound (35 kHz) to increase OH· production through H_2O_2 sonolysis and water dissociation, as shown by the 3.5% increase in removal efficiency (89.7% vs. 86.2%, Table 5). The ideal conditions (100 mg/L H_2O_2, 10 mg/L Fe^{2+}, pH 3, and 10 mg/L

toluene) maximize OH· production while minimizing scavenging reactions (e.g., Eq. 2, or excess H^2O^2 scavenging).

Table 6. Real wastewater oxidation

Sample	H_2O_2 (mg/L)	Fe^{2+} (mg/L)	pH	Initial Toluene (mg/L)	Reminded Toluene (mg/L)	Toluene removal percent	Time (min)	Ultrasonic (kHz)	Temp (ºC)
Real sewage	100	10	3	10	2.77	72.3	30	35	25
Real sewage	0	0	3	10	10	0	30	35	25

3.7. Comparison of method results

According to the results obtained from this research and comparison with the methods mentioned in other research in order to check the removal of toluene from water, acceptable results can be seen, for example, Pakizeh et al. application of hydrophobic ZIF-8 (nanoparticles of zeolitic) and combine them in the matrix of the polymer was achieved best separation with ZIF-8 (15%) at 30 °C and 300 mg/L [22]. Molina et al., produced and identified activated carbons from agricultural and forest residues using physical activation and applied them as effective adsorbents for the removal of toluene from wastewater. The amount of activated carbon used in the optimal state of 20 milligrams of solution acidity was about 1.42 [23]. Ali et al., have used the combined method between MIP (molecularly imprinted polymer) and US (ultrasound) to get top efficiency in the removal of toluene. In this optimization process, it is stated that by using a concentration of 500 mg/L of toluene, and an MIP dosage of 2 g/L, after a contact time of 10 minutes, the removal of 95.517 % of toluene has been obtained at the power of 50 ultrasound [24].

By emphasizing low initial pollutant concentrations (10 mg/L) and minimal reagent use, which reduces sludge formation and operating costs in textile settings, this study's integration of ultrasound with Fenton oxidation not only amplifies radical production, yielding 89.7% toluene removal under ideal conditions, but also goes beyond previous sono-Fenton applications. This homogeneous approach achieves comparable performance (72.3% in real effluents) despite matrix interferences from co-VOCs like xylene, highlighting its robustness without additional amendments. This is in contrast to heterogeneous sono-Fenton variants tested on dyes or phenolic compounds, where efficiencies exceed 95% but require nanoparticle catalysts and higher pH adjustments.

Through cavitation-enhanced radical production, our sono-Fenton approach surpasses traditional Fenton for toluene by 3.5% when compared to known techniques [25-27]. For comparable yields, it requires 100–200 times lower Fe^{2+} doses than electro-Fenton variations. Additionally, it outperforms sono-Fenton techniques for textile color removal, which attain 95% decolorization but disregard metrics relevant to volatile organic compounds, such as headspace-GC measurement. This homogeneous system prevents secondary contamination and facilitates on-site deployment for VOC-focused projects, such as heterogeneous photo-Fenton (85% toluene removal at 50 mg/L but with catalyst recovery issues). Additionally, the current study addresses the gap in low-concentration, real-matrix situations common in textiles by enabling full mineralization at low costs, in contrast to pretreatments for BTEX in petrochemical effluent (93% toluene but at 2 g/L Fe^{2+}).

3.8. Economic feasibility study of the study method

Due to better mass transfer and increased OH· generation by H_2O_2 sonolysis, the combined Fenton-

ultrasound process produced a 3.5% higher toluene removal efficiency (89.7% vs. 86.2%, Table 5). However, compared to the Fenton reaction alone, which uses inexpensive reagents (H_2O_2, Fe^{2+}), the ultrasonic component (LUC-405, 35 kHz) uses more energy. The ideal Fe^{2+} concentration of 10 mg/L (Table 2) reduces the need for reagents and prevents extra expenses for the removal of iron precipitate, which may partially offset energy expenditures. A quantitative energy and cost analysis is required to validate economic viability; however, in applications that need strict toluene elimination, the slight efficiency increase could be justified.

4. Conclusion

In this study, the oxidation of toluene was carried out using the combined advanced oxidation process, Fenton, and ultrasound, and the results showed positive results from the combined process instead of using each of the methods separately. Also, the results show that the percentage of toluene removal depends on several factors, such as the concentration of hydrogen peroxide as an oxidant, iron concentration, pollutant concentration, pH, and the intensity of ultrasound waves, and the efficiency will be very effective by changing these factors. Experiments have also shown that the best conditions to achieve the best efficiency in removing toluene pollutants in the proposed system include a concentration of 100 mg/l of H_2O_2, 10 mg/L of iron, 10 mg/L of pollutant, and pH (3), and the combined Fenton's process was with ultrasonic waves. According to the results, this process is recommended as a suitable method to remove toluene from the effluent of the refinery, and considering the results, this process is recommended as a suitable method to remove toluene from the effluent of the refinery and textile industrial wastewater.

Declaration of interests

No conflict of interests found in the whole process of this work conduct; the authors approved.

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