Research

Electrochemical Harvesting of Microalgae

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Abstract:
The rapid growth of the global population is driving increased energy and resource consumption, necessitating alternative feedstocks. Microalgae, with their high growth rates and valuable natural compounds, offer a sustainable solution for producing biofuels, pharmaceuticals, cosmetics, fertilizers, and animal feed. However, harvesting microalgae remains a significant challenge, accounting for 20–30 % of production costs due to factors such as small cell size, low density of microalgae solution, and negative surface charge of microalgae cell. Conventional methods, including centrifugation and filtration, are also high energy consuming. This study explores electrochemical processes as an alternative harvesting technique, focusing on electrocoagulation-flotation with aluminium and aluminium-graphite electrodes and on electroflotation with graphite electrodes. The additional focus of the research was to explore the harvesting efficiency with graphite electrodes to reduce environmental impact by the usage of aluminium. Experiments were conducted at voltages of 5, 8, and 10 V with harvesting durations of 4 and 8 minutes. Two types of microalgae samples were tested: lab-scale cultivated, and pilot-plant cultivated microalgae from high-rate algal pond at the central wastewater treatment plant Ajdovščina. Harvesting efficiency was determined by optical density at 680 nm. Results showed that higher voltages and extended durations improve efficiency, with a maximum at 98.2 % using aluminium electrodes. Combined aluminium-graphite electrodes achieved a comparable efficiency of 96.8 %, reducing aluminium usage by 50 %. Graphite electrodes alone achieved an efficiency of 66.5 %. Electroconductivity also plays a crucial role in the efficiency of electrochemical processes.

Keywords: Electrochemical process; Electroflotation**;** Electrocoagulation; Graphite electrodes; Aluminium electrodes; High-rate algal pond

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1. Introduction

The rapid population growth may lead to a rise in global energy and resource consumption. This growing demand can be addressed using alternative sources. Microalgae, for instance, contain natural compounds that serve as a valuable source of alternative raw materials for producing biofuels, pharmaceuticals, cosmetics, fertilizers, and animal feed. Additionally, microalgae are resistant to environmental conditions and can be cultivated in areas unsuitable for traditional plant farming. Microalgae are single-cell, photosynthetic microorganisms that grow very fast under the three essential elements: a light source, nutrients (primarily nitrogen, phosphorus, and trace metals), and a carbon source (provided in the form of CO_2) (Roy & Mohanty, 2019).

A bottleneck in microalgae production is harvesting, which involves separating algal biomass from the liquid medium in which it is suspended. According to some estimates, this process accounts for 20–30 % of total production costs (Suparmaniam et al., 2022). The high costs and low efficiency of certain harvesting processes are primarily due to: 1) the small size of cells (5–50 μm), 2) the negative charge on cell surfaces, 3) the cell density being similar to that of water (1.08–1.13 g/mL), 4) the relatively low concentration of microalgae in the liquid medium (0.5–5 g/L) (Young et al., 2021). Conventional harvesting techniques, such as centrifugation, filtration, flocculation, and gravitational sedimentation, have some drawbacks in terms of efficiency, toxicity, costs, and sustainability. To address these limitations, new harvesting methods must be explored, including bioflocculation, electroflotation-coagulation, ultrasonic aggregation, magnetic separation, and phototaxis (Zhu et al., 2024). Electrochemical (EC) microalgae harvesting is based on the application of a direct electrical current through electrodes into a culture broth. During this process, electrolysis occurs between the anode and cathode. Electrolysis can take place using either metallic or non-metallic electrodes. Metallic electrodes are sacrificial electrodes and thus releasing metal ions that can contaminate the final biomass and liquid, making the final products unusable. The objective is to use non-metallic, non-sacrificial electrodes that remain inert during electrolysis and do not react with the solution or biomass, such material like graphite (Coons et al., 2014).

Microalgae can thrive in diverse environments, including in nature-based solutions for wastewater treatment such as high-rate algal ponds (HRAPs). HRAPs are shallow, open ponds made for the treatment of wastewater where the wastewater represents a source of the nutrients for microalgal growth (Magalhães et al., 2024). The aim of this study is to separate microalgal biomass from wastewater originating from the HRAP system. The concentrated microalgal biomass holds potential as biofertilizer and biostimulant in agriculture, while the treated water can be repurposed for irrigation. Therefore, it is important to address potential environmental and health risks in compliance with Directive (EU) 2024/3019 on urban wastewater treatment, as well as Regulation (EU) 2020/741 on minimum requirements for water reuse.

EC processes for harvesting microalgae has been reported to be effective by some researches. Liu et al. (2018) investigated the efficiency of graphite and aluminium electrodes on *Scenedesmus sp.* and concluded that the process could achieve above 90 % of microalgal removal efficiency only if aluminium ions were present in the solution. Al-Yaqoobi & Al-Rikabey (2023) concluded that a maximum harvesting efficiency of 96 % was achieved in 12 min for aluminium and 14 min for graphite, with the addition of 2 g/L of NaCl.

2. Material and Methods

2.1. Lab-scale cultivation of microalgal biomass

The microalgae strains used in the current study, *Chlorella vulgaris* and *Scenedesmus sp.*, were supplied by the company Algen LLC. The algal culture was grown in Bold's Basal Medium with added nutrients (0.34 g/L $NaNO_3$) at a light intensity of 65 μmol/m^2s at room temperature. A photobioreactor (PBR) with a working volume of 27 L operated under semi-sterile conditions, with a stirring mechanism and controlled CO_2 supplementation. Electroconductivity (1200 μS/cm) and pH (9.3) were measured with WTW multi 3620 IDS multi-parameter portable meter.

2.2. *Pilot-plant cultivation of microalgal biomass*

Environmental samples were obtained from the HRAP located at the Central Wastewater Treatment Plant Ajdovščina, Slovenia. HRAP with volume of 3 m^3 was designed as a pilot plant to treat primary treated wastewater from the central wastewater treatment plant. It was operated in a batch mode receiving 300 L of every 3 days. HRAP was continuously steered with a paddle wheel and contained a mixture of bacteria and microalgal species, predominantly belonging to *Chlorella* sp., *Scenedesmus* sp., and *Pseudopediastrum* sp. that provided secondary (removal of organic substances) and tertiary (nutrient removal) treatment of wastewater.

The first sampling took place in mid-October 2024. The algae in the pond were visibly in poor condition and the solution was diluted due to the rainy weather. Therefore, 2 L of algae-bacteria sludge from the ultrasonic sedimentation tank was taken and mixed with 25 L water from the pond. The 2 L sample from the tank was sourced from the same HRAP system a few weeks earlier, during the period when algal biomass was in good condition. The second sampling took place at the beginning of November 2024. Samples were again taken from the ultrasonic sedimentation tank (algae-bacteria sludge, 2L), and HRAP (25 L). Due to the diluted media in the pond, the solution had a low electrical conductivity (200 μS/cm), which disabled the EC process. In the previous experiment, the electrical conductivity was approx. 1200 μS/cm. To achieve this conductivity in the second experiment and enable the EC process to function effectively, 0.5 L of a 2.5 % NaCl solution was added.

2.3. *Electrochemical experiments*

The experimental work was carried out using a 2 L glass beaker. Two types of electrode materials were used to compare the EC process: the sacrificial electrode (aluminium; Al) and the non-sacrificial electrode (graphite; Gr). When aluminium is used, the process involves electrocoagulation-flotation, whereas with graphite, the process is limited to electroflotation (Al-Yaqoobi & Al-Rikabey, 2023). Electrodes had dimensions of 10 cm × 5 cm × 0.3 cm. The distance between electrodes was 0.5 cm. There were three configurations of electrodes: 1) set of four Al electrodes; 2) set of four Gr electrodes and 3) set of two Al and two Gr electrodes arranged in the sequence: Al-Gr-Al-Gr. The applied constant voltage from DC power supply Basetech BT-3020 was set to 5, 8, and 10 V. A magnetic stirrer was used for constant mixing at a speed of 100 rpm. The experiments were performed with approx. 900 mL of initial sample at room temperature. The full setup of the laboratory experiment is shown in Figure 1a, and electrode sets on Figure 1b and 1c. During the process, the flocs of microalgae floated to the surface of microalgal suspension due to gases formed at the electrodes (H_2 and O_2). The samples were taken with a tube at the depth of approx. 5 cm and were collected at 4 and 8 minutes throughout the EC process.

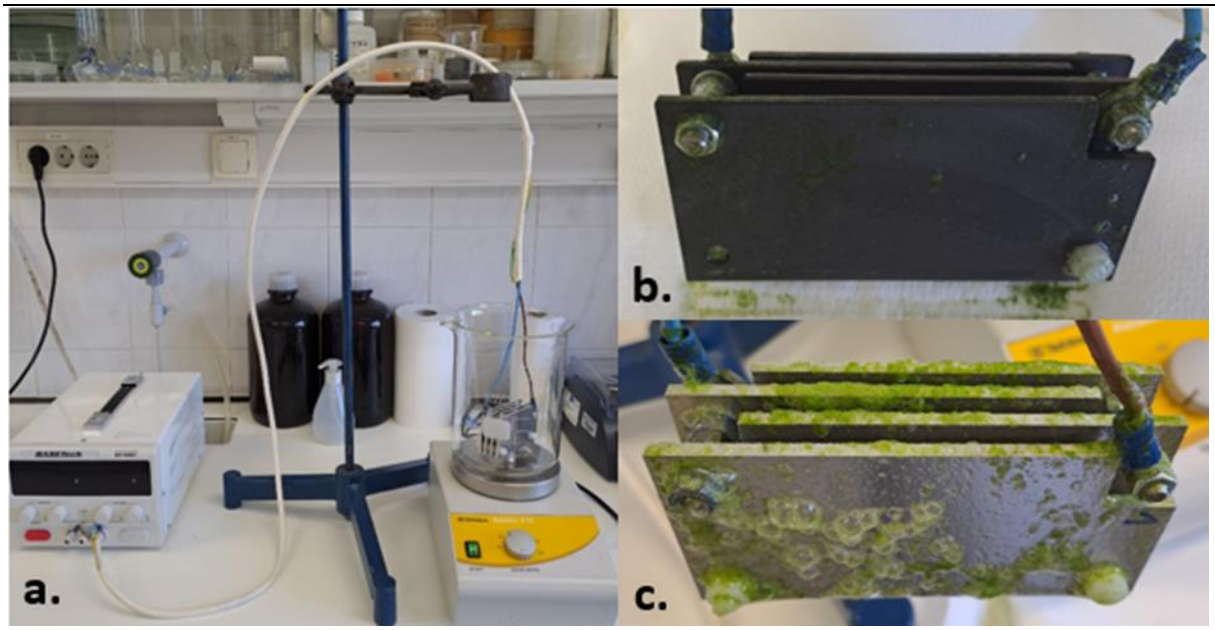

Figure 1. a) Laboratory setup. b) Graphite electrodes after EC process. c) Aluminium electrodes after EC process.

2.4. Determination of harvesting efficiency

The harvesting efficiency of the microalgae was calculated based on the change in optical density. Absorbance of the culture suspension was measured using UV-Vis spectrometer Nanocolor VIS (Macherey-Nagel, Germany), at 680 nm (Al-Yaqoobi & Al-Rikabey, 2023). The harvesting efficiency was subsequently calculated as Eq. (1):

$$\textbf{harvesting efficiency } [\%] = \frac{(ODi - ODt)}{ODi} \times 100 \quad (1)$$

where *ODi* is optical density of the initial suspension, and *ODt* is the optical density of the suspension at a time t (Al-Yaqoobi &Al-Rikabey, 2023).

3. Results

3.1. Results for lab-scale cultivated microalgae

After calculating the efficiency based on absorbance at 680 nm, the maximum harvesting efficiency for lab-scale organisms is as follows:

Table 1. Summarized maximum efficiency results for harvesting lab-scale organisms based on the absorbance at 680 nm.

Electrode material	Voltage [V]	Time [min]	Efficiency [%]	Experiment
Al	10	4	58.0	October
Al-Gr	5	4	0.6	October
Gr	10	4	2.9	October
Al	10	8	31.0	November
Al-Gr	10	8	14.2	November
Gr	10	8	9.8	November

In the initial experiment in October, several aspects were not fully optimized. The time limit was initially set to 4 minutes, as this duration was sufficient to harvest the environmental sample during preliminary testing. However, after harvesting the model organism for the first time, it became apparent that extending the time would improve the process.

As a result, for the next three experiments, the time limit was set to 4 and 8 minutes. Additionally, during the first experiment, the mixing of the initial sample was not sufficient, resulting in a denser sample by the end of the test when using the combined Al-Gr electrode. This inadequate mixing led to reduced efficiency with the combined electrode. In subsequent experiments, this issue was addressed by optimizing the sample mixing process.

3.2. Results for pilot-plant cultivated microalgae (environmental samples)

Based on the absorbance at 680 nm, the highest harvesting efficiency results for environmental samples from the HRAP system are as follows:

Table 2. Summarized maximum efficiency results for harvesting environmental samples from HRAP system based on the absorbance at 680 nm.

Electrode material	Voltage [V]	Time [min]	Efficiency [%]	Experiment
Al	10	8	89.0	October
Al-Gr	10	8	92.1	October
Gr	10	8	17.4	October
Al	10	8	98.2	November
Al-Gr	8	4	96.8	November
Gr	10	8	66.5	November

The most efficient electrodes were Al and the combined Al-Gr, both achieving approx. 90 % efficiency. The optimal voltages were 10 V and 8 V, when the process duration was set to 8 minutes. Gr electrodes were less effective with 17.4 and 66.5 % of removal efficiency. The harvesting efficiency of environmental samples from the HRAP system was better compared to lab-scale biomass (max. 98.2 % for environmental; max. 58.0 % for lab-scale).

4. Discussion

Harvesting efficiency increased with higher voltage, as the stronger electric field enhanced the production of H_2 and O_2 when using all three types of electrodes. These gases enabled microalgae flotation by producing a continuous stream of fine bubbles in the solution. In addition, higher voltage resulted in the release of more aluminium ions when using Al and Al-Gr electrodes, which likely caused microalgae coagulation (Liu et al., 2018).

Increasing the harvesting time also improved harvesting efficiency by increasing the dissolution of metal ions from the anode and providing additional time for flotation with gas bubbles. However, as harvesting time can impact the final quality of the algal biomass, the process duration needs to be controlled (Visigalli et al., 2021).

The selection of electrode materials has a significant impact on the EC process. Despite identical operating conditions during experiments, differences were observed between Gr and Al electrodes. Liu et al. (2018) emphasized that neutralizing the electric charge on the microalgae cell surface is important to achieve high harvesting efficiency. This process can be achieved using positively charged metal ions such as Al^{3+} releasing from Al electrodes together with an electric field. Gr electrodes are not producing positively charged metal ions which explains their reduced harvesting efficiency. However, in our experiment on environmental samples, the Al-Gr electrode demonstrated relatively high efficiency, while reducing Al usage by 50 %.

Major differences in harvesting efficiency were also evident when comparing lab-scale cultivation with environmental (HRAP) samples. Efficiency of environmental samples was higher, likely due to the chemical and microbial composition of the algal medium.

Electroconductivity proved to be an important parameter for successful EC reactions, as low electroconductivity inhibited the process. Nageshwari et al. (2022) highlighted that

higher salinity of the electrolyte increases conductivity, thereby promoting ion release required for microalgal removal. However, the addition of large amounts of NaCl can increase costs and introduce several drawbacks related to utilization of both water and biomass. Additionally, Al-Yaqoobi & Al-Rikabey (2023) reported that increasing the NaCl concentration from 2 g/L to 3-5 g/L significantly reduced harvesting efficiency.

5. Conclusions

The laboratory-scale study showed that EC processes (flotation and coagulation) can achieve high harvesting efficiency also with combination of non-sacrificial electrodes. The highest harvesting efficiency for environmental samples using combined Al-Gr electrode was 96.8 %, achieved at 8 V and 4 minutes. This efficiency is comparable to the maximum achieved with Al electrodes alone, which reached 98.2 % at 10 V, 8 minutes. The use of graphite alone is less sufficient, achieving only 66.5 % efficiency at 10 V in 8 minutes. Significant differences are observed in the harvesting efficiency between lab-scale microalgae and environmental samples from the HRAP. Further research needs to evaluate the compounds present in the HRAP system that enhances harvesting efficiency compared to lab-scale cultivated microalgae.

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