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Research Article

Electro-flotation harvesting of microalgae using a combination of electrode types

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Abstract

This study aimed to search for a proper system for electro-flotation harvesting of microalgae *Dunaliella salina* with a minimization of metal contamination. An applied electrical current caused cell aggregation and produced small bubbles which levitated cell clusters to the top surface. Since no electrolytic erosion for cathodes (negative electrodes), we tested the harvesting by using planar cathodes, lying in the bottom of the reactor, made from different materials and forms. For a given electrical current, all cathodes resulted in a similar harvesting efficiency as well as the energy consumption. Unlike the cathode, the anode (positive electrodes) usually erodes in the electrolytic process. To avoid contamination of released metal, we used only graphite plates as the anode. Different settings of graphite anode plates (located above the cathode) were studied. For a given electrical current, all cases of anode settings had a similar harvesting efficiency but the energy consumption increased with the distance between the anode and the cathode. For a given setup of electrodes, we showed that both the harvesting efficiency and the energy consumption increased with the applied electrical current. By considering the above results as well as the fact that the stainless steel plate is cheaper and easier to handle in comparison to the graphite plate, we suggest that a combination of graphite anode and stainless steel cathode is an appropriate set for microalgae harvesting with an avoidance of metal contamination. Furthermore, for a convenience of the collection of the levitated microalgae cells, the top surface should be leaved widely opened by setting the graphite anode plates near the wall of the reactor. Finally, a large scale harvesting of the microalgae cultured with outdoor conditions is demonstrated in Nakhon Ratchasima, Thailand.

Keywords: *Dunaliella salina*, harvesting, electro-flotation, stainless steel, graphite

Introduction

Microalgae have a potential in a wide variety of applications. Microalgae have been considered as the natural sources of energy and bioproducts including carotene (Lamers et al., 2008; Zhu & Jiang, 2008). Several research pathways have been explored to improve the economics of the microalgae production including microalgae harvesting (Christenson & Sims, 2011; Dassey & Theegala, 2013). Different techniques of microalgae harvesting are currently investigated, e.g., chemical coagulation/flocculation, flotation, filtration, centrifugation, and electrical based process. Microalgae utilization is not economically sustainable due to difficult and high cost of harvesting. The operation cost depends on the harvesting method as well as

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type of microalgal species. Quantitative and comprehensive cost comparisons of difference harvesting methods and microalgal types can be found in review articles, e.g., Lee et al., 2013; Barros et al., 2015 and Marrone et al., 2017. While centrifugation can harvest most algal types with rapid cell harvesting, its operational cost is much higher than others. The operation cost of other methods is quite similar.

Flotation is a process where gas bubbles fed to the liquid medium providing a lifting force needed for particle transport and separation. Electro-flotation generates bubbles through electrolysis (Baierle et al., 2015; Zhou et al., 2016; Vandamme et al., 2011). The generation of these bubbles can be done at both electrode and coupled with the electro-coagulation that occurs through the electrolytic oxidation that happens at the cathode (Gao et al., 2010). Separation by such flotation depends on bubble flux and size (Coward et al. 2014).

Electro-flotation harvesting of microalgae has been recently investigated by using electrodes made from various kinds of metals including Fe, Al, Cu, Mg and stainless steel. (Bleeke et al., 2015; Ryu et al., 2018; Ghernaout et al., 2014; Xu et al., 2010; Zenouzi et al., 2013; Shuman et al., 2015; Dassey & Theegala, 2014; Barros et al., 2015). During an application of electrical current, the positive electrodes release metal ions into the medium. These residual metal ions potentially act as coagulant and liberate adversely affecting microorganisms due to their toxicities in aqueous and microalgae biomass (Kim et al., 2012). An avoidance of this metal contamination has been demonstrated by using graphite plates for both positive and negative electrodes (Misra et al., 2014; Zhou et al., 2016).

In this article, we presented a study of microalgae harvesting using electro-flotation. To avoid adding metal contamination into the harvested microalgae, graphite plates were used as the anode which usually eroded by electrolysis. In contrast, an applied electrical current does not cause erosion of cathode. Therefore, we tested cathode plates made from different materials and forms. Then, the configuration of graphite anode as well as the electrical current were varied. A proper setup of electrodes was designed by considering the harvesting efficiency and energy consumption. Finally, a harvesting of the microalgae in a large scale was demonstrated.

Materials and methods

Microalgae *Dunaliella Salina* strain KU 11 (Wu et al., 2017) were cultured in transparent glass chambers (30 cm × 60 cm × 40 cm) using a modified Jonson's medium with 2 M NaCl (Wu et al., 2017). Each chamber contained 50 litres of medium and illuminated by fluorescent lamps with an average light intensity of 54 µmol m^{-2} s^{-1} from top for 24 h day^{-1}. Harvesting experiments were carried out when the microalgae cultured for 14 days and the cell density was about 14.1×10^{6} cells ml^{-1}.

As shown in Figure 1, the experiments were conducted using 4.0 liters of the microalgae culture in a transparent rectangular reactor with dimensions of 5 cm × 30 cm × 35 cm. The cathode plate was laid in the bottom of the reactor while the anode was set (either vertically or horizontally) above the cathode. An electrical current was applied using a DC power supply (Wanptek, Model KPS0D) in constant current mode.

The effect of electrode types as well as their configuration on the microalgae harvesting was studied. Four different cathodes (lying in the bottom of the reactor) were investigated; cathode I: a stainless steel plate with dimensions 4 cm × 28 cm × 0.1 cm, cathode II: a stainless steel plate with dimensions 2 cm × 28 cm × 0.1 cm, cathode III: a perforated stainless steel plate with dimensions 4 cm × 28 cm × 0.1 cm, 3-mm round holes and 40% open area, and cathode IV: a graphite plate with dimensions 4 cm × 28 cm × 1 cm

To avoid contamination of metals usually released from electrolytic erosion, we used the anodes made from only graphite. Four configurations of the anodes were tested in the setup using only cathode I. A couple of graphite plates with dimensions of 4 cm × 35 cm × 1 cm was set vertically at the left and the right of the reactor. The space between the cathode and the anode was varied for three cases: 1, 10, and 20 cm as anode I, II and III, respectively. The fourth configuration (anode IV) was a single graphite plate with dimensions of 4 cm × 28 cm × 1 cm, which was set horizontally with a distance of 20 cm apart from the cathode.

The influence of applied electrical current on the microalgae harvesting was examined in a setup with a couple of vertical graphite anodes setting at 1 cm (anode I) apart from a stainless steel cathode I (4 cm × 28 cm × 0.1 cm in dimensions). The applied electrical current was varied as 2, 4, and 8 A.

To determine the harvesting efficiency, 10 ml samples were collected from the middle of the reactor every minute during the experiments. The optical density of the samples was measured by using a spectrometer (Ocean optics, model HRS-4000). The harvesting efficiency was determined from the reduction in the optical density at a wavelength of 550 nm (OD_{550}) as

$$\text{harvesting efficiency (\%)} = (1\text{-}A/B) \times 100,$$

where A is the OD_{550} at time t and B is the OD_{550} before the application of the electrical current. The culture medium is used as the blank in the measurements so that in a null test (without microalgae), its optical density A is zero. The electrical energy consumption was estimated from the applied electrical current I (A), the applied voltage V (V), time t (h) and the microalgae volume L (m^{-3}) as

$$\text{energy consumption} = I \times V \times t \,/\, (1000 \times L)$$

which has a unit of kWh m^{-3} as usually reported (Barros et al., 2015).

To demonstrate the microalgae harvesting in a commercial scale, the microalgae *Dunaliella Salina* strain KU 11 were cultured in raceway tanks with a volume of 800 liters with outdoor conditions (Wu et al., 2017) in a salt production company located in Nakhon Ratchasima, Thailand (15◦ 8.4 N, 101◦ 9.4 E).

Microalgae biomass was harvested in a large transparent rectangular reactor with dimensions of 50 cm × 100 cm × 50 cm. Four graphite anodes (45 cm × 50 cm × 1 cm in dimensions) were placed vertically close to two opposite walls and above a stainless cathode plate (45 cm × 90 cm × 0.1 cm) which was laid at the bottom. A strong electrical current 120 A (the voltage of 12 V) was applied from a DC power supply (MEAN WELL SE-1500-12).

Results and discussion

Figure 1 demonstrates an example of the microalgae harvesting using electro-flotation. Prior to the application of the electrical current, the microalgae culture appeared dark green as in Figure. 1(a) and had a high optical density. When the electrical current was applied, many small bubbles generated from the electrolysis were distributed throughout the reactor. In the course of time, the bubbles as well as the levitated microalgae gradually accumulated as foam at the top surface. In Figure 1(b), the medium became lighter green at 5 min after the application of the electrical current and the harvesting efficiency was 49.3%. When most of the microalgae were lifted to the surface, the foam at the top surface appeared as two layers: a green layer with concentrated microalgae lying above a white layer composed of tiny bubbles

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(e.g. in Figure 1(c) and Figure 1(d) where the harvesting efficiency was 87.3% and 90.0%, respectively).

Figure 2 shows an example of cell aggregation of the microalgae *Dunaliella salina* induced by the applied electrical current. The microalgae are small single cells distributed throughout the culture medium, e.g., in Figure 2(a). These microalgae cells behave like suspension since they have negative charged surface (Vandamme et al., 2013). After an application of electrical current, e.g., 4 A for 5 min shown in Figure 1(b), the single cells were induced to form larger clusters, e.g., in Figure 2(b) and Figure 2(c). The cell clusters were much larger than the individual single cells. Since larger particles can easier to be lifted by bubbles so that the microalgae harvesting is facilitated by such the electrically induced cell aggregation, as reported earlier for other microalgae cells, e.g., *Chlorella Sp.* (Zhou et al., 2016) and *Scenedesmus acuminatus* (Bleeke et al., 2015).

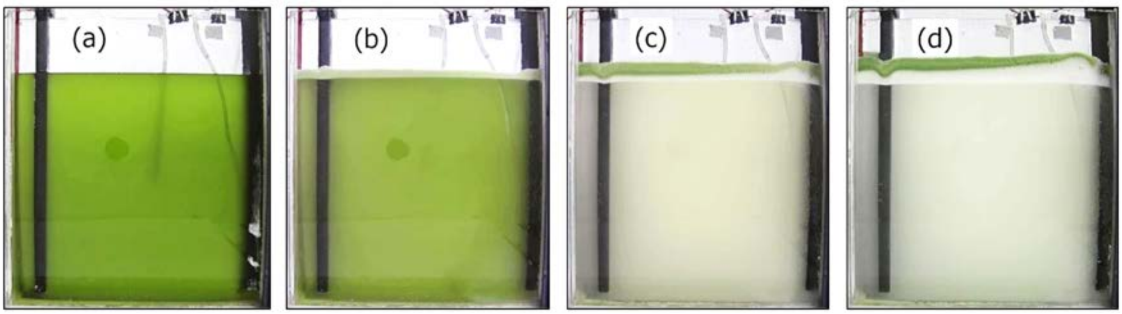

Figure 1. Microalgae harvesting using an electro-flotation. Sequential images (a) – (d) illustrate the experimental results at 0, 5, 10 and 15 min, respectively. A stainless steel cathode (4 cm × 28 cm × 0.1 cm in dimensions, cathode I) was laid in the bottom and a couple of vertical graphite anodes (anode I) was set at 1 cm above it. The applied current was 4 A.

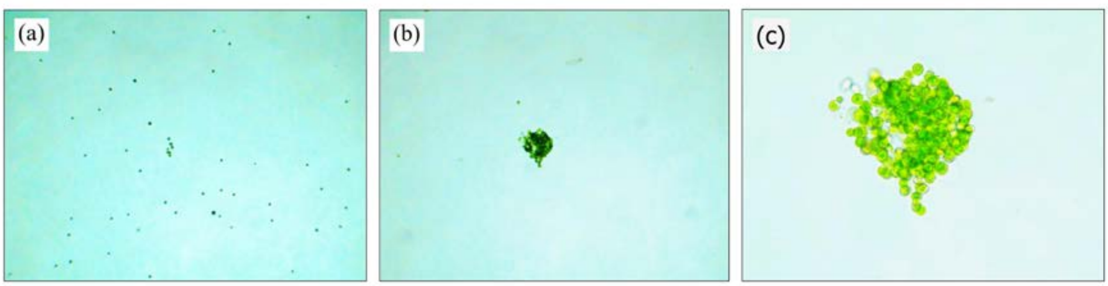

Figure 2. Images of microalgae *Dunaliella salina* under microscope: (a) a 10x image of single cells that stayed separately in the culture medium, (b) a 10x image and (c) a 40x image of a large cluster of the cells induced by an applied electrical current.

Figure 3 shows the harvesting efficiency obtained from the setup with four different cathodes. In these experiments, the anode I, a couple of vertical graphite plates at 1 cm above the cathode (as shown in Figure 1), was used and the applied electrical current was 4 A. All cathodes gave similar curves of the harvesting efficiency. At the beginning, the harvesting efficiency increased very fast. At 10 min, the harvesting efficiency reached about 80% for cathode IV (made from graphite) while other three cathodes gave a higher efficient of about 87%. For longer times, the harvesting efficiency still increased but with slower rate: at 20 min, the harvesting efficiency from all cathodes reached similar values of 89% and 97% at 15 and 20 min after the application of electrical current. Thus, in the initial phase of 10 min, the graphite cathode gives a lower harvesting efficiency than the stainless steel cathodes while all cathodes provide similar efficiency after 15 min.

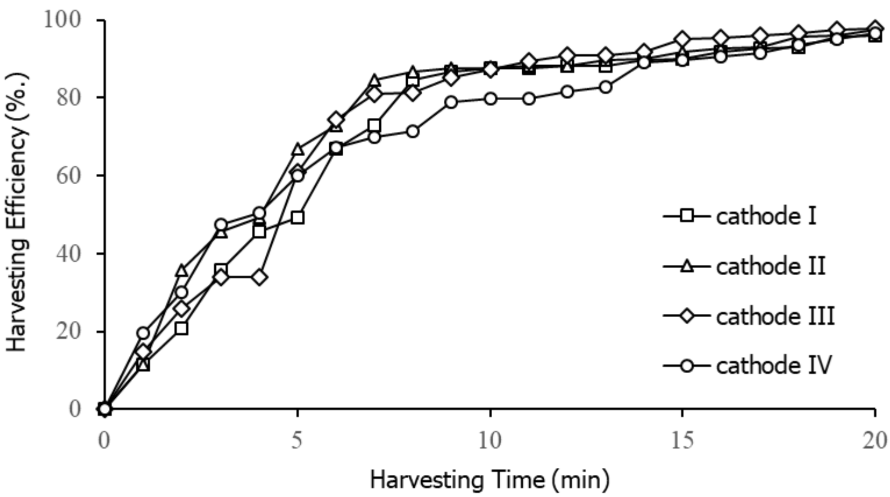

Figure 3. Efficiency of the harvesting using different cathodes; cathode I: a stainless steel plate (4 cm × 28 cm × 0.1 cm), cathode II: a stainless steel plate (2 cm × 28 cm × 0.1 cm), cathode III: a perforated stainless steel plate (4 cm × 28 cm × 0.1 cm, 3-mm round holes and 40% open area), and cathode IV: a graphite plate (4 cm × 28 cm × 1 cm). A couple of vertical graphite plates was placed 1 cm (Anode I) above the cathode.

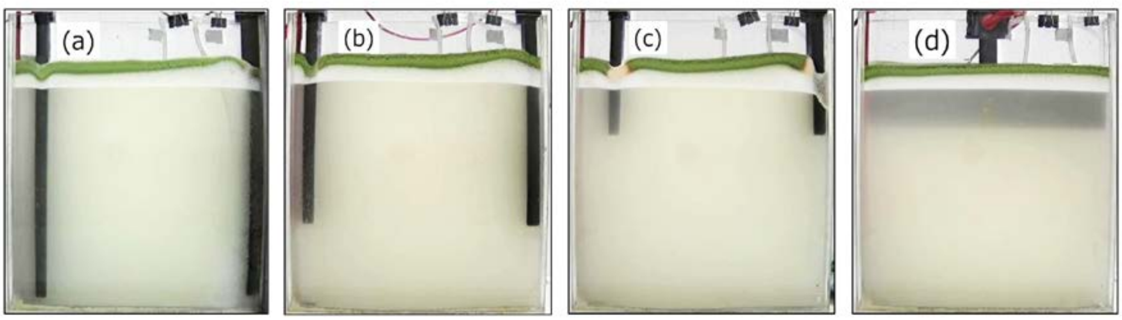

Figure 4. Microalgae harvesting with different anode configurations. A stainless steel cathode (4 cm × 28 cm × 0.1 cm in dimensions, cathode I) was laid in the bottom. (a) – (c) A couple of graphite anodes was set vertically at the left and the right with a distance of 1 cm (anode I), 10 cm (anode II), and 20 cm (anode III) above the cathode I. (d) A graphite anode was set horizontally with a distance of 20 cm (anode IV) above the cathode I. The images were taken at 15 min after electrical current application.

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Microalgae harvesting by using four different configurations of graphite anodes was studied in cooperation with cathode I. As shown in Figure 4, when most of the cells were harvested, i.e., when the harvesting efficiency was more than 80%, typical foam layers composed of a green layer of concentrated microalgae lying above a white layer of small bubbles occurred in all cases. Note that for the cases of a couple of vertical graphite plates in Figures. 4(a) – 4(c), the foam layers close to the anodes were thicker than those at other positions while for the case of horizontal graphite plate, the form was distributed evenly through the length of the reactor. This comes from the fact that the bubbles produced from the vertical graphite plates moved up nearby the plates but the horizontal plate generated the bubbles those raised evenly throughout the length of the plate.

The harvesting efficiency from experiments using four different anode configurations (see Figure 4) are shown in Figure 5. Similar to the results in Figure 3, after the application of electrical current was started, the harvesting efficiency grew up very fast and exceeded 80% within 10 min for all anode configurations. Then, it increased slowly. After 6 min, the harvesting efficiency of anode IV was higher than the others while that of anode III was the lowest. However, all curves reached similar values of 96-98% at 20 min.

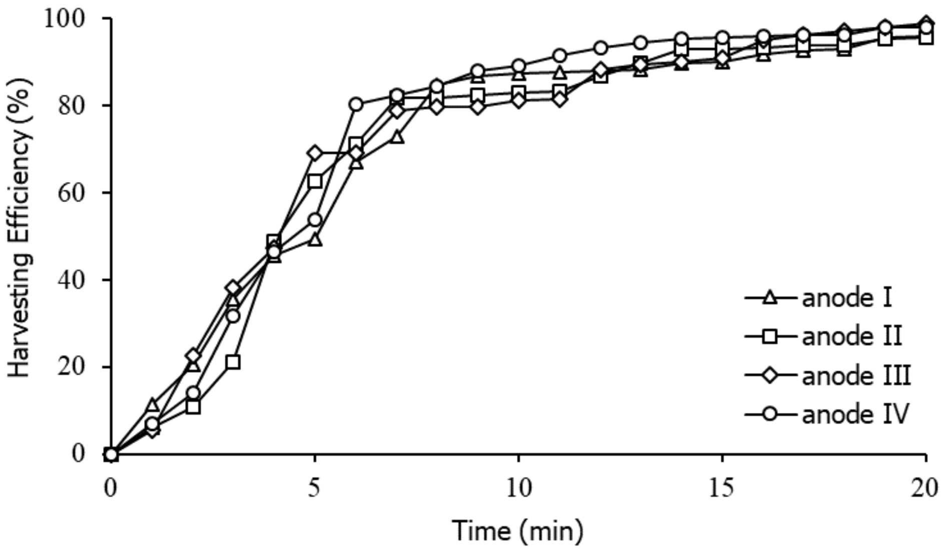

Figure 5. Efficiency of the harvesting using different configurations of the anodes. The curves labeled anode I, anode II, anode III, and anode IV were obtained from experiments using the setups in Figures 4(a) – 4(d), respectively.

Figure 6 shows the harvesting efficiency from experiments using different applied electrical current. The electrodes were set as in Figure 1. It can be seen clearly from the graphs that the higher electrical current results in the higher harvesting efficiency at all time of the experiments. Similar to the previous results in Figure 3 and Figure 5, the typical graph shape in which a fast increment of the harvesting efficiency was followed by a slower grow up. The first interval of such fast increment depends on the applied current: For 2, 4, and 8A, the harvesting efficiency reaches 77.4%, 84.7%, and 91% at 10, 8, and 5 min, respectively. The very strong current provided 100% of harvesting efficiency at 13 min while the cases of 2 and 4 A, the harvesting efficiency reaches 92.4% and 96.0% at 20 min. These present effect of electrical

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current on the harvesting agree to the study using other microalgae *Chlorella vulgaris* and *Phaeodactylum tricornutum* where the electrodes made from aluminum and iron (Vandamme et al., 2011).

The energy consumption of the electro-flotation harvesting are summarized in Table 1. It was calculated from the harvesting time of 15 min where in most of the experiments the harvesting efficiency was at least 90%, except for the lowest current of 2A with the harvesting efficiency of 82.8%. For the setups with anode I and the applied current 4 A, all cathode I-IV gave very similar energy consumption values of around 1.0 – 1.1 kWh m^{-3} (see setups 1-4 in Table 1). In contrast, the graphite anode configuration strongly affected energy consumption: an enlargement of the cathode-anode spacing from 1 to 20 cm resulted in an increment of both the voltage (from 4.1 to 7.0 V) and the energy consumption from 1.03 to 1.75 kWh m^{-3} in setups using cathode I and the current 4 A (setups 1, 5-7). Note that the setups 1-7, where the applied current was fixed at 4 A, provided similar harvesting efficiencies around 90-96%. For a given set of electrodes, both the harvesting efficiency and the energy consumption clearly increased with the applied electrical current. By comparing setups 1, 8, and 9, the increasing of the applied electrical current from 2 to 8 A caused a raise of the harvesting efficiency from 83% to 99%, however, the energy consumption increased from 0.41 to 1.63 kWh m^{-3}.

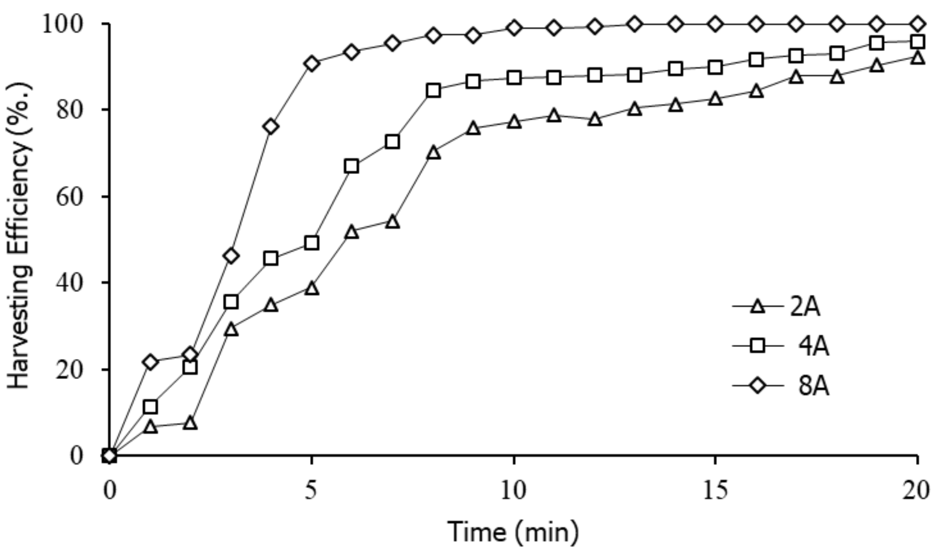

Figure 6. Effect of the applied electrical current on the harvesting efficiency. The setup composed of cathode I and anode I, as shown in Figure 1, were used.

Although the results show that the setups using different cathodes give approximately similar harvesting efficiency and consume similar energy, a stainless steel plate is more suitable to be used as the cathode as the following reasons. A graphite plate is fragile so it is prone to be broken during operation. It is difficult to produce a graphite plate thinner than 1 cm so it results in a relatively high production cost of a graphite plate, in comparison to, e.g., a stainless steel plat with the same area size. Finally, in comparison to other cheap metals, stainless steel is robust against corrosion caused by the culture medium of *Dunaliella salina* which contains high concentration of NaCl and other chemicals.

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The setups with different anode settings provided a similar harvesting efficiency but the energy consumption increased when the anode was set farer from the cathode. These results agree to previous reports where both the cathode and the anode are made from the same materials: graphite (Zhu et al., 2016) or stainless steel (Ghernaout et al. 2015). For a given distance from the cathode, the orientation of the anode, i.e., vertical or horizontal setup, does not affect the harvesting process. Furthermore, the top surface of the reactor should be leaved widely opened to facilitate the collection of the foam of concentrated microalgae cells (foam layers in Figure 1 and Figure 4) during the application of electrical current. Therefore, vertical graphite plates setting close to the stainless steel cathode and near the walls of the reactor are appropriate to be used as the anode.

Table 1. Energy consumption and harvesting efficiency of different setups at 15 min.

setup	electrodes	space (cm)	current (A)	voltage (V)	harvesting efficiency (%)	energy consumption (kWh m^{-3})
1	cathode I, anode I	1	4.0	4.1	90.00	1.03
2	cathode II, anode I	1	4.0	4.1	91.83	1.03
3	cathode III, anode I	1	4.0	4.2	95.03	1.05
4	cathode IV, anode I	1	4.0	4.3	89.76	1.08
5	cathode I, anode II	10	4.0	5.6	93.12	1.40
6	cathode I, anode III	20	4.0	7.0	90.88	1.75
7	cathode I, anode IV	20	4.0	7.0	95.76	1.75
8	cathode I, anode I	1	2.0	3.3	82.8	0.41
9	cathode I, anode I	1	8.0	4.9	99.00 [a]	1.63 [a]

[a] The results were obtained at 10 min.

To demonstrate the microalgae harvesting in a commercial scale, the microalgae *Dunaliella salina* was cultivated with outdoor conditions in a raceway tanks as in Figure 7(a). We enlarge the electrodes composed of a stainless steel cathode plate lying in the bottom of the reactor and four graphite anode plates setting vertical above the cathode and near the two opposite walls. The microalgae harvesting volume was scaled up 50 times of the laboratory scale so that the reactor was enlarged from 5 cm × 30 cm × 35 cm (as in Figure 1) to 70 cm × 100 cm × 50 cm. The scale up was mainly done in the width direction from 5 cm to 100 cm (20 times). To increase the stability of the large reactor, the length was extended from 30 cm to 70 cm (about 2.33 times). The height of the reactor was increased from 35 cm to 50 cm and the height of the culture medium in the reactor was set to about 30 cm so that the culture medium were about 200 L.

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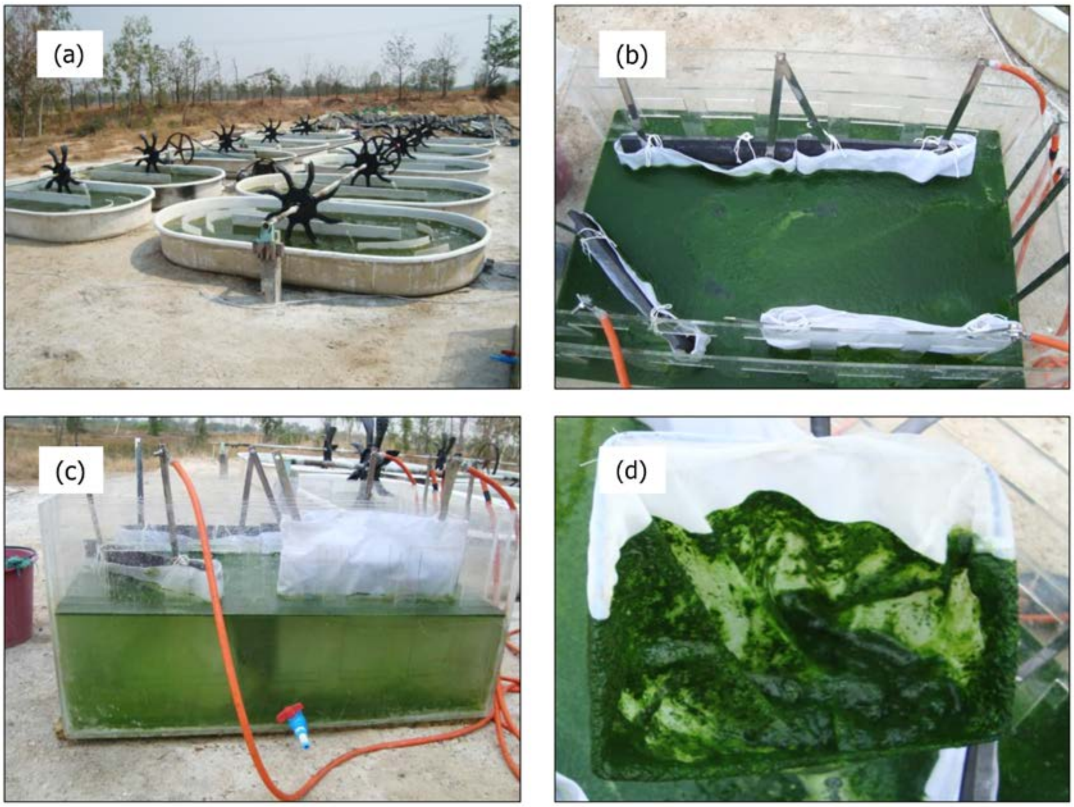

Figure 7. The microalgae harvesting in a commercial scale using a combination of electrode types. (a) The microalgae *Dunaliella salina* was cultured with outdoor conditions in raceway tanks. (b) During the harvesting, the cells were lifted at the top surface while (c) the medium below was clear. (d) The concentrated microalgae at the top surface was collected by using a colander.

Conclusion

We have presented an investigation of electro-flotation microalgae harvesting using different electrodes. When an electrical current was applied, the single-cell microalgae *Dunaliella salina* were induced to form larger clusters those subsequently were lifted by small bubbles to the top surface as a foam layer. While a graphite plate was used as the anode, all cathodes (stainless steel plate, perforated stainless steel plate and graphite plate) gave a similar harvesting efficiency as well as energy consumption. For a given electrical current, the energy consumption was reduced by setting the graphite anode close to the cathode while the harvesting efficiency was not altered. For a given setup of electrodes, an increasing of electrical current resulted in a reduction of the harvesting time and also an increment of the final harvesting efficiency; however, more energy was consumed. Finally, a large scale harvesting using a combination of graphite anode and stainless steel cathode, while metal released from the anode was avoided, was demonstrated for the microalgae *Dunaliella salina* cultured with outdoor conditions in a salt production company located in Nakhon Ratchasima, Thailand.

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