



## RESEARCH PAPER

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## Harvesting of microalgae by electro-coagulation-flocculation for biodiesel production: an investigation of the effect of operational parameters and forecast model using response surface methodology

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### Abstract

In this study, electro-coagulation-flocculation (ECF) technology has been used for the harvesting of *Dunaliella salina* microalgae from the culture medium. The effect of operational parameters such as current intensity, ECF time, electrode gap, stirring speed and electrode material were studied in an attempt to reach higher microalgae recovery efficiency. The experiments were designed based on multifactor response surface methodology (combining categorical with numeric factors). The results indicated that aluminum was an excellent electrode material for microalgae recovery as compared with iron. Furthermore, current intensity, ECF time, electrode gap, stirring speed had impact on microalgae recovery efficiency. Among the above-mentioned parameters, electric current intensity, time and electrode material had highest effects and electrode gap and stirring speed had the lowest effect on the ECF performance. The maximum microalgae recovery efficiency of 98.06% was estimated 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.

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## Introduction

Algae have demonstrated to be an efficient bio energy source (Chisti, 2007) because in contrast to sugarcane, soybean, canola and oil palm, algae are not edible, they are less expensive to produce, grow faster, allow higher yield and production rate per hectare (Sivakumar *et al.*, 2010), do not require clean water to grow, and have the potential of reducing carbon emission (Danquah *et al.*, 2009). This is the reason why developed countries have turned to algae as an efficient and adequate choice for the global energy crisis.

Because of their small size (typically a few micrometer) and low concentration in the culture medium (0.5–2 gL<sup>-1</sup>), harvesting microalgae biomass is a major challenge. Most existing microalgae production systems use energy intensive centrifuges to harvest microalgae (Heasman *et al.*, 2000). Consequently, harvesting represents a major fraction of the total energy demand of the production process (Grima *et al.*, 2003; Uduman *et al.*, 2010). If the microalgae could be pre concentrated 30–50 times by coagulation–flocculation and gravity sedimentation prior to centrifugation, the energy demand for harvesting could be strongly reduced (Harun *et al.*, 2010; Uduman *et al.*, 2010).

Microalgae can easily be flocculated using metal coagulants such as Fe<sup>3+</sup> or Al<sup>3+</sup> salts (Ahmad *et al.*, 2006; Bernhardt and Clasen, 1991; Papazi *et al.*, 2009). In wastewater treatment, electro-coagulation–flocculation (ECF) has been proposed as an alternative for chemical coagulants (Mollah *et al.*, 2001). In ECF, iron or aluminum ions are released from a sacrificial anode through electrolytic oxidation. Compared to coagulation–flocculation with Fe<sup>3+</sup> or Al<sup>3+</sup> salts, ECF has the advantage that no anions such as chlorine and sulphate are introduced in the process water. The electrolytic oxidation of the sacrificial anode, however, requires electricity.

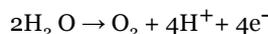
The main reactions occurring in ECF process with different electrode materials are as follows (Vasudevan *et al.*, 2008):

When aluminum is used as electrode material,

Anode:

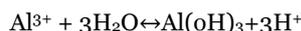


(1)



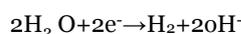
(2)

In solution:



(3)

Cathode:



(4)

Some studies have investigated the use of ECF for removal of microalgae from drinking or wastewater (Alfajara *et al.*, 2002; Azarian *et al.*, 2007; Gao *et al.*, 2010; Poelman *et al.*, 1997).

The main goal of this study was to demonstrate the proof of principle for harvesting of microalgae using electro-coagulation–flocculation and to investigate the influence of several important variables on the efficiency of the electro-coagulation–flocculation in harvesting and separating *Dunaliella salina* microalgae from the culture medium. This is a native species and halophyte microalgae with a different culture medium from the fresh water in terms of salinity and electrical conductivity.

## Materials and methods

### Cultivation of Microalgae

In this research was used a native species of *Salina Donalila* microalgae of Urmia Lake which is a halophyte species. Stock of microalgae were obtained from a collection of microalgae in Biotechnology Research Center, Tabriz. For the cultivation of *Donalila* was prepared three one-liter culture mediums in total sterile conditions. The first culture medium was included: NaCl, 87.75 g; MgCl<sub>2</sub>·6H<sub>2</sub>O, 9.8 g; CaCl<sub>2</sub>·2H<sub>2</sub>O, 0.53 g; Na<sub>2</sub>SO<sub>4</sub>, 3.2 g; SO<sub>4</sub> K<sub>2</sub>, 0.85 g; Tris-Base, 12.11g; KNO<sub>3</sub> (1M/L), 5cc; and the

second medium culture was included 5 ml for per liter of trace elements,

Including:  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , 0.004g;  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ , 0.00072;  $\text{NaMoO}_4 \cdot 2\text{H}_2\text{O}$ , 0.00025 g;  $\text{Na}_2\text{EDTA}$ , 4.57 g;  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , 1.259 g;  $\text{CuSO}_4 \cdot 7\text{H}_2\text{O}$ , 0.0104 g;  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , 0.017 g; and the second medium culture was included 5 ml for per liter of  $\text{KH}_2\text{PO}_4$  (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 optimum temperature for cultivation was adjusted, 25°C; pH, 7.5; Light intensity, 3500 Lux (light intensity was measured by a light meter pro, model of TES 1339). For measuring electrical conductivity was used the EC meter (Metrohm, 712). During the cultivation, aeration was designed as continuously by an air pump and for drawing the growth chart, counting the frequency of cells by Hemacytometer (Lam Neubauer) and optical density by spectrophotometer (JENWAY, 6305) at a wavelength 550 and 680 nm on a daily basis was measured. Dense cultivation in 20 liters containers and in germinator was performed in the same way which described above.

#### *Harvesting and Recovery Microalgae*

##### *Design Electro-Coagulation-Flocculation System Reactor*

In this study, the experiments were made inside 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<sup>2</sup> 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 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. The main pilot in shape of cubic rectangular which is made of Plexi glass with

dimensions 35 × 28 × 18 cm and the effective volume of 14 liters was designed and built, in order to test the results of optimal experiments. In order to better mixture solution, was used from a digital-mechanical stirrer and air pump. 4 numbers electrodes (2cathode and 2anode) were designed with dimensions of 10 × 20 cm and a surface area of 400 cm<sup>2</sup> inside the main pilot reactor (Fig. 1).

#### *Experimental procedure*

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 Lam Neubauer and measuring optical density by spectrophotometer device was determined the initial concentration rate in the culture medium based on cells/L. Then the culture medium was transferred into the reactor. In order to investigate the effect of 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 effect of current intensity, reaction time, the electrode gap, stirring speed and the electrode material as Control Variables (independent) were studied on the recovery efficiency as the response variable (dependent). At the end of electrical process of coagulation 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:

Microalgae recovery efficiency

$$(\%) \eta_a = \frac{C_o - C_t}{C_o} \times 100 \quad (5)$$

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-coagulation-flocculation (ECF).

#### Design of experiments

In order to investigate the effects of five control variables (independent) was included: material of the electrodes on both levels of aluminum and iron; current intensity in the range of 300 to 1000 mA; time for electro-coagulation-flocculation, 5 to 20 minutes; the electrode gap, 1 to 3 cm; stirring speed between 0 to 400, on the recovery efficiency as the response variable (dependent) experiments based on multifactors response surface method (combining categorical with numeric factors) was designed.

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 control and response variables. This approximation function is usually a polynomial function of control variables, such as which is used in the Eq. (6) (Montgomery, 2001).

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j + \varepsilon \quad (6)$$

where  $y$  represents the value of response;  $\beta_0, \beta_i, \beta_{ii}, \beta_{ij}$  regression coefficients;  $k$  the number of independent variables; and  $\varepsilon$  the amount of errors. In the response surface method in order to estimate the parameters of the function, the control variables must be have coded values these can be coded with Eq. (7) (Montgomery, 2001).

$$x_i = \frac{x_i - X_0}{\Delta X_i} \quad (7)$$

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,  $X_0$  is the actual center point value of the  $i$ th independent variable, and  $\Delta X_i$  is the step size of the  $i$ th independent variable.

In this study, for designing an experiment, statistical analysis and optimization was used from the software Design-Expert 8.

#### Results and discussion

In this study, the modified quadratic model was used to fit the Microalgae recovery efficiency data obtained from each batch test. The coefficients of determination ( $R^2$ ), adjusted and predicted were respectively more than 0.98, 0.96 and 0.90, which indicated that the modified quadratic model could describe the the Microalgae recovery efficiency in the batch tests of this study successfully.

**Table 1.** ANOVA of the fitting model for Microalgae recovery efficiency

Source	Sum of Squares	df	Mean Square	F Value	p-value
Model	13875.51	10	1387.55	101.04	< 0.0001
x1-Current	4238.5	1	4238.5	308.66	< 0.0001
x2B-Gap electrode	408.14	1	408.14	29.72	< 0.0001
X3-Time	2926.59	1	2926.59	213.12	< 0.0001
X4-Stirrer	159.38	1	159.38	11.61	0.0019
X5-Electrode Material	2627.33	1	2627.33	191.33	< 0.0001
x1 x3	1041.06	1	1041.06	75.81	< 0.0001
x1 x5	219.31	1	219.31	15.97	0.0004
x2 x3	100.06	1	100.06	7.29	0.0113
x22	87.19	1	87.19	6.35	0.0173
x42	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-Squared	0.9712				
Adj R-Squared	0.9616				
Pred R-Squared	0.9469				

### The comparison of different electrode materials

It has been established that the electrode material plays an important role in ECF process. Aluminum and iron have both been widely used as electrode materials in this process by many investigators (Alaton *et al.*, 2008; Zaied and Bellakhal, 2009). However, little information about the comparison of electrode material on algae removal could be found in the comparison up to now. In this investigation, comparative experiments were carried out using aluminum and iron as the electrode material, under the same operation conditions. ANOVA of the fitting model (table 1) showed that linear effect of electrode material and the interactive effect between electrode material and current on the recovery efficiency is very significant. With the use of statistical response surface method, the recovery efficiency function using

experimental data for aluminum and iron electrodes respectively to be predicted based on Eq. (8) and Eq. (9).

(8)

$$Y_{Al} = 8.409 + 0.056X_1 + 15.339X_2 + 3.471X_3 + 0.046X_4 - 0.00248X_1X_3 - 0.266X_2X_3 - 3.932X_2^2 - 8.72E05X_4^2$$

(9)

$$Y_{Iron} = -17.78 + 0.072X_1 + 15.339X_2 + 3.471X_3 + 0.046X_4 - 0.00248X_1X_3 - 0.266X_2X_3 - 3.932X_2^2 - 8.72E05X_4^2$$

where  $X_1$ ,  $X_2$ ,  $X_3$  and  $X_4$  respectively are the actual values of current intensity (mA), gap electrodes (cm), time (min), speed of stirring (rpm).

**Table 2.** optimized values of the ten top priority, according to maximize recovery efficiency

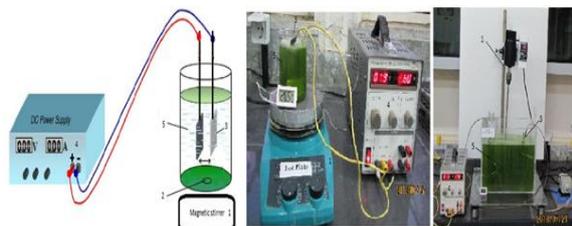
run	Current mA	Gap Electrode	Time min	Stirrer rpm	Electrode Material	Algae Recovery	Desirability
1	999.88	1.39	20	222	Al	98.0681	0.976*
2	1000	1.32	19	228	Al	97.9984	0.975
3	1000	1.4	20	211	Al	97.9632	0.974
4	1000	1.25	19	229	Al	97.8687	0.973
5	987.65	1.4	20	228	Al	97.8652	0.973
6	999.99	1.21	20	229	Al	97.7879	0.972
7	982.06	1.39	20	229	Al	97.7436	0.971
8	1000	1.17	20	228	Al	97.675	0.971
9	1000	1.34	20	186	Al	97.5391	0.969
10	999.82	1.19	20	205	Al	97.4739	0.968

\*selected.

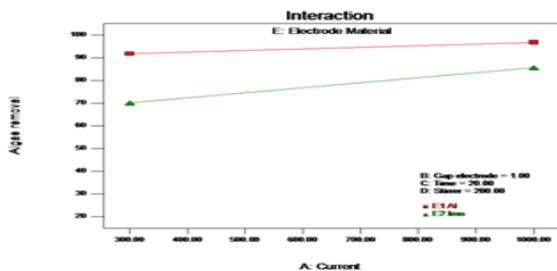
Fig. 2, represents the effect of electrode material on the recovery efficiency of microalgae. According Fig. 2 (A), in the condition that ECF Time, 20min; the electrode gap, 1 cm; and the speed of stirring in 200 rpm, was kept constant in current intensity of 300 mA, the efficiency recovery using electrodes of aluminum and iron, respectively are equal to 91% and 70%. In the same Fig., when the current intensity is 1000 mA, the efficiency recovery of aluminum and iron electrodes, respectively are equal to 97% and 86%. Thus, in comparing difference between recovery efficiency of both iron and aluminum electrodes under the same conditions of Fig. 2 (A), respectively are (86% vs. 97%) in current intensity 1000 mA and (70% vs. 91%) has been located in current intensity of 300 MA. in one other case based on Fig. 2 (B), when

the ECF Time, 5min; the electrode gap, 1 cm; and the speed of stirring in 200 rpm, was kept constant in current intensity of 300 mA, the efficiency recovery using aluminum and iron electrodes, respectively are equal to 55% and 33%. In the same Fig, when the current intensity is 1000 mA, the efficiency recovery of aluminum and iron electrodes, respectively are equal to 86% and 75%. Thus, in comparing difference between recovery efficiency of both iron and aluminum electrodes under operating conditions identical to Fig. 2 (B) respectively are, (75% vs. 86%) in current intensity 1000 mA and (33% vs. 55%) has been located in current intensity of 300 mA. The reason for this could be due to differences in material structure of aluminum and iron, and probably due to the much higher current efficiency generated by

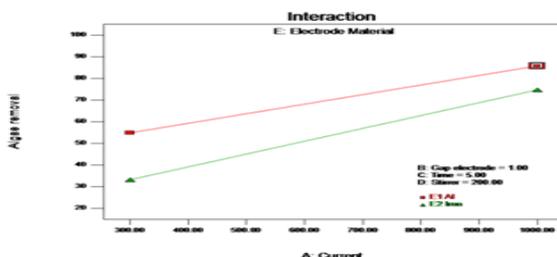
aluminum electrodes than that by iron electrodes. It is also possible under the Ph experiment 7.5; the amount of aluminum hydroxide was much higher than that of ferric/ferrous hydroxide, which may be another reason accounting for the better recovery of microalgae with aluminum electrodes (Duan and Gregory, 2003). Similar reports by (Vandamme *et al.*, 2011; Zongo *et al.*, 2009; Caizares *et al.*, 2005; Gao *et al.*, 2010) has been established in other applied industries that are consistent with the results of this research. Overall, aluminum was considered as the better one for the microalgae recovery.



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



**A.** In situations where reaction time is 20 min; the electrode gap, 1 cm and stirrer speed was kept constant 200 rpm.

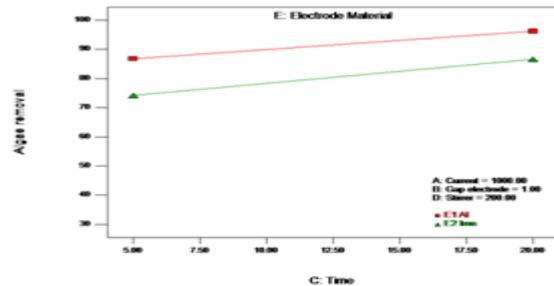


**B.** In situations where reaction time is 5 min; the electrode gap, 1 cm and stirrer speed was kept constant 200 rpm.

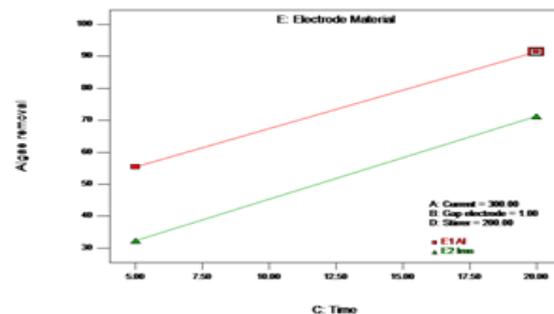
**Fig. 2.** Effect of Electrode Materials and current intensity on the recovery efficiency of microalgae.

*Effect of current intensity and ECF time on the recovery efficiency*

ANOVA of the fitting model (table 1) showed that the linear effect of two-controlled variables, current intensity and *reaction time* on the recovery efficiency is very significant, Also the coefficients of these two variables at the Eq. (8) and Eq. (9), showed that by increasing the current intensity and *reaction time*, the recovery efficiency has improved.



**A.** in situations when current intensity 1000 mA; the electrode gap, 1 cm and stirrer speed was kept constant 200 rpm.

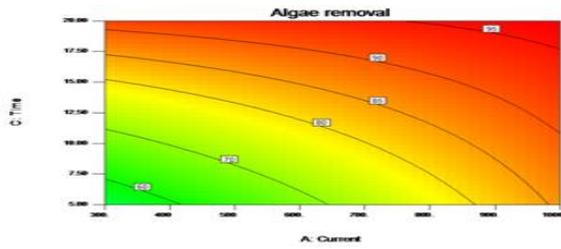


**B.** In situations where current intensity of 300 mA; the electrode gap, 1 cm and stirrer speed was kept constant 200 rpm.

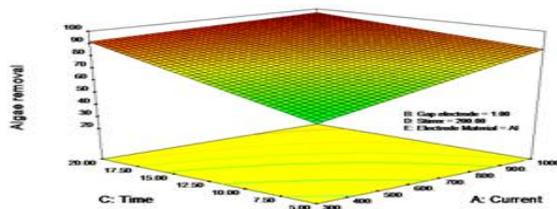
**Fig. 3.** Effect of reaction time on the recovery efficiency of microalgae at different Electrode Materials.

Fig. 2, represents the effect of current intensity on the recovery efficiency of microalgae. A section of this chart shows, in the conditions that electrode Material is aluminum; the electrode gap, 1 cm; and the speed of stirring in 200 rpm; and ECF time was kept constant in 20 minutes, by increasing the current intensity from 300 to 1000 mA, the recovery efficiency of microalgae has increased from 91% to 97%. Fig. 2 (B), indicates when the electrode Material is aluminum, ; the electrode gap, 1 cm; and the speed

of stirring in 200 rpm, and ECF time was kept constant 5 minutes, by increasing the current intensity from 300 to 1000 mA, recovery efficiency of microalgae has increased from 55% to 86%.



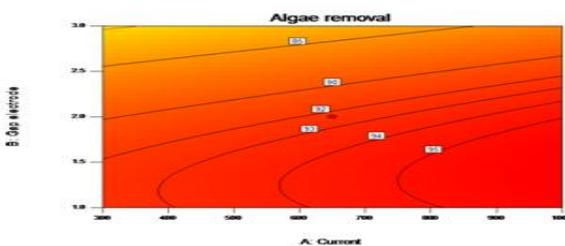
A. Contour plot.



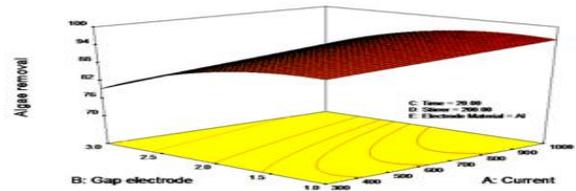
B. Response surface plot

**Fig. 4.** Response surface plot and corresponding contour plot of current intensity effect and the ECF time on recovery efficiency. (in the conditions that electrode Material is aluminum; the electrode gap, 1 cm; and the speed of stirring in 200 rpm).

Fig. 3 shows the effect of ECF time on the recovery efficiency. Fig. 3 (A), indicated, in the conditions that electrode Material is aluminum; the electrode gap, 1 cm; and the speed of stirring in 200 rpm; and current intensity in the 1000 mA was kept constant with increasing length of the ECF from 5 to 20 min, the recovery efficiency of microalgae has been increased from 86 % to 97%. Fig. 3(B), indicated, in the conditions that electrode Material is aluminum; the electrode gap, 1 cm; and the speed of stirring in 200 rpm; and current intensity in the 300 mA was kept constant with increasing length of the ECF from 5 to 20 min, the recovery efficiency of microalgae has been increased from 55 % to 91%.



A. Contour plot.



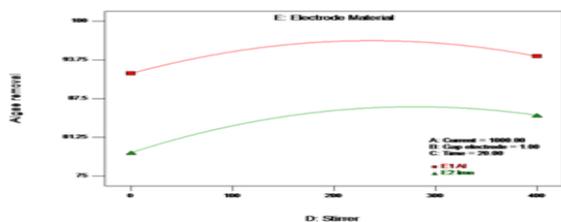
B. Response surface plot.

**Fig. 5.** Response surface plot and corresponding contour plot of Gap electrode effect and the ECF time on recovery efficiency.

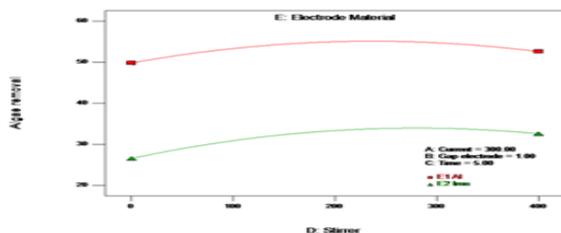
(In situations where electrode Material is aluminum, time of ECF is 20 minutes and stirrer was kept constant 200 rpm)

Also table ANOVA of the fitting model (table 1) showed that interaction effect between the current intensity and time of ECF on the recovery efficiency is significant, in other words 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. (8) and Eq. (9)., that interactive effect between these two factors has a negative effect on the recovery efficiency. Fig. 4, indicated that the interactive effect between the current intensity and time of ECF on the recovery efficiency in the conditions that electrode Material is aluminum; the electrode gap, 1 cm; and the speed of stirring in 200 rpm. Considering the significant linear effects, quadratic and interaction between current intensity and ECF time was expected to be curvature on contour and response surface plots. As you can see by holding time of ECF for 5 minutes and with increasing current intensity from 300 to 1000 mA, the recovery efficiency curve with a steep slope has increased from 55% to 86%, So simple effect of the minimum and maximum of current intensity (difference between minimum and maximum current intensity) in the minimum level of the ECF time (5 min) is equal to 31%. in the second step by holding time in the 20 minute and with increasing current intensity from 300 to 1000 mA, the recovery efficiency curve with a light slope has been improved

from 91% to 97%, Therefore, simple effect of the minimum and maximum current intensity (the difference between the minimum and maximum current intensity) at the maximum level ECF time (20 minutes) is equal to 6%. This could be attributed to the fact that the dissolved aluminum from anode increased with the current intensity and electrolysis time according to the faraday's law. As the concentration aluminum and iron ions increased in the ECF, the coagulant surface area and number of active sites increased correspondingly (Zhu *et al.*, 2005) and ion-charged algae particles, depend on the ions interaction which are produced by the dissolution of anode electrode will be neutralize and by absorbing each other, it means that action of coagulation, are separated from the solution.



**A:** in situations when current intensity 1000 mA; the electrode gap, 1 cm and time of ECF is 20 minutes was kept constant 1 cm.



**B:** In situations when current intensity 300 mA; the electrode gap, 1 cm and time of ECF is 5 minutes was kept constant 1 cm.

**Fig. 6.** Response surface plot and corresponding contour plot of Gap electrode effect and the ECF time on recovery efficiency.

Also hydrogen gas will be free surrounding the cathode electrode, makes some clots floating on the surface of the solute and separating them. Similar reports is published in this issue by (Kim *et al.*, 2012; Vandamme *et al.*, 2011; Gao *et al.*, 2010) while being consistent with these results represent direct relation of the recovery efficiency improvement by increasing

the time and current intensity, So that the initial minutes of harvesting efficiency has high speed and over time harvesting efficiency are more homogeneous.

#### *The effect of electrode gap on the recovery efficiency*

The results of ANOVA of the fitting model (table 1), indicated that the linear effect and quadratic electrode gap on the recovery efficiency is statistically significant ( $p < 0.01$ ). the coefficients of the control variables in the Eq. (8) and Eq. (9), be inferred that the electrode gap has positive linear effect and negative quadratic effects on the recovery efficiency. As can be seen response surface and contour plots (Fig. 5), in the conditions that electrode Material is aluminum; the speed of stirring, 200 rpm; the current intensity, 1000 mA and and ECF time, 20 minutes was kept constant, With increasing electrode gap from 1 to 2 cm, the recovery efficiency has decreased from 97% to 95% and with increasing electrode gap to 3 cm, the recovery efficiency is reduced to 85%, so that the difference of the recovery efficiency from 1 to 2 cm low and about 2%, was occurred with a light slope but from 2 to 3 cm slope of the curve is steep and the recovery efficiency has decreased very significant about 10%. In the Fig. 5, with the changes in current intensity to 300 MA, in the conditions that electrode Material is aluminum, stirrer speed of 200 rpm and ECF time, 20 minutes was kept constant, It was observed that by increasing the electrode gap from 1 to 2 cm, the recovery efficiency has decreased from 92% to 89%, and with increasing distance between the electrodes to 3 cm, the recovery efficiency has decreased to the 79%, so that difference between the recovery efficiency from 1 to 2 cm is low and about 3%, carried out with a light and smoothly slope, but from 2 to 3 cm slope of the curve is steep and the recovery efficiency has reduced very significant about 10%. Previous research results on electro coagulation for other applications also indicates that in the electrochemical process and in the same time, when the distance between the electrodes is increased the coagulation efficiency is reduced (Kim *et al.*, 2002). From the results it can be inferred that by increasing the distance between the

electrodes the electrical resistance between the electrodes is increased, as a result, the solution conductivity is reduced and the amount current consumption decreases that this reduction of the flow makes absence of ions production (aluminum or iron) and enough Hydroxyl for flocculation and the coagulation of microalgae. So that, in this study it was observed in a constant current intensity by increasing the distance between the electrodes, the voltage is increased, this issue means that more consuming of electric current, and thus will consume more electrical energy. In addition by increasing distance between the electrodes is expected that from one side fewer interaction between the produced ions (aluminum or iron) was occurred with the hydroxyl ions, And fewer flocculation be formed on the other hand interaction between microalgae with polymers hydroxide is reduced, thereby reducing the electrostatic absorption and the recovery efficiency will be low (Song *et al.*, 2007).

#### *Effect of stirrer speed on the recovery efficiency*

Also table 1, revealed that the linear effect and quadratic stirrer speed on the recovery efficiency is significant. The coefficients of the control variables in Eq. (8) and Eq. (9), will be inferred that linear of stirring speed is positive and negative quadratic effects are on the recovery efficiency. Considering the significant linear effects, quadratic of this variable, were expected the existence of curvature in the response surface and contour plot.

Fig. 6(A), indicated, in the series of tests with aluminum electrodes, in which the current intensity is 1000 mA, electrode gap is 1 cm and duration of the ECF was kept constant in 20 minutes, by increasing the stirrer speed from 0 to 200 rpm, the recovery efficiency curve with a steep slope has increased from 91% to 97% but with increasing the speed from 200 to 400 rpm, the recovery efficiency curve has decreased 97 to 94 % with a light slope. Also in the series of tests with aluminum electrodes, where current intensity of 300 mA and duration of the ECF was kept constant in 5 minutes, with increasing stirrer speed from 0 to 200 rpm, the recovery efficiency curve has increased with

a steep slope from 49% to 55% but with increasing the speed from 200 to 400 rpm, the recovery efficiency curve has decreased with a light slope from 55 to 52% (Fig. 6 B). The results are consistent with the research report of (Canizares *et al.*, 2006; Mollah *et al.*, 2004; Vandamme *et al.*, 2011). Generally was concluded from the results that in coagulation process, the clotted material through mixture close to each other slowly and create larger particles, that are could be easily floating or sedimentation, Therefore, to make a gentle mixture the existence of stirred in the mechanism with the rotational speed of 200 rpm improves the recovery efficiency, But with increasing rotational speed of stirrer from 200 to 400 rpm due to the high stirrer speed, coagulant material is not able to flocculation the microalgae or even possibly created flocculation in this rate of stirring, was broken and re-formed the suspension and were deposited.

#### *Optimizations*

The relations that were discussed in the previous sections, only for predicting recovery efficiency 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. In this research for optimizing the Control Variables under study with aims of maximization of the recovery efficiency of *Dunaliella salina* microalgae has been done by using electro-coagulation-flocculation (ECF) technology in biodiesel production cycle, Table 2 is shown the results of ten top priority of maximizing recovery efficiency. The maximum microalgae recovery efficiency of 98.06% was obtained 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 with aluminum as electrode material, and selected as a priority.

#### **Conclusion**

In this study was examined the effect of five control

variables (independent) including: current intensity, electrode gap, ECF time, stirring speed and electrode material on the response variable (dependent) the recovery efficiency of *Dunaliella salina* microalgae from the culture medium. The modified quadratic model was used to fit the microalgae recovery efficiency data obtained from each batch test. The coefficients of determination ( $R^2$ ), adjusted and predicted were respectively more than 0.98, 0.96 and 0.90, which indicated that the modified quadratic model could describe the microalgae recovery efficiency in the batch tests of this study successfully. The results indicated that the linear effect of control variable on the recovery efficiency is very statistically significant. Moreover with increasing the electric current intensity variable and ECF time, 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. The results showed that aluminum electrodes on the recovery of microalgae from the culture medium are more efficient than iron electrodes. In this study, were searched the optimal operating conditions with aims of maximization of the microalgae recovery efficiency. The maximum microalgae recovery efficiency of 98.06% was obtained 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 with aluminum as electrode material. If you want to achieve the maximum efficiency with considering economic factors, energy and environment, the second part of an article by the same research group that focuses on this topic is recommended.

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