

Article

Characterization of Sludge Resulting from Chemical Coagulation and Electrocoagulation of Pumping Water from Fishmeal Factories

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Abstract: In the fishmeal industry, seawater is used to transport fish to the factories. Due to this, “pumping water” with high concentrations of organic matter is generated that is treated via chemical coagulation before letting it into the sea. The objective of this study is to characterize and compare the sludge obtained from conventional chemical coagulation and the sludge from the electrocoagulation process. A pilot electrocoagulation plant was built next to a chemical coagulation plant. The sludge obtained from both methodologies was analyzed for its proximal composition, its iron and aluminum content, and the fatty acid profile in its contained fat. Electrocoagulation was found to produce sludge with a higher concentration of lipids and ash, which indirectly confirmed that it removes more organic pollutants and salts than chemical coagulation. The contents of aluminum and iron in the sludge obtained by electrocoagulation were 4.2% and 0.025%, respectively, while those in the sludge obtained from chemical coagulation were 0.01% and 4.8%, respectively. Aluminum comes from the sacrificial electrode of the electrocoagulation tank, while iron comes from the salts used in chemical coagulation. The sum of w-3 fatty acid values (EPA + DHA) was 12.5% and 18.8% for sludges from the electrocoagulation and chemical coagulation processes, respectively, so we can assume that electrocoagulation is a more oxidizing process than chemical coagulation. Due to their high organic load, both sludges must be assessed as an alternative feed ingredient.

Keywords: electrocoagulation; pumping water fishmeal industry; sludge from pumping water; chemical coagulation



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1. Introduction

In the fishmeal industry, ~30% of the world’s production comes from Peru and primarily comprises anchovy (*Engraulis ringens*) [1]. The transport and discharge of water fish from the boats to the processing plants, after sieving, is called “pumping water” (PW) [2]. On average, the process uses two volumes of seawater for every volume of fish transported [3,4]. However, updated reports show this ratio has turned out to be 1. Depending on the capture time and the subsequent treatment of fish, PW can contain between 9000 and 30,000 mg/L of Total Suspended Solids (TSS) and between 400 and 650 mg/L of Fats, Oils and Grease (FOG) [5,6]. Given that the annual anchovy catch had an average annual landing of 4.21 million tons during the last decade [7], these are significant statistics to consider.

Before returning to the sea, PW must be treated to reduce its organic material content, and thus, avoid pollution. The treatment of PW includes several consecutive processes (Figure 1). Initially, the fish is unloaded from the boat and taken to the processing plant. Once inside the plant, water is removed via rotating filters that recover scales and larger solids incorporated into fishmeal production. Then, water is taken to inclined plane pools, “grease traps”, where grease is recovered by flotation at a water flow rate of 200–250 m³/h. The water that comes from the grease traps is stored in an “equalizer tank”, from where it can be diverted to discharge by recirculation, depending on its physicochemical parameters, making efficient use of the water resource. In these instances, the water passes to a Dissolved Air Flotation “DAF” system to continue the recovery of lipophilic foams. Then, the PW is sent to the clarifier, where chemical agents are added to allow the coagulation and flocculation of the solids present in the water, called the “Chemical Coagulation Process” (CCP), which, by the action of injected air microbubbles, allow flocs to float and be collected and taken to centrifugal decanters where the solids are separated to form the sludge [8]. Once clarified, the water is returned to the sea, complying with government regulations that establish values that should not exceed the Maximum Permissible Limit (MPL): 700 mg/L of TSS and 350 mg/L of FOG [9].

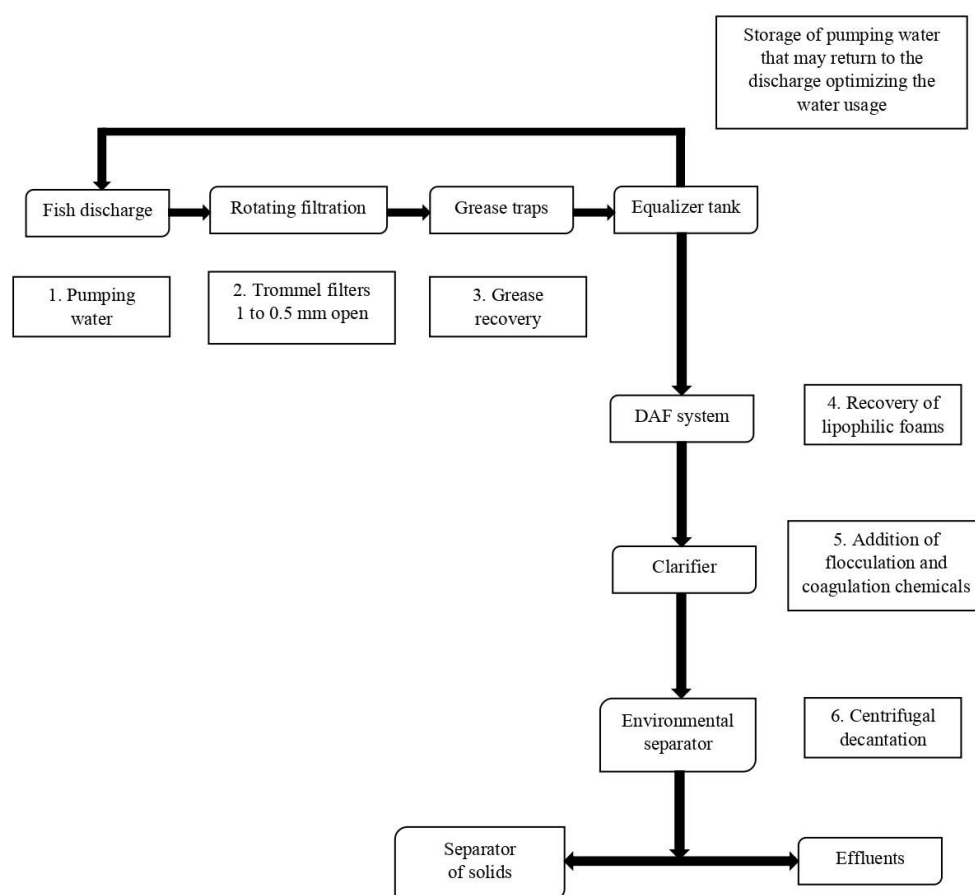


Figure 1. Scheme for the fish PW treatment system. Source: Pesquera Diamante.

The most studied coagulation/flocculation chemical agents in the fishmeal industry are iron salts, which also reduce the pH of the medium and bring most proteins to its isoelectric point, increasing their precipitation [10]. Currently, for the chemical treatment of PW, ferric sulfate and polyelectrolytes of an anionic or cationic nature are added for the destabilization and flocculation of proteins [11]. Chemical agent application increases treatment costs and encourages the recovery of iron-rich sludge, which has only a limited value.

An alternative method to the CCP is electrocoagulation (EC), where the electric current destabilizes the polluting particles in the water through metal plates used as electrodes, with iron and aluminum being the most used elements due to their environmental and economic advantages [12,13]. In a sacrificial electrode, the metal ions are released and dispersed in the liquid medium. They tend to form metal hydroxides that electrically attract the polluting solids that have been destabilized.

Flocs larger than those observed in the CCP are formed [14,15]. Because of this, they precipitate or float, forming sludges which contain the polluting material and are easier to dehydrate with other secondary processes [16,17]. In addition, hydrogen and oxygen bubbles are generated on the electrodes, which help lift the coagulated particles via electroflotation [18,19].

Compared to CCP, EC provides a compact and robust treatment alternative where electrochemical reactions are started at the anode that provides active metal cations for coagulation and flocculation and where the resulting use of high-cost chemicals and secondary pollution is avoided [13].

In the search for alternative systems for treating PW from fishmeal factories, the EC process has been proposed as a more economical system [14]. Previous studies have shown that it reduces the organic levels in residues from fish processing. However, on a pilot scale, this treatment did not turn out to be as effective as initially anticipated Beck et al. 1974, mentioned by [8]. Recent improvements to the EC processes are again making this technology an alternative to treating this type of water [12,13].

Pumping water in fishmeal factories usually comes from the sea, where NaCl is higher than other industrial wastewater. These ionic strength conditions favor conductivity in the EC process and reduce the formation of calcium carbonate-type anions that can precipitate on the cathode surface and increase ohmic resistance [20]. The conductivity of the PW varies between 20,000 and 50,000 uS/cm. For this reason, this technology must be adapted to avoid high demands for electric current and excessive electrode consumption in the electrocoagulation process. Recent studies [21] removed organic material in PW using a laboratory-level EC system. They sufficiently complied with the limits proposed by the Peruvian legal regulations for treated water [9].

As environmental restrictions become stringent, there is a growing interest in researching wastewater sludge from water treatment to assess its potential use and recovery within a circular economy model. This would result in cheaper costs and sufficient final waste disposal [22].

In this regard, high concentrations of silica, aluminum, ferric oxide, lime, and heavy metals have been found in the sludge from CCP drinking water plants. Thus, a safe disposal route for this sludge would be manufacturing bricks or their use as a substitute for building materials [23]. Alternatively, a product has been obtained from sludge treated with an alum; the said product can remove colloidal suspensions and apply them to river water [24]. Regarding the use and reassessment of sludge obtained by EC, it has been proposed as a partial substitute for cement [25], used for the adsorption of dyes in aqueous solutions [26] and the manufacture of alumina microcapsules for corrosion protection [27].

PW has a high conductivity due to dissolved salts and high concentrations of organic substances. In previous studies at the laboratory level [1], EC technology managed to remove 99% of TSS under conditions of $I = 13$ A and $t = 30$ min, generating sludge that contained concentrations of 0.3% of iron and 1.7% aluminum, which can be considered as a technology for the purification of this type of water and generating sludge that can serve as a feed component. Thus, to use EC technology in PW, a pilot electrocoagulation system was designed and built to evaluate the efficiency of solids removal in PW while characterizing the sludge obtained to evaluate its possible use as feed and compare them with sludges obtained by the CCP.

2. Materials and Methods

2.1. Electrocoagulation Pilot System

The system consists of an equalizer tank with a 0.6 m³ capacity, where the PW is received and homogenized. This water is sent to the reaction tanks, where aluminum electrodes are located (three tanks connected in series). The electric current used is continuous and was supplied to the electrodes with a power source with a capacity of up to 100 A. After passing through the electrocoagulation process of the third tank, the water is diverted to a clarifying tank for the final sedimentation process and then sent to the discharge point by pumping. The start-up of the loading and unloading pumps, as well as the retention time in the reactor tanks, are monitored with a control panel. The sludge (recovered solids) formed managed to float as foam in the three tanks until overflowed by the action of the microbubbles generated in the electrodes. These foams are collected to obtain the sludge (Figure 2A,B).

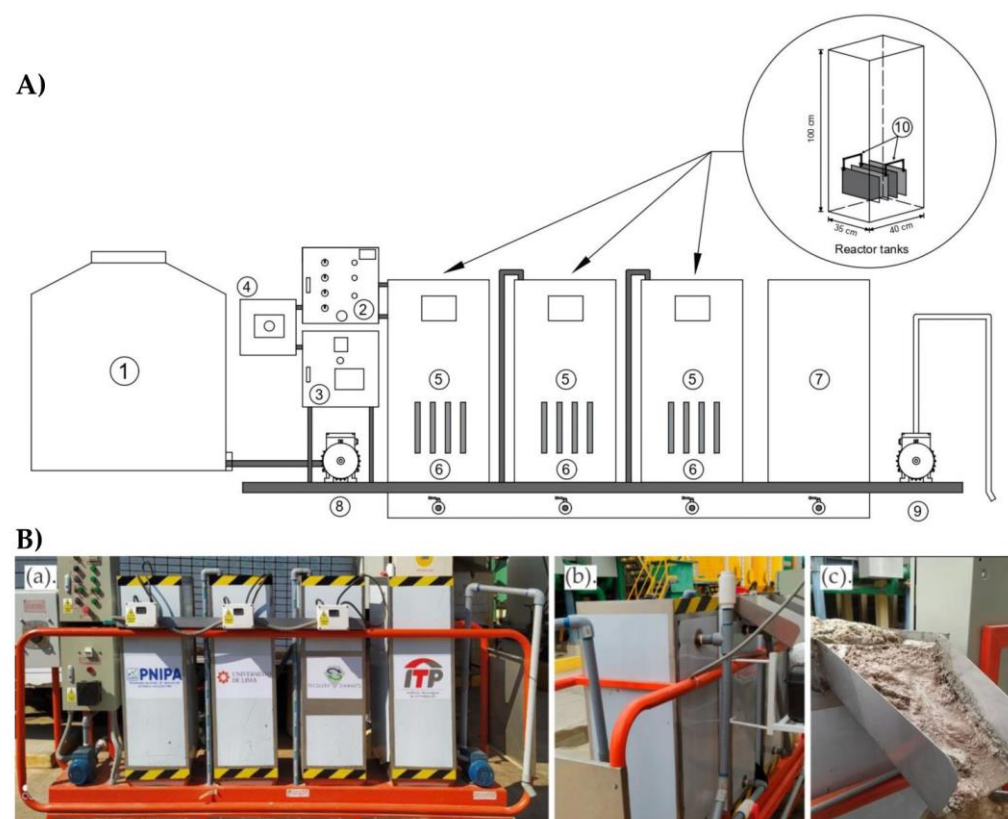


Figure 2. (A): Scheme of the electrocoagulation pilot plant. (1) Equalizer tank. (2) Control board. (3) Power source. (4) Voltage variator. (5) Reactor tanks. (6) Aluminum electrodes. (7) Clarifying tank. (8) Charging pump. (9) Discharge pump. (10) Cable connection. (B): Electrocoagulation pilot plant images. (a) Image of front. (b) Back image. (c) Sludge collector.

2.2. Obtaining and Preparing Samples

The samples were obtained during the first week of October 2021. To obtain sludge by EC, the PW was placed in the equalizer tank, then sent to the reactor tanks at a flow of 125 L/h and a current of 26 A (Figure 2) so that the PW in the successive reactor tanks had an increasingly lower concentration of suspended solids to comply with MPL [9]. The time to treat 600 L of PW contained in the equalizing tank was 3 h. Simultaneously, for each batch, the sludge from the CCP was obtained from the separator centrifuge PW treatment plant (Figure 1). The sludge generated by the EC floats in the form of foam due to the action of the microbubbles generated by the electrodes. These foams increased until they overflowed from the reactor tanks and were collected in polyethylene bags and placed in

thermal boxes at 4 °C, where they returned to a liquid state. These samples were sent to the laboratory and centrifuged at 2000 rpm for 15 min. The supernatant liquid was discarded, and the precipitated sludge was collected.

The sludge from both processes was spread on nylon meshes placed on aluminum trays and dried at 70 °C for 18 h in an oven with forced air convection (VENTICELL®). The dried samples were ground and sieved through a mesh of <1 mm, bagged in polyethylene bags and stored at room temperature (20 °C ± 2) for 30 days before being analyzed. The samples were called LCQ (chemical coagulation sludge) and LEC (electrocoagulation sludge).

2.3. Physicochemical Tests

The proximate composition of LCQ and LEC was conducted according to the methodologies recommended by the FAO [28]: moisture by oven drying at 104 °C (VENTICELL®) and ash content using a muffle furnace at 600 °C (Barnstead/thermolyne 48000®), both until constant weight. The protein was obtained using the Kjeldahl method using the kjeldatherm/Turbosog/Vapodest® equipment system (C. Gerhardt GmbH & Co. KG Cäsariusstraße 97, 53639 Königswinter, Germany), using a total nitrogen to protein conversion factor of 6.25. The crude fat was realized using hexane as an extraction solvent in Soxtherm® equipment (C. Gerhardt GmbH & Co. KG). All tests were duplicated and expressed in g 100 g⁻¹ of sample.

Iron (Fe) was quantified by atomic absorption spectrometry (AA) following the 999.11 AOAC methodologies [29]. Aluminum quantification was performed by AA on the sample calcined at 600 °C and dissolved in 1N HCl, which was read at 396.2 nm, using a nitrous oxide-acetylene flame following the recommendations of the team [30].

The fatty acid profile was obtained by gas chromatography (Perkin Elmer Autosystem XL GC with FID detector) using a Carbowax 20 M column following the Ce 1f-96 AOCS methodology. The sample analyzed was the fat extracted from the sludge by the Bligh and Dyer methodology [31] before drying it in the oven to avoid oxidation.

The Tukey test verified the differences at 95% confidence intervals using the Minitab® 21 Statistical Software (Chicago, IL, USA).

3. Results and Discussion

The chemical coagulation process (CCP) and electrocoagulation (EC) have been studied and compared with variable results depending on the type of water to be treated, ionic charge, electrical conditions, and pH [13,23,32–35]. A quantitative comparison is not feasible because both operations are different; in CCP, the coagulant is administered discretely, whereas in EC, the addition of the coagulant is a function of time and current [36].

The content of solids and fats in PW varies greatly. It depends on factors after the fish catch, such as time, type and storage temperature, discharge speed, type of pumps used in the discharge, and flow rate of water used in the discharge, among others. The water samples analyzed presented values of about 4800 mg/L for TSS, 265.4 mg/L for FOG, 6.4 for pH value, and 20,500 uS/cm for conductivity.

At the flow rate and current intensity conditions analyzed (125 L/h and 26 A), the treated water obtained in the clarification tank sufficiently met the maximum permissible limits (MPL) required by the regulations, as it presented values of 41 mg/L in SST (MPL < 700 mg/L), 2 mg/L FOG (MPL < 350 mg/L), and a pH value of 7.08 (MPL pH 5–9). Even though CCP was accomplished with MPL, reaching values of 417.4 mg/L TSS, 30 mg/L A&G, and a pH value of 5.5, these values were more elevated than EC. These results indicate that EC more effectively removed the contaminants from PW than CCP.

The estimated cost of the EC process was USD 1.24/m³ PW, including electrode consumption and energy cost USD/KWh. The fishing industry works with CCP and reports a cost of around USD 1.5/m³ PW by chemical consumption, energy, and operations.

3.1. Proximate Composition of Sludge

The proximate composition included moisture and served to convert the protein, fat, and ash on a dry basis. The LEC sample's moisture presented lower values than their LCQ pairs (2.33 ± 0.6 vs. 5.32 ± 1.3 , respectively). The higher fat content in LEC proved to be a barrier to the balance with ambient humidity, given that the samples were stored for similar amounts of time. Pronounced differences were observed in the composition of the LCQ and LEC samples (Table 1), and it was confirmed that both types of coagulation operate differently. Under the conditions analyzed, LEC presented a higher fat and ash than LCQ. Furthermore, EC was more effective than CCP in removal of pumping water contaminants, so it is deduced that EC has a better performance than CCP.

Table 1. Proximate composition (dry basis) of sludge flour from PW.

	Batch	% Fat	% Ash	% Proteins
Chemical coagulation sludge (LCQ)	1	13.75 ± 0.40^a	14.14 ± 0.04^a	$41.26 \pm 1.44^{a,b}$
	2	10.23 ± 0.33^b	16.94 ± 0.08^b	$38.72 \pm 0.96^{b,c}$
	3	21.46 ± 0.44^c	13.95 ± 0.09^c	44.17 ± 2.21^a
	4	19.76 ± 0.71^d	13.76 ± 0.03^c	35.98 ± 0.96^c
Electrocoagulation sludge (LEC)	1	24.71 ± 0.54^a	22.89 ± 0.31^b	$35.43 \pm 0.52^{b,c}$
	2	25.07 ± 2.21^a	22.85 ± 0.36^b	34.89 ± 0.61^c
	3	26.19 ± 0.60^a	27.11 ± 0.14^a	39.84 ± 0.70^a
	4	25.41 ± 0.67^a	27.19 ± 0.24^a	$37.59 \pm 1.66^{a,b}$

Different letters in LCQ and LEC columns indicate significant differences ($p < 0.05$). Each batch number worked in LEC had its corresponding sampling in LCQ.

Proximate composition on a dry basis in LCQ samples presented values of 10.2–21.5% fat, 13.8–16.9% ash, and 35.9–44.2% protein. Meanwhile, LEC samples showed 24.7–26.2% fat, 22.8–27.2% ash, and 34.9–39.8% protein. LCQ samples presented variable results and greater significant differences between batches because they represented larger processed volumes, even though they came from the same work period (Table 2: LCQ part). Meanwhile, LEC samples showed more homogeneous results: 24.7–26.2% fat, 22.8–27.2% ash, and 34.9–39.8% protein. They represent 600 L of PW only. We must remember that the chemical coagulation process worked at 150–250 m³/h, while the electrocoagulation process worked at a flow of 0.125 m³/h.

Table 2. Composition of Fe and Al (dry base) in sludge flour from PW.

	Batch	Fe %	Al %
Chemical coagulation sludge (LCQ)	1	4.60 ± 0.08^b	0.01
	2	4.64 ± 0.06^b	0.01
	3	5.31 ± 0.03^a	0.01
	4	4.69 ± 0.13^b	0.01
Electrocoagulation sludge (LEC)	1	0.03	4.12 ± 0.11
	2	0.03	4.31 ± 0.17
	3	0.03	4.36 ± 0.10
	4	0.03	4.02 ± 0.02

Different letters in LCQ and LEC columns indicate significant differences ($p < 0.05$). Each batch analyzed in the electrocoagulation had its corresponding sampling in the chemical coagulation.

There is evidence of a higher percentage of fat and ash content in the LEC samples; consequently, there is a percentage decrease in the protein content (Figure 3). The average results of LCQ versus LEC were $16.3 \pm 4.7\%$ and $25.3 \pm 1.2\%$ in fat; $14.7 \pm 1.4\%$ and $25.01 \pm 2.2\%$ in ash; and $40.0 \pm 3.4\%$ and $36.9 \pm 2.2\%$ in protein.

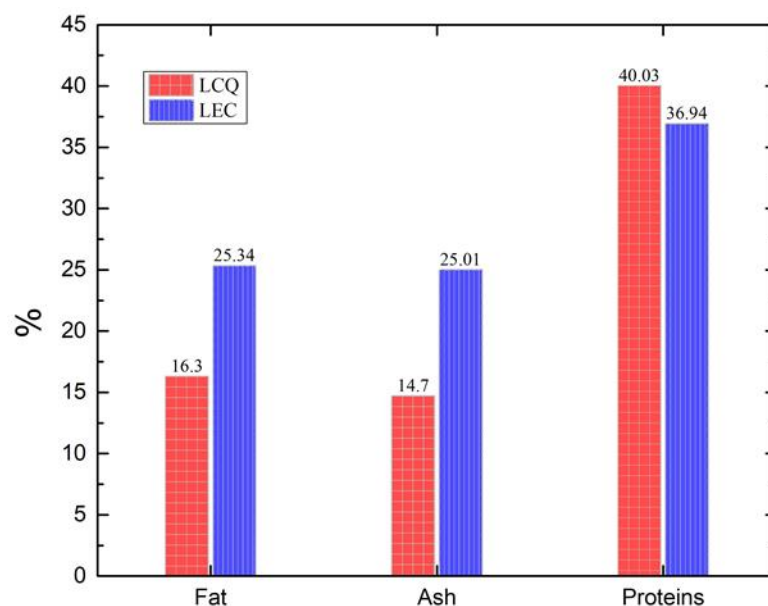


Figure 3. Proximal composition on a dry basis of the sludge obtained by LCQ and LEC. The bar values represent the average of all the batches worked.

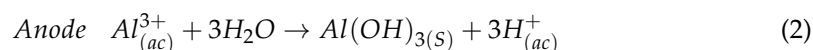
3.2. Iron and Aluminum Content in Sludge

The treatment of PW in fishmeal factories is carried out by chemical coagulation by adding mainly iron salts. In contrast, in the tests carried out by electrocoagulation, aluminum electrodes were used, so the formation of aluminum salts from the sacrificial electrode was expected. Consequently, the resulting sludge's iron and aluminum content differed (Table 2).

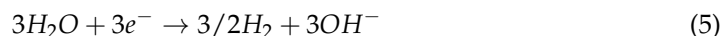
The Fe content in the LCQ was around $4.8\% \pm 0.3\%$ and mainly came from ferric sulfate added during the treatment of PW. Previous tests on the iron content in this type of sludge showed iron values ranging from 2.5% to 3.5% on a dry basis. These values show a notable difference from those found in this study. Chemical coagulation processes in the plant are complicated due to the large volumes treated in short times ($250 \text{ m}^3/\text{hour}$); thus, over an hour, PW can show changes of up to 1500 mg/L of SST, depending on the raw material quality, according to data reported by the company. Although the jar test in the laboratory suggests adding precise coagulant contents, the answer to treating these large volumes in adequate time depends in part on technical experience, so, oftentimes, the addition of coagulants is performed empirically, resulting in varying concentrations of coagulants in the sludge obtained. Alternatively, Fe content in the LEC had an average value of $278 \pm 16 \text{ mg/kg}$, higher than that found in fishmeal, possibly due to some pollution in the line and due to the recovery of Fe associated with proteins and iron salts present in seawater.

Aluminum content in LCQ had an average value of $106 \pm 12 \text{ mg/kg}$, an average found in marine foods [36], while aluminum content in LEC was around $4.2 \pm 0.2\%$, mainly from the sacrificial anode. The oxidation and reduction reactions that occur in the aluminum electrodes of the electrocoagulation reactor depend on the pH of the medium. The reactions at the anode and cathode are observed below:

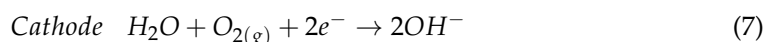
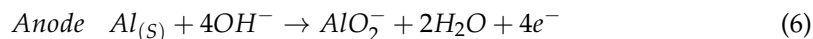
Acidic pH



Cathode



Neutral/Alkaline pH



The PW in fishmeal factories has pH values of 6.1–6.5. Under these conditions, the sacrificial electrode (anode) releases aluminum ions that, in contact with the hydroxyls formed by electrolysis, favor the formation of hydroxides or polyhydroxides, which have a great affinity for dispersed particles and ions and allow their coagulation. This explains the high concentration of aluminum in the recovered sludge (Figure 4).

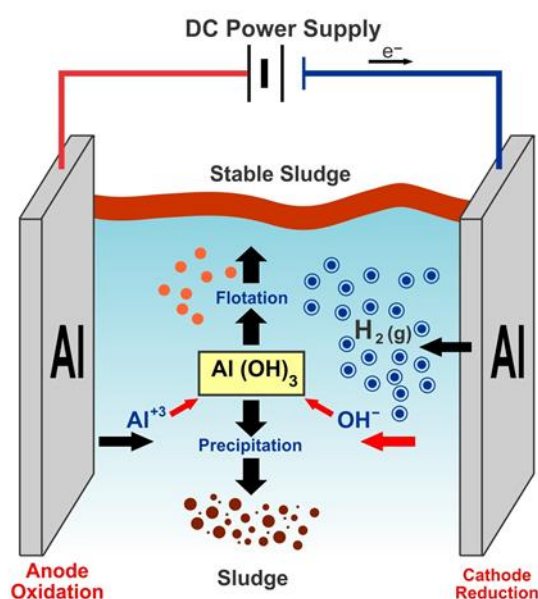


Figure 4. Simplified diagram of the electrocoagulation process with an aluminum electrode.

3.3. Fatty Acid Profile in the Fat Extracted from the Sludge

The initial tests of the fatty acid profile in LCQ and LEC showed values in the content of omega-3 fatty acids (W-3) [eicosapentaenoic (EPA) + docosahexaenoic (DHA)], well below the expected value in the anchovy fat (*E. ringens*), whose W-3 values are around 30% [37]. It is possible that the drying process during the preparation of the flour samples oxidized the polyunsaturated fatty acids and greatly decreased them. Thus, the fat was extracted from the newly arrived samples at the laboratory using the Bligh and Dyer methodology to avoid oxidation; then, its methylation was performed, and the fatty acid profile was determined.

The ions formed by electrolysis are responsible for the oxidation of polyunsaturated fatty acids. The fatty acid profile of the fat extracted from the sludge (Table 3) shows that the EC was more susceptible to oxidation than the CCP. The EPA + DHA content in LEC was 12.46%, while it was 18.78% in LCQ. The average percentage content of EPA was $9.98 \pm 0.89\%$ and $4.38 \pm 0.09\%$, while DHA was $8.80 \pm 1.1\%$ and $8.08 \pm 0.34\%$ in LCQ and LEC, respectively. The unsaturation of fatty acids directly influences their oxidation, so it is striking that DHA (22:6 W-3) has not decreased as much as or more than EPA (20:5 W-3). The DHA: EPA ratio in anchovy is higher in phospholipids than in triglycerides [38]. The increased EPA concentration may have been the target of the oxidative assault, because it is possible that the lipids recovered in EC are primarily derived from triglycerides, even if there is no more information on the lipids present in the sludge.

Table 3. Fatty acid profile of the fat extracted from the sludge from PW. The values are expressed in percentage.

Ac. Fatty/Batch	Chemical Coagulation Sludge (LCQ)				Electrocoagulation Sludge (LEC)			
	1	2	3	4	1	2	3	4
C 14:0 (Myristic)	10.65	9.73	10.89	10.44	10.72	10.62	10.63	10.63
C 16:0 (Palmitic)	24.88	23.69	25.8	25.03	29.85	30.11	30.29	31.1
C 16:1 (Palmitoleic)	10.93	8.96	11.24	11.01	9.37	9	9.13	9.01
C 18:0 (Stearic)	5.19	5.04	5.37	5.26	7.28	7.37	7.46	7.62
C 18:1 w-9 (Oleic)	10.11	11.67	10.28	10.07	9.87	9.28	9.34	9.58
C 18:1 w-7 (Vaccenic)	3.72	3.33	4.11	4.08	4.4	4.28	4.34	4.49
C 18:2 w-6 (Linoleic)	1.24	1.04	1.24	1.28	1.05	0.96	1.03	1.05
C 20:5 w-3 (EPA)	9.69	11.12	9	10.11	4.5	4.4	4.35	4.28
C 22:5 w-3 (Cupladonic)	1.06	1.43	1.02	1.13	1.61	1.63	1.73	1.78
C 22:6 w-3 (DHA)	8.59	1.43	7.64	8.66	8.08	8.41	8.21	7.62

4. Conclusions

This study compared sludges obtained from the treatment of PW treated with a 125 L/h continuous system electrocoagulation pilot plant and conventional chemical coagulation used in the fishing industry. EC was found to produce sludge with a higher concentration of lipids and ash, which indirectly confirmed that it performs a greater removal of biomolecules and salts compared to CCP. The aluminum content in LEC was 4.2%, while the iron content in LCQ was 4.8%. The fatty acid profile of the lipids extracted from the sludge indirectly showed that the electrocoagulation process, under the test conditions, turned out to be more oxidizing than the chemical coagulation process, which was verified by a lower concentration of W-3 fatty acids (EPA + DHA).

Although it should be noted that, due to the large volumes of water handled by this type of industry, electrocoagulation tanks must have a continuous flow to meet the water purification criteria and simultaneously obtain sludge that serves as a feed ingredient under a circular economy concept. Electrocoagulation has shown to be an up-and-coming technology for treating PW in fishmeal factories.

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