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Electro-Fenton treatment process for brewery wastewater: effects of oxidant concentration and reaction time on BOD and COD removal efficiency

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Abstract

The brewing process consumes and releases a large amount of wastewater into the environment. The objective of this study was to determine optimum operating conditions for the treatment of brewery wastewater using the Electro-Fenton (EF) process. The EF process has been applied to a wide variety of pollutants due to its ability to remove complex and recalcitrant water contaminants. Brewery wastewater contains large amounts of biodegradable and non-biodegradable compounds which are suitable for the EF process. The effect of hydrogen peroxide (H_2O_2) concentration and reaction time (RT) on the biological oxygen demand and chemical oxygen demand of the EF process was investigated. The brewery wastewater with an initial BOD and COD of 423.76 mg/l and 854.86 mg/l respectively was subjected to changing H_2O_2 concentrations of 250, 500, 1000, 2000, 3000, and 4000 mg/l and reaction times of 30, 45, 60, 90, and 120 min. Other conditions such as ferric-sulfate catalyst concentration, voltage, electrode spacing, and pH were maintained constant. The BOD and COD showed considerable changes after applying the EF process. Removal efficiencies for BOD and COD were significant ($p < 0.05$), up to 96.91% (423.76–13.04 mg/l) and up to 93.16% (854.86–58.5 mg/l), respectively. Results further showed increasing concentrations of hydrogen peroxide and reaction time favored BOD and COD removal. The efficiency was analyzed using analysis of variance (ANOVA) and graphical plot. The optimum BOD and COD removal of 88.96% (46.79 mg/l) and 93.16% (58.51 mg/l) was observed at 2000 mg/l hydrogen peroxide and 120 min reaction time. Optimized experimental conditions and performance have been discussed in the literature. The analysis of the treated wastewater shows that there is a significant reduction of BOD and COD compared with the raw wastewater. Therefore, it is suggested that the EF process be applied in the treatment of brewery wastewater for removing BOD, COD, and some other complex pollutants. The treated water can be reused or safely disposed into the environment.

Keywords: Brewery wastewater, Wastewater treatment, Electro-Fenton process, Biological oxygen demand (BOD), Chemical oxygen demand (COD)

Introduction

The world's water is a finite and precious resource, constantly under industrial, agricultural, and domestic pressure. Water scarcity concerns have soared in recent years as more countries around the world have implemented more stringent regulations to meet their demands. Water scarcity coupled with rapid population growth in some of these countries has brought increased attention to appropriate water use and disposal practices. According to the [58] report, water demand in the last decade has increased by more than 100%, and projections show that it is expected to increase in countries with developing economies. Industrial and agricultural processing industries have played a vital part in the economic development of these countries in past years, and with large-scale development and population growth, it is also expected to increase in the coming years [57]. Water quality concerns have been raised by international and governmental bodies around the world largely due to the accumulation of organic and inorganic suspended matter and nitrate as well as soluble phosphorus in the natural water bodies [26]. Different studies have shown that only a few industries in developing regions have their wastewater treated before disposal into the aquatic environment [17, 58]. Many of these contain chemicals that pose a risk to human health and have often been associated with loss of biodiversity and ecological damage. Some of them are persistent, toxic, and partly biodegradable and hence are not easily removed by conventional wastewater treatment plants [2, 44]. With recent environmental pollution problems, there is therefore a need to monitor, control, and develop sustainable and economically efficient methods for the treatment of industrial wastewater before its disposal into the environment [1, 52].

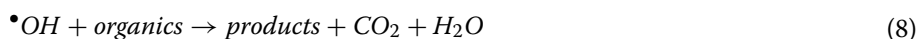
The brewery industry is water-intensive as a large volume of water is required for the daily production of beer. For instance, an average of 6.0 hl is required to produce 1 hl of clear beer. In the brewing process, water not only serves as the main ingredient of the beer but is also used in steam raising, cooling, washing of floors, packaging, and cleaning during and after each batch operation [18]. The wastewater effluents from the brewery process also contribute to soil pollution in the cases of inappropriate treatment and land discharge [43]; it is also reported to cause inhibition of seed germination, reduction of soil alkalinity, loss of soil manganese, and damage in agricultural products [1, 52].

A wide range of conventional and advanced methods have been adopted for the treatment of brewery wastewater [52, 56]. Some of the conventional treatment methods include anaerobic treatment with the recovery of biogas, followed by aerobic treatment. However, some phenolic compounds have been reported in this type of wastewater [8, 25, 53] which are not readily biodegradable and therefore cannot be removed by conventional methods. Some other effective technologies have also been proposed for brewery wastewater treatment; however, for such technologies to be implemented, it would require environmental regulations which can be costly and relatively complex [27, 52].

Advanced oxidation treatment processes have also been widely used in the treatment of distillery and brewery wastewater; they operate through the generation of hydroxyl radicals and other oxidant species to degrade organic compounds in wastewater [48]. Advanced oxidation process (AOPs) technologies can also be applied by a combination of hydrogen peroxide/ultraviolet irradiation ($\text{H}_2\text{O}_2/\text{UV}$), ozone/ultraviolet irradiation (O_3/UV), and ozone/hydrogen peroxide ($\text{O}_3/\text{H}_2\text{O}_2$), and ozone and hydroxyl radicals

($\bullet OH$), which are robust oxidants capable of oxidizing a wide range of organic compounds when dissolved in water [3, 10]. Several technologies have been successfully used in the removal of highly complex molecules that are bio-refractory in nature [9, 23, 29, 32]. AOPs offer an attractive approach owing to their high oxidation potential and hydroxyl radicals produced, which helps in the degradation and mineralization of pollutants [11, 21, 47].

Fenton's oxidation process is a well-known AOPs based on the Fenton reaction. The Fenton process is a catalytic cycle of reaction between iron (Fe^{2+}) and hydrogen peroxide (H_2O_2) to produce hydroxyl radicals. Fenton oxidation technology produces hydroxyl radicals ($\bullet OH$), with the reaction generally occurring in an acidic medium between pH 2 and 4 [37, 49] resulting in the precipitate formation and de-colorization of effluent [7]. Fenton technology produces a homogeneous reaction that is ecologically friendly. The efficiency of the Fenton reaction depends mainly on (H_2O_2) concentration, the Fe^{2+}/H_2O_2 ratio [31], pH, and reaction time. In addition, the initial concentration of the pollutants and their character, as well as temperature, has a substantial influence on final efficiency [45, 46]. Fenton's reagent is characterized by its cost-effectiveness, simplicity, and suitability for treating aqueous wastes with variable compositions [3, 9]. The Fenton process involves the application of Fe^{2+} and H_2O_2 for the production of hydroxyl radicals. Ferrous ion is oxidized by H_2O_2 to ferric ion to hydroxyl radical, and a hydroxyl anion [9]. The reaction is shown in the following steps.



Ferric ion is reduced back (typically in the presence of irradiations) to ferrous ion, a peroxide radical, and a proton by the same H_2O_2 [37, 49]. The rate of reaction (1) is around $63 \text{ M}^{-1} \text{ s}^{-1}$, while the rate of reaction (2) is about $0.01\text{--}0.02 \text{ M}^{-1} \text{ s}^{-1}$ (Kang et al. 2002 [36, 48, 51];). This shows that the ferrous ions are consumed faster than they are being generated. The hydroxyl radicals then degrade the organic compounds in reaction (8), and H_2O_2 also reacts with Fe^{3+} via reaction (2) [20]. Fenton chemistry has been studied by many researchers for oxidation of different organic pollutants, including aromatic

and phenolic compounds, pesticides, herbicides, and organic dyes [7, 12, 28, 30, 35, 36, 54, 59].

While the Fenton process has recorded success on a laboratory scale, the process still finds lesser application on an industrial scale largely due to its ineffectiveness in reducing certain refractory pollutants, such as acetic acid, acetone, carbon tetrachloride, methylene chloride, n-paraffins, maleic acid, malonic acid, oxalic acid, and trichloro-ethane, and also due to the high amount of total dissolved solids generated during the process [48, 51]. To manage and improve the quality of sludge generated using the Fenton reagent, the electro-Fenton (EF) process was developed from the principle of ionization, oxidation, and separation of wastewater constituents at the atomic level using electric currents [41]. The development of the EF process solved the problem of imbalance in the Fe/H_2O_2 ratio (which leads to a lower rate of oxidation) and extensive use of oxidants (such as H_2O_2) in the Fenton's process [16].

The EF process reduces the quality of sludge generated by recycling the ferric back to ferrous salt electrochemically. The converted ferrous salt then participates in the oxidation process again, this recycling process can be done in two ways: (1) in situ recycling and (2) ex situ recycling. In in situ recycling, the quantity of the added ferrous catalyst is lower compared to the conventional Fenton process. During this process, the ferric salt formed after the Fenton process is converted back into ferrous salt at the cathode [6, 15]. This reaction process is also called the Fered Fenton process. The reaction taking place at the anode is:



while the reaction taking place at the cathode is:



The main advantage of the Fered Fenton process is the molar ratio of Fe ions and H_2O_2 available at any time in the reactor, which can be easily controlled and maintained at the optimal level; with this, the Fered Fenton process not only reduces the quantity of ferrous salt produced, but it also accelerates and increases the degradation of organic compounds compared to the conventional Fenton process [23, 29, 50, 60].

For the ex situ recycling process, the sludge recycling process takes place in the same reactor as the in situ recycling process. However, the sludge after the neutralization stage is acidified to the required pH and then passed through an electrochemical cell where the ferric is converted back to ferrous salt [48, 51].

However, despite the potential the EF process presents, there is still a need to seriously assess high-cost features such as the need to add and maintain appropriate ionic Fe concentrations, reaction time, the concentration of oxidant, and the requirement to remove the iron species and neutralize the acid of the aqueous effluent after treatment [14, 40].

The objective of this study is to analytically examine the influence and interaction of oxidant (H_2O_2) and reaction time on biological oxygen demand (BOD) and chemical oxygen demand (COD) removal efficiency during the EF process. The study looks into the overall removal efficiency of the EF process (by adding iron from external sources) on the treatment of brewery wastewater. The study also characterizes the interaction

between the concentration of oxidant and reaction time during the EF process in a fully submerged electrolytic cell. The treatment of high-strength brewery wastewater can be further developed from the results this study presents.

Methods

Wastewater sampling

The wastewater used in this study was collected from a brewery factory along Omi-Asoro sawmill road in Ilesha, Southwest Nigeria. A large volume of effluent is discharged daily into a nearby river; the effluent generation is high due to the large volume of malts and beer produced. The collected brewery wastewater is observed to have a brownish color and strong odor which suggests it contains high levels of organic content. The wastewater used in this study was collected in 2-sterilized 25-l containers and stored at about 4–6°C to inhibit biological activity. The wastewater had a BOD and COD of 423.76 mg/l and 854.86 mg/l, respectively; other characteristic properties are shown in Table 1.

Chemicals and materials used

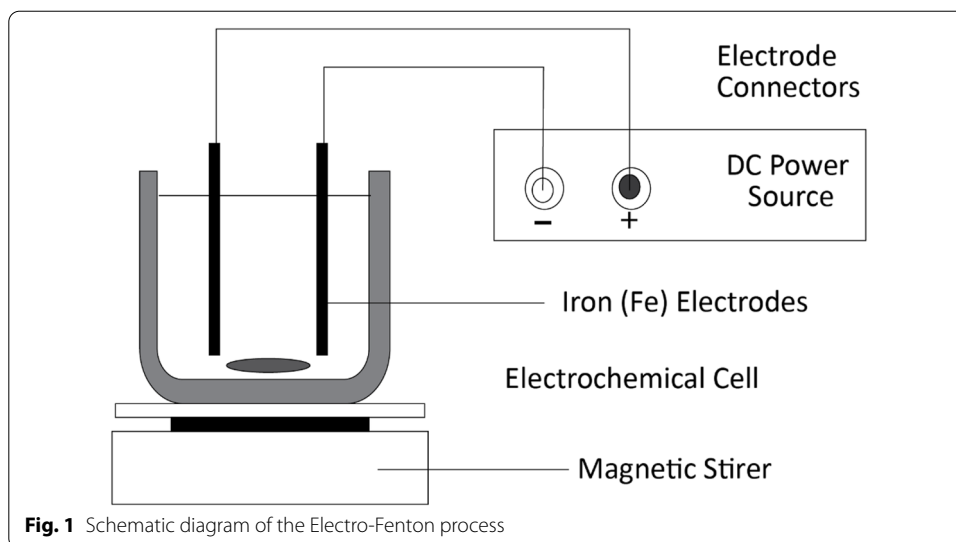
The following chemical reagents purchased from Merck chemicals (Germany) were used: ferrous-sulfate heptahydrate (analytical grade, 99%), hydrogen peroxide (analytical grade, 30%), sodium hydroxide (chemical purity grade, 95%), sulfuric acid (analytical grade, 95–98%), and potassium dichromate (analytical grade, 99.8%). Distilled water was used in cleaning and preparing the reagents during the experiments. Iron electrodes with diameter and length of 7.5 mm and 60 mm, respectively, were bought and a direct current (DC) power source (ISO-TECH-IPS30) was used.

Experimental setup

The experiments were conducted in a batch reactor containing a 1000-ml sample of the BW as shown in Fig. 1. The set-up consisted of a 1500-ml electrochemical cell, 2-iron electrode rods, electrode connectors, magnetic stirrer, and a DC power source. The iron electrodes were cleaned using distilled water and dried overnight at 50°C in an oven [15]. The experiment was carried out in an open reactor at room temperature ($24 \pm 2^\circ\text{C}$). The iron electrodes were spaced 60 mm apart to allow effective oxidation and arranged in

Table 1 Characteristics of brewery wastewater

Parameter(unit)	Value
pH (mg/l)	5.84
Chemical oxygen demand (mg/l)	854.86
Biological oxygen demand (mg/l)	423.76
BOD/COD	0.49
Total dissolved solids (mg/l)	493.0
Total soluble solids	568.0
Turbidity (mg/l)	27.60
Conductivity (mS/cm)	0.850
Copper (mg/l)	0.08
Zinc (mg/l)	0.350
Manganese (mg/l)	0.050



parallel connection to the DC power source at 15 V 2.0 A. 30% analytical grade, hydrogen peroxide was added to the cell containing a prepared sample of the collected BW. Before the EF process, the pH of the cell was adjusted to 3.5 ± 0.5 using 1M H_2SO_4 or 1M $NaOH$ [62] to improve the generation of hydroxyl radical throughout the electrochemical cell [19, 33, 39]. The solution was stirred continuously throughout the process at 300 rpm using a magnetic stirrer. 0.002M of ferrous-sulfate heptahydrate catalyst was added to the cell to initiate the EF reaction.

Analytical tests

For this study, the concentration of hydrogen-peroxide was maintained at 250, 500, 1000, 2000, 3000, and 4000 mg/l and reaction times of 30, 45, 60, 90, and 120 min to investigate optimal removal of biological oxygen demand and chemical oxygen demand using the EF process (all experiments were carried out in triplicates to avoid error). At the end of each EF treatment, BOD and COD tests were carried out using APHA manual standard [4]. The data collected from the BOD and COD tests were then statistically analyzed using Microsoft Excel. A one-way analysis of variance (ANOVA) was used to compare the removal efficiency of the EF process in terms of BOD and COD (at 95% confidence interval). The result showing the relationship between the concentration of hydrogen peroxide and reaction time is as shown in Table 2. A graphical plot showing the removal efficiency of BOD and COD measured before and after the EF treatment process is shown in Figs. 2 and 3. The removal efficiency of the EF process was evaluated using (11).

$$\text{Removal Efficiency} = \frac{C_{raw} - C_i}{C_{raw}} \times 100 \quad (11)$$

where C_{raw} is concentration in the raw sample
 C_i is concentration in the treated sample

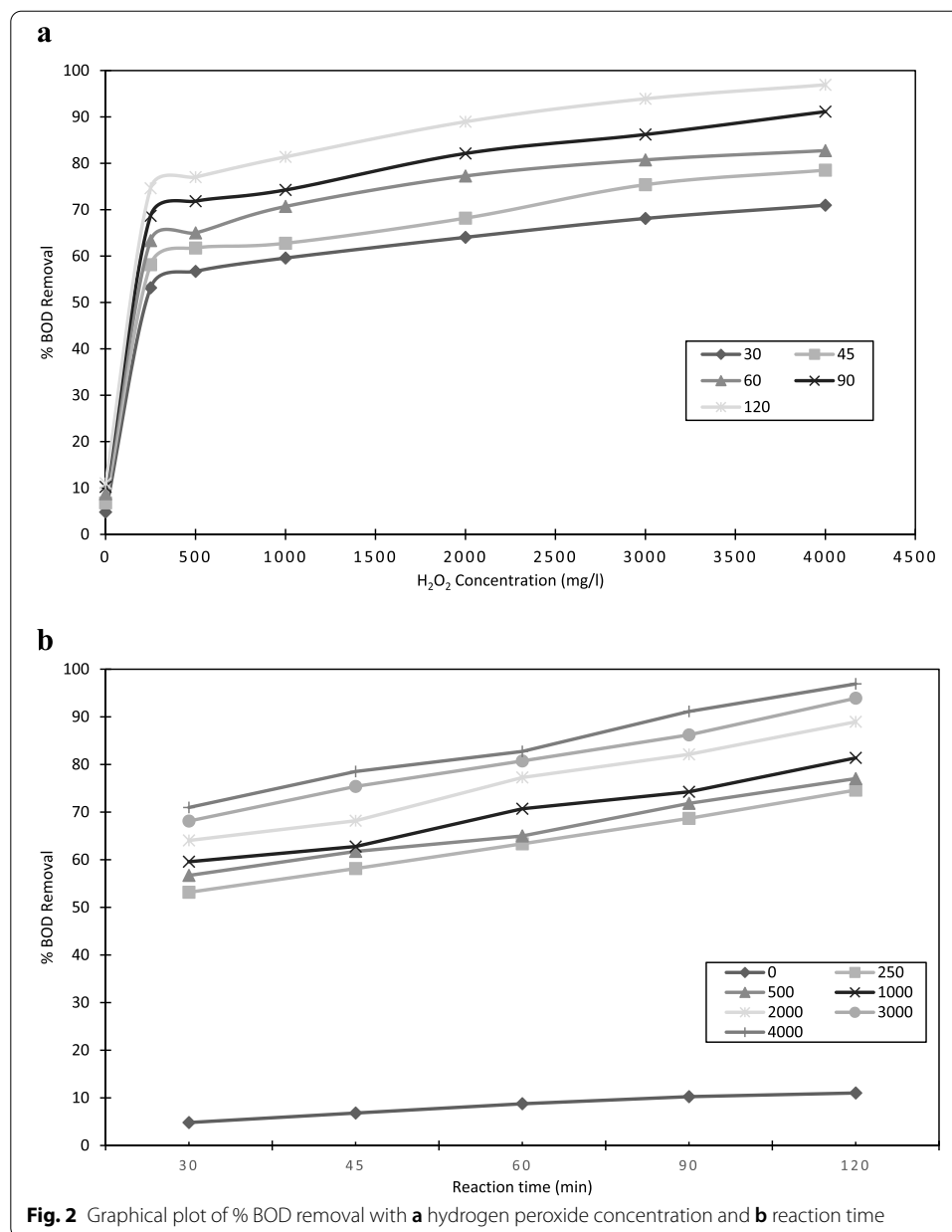
Table 2 Experimental design and obtained results

No	H ₂ O ₂ concentration (mg/l)	Reaction time (min)	BOD (mg/l)	COD (mg/l)	% BOD	% COD
1	0	30	403.34	828.38	4.82	3.10
2	250	30	198.46	275.55	53.17	67.77
3	500	30	183.45	258.33	56.71	69.78
4	1000	30	171.27	227.06	59.58	73.44
5	2000	30	152.35	202.11	64.05	76.36
6	3000	30	135.11	248.61	68.12	70.92
7	4000	30	123.02	268.00	70.97	68.65
8	0	45	394.90	816.65	6.81	4.47
9	250	45	177.34	248.38	58.15	70.95
10	500	45	162.10	236.17	61.75	72.37
11	1000	45	157.77	205.92	62.77	75.91
12	2000	45	134.82	168.35	68.18	80.31
13	3000	45	104.30	212.57	75.39	75.13
14	4000	45	91.02	251.59	78.52	70.57
15	0	60	386.62	805.42	8.76	5.78
16	250	60	155.33	224.49	63.35	73.74
17	500	60	148.34	187.31	64.99	78.09
18	1000	60	124.17	156.22	70.70	81.73
19	2000	60	96.33	142.87	77.27	83.29
20	3000	60	81.63	178.87	80.74	79.08
21	4000	60	73.18	242.79	82.73	71.60
22	0	90	380.32	792.78	10.25	7.26
23	250	90	132.72	183.50	68.68	78.54
24	500	90	119.35	158.82	71.84	81.42
25	1000	90	109.09	115.15	74.26	86.53
26	2000	90	75.76	91.46	82.12	89.30
27	3000	90	58.44	164.46	86.21	80.76
28	4000	90	37.56	228.65	91.14	73.25
29	0	120	377.04	771.08	11.03	9.80
30	250	120	107.55	145.50	74.62	82.98
31	500	120	97.35	109.21	77.03	87.22
32	1000	120	78.90	71.12	81.38	91.68
33	2000	120	46.79	58.51	88.96	93.16
34	3000	120	25.79	145.51	93.91	82.98
35	4000	120	13.04	204.98	96.92	76.02

Results and discussion

BOD and COD removal efficiency

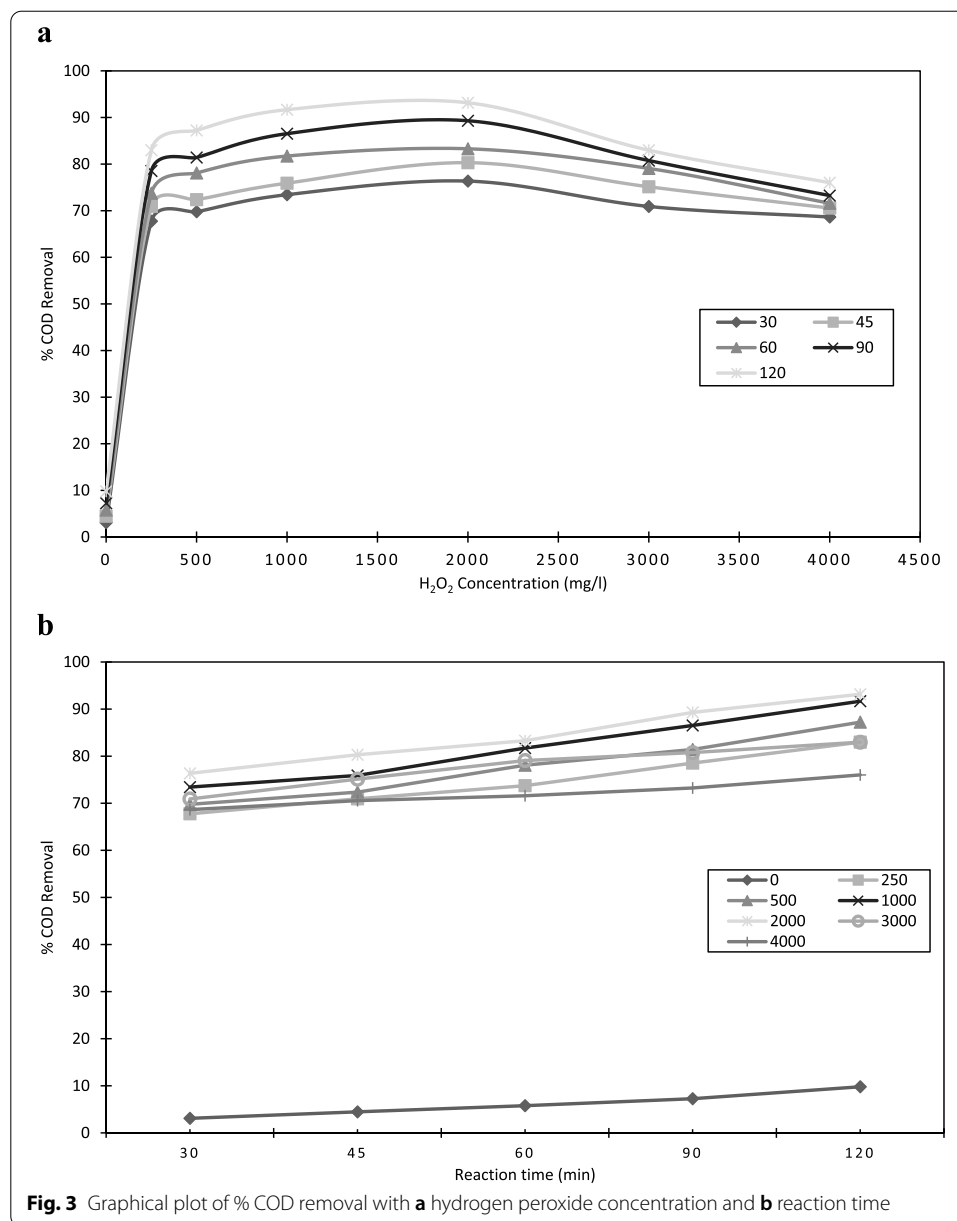
The BOD and COD removal efficiency was investigated at 0, 250, 500, 1000, 2000, 3000, and 4000 mg/l of H₂O₂ while maintaining constant pH, current, and ferrous-sulfate concentrations at 3, 2.5 A, and 0.002M respectively. The results are shown in Table 2. In the absence of H₂O₂, BOD removal efficiency of 4.82%, 6.81%, 8.76%, 10.25%, and 11.025% was observed at 30, 45, 60, 90, and 120 min reaction time, respectively. The low removal efficiency is due to the insufficient generation of hydroxyl radicals at the anodic end of the EF system. Gümüş and Akbal [22] compared the Fenton and EF processes for the oxidation of phenol, and they observed 18.96% removal of phenol at 5 min reaction time in the absence of H₂O₂ in the



electrochemical cell. They also suggested that low removal efficiency in the system is due to insufficient generation of H_2O_2 (oxidizing agent) for the degradation of organics in the BW as shown in Eqn. (12). With iron used as anode in the EF process, Fe^{2+} is dissolved from the iron anode, while hydroxide ions and H_2 gas are generated at the cathode end of the reactor. Rahmani et al. [47] noted that an increase in the presence of hydrogen peroxide in the EF process will lead to improving the generation of Fe^{3+} in the system.



The COD removal was also investigated similarly to BOD, and removal of 4.82 %, 6.81 %, 8.76 %, 10.25 %, and 11.03 was observed at 30, 45, 60, 90, and 120 min reaction time,



respectively. The COD removal without the addition of hydrogen peroxide was also low due to the insufficient generation of hydroxyl radicals. Zhang et al. [61] studying the removal of COD from landfill leachate also reported that the EF process alone could only remove 10% of COD from the leachate without the addition of hydrogen peroxide.

Effects of hydrogen peroxide

The concentration of H_2O_2 is crucial to deciding the overall efficiency of BW degradation using the EF process. The removal of BOD and COD in the EF process was evaluated in three replicate samples before and after application of EF treatment. The EF process combines the Fenton and electrocoagulation methods to improve the degradation of

organic compounds in the BW as presented in terms of BOD and COD removal. Maximum BOD and COD removal was observed up to 96.9% and 93.16% in the replicate treatments without any significant change ($p > 0.05$). The results of the mean composition of the raw and treated BW samples are presented in Table 2. The raw BW contained 423.76 mg/l and 854.86 mg/l of BOD and COD, respectively.

Analysis of variance ANOVA showed that both BOD and COD after the EF process were much lower compared to that of the raw BW concentration values ($p < 0.05$). Removal efficiencies calculated using Eq. (11) showed considerable removal efficiencies for BOD (53–96%) and COD (67–93%) at varied oxidant doses (i.e. 250, 500, 1000, 2000, 3000, and 4000 mg/l). Comparing results to other previous studies showed that the average BOD and COD reduction was 70.8% and 75%, respectively, in the treatment of olive mill wastewater using high power ultrasound and EF method [38]. The removal efficiencies in the present study are also substantiated by previous studies on removal of fatty acids from palm-oil effluent by combined EF and biological oxidation process, and the EF process showed removal of 86% COD and 85% BOD [7]. Furthermore, it was reported that the EF process presented reasonable removal efficiencies of 72% for COD [5] for landfill leachate treatment. During the analysis, COD concentrations after the EF process were lowest at 250 mg/l (67.77 %) H_2O_2 concentration, and concentration of 2000mg/l (96%) showed the highest COD removal.

Figures 2b and 3b show that increased oxidant dose from 0 to 2000 mg/l was favorable to BOD and COD removal in the BW following the EF process. However, in Fig. 3b, further addition of H_2O_2 (> 2000 mg/l) showed reduced COD removal till 4000 mg/l. This reduction was described as related to the insufficient ferrous-sulfate catalyst and a scavenging effect [24]. An increase in the H_2O_2 dose will usually improve the overall efficiency in the process [24] due to the increase of OH^\bullet , but large quantities of H_2O_2 unused reduce removal efficiency by scavenging generated OH^\bullet or recombining OH^\bullet . In a related study, it was noted that unused portion of H_2O_2 with its reductive ability consume chemical oxidant during COD analysis thus leading to an overestimation of COD values. Hence, the degradation rate of organic compounds in BW increases with increasing concentration of hydrogen peroxide; however, excess amount of hydrogen peroxide should be avoided, as excess addition contributes to COD. Matavos-Aramyan [34] noted that the presence of hydrogen peroxide in water released to the environment is harmful to many organisms and will affect the overall degradation efficiency of the treatment process.

Effects of reaction time

The effect of reaction time on BOD and COD removal was investigated at 0, 250, 500, 1000, 2000, 3000, and 4000 mg/l H_2O_2 to determine the duration needed for optimum operation of the EF process. The reaction time was observed at 30, 45, 60, 90, and 120 min. The results from Figs. 2a and 3a showed that organic materials were rapidly degraded in the first 30 min but maintained a gradual reduction as the reaction time increased during the EF process. At 30 min reaction time, BOD_5 was reduced from 423.76 mg/l to 198.46, 183.45, 171.27, 152.35, 135.11, and 123.02 mg/l at 250, 500, 1000, 2000, 3000, and 4000 mg/l H_2O_2 concentration, respectively. At 120 min reaction time, BOD was reduced from 423.76 to 46.8 mg/l at 2000 mg/l H_2O_2 . The reduction in

the BOD concentration after EF treatment indicated that an increase in reaction time improved BOD removal in the BW. Figure 2b shows the relationship between reaction time and percentage removal of BOD in the BW. Similar studies on the bio-electro-Fenton process [13] have shown that an increase in reaction time with H_2O_2 concentration improved biodegradability in enhanced medicinal herbs wastewater.

The COD removal efficiency from Fig. 3b shows that as the reaction time increased COD removal in the EF process improved. During the first 30 min of treatment, the wastewater was rapidly degraded. Umar et al. [55] described a similar initial rapid degradation in landfill leachate as largely due to the easily degradable organics. After the first 30 min, the degradation then gradually slowed down, following a steady trend through the EF treatment. Umar et al. [55] also described the reaction time to be mainly dependent on H_2O_2 dose, and Zhang et al. [61] also showed that an increase in reaction time improved on COD removal efficiency in removal of landfill leachate using the EF process.

Conclusions

In this study, the electro-Fenton (EF) process was applied to the treatment of brewery wastewater. The effects of some of its operational parameters were examined to determine the efficiency of the EF process. The study observed effects the hydrogen peroxide and reaction time was observed on BOD and COD removal. The study was performed at 30 to 120 min reaction time and 0 to 4000 mg/l hydrogen peroxide. The results indicated that 93.15% COD removal was achieved at optimal conditions of pH 3, catalyst dosage 0.002M of ferrous sulfate, a voltage of 20 V, and electrode spacing of 6cm. The maximum COD removal was observed at 2000 mg/l hydrogen peroxide and 120 min reaction time from 854.86 to 93.16 mg/l. Maximum BOD removal was also observed at 4000 mg/l at 120 min reaction time. However, due to harm posed to water organisms and the scavenging effect caused by excess hydrogen peroxide, an optimum level of hydrogen peroxide at 2000 mg/l was accepted. At the 2000 mg/l hydrogen peroxide (88.95%), removal was observed in the EF treatment system. Results from the study showed that the EF process is promising in the removal of BOD and COD from BW, and this also suggests the EF process may be suitable for the treatment of high-strength industrial wastewater. The application of this process is also suggested to the treatment of high-strength BW.

Abbreviations

AOPs: Advanced oxidation process; BW: Brewery wastewater; EF: Electro-Fenton; BOD: Biological oxygen demand; COD: Chemical oxygen demand; DC: Direct current; ANOVA: Analysis of variance.

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Authors' contributions

OA wrote up the manuscript and conducted the laboratory experiments. KA and DO supervised the laboratory experiment and structured, edited, read, and approved the final manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets supporting the conclusions of this article are included within the article.

Declarations**Competing interests**

The authors declare that they have no competing interests.

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