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Energetic and economic assessment of electrocoagulation-flocculation process for separation of microalgae biomass

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Abstract

The high energy input for harvesting biomass makes current commercial microalgae biodiesel production economically unfeasible. In this study, the effect of current intensity, electrode gap, application time, stirring speed and electrode material on the recovery efficiency, energy consumption, amount of electrode dissolution and operation costs was investigated in batch tests. In addition the optimization of recovery of *Dunaliella salina* microalgae by the electrocoagulation-flocculation (ECF) process was conducted using multifactor response surface methodology (combining categorical with numeric factors) based on the D-optimal design. The results indicated that, maximum microalgae recovery efficiency achieved up to 98.06% with, electrical energy consumption of 2.4 kWh kg^{-1}; electrodes dissolution of 17.117 mmol L^{-1}; electrical energy consumption costs of 0.033 \$ kg^{-1}; electrodes dissolution costs of 0.98 \$ kg^{-1} and the total consumption cost of 1.013 \$ per kg of microalgae biomass. Multiple response optimization for maximizing recovery efficiency, and minimizing energy consumption and electrodes dissolution showed 93.84% recovery efficiency with electrical energy consumption of 0.3 kWh kg^{-1}; electrodes dissolution of 5.3 mmol L^{-1}; electrical energy consumption costs of 0.004 \$ kg^{-1}; electrodes dissolution costs of 0.28 \$ kg^{-1} and the total operation cost of 0.284 \$ per kg of microalgae biomass with aluminum as electrode material. Results of the prediction models were validated through laboratory scale batch experiments.

Key words: ***Energy consumption, Electrode dissolution, Electrocoagulation-flocculation, Recovery efficiency, Microalgae, Separation***

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Introduction

Biodiesel is one of the new types of renewable energy which is considered to be a possible substitute of conventional diesel because of its biodegradable nature, non-toxic, renewable and reduced emission level of CO, SO2 [15, 18, 23]. Nowadays microalgae are focused by many researchers all over world for its source of biodiesel-convertible lipids. They are a group of diverse photosynthetic organisms that can accumulate substantial amounts of lipids-up to 20–50% of dry cell weight in certain species [16, 19, 26]. Moreover, microalgae have advantages of high growth rate over traditional biodiesel feedstock (able to double their biomass within a period of 24 h) [2, 39]. The microalgae size is very small (i.e., 3–30 μm in diameter) and possesses similar density to water [43, 45]. Hence the efficient separation of cells from culture broth as well as to maintain their viability is found to be difficult in large scale production of microalgae. Moreover, harvesting of biomass represents one of the significant cost factors in the production of biodiesel. Therefore, microalgae harvesting process became a challenging task and Different studies showed that the harvesting cost of algal production in open ponds accounts for more than 20–30% of the total cost of biodiesel production [37]. Therefore, it is necessary to develop effective and economic technologies for harvesting process. There are currently several harvesting methods, including mechanical, electrical, biological and chemical based. In mechanical based methods, microalgae cells are harvested by mechanical external forces, such as centrifugation, filtration, sedimentation, dissolved air flotation and usage of attached algae biofilms and ultrafiltration membranes [41]. Microalgae can easily be flocculated using metal coagulants such as Fe^{3+} or Al^{3+} salts [1, 6, 34]. In wastewater treatment, electrocoagulation (EC) has been proposed as an alternative for chemical coagulants [28].

Electrocoagulation of microalgae can be achieved by passing an electrical current between two electrodes; a sacrificial metal anode (most commonly aluminum or iron) and a cathode. Half-reactions occur at each electrode and the dominate anode and cathode half reactions are shown in Eq. (1-3).

Anode half reaction:

$$M_{(s)} \rightarrow M_{(aq)}^{n+} + ne^- \quad (1)$$

$$2H_2O_{(l)} \rightarrow O_{2(g)} + 4H_{(aq)}^{+} + 4e^- \quad (2)$$

Cathode half reaction:

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2(g)} + 2OH^- \quad (3)$$

Where *M* is the metal anode, *n* is the charge of the metal ion.

Hydrogen gas is generated at the cathode and the sacrificial anode releases cations which can destabilize microalgae by reducing or neutralizing the negative surface charge. The destabilized microalgae can then flocculate. Depending on the design of the system the flocs once formed may sink to the bottom of the vessel, or attach to the hydrogen bubbles produced at the cathode and float to the surface Electro-flocculation has an advantage over inorganic (chemical) flocculation as whilst metal cations are released from the anode, no anions are introduced to the growth media and less 'coagulant' may also be required [42]. The major factors which affect the electro flocculation of microalgae include voltage, current, residence time, electrode materials and system design. High biomass recoveries are possible, with biomass recovery up to 99 and 98% being observed under laboratory conditions for *Tetraselmis sp*.and *Chlorococcum sp.,* respectively [40]. Some studies have investigated the use of ECF for removal of microalgae from drinking or wastewater [4, 5, 14, 25, 35].The greatest issues surrounding the use of electro flocculation is the contamination of the recovered biomass and growth media with metal salts from the sacrificial anode, the high cost of anode replacement and the formation of an oxide layer on the cathode. While the energy consumed during electro flocculation can be calculated based on the applied current, voltage, residence time and volume of growth medium, the reported energy consumptions vary significantly and depend on the system design and operational parameters. The initial biomass concentration, mode of biomass recovery (i.e. flotation or sedimentation), percent biomass recovery and the salinity of the media have a significant bearing on the operational cost. Freshwater systems generally have a higher energy demand over saline systems as the electrical resistance in freshwater systems is higher and a higher voltage is required in order to achieve the same current [32].

As stated earlier, the success of microalgae biofuels depends very much on the economy of production, but available literatures on microalgae recovery by electrocoagulation-flocculation have only discussed the energy consumption with the cost of metal dissolution neglected. The main goal of this study was to demonstrate the proof of principle for harvesting of *Dunaliella salina* microalgae from the culture medium using electrocoagulation–flocculation. Specific goals were (1) to study the influence of several important variables on the efficiency of the ECF process, (2) to survey the electricity demand of the ECF process, (3) to evaluate electrode dissolution and metals released from the sacrificial anode, (4) to estimate the cost of energy consumption and electrode dissolution for such a process; subsequently, by incorporating

these major costs, determine the cost of microalgae harvesting on a $\$kg^{-1}$ basis. Response surface methodology (RSM) was used to design the experiments, to perform statistical analysis and to determine the optimum conditions.

Materials and methods

Microalgae growth condition

The ECF recovery studies were performed using the native species of *Dunaliell salina* microalgae of *Urmia Lake* which is a halophyte species. The starting culture was obtained from the collection of microalgae in Biotechnology Research Center, Tabriz. For the cultivation of Dunaliella salina was prepared Johnson medium in sterile conditions (Johnson et al., 1968). The culture medium was included: Nacl, 87.75 g; $MgCl_2.6H_2O$, 9.8 g; $CaCl_2.2H_2O$, 0.53 g; Na_2SO_4, 3.2 g; SO_4 K_2, 0.85 g; Tris-Base, 12.11g; KNO_3 (1M/L), 5cc; and the second medium culture was included 5 ml for per liter of trace elements, Including: $CoCl_2.6H_2O$, 0.004g; $MnCl_2.4H_2O$, 0.00072; $NaMoO_4.2H_2O$, 0.00025 g; Na_2EDTA, 4.57 g; $FeCl_3.6H_2O$, 1.259 g; $CuSO_4.7$ H_2O, 0.0104 g; $ZnSO_4.7H_2O$, 0.017 g; and the second medium culture was included 5 ml for per liter of KH_2PO_4 (100mM). All chemicals used in this study were purchased from Merck Company. Primary cultivation was performed in 250 ml Erlenmeyer flask with 10% of Stock microalgae insemination. From one germinator device (Grouc Company Model, 400 GC, Iran) which is equipped with light, temperature, humidity and ventilation control systems was used as the culture room. The algal culture has a pH of 7.5 and the temperature was kept constant at 25°C and fluorescent light with an intensity of, 3500 lux.

Electrocoagulation-flocculation experiment

All the ECF experiments were carried out at room temperature in a batch reactor with an effective volume of 250 mm which is made of Pyrex glass. Two electrodes with dimensions of 5×5 cm and a surface area of 25 cm^2 with distance 2 cm from bottom of the reactor in vertically state and in different stages were placed inside the reactor with distance of 1, 2 and 3 cm. The electrode material were used from the aluminum electrodes with 99.5% purity (Al-1050) and iron electrodes (ST 37-2). The impurities level in the surface of the electrodes before using in the reactor is cleaned by sanding, and then the electrodes were put for 15 min in a solution of dilute hydrochloric acid (15 wt %), then were washed with distilled water after brushing, and were dried in the oven-device. The Voltage and required current in the reactor were provided with a digital DC power supply (Afzar Azma, Model JPS-403D). Order to establish a uniform mixture within the reactor, was used from a magnetic stirrer (Fig. 1).

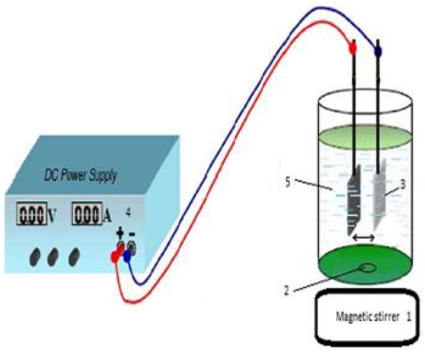


Fig. 1. Schematic diagram of experimental set-up. (1) magnetic stirrer; (2) magnetic; (3) anode and cathode electrodes; (4) DC Power supply; (5) microalgae culture.

With the growth of microalgae, the cultivation concentrations are increased and after 10 days the logarithmic growths of microalgae have reached a maximum and after this microalgae growth stage were stopped and their density is practically remained constant and harvesting time was started. At this stage by counting the number of cells by Hemacytometer (Lam-neubauer) and measuring optical density by spectrophotometer (JENWAY, 6305) at a wavelength 550 and 680 nm was determined the initial concentration rate in the culture medium based on cells/L. Then the culture medium was transferred into the reactor.

According to the experimental design levels in Table 1, Forty one batch tests were conducted in 250 mL glass bottles. At the end of tests time, was given 30 minutes for the sedimentation of samples and then counting the number of cells was performed by Lam-Neubauer and optical density of by spectrophotometer device.

The recovery efficiency was subsequently calculated as [14]:

Microalgae recovery efficiency (%) $\eta_a = \frac{C_o - C_t}{C_o} \times 100$ (4)

where C_o is the cell density of microalgae (cell/L) in the initial culture medium, and C_t is cell density of microalgae (cell/L) after the electro-flocculation.

Table 1. Independent variables and experimental design Levels

Independent variables	Unit	Level	
		-1	+1
Current intensity	*mA*	300	1000
Electrode gap	*cm*	1	3
Time	*min*	5	20
Stirring speed	*rpm*	0	400
Electrode material	*material*	Aluminum	Iron

Calculation of the energy consumption, material dissolution and operation cost

Electrical energy consumption and current efficiency are very important economical parameters in EFC process like all other electrolytic processes. The value of energy consumption as a function of treated solution volume was also calculated in different current intensity for batch tests. The electrical energy consumption EEC (in kWh kg^{-1}) of recovered microalgae) was calculated [42] as:

$$EEC = \frac{UIt}{1000V\eta_a C_i} \ (kWh/kg) \quad (5)$$

where U is the voltage (V), I the current (A), t the time of the ECF treatment (h), V the volume of the microalgae solution treated (m^3), η_a the microalgae recovery efficiency, and C_i the initial microalgae biomass concentration (kg m^{-3}).

The current efficiency (ϕ) of EFC process was calculated Eq. (6). This calculation was based on the comparison of experimental weight loss of electrodes (ΔM_{exp}) during EC process with theoretical amount of electrode dissolution (ΔM_{theo}) according to the Faraday's law Eq. (7). [3, 9, 27, 36].

$$\varphi = \frac{\Delta M_{exp}}{\Delta M_{theo}} \quad (6)$$

$$\Delta M_{theo} = \frac{MIt}{nF} \quad (7)$$

where M is the molecular weight of the electrode material (g mol^{-1}), n the number of electron moles and F is the Faraday constant (F = 96487 C mol^{-1}).

The operating cost includes material cost (mainly electrodes), utility cost (mainly electrical energy), as well as labor, maintenance and other fixed costs [21]. In this preliminary economic study, energy and electrode material costs are taken into account as major cost items in the calculation of the operating cost as kWh per kg of microalgae recovery.

$$Cost_{material\ electrode} = m_{exp} \times A_{fee} \quad (8)$$

$$Cost_{EEC} = EEC_m \times B_{fee} \quad (9)$$

$$Operating\ Cost_{\text{total}} = Cost_{material\ electrode} + Cost_{EEC} \quad (10)$$

where $Cost_{EEC}$ and $Cost_{material\ electrode}$ are consumption quantities per kg of microalgae recovery, which are obtained experimentally. Given Iranian market in march 2020, unit prices A_{fee} and B_{fee} are as follows: A_{fee}, Aluminum and Iron electrode price was 2.03 and 0.625 US$ kg^{-1} respectively; B_{fee}, electrical energy price was 0.014 US$ per kWh.

Experimental design

Experimental design of the EFC process for microalgae recovery was carried out by using the Response surface methodology with combining categorical and numeric factors based on the D-optimal design.

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response. The purpose of applying this method is variance analysis model and the optimization of the response (response variable). The first step is finding a suitable approximation function between a set of independent and

response variables. This approximation function is usually a polynomial function of independent variables, such as which is used in the Eq. (11) [30].

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum\sum_{i<j} \beta_{ij} x_i x_j + \varepsilon \quad (11)$$

where y, represents the value of response; $\beta_0, \beta_i, \beta_{ii}, \beta_{ij}$ regression coefficients; k the number of independent variables; and ε the amount of errors.

In the response surface method in order to estimate the parameters of the function, the independent variables must be have coded values these can be coded with Eq. (12) [30].

$$x_i = \frac{X_i - X_o}{\Delta X_i} \quad (12)$$

where x_i is the coded value of the *i*th test variable, X_i is the actual value of the *i*th independent variable, X_o is the actual center point value of the *i*th independent variable, and ΔX_i is the step size of the *i*th independent variable.

In this study, recovery efficiency, energy consumption, electrode dissolution were chosen as the response variable (y_i), while Current intensity (X_1), the electrode gap (X_2), time for electrocoagulation-flocculation (X_3), stirring speed (X_4), material of the electrodes (X_5) were chosen as fife independent variables.

Optimization procedure

Optimization of the EFC for microalgae recovery was carried out by using the Design-Expert program 8.0.7.1 trial version. The optimization module in Design-Expert searches for a combination of factor levels that simultaneously satisfy the requirements placed on each of the responses and factors. According to the optimization step of the program, the desired goal for each variable (current intensity, electrode gap, application time, stirring speed and electrode material) and responses (microalgae recovery efficiency, energy consumption, electrode dissolution) should be chosen. The possible goals are: maximize, minimize, target, within range, none (for responses only) and set to an exact value (factors only). The goals are then combined into an overall desirability function. The program seeks to maximize this function. The goal seeking begins at a random starting point and proceeds up the steepest slope to a maximum. There may be two or more maximums because of curvature in the response surfaces and their combination into the desirability function. By starting from several points in the design space chances improve for finding the "best" local maximum [10].
In optimization, the desired goals for the responses were chosen to maximize the recovery efficiency, minimize the energy consumption, minimize the electrodes dissolution and the variables of applied current intensity, electrode gap, application time, stirring speed and electrode material were selected to be within range.

Results and discussion

This study in following previous research Mohammad-ghasemnejadmaleki et al. [24] was conducted in order to evaluation of effect of five independent variables including: current intensity, electrode gap, ECF time, stirring speed and electrode material on the energy consumption, electrode dissolution and operation cost as a response variables in electrocoagulation-flocculation process for recovery *Dunaliella salina* microalgae. In the previous study had been discussed the effect independent variables on the recovery efficiency of microalgae from the culture medium. To easily access the summary of the results are presented in the following.

Effect of current intensity, electrode gap, time, stirring speed and electrode material on recovery efficiency

In optimizing a response surface, an adequate fit of the model should be obtained to avoid poor or ambiguous results [31]. This is important to ensure the adequacy of the employed model. The quadratic model was used to fit the recovery efficiency of microalgae data obtained from each batch test using Eq. (4). The test results showed that lack of fit test of quadratic polynomial is not significant. Also the coefficients of determination (R^2), adjusted and predicted were respectively more than 0.98, 0.96 and 0.90, which indicated that the quadratic model could describe the recovery efficiency of microalgae in the batch tests of this study successfully (table 2).
After eliminating insignificant phrases from quadratic function, Eq. (13) was obtained by using Eq. (11) to fit the experimental data of recovery efficiency.

$$y(\text{recovery efficiency}) = 74.3 + 11.61x_1 - 3.71x_2 + 9.98x_3 + 2.24x_4 - 8.18x_5 - 6.50x_1x_3 + 2.65x_1x_5 - 1.99x_2x_3 - 3.93x_2^2 - 3.49x_4^2 \quad (13)$$

where x_1 ،x_2 ،x_3 ،x_4 *and* x_5 respectively are coded values of current intensity (mA), distance between the electrodes (cm), time (min), and speed of stirring (rpm) and the electrode type.

ANOVA of the fitting model (Table 3) showed that the fitting model was highly significant ($p<0.01$), while the lack of fit was not significant ($p > 0.05$). Comparing the observed values (actual) with predicted values in Fig. 2a, and these observations has been mentioned a very good correlation between the obtained results with experimental method in laboratory and the predicted values with the statistical method, is according to Eq.(13).
Coefficient of determination (R^2) was 0.971, which can explain 97.1% variability of the response variable. All these indicated that Eq. (13) could describe the effect of current intensity, electrode gap, ECF time, stirring speed and electrode material on the recovery efficiency of this study very well.

Table 2. Lack of fit tests and model Summary Statistics for selection of an appropriate model in studying the effect of five independent variables on recovery efficiency, energy consumption, electrode dissolution in ECF

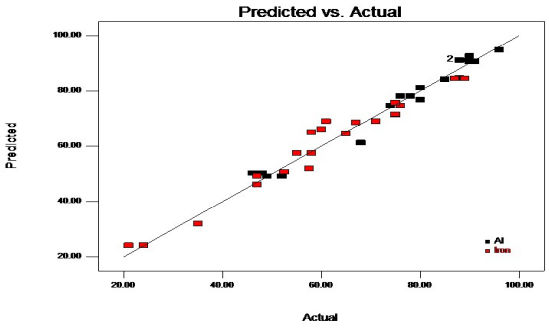
Source	df	Mean Square	F Value	p-value Prob > F	R-Squared	Adjusted R-Squared	Predicted R-Squared
Recovery efficiency:							
Linear	26	74.8069	9.61803	0.0007	0.859	0.8388	0.8019
2FI	16	27.81036	3.575618	0.0289	0.964	0.9423	0.9073
Quadratic	12	15.61737	2.007947	0.1506*	0.982	0.9657	0.9066
Cubic	0				0.9951	0.9782	+
Pure Error	9	7.777778					
Energy consumption:							
Linear	25	0.026	31.65	<0.0001	0.9083	0.8948	0.8658
2FI	15	3.99E-03	4.89	0.0106	0.9906	0.9847	0.9718
Quadratic	11	2.69E-03	3.29	0.042*	0.9948	0.9899	0.9677
Cubic	0				0.9990	0.9955	+
Pure Error	9	8.17E-04					
Electrode dissolution:							
Linear	26	0.096	14.93	0.0001	0.9474	0.9399	0.9246
2FI	16	0.017	2.64	0.0716	0.9932	0.9892	0.9755
Quadratic	12	9.04E-04	0.14	0.9987*	0.9986	0.9973	0.9946
Cubic	0	Aliased			0.9988	0.9947	+
Pure Error	9	6.44E-03					

*Suggested

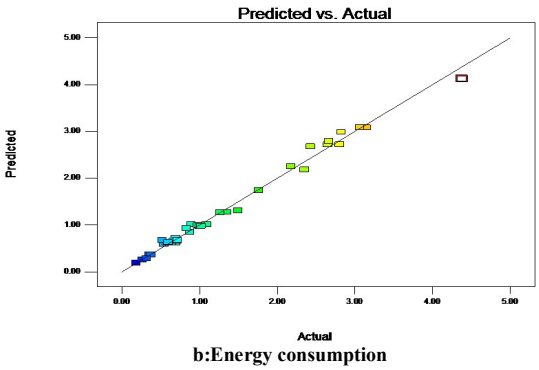
ANOVA of the fitting model (Table 3) also showed that the linear effect of independent variables, current intensity, reaction time, electrode gap, stirring speed and electrode material, and interactive effect between current and time, current and electrode material, electrode gap and time, and quadratic effect of electrode gap on the recovery efficiency were significant ($p < 0.05$), indicating that these terms had great impact on recovery efficiency. The fact that there was a significant interaction between the current intensity and time of ECF on the recovery efficiency is significant, means that the simple effect of current intensity at different times is not the same response, Thus, between the current intensity and the duration of ECF, it is possible the existence of interactive effect. The coefficient of interaction effect between current intensity and the time of EDF indicated based on the Eq. (13), that interactive effect between these two factors has a negative effect on the recovery efficiency.
Subsequently, the maximum microalgae recovery efficiency of 98.06% was estimated from Eq. (13) at the current intensity of 999 mA, the time of 20 min, the electrode gap of 1.39 cm, the stirring speed of 222 rpm and aluminum as electrode material.
Fig. 3, shows the response surface plot and corresponding contour plot based on Eq. (13) with one variable being kept constant at its optimum level and varying the other two variables within the experimental range.
In Fig. 3, a_1 and a_2, $b_1$and b_2 were plotted with current intensity, electrode gap, ECF time, stirring speed and electrode material being kept constant at 999 mA, 20 min, 1.39 cm, 222 rpm and aluminum respectively. As shown in Fig. 3, in the design boundary, each response surface plot had a clear peak and the corresponding contour plot had a clear highest point, which means that the maximum microalgae recovery efficiency could be achieved inside the design boundary.
Similar reports are published in this issue by [13, 20, 33, 43, 44]. The results showed that aluminum electrodes on the recovery of microalgae from the culture medium are more efficient than iron electrodes. Moreover with increasing the electric current intensity and time for electro-flocculation, or reduce the distance between the electrodes, the recovery efficiency has increased significantly. Also by increasing stirrer speed from 0 to 200 rpm the amount of recovery efficiency is increased, and by increasing stirrer speed from 200 to 400 rpm the amount of recovery efficiency has decreased. Previous studies on ECF for other applications have also demonstrated that stirring can improve the coagulation–flocculation efficiency [7]. Stirring improves the recovery efficiency by enhancing contact rates between the coagulants and the microalgae cells [29].

Table 3. ANOVA of the fitting model for recovery efficiency

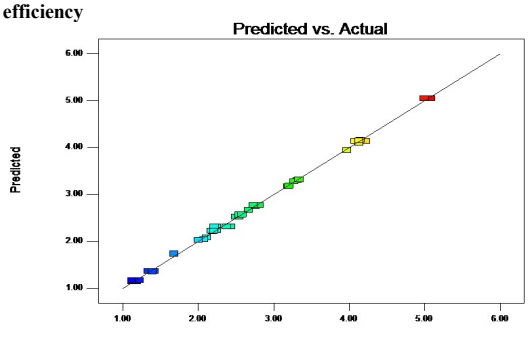
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	13875.51	10	1387.55	101.04	< 0.0001
x_1	4238.5	1	4238.5	308.66	< 0.0001
x_2	408.14	1	408.14	29.72	< 0.0001
x_3	2926.59	1	2926.59	213.12	< 0.0001
x_4	159.38	1	159.38	11.61	0.0019
x_5	2627.33	1	2627.33	191.33	< 0.0001
$x_1 x_3$	1041.06	1	1041.06	75.81	< 0.0001
$x_1 x_5$	219.31	1	219.31	15.97	0.0004
$x_2 x_3$	100.06	1	100.06	7.29	0.0113
x_2^2	87.19	1	87.19	6.35	0.0173
x_4^2	51.22	1	51.22	3.73	0.0629
Residual	411.96	30	13.73		
Lack of Fit	341.96	21	16.28	2.09	0.1266
Pure Error	70	9	7.78		
Cor Total	14287.48	40			
C.V. %	5.45				
R^2	0.9712				
Adj R^2	0.9616				
Pred R^2	0.9469				



a: Recovery efficiency



b:Energy consumption



c:Electrode dissolution

Fig. 2. Comparing the predicted values with actual (observed)

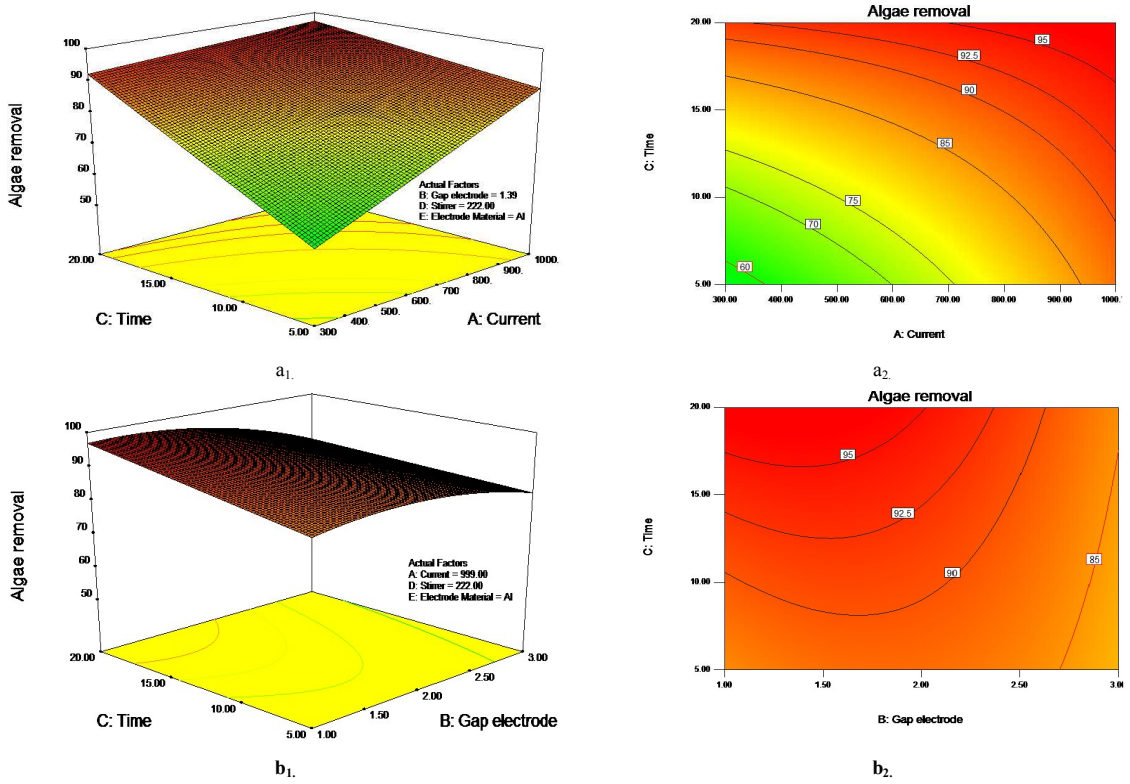


Fig. 3. Response surface plot and corresponding contour plot for recovery of microalgae

Effect of current intensity, electrode gap, time, stirring speed and electrode material on energy consumption

The experimental results indicated that similar microalgae recovery efficiencies could be obtained by applying a high current density during a short time as by applying a low current density during a longer time. From an energy consumption point of view, it is unclear which strategy is best. Therefore, for the experimental design, the global energy consumption, expressed as kWh/kg^{-1} dry weight microalgae biomass recovered during the ECF process was calculated using Equation (5) for each sampling. For each ECF run, a point in time could be identified at which the energy consumption per unit of microalgae biomass recovered was minimal. This point in time generally corresponded to the time at which recovery efficiency reached the saturation phase.

In this study, the square root transformation as suggested by Box-Cox plot of the energy consumption was performed. Quadratic model was found to be the best with insignificant lack of fit. Also the coefficients of determination (R2), adjusted and predicted were respectively more than 0.994, 0.989 and 0.9677, which indicated that the quadratic model could describe the energy consumption in the batch tests of this study successfully (table 2). After eliminating insignificant phrases from quadratic function, Standardized equation for energy consumption is represented by Eq. (14)

$$Sqrt(Electrical\ Energy\ Con.) = 1.07 + 0.35x_1 + 0.088x_2 + 0.26x_3 - 0.027x_4 + 0.1x_5 + 0.14x_1x_3 - 0.0049x_1x_5 + 0.022x_2x_5 + 0.064x_2^2 - 0.097x_3^2 + 0.046x_4^2 \quad (14)$$

where x_1 ،x_2 ،x_3 ،x_4 *and* x_5 respectively are coded values of current intensity (mA), distance between the electrodes (cm), time (min), and speed of stirring (rpm) and the electrode type.

The ANOVA table for reduced quadratic model for energy consumption (Table 4) indicated that the model is significant at $p \leq 0.0001$, and its Lack of fit ($p \leq 0.0652$) is not significant. Responses predicted by the reduced quadratic model (Eq. 14) are given in Fig. 2b, the reduced quadratic model gave R^2 equal to 0.992, which can explain 99.92% variability of the response variable, thus the model is highly efficient in response predictions. All these indicated that Eq. (14) could describe the effect of current intensity, electrode gap, ECF time, stirring speed and electrode material on the energy consumption of this study very well.

ANOVA of the fitting model (Table 2) also showed that that the linear effect of independent variables, current intensity, reaction time, electrode gap, stirring speed and electrode material, and interactive effect between current intensity and time, electrode gap and electrode material, and quadratic effect of electrode gap, reaction time and stirring speed on the energy consumption were significant ($p < 0.05$), indicating that these terms had great impact on recovery efficiency. The fact that there was a significant interaction between the current intensity and time of ECF on energy consumption is significant, means that the simple effect of current intensity at different times is not the same response, Thus, between the current intensity and the duration of ECF, it is possible the existence of interactive effect. The coefficient of interaction effect between current intensity and the time of EDF indicated based on the Eq. (14), that interactive effect between these two factors has a plus effect on the energy consumption.

Table 4. ANOVA of the fitting model for energy consumption

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	7.08	9	0.79	443.3	< 0.0001
x_1	3.74	1	3.74	2110.03	< 0.0001
x_2	0.22	1	0.22	126.26	< 0.0001
x_3	1.9	1	1.9	1073.15	< 0.0001
x_4	0.024	1	0.024	13.49	0.0009
x_5	0.4	1	0.4	223.29	< 0.0001
x_1x_3	0.51	1	0.51	285.28	< 0.0001
x_2x_5	0.014	1	0.014	8.07	0.008
X_2^2	0.017	1	0.017	9.38	0.0046
X_3^2	0.038	1	0.038	21.41	< 0.0001
X_4^2	0.013	1	0.013	6.70	0.0147
Residual	0.053	30	1.78E-03		
Lack of Fit	0.046	21	2.19E-03	2.67	0.0652
Pure Error	7.36E-03	9	8.17E-04		
Total	7.13	39			
C.V. %	4.02				
R^2	0.9925				
Adj R^2	0.9903				
Pred R^2	0.9872				

Running the reduced quadratic model for minimizing energy consumption gave optimal points as the current intensity of 307 mA, the time of 5.09 min, the electrode gap of 1.07 cm, the stirring speed of 39.98 rpm and aluminum as electrode material. These parameters predicted energy consumption of 0.2 kWhkg^{-1} and recovery efficiency of 51.72%. Fig. 4, shows the response surface plot and corresponding contour plot based on Eq. (14), with one variable being kept constant at its optimum level and varying the other two variables within the experimental range. In Fig. 4, a_1 and a_2 were plotted with current intensity, electrode gap, ECF time, stirring speed and electrode material being kept constant at 307 mA, 5.09 min, 1.07 cm, 39.98 rpm and aluminum respectively.

As shown in Fig. 4, a_1 and a_2 in the design boundary, each response surface plot had a clear peak and the corresponding contour plot had a clear lowest point, which means that the minimum energy consumption could be achieved inside the design boundary.

In spite of more effective recovery of algae with the higher current density, increasing the current intensity could also lead to the increase of applied potential, which resulted in the sharp increase of the energy consumption of the ECF system Eq. (5).Therefore, it might be important to optimize the current input for the ECF process, to avoid extra- higher energy consumption.

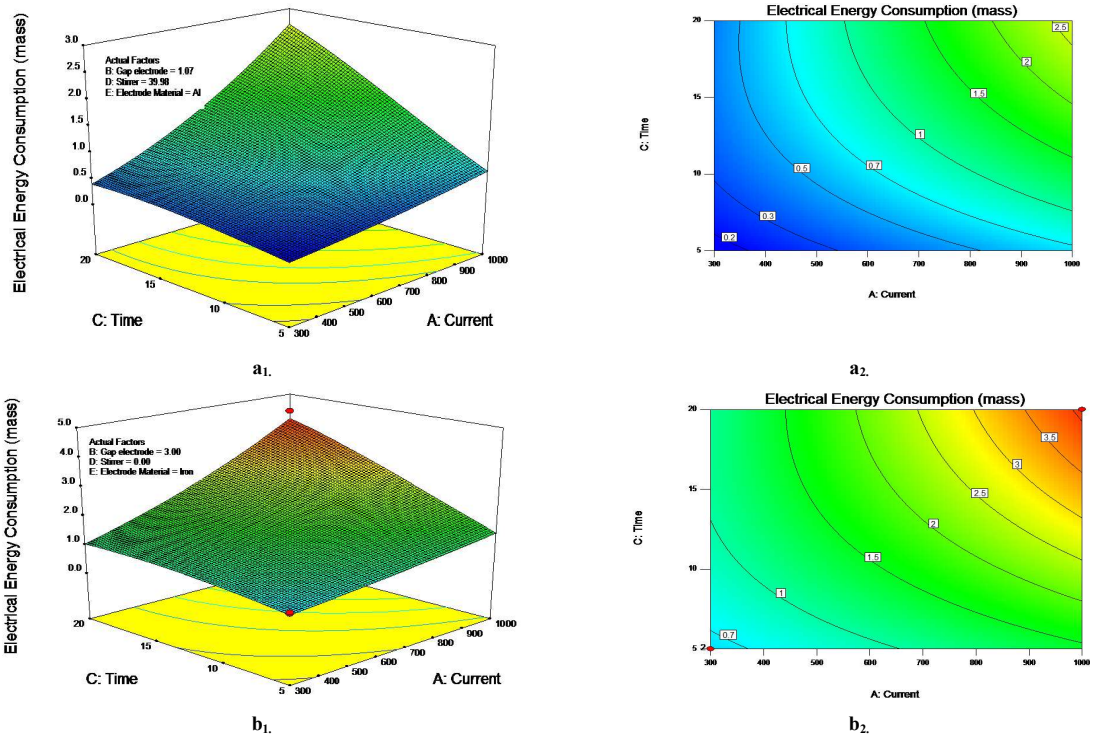
The maximum energy consumption was 4.1KWh kg^{-1} at the current intensity of 1000 mA, the time of 20 min, the electrode gap of 3 cm, the stirring speed of 0 rpm and Iron as electrode material (Fig. 4, b_1 and b_2).

Also the electrode gap was increased high potential energy was required for transferring the electrons between electrodes. Therefore, the energy consumption was increased. Increasing the formation of bobbles around the electrodes by decreasing the electrode gap under 1 cm caused the increase in resistance between the electrodes, thereby increasing energy consumption.

Regarding high cationic property of Fe^{3+} and Al^{3+}, these two metals were compared to determine the effect of electrode type on energy consumption. The Fe electrodes consume more electrical energy than Al electrodes.

Similar report are published in this issue by Vandamme et al. [42] and Pandey et al.[33]. Their research showed that, the minimum value of the power consumption was 2.1 kWh kg^{-1} of biomass harvested for Chlorella vulgaris and 0.2 kWh kg^{-1} of biomass harvested for *Phaeodactylum tricornutum*, at a current density of 1.5 and 0.6 mA cm^{-2}, respectively. These data confirm the low power requirements of ECF, especially for the marine species. The lower power

consumption needed for the marine species is mainly due to the higher conductivity of the marine medium when compared to the freshwater medium, which results in a higher efficiency of the electrolytic release of aluminum from the anode [20], but other phenomena could also play a role here. Vandamme et al. [42] reported that chloride ions present in seawater attack the aluminum oxide layer formed on the surface of the anode, thereby enhancing the release of aluminum from the anode aluminum oxide layer formed on the surface of the anode, thereby enhancing the release of aluminum from the anode.



a1. **a2.**

b1. **b2.**

Fig. 4. Response surface plot and corresponding contour plot for energy consumption

Effect of current intensity, electrode gap, time, stirring speed and electrode material on Amount of electrode dissolution

In this study also was investigated the amount of electrodes dissolution during the course of the ECF process. As predicted by Faraday's law Eq. (7), the theoretical Amount of electrode dissolution increased with time and with current density. The experimental amount of electrode dissolution (weight loss of electrodes in mmol l^{-1}) was calculated from the weight difference of electrodes before and after each the batch tests.

The square root transformation as suggested by Box-Cox plot of the amount of electrode dissolution was performed. Quadratic model was found to be the best with insignificant lack of fit. Subjecting the model to ANOVA indicated that only 11of the 19 model terms are significant (table). Also the coefficients of determination (R2), adjusted and predicted were respectively more than 0.998, 0.997 and 0.994, which indicated that the quadratic model could describe the amount of electrode dissolution in the batch tests of this study successfully (table 2). After eliminating insignificant phrases from quadratic function, Standardized equation for amount of electrode dissolution is represented by Eq. (15)

$$Sqrt(Electrode\ dissolution) = 2.93 + 0.78x_1 + (5.985E-003)x_2 + 0.89x_3 + (5.628E-003)x_4 + 0.25x_5 + 0.25x_1x_3 + 0.088x_1x_5 - 0.025x_2x_5 + 0.086x_3x_5 - 0.088x_1^2 - 0.16x_3^2 \quad (15)$$

where x_1 ،x_2 ،x_3 ،x_4 *and* x_5 respectively are coded values of current intensity (mA), distance between the electrodes (cm), time (min), and speed of stirring (rpm) and the electrode type.

The ANOVA table for reduced quadratic model for amount of electrode dissolution (Table 5) indicated that the model is significant at $p \leq 0.0001$, and its Lack of fit ($p \leq 0.999$) is not significant. Responses predicted by the reduced quadratic model (Eq. 15) are given in Fig. 2c, the reduced quadratic model gave R^2 equal to 0.998, which can explain 99.8%

variability of the response variable, thus the model is highly efficient in response predictions. All these indicated that Eq. (15) could describe the effect of current intensity, electrode gap, ECF time, stirring speed and electrode material on the amount of electrode dissolution of this study very well.

ANOVA of the fitting model (Table 5) also showed that that the linear effect of independent variables, current intensity, reaction time and electrode material, and interactive effect between current intensity and time, current intensity and electrode material, electrode gap and stirring speed, time and electrode material, and quadratic effect of current intensity and reaction time on the amount of electrode dissolution were significant ($p < 0.05$), indicating that these terms had great impact on amount of electrode dissolution. The fact that there was a significant interaction between the current intensity and time of ECF on amount of electrode dissolution is significant, means that the simple effect of current intensity at different times is not the same response, Thus, between the current intensity and the duration of ECF, it is possible the existence of interactive effect. However, the liner effect of electrode gap and stirring speed was not significant ($p>0.05$), indicating that these terms had little impact on amount of electrode dissolution.

Table 5. ANOVA of the fitting model for electrode dissolution

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	48.57	11	4.42	1696.12	< 0.0001
x1	19.34	1	19.34	7428.73	< 0.0001
x2	1.07E-03	1	1.07E-03	0.41	0.5271
x3	22.73	1	22.73	8732.08	< 0.0001
x4	9.95E-04	1	9.95E-04	0.38	0.5413
x5	2.39	1	2.39	916.66	< 0.0001
x_1x_3	1.63	1	1.63	625.89	< 0.0001
x_1x_3	0.23	1	0.23	89.01	< 0.0001
x_1x_3	0.017	1	0.017	6.57	0.0158
x_1x_3	0.2	1	0.2	77.83	< 0.0001
x_1^2	0.04	1	0.04	15.5	0.0005
x_3^2	0.15	1	0.15	57.82	< 0.0001
Residual	0.075	29	2.60E-03		
Lack of Fit	0.018	20	8.77E-04	0.14	0.9999
Pure Error	0.058	9	6.44E-03		
Cor Total	48.65	40			
C.V. %	1.87				
R^2	0.9984				
Adj R^2	0.9979				
Pred R^2	0.9968				

Running the reduced quadratic model for minimizing amount of electrode dissolution gave optimal points as the current intensity of 300 mA, the time of 5 min, the electrode gap of 1 cm, the stirring speed of nothing and aluminum as electrode material. These parameters predicted amount of electrode dissolution of 1.31mmolL^{-1} and recovery efficiency of 49%.

Fig. 5, a_1 and a_2 shows the response surface plot and corresponding contour plot based on Eq. (15), with one variable being kept constant at its optimum level and varying the other two variables within the experimental range. In Fig. 1, a_1 and a_2, $b_1$and b_2, and $c_1$and c_2 were plotted with current intensity, electrode gap, ECF time, stirring speed and electrode material being kept constant at 300 mA, 5 min, 1 cm, nil rpm and aluminum respectively. As shown in Fig. 5, a_1 and a_2 in the design boundary, each response surface plot had a clear peak and the corresponding contour plot had a clear lowest point, which means that the minimum amount of electrode dissolution could be achieved inside the design boundary.

The maximum amount of electrode dissolution was 25.488 mmol L^{-1} at the current intensity of 1000 mA, the time of 20 min, the electrode gap of 3 cm, the stirring speed of nil rpm and Iron as electrode material (Fig. 5, b_1 and b_2).

Similar report is published in this issue by Matos et al. [20]. Vandamme et al. [42] reported that the Aluminum content in the recovered microalgae biomass was about twice as high at the higher current density than at the lower current density. For both species, the aluminum content in the microalgae biomass continued to increase after the maximal recovery efficiency was reached, which can be ascribed to continued precipitation of aluminum hydroxides.

The difference in aluminum concentration in the water between the marine and fresh water species are most likely due to differences in the chemical composition of the freshwater and the seawater medium [42]. The seawater medium contains high concentrations of sulphate anions. These sulphate anions are known to facilitate precipitation of aluminum hydroxides [12].

In the experiments described in this research, microalgae were coagulated-flocculated by aluminum hydroxides. This mechanism of coagulation–flocculation is comparable to coagulation–flocculation of microalgae using aluminum salts like alum. According to the literature [38], 80–250 mg L^{-1}alum, corresponding to 7.2–23 mg Al L^{-1} is needed to

coagulate/flocculate a microalgae suspension. For harvesting *Chlorella minutissima*, Papaziet al. [34] used 750 mg L^{-1} alum, which corresponds to 120 mg L^{-1} of aluminum. If we assume that only aluminum oxidation occurred at the anode, we estimated that in the experiments in which the lowest current density was used, only 1.31mmol L^{-1} Al was released in the experiment with optimal condition (minimizing amount of electrode dissolution). This suggests that ECF is more efficient in terms of aluminum consumption than coagulation-flocculation using alum. Similar report is published by Canizares et al. [8] on the use of ECF for treatment of textile waters.

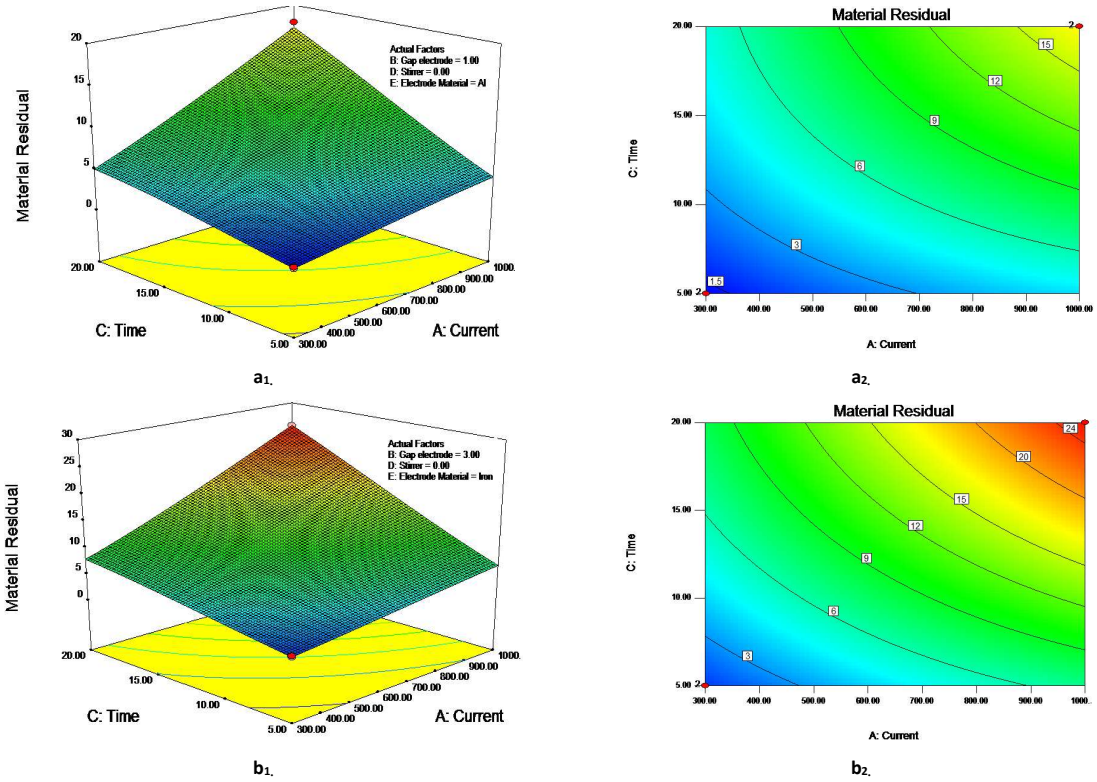


Fig. 5. Response surface plot and corresponding contour plot for electrode dissolution

Multiple response optimization

In Phase-II, model equations for recovery efficiency and energy consumption and amount of electrodes dissolution were simultaneously solved to find the optimal process variables. The relations that were discussed in the previous sections, only for predicting recovery efficiency, energy consumption and electrodes dissolution was tested in different situations, however, these mathematical relationships are not provided information about the optimal amount of each independent variable. In this study, were searched the optimal operating conditions by using numerical optimization techniques. For this purpose at first it is needed to define different scenarios for the optimization model.

Design-Expert software was used for maximizing microalgae recovery efficiency while minimizing energy consumption and electrodes dissolution, and the optimal variables obtained were current intensity of 300 mA, the time of 20 min, the electrode gap of 1.29 cm, the stirring speed of 228 rpm and with aluminum as electrode material, providing 93.84% recovery efficiency and electrical energy consumption of 0.3 kWh kg^{-1}; electrodes dissolution of 5.3 mmol L^{-1}; electrical energy consumption costs of .0.004 \$ kg^{-1}; electrodes dissolution costs of 0.280 \$ kg^{-1} and the total consumption cost of 0.284 \$ per kg of biomass of microalgae (Fig. 6).

The study has shown that, maximum microalgae recovery efficiency can be achieved up to 98.06% and with electrical energy consumption of 2.4 kWh kg^{-1}; electrodes dissolution of 17.117 mmol L^{-1}; electrical energy consumption costs of 0.033 \$ kg^{-1}; electrodes dissolution costs of 0.98 \$ kg^{-1} and the total consumption cost of 1.013 \$ per kg of biomass of

microalgae under condition the current intensity of 999 mA, the time of 20 min, the electrode gap of 1.39 cm, the stirring speed of 222 rpm and with aluminum as electrode material (Fig. ۳).
These analyses clearly indicated that the minimal energy consumption per unit of microalgae biomass recovered is much lower if lower currents intensity are used than when higher current densities are used.
Previous studies, in which ECF was used to remove microalgae from surface waters, have also indicated that the energy consumption to achieve coagulation–flocculation is lower when a lower current density is used [14]. Although a higher current intensity thus leads to a more rapid coagulation-flocculation of the microalgae, the use of a low current intensity is more efficient, from an energy consumption point of view. It should be noted, however, that the use of a low current intensity requires relatively long retention times of the water in the reactor. It is not unusual, however, to use retention times in other applications of ECF [11, 46]. Nevertheless, the retention time should be taken into account when the process is applied at an industrial scale. A long retention time will require a larger reactor to process the same volume of water. A long retention time may also influence the quality of the algal biomass that is harvested.

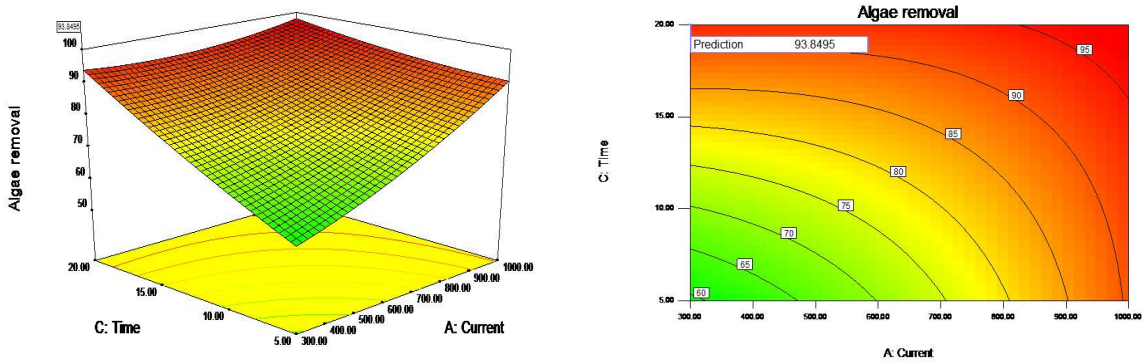


Fig. 6. Response surface plot and corresponding contour plot for optimal condition

(300 mA current intensity, 20min time, 1.29 cm electrode gap, 228 rpm stirring speed and with aluminum)

Conclusions

The success of microalgae biofuels depends very much on the economy of production, but available literatures on microalgae recovery by electrocoagulation-flocculation have only discussed the energy consumption with the cost of metal dissolution neglected. The main goal of this study was to demonstrate the proof of principle for harvesting of *Dunaliella salina* microalgae from the culture medium using electrocoagulation-flocculation. Specific goals were (1) to study the influence of several important variables on the efficiency of the ECF process, (2) to survey the electricity demand of the ECF process, (3) to evaluate electrode dissolution and metals released from the sacrificial anode, (4) to estimate the cost of energy consumption and electrode dissolution for such a process; subsequently, by incorporating these major costs, determine the cost of microalgae harvesting on a $\$kg^{-1}$ basis.
In this study, Response Surface Methodology (RSM) was employed to investigate the effects of different operating conditions on the recovery of *Dunaliella salina* microalgae by the electrocoagulation-flocculation (ECF) process. Combining categorical and numeric factors based on the optimal design was used for the optimization of the ECF process and to evaluate the effects and interactions of process variable: current intensity, electrode gap, ECF time, stirring speed and electrode material on the recovery efficiency, energy consumption, amount of electrode dissolution and operation costs.
The modified quadratic model was used to fit the microalgae recovery efficiency data obtained from each batch test. The experimental data and the predicted data by the models was highly correlated (R^2>0.98). The results indicated that the linear effect of independent variable on recovery efficiency is very statistically significant ($p < 0.01$). The separation efficiency of aluminum electrode in all experiments is significantly higher than iron electrode. Electrical energy consumption and electrodes dissolution in iron electrodes is significantly higher than aluminum electrodes. As a result, the electrical energy consumption costs; electrodes dissolution costs and the total operation cost in electrical coagulation with aluminum electrodes are lower than iron. With increasing the electric current intensity and time of electro-flocculation, or reduce the distance between the electrodes, the recovery efficiency has increased significantly as

according to Faraday's law, electrodes dissolution and electrical energy consumption has increased significantly. Also by increasing stirring from 0 to 200 rpm, the recovery efficiency is increased and then decreased.

The study has shown that, maximum microalgae recovery efficiency can be achieved up to 98.06% and with electrical energy consumption of 2.4 kWh kg^{-1}; electrodes dissolution of 17.117 mmol L^{-1}; electrical energy consumption costs of 0.033 $ kg^{-1}; electrodes dissolution costs of 0.98 $ kg^{-1} and the total consumption cost of 1.013 $ per kg of biomass of microalgae under condition the current intensity of 999 mA, the time of 20 min, the electrode gap of 1.39 cm, the stirring speed of 222 rpm and with aluminum as electrode material.
In optimization, the desired goals for the responses were chosen to maximize the recovery efficiency, minimize the energy consumption, and minimize the electrodes dissolution. The optimal variables obtained were current intensity of 300 mA, the time of 20 min, the electrode gap of 1.29 cm, the stirring speed of 228 rpm and with aluminum as electrode material. The result of optimization indicated 93.84% recovery efficiency and electrical energy consumption of 0.3 kWh kg-1; electrodes dissolution of 5.3 mmol l-1; electrical energy consumption costs of .0.004 $ kg-1; electrodes dissolution costs of 0.28 $ kg-1 and the total operation cost of 0.284 $ per kg of microalgae biomass.
The results of research are introduced using ECF with current intensity of 300 mA, the time of 20 min, the electrode gap of 1.29 cm, the stirring speed of 228 rpm and with aluminum as electrode material as an one convenient method and optimum conditions in the process of harvesting and separation of biomass from the culture medium in the cycle of biodiesel production of microalgae that can facilitate competitive conditions of biofuels with fossil fuels and supply the environmental issues and community health.

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