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# Kinetic and statistical analysis of Bisphenol A in wastewaters by ozonation and electrooxidation–ozonation processes

N. Pınar Tanatti[a,*], Emirhan Balkaya[b], İ. Ayhan Şengil[b]

[a]*Department of Environmental Protection Technologies, Sakarya University of Applied Sciences, 54100 Sakarya, Turkey, Tel. +90 264 2957356; Fax: +90 264 2955601; email: ptanatti@sakarya.edu.tr (N.P. Tanatti)*
[b]*Department of Environmental Engineering, Engineering Faculty, Sakarya University, 54100 Sakarya, Turkey, emails: emirbalkayaa@gmail.com (E. Balkaya), asengil@sakarya.edu.tr (İ.A. Şengil)*

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**ABSTRACT**

The purpose of this study is to investigate the treatability of synthetic Bisphenol A (BPA) solution by electrooxidation–ozone (EO+$O_3$) and ozone ($O_3$) processes. The BPA removal efficiencies of both combined method and $O_3$ methods were compared and the combined process gave better results than the $O_3$ process. For the BPA removal efficiencies, the experimental parameters including pH, current density, ozone dose, and reaction time parameters were optimized by changing the one parameter at a time. The BPA removal efficiencies have been obtained for EO+$O_3$ as 90.68%, and for $O_3$ as 83.92% under the optimum conditions (pH 7, 1.5 g/L h ozone dose, 4.57 mA/cm$^2$ current density, and 25 min reaction time) by using the Ti/$RuO_{2(0,70)}$–$IrO_{2(0,30)}$ anode electrode. The experimental data, obtained from EO+$O_3$ and $O_3$ process has been evaluated by applying three different kinetic theories, that is, first, second, and pseudo-second-order. The second-order-kinetic model has been found to be the most suitable model for both processes and the regression coefficients ($R^2$) has been found as 0.94 and 0.92 for EO+$O_3$ and $O_3$, respectively. The reaction rate constants ($k$) have been also calculated as $1.8 \times 10^{-2}$ and $9 \times 10^{-3}$ L/mg min for EO+$O_3$ and $O_3$, respectively. The obtained results showed that the BPA removal efficiency of EO+$O_3$ process has superiority over $O_3$ process. The detailed investigation revealed that no by-product has been observed during the BPA removal.

*Keywords:* Bisphenol A; Electrooxidation; Ti/Ru-Ir electrode; Ozone oxidation

## 1. Introduction

BPA is an organic colorless crystalline synthetic compound with the chemical formula of $(CH_3)$–C–$(C_6H_4$–$OH)_2$. The chemical structure of BPA consists of two methyl functional groups that are connected by a bridge to the phenol rings [1]. BPA is one of the estrogenic endocrine-disrupting chemical (EDC) and it is used largely in the production of polycarbonate plastics and epoxy resins. In recent years, polycarbonate materials are used to produce packing materials been exposed to food such as storage containers, cups, plates, feeding bottles, plastic bottles, and microwave ovenware [2]. The epoxy resins also used inside the food and beverage containers, which might be more dangerous [3,4]. BPA is not only used for these mentioned products but also used for the production of dental materials, medical devices, sunglasses, building materials, and thermal paper. As can

* Corresponding author.

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be seen, BPA is one of the largest amounts of produced chemicals and the amount of it increases every other year. During those material productions, tons of BPA are released to the environment in the gas form or dissolved in water. Due to the high amount of BPA usage, the amount of it should be monitored in nature continuously. The BPA contamination may occur through food contamination, which might be leached into the food from packing materials or contamination thorough other means to the soil, water, and air [2,5].

BPA is not produced naturally; it is released by a human being in different ways. Although BPA containing wastewater is not allowed to be discharged to the environment, traces of BPA are directly given to receiving water bodies via permitted amounts. It is released not only by wastewater but also fugitively released to the air during the processing of BPA. Although BPA can be removed easily in wastewater treatment plants, it can be still detected in industrial wastewaters and it accumulates in those waters and still needs to be treated to remove it completely. Although it is easily biodegradable and rest can be adsorbed onto sediments in receiving waters, it is better to be removed or filtered out before discharging to the environment completely [6,7].

The importance of BPA removal from wastewaters comes from its effect on the human body. It is an endocrine disruptor and it imitates the body's hormones [8]. This will change all the production, secretion, action, and all other functions of body. It behaves in a very similar way to estrogen and other various hormones [9]. Therefore, it has to be removed completely from the environment. In recent years, BPA has been studied by many researcher and a number of publications have been published in the literature in both analysis and removal of BPA from wastewaters.

Physical treatment, biological treatment, and advanced oxidation processes are used for BPA removal from water [10]. Advanced oxidation processes together with the biological treatment is an alternative option for the BPA removal for the water and the wastewater. The combined methods have improved the performance of the BPA removal and inhibition of the estrogenic activation [11]. Although, the activated carbon adsorption is one of the BPA removal methods that has been considered by many researchers, it has a limitation because of the low hydrophobicity of the BPA compound [12].

BPA removal has been studied with ozonation ($O_3$) and electrooxidation (EO) processes as advanced oxidation processes. Due to high efficiency in BPA treatment, ozone application has a high potential [13,14]. BPA removal has been also examined by using different electrode systems like BDD, Pt, Ti/BDD, and Ti/$RuO_2$ electrodes [15].

In this paper, we examine the BPA removal using a synthetic BPA sample using ozone ($O_3$) and electrooxidation (EO)+ ozone ($O_3$) processes. We use Ti/$RuO_{2(0.70)}$–$IrO_{2(0.30)}$ anode and stainless-steel cathode in the EO+$O_3$ process. BPA removal efficiency was studied using $O_3$ and combined method, EO+$O_3$ processes, and all the important parameters were optimized including pH, $O_3$ dose, and reaction time for $O_3$ process and conductivity, pH, $O_3$ dose, current density, and reaction time parameters for EO+$O_3$ process. The kinetic consideration, mechanisms, and the statistical analysis were performed on all the collected data for BPA removal from synthetic mixtures.

## 2. Materials and methods

### 2.1. Material

BPA sample was purchased from Acros Organics (Belgium). The purity of the sample is 97% Bisphenol A and it was used without further purification. The synthetic solutions were prepared from this sample. During the electrocoagulation process to provide the solution conductivity, $Na_2SO_4$ (sodium sulfate) has been used in BPA solutions. The pH of the solutions was adjusted by using $H_2SO_4$ (sulfuric acid) and NaOH (sodium hydroxide).

BPA concentrations have been measured by Shimadzu UV-Vis (Japan) 1700 spectrophotometer. The pH measurements of BPA samples have been done by using the Hanna brand (US) pH meter. MTOPS magnetic stirrer has been used in the reactor, and the required current for electrooxidation has been obtained by the DC power supply of Yıldırım Elektronik (Turkey). Additionally, the Sabo Electronics (Turkey) SL-10 model ozone generator has been used for $O_3$ production.

### 2.2. Synthetic BPA sample preparation and measurement

The stock solution of BPA was prepared by dissolving 25 mg of BPA in 1 L Milli-Q water. All the other solutions were prepared by diluting from this stock solution. The maximum absorption of BPA was determined to form the absorption spectrum as 278 nm in water. Hence, the calibration graph has been prepared between 0.5 and 25 mg/L at 278 nm. The calibration equation was obtained from the calibration graph as in the following equation.

$$\mathrm{BPA}\left(\mathrm{mg/L}\right) = \frac{0.0129}{\mathrm{ABS}} \tag{1}$$

### 2.3. Reactor design

The organic pollutants are oxidized either by direct reaction with molecular ozone or by indirect reaction with $OH^{\bullet}$ radical which is occurred as a decomposition of ozone under the alkaline conditions. Molecular ozone has selectively reacted with compounds containing C=C bonds, some functional groups (OH, $CH_3$, and $OCH_3$) and anions (N, P, O, and S) [16]. However, $OH^{\bullet}$ radical does not have selectivity and the competition remains practically the same during the entire oxidation [17].

The reactor designs of the $O_3$ and EO+$O_3$ processes have been shown in Figs. 1 and 2, respectively. They consist of an ozone generator with 15 g/L h capacity, magnetic stirrer, and a glass reactor. The basic principle of electrooxidation treatment is to oxide the contaminants in water and wastewater using the insoluble metal/metal oxide electrodes (such as Ti, Ru, Pt, and stainless steel) [18].

The electrodes are connected in monopolar position in the reactor and the distance between them was 1.20 cm during all the experiments. The dimensions of Ti/$RuO_{2(0,70)}$–$IrO_{2(0,30)}$ anode electrode is 12 cm × 6 cm × 0.21 cm, and the stainless steel cathode electrode is 12 cm × 6 cm × 0.20 cm. The DC current was applied to the electrodes. The maximum applied voltage and current were 30.3 V and 5 A, respectively. The reactor was a glass container with a volume of

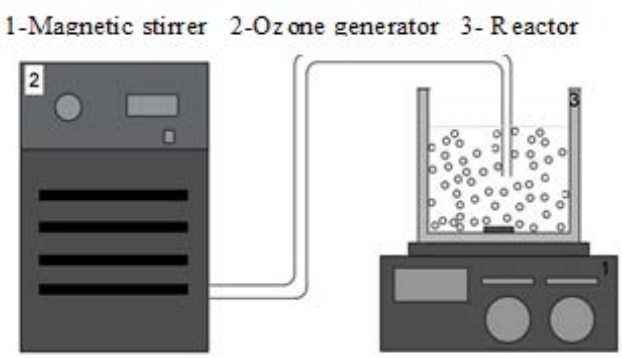


Fig. 1. Schematic diagram of $O_3$ experimental setup.

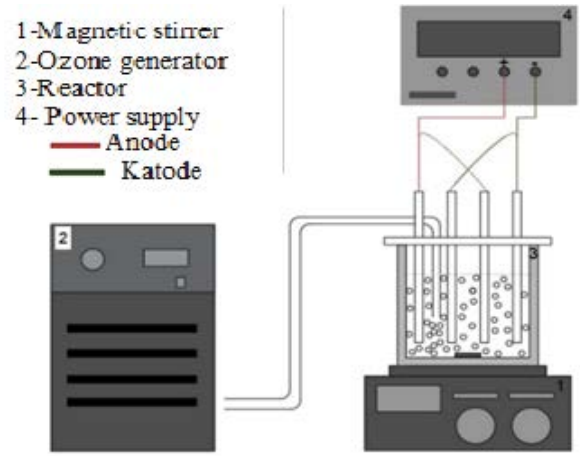


Fig. 2. Schematic diagram of EO+$O_3$ experimental setup.

600 mL. The depth of the immersion of the electrodes were 4.3 cm. The total surface area of four electrodes (two anodes and two cathodes) was 218.37 $cm^2$.

## 3. Results and discussion

### 3.1. Effect of pH in $O_3$ and EO+$O_3$ processes

The mechanism of electro oxidation and ozone processes can be controlled by changing the pH of the solution. BPA removal efficiency highly depends on the pH of the solution, therefore different pH values have been tried to optimize the method. The pH optimization has been done by keeping the other parameters constant as 25 mg/L as an initial BPA concentration, 0.9 g/L h as ozone dose, 11.44 mA/$cm^2$ as current density, 20 min as reaction time, and 0.75 mg/L as a $Na_2SO_4$ concentration.

The effect of pH on BPA removal efficiency has been shown in Fig. 3. Using the $O_3$ process, the BPA removal efficiency has been obtained as 35.53% at pH 3 and 49.87% at pH 9. Also, using the EO+$O_3$ process, the BPA removal efficiency has been obtained as 38.82% at pH 3 and as 52.08% at pH 9. Since the pH of the synthetic BPA solution is 6.97, pH 7 has been chosen for both processes. Therefore, the BPA removal efficiencies at pH 7 for $O_3$ and EO+$O_3$ processes are 50.36% and 57.32%, respectively. The results are showing that the BPA removal efficiency in EO+$O_3$ process is higher than the $O_3$ process.

The BPA removal efficiency of a BPA sample was investigated for the initial concentration of 8 mg/L with 2.2 mg/L ozone dose and 14 min ozone duration. In these experiments, it has been shown that the higher the solution pH, the higher the BPA removal efficiency. Therefore, the BPA removal efficiencies have been observed at pH 2, 5, and 7 as 53%, 79%, and 92%, respectively in the ozone process. However, the removal efficiency of BPA slowed down over pH 10.0 [19]. Similar pH-dependent results have been found by Zhang et al. [20] for pH-dependent BPA removal.

BPA removal sample has been mineralization by anodic oxidation using BDD electrode. In this study, the experiments have been done for pH 2, 6, and 10 under the conditions which are 20 mg/L as initial concentration, 0.1 M as $Na_2SO_4$ concentration, and 360 min as reaction time. The TOC removal efficiencies at pH 2, 6, and 10 have been obtained as 76%, 92%, and 86%, respectively [15].

### 3.2. Effect of $O_3$ dose in $O_3$ and EO+$O_3$ processes

The organic pollutants are oxidized either by direct reaction with molecular ozone or by indirect reaction with $OH^{\cdot}$ radical which is occurred as a decomposition of ozone under the alkaline conditions [21,22].

The $O_3$ and EO+$O_3$ processes have been studied between 0.3 and 1.5 g/L h $O_3$ dose. The effect of $O_3$ dose on BPA removal efficiencies have been shown in Fig. 4. While the BPA removal efficiency has been found for $O_3$ and EO+$O_3$ processes as 19.90% and 21.11%, respectively at the dose of 0.3 g/L h $O_3$, the removal amounts were found as 83.90% and 93.75%, respectively at the dose of 1.5 g/L h $O_3$. The results are showing that the removal efficiency of EO+$O_3$ process has been found % 10 more effective than the $O_3$ process at the dose of 1.5 g/L h $O_3$. Fig. 4 shows the removal efficiency of BPA for both $O_3$ and EO+$O_3$ process for 1.5 g/L h $O_3$ dose and the final concentrations were found as 4.03 and 1.56 mg/L, respectively.

In another study, 22.8 mg/L initial concentration was used to investigate the BPA removal. The $O_3$ does was $8.96 \times 10^{-1}$ and total reaction time was kept as 80 min. The results showed that the total $O_3$ amount was 14.94 mol for the complete removal of 1 mol BPA from wastewater [23]. The different BPA concentrations were also studied ranging from 10 to 60 mg/L and %100 removal was obtained throughout all the concentrations. The results showed that 1 mol of BPA requires 1.5–1.8 mol $O_3$ for the complete removal [14].

### 3.3. Effect of the current density in $O_3$ and EO+$O_3$ processes

The current density studies were conducted in the range of 2.28–13.73 mA/$cm^2$ for both $O_3$ and EO+$O_3$ process. The effect of the current density on the BPA removal efficiency is shown in Fig. 5. The various current densities 2.28, 4.57, 9.15, 11.44, and 13.73 mA/$cm^2$ have been investigated for the removal of different BPA initial concentrations and the final remaining amounts were found as 3.45, 3.06, 2.58, 1.35, and 1.88 mg/L, respectively. The final results are showing that the higher the current density, the higher the BPA removal efficiency till a

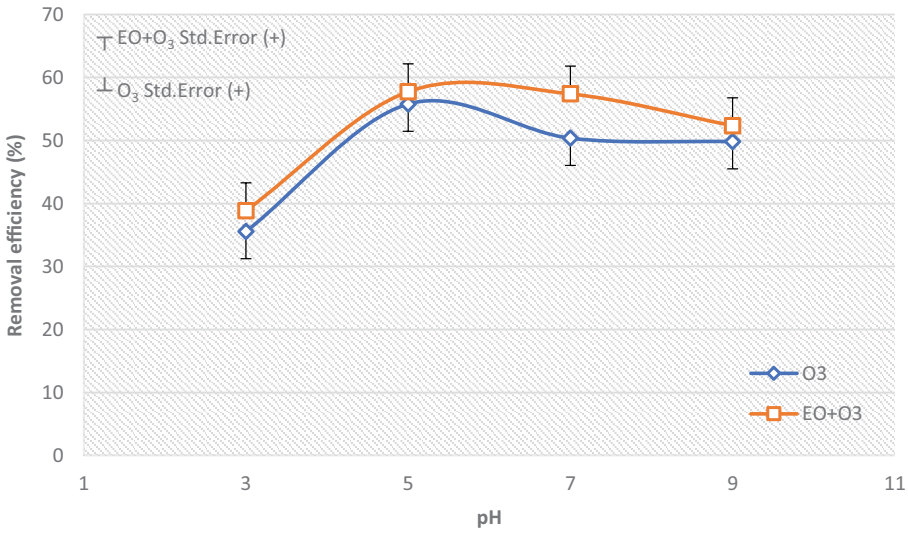


Fig. 3. Effect of pH on the BPA removal efficiency ($C_{0BFA}$ = 25 mg/L, $O_3$ dose = 0.9 g/L h, $i$ = 11.44 mA/cm$^2$, and $t$ = 20 min).

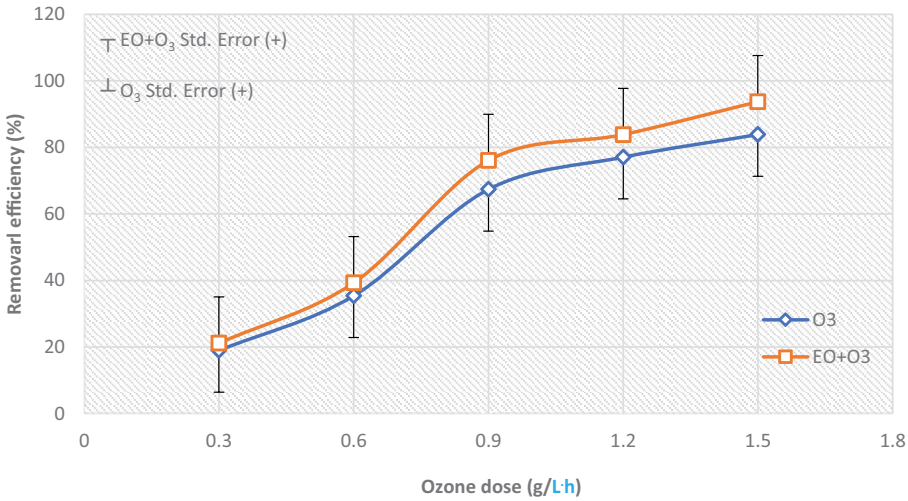


Fig. 4. Effect of $O_3$ dose on the BPA removal efficiency ($C_{0BFA}$ = 25 mg/L, pH = 7, $i$ = 11.44 mA/cm$^2$, and $t$ = 25 min).

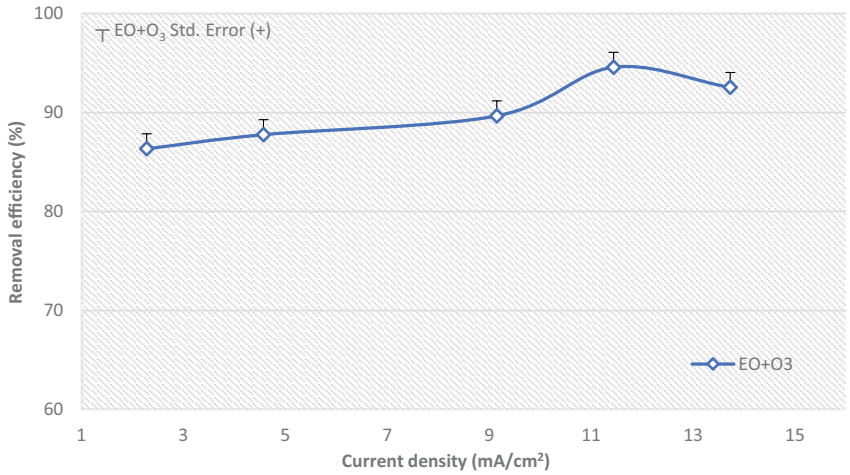


Fig. 5. Effect of current density on the BPA removal efficiency in EO+$O_3$ process ($C_{0BFA}$ = 25 mg/L, pH = 7, $O_3$ dose = 1.5 g/L h, and $t$ = 25 min).

certain current density. When the current density reaches to 11.44 mA/cm², the removal efficiency starts decreasing. The results are showing that the highest BPA removal efficiency was obtained as 94.59% at the current density of 11.44 mA/cm². Although, the higher current density provides higher removal efficiency at 11.44 mA/cm², this high current density is not feasible economically. Therefore, in all the experiments 4.57 mA/cm² current density was used with 87.76% BPA removal efficiency.

In another study using 20 mg/L initial concentration, BPA has mineralized by using BDD electrodes. In this study, the experimental conditions were set as pH 6, 0.1 M $Na_2SO_4$ salt, 360 min reaction time, and 14.28, 25, and 37.5 current densities. The corresponding TOC removal efficiency for the BPA removal was examined using 14.28, 25, and 37.5 mA/cm² current densities and the obtained values were 50%, 62%, and 92%, respectively [15].

### 3.4. Effect of reaction time in $O_3$ and EO+$O_3$ processes

The reaction time parameter was studied for the BPA removal efficiency using $O_3$ and EO+$O_3$ processes. The result of this study is summarized in Fig. 6. As seen in the figure, the BPA removal efficiency increased rapidly in the first 25 min, after that the removal efficiency rate slowly decreased. The BPA removal efficiency has examined for both processes at 10th min and removal efficiencies were found as 44.19% and 42.50%, for $O_3$ and EO+$O_3$ process, respectively. The removal efficiency of both $O_3$ and EO+$O_3$ processes were investigated at 35th min and the removal efficiency of both processes increased to 86.20% and 91.92%, respectively. When the reaction time parameter is evaluated for the process cost, although 35 min time duration provides higher removal efficiency, 25 min reaction time was used in all the experiments. In this optimum reaction time, the removal efficiencies were obtained as 83.92% and 90.68% for $O_3$ and EO+$O_3$ process, respectively.

Fig. 7 shows the pH and $O_3$ dose on the BPA removal efficiency at 25th min. As seen in the Fig. 7, the most important factor that affects the BPA removal is $O_3$ does. Increase in the $O_3$ dose increases the BPA removal efficiency, whereas the process has not affected by the changes in the level of pH.

The pH, $O_3$ dose, and current density parameters were examined for EO+$O_3$ process in 25 min time duration and the results are shown in Figs. 8a and b. As seen from the given figures, $O_3$ and current density parameters affect the process more.

Fig. 8a shows the pH effect on BPA removal efficiency. The low pH values do not affect the BPA removal and the real pH effect becomes significant over pH 7. Fig. 8b shows the $O_3$ dose and current density on the BPA removal efficiency. In case of an increase in both parameters, the BPA removal efficiency increases.

The byproduct formation was investigated for both processes for BPA removal as seen in Fig. 9. Using the optimum conditions, each sample UV-vis spectrum was recorded between 200 and 800 nm, and spectrum profiles were

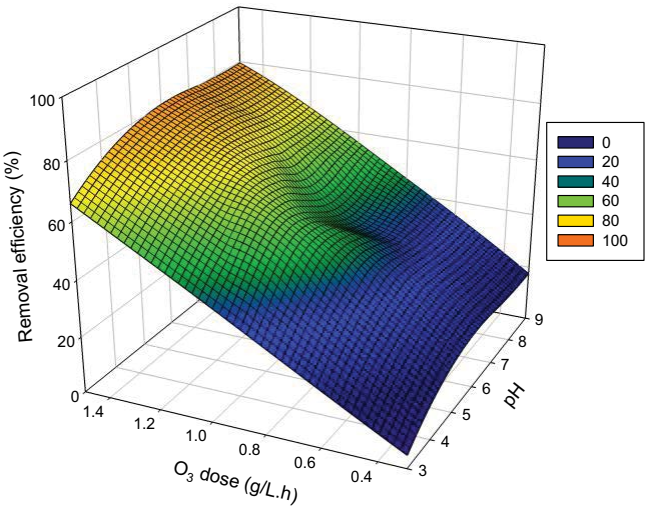


Fig. 7. Effect of pH and $O_3$ dose on BPA removal efficiency in $O_3$ process.

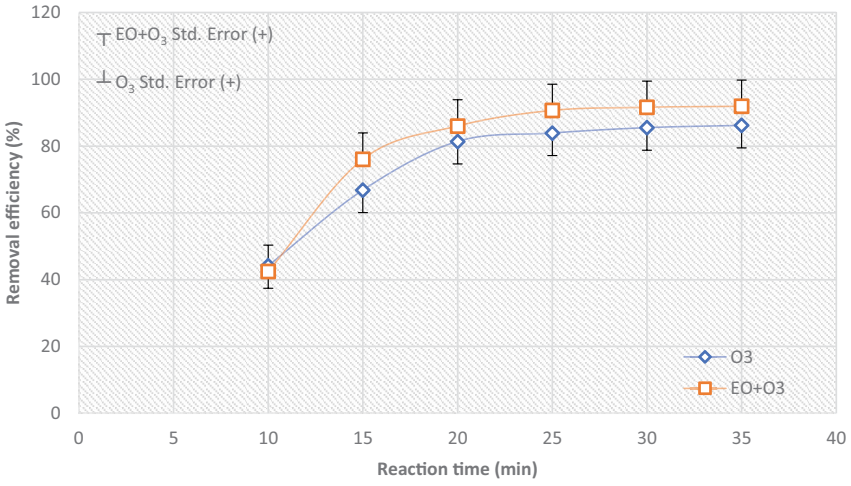


Fig. 6. Effect of reaction time on the BPA removal efficiency ($C_{0BFA}$ = 25 mg/L, pH = 7, $O_3$ dose = 1.5 g/L h, and $i$ = 11.44 mA/cm²).

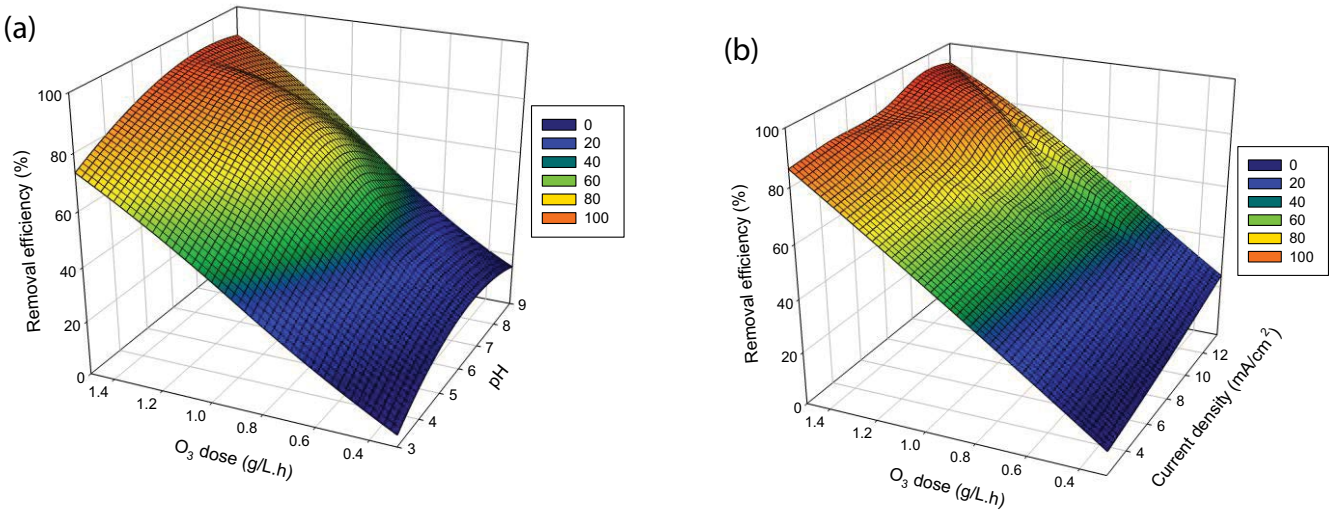


Fig. 8. Effect of (a) pH and $O_3$ dose and (b) $O_3$ dose and current density on BPA removal efficiency in EO+$O_3$ process.

compared for any changes. We did not observe any profile changes during the and after the experimental procedure. The maximum absorbance of BPA was recorded at 278 nm and the concentration decrease was calculated using this wavelength for each sample. The results showed that the BPA concentration decrease was higher for EO+$O_3$ process than the $O_3$ process. The results also proves that there was no byproduct formation for BPA removal in both process.

### *3.5. Kinetic analysis of BPA removal efficiency in $O_3$ and EO+$O_3$ processes*

Results obtained from $O_3$ and EO+$O_3$ process for the BPA removal have been used to study the kinetics of both process by three different models including the first-order, second-order, and pseudo-second-order models. All the data for kinetic studies were obtained using 25 mg/L initial concentration. The three different models given by the following equations:

$$\ln\frac{C_0}{C} = k.t \quad [24] \quad (2)$$

$$\frac{1}{C} - \frac{1}{C_0} = k \cdot t \quad [25] \quad (3)$$

$$\frac{t}{C} = \frac{1}{k_2 \cdot C_e^2} + \frac{1}{C_e} \cdot t \quad [26] \quad (4)$$

where $C_0$ is the initial BPA concentration (mg/L), $C$ is the final BPA concentration (mg/L), $k$ is the reaction rate constant (L/mg min), $t$ is the reaction time (min), $C_e$ is the concentration coefficient (mg/L), $k_2$ is the mean mass transport coefficient (1/min).

$O_3$ and EO+$O_3$ processes have been examined by using these three kinetic models and the regression coefficients ($R^2$) and the rate constants ($k$) are given in Tables 1 and 2, respectively. For both processes, the second-order kinetic

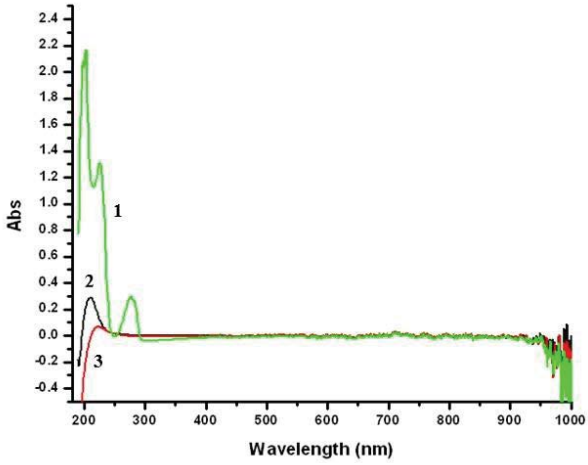


Fig. 9. Spectrum scanning wavelength-abs graph (1) synthetic BFA sample, (2) $O_3$ process applied BPA sample, and (3) EO+$O_3$ process applied BPA sample.

model was found as the most appropriate model. The regression coefficient and rate constant were found as 0.926 and 0.0091 L/mg min for $O_3$ process and 0.947 and 0.018 L/mg min for EO+$O_3$ process, respectively.

Therefore, the theoretical and experimental results of BPA effluent concentrations are given in Table 3.

Theoretical and experimental removal efficiencies obtained by the second-order kinetics models for $O_3$ and EO+$O_3$ processes and results are given in Fig. 10. As seen in the figure, the BPA removal efficiency rapidly increases in the first 20 min, and then it slows down. In the comparison of the experimental and theoretical results, both results overlap in the longer time periods.

### *3.6. Statistical analysis of BPA removal efficiency in $O_3$ and EO+$O_3$ processes*

The effect of the independent multiple variables onto the dependent variables has been studied using multiple

Table 1
$R^2$ and $k$ values as per kinetic models in $O_3$ process

| Kinetic model | $R^2$ | $k$ |
|---|---|---|
| First-order | 0.851 | 0.0550 |
| Second-order | 0.924 | 0.0091 |
| Pseudo-second-order | 0.752 | 0.0171 |

Table 2
$R^2$ and $k$ values as per kinetic models in EO+$O_3$ process

| Kinetic Model | $R^2$ | $k$ |
|---|---|---|
| First-order | 0.858 | 0.076 |
| Second-order | 0.947 | 0.018 |
| Pseudo-second-order | 0.689 | 0.017 |

regression analysis. The multiple regression analysis has performed with SPSS 22. In this study, the independent variables are pH, $O_3$ dose, current density, and reaction time, and the dependent variable is BPA removal efficiency. The regression coefficients ($R^2$) between the independent variables and the dependent variables are determined as 96.2% for $O_3$ process and as 96.1% for EO+$O_3$ process. The effect of the independent variables onto the dependent variables has given in Table 4 that represents the significance levels and the coefficients that are obtained for both processes. The obtained values of the independent variables and the BPA removal efficiency have shown some important statistical significance. The statistical relation between pH and removal efficiencies are not statically significant. Because the significance levels for both processes are more than 5%.

The coefficients obtained from the statistical analysis have been established between the independent and dependent variables given below:

$O_3$: 1.657 pH + 46.725 $O_3$ dose + 2.142 reaction time – 52.776

EO+$O_3$: 1.919 pH + 56.792 $O_3$ dose + 1.485 current density + 2.611 reaction time

The actual value and the calculated value of BPA removal efficiencies for both processes $O_3$ and EO+$O_3$ are shown in Fig. 11. The tendencies of the actual values and the calculate values for BPA removal efficiencies have almost seems the similar.

## 4. Conclusions

Two different advanced oxidation processes have been used on the BPA removal efficiencies. The optimum conditions for $O_3$ process have determined as pH 7, 1.5 g/L h $O_3$ dose, and 25 min reaction duration. Moreover, the optimum conditions for EO+$O_3$ are the same as the conditions which are given for $O_3$ process and additionally the current density condition was included as 4.57 mA/cm$^2$. Therefore, under the optimum conditions, the BPA removal efficiencies for $O_3$ and EO+$O_3$ processes was obtained as 83.92% and 90.68%, respectively. The result of this study was verified that the BPA removal was reached the highest efficiency for both processes, whereas, comparing to the $O_3$ process, it has seen that EO+$O_3$ process removal efficiency is higher than the $O_3$ process.

Table 3
Theoretical and experimental BPA concentrations as per second-order kinetics

| | Experimental ($O_3$) | Theoretical ($O_3$) | Experimental (EO+$O_3$) | Theoretical (EO+$O_3$) |
|---|---|---|---|---|
| Time (min) | BFA (mg/L) | BFA (mg/L) | BFA (mg/L) | BFA (mg/L) |
| 10 | 13.95 | 6.45 | 14.26 | 4.48 |
| 15 | 8.29 | 4.71 | 5.97 | 3.18 |
| 20 | 4.65 | 3.70 | 3.49 | 2.46 |
| 25 | 4.02 | 3.05 | 2.33 | 2.01 |
| 30 | 3.39 | 2.60 | 2.09 | 1.70 |
| 35 | 2.76 | 2.26 | 2.02 | 1.47 |

Table 4
Coefficient and significant values

| | $O_3$ process | | | EO+$O_3$ process | | |
|---|---|---|---|---|---|---|
| | Coefficient | Significance | Standard error | Coefficient | Significance | Standard error |
| Constant | –52.776 | 0.000 | | –83.699 | 0.000 | |
| pH | 1.657 | 0.073 | 0.895 | 1.919 | 0.058 | 0.991 |
| $O_3$ dose (g/L h) | 46.725 | 0.000 | 3.226 | 56.792 | 0.000 | 3.126 |
| Current density (mA/cm$^2$) | – | – | | 1.485 | 0.004 | 0.362 |
| Time (min) | 2.142 | 0.000 | 0.195 | 2.611 | 0.000 | 0.181 |

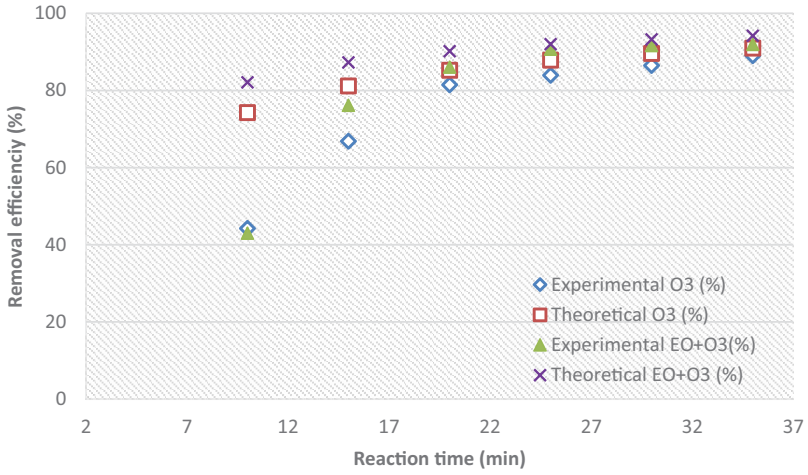


Fig. 10. Theoretical and experimental BPA removal efficiency as per second-order kinetics.

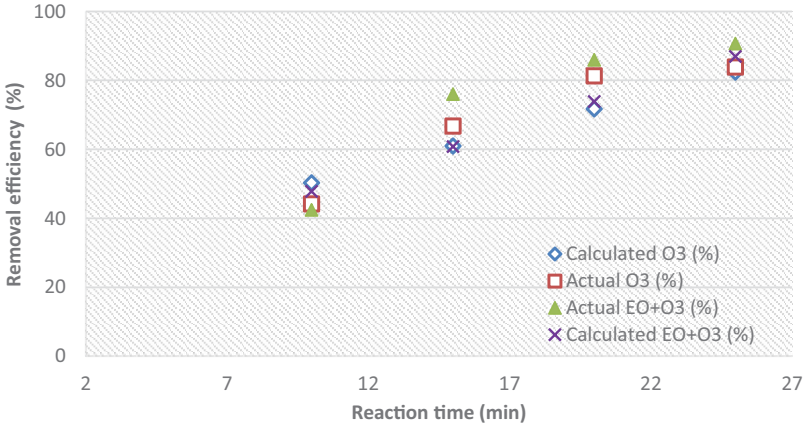


Fig. 11. Effect of electrolysis time on the BPA removal for multiple regression analysis.

## Symbols

| | | |
|---|---|---|
| BPA | — | Bisphenol A |
| $O_3$ | — | Ozone |
| EO+$O_3$ | — | Electrooxidation–ozone |
| $R^2$ | — | Regression coefficient |
| $k$ | — | Reaction rate constant |
| $C_{0BPA}$ | — | Initial BPA concentration |
| $C$ | — | Final BPA concentration |
| $C_e$ | — | Concentration coefficient |
| $k_2$ | — | Mean mass transport coefficient |
| $t$ | — | Reaction time |
| $i$ | — | Current density |

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