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SCIENTIFIC PAPER

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ELECTROCHEMICAL HARVESTING OF *CHLORELLA* SP.: ELECTROLYTE CONCENTRATION AND INTERELECTRODE DISTANCE

Article Highlights

- The presence of Cl^- ions have a noticeable effect on the performance of the harvesting process
- Maximum efficiency achieves in a short electrolysis time at 2 g NaCl/l for both electrodes
- It takes a longer time to achieve maximum efficiency at 5 g/l NaCl
- The lower energy consumption is achieved with Al, which decreases with increasing NaCl concentration
- Reducing the electrode gap decreases the time required to reach the maximum efficiency

Abstract

Two modes of electrochemical harvesting for microalgae were investigated in the current work. A sacrificial anode (aluminum) was used to study the electrocoagulation-flotation process, and a nonsacrificial anode (graphite) was used to investigate the electroflotation process. The study inspected the effect of chloride ions concentration and the interelectrode distance on the performance of the electrochemical harvesting processes. The results demonstrated that both electrodes achieved maximum harvesting efficiency with a 2 g/L NaCl concentration. Interestingly, by increasing the NaCl concentration to 5 g/L, the harvesting efficiency reduced dramatically to its lowest value. Generally, the energy consumption decreased with increasing of NaCl concentration. Moreover, the energy consumption achieved with aluminum anodes is lower than that achieved with graphite. However, by increasing the gap between the electrodes from 15 mm to 30 mm, the time required to achieve the maximum efficiency doubled, and energy consumption increased consequently.

Keywords: electrochemical harvesting, electrocoagulation, Chlorella sp., non-sacrificial electrode, energy consumption.

The two significant challenges facing human developments in recent decades are world energy and global warming. These challenges are intensified because of the fast population growth, industries, and the consequent increase in conventional fossil fuel production demands [1]. The complete reliance on fossil-based fuels associated with escalating emissions of Green House Gases (GHG) in the atmosphere is considered the primary factor of severe global climate problems in the present and future years. Moreover, because of the probability of depletion in the fossil fuel reserves, many developed countries have invested considerable funds to investigate alternative renewable and environment-friendly energy sources [2]. Biofuels are considered promising forms of energy that can overcome the negative impacts of fossil fuels on living organisms, including human health. Among various biofuel sources, microalgae have been proposed as an ideal renewable energy route that can efficiently replace conventional fossil-based fuels. Several features make microalgae the key to the next generation of energy, such as high oil content, rapid biomass growth, ability to mitigate the emissions of

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CO_2, and other features that make microalgae superiority over other types of biomass for sustainable development [3,4].

The critical barrier to the commercial production of biofuels from microalgae is their economic feasibility compared with conventional fossil-based fuels. The process of biofuel production involves several steps. The harvesting of microalgae biomass considers the most costly and complicated step in the biorefinery process of biofuel production. Namely, microalgae are in a high diluted mixture (usually 0.1–2.0 g/L) and small size (less than ten micrometers in diameter) for most microalgae strains [5,6].

Different techniques have been exploited for microalgae harvesting, such as centrifugation, sedimentation, flocculation, filtration, flotation, etc. [2, 7–9]. However, the disadvantages of most of these techniques are associated with high operating costs and low efficiency [10].

Electrocoagulation-flocculation approach has been reported as one of the successful tools in wastewater treatment. Moreover, this technique is considered an alternative to conventional chemical coagulants, which use metal salt as chemical coagulants, such as Fe^{3+} or Al^{3+} salts [11–13]. The benefits of the electrocoagulation-flocculation process include easy operation, avoiding adding chemicals, and high efficiency [14].

During the electrocoagulation-flocculation process, Fe^{3+} or Al^{3+} ions are produced by dissolving a sacrificial anode through oxidation reaction as the following reaction on the anode [12,15]:

$$Al \rightarrow Al^{3+} + 2e^{-} \tag{1}$$

$$2H_2O \rightarrow 4H + O_2 + 4e^{-} \tag{2}$$

The following reaction accrue in the solution:

$$Al^{3+} + 3H_2O \leftrightarrow Al(OH)_3 + 3H^{+} \tag{3}$$

The electrochemical reduction of water takes place at the cathode:

$$2H_2O + 2e^{-} \rightarrow H_2 + 2OH^{-} \tag{4}$$

The Al^{3+} ions are dissolved from the anode and react to produce positive charge coagulants, the latter attracts the negative charge microalgae, and the resulting flocs were floated by the aid of the microbubbles (H_2 and O_2) realized on electrodes surfaces.

When chlorides are present in the culture; chlorine, hypochlorous acid, and hypochlorite can be produced from the anodic oxidation of chlorides as in reactions below [11,16]

$$2Cl^{-} \rightarrow 2e + Cl_2 \tag{5}$$

$$Cl_2 + H_2O \rightarrow HOCl + Cl^{-} + H^{+} \tag{6}$$

$$HOCl \leftrightarrow OCl^{-} + H^{+} \tag{7}$$

The dissolved electrode material is one of the parameters that enhance the cost of the electrocoagulation-flocculation process. However, this type of electrode material requires parodic replacement, and the extensive concentration of dissolved material ions hurts the microalgae and the environment [17,18]. Consequently, other types of electrode materials have been used for the electrochemical harvesting of microalgae. In addition, non-sacrificial electrodes like a boron-doped diamond [19], carbon [18], and other electrodes have been examined for the same purpose.

The polarity of the non-sacrificial electrode drives the negative charge microalgae toward the surface of positive charge anodes and neutralizes there. The resulting algal aggregates are driven upward by the action of the microbubble formatted on the electrodes [20,21].

In the current study, microalgae's electrochemical harvesting is investigated using sacrificial (aluminum) and non-sacrificial (graphite) electrodes. Furthermore, the harvesting efficiency and energy consumption were evaluated based on the effect of the variation in inter-electrode distance and electrolyte concentration.

MATERIAL AND METHODS

Cultivation of microalgae

The microalgae strain used in the current study was *Chlorella* sp., which was offered kindly from the department of Biology/Ibn-Haitham College of Education/University of Baghdad. Ten ml of OD= 1 microalgae inoculate were transferred to two 250 ml flasks containing 100 ml sterile Chu-13 media. Then, two glass jars of 5 liters were used for incubation with fresh culture media at a temperature of 25±2 °C. The algal culture was grown at a fixed light intensity of 2500 Lux with 16/8 light-dark conditions. A continuous air stream was supplied to both growth containers at 500 ml/min for 6 hours daily.

Electrocoagulation experiments

The experimental work was conducted using a Plexiglass cylindrical cell of 9 cm diameter and 10.5 height, as described previously [22]. Two types of electrode material were used as anode martial to compare the electrochemical behaviors of both processes, representing the sacrificial electrode (aluminum) and non-sacrificial electrode (graphite). Both electrodes were of 9 cm × 1 cm × 6 cm dimensions. The cathode used in this work was made of aluminum and had a similar size to the anode electrode. A DC power was supplied to the cell at constant current mode by(Smart Power System) model

EMA-01-32-15-P. The applied current was fixed at 0.2 A for all experiments. A magnetic stirrer (Corning, model PC-410) was used for applied constant mixing at a speed of 200 rpm.

For the aluminum electrode cleaning, the electrodes were washed with 1 M HCl solution for 30 minutes, sanded with abrasive paper, and then rinsed with distilled water.

The harvesting efficiency of the microalgal was calculated based on the change in optical density of the culture suspension. The samples were collected from the electrochemical cell at a constant level of 5 cm from the liquid surface. A spectrometer UV-VIS (Bio-Rad, Smartspec plus) was used to measure the optical density of samples at 680 nm. The harvesting efficiency was calculated according to the following equation:

$$harvesting\ efficiency\ (\%) = \frac{(OD_i - OD_t)}{OD_i} \times 100 \quad (8)$$

where OD_i is the optical density of the initial suspension, and OD_t is the optical density of the suspension at a time (t).

The power consumption (in kWh/kg of recovered microalgae) was calculated using Eq. (9):

$$PC = \frac{(P \times t)}{1000 \times V \times \mu_\alpha \times C_i} \quad (9)$$

where P is the power (W), t is the electrolysis time (h), V is the volume of the microalgal broth treated (m^3), μ_a is the microalgae recovery efficiency, and C_i is the initial microalgae biomass concentration (kg/m^3).

RESULTS AND DISCUSSION

Electrolyte concentration

The effect of electrolyte concentration on the harvesting efficiency of *Chlorella* sp. was investigated with different NaCl concentrations (1, 2, 3, and 5) g/L at a constant applied current of 0.2 A, and the pH of the culture medium was around 10. The results in Figure 1 revealed that the harvesting efficiency of *Chlorella* sp. at 1–2 g/L NaCl concentration increased sharply, and consequently, the maximum removal efficiency was reached in a short electrolysis time. The harvesting efficiency of around 96% and the concentration of 2 g/L were achieved in 12 min. However, increasing the NaCl concentration to 3–5 g/L had a markedly negative influence on the removal efficacy. It required 14 min to obtain around 89% harvesting efficiency with an electrolyte concentration of 5 g/L. Furthermore, the removal efficiency of 3.5 g/L NaCl barely reached the response without NaCl addition by the end of the experimental time.

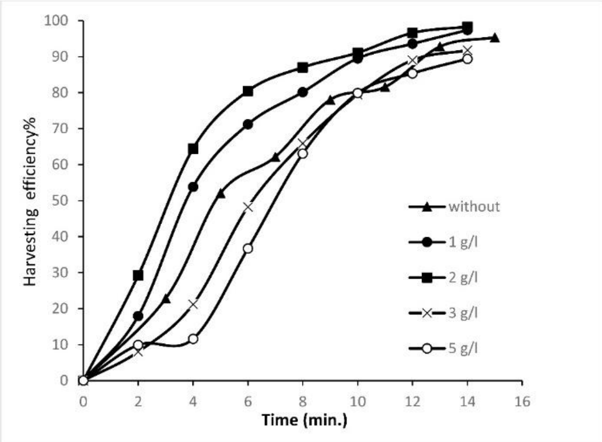

Figure 1. The effect of electrolyte concentration on the harvesting efficiency for the aluminum anode.

For the graphite electrode, the trend of harvesting efficiency is similar to that obtained with the aluminum electrode. Figure 2 shows the effect of different electrolyte concentrations (1, 2, 3, and 5 g/L) on the harvesting efficiency of microalgae. With the electrolyte concentrations of 1, 2, and 3 g/L, the harvesting efficiency was higher than that obtained without adding NaCl. However, with a NaCl concentration of 3 g/L, the removal efficiency was lower than that at 1 g/L and 2 g/L. The harvesting efficiency of more than 98% was achieved in 14 min with electrolyte concentrations of 1 and 2 g/L. In comparison, it required 20 min to obtain about 95% harvesting efficiency with the electrolyte concentration of 3 g/L. Moreover, in compression to zero NaCl addition broth, an increase of the NaCl concentration to 5 g/L has a marked reduction in the removal efficiency for the first 10 minutes of electrolysis time. However, after that, the removal efficiency recovered was almost comparable to that obtained without adding NaCl.

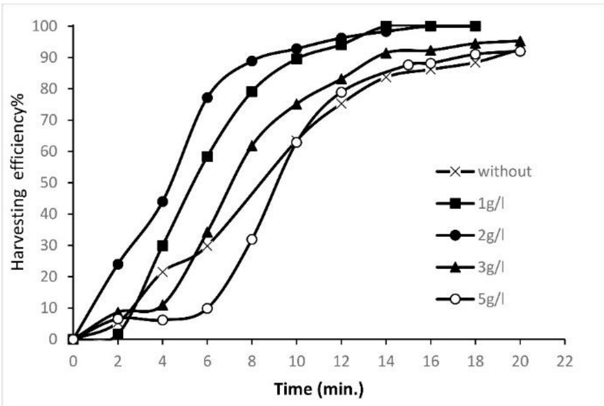

Figure 2. The effect of electrolyte concentration on the harvesting efficiency for the graphite anode.

The above experiments show that both electrodes' highest and lowest harvesting efficiencies were achieved with concentrations of 2 g/L and 5 g/L, respectively. Consequently, the harvesting efficiency of the two electrode types were compared between these two limiting concentrations, i.e., 2, 5 g/L of NaCl.

Figure 3 compares using aluminum and graphite electrodes at the electrolyte concentrations of zero, 2, and 5 g/L. The harvesting efficiency with the aluminum electrode is higher than that obtained with the graphite electrode in all electrolyte concentrations. For example, with electrolyte concertation of 5 g/L, it required 12 min to achieve a harvesting efficiency of 79% by a graphite electrode. In contrast, for the aluminum anode, the harvesting efficiency was around 85% for the same electrolysis time. However, with an electrolyte concentration of 2 g/L after 6 min of electrolysis time, the graphite anode's harvesting efficiency was enhanced to a level obtained with the aluminum anode.

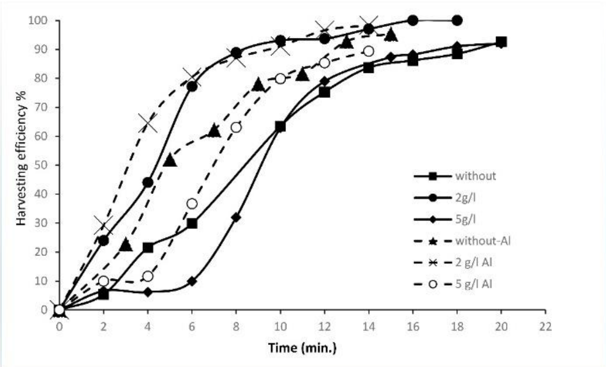

Figure 3. Comparison of graphite and aluminum anodes' harvesting efficiencies with different electrolyte concentrations. The solid line represents the graphite electrode, and the dashed line represents the aluminum electrode.

The presence of chlorine ions in the mixture improved the removal process by increasing the amount of Al^{3+} released by the aluminum electrode and enhanced the current efficiency. The current efficiency increased by approximately 15% when 2 g/L of NaCl was added. It was attributed to the rising conductivity of the electrolyte, which reduced cell voltage. Consequently, at low cell potential, the aluminum dissolution reaction was preferred over the oxygen evaluation reaction [23]. However, the presence of Cl^- in the culture medium reduced the effect of other ions, which could impede the aluminum dissolution and the formation of aluminum hydroxide. Furthermore, the vital rule of chlorine ions was in the de-passivating action of the oxide layer formed on the electrode surface [24,25]. As a result, the passive film built on the electrode led to an increase in energy consumption and a decrease in process efficiency. In addition, when Cl^- is present in the solution, pitting corrosion occurs at the aluminum anode surface, accelerating the dissolution and formation of Al^{3+} [26].

In the electrochemical harvesting process using a sacrificial or nonsacrificial electrode, the increased NaCl concentration in the algal culture may improve the harvesting efficiency due to the increased mass transport of ions to the anode surface [21].

On the other hand, in the electrochemical harvesting of microalgae process, a noticeable decrease in the active chlorine concentration. It could be caused by the reaction between active chlorine and the microalgae cells immediately after its generation, which also has been proved for *Chlorella vulgaris* microalgae [26–29]. It was observed that the algae cells were damaged and lysed due to the presence of Cl^- in the electrochemical process. The pores formed in cell membranes by the external electric field permitted the generated oxidants to penetrate the cells and led to the oxidation of the cell's cellular constituents. Additionally, due to damage to cell membranes, the chlorophyll-a may be subtracted from the cell to the solution and oxide by the active chlorine species generated through the process [26,30,31].

Furthermore, the oxidation agents generated during the process in the presence of Cl^- had an adverse effect on the floc formation [32]. It has been reported that a decrease in Al^{3+} released occurs at higher Cl^- concentration [33]. The Cl^- ions could deposit on the surface of polymeric aluminum hydrolysis and aluminum hydroxide species, which affects their function in generating active flocs [34]. In fact, according to *Aitbara* et al. [35], when chlorides are in contact with $Al(OH)_3$ in the solution, different intermediate species could be produced, such as $Al(OH)Cl_2$, $Al(OH)Cl_2$, and $AlCl_3$. Also, $AlCl_4$ anion can be produced at a high Cl^- concentration, which contributes to the dissolution of aluminum species and impedes the floc's formation. Moreover, the presence of active chlorine species and radicals (Cl_2, HOCl, ClO^- •Cl, •Cl_2^-) and other reactive species in the electrolyte have multiple effects on the microorganism, as listed in Table 1 [29].

All of the reasons mentioned above clearly show that adding NaCl can improve the process removal efficiency. However, microalgae cells can be damaged at specific NaCl concentrations, negatively influencing the electrocoagulation process when aluminum electrodes are used. That may explain the sensitivity of the electrocoagulation process to NaCl, where the removal efficiency of 3 g/L of NaCl was lower than that obtained without adding NaCl when the aluminum is used.

The influence of NaCl addition on microalgae removal efficiency obtained in the current study is harmonious with the results stated by several previous studies. For example, the process efficiency of harvesting freshwater *M. aeruginosa* improved by adding NaCl from 1–5 g/L, but the efficiency started to reduce at an excess concentration of 8 g/L [26]. On the other hand, the removal efficiency of *Chlorella sorokiniana* microalgae using the carbon electrode increased by adding NaCl to the *Chlorella* culture [21].

Table 1. Effect of reactive species on the microorganism in the electrolysis process

Reactive species	Effect
Chlorine	DNA replication enzymes inhibition
ClO_2^-	Changes in cell walls due to oxidation of amino groups
	Disruption of proteo-synthesis
	Disruption of glucose oxidase
O_3	Destruction of cell membranes
	Reaction with glutathione
	Damages of chromosomal DNA
Hydroxyl radicals	Strand breaking in DNA radicals
	Reaction with nucleic acids
	Cell deformation leads to their rupture

Economic feasibility is the most affecting parameter in any process that determines the process efficiency. The energy consumption is calculated based on 90% harvesting efficiency using Eq. 9. Figure 4 shows the effect of NaCl concentration on energy consumption (kWh/kg). It can be observed that the energy consumption obtained with the aluminum electrode is lower than that of graphite with different NaCl concentrations. The energy consumption achieved with graphite at 1 g/L NaCl was 1.7 kWh/kg, decreasing dramatically to around half when the aluminum electrode was used at the same electrolyte concentration. By increasing NaCl concentration, the electrolyte's conductivity increased, the cell voltage decreased, and the energy consumption decreased. However, according to Eq. 9, the time required to achieve 90% is another vital parameter determining energy consumption. The optimum energy consumption achieved with the graphite electrode was 0.413 kWh/kg at an electrolyte concentration of 2 g/L. While with the aluminum electrode, the optimum energy consumption was 0.246 kWh/kg at an electrolyte concentration of 5 g/L. Nevertheless, the optimum removal efficiency was achieved at a NaCl concentration of 2 g/L, at which the energy consumption was 0.258 kWh/kg.

Distance between electrodes

The effect of inter-electrode distance on the harvesting efficiency and energy consumption was investigated with two inter-electrode gaps of 15 mm and

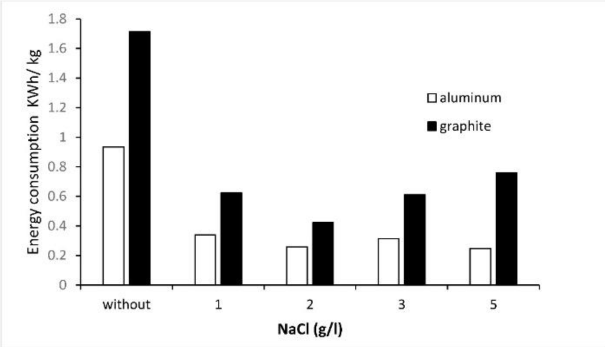

Figure 4. Comparison between the energy consumption of graphite and aluminum anodes with different electrolyte concentrations.

30 mm. The experiment conditions were kept constant at an applied current of 0.2 A without adding NaCl. Figure 5 demonstrated that increasing the distance between the electrodes increases the electrolysis time required to reach the maximum harvesting efficiency. For example, the graphite electrode needed about 20 min to achieve around 92% harvesting efficiency when the gap between the electrodes was 15 mm. At the same time, it took approximately 26 min to attain similar harvesting efficiency when the distance increased to 30 mm. Likewise, with the aluminum electrode, similar behavior was noted. However, the electrolysis time required to reach around 92% decreased from 18 to 13 min when the inter-electrode distance was reduced from 30 mm to 15 mm.

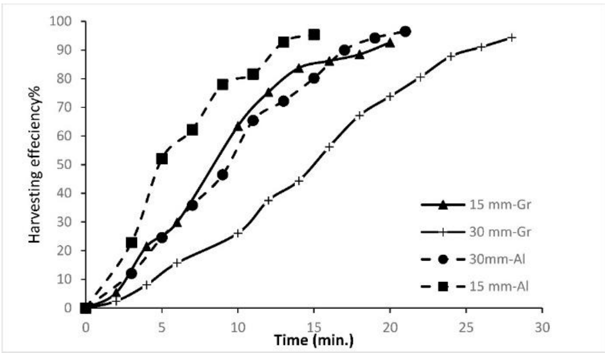

Figure 5. Harvesting efficiency of graphite and aluminum anodes with a distance between the electrodes of 15 mm and 30 mm. The solid line represents the graphite electrode, and the dashed line represents the aluminum electrode.

The reduction of distance between the electrodes increases the process efficiency by increasing the amount of dissolved Al^{+3} and enhancing the performance of flotation and floc formation [11,36,37].

Furthermore, the distance between the electrodes showed a clear impact on the energy consumption for both electrode types, as shown in Figure 6. It can be observed from the results that reducing the gap between the electrodes results in a high reduction in energy consumption. For aluminum electrodes, the energy consumption reduced from 1.4 kWh/kg to

0.93 kWh/kg when the distance was reduced from 30 mm to 15 mm. Likewise, with the graphite electrode, the energy consumption decreased from 2.75 kWh/kg to 1.71 kWh/kg when the electrode gap was reduced from 30 mm to 15mm. The energy reduction is attributed to the decrease in cell resistance with decreasing the inter-electrode distance.

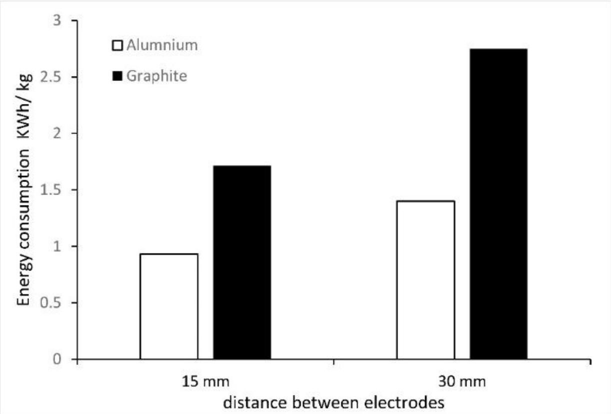

Figure 6. Effect of distance between electrodes on energy consumption for aluminum and graphite electrodes.

CONCLUSION

Although both types of electrodes (aluminum and graphite anodes) have achieved a maximum removal efficiency of the *Chlorella* sp. suspensions, the aluminum anode is more efficient. Cl^- ions have a noticeable effect on the performance of the electrochemical harvesting process. However, the concentration of chloride ions has to be controlled to a specific limit. Maximum harvesting efficiency of 96% has been achieved in a short electrolysis time of 12 min and 14 min with 2 g/L of NaCl for aluminum and graphite electrodes, respectively. With increasing the NaCl concentration to 5 g/L, harvesting efficiency takes longer to achieve the desired value. In general, aluminum anode energy consumption is lower than that of the graphite one. The increase in the NaCl concentration decreases energy consumption. However, electrolysis time is an important parameter in determining energy consumption. The lowest energy consumption value of 0.413 kWh/kg was achieved at 2 g/L of NaCl with graphite, while it was 0.258 kWh/kg with the aluminum one under the same conditions.

The electrolysis time required to achieve the maximum harvesting efficiency was reduced from 26 min to 20 min when the inter-electrode distance decreased from 30 mm to 15 mm for the graphite electrode and from 18 min to 13 min for the aluminum electrode. The energy consumption also decreases with the gap between the electrodes. It was reduced from 1.4 kWh/kg to 0.93 kWh/kg with the aluminum electrode and from 2.75 kWh/kg to 1.71 kWh/kg with the graphite electrode.

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NAUČNI RAD

ELEKTROHEMIJSKO IZDVAJANJE *CHLORELLA* SP.: KONCENTRACIJA ELEKTROLITA I MEĐUELEKTRODNA UDALJENOST

U ovom radu su istraživana dva načina elektrohemijskog izdvajanja mikroalgi. Žrtvena anoda (aluminijum) je korišćena za proučavanje procesa elektrokoagulacije i flotacije, a nežrtvena anoda (grafit) za istraživanje procesa elektroflotacije. Istražen je uticaj koncentracije hloridnih jona i međuelektrodnog rastojanja na performanse procesa elektrohemijskog izdvajanja. Rezultati su pokazali da su obe elektrode postigle maksimalnu efikasnost izdvajanja sa koncentracijom NaCl od 2g/l. Povećanjem koncentracije NaCl na 5 g/l, efikasnost izdvajanja je dramatično smanjena na najnižu vrednost. Generalno, potrošnja energije opada sa povećanjem koncentracije NaCl. Štaviše, potrošnja energije postignuta sa aluminijumskim anodama je niža od one koja se postiže sa grafitnim. Međutim, povećanjem razmaka između elektroda sa 15 mm na 30 mm, vreme potrebno za postizanje maksimalne efikasnosti se udvostručilo, a samim tim je povećana potrošnja energije.

Ključne reči: elektrohemijsko izdvajanje, elektrokoagulacija, Chlorella sp., nežrtvena elektroda, potrošnja energije.