The Treatment of Olive Oil Mill Waste Water (OMW) by Electrocoagulation

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Abstract

The effective performance of electrocoagulation (EC) technique in the treatment of olive mill wastewater (OMW) has been investigated using aluminum electrodes. The electrocoagulation progress was followed by the measurement of COD, turbidity, color, suspended solids, pH, height of liquor, TDS, conductivity, electrode and energy consumption, temperature and the quantity of electricity used. The effects of operating parameters such as current density, initial pH, electrolysis time, and distance between electrodes (D), on COD removal efficiency have been investigated. It has been shown that the removal efficiency of COD increased with the increasing applied current density and coagulation time. The obtained results of treatment of OMW, showed that the effectiveness of the reduction of COD and color by electrocoagulation increased with time and D at a constant current density (i=23.55 mA/cm2), the effectiveness of treatment was also increased with decreasing D at a constant voltage (U=15 Volt).The BOD5 treated/BOD5 raw ratio of the electrocoagulated OMW increased to 2. The electrodes consumption was 0.11 kgAl/kg CODremoved and the specific energy consumed was 3.93 kWh/kg CODremoved. the pH of effluent increased during electrolysis treatment.

Keywords: Olive mill wastewater; OMW, Electrocoagulation; Aluminum electrode.

1. Introduction

The olive oil industry, which is one of the most traditional agricultural industries in Syria, produces a lot of liquid waste. In the last years the world productions ranged from 2.5 to 3 millions tons of olive oil (3.0 in 2004 and 2.58 in 2005) [1, 2].

The quality and quantity of the constituents in olive oil wastewater are influenced by the type of production process, the type of olives and stage of maturity, the nature of the area under cultivation, use of pesticides and fertilizers, climatic conditions, and harvest time [1, 2, 3, 4].

The actual industrial process for the production of olive oil, based on the olive milling, cause a large amount of waste water, more than 3×107 m^3/year of OMW in the Mediterranean area. OMW is a dark colored, mildly acidic liquid. Because of the high organic and polyphenol content of OMW, its direct disposal may pollute both land and aquatic environments. The phytotoxicity of OMW is due to the phenolic substances and some organic acids such as acetic and formic acids. The inorganic constituents at the concentration levels found in OMW are not toxic. In fact, it has been proven that they may potentially act as good sources of plant nutrients [5, 6].

Electrocoagulation (EC) of Olive Oil Mill Wastewater:

EC is one of the simple and efficient electrochemical methods for the purification of several types of water and wastewaters. During EC, when a potential difference is applied between a soluble anode, such as Fe or Al and the cathode, ferrous or aluminum and hydroxyl ions are generated, respectively, at the anode and the cathode [4, 7, 8, 9, 10, 11].

In the EC process, electrochemically generated aluminum can remove most   contaminants present in OMW by precipitation and adsorption [12, 13, 14].

The aluminum species acts as a coagulant by combining with the pollutants to form large size flocks and can then be removed by settling and flotation (EC/flotation process)[11, 13, 14, 15, 16].

The method includes several processes: oxidation, sterilization, and coagulation [4, 17, 18]. In this process, wastewater flows in the reactor which is designed in particular for the treatment of the OMW by electrodes connected to a direct current power. In a recent study, Nafaˆa Adhoum et al. [20] investigated decolourization and removal of phenolic compounds from olive mill wastewater by electrocoagulation. The optimum current density allowing the quickest treatment with a low cost was found to be 75 mAcm^{-2}, and the electrode consumption was found to be 2.11 kgm−3. The electrocoagulation has successfully been used for the treatment of wastewaters including paper mill effluents wastewaters [4], cutting oil emulsions oil suspensions wastewaters [8], defluoridation of semiconductor wastewater [11], textile wastewaters [12], oil suspensions wastewaters [17], poultry slaughterhouse wastewaters [19], and also of drinking water [21]. Treatment of oil refinery wastewater at a fixed current density using Ti/TiO^{2}RuO^{2}IrO$_2$ electrode and an undivided reactor was studied by Rajkumar and Palanivelu [22].

Waste from noncyanide stripper, a toxic liquid associated with the production of olive oil, was treated by an electrochemical method using Ti/Pt as anode and stainless steel 304 as cathode by Vlyssides et al. [23].

In this study, treatment of OMW in an electrocoagulation cell was investigated at constant and variable

currents to determination of the optimum conditions for the EC using aluminum electrodes.

2. Experimental Setup
2.1. Reagents and analytical procedures:
Fresh olive mill wastewater was collected from a local olive extraction plant located in the city of Homs (Syria) which uses a traditional process. OMW was collected in a closed plastic container.

The main characteristics of fresh olive mill wastewater used in this work are presented in Table 1. Analysis of COD was determined by the procedure described in the standard method [21].

Analysis of TOC was determined by Liqui TOC Elementar equipment. The total solid content was measured by drying a 100 ml aliquot of OMW at 105 ∘C until a constant mass was obtained. The dark color intensity was determined by measuring the sample absorbance at 455 nm (UV-Vis spectrophotometer, DR-5000). The TSS was determined by measuring the sample absorbance at 810 nm (DR-5000). The turbidity was determined by measuring the sample absorbance at 695 nm (DR-5000). A digital calibrated pH-meter (Inolab pH Level 2) and a conductivity-meter (Hach .Conductivity/TDS Meter) were used to measure the pH and the conductivity of the OMW wastewater samples.

2.2. Electrocoagulation procedure:
The experiments were conducted in a home-made Plexiglass electrocoagulator with the dimensions of 25 cm×10 cm ×10 cm, was equipped with two aluminum electrodes, one serving as a cathode and the other as anode (Fig. 1).

The total effective electrode area was 96 cm^2 (8 cm×6 cm×2 f). The electrodes were fixed in the cell at a distance of 2 cm from the bottom of the cell. The electrodes were connected to a digital DC power supply (5 A, 20 V). For each run, 1000 cm3 of 100 % (v/v) of OMW were placed into the electrolytic cell and a gentle stirring rate of about 200 rpm (revolutions per minute) was applied to allow the chemical precipitate to grow large enough for removal (the dimensions of stirring bar were 6 mm×15 mm). The samples were centrifuged at 3000 rpm (Labofuge 200 Heraeus Sepatech) for 5 min before being used for analysis.
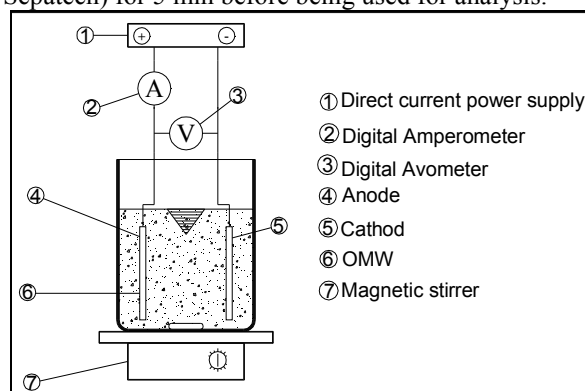

① Direct current power supply
② Digital Amperometer
③ Digital Avometer
④ Anode
⑤ Cathod
⑥ OMW
⑦ Magnetic stirrer

Fig. 1 Electrolytic cell.

3.Result and discussion:
3-1. Determination of optimum distance between the electrodes:
3-1-1. Determination of optimum distance between the electrodes depending on the variations of CODremoved:
Fig. 2 shows the variations of COD with the distance between the electrodes in two cases:((1) constant current intensity at variable voltage, (2) variable current intensity and constant voltage)). The curves show a decrease in the COD conversion with the increase in D at (U=15 V), and a decrease in COD conversion with decrease D at (I=constant). The two types of curves intersect when t =10- 20- 30- 45- 60 min at: Dopt = 1.45-1.65-1.85-1.48-1.75 cm. and COD Conversion equal to 9.12- 11.21- 13.87- 17.78- 21.97 % respectively.

When the inter-electrode distance D was1.0 cm, the removal efficiency of COD reached 32 % after 60 min reaction, while it was only 12 % when the distance was 6.0 cm. These results could be explained by the effectiveness of floatation by gas bubbles generated at the electrodes.
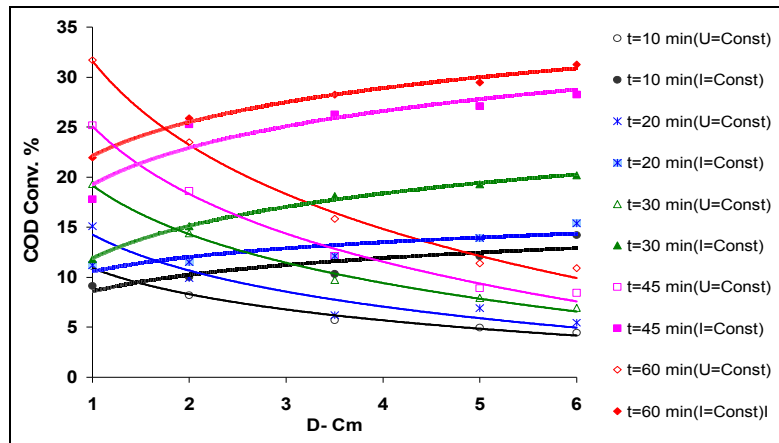
Fig. 2 Effect of D on COD conversion at constant current or voltage

3-1-2 Determination of optimum D depending on electrode consumption:
Fig.3 shows the practical and theoretical consumption of the electrode at constant current (I = 3 A) and constant voltage (U=15 V).

It is clear that the two curves are crossing at   D = 2.2 and 2.4 cm, respectively, and these are the optimum values. Moreover, Fig. 4 shows that the curves intersect at 2>D>1 cm and D≤1 cm at constant current (I = 3A) and constant voltage (U = 15 Volt), respectively. Since the best value of D was found to be between 1 and 2 cm, the optimum value was chosen 1.5 cm.
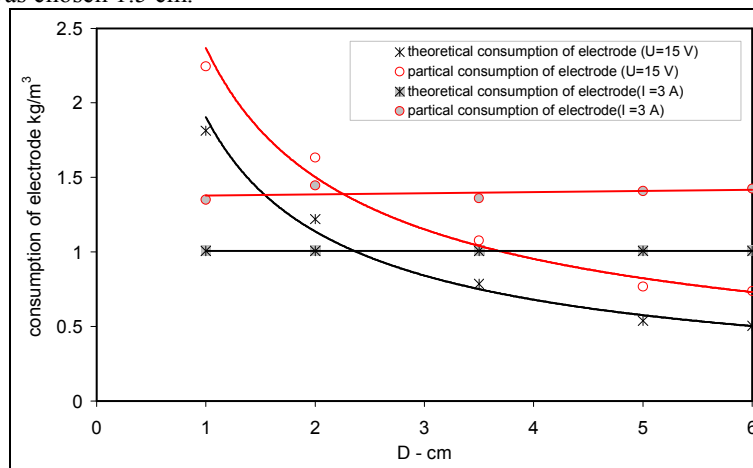

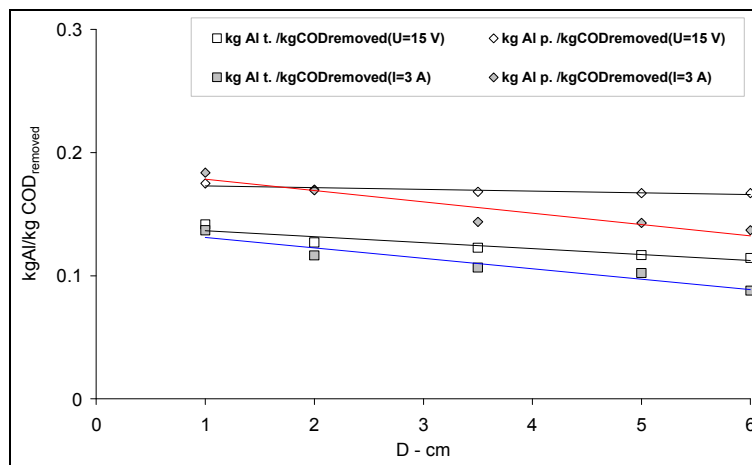**Fig. 3** Effect of D on electrode consumption at (I = 3 A) or (U=15 V).

Fig. 4 Effect D on electrode consumption for a unit of COD removed

3-1-3. Evaluation of the specific consumption of electrode mAl (kgAl/m3):
As mentioned above, Fig. 4 shows the variations of specific consumption of electrode with D in two cases (constant current intensity and variable voltages, and constant voltage and variable current intensities) and it

shows that the electrode consumption is almost constant at 60 min for different D values with constant current intensity 3A. The theoretical consumption of electrodes was calculated by Faraday′s low [20]:

$$m_{Al} = \frac{M_{Al} \times I \times t \times 3600}{Z \times F}$$

where M= the atomic mass of the Al, I the current intensity, t the time of treatment, the charge number Z and F Faraday's constant.

As mentioned in the theoretical part, the treatment efficiency should increase with increasing specific consumption of electrode, which is practically constant at I = 3 A. Therefore, this efficiency is expected to be constant. The observed increase of efficiency must be due to another factor which could be the temperature rise due to the increase of U, and collision frequency between the pollutants and the hydroxyl polymer.

Accordingly, the simple increase of COD conversion with increasing D at I = 3 A undesirable because of the large energy consumption. At the same time, the consumption of aluminum decreased at a constant voltage with increasing D due to the decline of I.

3-1-4. Evaluation of the specific consumption of electrical power Wsp:
Fig. 5 Shows that Wsp increases with increasing D at I = 3A and decreasing D at U = 15 V.

Comparison of the curves, shows that the curve which represents the variation of Wsp by the time, at (I = 3A & U = 7.4 Volt & D = 1cm), corresponds exactly to the curve which represents the variation of Wsp with time, at (U = 15 Volt & I = 1.5A &   D = 6 cm). However, the COD conversion in the first case ranged between (7.38 - 19.69 %), whereas in second case it ranged between (4.45 - 10.89 %) when (t = 10 - 60 min) respectively.

It can be concluded that the treatment must be at high current intensity and low voltage, and at low D values which result in increase of the treatment efficiency, low power consumption and economical viability.

The electrical energy consumed, expressed as kWh per m3 of the treated wastewater was calculated using the equation EC = V×I×t /volume of OMW. The COD concentration of 36.8 g L−1 was reduced to 25.5 g L−1 after 1 h of electrolysis. The removal efficiency was 30.58 % for energy consumption equal to 22 kWhm−3.
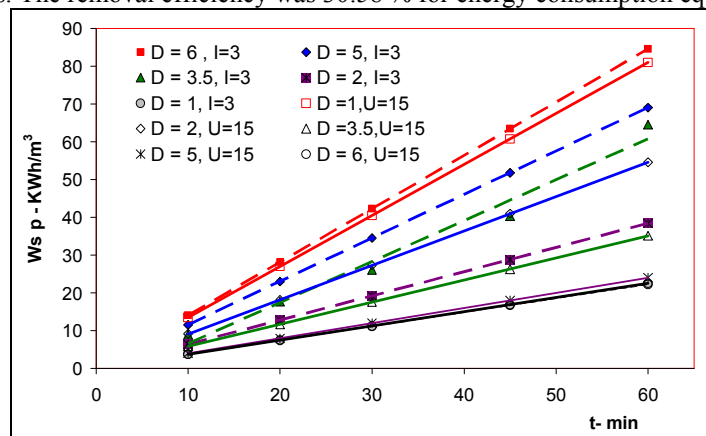

Fig. 5 Change of Wsp with time at I= 3A, U=15 Volt

3-2. Determination of optimum time:
3-2-1. Determination of optimum time depending on the TOC and COD conversions:
The optimal time is the time at which highest COD conversion value can be obtained at lower energy consumption.

It was found that during the first 30 min, the conversion of TOC was 33.5 % and 42.4 % at D= 1 and 2 cm respectively. While this conversion was only 10.9 % and 10.7 % in the next 30 min. similar trend was observed for the conversion of COD. Therefore the optimal time is believed to be 30 min.
3-2-2. Determination of optimum time depending on electrode and electrical energy consumption:
Practically, the electrode consumption at I=3A, and D=1 cm was increased from 0.765 kg/m^3 during the first 30 min to 1.3503 kg/m3 at t=60 min.

The ratio of m Al (t=60 min)/mAl (t=30 min) is equal to 1.766. This means that the amount of consumed aluminum in the second thirty minutes is less than that in the first thirty minutes, which explains the reason of decreased COD conversion in the next thirty minutes.

Moreover, the electrical energy consumption at 60 min is double that at 30 min, whereas the COD conversion is not. This is why the optimal treatment time has been chosen as 30 min.

4- Electrocoagulation of OMW at various current densities:

Electrocoagulation tests were performed on OMW at t=30 min, D=1cm, S=128 cm2 with variable current intensities (I=1-2-3-4 A) and current densities: (i = 7.8 -15.6 - 23.4 - 31.25 mA/cm2) respectively. (Table 1) demonstrates the results of the increase in temperature differences of treated water compared with that of the raw sample at I=1, 2, 3, 4 A. It also shows the increase of the ratio the amount of Alkg/kg CODremoved with increasing current density.

Raw OMW was highly colored due to its high content of polyaromatic compounds. At I= 1, 2 A of the electrolytic treatment, the color intensity of the effluent increased. This could be explained by the oxidative polymerization of phenols and tannins originally present in the sample, which resulted in dark colored organic compounds [20]. However, color intensity decreased to 50 % of the initial value at I = 4 A.

EC OMW had a turbidity value of 312, 714, 1202, 1245 FAU, at I=1, 2, 3, 4 A respectively, which was higher than the initial value. It should be noted that turbidity depends on the amount of TSS produced during the electrolysis process. Thus, an appropriate technique should be adopted for TSS and turbidity removal after electrolysis treatment.

80 % of TSS was removed after 10 min of centrifugation time at 3000 rpm for I = 4 A. The removal of 17.39 %, 50 % and 91 % of COD, color, and turbidity, respectively were obtained by centrifugation.

After electrocoagulation, sedimentation step was necessary to separate the suspended solids from the wastewater. For this reason, EC pre-treated OMW was allowed to settle for 3 h. The behavior of both raw and EC OMW inside the settling column was observed through the analysis of the collected samples. Experimentation showed that small suspended particles appeared in wastewater which increased in number and size as the electrolytic treatment continued. Crude OMW contained a smaller quantity of suspended solids (4.4 g L^{-1}) compared to EC OMW (38.6 g L^{-1}) for I = 4 A.

Table 1: Effect of current density on electrocoagulation of OMW after 30 minutes

parameter	OMW	I = 1	I = 2	I = 3	I = 4	Units
Δ Temprature	-	11	12	17	23	°C
Color *100	84	126	141	59	42	Pt Co
TSS	4.4	8	18.4	29.8	38.6	g/L
pH	3.9	4.1	4.3	4.5	4.7	-
Turbidity*100	149	312	714	1202	1245	FAU
COD	36.8	33.8	33.2	32	30	g/L
Conv. COD		8.15	9.8	13	17.4	%
U		3.5	6.2	8.4	11.5	mV
i		7.8	15.6	23.4	31.2	mA/cm2
Wsp		1.75	6.2	12.6	23	kWh/m3
Wsp/CODremov.		0.58	1.72	2.63	3.59	kWh/kg CODremov.
m Electrodes		0.25	0.49	0.77	1.07	kg/m3
mp. Al/CODremov.		0.08	0.14	0.16	0.17	kg Al/kg CODremov.

4-1. Effect of current density on the removal efficiency of COD and electrical power consumption Wsp:

Fig. 6 and Table 1 show that the removal efficiency of COD significantly increased with increasing current density, they also show the change of the specific energy consumption versus i for Al electrodes during the EC expressed in kWh consumed per m3 OMW. The minimum energy consumption and maximum COD removal were observed at (i ≈ 31.26 mA/cm2) for 30 min of electrolysis time.
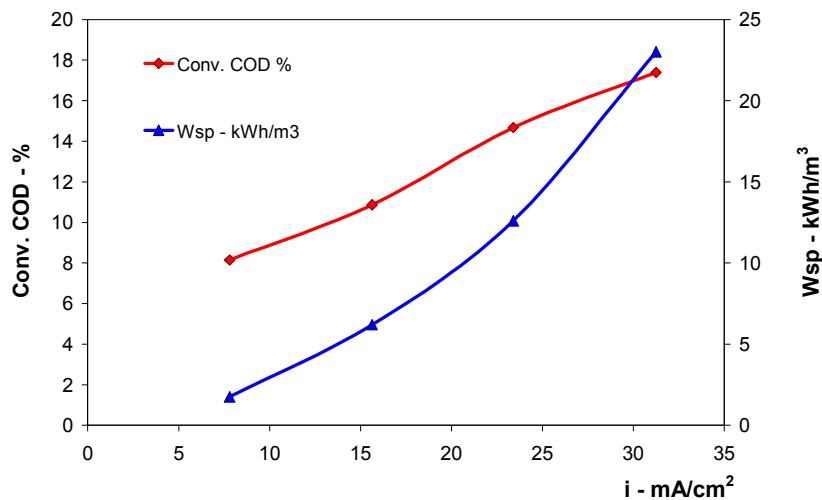
Fig. 6 Effect of current density on energy consumption and COD conversion

4-2. Effect of current density on electrode and energy consumption:

Fig. 7 illustrates the effect of current density on electrode consumption expressed in kg of Al per kg of COD removed, and the effect of current density on energy consumption (kWh/kg CODremov.).

As expected, the amount of aluminum oxidized increased with current density, resulting in a greater amount of precipitate for the removal of pollutants, and greater energy consumption kWh/gCODremov.. The curves intersect at optimum I (i=31.25 mA/cm^2 approximately).
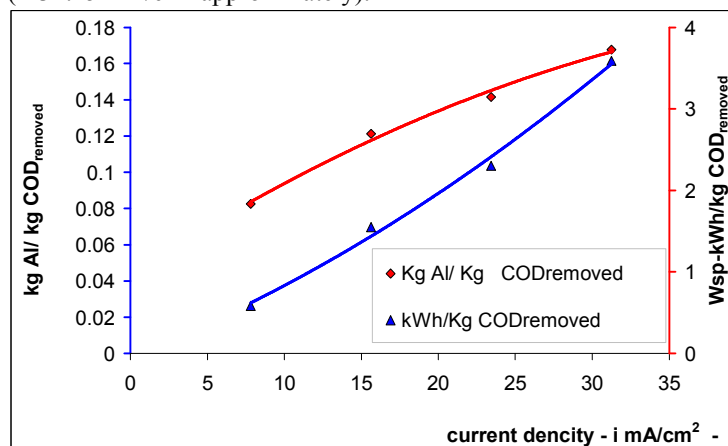
Fig. 7 Effect of current density on electrode and energy consumption

4-3. Analysis of the sludge:

The amount of floated scum increased and the settled sludge decreased with increasing current density, and the ratio of organic part in the floated sludge increased compared to the settling sludge, while the ratio of the inorganic part in the settled sludge was the highest.

The amount of sludge decreased gradually during the settling time at the top of the column. The settled and floated scum were dried over night at 105 ℃. These represented the total suspended solids (TSS). The ash content was determined after calcinations of the dry sludge at 600 °C for 2h. Fig. 8 illustrates the obtained results.
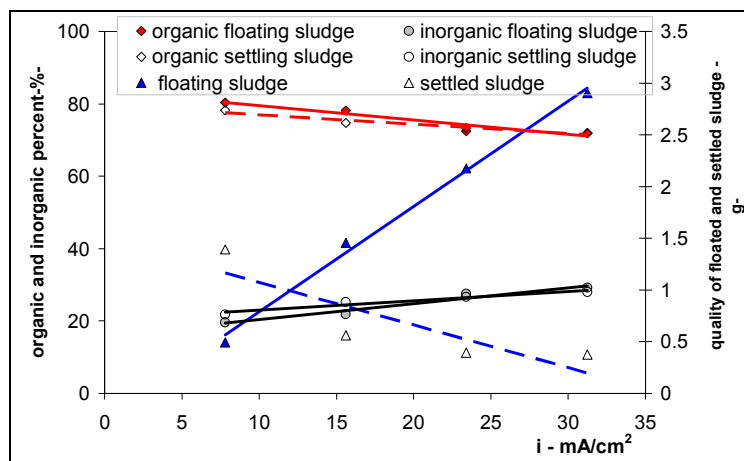
Fig. 8 The composition of organic and inorganic sludge, and scum and sludge at different currents

5-Effect of the total effective electrode area on the electrocoagulation:

Aluminum electrodes with dimensions 8×8, 8×6, 8×4, 8×2 cm^2 were used to study the effect of electrode surface on the treatment effectiveness, energy and electrode consumption (at I=4 A, t=30 min, D=1 cm) where the values of current density were: i = 31.25-41.66-62.5-125 mA/cm^2.

The results are shown in Table 2. They indicate that there is no relationship between the ratios of current densities and aluminum consumption at different electrode surfaces and constant current intensity. The aluminum consumption increases by increasing of the current intensity i, because of decreasing the electrode surface area which caused the increase of temperature resulted from increasing U. Table 2 the values of the indicators related to at these conditions.

5-1. Effect of current density on removal efficiency of COD and Wsp:

As is clear from Table 2, the change of current density increases with the decrease in effective electrode surface area (i=31.25, 41.66, 62.5, 125 mA/cm^2). the percentage increase in this density as follows: 33.33–50 and 100 % for the electrode area 8×6, 8×4, 8×2 cm^2 respectively, with respect to, the density at electrode area equal to 8×8 cm^2. The corresponding COD conversion decreases from 23.1 to 12.5 to 8.36 %, and the percentage change of Wsp was 6.78 - 39.68 - 43.18 % respectively.

As displayed in Fig. 9 the curves intersect at i = 41.66 mA/cm^2 (at which the effective electrode area is 96 cm^2) which is the best current density.

The Table 2 includes calculation of cost of electrolytic treatment in terms of consumption of electrode and energy.

Table 2 Indicators related to electrode area

| Parameter	8×8	8×6	8×4	8×2	Units
Δ Temp.	23	26	31	44	°C
color Abat.	50	56.5	60	62	%
Ph	4.6	4.6	4.7	4.6	-
COD Conv.	13.9	17.1	19.3	20.8	%
I	31.25	41.66	62.5	125	mA/cm2
U	11.8	12.6	17.6	25.2	Volt
Wsp/ CODremov.	4.53	3.93	4.88	6.46	kWh/kg CODremov.
mp electrode /CODremov.	0.199	0.16	0.156	0.139	kgAl/kg CODremov.
mt. electrode /CODremov.	0.129	0.11	0.09	0.086	kgAl/kg CODremov.
Cost of electrode p.	0.99	0.8	0.78	0.7	$/kg CODremov.
Cost of electrode th.	0.65	0.5	0.47	0.43	$/kg CODremov
Cost of energy	0.27	0.24	0.29	0.39	$/kg CODremov
Cost (elect. & energy)p.	1.26	1.04	1.07	1.09	$/kg CODremov
Cost (elect. & energy)th.	0.92	0.76	0.76	0.82	$/kg CODremov
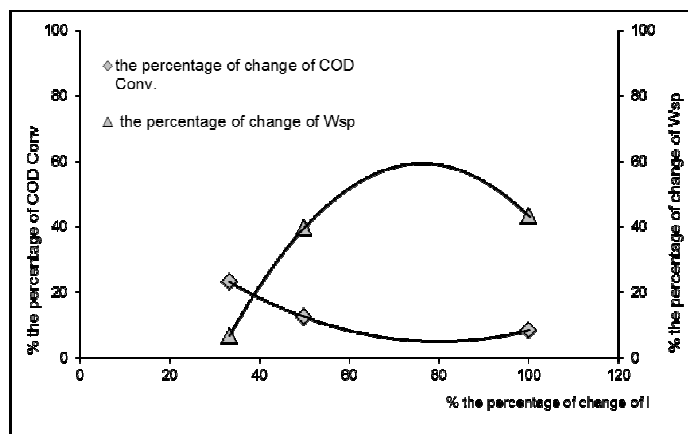
Fig. 9 Change percentage of COD Conversion and Wsp

5-2. Effect of current density on the electrode and energy consumption for one unit of CODremoved:

It is clear from Fig. 10 again that the curves intersect at i = 41.66 mA/cm^2 (at which the surface of the electrode S=96 cm2 and dimensions 8×6 cm.) which is the best current density.

From the above mentioned, it can be concluded that no relationship between the effective surface area and the amount of consumed aluminum. It remains constant in theory and practice because of the stability in current intensity I. We can relate a simple increase in the amount of consumed aluminum in practice with the increase in the intensity of the current to the significant rise in temperature with increasing i (due to the increase of the voltage U). This leads to more rapid aluminum oxide dissolution (virtually impermeable layer formed on the surface of the electrode), and is also confirmed by the increased consumption of the cathode metal with increasing i. This means the possibility of interaction of aluminum ion Al^{+3} on the surface of the runway, since the analysis of the electric barrier without being separated from the region cathode anode.
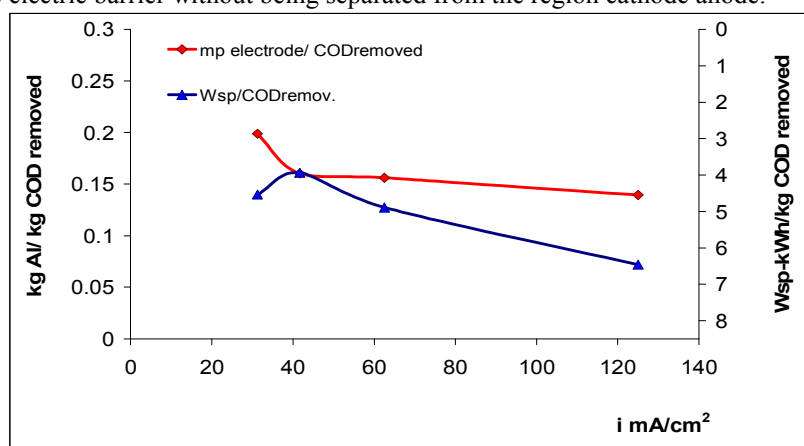

Fig.10 Effect of current density on the electrode and energy consumption

Table 3 summarizes the qualities of OMW before and after treatment under optimum conditions.
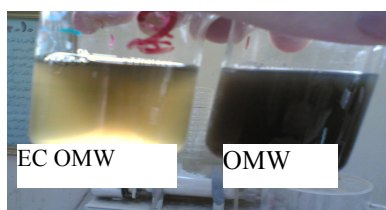
Table 3 specifications of the sample before and after treatment

Parameter	OMW	ECOMW	unit	Abatement %
Temperature	22	46	°C	-
Color *100	82	36	Pt Co	56
TSS	2	0.4	g/L	80
pH	3.96	4.52	-	-
Turbidity* 100	25.9	2.25	FAU	91
CODraw	34.4	26.6	g/L	22.7
TOC	9.15	6.54	g/L	28.5
BOD5	10.5	20.5	g/L	-
Cl^-	1020	570	mg Cl^-/L	44
$PO4^-3$	301	18	mg $PO4^-3$/L	94.02
$NO3^-$	50	40	mg $NO3^-$/L	20
NH3- N	169	94	mg NH3-N/L	44.38
Cu	4	2.5	mg Cu/L	37.5
phenol	44.3	39.2	mg/L	11.5
Tannin and lignin	1.85	1.4	g/L	24.3
Volatile acids	9	6.6	g/L	26.7
Dry residue	15.35	13.66	g/L	11
TS	6.75	5.47	g/L	18.9
TNb	1178	0	mg/L	100

4. Conclusion:

This research has shown that electrocoagulation treatment achieves a fast and effective reduction of pollutants 22.7 % of COD, 56 % of dark color, 80 % of TSS, 91 % of turbidity, 28.5 % of TOC, 11.5 % of Phenol, 24.3 % of Tannin and lignin, 26.7 % of Volatile acids, 100 % of TIC and 100 % of TNb present in fresh olive mill wastewater.

The optimum value of current density was found to be 41.66 $mAcm^{-2}$, and the electrode consumption to be 0.11 kgAl/kg CODremoved.of treated OMW.

The optimum operating conditions were 30 min of treatment, and 3.93 kWh/kg CODremoved.

Biodegradability is measured according to the ratio between the biochemical oxygen demand BOD5 and COD. As mentioned above, the soluble COD of OMW drops to approximately 22.7 % of the initial value, whereas, the BOD5 value increased from 10.5 to 20.5 g L^{-1}. Thus, BOD5/COD ratio increased from 0.33 to 0.77 after treatment. It appears therefore that a significant proportion of the non-biodegradable matter present in OMW was removed by electrocoagulation.

Consequently, electrocoagulation can be considered as a suitable alternative to existing methods or applied as pre-treatment step of biological and advanced oxidation processes used for the treatment of OMW.

Abbreviations: BOD5, biological oxygen demand; COD, chemical oxygen; demand; EC, electrocoagulation; OMW, olive mill wastewater; TSS, total suspended solids; TOC, total organic carbon; TNb, total nitrogen bound; TIC, total inorganic carbon; TDS, total dissolved solids.

Acknowledgment

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