Removal of Bisphenol A from aqueous solutions using ultrasonic waves and hydrogen peroxide


Abstract

Efficiency of technologies using ultrasonic waves superimposed with hydrogen peroxide for the removal of Bisphenol A (BPA) from the aqueous solution was well studied and investigated. BPA is a non-biodegradable antioxidant, which was highly resistant to the biological and chemical breakdown; the non-biodegradability of the dye made it noxious and hazardous material for the both aquatic flora as well as fauna. Tests were done at two levels i.e. ultrasonic power 300 and 500 W; frequencies of 35 and 130 kHz respectively and at the optimum pH levels of 3, 7 and 11. Hydrogen peroxide levels were tested at concentrations of 5, 15 and 30 mg/l. Obtained results demonstrated that the efficiency of BPA breakdown using a process combining ultrasonic waves assisted by hydrogen peroxide was more effective than separately applied processes. The highest rate of decomposition was achieved at the frequency of 130 kHz and 500 W assisted by 30 mg/l H2O2 at pH level 11. The removal efficiency of BPA in natural samples decreased compared to efficiency in synthetic samples.

Keywords: Bisphenol A, Ultrasonic wave, Hydrogen peroxide, Water

1. Introduction

The IUPAC name for BPA is 2,2-(4, 4-dihydroxyphenyl) propane. The formation is 2 mol phenol and 1 mol acetone, expressed as the following formula C13H10O2 [1]. BPA is a substance that contaminates the aqueous ecosystems or biota and its acute toxicity for aquatic organisms was about 1–10 mg/l in freshwater and marine species [2–4]. Even at very low concentrations BPA leads to the proliferation of prostate cancerous cell in humans, it also has adverse effects on the cardiac patients, type 2 diabetes and contributes to enzymatic rudeness in the liver [5].

BPA was classified as an endocrine disrupting compound in humans and other animals [6]. BPA in the blood of both men and women is associated with disorders of the reproductive system such as, endometrial hyperplasia, repeated abortion, unnatural genetics and poly-cystic ovarian syndrome. Recent reports show that BPA carries a health risk even at the low dose of 0.05 mg per kg body weight [7]. Recent research has indicated that BPA can affect estrogenic receivers membrane cells at concentrations of pg/ml in physiological response to BPA simulation [8].

There are several different ways commonly applied to destroy BPA. Ultrasonic technology is considered to be a best method to treat water sources contaminated with compounds such as BPA. Ultrasonic waves stimulate cavitations in chemical compounds resulting in the formation of strong and active radicals such as OH°, H° and H2O2. These radicals may react with collapsing bubbles and form new molecular structures; or radicals may be transformed to a state whereby the inner liquid becomes active. In water sonolysis produces hydroxyl and hydrogen active radicals [9–12].

Procedures using ultrasonic waves are very efficient at removing chemicals and eliminating toxic contamination. Nowadays, techniques using hybrid ultrasonic waves, together with other methods such as advanced oxidation process are the most common uses of ultrasonic waves [13–28].

The objective of this study was to investigate the efficiency of ultrasonic reactor and hydrogen peroxide as single and hybrid processes for the removal the BPA from synthetic and natural samples. Variables in this study were: power, frequency, pH, sonication time, BPA concentration and hydrogen peroxide.
2. Materials and methods

This research was an experimental–applicative study to test contaminated water samples in the LAB of Tehran University of Medical Sciences in 2012. Tests were done on processes to remove BPA using an ultrasonic bath [Alma com] applying irradiation capability of ultrasonic waves at two frequencies 35 and 130 kHz with inlet power supplies of 300 and 500 W. Specifications of the ultrasonic bath are presented in Table 1. Solution pH was adjusted using NaOH and HCl 0.01 N. All chemicals used in this research were obtained from Merck and were high grade. Tests were carried out in several steps according to the following description.

First Step: Use of ultrasonic process.
A stock solution of BPA was made and then samples were produced at concentrations of 2, 5, 20 and 50 mg/l; solution pH was adjusted with a pH meter model E520, Metrohm Herisau to levels 3, 7 and 11. At the next step samples were poured into the reactor 500 ml inside the ultrasonic bath, (Fig. 1) at frequencies of 35 and 130 kHz, powers 300 and 500 W and sonication times of 15, 30, 45, 60, 90, 120, 150 and 180 min.

Second Step: Use of hydrogen peroxide.
In this step, BPA samples were adjusted according to concentrations and pH levels as mentioned above. Then concentrations of 5, 15 and 30 mg/l were made from the stock solution with hydrogen peroxide (30%) and added to samples of BPA and mixed in a shaker (model GFL 3018) for the following durations; 5, 10, 20, 30, 45, 60, and 90 min. Removal balance of BPA was determined by investigation of investigated by H₂O₂.

Third Step: ultrasonic waves assisted by hydrogen peroxide.
This step tested effects ultrasonic waves and hydrogen peroxide in combination on removal of BPA. Hydrogen peroxide was added to initial concentrations of BPA in amounts of 5, 15 and 30 mg/l and tested in the ultrasonic unit at frequencies of 35 and 130 kHz and two power supplies of 300, 500 W for durations of 15, 30, 45, 60, 90, 120, 150, 180 min and at pH of 11 (based on results for tests to determine an optimum pH for processes of ultrasonic and hydrogen peroxide treatments). BPA concentrations were measured by colorimetry with 4-Amino antipyrene and mean Spectrometer UV–vis Lambada 25 Perkin Elmer, Shelton. Finally all data gained from ultrasonic and hydrogen peroxide tests were analyzed with Excel software.

Forth step: Analysis on water nature sample.
In this step, the water used for analysis was taken from the urban water supply and BPA was added to make a concentration of 2 mg/l in a natural sample and in optimum conditions in terms of pH, time, and concentration of hydrogen peroxide, power and frequency that had been determined during the previous tests. Ultrasonic processes were then applied to the samples, hydrogen peroxide and a combination of ultrasonic treatment with hydrogen peroxide and to determine amounts of removal of BPA from natural samples. Measurements of concentrations of BPA in natural water samples were taken according to the applied method in studies from GC machine model VARIAN CP–3800 (Australian) [29]. The Specification of natural water samples is introduced in Table 2.

3. Result and discussion

3.1. Effect of ultrasonic process

Effects of the ultrasonic process to remove BPA in the treatments at various pH levels, concentrations, power supplies and frequencies are shown in Tables 3 to 6. According to Table 6, destruction efficiency of BPA with 2 mg/l primary concentration for the duration of 150 min, at the frequency of 130 kHz and a 500 W power supply at pH levels 3, 7 and 11 were 41.01%, 43.32% and 47.14% respectively. These results demonstrate that the efficiency of ultrasonic waves to remove BPA, in pH 11 was more than 3. Results also showed that removal efficiency of BPA was optimized at pH 11, at the frequency of 130 kHz and power supply levels of 300 and 500 at 40.79% and 47.14% at frequency 35 kHz, power supplies of 300 and 500 at about 22.88% and 23% respectively.

Results demonstrate that the effect of frequency on increasing efficiency was more than that of power and that there was a higher rate of destruction of BPA at high frequency. It was noticed that the usually the rate of destruction of BPA by ultrasound waves is slow. Results showed that it was the effect of ultrasonic frequency in oxidation of BPA that was the main reaction to supply hydroxyl radicals during cavitations. This condition shows that the rate of the reaction related to hydroxyl radical (production of hydrogen peroxide and BPA) was more at higher frequency. Different rates of destruction can be attributed to low efficiency. The effect of ultrasonic frequency on the removal of Bisphenol A can be attributed to several factors; for example, when the frequency increases, the cavitation threshold becomes higher, the maximum temperature attained in the collapse decreases, the lifetime of bubbles becomes shorter and the size of cavitating bubbles decreases.

![Fig. 1. Typical scheme used for degradation of surfactant.](image-url)
In this case, the bubbles did not have enough time to store energy because the duration of collapse was shorter at higher frequencies. All these factors lead to relate this behavior with a lower yield of \( \cdot \text{OH} \) generation as the frequency is increased. The production of hydroxyl radicals and the low possibility of ejection of these radicals from inside the cavitations globe in the solution were affected by the frequency 35 kHz. The destruction of BPA occurred at the frequency 130 kHz at a dose of more than 35 kHz [30,31].

The effect of sonication time was tested for various durations ranging from 1 to 180 min for the removal of BPA in optimizing conditions. Fig. 2 reveals the effect of sonication time on removal efficiency of BPA by ultrasonic waves. Fig. 2 demonstrated that removal efficiency increased with increased sonication time and the most efficient removal of BPA occurred at the sonication duration of 150 min. However, removal efficiency decreased with an increased concentration of BPA, for example evaluations for removal efficiency in optimizing conditions for BPA with concentrations of 2, 5, 20 and 50 mg/l, were 47.14, 37.6, 33.89 and 28.66 respectively.

Results demonstrated a decrease in the amount of removal with increased concentration of BPA indicating that there was increased competition for reaction with hydroxyl radicals at high concentrations. Furthermore when concentration in the liquid phase is increased it might put a partial pressure increase on the cavitations bubbles thus decreasing the temperature at which destruction occur and this can affect removal efficiency. Also, BPA concentration can cause destruction in the zone material (inner bubble, between zone and inner solution) effecting the removal rate. Generally, the process of removal of BPA by ultrasonic waves is slow. In an aquatic solution, the formation of \( \cdot \text{H} \) and \( \cdot \text{OH} \) radicals caused by cavitations serves to destroy organic materials in inner or outer bubbles [32,33].

Those materials in which destruction occurs inside the bubbles demonstrate a higher destruction rate than do processes in which materials are destroyed outside bubbles. Material nearer the bubbles, have a higher removal rate because of access to radicals, the oxidation process of BPA becomes more difficult with the ultrasonic technique [33].

### 3.1.1. Role of ultrasonic wave combined with \( \text{H}_2\text{O}_2 \)

When the aqueous solution is ultrasonically irradiated in the presence of \( \text{H}_2\text{O}_2 \), hydroxyl radicals are produced by cavitation. The hydroxyl radical exhibits a high oxidation potential and can directly degrade organic pollutants; however, hydroxyl radicals has a very short lifetime and tend to combine with one another to form \( \text{H}_2\text{O}_2 \). The following reactions occur under ultrasonic irradiation in the presence of \( \text{H}_2\text{O}_2 \) [34–35].

\[
\text{H}_2\text{O} \rightarrow \text{H}^\cdot + \text{OH} \\
\text{H}_2\text{O}_2 \rightarrow 2\text{HO}^\cdot \\
2\text{HO}^\cdot \rightarrow \text{H}_2\text{O}_2 \\
\text{O}_2 \rightarrow 2\text{O}^\cdot \\
\text{O}^\cdot + \text{H}_2\text{O} \rightarrow 2\text{OH} \\
\text{H}^\cdot + \text{H}_2\text{O} \rightarrow \text{H}_2 + \text{OH} \\
\text{H}^\cdot + \text{O}_2 \rightarrow \text{OOH} + \text{O} \\
2\text{OOH} \rightarrow \text{H}_2\text{O}_2 + \text{O}_2 \\
\text{H}_2\text{O}_2 + \text{O}^\cdot \rightarrow \text{H}_2\text{O} + \text{HO}_2^\cdot \\
\text{HO}_2^\cdot + \text{H}_2\text{O} \rightarrow \text{H}_2\text{O} + \text{OH} + \text{O}_2 \\
\text{BPA} + \text{OH}^\cdot \rightarrow \text{BPAOH}^\cdot \\
\text{BPAOH} + \text{O}_2 \rightarrow \text{BPA–OH} + \text{OOH}^\cdot
\]

Hence from the above reactions it is clear that the ultrasonic assistance leads to the more breakdown of the \( \text{H}_2\text{O}_2 \). Hence the organic degradation and removal of Bisphenol A increases in the presence of combined effect of ultrasonic radiation with \( \text{H}_2\text{O}_2 \).

Similar studies have been done in this field by other researchers and results demonstrate various outcomes. For example, Kejiazhang et al. (2010) concluded that in field destruction BPA with application ultrasonic irradiation at frequencies of 400, 670, 800 kHz and intensities of 0.9, 1.8 and 3 W/cm² assisted by low concentration of hydrogen.

### Table 2
**Specification of water natural samples.**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Concentration (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
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</tr>
<tr>
<td>( \text{SO}_4 )</td>
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</tr>
<tr>
<td>( \text{NO}_3^- )</td>
<td>17</td>
</tr>
<tr>
<td>( \text{Cl}^- )</td>
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</tr>
<tr>
<td>( \text{Mg}^{2+} )</td>
<td>75.9</td>
</tr>
<tr>
<td>( \text{Na}^+ )</td>
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<tr>
<td>( \text{Ca}^{2+} )</td>
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</tr>
<tr>
<td>TDS</td>
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</tr>
<tr>
<td>EC(μs/cm)</td>
<td>1459</td>
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<tr>
<td>Alkalinity (mg/l as CaCO₃)</td>
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</tbody>
</table>

### Table 3
**Average removal efficiency (percent) of BPA in ultrasonic process with frequency 35 kHz and power 300 W at retention time optimizing 150 min at pH and different concentrations.**

<table>
<thead>
<tr>
<th>pH</th>
<th>Concentration initial of BPA (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2</td>
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<td>3</td>
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<tr>
<td>7</td>
<td>22.67</td>
</tr>
<tr>
<td>11</td>
<td>22.88</td>
</tr>
</tbody>
</table>

### Table 4
**Average removal efficiency (percent) of BPA in ultrasonic process with frequency 35 kHz and power 300 W at retention time optimizing 150 min at pH and different concentrations.**

<table>
<thead>
<tr>
<th>pH</th>
<th>Concentration initial of BPA (mg/l)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>2</td>
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<tr>
<td>3</td>
<td>20.26</td>
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<tr>
<td>7</td>
<td>22.59</td>
</tr>
<tr>
<td>11</td>
<td>23</td>
</tr>
</tbody>
</table>

### Table 5
**Average removal efficiency (percent) of BPA in ultrasonic process with frequency 130 kHz and power 300 W at retention time optimizing 150 min at pH and different concentrations.**

<table>
<thead>
<tr>
<th>pH</th>
<th>Concentration initial of BPA (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2</td>
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<tr>
<td>3</td>
<td>36.95</td>
</tr>
<tr>
<td>7</td>
<td>40.61</td>
</tr>
<tr>
<td>11</td>
<td>40.79</td>
</tr>
</tbody>
</table>

### Table 6
**Average removal efficiency (percent) of BPA in ultrasonic process with frequency 35 kHz and power 300 W at retention time optimizing 150 min at pH and different concentrations.**

<table>
<thead>
<tr>
<th>pH</th>
<th>Concentration initial of BPA (mg/l)</th>
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<tbody>
<tr>
<td></td>
<td>2</td>
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<td>3</td>
<td>20.26</td>
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<td>7</td>
<td>22.59</td>
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<td>11</td>
<td>23</td>
</tr>
</tbody>
</table>
peroxide, showed that the highest rate of BPA removal was achieved at the frequency of 800 KHz [36]. Guo and Feng (2009) investigated the effect of ultrasonic waves, with a frequency of 20 kHz and different intensities in the removal of BPA [37].

Results showed that with an increased intensity of the ultrasonic wave from 20 to 80 W/cm², removal efficiency of BPA also increased [37]. Inoue et al., (2008) also studied the removal of BPA by ultrasonic waves. The research concluded that removal efficiency increased at a frequency of 404 kHz with an increase of ultrasonic intensity from 3.5 up to 12.9 kW/m² and after 10 h of ultrasonic wave irradiation, BPA destruction was complete [38].

### 3.2. Effect of H₂O₂

The effects of different concentrations of H₂O₂ at different pH levels to remove BPA are presented in Table 7. As demonstrated in Table 7 the efficiency of H₂O₂ with an initial concentration of 5 mg/l at pH = 11 for removal of BPA at concentrations of 2, 5, 20 and 50 mg/l was 10.96, 9.03, 8.4 and 6.11% respectively, indicating low efficiency of H₂O₂ with an initial BPA concentration of 5 mg/l. So removal efficiency of H₂O₂ was dependent on its initial concentration.

Firstly, pH affects the formation of ·OH. In the presence of dissolved oxygen, water molecules can generate ·OH, hydrated electrons (e⁻aqu), and H· under UV irradiation.

\[
\text{H}_2\text{O} + \text{hv} \rightarrow \text{e}^-_{\text{aqu}} + \text{H}^+ + \text{·OH} + \text{H}^+ \quad (13)
\]

Hence in acidic solutions, the formation of ·OH will be suppressed as shown in Eq. (13). Moreover, H⁺ will have a scavenging effect on ·OH, causing the decrease in degradation rate. Secondly, pH influences the ionization of reactant compounds and products. As a result, alkaline condition is favorable to the photo-degradation of target organic Bisphenol A dye. As a high level pH produces more hydroxyl radicals resulting in better removal of BPA, this antioxidant effect on the removal of BPA can be considered as important [39–41]. Evaluations for removal efficiency of BPA in optimum conditions, in pH levels 3, 7 and 11 were 20.65, 25.10 and 29.66 respectively, indicating an increase of efficiency in an alkaline pHs.

<table>
<thead>
<tr>
<th>pH</th>
<th>Concentration initial of BPA (mg·l⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>41.01</td>
</tr>
<tr>
<td>7</td>
<td>43.32</td>
</tr>
<tr>
<td>11</td>
<td>47.14</td>
</tr>
</tbody>
</table>

The effect of retention time was investigated at durations of 1–90 min for the removal of BPA in optimizing conditions. Effects of retention time over removal efficiency are shown in Fig. 3. Just as demonstrated in Fig. 3, the removal efficiency is increased with an increased in the retention time from 15 min to 45 min and then remained approximately fixed after 45 min. Thus results demonstrated that efficiency of H₂O₂ decreased with an increased concentration of BPA. This decrease in the amount of removal relating to the initial concentration of BPA can be explained as follows; as removal rates were similar in all solutions, amounts of H₂O₂, pH levels and retention durations, so amounts of radicals produced in each of the three samples were also similar, therefore insufficient hydroxyl radicals were produced at high concentrations of BPA. So more efficient removal was evident in those solutions containing lower concentrations of BPA [42,43].

### 3.3. Effect of combined ultrasonic process and H₂O₂

Results of this experiment indicated the efficiency of BPA destruction at initial concentration of 2 mg/l, at pH = 11, ultrasonic frequency of 130 kHz and power 500 W in 30 mg/l H₂O₂ was 98.65%, but in the same conditions the efficiency removal by ultrasound wave irradiation alone was about 47% and by H₂O₂ alone it was 30%. Fig. 4 demonstrates the efficiency of processes to remove BPA at different treatment durations.

Results demonstrated that these processes were more effective for BPA removal in combination than they were individually and there was significant different between single and combined processes. This was because of the synergic effect of the two mechanisms on oxidation. These mechanisms involve ultrasonic destruction and oxidation of H₂O₂. During the research, amounts of hydroxyl radicals affected the

<table>
<thead>
<tr>
<th>BPA (mg/l)</th>
<th>H₂O₂ (mg/l)</th>
<th>pH</th>
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</thead>
<tbody>
<tr>
<td>2</td>
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<tr>
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</tr>
<tr>
<td>30</td>
<td></td>
<td>10.5</td>
</tr>
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</table>
oxidation rate of the matter. In terms of amounts of radicals, the combined system was more than the ultrasonic single system and the rate of combined oxidation systems was more than ultrasonic single system [43–45].

Results for the effect of concentration of hydrogen peroxide on removal efficiency of BPA by the combined process are shown in Fig. 5. It demonstrates considerable removal efficiency of BPA in optimum conditions by the ultrasonic process in the presence of hydrogen peroxide at concentrations of 5, 15 and 30 mg/l is 62.75%, 90.91% and 98.65% respectively, as more hydroxyl radicals were produced, indicating an increase of removal efficiency with an increased concentration of hydrogen peroxide.

Oxidation efficiency is increased as a result of increased hydrogen peroxide. While with an increased concentration of hydrogen peroxide the process subsided and the rate of removal decreased. Because hydrogen peroxide acts as a scavenger on radicals at very high concentrations it increases the amount of very active radicals in an environment [45–47].

Fig. 6 shows the effect of pH and initial concentration on removal efficiency in the combined process. As indicated in Fig. 6, with an increase of pH, there is increased efficiency of ultrasonic and hydrogen peroxide in the hybrid process. It also demonstrates that hybrid process efficiency decreases with an increased initial concentration of BPA. The process is more efficient in low concentrations because there are more free radicals produced at higher concentrations of BPA. And it is more effective in treating contaminants by facilitating oxidation and destruction of BPA [44,50,51].

Fig. 7 shows the effects of frequency and power on the efficiency of hybrid process. Results demonstrate that efficiency is increased with increasing power and frequency. It can be concluded that reactions were affected by an addition of hydrogen peroxide in reactions 14 and 15 [44–50].

\[
\begin{align*}
\text{OH} + \text{H}_2\text{O}_2 & \rightarrow \text{H}_2\text{O} + \text{HO}_2 \quad (14) \\
\text{HO}_2^- + \text{OH} & \rightarrow \text{H}_2\text{O} + \text{O}_2 \quad (15)
\end{align*}
\]

In a study by Zhang et al., (2011) on removal of BPA by ultrasonic waves assisted by low concentration of H$_2$O$_2$ results showed that by adding of H$_2$O$_2$ removal efficiency was improved by treatment with ultrasonic waves [36]. Gültekin and İnce (2008) also investigated effects of different factors in removal of BPA by ultrasonic waves; results demonstrated that adding H$_2$O$_2$ and the subsequent formation of hydroxyl radicals could increase removal efficiency. So it was demonstrated in that research that with an increased concentration of BPA, removal efficiency decreased [49].

Torres and et al. (2008) studied effects of saturation gases (oxygen, argon and air), concentration of BPA (0.15–460 μMol/l), ultrasonic frequency (300–800 kHz) and power (20–80 v) in removal of BPA with ultrasonic waves. Results showed that removal was most efficient for 118 μMol/l of BPA at frequency 300 kHz and power 80 W with oxygen as the saturated gas [50].

3.4. Effect of ultrasonic and H$_2$O$_2$ processes on natural water sample in optimizing conditions

Removal efficiency of BPA from natural and synthetic samples in optimum conditions is shown in Fig. 8. As indicated in Fig. 8, removal
efficiency of BPA from synthetic water samples in optimizing conditions by ultrasonic, H₂O₂ and ultrasonic assisted by H₂O₂ processes 47.14%, 30.1%, 98.65% respectively and for natural water samples 29.51%, 14.81% and 78% respectively which showed that removal efficiency of BPA in the natural sample decreased more than in the synthetic samples. The cause of this decrease of removal efficiency can be explained by the presence of anions and cations in the natural water sample served as an interference that decreased the rate of removal in similar conditions relative to synthetic samples. Also, turbidity exists in natural water samples, and is a nuisance factor in the removal of BPA, as propounded by other tri-process studies. Since the decrease of removal efficiency in natural water samples was not so considerable, studying the processes of removal of BPA is useable from water with such conditions [47-50].

3.5. Comparable studies with other methods

The combined effect of ultrasonic wave with that of H₂O₂ was well studied in the present work, the developed method leads to more removal of Bisphenol A in comparison to previously developed ozone methods and concentration. Table 8 lists the comparison of removal percentage of Bisphenol A using various ozone concentration methods. The removal percentage for the proposed method in comparison with all the others methods is preferable and superior to the literature which shows satisfactory removal performance for Bisphenol A as compared to other reported adsorbents [52–53].

4. Conclusions

The ultrasonic treatment combined with H₂O₂ had the highest efficiency for the removal of BPA. Also, efficiency of removal of BPA, with pH, H₂O₂ concentration, frequency, power and sonication time had a direct relation and to concentration of BPA. Effects of frequency in removal BPA were more than the effect of power. Analysis of results of natural water samples indicates towards a decrease of removal efficiency as compared with synthetic samples. With attention to frequency and power in processes tested in this study, a general conclusion can be drawn that removal efficiency of BPA can be explained as follows:

US130 + H₂O₂ > US35 + H₂O₂ > US130 > US35 > H₂O₂

With attention to the decrease of removal efficiency in natural samples water and high-energy consumption, it suggests that this method can be used in small-scale industrial systems or combined with other advanced oxidation processes.

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References