



## Algal Removal from Water by Electrochemical Generation of Hydrogen Peroxide and Persulfate

Intesar K. Abdulhassan<sup>1</sup>, Adel F. Hamed<sup>2</sup>, Hesham M. Abd El Fatah<sup>2</sup>, Gamal O. El-Sayed<sup>3\*</sup>

<sup>1</sup>Sciences Department, College of Basic Education, University of Diyala, Iraq

<sup>2</sup>Botany Department, Faculty of Science, Ain Shams University, Cairo, 11566, Egypt

<sup>3</sup>Chemistry Department, Faculty of Science, Benha University, Benha, Egypt

\*Corresponding Author: gamaloelsayed@yahoo.com

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### ABSTRACT

Removal of algae by electrochemical treatments through electrochemical generation of persulfate and hydrogen peroxide was investigated in a fixed bed reactor. The cell was equipped with two electrodes (two identical graphite anode and cathode). The effect of different operative parameters, such as applied potential and amount of initial sodium sulfate added in the case of persulfate production was evaluated. In addition, the influences of potential differences in the case of hydrogen peroxide production were addressed. The two treatments were optimized, and their high efficacy in removing algae-laden water was proved upon application to collected samples obtained from the reservoir of Rod El-Farag drinking water station. The pro-type system used with the proposed methods is simple, fast, and effective to remove algae from water sources.

### INTRODUCTION

Drinking water quality varies greatly from place to another, depending on the source of water and the conditions. Thus, it's important to ensure the safety of drinking water through various certified methods of analysis in order to choose the appropriate methods of treatment. Sources of municipal water may be contaminated by natural pollutants including chemical and biological. These methods are limited by several factors such as efficiency, formation of secondary pollutants, costs and treatment time (Robertson *et al.*, 2012). Therefore, recent studies have focused on the instant need for new technical applicable methods to solve the problem of overgrowing and propagation of algae inhabiting the surface waters.

Algae in water are of great concern since they adversely affect drinking water quality and water treatment processes. In particular, in tropical and semi-tropical zones, algae can grow excessively under high nutrient contents in surface water due to contamination by agricultural activity, domestic wastewater discharge and industrial effluents. The high concentrations of nitrogen and phosphorous can provide the ideal medium for the excessive growth of algae, which is detrimental not only from an environmental point of view but also for human health (Sathe *et al.*, 2016).

Algae may cause problems such as poor taste and odor in the water, and several studies in the literature have addressed the possible problems connected with toxins released by algae. The presence of sub-lethal doses of cyanotoxins in drinking water is implicated as one of the key risk factors for the high occurrence of primary liver cancer.

Conventional treatment methods of fresh water include physical, chemical and biological techniques as coagulation, air flotation, flocculation, sedimentation, chemical oxidation, electrochemical techniques, photolysis using ultraviolet (UV) radiation and chlorine disinfection, which can remove most of the algal cells (**Mascia *et al.*, 2013; Wang *et al.*, 2018**). It is important to remove the algal cells by filtration prior to the addition of any oxidant such as chlorine.

Oxidation disinfection of biological contaminants by chemical agents has been studied using halogens, ozone, hydrogen peroxide, hypohalous salts and silver cations. Physical processes comprise thermal treatment, ultrasonic and electromagnetic radiation as ultraviolet (UV) light and X-ray in addition to filtration with suitable filters (**Yu *et al.*, 2022**). Recent researches have been published about algal removal or deactivation by several techniques including electrocoagulation (**Azarian *et al.*, 2007**), electrochemical oxidation (**Mascia *et al.*, 2013**), coagulation (**Dai *et al.*, 2020; Ren *et al.*, 2022**) and photocatalysis (**He *et al.*, 2021; Mohan *et al.*, 2022**).

Electrochemical disinfection of water is a new technique which has gained increasing interest because of the growing need to disinfect the drinking water, wastewater and pool water (**Bergmann, 2021**). Electrochemical disinfection is mainly based on the oxidation power of disinfectants produced on the electrode surface or the bulk of an electrolyte (**Bruguera-Casamada *et al.*, 2016; Ghernaout, 2017**). In case of chlorine-based electrochemical drinking water disinfection, new activities in the field were observed to replace pressurized chlorine gas with chlorine containing solutions produced electrochemically (**Martínez-Huitle & Brillas, 2008**), often working at low chloride concentration. Chlorine dioxide ( $\text{ClO}_2$ ) can be electrochemically produced onsite using undivided electrochemical or divided cells by anodic chlorite oxidation or cathodic chlorate reduction (**Sivey *et al.*, 2010**). The production of ozone on  $\text{PbO}_2$ , platinum,  $\text{SnO}_2$  and other anodes has been studied (**Park *et al.*, 2019**). Another work on the production of  $\text{O}_3$  gas on BDD anodes in divided cells was found to be more efficient (**Lara-Ramos *et al.*, 2020**).

The synthesis of  $\text{H}_2\text{O}_2$  by the electrochemical reduction of  $\text{O}_2$  is also a promising technique. Hydrogen peroxide is one of the most environmentally friendly as it decomposes into oxygen and water and does not release any harmful compounds after reaction (**Drougi *et al.*, 2001**). In this process, a noble metal, metal alloy or carbon-based material is used as a catalytic material either individually or in combination (**Chen *et al.*, 2018**). The use of carbon-based materials has been accelerated because of their high performance, appropriate strength and economic benefit. Many research attempts for improving yield of  $\text{H}_2\text{O}_2$  are focused on higher productive and cheaper electrocatalysts (**Jung *et al.*, 2020; Zhang *et al.*, 2020**). Among the catalysts recently considered, noble metals, alloys and metal oxides could be used. When this process occurs on graphite cathodes, it is performed through the reduction of molecular oxygen (**Perry *et al.*, 2020**).

The reduction of oxygen is described by the following equations depending on the applied potential:

• 4-electron reaction:



• 2-electron reaction:



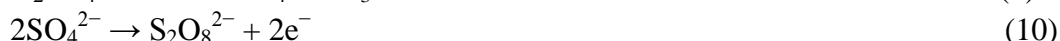
The overall process involves the formation of various radical species such as superoxide  $\text{O}_2^{\cdot-}$  and hydroperoxide  $\text{HOO}^{\cdot}$  (Collin, 2019). Hence, controlled electrochemical oxidation of water may control the production of hydroperoxyl radical, hydrogen peroxide and hydroxyl radical (Anantharaj *et al.*, 2021). Depending on the applied potential, the reaction products may vary according to the equations (3-6):



The order of oxidizing capability of the reactive oxygen species are arranged as hydroxyl radical > hydrogen peroxide > hydroperoxyl radical > dioxygen (Wang *et al.*, 2021).

The theoretical required potential for the chemical reaction shown in Eq. (5) is 1:23 V. Actually, about 1.5–1.7 V is required for the reaction because of various voltage losses. About 1.7–2:0 V is required in the onsite hydrogen peroxide production system, which is considered due to oxygen overpotential (Song, 2020; Alayande & Hong, 2022).

The electrochemical production of persulfate is performed in sulfate-containing solutions from the oxidation of  $\text{SO}_4^{2-}$  and  $\text{HSO}_4^-$  ions through the reactions described by the equations (7) and (8), respectively (Fernandes *et al.*, 2021).



Electrochemically produced sulfate radical (Equations (7) and (8)), which presents higher oxidizing potential (E 2.6 V/SHE) than that of a hydroxyl radical (E 2.01 V/SHE), promotes a non-selective oxidation resulting in an efficient removal of a wide range of organic compounds (Shafiee *et al.*, 2018). Furthermore, the half-life time of  $\text{SO}_4^{\cdot-}$  is usually longer than that of  $\cdot\text{OH}$ , enabling better mass transfer and contact with the organic substrate in solution.

The aim of the present work was to examine the ability of electrochemically generated persulfate and hydrogen peroxide to remove different types of algae. The target of this study was the drinking water source supplying fresh water to Rod El-Farag drinking water station in Great Cairo area. The experiments were performed at the laboratory scale.

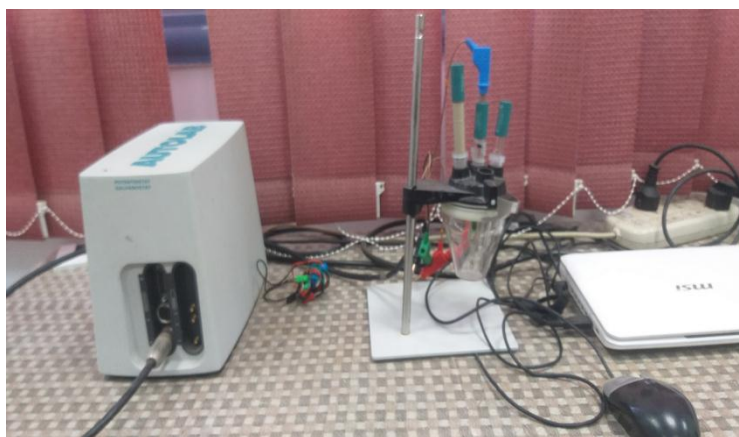
## MATERIALS AND METHODS

### Chemicals

All reagents and chemicals used in the experiment were of analytical grade purity and were used without further purification. Chemicals used in this study were: sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), sodium persulfate ( $\text{Na}_2\text{S}_2\text{O}_8$ ), sodium thiosulfate  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  and iodine ( $\text{I}_2$ ),

### Apparatus

A 100mL glass cylinder cell was used as the electrolysis cell with two graphite electrodes ( $2 \times 0.8 \times 0.4$  cm). Both the anode and cathode had a working surface area of about  $6\text{cm}^2$ . 100mL of working solution (50mL of raw water diluted with 50mL of distilled water) was taken for the experimental work. The working electrodes were connected together through external wires, while the potential was adjusted to the desired value. All experiments were conducted at room temperature ( $25^\circ\text{C} \pm 2^\circ\text{C}$ ). The apparatus used in this research is shown in Fig. (1).



**Fig. 1.** Electrolysis apparatus with the working cell

### Sampling of algae

Algal samples were seasonally collected during the years 2021-2022 from the drinking water station (Rod El-Farag station, Cairo Governorate, Egypt) by using phytoplankton net of 15 micrometer mesh. In a cleaned plastic bottle, each sample collected was concentrated several times to reach a volume of 100ml. The samples were subjected and transferred to the laboratory in an ice box for the identification and counting of the total algal biomass (**Round *et al.*, 1990; Li *et al.*, 2021**).

### Physicochemical characters of water samples

The essential parameters of raw water taken from the station were measured in two seasons (spring and winter) to determine the water quality of the analyzed samples according to the methods provided by **APHA (2005)**. Some parameters (pH, turbidity, dissolved oxygen) were measured in the field, while other parameters were stored in clean plastic bottles in the refrigerator and analyzed at the Lab a few hours after arriving from the field.

## Electrolysis method

1- In case of persulfate, 50ml of the required concentration of Na<sub>2</sub>SO<sub>4</sub> solution was placed with 50ml of pure water or raw water sample and placed in the electrolysis cell. The final concentration of sodium sulfate was 2.5, 5.0, 7.5 and 10% (by weight), then the electrode was lowered into the cylinder, and the electrical voltage was adjusted at 2.0 or 2.5 volts. The time of electrolysis was adjusted at a fixed period of an hour. An excess of solid KI was added to react with the liberated persulfate, giving an equal amount of I<sub>2</sub>. The liberated iodine was titrated with a standard solution of sodium persulfate to calculate the amount of persulfate formed by electrolysis.

2- In case of hydrogen peroxide, potassium chloride (KCl) was used, where 0.025g of the substance was weighed and dissolved in 100ml of distilled water and placed in the device cylinder, and the voltage was set to 1.2 volt for half an hour. The pH of the solution was 5.6. The sample was left after analysis for 24 hours. The next day, 20ml of the analyzed sample was taken, and 1ml of H<sub>2</sub>SO<sub>4</sub> was added to the sample. The sample was calibrated with a standard solution of potassium permanganate KMnO<sub>4</sub> (0.952x10<sup>-3</sup> M).

## RESULTS AND DISCUSSION

### 1. Water treatment processes

#### 1.1. Electrochemical production of persulfate

Sodium persulfate has been prepared by direct electrolysis of an aqueous solution of sodium sulfate as a starting solution (**Bashir et al., 2017; Chaplin, 2019**). The cell consists of a platinum anode and a graphite cathode. The persulfate ions were generated at the anode according to Eq. (10). To evaluate the amount of persulfate formed in the electrolysis solution, a starch-titration process was performed depending on the oxidizing properties of persulfate, which turn iodide ions to iodine as shown in the equation:

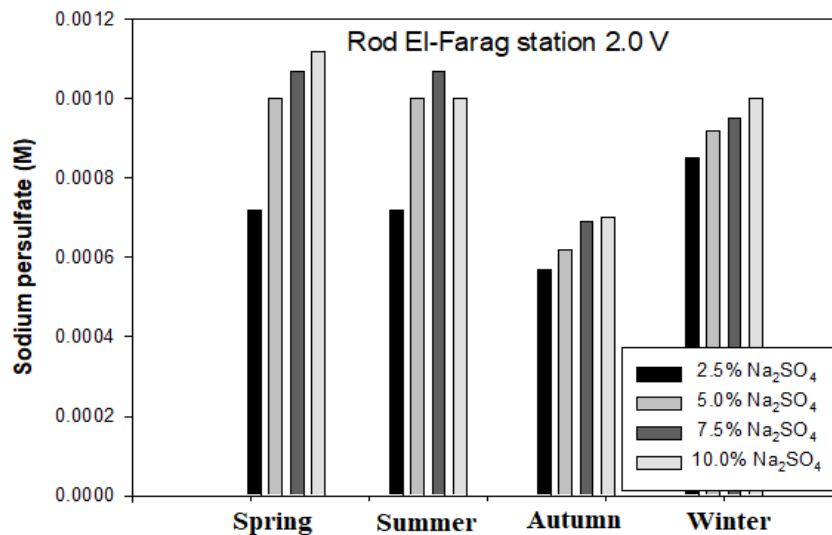


At the end of the titration, the liberated iodine was titrated with a standard solution of sodium thiosulfate, eq. (12):

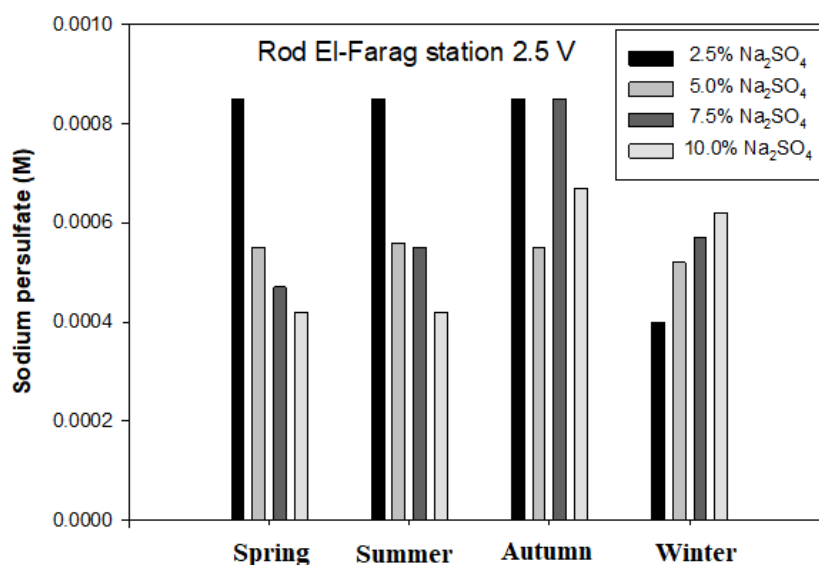


The oxidation reaction has a large negative potential (-2.05 volts) and theoretically cannot compete with the oxidation of water (-1.23 volt). However, higher potentials (2.0 and 2.5 V) were applied. The relation between electrochemical generated sodium persulfate concentrations (M) and initial concentrations of sodium thiosulfate (%) for Rod El-Farag station in different seasons, applying potential differences 2.0 and 2.5V, are shown in Figs. (2, 3), respectively. By comparing the results in Figs. (2, 3), it could be noticed that, the amount of persulfate generated in the solution increases with the increase of the initial sodium sulfate added in case of using 2.0 as applied potential during all seasons. Possible explanation might be that the formed thiosulfate radicals have more reactive surface area, which may adhere to algal cell surface and promote the cell coagulation (**Wei et al., 2023**). **Yang et al. (2022)** reported that, the addition of small amount of peroxymonosulfate increase the efficiency of algal removing by 18%, and with their simultaneous addition, the removal of algae increased to about 97%.

Additionally, some natural organic matter in water can activate thiosulfate to produce  $\text{SO}_4^{\cdot-}$  (Srithep & Phattarapattamawong, 2017). Rapid reaction of  $\text{SO}_4^{\cdot-}$  with  $\text{H}_2\text{O}$  and its low rate of production, adding to its rapid reaction at neutral pH lead to a weak  $\text{HO}^{\cdot}$  signal, and this may play an important role in algal removal (Yang *et al.*, 2022). In case of using a potential difference of 2.0 volts which is higher than that in the case of 2.5 volts, the volume increases with the increase in sodium sulfate content from 2.5% to 10.0%. This result indicates that the suitable potential for maximum production of persulfate is 2.0 volts.



**Fig. 2.** Relation between electrochemical generated sodium persulfate and initial content of sodium sulfate in different seasons for Rod El-Farag station (2.0 V)



**Fig. 3.** Relation between electrochemical generated sodium persulfate and initial content of sodium sulfate during different seasons for Rod El-Farag station (2.5 V)

## ***1.2. Electrochemical production of hydrogen peroxide***

Electrochemical hydrogen peroxide synthesis using two-electron oxygen electrochemistry is an intriguing alternative to currently dominating environmentally unfriendly and potentially hazardous anthraquinone process and noble metals catalyzed direct synthesis. Electrocatalytic two-electron oxygen reduction reaction (ORR) and water oxidation reaction (WOR) are the source of electrochemical hydrogen peroxide generation. Various electrocatalysts have been used for the same target and were characterized using several electroanalytical, chemical, spectroscopic and chromatographic tools. Despite the existence of few reviews summarizing the recent developments in this field, none of them has unified the approaches in catalysts' design, criticized the ambiguities and flaws in the methods of evaluation or emphasized the role of electrolyte engineering. Hence, we dedicated this review to discuss the recent trends in the catalysts' design, performance optimization, evaluation perspectives and their appropriateness and opportunities with electrolyte engineering. In addition, particularized discussions on fundamental oxygen electrochemistry, additional methods for precise screening, and the role of solution chemistry of synthesized hydrogen peroxide were presented. Thus, this review disclosed the state-of-the-art in an unrepresented view, highlighting the challenges, opportunities and alternative perspectives.

## **2. Treatment of raw water by persulfate and hydrogen peroxide**

### ***2.1. Removal of chemical pollution***

The main physicochemical parameters of the collected water samples were determined before and after treatment. The results in Table (1) show that some parameters are nearly the same for the two seasons, whereas some others such as turbidity, bicarbonate content, COD, oil and grease are quite different from season to another.

### ***2.2. Removal of algae and chlorophyll from raw water***

Concerning the algal population from the collected samples, it was found that, the highest algal count was observed in autumn and decreased by increasing the temperature to reach its lowest populations during summer (Table 2). In this context, **Shen *et al.* (2011)** elucidated that, the total algal biomass reaches the highest value with the decrease in water temperature, and added that, the concentration of algae cells decreases gradually with the increase in water temperature. The same results are those recorded in the study of **Mohamed *et al.* (2020)** conducted on the River Nile water.

After counting the algal content and the total amount of chlorophyll, the water samples were treated by electrochemically generated persulfate and hydrogen peroxide. The initial concentrations of Na<sub>2</sub>SO<sub>4</sub> were 2.5, 5.0, 7.5 and 10%, and the experiments were performed at 2.0 and 2.5 volts for the samples taken during the four seasons.

**Table 1.** Essential parameters for raw water sampled in spring and winter from Rod El-Farag station

<b>Parameter</b>	<b>Season</b>	<b>Raw water</b>	<b>After Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (10%) treatment</b>	<b>After H<sub>2</sub>O<sub>2</sub> treatment</b>
<b>pH</b>	Spring	8.27	2.7	6.9
	Winter	7.7	2.7	6.6
<b>Turbidity (NUT)</b>	Spring	10.20	8.4	3.3
	Winter	2.03	10.3	7.4
<b>Electrical conductivity (mS/cm)</b>	Spring	500	84600	819
	Winter	495	84600	819
<b>HCO<sub>3</sub><sup>-</sup> (mg/L)</b>	Spring	95.16	Nil	75.4
	Winter	247.7	Nil	93.9
<b>SO<sub>4</sub><sup>2-</sup> (mg/L)</b>	Spring	64.0	39000	90.0
	Winter	65.0	39000	90.0
<b>Cl<sup>-</sup> (mg/L)</b>	Spring	28.5	4200. 0	134. 8
	Winter	29.7	4200.0	135. 8
<b>DO (mg/L)</b>	Spring	9.60	0.43	8.15
	Winter	9.62	0.43	8.15
<b>BOD<sub>5</sub> (mg/L)</b>	Spring	Nil	Nil	Nil
	Winter	1.8	2.4	5.2
<b>COD (mg/L)</b>	Spring	354	90	20
	Winter	11	51	20
<b>Oil &amp; grease (mg/L)</b>	Spring	81	83	Nil
	Winter	6	8	Nil
<b>TDS (mg/L)</b>	Spring	320	65265	461.7
	Winter	341	65268	461.7



**Table 2-a.** Algae and chlorophyll removal from Rod El-Farag station by persulfate (Spring)

Na <sub>2</sub> SO <sub>4</sub>	Algae count (individual/L)			Chlorophyll (mg/L)		
	Before	After	Removal%	Before	After	Removal%
	2.0 V					
2.5%	18480	7.2	99.96	9.07	2.03	77.61
5.0%		5.6	99.97		1.92	78.83
7.5%		6.2	99.96		2.73	69.90
10.0%		3.8	99.98		2.46	72.87
	2.5 V					
2.5%	18480	10.0	99.94	9.07	2.81	69.01
5.0%		8.6	99.95		2.24	75.30
7.5%		9.2	99.95		2.75	69.68
10.0%		11.4	99.93		2.92	69.80

**Table 2-b.** Algae and chlorophyll removal from Rod El-Farag station by persulfate (Summer)

Na <sub>2</sub> SO <sub>4</sub>	Algae count (individual/L)			Chlorophyll (mg/L)		
	Before	After	Removal%	Before	After	Removal%
	2.0 V					
2.5%	3960	9.6	99.75	9.07	1.78	80.37
5.0%		18.6	99.53		1.93	78.72
7.5%		11.6	99.70		2.20	75.74
10.0%		7.4	99.81		4.81	46.96
	2.5 V					
2.5%	3960	14.8	99.62	9.07	4.53	50.05
5.0%		16.4	99.58		5.11	54.80
7.5%		12.0	99.69		5.73	56.8
10.0%		10.8	99.72		2.24	65.00

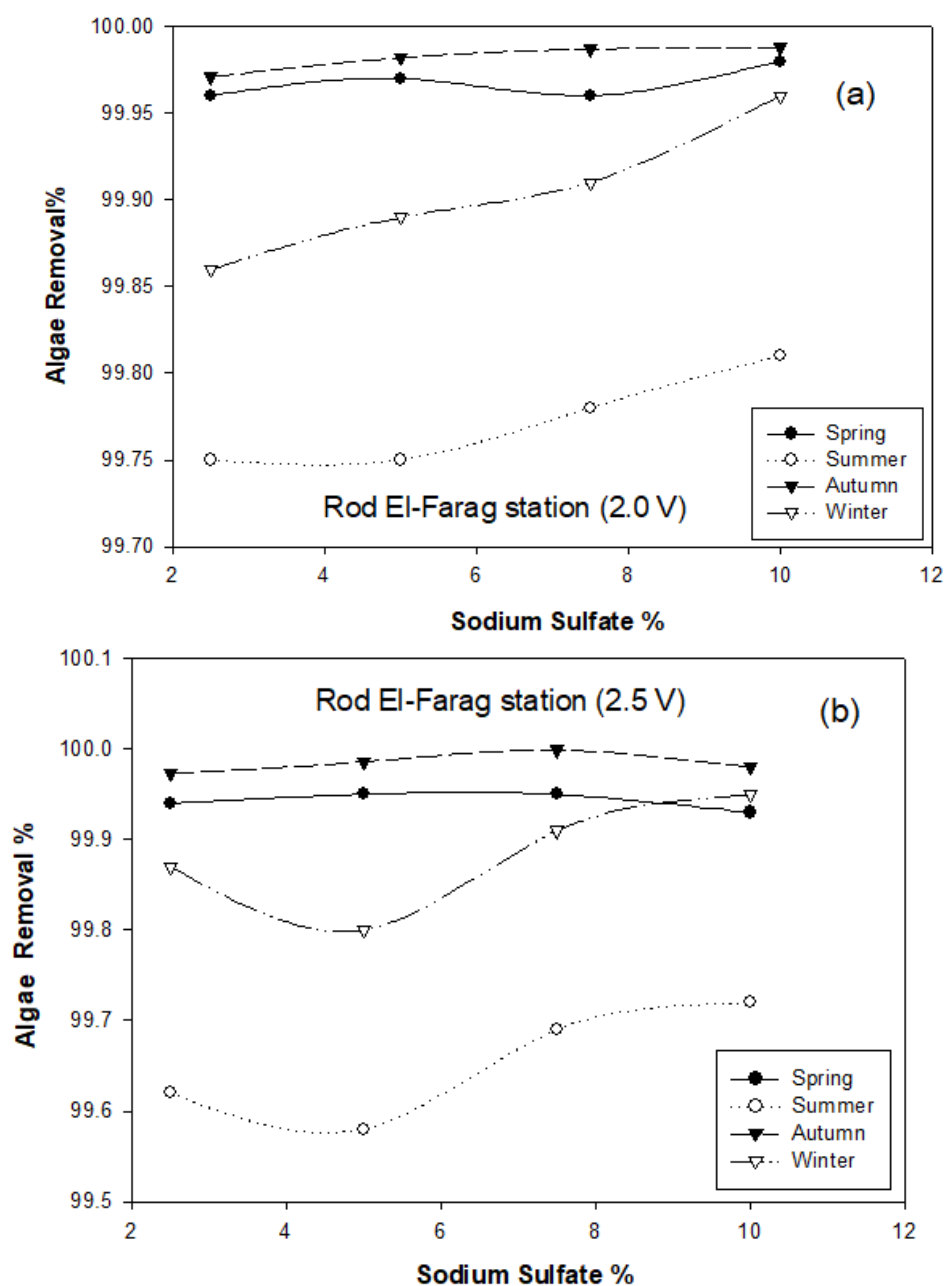
**Table 2-c.** Algae and chlorophyll removal from Rod El-Farag station by persulfate (Autumn)

Na <sub>2</sub> SO <sub>4</sub>	Algae count (individual/L)			Chlorophyll (mg/L)		
	Before	After	Removal%	Before	After	Removal%
2.0 V						
2.5%	69800	19.8	99.971	13.8	3.39	75.43
5.0%		12.2	99.982		3.32	75.94
7.5%		9.0	99.987		3.94	71.44
10.0%		8.0	99.988		6.14	55.50
2.5 V						
2.5%	69800	18.6	99.973	13.8	3.39	75.43
5.0%		9.2	99.986		3.30	76.08
7.5%		17.8	99.999		4.82	65.21
10.0%		13.6	99.980		8.92	35.36

**Table 2-d.** Algae and chlorophyll removal from Rod El-Farag station by persulfate (Winter)

Na <sub>2</sub> SO <sub>4</sub>	Algae count (individual/L)			Chlorophyll (mg/L)		
	Before	After	Removal%	Before	After	Removal%
2.0 V						
2.5%	10720	14.2	99.86	20.75	4.16	79.95
5.0%		11.6	99.89		1.70	91.80
7.5%		9.6	99.91		0.60	97.11
10.0%		4.4	99.96		0.32	98.45
2.5 V						
2.5%	10720	13.0	99.87	20.75	3.96	80.91
5.0%		21.6	99.80		2.51	95.18
7.5%		9.8	99.91		0.81	96.09
10.0%		5.0	99.95		0.31	98.50

The data related to algal removal depending on the initial sodium sulfate concentration are displayed in Fig. (4a, b) for the four seasons using 2.0 and 2.5 volts, as applied potential, respectively.



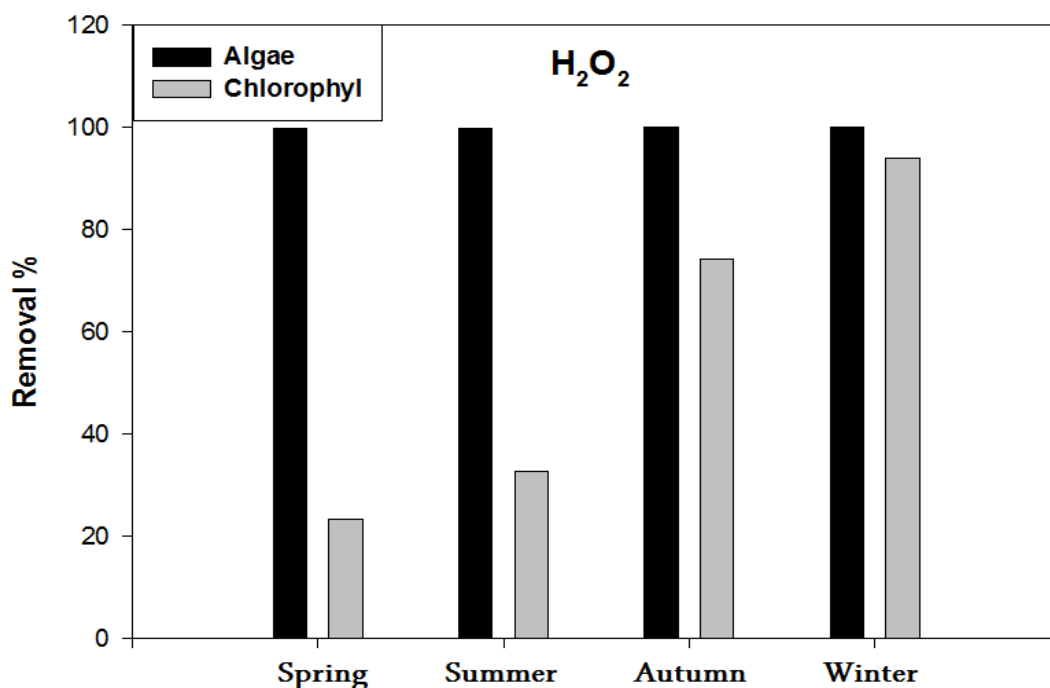
**Fig. 4.** Algal removal efficiency by electrochemical generated sodium persulfate using (a) 2.0 and (b) 2.5 applied volts

As shown in Fig. (4), the removal efficiency was high at the two potentials and reached its maximum value in autumn and spring.

Table (2) shows that, after treatment with sodium sulfate, the pH value turned to acidic, and this acidic pH favors the formation of radicals, resulting in higher radical oxidation potentials and lower recombination rates. The higher density of those radicals helps in algal degradation process (Shokoohi *et al.*, 2023).

When  $H_2O_2$  was applied to water samples, the removal efficiency was evaluated for algae and chlorophyll. In case of algae, the removal reached almost about 100% in all seasons, while in case of chlorophyll removal, the efficiency changed from season to season.

Minimum removal was observed in spring (about 23%), while maximum removal was detected in winter (about 92%). These results are shown in Fig. (5). Removal efficiency of  $H_2O_2$  may be due to the production of hydroxyl radicals ( $\bullet OH$ ), which can destroy algal cells, affecting the physiological and biochemical activities of cells, chlorophyll and algae proteins (Chen *et al.*, 2020). Moreover,  $\bullet OH$  acts as an electrophilic group, which can attack nucleophilic sites in algal organic material, leading to a reduction in organic by-products of algae. The  $\bullet OH$  can oxidize algal organic matter to increase the content of methylketone structure, which results in decreasing algal contents (Wang *et al.*, 2023). On other hand,  $\bullet OH$  primarily destroys macromolecular organic carbon-carbon double bonds and aromatic rings, resulting in the conversion of large molecules to smaller ones, leading to a higher organic matter removal rate (Yang *et al.*, 2022; Wei *et al.*, 2023). By increasing the concentration of hydrogen peroxide, the removal efficiency increases (Mukherjee *et al.*, 2020; Du *et al.*, 2021).



**Fig. 5.** Removal efficiency of algae and chlorophyll during different seasons using electrochemically generated  $H_2O_2$ .

### 3. Comparison with other disinfection methods

**Table 3.** A comparison between the results of the efficiency of the present study and other published works in terms of removing algae

Reagent	Removal efficiency	Reference
Fe(II)/ peroxymonosulfate	94.3%	(Wang <i>et al.</i> , 2018)
UV/H <sub>2</sub> O <sub>2</sub>	94.7%	(Jia <i>et al.</i> , 2018)
H <sub>2</sub> O <sub>2</sub> /Fe(II)	94.0%	(Zhang <i>et al.</i> , 2020)
photo-bioreactor/air flotation	61%	(Badawi <i>et al.</i> , 2022)
	Algae	Chlorophyll
This study: persulfate	>99.0%	>70.0%
This study: ydrogen peroxide	>99.0%	>80.0%

## CONCLUSION

It could be concluded that, the proposed methods for electrochemical production of water disinfectants are simple and effective for the removal of algae from raw water. The production of sodium persulfate and hydrogen peroxide is easy, fast and cost-effective as the voltage needed for it is very low and could be applied by a solar cell.

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