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Technical Note

Electrocoagulation of wastewater from almond industry

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ABSTRACT

This work was carried out to study the treatment of almond industry wastewater by the electrocoagulation process. First of all, laboratory scale experiments were conducted in order to determine the effects of relevant wastewater characteristics such as conductivity and pH, as well as the process variables such as anode material, current density and operating time on the removal efficiencies of the total organic carbon (TOC) and the most representative analytical parameters. Next, the wastewater treatment process was scaled up to pre-industrial size using the best experimental conditions and parameters obtained at laboratory scale. Finally, economic parameters such as chemicals, energy consumption and sludge generation have been discussed.

1. Introduction

Almond industry, which is an important part of the food industry, uses almonds as raw material for the manufacturing of different products such as nougats, snacks and bakery products. Global production of almonds is estimated to be around 1.7 million tonnes per year, 50% of them are produced in California and 10% in Spain, whereas the rest is mainly distributed among other Mediterranean countries (USDA, 2004).

The industrial processing of almonds includes several steps, among them cracking and blanching. The organic content of the wastewater from the blanching step is high and for this reason, the wastewater must be treated before its reuse or discharge to sewage systems. Moreover, almond industry is seasonal, working mainly during the harvest season and for this reason the wastewater treatment facilities must cope with a huge amount of wastewater during short periods of time.

The wastewater generated can be treated using different techniques which should allow either its reuse or its direct disposal into the sewage system. Some of these techniques are evaporation, which has high energy consumption, or physical–chemical treatment that sometimes cannot achieve a satisfactory level of purification.

Nowadays, the social concern about the environmental impact caused by industry is growing and new laws demanding more strict environmental protection are being approved. For this reason the search of "greener" and more efficient methods for wastewater treatment is increasing (Lancaster, 2002). Among the different techniques for wastewater treatment, electrochemical methods have achieved a relevant place (Rajeshwar and Ibanez, 1997; Chen, 2004; Martínez-Huitle and Brillas, 2008). In particular, the oxidation of organic pollutants either by anodic oxidation using different electrodes (Iniesta et al., 2001, 2002; Andrade et al., 2007; Panizza and Cerisola, 2009) or by cathodic generation of hydrogen peroxide (Oturan et al., 2000; Expósito et al., 2007; Sánchez-Sánchez et al., 2007) and desalination among other membrane methods (Ortiz et al., 2005; Banasiak et al., 2007).

Electrocoagulation (EC) is an electrochemical technique closely related to chemical coagulation, that involves the supply of coagulant ions (Al^{3+}, Fe^{3+}) by application of an electric current to a sacrificial anode (made of aluminium or iron) placed into a process tank. Electrocoagulation theory and fundamentals are well described in bibliography (Chen, 2004; Mollah et al., 2004). The metallic ions produced by the corrosion of Al or Fe behave in a similar way to the aluminium or ferric ions employed in chemical coagulation. However, the characteristics of the particle aggregates (flocs) generated during the electrocoagulation process differ dramatically from those generated by chemical coagulation. Thus, flocs generated in an electrocoagulation process tend to contain less bound water and are more shear resistant and are more readily filterable. Other advantages of electrocoagulation over chemical coagulation are: the amount of chemicals needed is lower, the salinity of the wastewater does not increase and the economic cost for the treatment is also lower. In addition, the gas bubbles generated at the cathode can contribute to the flotation of the flocs,

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which can be easily recovered. Finally, at the end of the process the generated sludge is subsequently separated by filtration.

Although EC has been implemented for most of the 20th century with limited success and popularity, this technology has been increasingly used for treatment of industrial wastewaters from different origins in the last decade (Mollah et al., 2004; Holt et al., 2005). Thus, EC has been applied to treat wastewaters containing food waste (Hanafia et al., 2010), dyes (Martínez-Huitle and Brillas, 2009), oil, suspended particles, etc. (Xu and Zhu, 2004; Can et al., 2006; Cañizares et al., 2008a,b). The electric energy needed for the corrosion of Al or Fe can be obtained from classical or renewable electricity sources (Valero et al., 2008).

The aim of this paper is to demonstrate that electrocoagulation is a suitable technology for the treatment of wastewater generated in the almond industry. First, the influence of experimental parameters such as anode material, pH and current density on the removal of pollutants was studied at laboratory bench scale. Next, using the best experimental conditions and parameters obtained at this scale, the wastewater treatment process was scaled up to pre-industrial size using an electrocoagulation system developed by our Research Group in collaboration with the company Essentium Depuración S.L. This last study allowed us to know and to extrapolate to industrial scale the values of removal efficiency and economic figures of merit such as energy consumption, the replacement cost of the electrodes, the amount of chemicals, the ratio kg of sludge generated/volume of wastewater treated, at a scale that allowed us to know fairly accurately the results that could be obtained using an electrocoagulation system on an industrial scale.

2. Experimental

2.1. Wastewater description

In order to carry out this work, samples of wastewater from an almond industry located in the Southeast of Spain were used. Wastewater samples were taken from a homogenisation tank placed after the blanching process. The wastewater samples were brown, murky and contained a large amount of suspended solids. The samples used in the laboratory and pilot-scale studies had different composition since they were taken from the homogenisation tank on different days.

2.2. Electrocoagulation at laboratory bench scale

EC experiments were performed in batch mode. The laboratory EC cell consisted of a methacrylate parallelepiped tank, where the electrodic stack was submerged. The electrode stack consisted of four parallel-plate electrodes: two aluminium electrodes alternating with two iron electrodes. When aluminium electrodes acted as anodes, iron electrodes were the cathodes and viceversa. The dimensions of the electrodes were 130 mm × 90 mm × 2 mm. The total anodic area was 351 cm^2, the inter-electrodic gap was 1 cm and the volume of the tank was 700 cm^3. Experiments were performed at room temperature. A magnetic stirrer was used to agitate the solution at constant agitation speed. The duration of the experiments was 30 min and samples were taken every 5 min. A solution of floculant was added to form the flocs and the last step of the process was a filtration using filtration paper. TOC was measured for every sample, whereas for the initial and final samples pH, conductivity and temperature were also measured.

For EC laboratory experiments, two DC-power sources (Blausonic 0–30 V 2.5 A DC, and EA PS power supply 0–16 V 10 A DC) were employed. pH was measured using a CRISON microph 2000, whereas conductivity was measured using a CRISON 525 conductivity metre.

To carry out the laboratory experiments, pH was adjusted by adding the necessary amount of 5% wt NaOH and 96% wt H_2SO_4. Chemicals used were 99% wt NaOH Panreac PRS and 96% wt H_2SO_4 Panreac PRS. NaCl PANREAC QP was used to adjust conductivity values. A 2 g L^{-1} solution of Dalfloc 13D was used as flocculant.

2.3. Electrocoagulation at pre-industrial scale

In this stage of the work an EC plant working in continuous mode of operation was used. The EC system was designed and built by our research group in collaboration with the company Essentium Depuración S.L., which is currently commercialising these EC systems (with a treatment capacity in the range 1–4 m^3 h^{-1}), under the acronym TREAC. The wastewater treatment capacity of our system was 1 m^3 h^{-1}. Fig. 1 shows the flow chart of the TREAC–EC system.

The efficiency of a wastewater treatment process by electrocoagulation is strongly influenced by the physical–chemical parameters of the solution such as the types of contaminants, pH and electrical conductivity. Because of this, the first unit operation of the TREAC system is the preconditioning of the wastewater – pH and/or electrical conductivity –, before electrocoagulation. TREAC systems perform this task in a continuous way by means of a homogenisation tank (see **D-4** in Fig. 1), a microprocessor PID (Proportional–Integral–Derivative) controller for pH and conductivity and acid/base and brine dosing pumps **P-2** and **P-3**. Next, a centrifugal pump **P-4** and a flow metre for the extraction and circulation at controlled flow of the conditioned wastewater are used.

The next step of the process is the electrocoagulation of the conditioned wastewater. The electrocoagulation cell **E-1** consisted of a parallelepiped-shaped tank −85 cm long, 25 cm wide, and 95 cm high-made of polypropylene. The upper side of the EC cell was open to provide access to the electrode stack. The conditioned wastewater circulated through the electrocoagulation cell with an upward flow, while electric current flows through the electrode stack. Finally, the electrocoagulated wastewater overflowed from the top of the EC cell into a drain.

The electrode stack consists of 40 parallel plate electrodes connected in monopolar mode: 20 aluminium electrodes (anodes) alternating with 20 iron electrodes (cathodes). The dimensions of the electrodes are 48.5 cm × 23.0 cm × 0.4 cm, the total anodic area is 4.46 m^2 and the inter-electrodic gap 1.5 cm. The electrical connection is monopolar and the DC-power is a Sidasa 10 V-250 A Quasar Q500.

The next unit operation is the flocculation of the electrocoagulated wastewater. This stage takes place in an auxiliary tank **D-6**, to where the electrocoagulated wastewater overflows by gravity from the top of the EC cell. Solution 1 g L^{-1} Dalfloc 13D is added by means of a dosing pump **P-5** (EMEC FIC 0505). The amount of flocculant added was 5 g m^{-3}. The tank is provided with a stirrer with paddle shaft and blade linked to a variable speed motor to allow homogenisation. After this, the flocculated wastewater is sent to a sedimentation tank **E-3,** where macroflocs are separated from the treated wastewater. Macroflocs sediment at the bottom of the tank forms sludge, while treated wastewater overflows from the top of the sedimentation tank.

The last step of the process is the filtration of the generated sludges. A pneumatic pump **P-7** sends the sludge to a filter press **E-4**, using Polypropylene multifilament filter cloth, where treated wastewater and wet sludge are obtained.

The plant was controlled using a PLC-based (Programmable Logic Controller) control system that automatically compensates

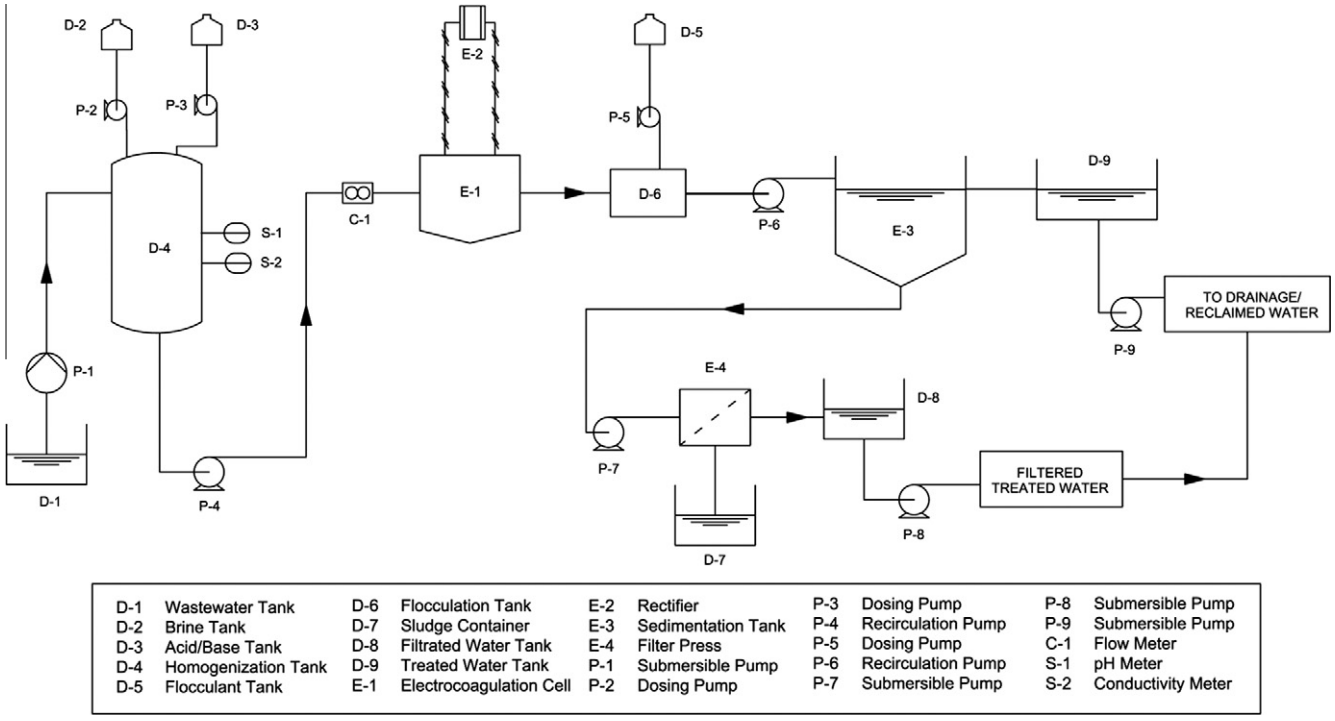

Fig. 1. Flow diagram of the EC system.

for changes in various parameters of the process, operates the system automatically and stops it in case of any emergency situation.

2.4. Analytical techniques

Total Organic Carbon (TOC) was measured by a SHIMADZU TOC-V CSH analyzer. COD, total N and the rest of analytical parameters were measured by a HACH DR 2000 spectrophotometer using colorimetric methods. The method used for COD determination needs a digestion in sulphuric acid at 150 °C and includes $HgSO_4$ in order to reduce chloride interferences in the measurement.

The method used for determining metals in water included a digestion and detection by ICP. For determining metals in sludge sample, the method included an acid digestion in MW and detection by ICP. The microwave oven was an Anton Paar Multiwave 3000 and ICP was a Varian Liberty II Axial.

Table 1
Analytical results of a sample treated using the best experimental conditions. FTU: Formazin turbidity units; TSS: total suspended solids; ND: non-detected.

	Raw wastewater	Treated water	% Removal
pH	5.7	6.1	–
Conductivity (mS cm^{-1})	2.1 (23 °C)	2.2 (25 °C)	–
TOC (ppm)	1400	360	74
COD (ppm)	5300	1000	81
BOD5 (ppm)	1000	200	80
Total nitrogen (ppm)	240	60	75
Total phosphorous (ppm)	3.0	0.01	99.7
Apparent colour (Pt–Co units)	18 000	80	99.6
TSS (ppm)	3400	10	99.7
Turbidity (FTU units)	3200	20	99.4
Heavy metals			
(Cd, Co, Cr, Hg, Ni, Sn, Ti, Pb)	ND	ND	–

3. Results and discussion

3.1. Laboratory-scale study

As it was previously stated, the first step of our work was to study the influence at laboratory scale of experimental parameters such as anode material, pH, current density and reaction time, on the removal efficiency of the electrocoagulation process. Table 1 shows the values of the analytical parameters of interest of the initial samples. COD and TOC follow similar elimination curves and as TOC analysis are faster, more accurate and require less manipulation of the samples, TOC removal was selected as the main performance criteria in our study.

Several series of experiments were carried out to study the electrocoagulation process as a function of the operating conditions. The sacrificial anode material and solution pH are the most important experimental parameters in an electrocoagulation process. In this work, Al and Fe were tested as sacrificial anodes. Fig. 2a and b show the %TOC removal in experiments carried out at several initial pH values using aluminium and iron anodes, respectively. All the experiments were carried out at j (current density) = 5 mA cm^{-2} and cell voltage remained constant during the experiments. Figures show that for both anodes, the electrocoagulation of raw wastewater (pH = 5.7 with no pH adjustment) displays the best results of %TOC removal. This is a very convenient result because it shows that there is not need of chemicals addition to change the initial pH of the wastewater. The results obtained at pH = 5.7 (initial pH) were very similar for both anodes. It should be pointed out that a thick layer of brown foam is formed during the electrocoagulation and disappears during the filtration step. The colour of the filtered solution ranges from transparent to pale yellow. It is important to note that in experiments where iron was used as anode, a small concentration of Fe^{2+} remained in solution after the filtration of the electrocoagulated wastewater. The Fe^{2+} ion was slowly oxidised to Fe^{3+} by the dissolved oxygen. Thus, after a few hours, a red precipitate of ferric oxide appeared in the filtered solution that became murky. In spite of the similar behav-

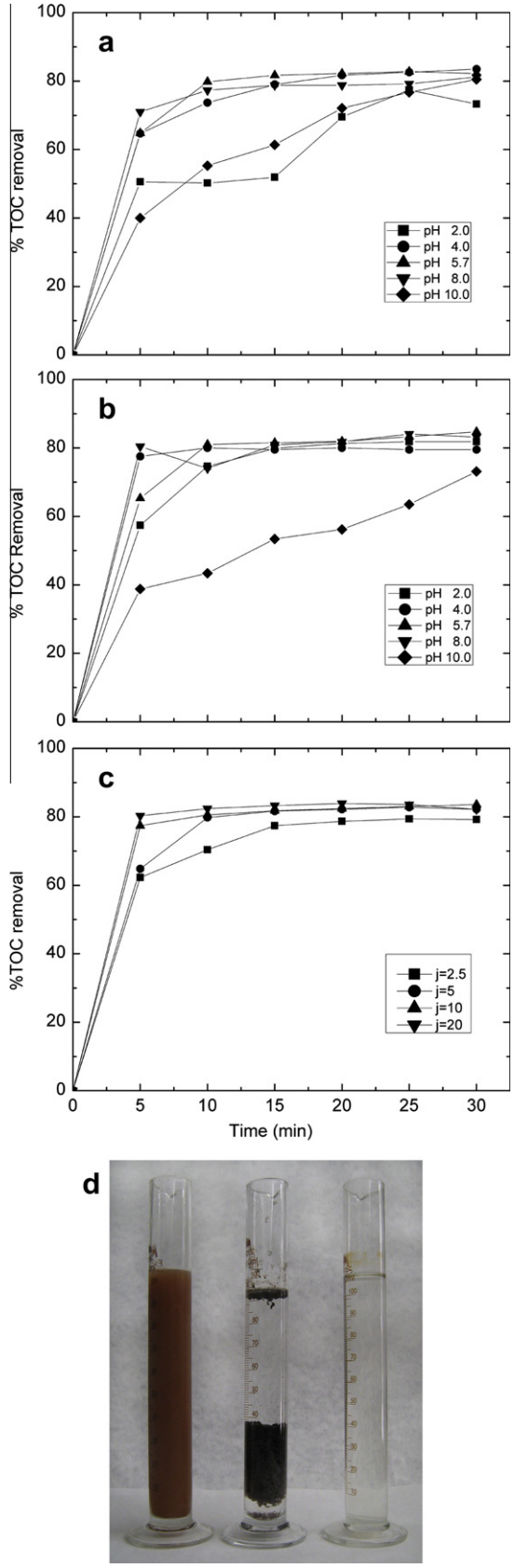

Fig. 2. %Toc removal vs time at different: (a) initial pH values using an Al anode. (b) initial pH values using a Fe anode and (c) current density values in mA cm^{-2} using an Al anode. (d) picture of: raw (left), electrocoagulated (middle) and treated and filtered wastewater (right).

iour, aluminium was selected as anode material because apart from this change of colouration there are other practical considerations that make more interesting its use instead of Fe (lower weight, longer life and better performance after periods of prolonged inactivity).

The feasibility of the wastewater treatment without pH adjustment has some important practical advantages: (i) saving of chemicals for pH adjustment, (ii) the conductivity of treated water does not increase because of the addition of chemicals, and (iii) simplifies the flow chart of the EC plant, reducing the investment costs (pumps, tanks, pH control, etc.).

Next, the influence of the working current density on the efficiency of the treatment was studied. To this end, a series of experiments at several values of j were carried out at initial pH and using Al anodes. The results of these experiments are shown in Fig. 2c.

The figure shows that at the beginning of the experiments, %TOC removal is greater at higher values of current density. However, for values of $j \geqslant 5$ mA cm^{-2}, TOC removal attains a constant and minimum value after 15 min of treatment. Since higher values of current density do not improve the removal yield but increase the electric cost of the electrocoagulation process, a current density of 5 mA cm^{-2} was considered as an optimum value.

From Fig. 2, it can be deduced that for all the experimental conditions studied, a reaction time of 15 min is long enough to complete the reaction. From this time value, asymptotic %TOC removal values are reached, and an increase of the electrocoagulation time only gives rise to the increase of the energy consumption of the process. According to the obtained results, the best working conditions for the treatment of these wastewaters are the following: aluminium anodes, $t = 15$ min, initial pH = 5.7 and $j = 5$ mA cm^{-2}. Fig. 2d shows the appearance of the wastewater treated under the chosen conditions: before the treatment (left); after the electrocoagulation process (middle), where flocs have been formed; and finally, once they have been filtered and the process is finished (right). Table 1 shows the analytical results of a sample treated under these experimental conditions.

3.2. Pre-industrial scale study

In the previous section, the best experimental conditions to carry out the electrocoagulation treatment of this wastewater have been established. In this section, the scaling-up of the electrocoagulation process to a pre-industrial size will be performed using these experimental conditions and the experimental results obtained are shown in Table 2. The ratio between current intensity (A), and flow (L h^{-1}) indicates the amount of electrical charge passed per unit of volume of treated wastewater and therefore the amount of aluminium consumed according to the Faraday law if neither parasitic reactions nor chemical corrosion of the anode exist. Other important parameters are the pH and conductivity of both initial and treated wastewaters. Cell voltage was constant during the reactions. Finally, TOC values were used as an indicator of the level of decontamination of the wastewater.

As shown in Table 2, in EXPERIMENTS 1 and 2 the conductivity of the wastewater was not adjusted by addition of electrolyte. These experiments were carried out for the same flow and for two different current densities. The table shows that the higher the current density, the higher the%TOC removal. However, this improvement in the removal of organic matter was produced at significantly higher values of both, cell voltage and energy consumption.

Since the energy consumption of the EC cell is directly related to the conductivity of the wastewater, in order to decrease the ohmic resistance of the wastewater the conductivity was increased in EXPERIMENTS 3–6 up to values allowed by the existing legislation

Table 2
Experimental results obtained using the pre-industrial scale EC system.

Experiments Id.°	1	2	3	4	5	6
Flow (L h^{-1})	250	250	250	325	400	625
Current intensity (A)	125	185	200	200	200	400
Current density (mA cm^{-2})	2.8	4.1	4.5	4.5	4.5	9.0
Residence time (min)	13.0	13.0	13.0	10.0	8.2	5.2
Final pH	6.9	7.0	6.6	6.4	6.5	6.6
Initial conductivity (mS cm^{-1}, 25 °C)	2.1	2.1	3.2	3.2	3.2	3.2
Final conductivity (mS cm^{-1}, 25 °C)	2.2	2.2	3.5	3.4	3.7	3.3
Voltage EC (V)	6.5	9.4	5.6	5.6	5.6	10.0
Energy consumption (kW h m^{-3})	3.2	6.9	4.4	3.4	2.8	12.3
Electrode mass loss (kg m^{-3})	0.17	0.25	0.27	0.21	0.17	0.21
Initial TOC (ppm)	2230	2230	2260	2260	2260	2260
Final TOC (ppm)	550	440	450	470	470	490
%TOC removal	75	80	80	79	79	78

Table 3
Analytical results of EXPERIMENT 2 sample. FTU: Formazin turbidity units; TSS: total suspended solids; ND: non-detected.

	Raw wastewater	Treated water	% Removal
pH	5.7	6.5	–
Conductivity (mS cm^{-1})	2.3 (26 °C)	3.2 (26 °C)	–
TOC (ppm)	2260	470	79
COD (ppm)	6300	1200	81
BOD5 (ppm)	1200	400	67
Total nitrogen (ppm)	330	50	85
Total phosphorous (ppm)	4	0.03	99.3
Apparent colour (Pt–Co units)	22 500	530	98
TSS (ppm)	4200	30	99.3
Turbidity (FTU units)	4000	100	98
Heavy metals			
(Cd, Co, Cr, Hg, Ni, Sn, Ti, Pb)	ND	ND	–

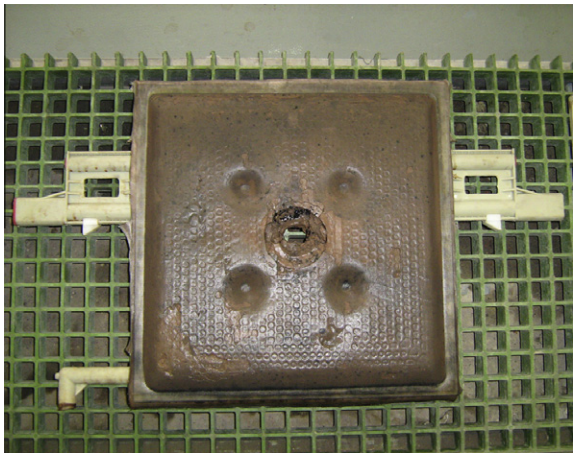

Fig. 3. Photograph of a filter press plate after the filtration step.

of wastewater discharges in Spain (Iglesias et al., 2010). Sodium chloride was used and the amount needed was 0.41 kg NaCl m^{-3}.

EXPERIMENTS 3–5 were performed at the same current density value (4.5 mA cm^{-2}) and different flow values. EXPERIMENTS 4 and 6 were carried out by maintaining a constant ratio of the current intensity (A) to the flow (L h^{-1}). Table 2 shows that %TOC removal was approximately 80% in all the experiments. Table 3 shows the complete analytical results for EXPERIMENT 2.

Fig. 3 shows a photograph of a filter press plate with the filtered sludge on a filter press plate (final filtration pressure 4 bar). Both mechanical properties and moisture content of the sludge were suitable for gravity separation after filter press opening. The sludge production was about 11.4 kg of wet sludge per cubic metre of treated wastewater. Otherwise, the sludge produced is non-toxic with neutral pH and can be managed as a non-hazardous solid waste. Table 4 shows the analytical results of a sludge sample.

Table 4
Analytical results of a sludge sample.

pH	6.1
Moisture (%)	77
Calcination, organic matter (wet sludge) (%)	18
Calcination, organic matter (dry sludge) (%)	78
Total nitrogen (g kg^{-1})	3
Total phosphorous (g kg^{-1})	0.3
Heavy metals	
(Cd, Co, Cr, Hg, Ni, Sn, Ti, Pb)	ND

4. Conclusions

The results of the laboratory tests presented here show that the electrocoagulation process is currently an effective technology for treating wastewater from the almond industry because: (i) the analytical parameters of the treated water satisfy wastewater discharge legislation and (ii) the sludge generated is neutral, non-toxic and can be recovered for agricultural purposes. Our work has proved that this technology can be transferred to industrial scale solving the problem of disposal of the wastewater produced in the processing of almonds, which due to its high level of contamination cannot be directly disposed in the sewage system.

Electrocoagulation requires minimal amounts of chemical reagents for the treatment of this type of wastewater: (i) no pH adjustment is required, and (ii) conductivity adjustment is not necessary, although is recommended in order to reduce the energy consumption of the process. Indeed, it could be assumed that the main chemical reagent in electrocoagulation technology is the electron, which is an inexpensive, safe, environmentally friendly reagent and does not require storage.

Moreover, the developed pre-industrial pilot electrocoagulation reactor is compact, versatile, energy efficient, robust and easy to automate.

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