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RESEARCH ARTICLE

OPTIMIZATION ELECTROCOAGULATION PROCESS WHEY ACID WITH PHOSPHORUS RECOVERY

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ABSTRACT

The objectives were to optimize the EC process parameters using Taguchi methodology, evaluating the efficiency percentage removal of COD and phosphorus removal. The whey was analyzed in initial and final values de pH, COD and phosphate ions. We used a electrochemical reactor de 2 L, batch type and performed the experimental design optimization, fractional factorial random parameters, the type of $L_9(3)^4$, ie four variable at three levels. The results have allowed, working with aluminum anode and cathode iron, 8 hours, voltage 4.67 V, 57.6 recirculation flow Lh^{-1} and a distance of 1 cm between electrodes, achieve a 63% removal initial COD and a recovery above 83% phosphorus as phosphate.

INTRODUCTION

The aluminum electrodes are used for processes of electrocoagulation (EC) because they dissolve (sacrificial anode) giving rise to metal ions (Al^{3+}), which form hydroxides or polyhydroxides, which are excellent coagulating agents that destabilize the colloidal substances (Mollah et al, 2004) and are readily available for its relatively low cost and availability. Also other materials have been studied to be used as anodes such as iron (Luck *et* Foegeding, 2002; Dockhorn, 2007; Callejas *et al*., 2012).

The initial conductivity of the sample (8.05 $mS.cm^{-1}$), was unchanged. According to literature, from a minimum value of 1.00 $mS.cm^{-1}$ and higher the incidence of this process parameter in the EC is minimal. It also seeks to reduce the addition of chemicals to the process. This parameter is important for good performance of the EC process which favors the conduction of electricity. (Zumdahl, 2000; Mullah, 2001; Mullah, 2004; Holt, 2005). The initial pH of the solution is an important variable for determining the ionic species formation at the EC and avoids the addition of chemicals for the same.

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This reduces operating costs (Mollah, 2001). The original pH of an acid whey (4.5-5.0) coincides with that reported in the literature (Golder, 2006), so the EC tests are performed without changing the initial pH of the sample, but should be monitored. The voltage is directly proportional to the energy cost and may vary by factors such as conductivity of the solution, the distance between the electrodes, the material and the geometry thereof. (Mullah, 2001; Mullah, 2004; Holt, 2005; Zumdahl, 2000). In EC is necessary to apply a potential drop to overcome the resistance of the medium, which is directly proportional to the distance between electrodes and energy consumption, resulting in lower values for the latter potential drops (at distances between electrodes minor). Moreover, agitation (flow) the system can improve the contact between the particles of the ion M^{n+} to be freeing the sacrificial anode both Fe and Al and the hydroxyl groups (which are released from the cathode) of any materials allowing use coagulation by forming insoluble metal oxyhydroxides that cooprecipitan with organic matter (COD) and inorganic (P) present in the electrolyte (Mullah *et al*, 2004).

Because the process of electrocoagulation (EC) is directly affected by a large number of variables and with the aim of reducing the number of experimental tests and improve the design of the EC cell, must perform design optimization experiment achieve good removal of organic matter (COD) and phosphorus (as PO_4^{3-}). The objectives of this study were to optimize the EC process using Taguchi methodology, taking into account the higher incidence variable in the process and with the minimum amount of testing and analyzing the behavior of effluent, evaluating the efficiency percentage removal of COD and percent efficiency of phosphorus removal and recovery.

MATERIAL AND METHODS

The work was performed in a batch electrochemical reactor of 2 liters as shown in Figure 1. We performed the optimization study EC process working with Taguchi methodology, taking into account the various factors that influence the process (Cesatrone, 2001; Sreenivas *et al*., 2004), in order to reach optimal values of factors and levels. It was based on a fractional factorial randomized design (Chuaqui *et al*., 2004, Medina *et al*., 2007; Ravella *et al*., 2008). It was considered as factors of answers: the highest percentage of COD removal efficiency or percentage of EC process and greater removal of phosphor as phosphate. We used the ANTM 3071, version 2.5 (ANTM 3071, 1993) for information processing. Were considered as response factors: higher percentage COD removal and greater removal of phosphorus as phosphate. We used the ANTM 3071, version 2.5 (ANTM 3071, 1993) for information processing.

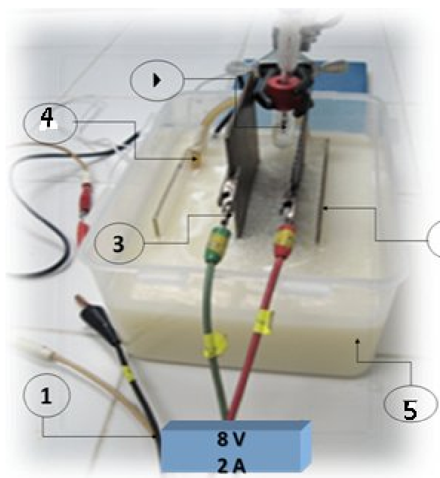

Figure 1. Electrochemical cell with recirculation of the solution of whey.

1. Potentiostat/galvanostat 2. Cathode: Ti/Ruthenium Oxide, Iron or Graphite 3. Anode: iron or aluminum 4. Recirculation pump 5. Effluent (whey) from cheese making

We considered four control factors (EC process time, system temperature, flow of the electrolyte and distance between electrodes) and are listed in Table 1, where are also the three levels which we worked. It also indicates the noise factor (cathode material) in three levels (iron, graphite and Ti/ruthenium oxide IV). Factors were considered unchanged, the initial pH of the acid whey (4.70 ± 0.20), the initial conductivity (8.05 $mS.cm^{-1}$) and the anode material, in this case aluminum. With four control factors at three levels, resulting in an orthogonal arrangement of the type $L_9(3)^4$, which gives rise to nine experiments, three levels of noise to result in an array of 27 experimental experiences as indicated in the table 2 (ANTM 3071, 1993).

Table 1. Matrix parameters for experimental design

	FACTOR	VARIABLE	LEVELS			NOISE
A	TIME	t (HOURS)	4	6	8	IRON
B	VOLTAGE	V (VOLTS)	2.67	3.67	4.67	GRAPHITE
C	FLOW	$L.h^{-1}$	57.6	39.6	76.8	Ti|RuO_2
D	DISTANCE	d (cm)	1	1.5	2	-

Table 2. Matrix experimental design $L_9(3)^4$ and results matrix to obtain

Nº	CONTROL VARIABLES				NOISE (Cathode Material)		
SERIES	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
1	4	2.67	57.6	1	1	2	3
2	4	3.67	39.6	1.5	4	5	6
3	4	4.67	76.8	2	7	8	9
4	6	2.67	39.6	2	10	11	12
5	6	3.67	76.8	1	13	14	15
6	6	4.67	57.6	1.5	16	17	18
7	8	2.67	76.8	1.5	19	20	21
8	8	3.67	57.6	2	22	23	24
9	8	4.67	39.6	1	25	26	27

The analysis and statistical processing of the experimental data obtained, was conducted in two stages:

- Regular analysis, which allowed us to evaluate the influence of the factors on the average value of the responses in a result table and a graph representing factorial and interactions, these values.
- Analysis Signal/Noise, which allowed evaluation as factors affecting the average value and the variation around this mean value.

In both stages was performed an analysis of variance (ANOVA) also indicated the percentage contribution of each factor to the total variation and variation due to residual error (ANTM 3071, 1993). Also important information on the corrosion behavior of the aluminum anodes (dissolution of sacrificial anodes) and the evaluation of the effects of the nature and concentration of the medium electrolyte (acid whey), pH and current density, determining in the electrochemical dissolution of aluminum. For this, we assessed the degree of wear on the anode material (aluminum) for different weights and observation of destabilization of suspensions and emulsions. The COD was determined by the method of open reflux (Eaton *et* Clescerl, 1992, APHA, 1998; NMX-AA-030-SCFI-2001). Determination of P as phosphate ions (PO_4^{3-}) was performed by the method of forming a blue colored complex phosphomolybdic in reduced leuco form and is measured photometrically (APHA, 1998).

RESULTS AND DISCUSSION

Removal of organic load (COD)

Table 3 shows the respective efficiencies of COD removal reaching a maximum of 48.85% efficiency after applying EC treatment during 8 hours (A3), with a voltage of 4.67 V (B3) and a recirculation flow 39.6 $L.h^{-1}$ (C1) with a distance of 1 cm between electrodes (D1) using a cathode of iron (R1), with the same conditions but using a graphite cathode was obtained 47.36% efficiency. Importantly the difference when using a graphite cathode in iron or efficiencies corresponding to 1.5%, so it can use either a cathode or graphite iron.

Table 3. COD response, expressed in terms of % removal efficiency at the end of each experiment

Nº SERIES	CONTROL VARIABLES				% COD removal efficiency		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO$_2$
1	4	2.67	57.6	1	2.10	22.17	17.73
2	4	3.67	39.6	1.5	15.42	19.96	31.45
3	4	4.67	76.8	2	21.71	22.64	33.06
4	6	2.67	39.6	2	17.98	18.59	16.53
5	6	3.67	76.8	1	43.18	16.94	34.15
6	6	4.67	57.6	1.5	41.44	44.29	26.57
7	8	2.67	76.8	1.5	24.67	22.70	17.98
8	8	3.67	57.6	2	27.76	33.13	20.32
9	8	4.67	39.6	1	48.85	47.36	33.71

Then follows an efficiency of 44.29% when working with the same voltage (4.67 V) for 6 hours (A2) and a distance of 1.5 cm (D2) between electrodes with a graphite cathode (R2). With an efficiency of 43.18% followed by an iron cathode but with lower voltage (3.67 V) and distance between electrodes (1 cm) and higher flow (76.8 L.h^{-1}) again shows that there is a difference in at least 1% efficiency when working with graphite cathodes and/or iron. For the election of the top five experiments (highlighted in Table 3), we used the criterion of a removal efficiency greater than 40%. Thus are better iron and graphite cathodes under conditions of time between 6 and 8 am, between 3.67 and 4.67 voltages V and distances of 1 and 1.5 cm, with minimal differences in% COD removal efficiency (1-1.5 %).

Removal and recovery of phosphorus

In order to obtain simultaneously during the optimization process of the EC, the highest COD removal and the highest percentage of phosphorus removal in the whey can be observed in experiment 9 with graphite cathode match both parameters (see Tables 3 and 4) with higher performance. It may be noted that the material used as cathode (graphite) significantly influences the process as the voltage (4.67 V), time (8 hours) and the distance between anode and cathode (1 cm). In the removal efficiency of phosphorus in the form of phosphate (PO_4^{3-}) (see table 4) was taken as criterion removal efficiency greater than 70% (indicated in Table 4). One can see that the highest efficiency (90.13%) is obtained by applying graphite cathode aforementioned conditions. For phosphorus removal, second order of importance of the cathode is 82.90% Fe with a time of 6 h, with a voltage of 4.67 V and 1.5 cm distance between electrodes noting here that the distance also plays a very important in treatment. Using a graphite cathode is best for phosphorus removal, since it has more than ≈ 10% removal efficiency when compared with iron cathodes and Ti|RuO$_2$ both with ≈ 80% of maximum efficiency.

Table 4. Phosphorus uptake, expressed as phosphate (PO_4^{3-}) in terms of % removal efficiency at the end of each experiment

Nº SERIES	CONTROL VARIABLES				% Phosphorus removal efficiency		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO$_2$
1	4	2.67	57.6	1	8.54	17.83	9.41
2	4	3.67	39.6	1.5	15.77	6.50	11.90
3	4	4.67	76.8	2	1.51	6.92	18.13
4	6	2.67	39.6	2	12.76	6.28	1.49
5	6	3.67	76.8	1	52.82	70.98	80.16
6	6	4.67	57.6	1.5	82.90	69.96	29.99
7	8	2.67	76.8	1.5	18.32	9.04	56.54
8	8	3.67	57.6	2	5.35	33.09	13.87
9	8	4.67	39.6	1	77.99	90.13	71.68

Dissolution of Aluminum (sacrificial anode)

We analyzed also the loss of Al by weight difference of the anode at the beginning and end of each of the 27 experiments, obtaining the results shown in Table 5. As in the previous cases five experiments were chosen, which have higher aluminum solution to 3 g/2L (> 1.5 g.L^{-1}). Experiments 25, 26 and 27 (depending on the design matrix in Table 2) belonging to the run 9 are those having higher dissolution 5.96, 4.71 and 5.54g.(2L)$^{-1}$ respectively, these correspond to the three cathodes under study. Require a time of 8 h of treatment with a maximum voltage of 4.67 V and a minimum distance of 1 cm between electrodes. For this parameter the experiment 25 matches as COD removal, but not for the high phosphorus removal, which would correspond to the experiment 26 (4.71 g.(2L)$^{-1}$). Under the smaller criterion value sacrificial anode wear (lower metal loss), the latter would be an acceptable option.

Table 5. Aluminum anode dissolution at the end of electrocoagulation process in g.2L^{-1}

Nº SERIES	CONTROL VARIABLES				Dissolved aluminum g.(2L)$^{-1}$		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO$_2$
1	4	2.67	57.6	1	1.08	0.68	1.02
2	4	3.67	39.6	1.5	1.59	1.10	1.32
3	4	4.67	76.8	2	1.60	1.39	1.62
4	6	2.67	39.6	2	1.15	0.88	1.21
5	6	3.67	76.8	1	3.20	2.34	3.22
6	6	4.67	57.6	1.5	2.89	2.08	2.17
7	8	2.67	76.8	1.5	1.95	1.42	1.96
8	8	3.67	57.6	2	2.11	1.46	2.21
9	8	4.67	39.6	1	5.96	4.71	5.54

When considering that the higher the aluminum dissolution could occur redissolution of the $Al(OH)_3$ formed by increasing the pH of the medium and consequently a reduction of organic matter and phosphorus coprecipitates thus affecting the efficiency of COD removal (to values less than 40%) and phosphorus (at less than 70%).

Significantly, in this sense it is desirable that the anodic dissolution is the smallest possible because the process is encarecería CD. If so are better for the three effects (most COD removal together with the highest phosphorus removal and recovery while there is less anodic dissolution) experiments 16 and 26, which coincide with the objectives.

Relationship of aluminum dissolution% efficiency of COD

Table 6 shows the results of dissolution of aluminum (sacrificial anode) to the higher COD removal (see Table 1) and highlight that match the higher dissolution of the aluminum electrode and the matching lower wear the anode. It can be seen that for the removal of higher organic loading (48%) requires greater amount of aluminum dissolved being fulfilled for experiment 25 (5.96 g.(2L)$^{-1}$) and for experiment 26 with a solution of 4.71 and a removal g.(2L)$^{-1}$ very COD similar between 48-47%, both experiments under the same conditions but with different cathode materials, iron and graphite, respectively.

Table 6. Dissolution of aluminum in the experiments with higher COD removal

N° EXP	CONTROL VARIABLES				Al^{3+} dissolved & % COD efficiency		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
13	6	3.67	76.8	1	3.20*	-	-
16, 17	6	4.67	57.6	1.5	2.89	2.08	-
25, 26	8	4.67	39.6	1	5.96*	4.71*	-

Third in % removal efficiency of COD (44%) with a solution of Al^{3+} g.(2L)$^{-1}$ 2.08 shows that it takes less time (6 h) but higher flow and greater distance between electrodes (1.5 cm) with over previous. Fourth is to be with the iron cathode, is reached a solution of Al^{3+} 3.20 g.L^{-1} and a removal efficiency of 43% in the same period of time but at a lower voltage (3.67 V) and increased flow (76.8 L.h^{-1}) and 1 cm distance between electrodes and finally with an iron cathode again a removal of 41% of the COD, presents a lower solution (2.89 g.L^{-1}) of the electrode of Al^{3+} in less time, with the same voltage but higher flux and greater distance between electrodes. It is observed in the latter, which decreases the dissolution of the electrode by about 50%. This is also reflected in the experiments with graphite. It is important to note that as the voltage variable (B), the treatment time (A) and the distance between electrodes (A) play a very important role in the optimization process. Not so the variable flow (C) as they could work at any level.

Relationship of aluminum dissolution% efficiency in removing phosphorus

The dissolution results for the aluminum top phosphorus removal efficiencies are shown in Table 7 and highlight that match the major and minor dissolution of the aluminum electrode. It can be seen that when using graphite cathode is achieved remove up to 90% phosphorus and 4.71 g.(2L)$^{-1}$ was obtained. With a 83% removal of phosphorus, using an iron cathode, dissolve 2.89 g.(2L)$^{-1}$, two experiments were performed with the maximum value of voltage, but at different times and distances between electrodes.

Table 7. Dissolution of aluminum for greater % removal of phosphorus

N° EXP	CONTROL VARIABLES				Al^{3+} dissolved & % PO_4^{3-}efficiency		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
15	6	3.67	76.8	1	-	-	3.22*
16	6	4.67	57.6	1.5	2.89	-	-
25, 26, 27	8	4.67	39.6	1	5.96*	4.71*	5.54*

Third in% removal efficiency (80%) had a total solution of Al^{3+} in 3.22 g.(2L)$^{-1}$ and shows that less time is required (6 h) and lower flux but higher voltage with respect to the above. Moreover it is a Ti|RuO_2 cathode, this material has disadvantages compared with other materials, such as a low COD removal efficiency and requires longer treatment. In fourth place is the cathode iron (78%) with 5.96 g.(2L)$^{-1}$, in this experiment (25) corresponds approximately to the double amount of Al^{3+} dissolved in experiment 16, where the same voltage was applied at different times, flows and distance between electrodes, which suggests a strong influence of the variable voltage in treating EC.

Finally, the Ti|RuO_2 electrode is again, in experiment 27 with a total dissolution of Al^{3+} of 5.54 g. (2L)$^{-1}$ and an efficiency of 72% removal of phosphorus, it coincides present as anode dissolution under the same conditions, but with different materials. It is imperative to note that the voltage control variables (B), treatment time (A) and distance between electrodes (C) play an important role in the process.

Variation of pH

Table 8 shows the results of the pH at the end of the experiment. The variations are determined primarily by the type of material used as cathode and the treatment time. By using a cathode of Ti|RuO_2 during 4 h the pH range is maintained between 7 and 8, whereas a graphite electrode with pH maintained below 7 units. In the case of iron, the pH ranges from 7 to 7.5.

Table 8. Results of pH at the end of each experiment

N° SERIES	CONTROL VARIABLES				pH		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
1	4	2.67	57.6	1	6,93	5,96	7,15
2	4	3.67	39.6	1.5	7,50	6,85	7,54
3	4	4.67	76.8	2	7,48	6,79	7,76
4	6	2.67	39.6	2	7,02	6,04	6,83
5	6	3.67	76.8	1	8,71	8,56	8,82
6	6	4.67	57.6	1.5	8,95	8,42	9,10
7	8	2.67	76.8	1.5	7,97	6,70	7,44
8	8	3.67	57.6	2	7,81	6,53	7,75
9	8	4.67	39.6	1	9,03	8,86	8,94

The variable voltage on the pH has a significant impact since increasing the time while keeping the voltage at the lowest level, the pH is maintained below 7 for the three cathode materials with higher voltage and higher time the pH is increased above and from 8.5 to 9.1 units. After applying the treatment for 8 hours presented a similar behavior lowest voltage level and subsequently increases above 8.5.

Relation of pH/Eh and% COD removal efficiency

Table 9 is a comparison of the values of pH/Eh respect to maximum efficiency of COD. It can be seen that the pH range is between 8.4 and 9.0 units when it has the highest efficiency of removal of organic material. As higher the efficiency of COD removal the pH also falls within the maximum values, while the Eh having an inverse behavior corresponds to the minimum values.

Table 9. Values of pH/Eh when you have a high COD removal efficiency

N° EXP	CONTROL VARIABLES				pH/Eh & COD removal efficiency		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
13	6	3.67	76.8	1	8.71/-105	-	-
16, 17	6	4.67	57.6	1.5	8.95/-117	8.42/-91	-
25, 26	8	4.67	39.6	1	9.03/-120	8.86/-112	-

Relation of pH / Eh and% phosphorus removal efficiency

Table 10 shows the pH/Eh, corresponding to the maximum phosphorus removal efficiency. It can be seen then that for maximum phosphorous removal requires slightly higher values of pH between 8.8 and 9.0.

Table 10. Values of pH/Eh for maximum phosphorus removal

N °EXP	CONTROL VARIABLES				pH/Eh & % efficiency PO_4^{3-}		
	A	B	C	D	IRON	GRAPHITE	Ti|RuO_2
15	6	3.67	76.8	1	-	-	8.82/-110
16	6	4.67	57.6	1.5	8.95/-117	-	-
25, 26, 27	8	4.67	39.6	1	9.03/-120	8.86/-112	8.94/-116

Relation of the pH and dissolution of aluminum

Table 11 shows pH values which belong to the maximum dissolution of sacrificial electrode (Al). As in the removal of phosphorus is observed that for maximum dissolution required pH values between 8.7 and 9.0.

Table 11. When pH is increased aluminum dissolution

N° EXP	CONTROL VARIABLES				Al^{3+} dissolved & % PO_4^{3-}efficiency		
	A	B	C	D	HIERRO	GRAFITO	Ti|RuO_2
15	6	3.67	76.8	1	8.71	-	8.82
25, 26, 27	8	4.67	39.6	1	9.03	8.86	8.94

Statistical analysis of experimental design

For optimization of the EC process of acid whey were taken into account two factors of response, maximum% of removal efficiency of COD, and greater% efficiency in the removal of phosphorus. This type of design (Taguchi's methodology) allows uncontrolled use a known factor and noise factor in this case is used the type of material used as cathode, were used iron, graphite and Ti|RuO_2, with the aim of assessing which of them is better.

Analysis of variance for COD removal

An analysis of variance using ANOVA program, considering first the variables along with the noise factor; first performed for COD analysis, and the results are shown in Table 12. It is noted that the factor B (voltage) is the largest single contribution that has to variations in the response signals, with a percentage of 41.95%, followed by the time factor (A) with 14.98% and had less influence factors on the system were the distance between the electrodes (4.61%) and flow (negligible value).

To obtain good removal efficiencies of COD, the voltage applied and the time of treatment, are the most important factors to consider, without regard to the distance at which the electrodes are attached (anode |cathode) and flow recirculation. It is also noted that the type of material used as cathode does not have a significant effect on the removal of organic load since their contribution is minimal (0.05%).

Table 12. Analysis of variance main effect of each factor and the noise factor

Fuente de variación	Degrees of freedom	Est Dev (S)	Variance	F	S`	%
TIME (hours)	2	511,77	255,89	37,11	497,98	14,98
VOLTAGE (Volts)	2	1408,06	704,03	102,12	1394,27	41,95
FLOW ($L.h^{-1}$)	2	13,79	6,89	1,00	1,61	0,02
DISTANCE (cm)	2	167,09	83,55	12,12	153,3	4,61
NOISE	2	15,62	7,81	1,13	1,83	0,05
e_1	16	1207	75,44	10,94	1096,69	33
e_2	0	0,00				
e	2	13,79	6,89		179,25	5,39
TOTAL	26	3323,33	127,82			100,00

Figure 2 shows the individual effect of each factor on the variable to optimize:% COD removal efficiency, including the noise factor. Considering that we work under the criteria of more is better, you can set the levels of each factor to work and earn higher removal efficiencies deDQO, corresponding to the combination A3-B3-C2-D1 and R2 would be the noise factor (graphite) . This means, it must work at a time of 8 h (A3), a voltage of 4.67 V (B3), a flow of 57.6 Lh-1 (C2) and a distance between electrodes of 1 cm (D1). The material that could give best answer as is the graphite cathode (R2).

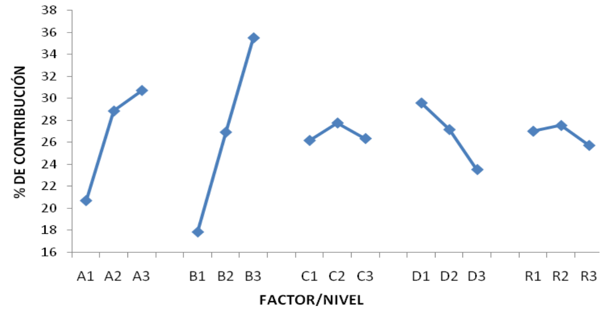

Figure 2. Variation of factors and levels including noise factor in the analysis of response % efficiency in the removal of COD (higher is better).

The analysis of variance at any level of noise factor (any cathode material) to visualize the effect of each factor under optimization and are shown in Table 13. Again factor B (voltage) is the major contribution to system presents with 38.27%, followed by the time factor (A) with 23.25%. Factors that had less influence on the flow and treatment were distance. As can restate the above that to obtain good efficiencies the COD removal in the applied voltage and the treatment time, are the most important factors to consider as they have the greatest effect on the removal, regardless of the distance to be placed electrodes (anode | cathode) and the recirculation flow.

Table 13. Analysis of variance main effect of each factor

Fuente de variación	Degrees of freedom	Est Dev (S)	Variance	F	S`	%
TIME (hours)	2	95,25	47,63	3,64	69,09	23,25
VOLTAGE (Volts)	2	139,87	69,94	5,35	113,71	38,27
FLOW ($L.h^{-1}$)	2	35,89	17,94	1,37	9,73	3,27
DISTANCE (cm)	2	26,16	13,08	1,00	7,09	2,38
NOISE						
e_1	0	0,00				
e_2	0	0,00				
e	2	26,16	13,08		104,63	32,83
TOTAL	8	297,17	37,15			100,00

Figure 3 shows again the effect of each factor on% COD removal efficiency. Considering that the feature worked under more is better. After defining the levels of each factor were obtained removal efficiencies of organic load, taking into account those who are above the trend line of the data.

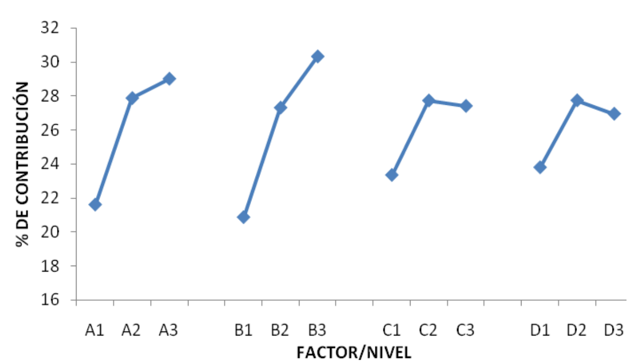

Figure 3. Variation of factors and levels, independent of the noise in the response analysis:% of removal efficiency of COD (higher is better).

In this case corresponds to the combination A3-B3-C2-D2. This means that to have the highest percentages of removal efficiency of organic load should work at a time of 8 h (A3) with a voltage of 4.67 V (B3), a flow of 57.6 $L.h^{-1}$ (C2) and a distance between electrodes 1.5 cm (D2). In analyzing Table 2 again corresponding to the experimental design matrix is observed that no experiment was carried out under these conditions: A3, B3, C2, D2 and R2 corresponds therefore to analyze the results obtained in confirmatory experiments.

Analysis of variance for the removal of phosphorus

The data for the% efficiency in the removal of phosphorus are shown in Table 14. In assessing the contribution of each factor including the removal of noise in the phosphor can be seen that the factor D (distance between electrodes) is the largest contributor to the system, 33.19%, followed by time factor (A) and the voltage (B) 26.82 and 21.81% respectively. The effect of the flow and the noise factor is negligible. To achieve good removal efficiencies phosphorus should consider the distance between electrodes (anode|cathode), the treatment time and the voltage to be applied, regardless of the recirculation flow and the material used as cathode and which have no significant effect in removing phosphorus because their contribution is minimal (0.06%).

Table 14. Analysis of variance main effect of each factor and the noise factor

Fuente de variación	Degrees of freedom	Est Dev (S)	Variance	F	S`	%
TIME (hours)	2	6508,04	3254,02	96,89	6440,87	26,82
VOLTAGE (Volts)	2	5305,85	2652,93	79,00	5238,68	21,81
FLOW ($L.h^{-1}$)	2	105,27	52,64	1,57	38,11	0,16
DISTANCE (cm)	2	8037,85	4018,92	119,67	7970,68	33,19
NOISE	2	67,17	33,58	0,03	14,48	0,06
e_1	16	3992,64	249,54	7,43	3455,31	14,32
e_2	0	0,00	0,00			
e	2	67,17	33,58		873,16	3,64
TOTAL	26	24016,82	923,72			100,00

Figure 4 shows the individual effect of each factor on the signal to be optimized. In this case corresponds to the combination A2, B3, C3 and D1 and R2. Therefore, to have the highest percentages of phosphorus removal efficiency should work in time of 6 h (A2), a voltage of 4.67 V (B3), a flow of 76.8 $L.h^{-1}$ (C3) and a distance between electrodes 1 cm (D1). The material that could give better response was graphite (R2).

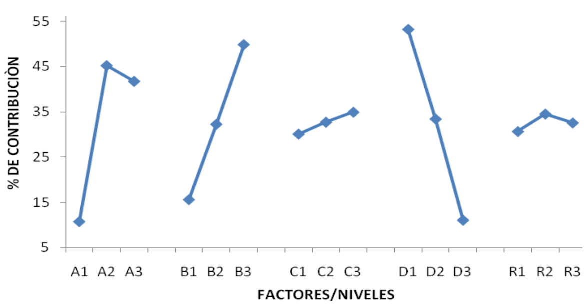

Figure 4. Variation factors and levels including noise factor in response analysis: % removal of phosphorus (higher is better).

The analysis of variance for any level of noise factor is presented in table 15. It is observed that there is greater individual contribution of factor D (distance between electrodes) with 62.01%, followed by the time factor (A) with 20.33% and less influence on the voltage applied treatment, 14.37%. C variability factor is minimal, so that its value is discarded.

Table 15. Analysis of variance of the main effect

Fuente de variación	Degrees of freedom	Est Dev (S)	Variance	F	S`	%
TIME (hours)	2	209.03	104.52	25.68	200.89	20.33
VOLTAGE (Volts)	2	150.10	75.05	18.44	141.96	14.37
FLOW ($L.h^{-1}$)	2	8.14	4.07			
DISTANCE (cm)	2	620.95	310.47	76.28	612.81	62.00
NOISE						
e_1	0	0.00				
e_2	0	0.00				
e	2	8.14	4.07		32.56	3.30
TOTAL	8	988.22	123.53			100,00

Figure 5 shows the effect of each factor on% phosphorus removal efficiency. In this case corresponds to the combination A3-B3-C1-D1. That is, to have the highest percentages of phosphorus removal efficiency should work at a time of 8 h (A3) with a voltage of 4.67 V (B3), a flow of 39.6 $L.h^{-1}$ (C1) and a distance 1.0 cm between electrodes (D1). In reviewing the experimental design matrix, it is observed that this experimental run and was performed for all three cathode materials (noise) and corresponds to the experiments 25 to 27. As seen in Figure 4, working noise 2 (graphite cathode) gives the best result in removal of phosphorus (90.13%).

This indicates that there may be other combinations of variables and permitting higher levels% removal of phosphorus. To test confirmatory experiments were performed 3, where the following combinations were tested to find the best answer for both factors (higher% COD removal and phosphorus simultaneously): A3-B3-C2-D2, A3-B3-C2-D1 and A3-B3-C1-D2, the combination A3-B3-C1-D1 has already been done in run 9 and corresponded to the experiments 25 and 26 with iron and graphite materials.

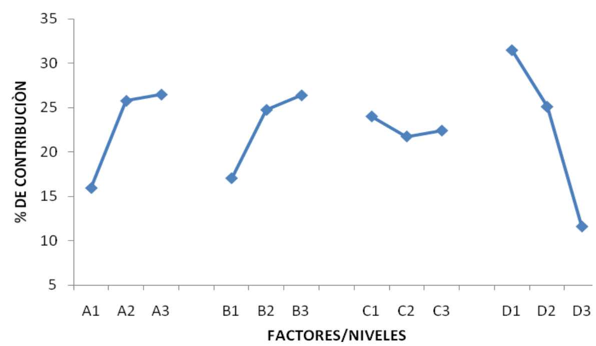

Figure 5. Variation factors and levels, independent noise in the response analysis: % removal of phosphorus (higher is better).

Table 16 shows the results for confirmatory experiments under the conditions encountered and using iron and graphite cathodes. It was found that by using iron under the conditions A3-B3-C2-D1 (8h, 4.67V, 57.6L/hy 1 cm) can achieve a COD removal efficiency of 63%, significantly improving efficiency% found above. With the same cathode but different flow and distance (A3-B3-C1-D2) was obtained 60% efficiency. So we can say that for greater COD removal efficiency increased flow is needed but less distance between electrodes. This statement can be corroborated with the confirmatory experiment A3-B3-C2-D2, employing more flow but greater distance between electrodes, and efficiency achieved was lower (42%). With the graphite cathode was obtained a maximum efficiency of 46% under the same conditions as iron. This finally allowed to discard the graphite material for the purification of whey by electrocoagulation, as the COD removal efficiency in all confirmatory experiments is less than 49% efficiency.

Table 16. Experimental conditions for confirmatory experiments and response in% COD removal efficiency and phosphorus

EXPERIMENTAL CONDITIONS					% COD removal efficiency	
	CONTROL VARIABLES				NOISE	
	A	B	C	D	IRON	GRAPHITE
A3-B3-C2-D2	8	4.67	57.6	1.5	41.97	19.91
A3-B3-C2-D1	8	4.67	57.6	1	63.02	45.58
A3-B3-C1-D2	8	4.67	39.6	1.5	60.24	35.97
A3-B3-C1-D1	8	4.67	39.6	1	48.85	47.36
					% Phosphorus removal	
	CONTROL VARIABLES				NOISE	
	A	B	C	D	IRON	GRAPHITE
A3-B3-C2-D2	8	4.67	57.6	1.5	46.57	4.72
A3-B3-C2-D1	8	4.67	57.6	1	83.22	78.98
A3-B3-C1-D2	8	4.67	39.6	1.5	79.15	83.83
A3-B3-C1-D1	8	4.67	39.6	1	77.99	90.13

Regarding the phosphorus removal efficiency was confirmed that the best conditions correspond to those already made in run 9 (A3-B3-C1-D1) and there are no other conditions that can overcome the foregoing. In this case again the graphite material which showed higher removal efficiency of 83.83% with the conditions: A3-B3-C1-D2, followed by the iron with 83.22% efficiency and other conditions A3-B3-C2-D1, but with a minimum difference of 0.6%. From these results it can again discarding the iron material for use as cathode. Since the objective is to achieve a simultaneous: higher% COD removal efficiency and higher% phosphorus removal efficiency, the iron can be used as cathode material as the best option. It meets both parameters, higher COD removal efficiency (63%), and although not the most efficient phosphorus removal, but among the best answers to this material (83%). Efficiencies can be achieved both with the same working condition: A3-B3-C2-D1 (8 h, 4.67 V, 57.6 $L.h^{-1}$ y 1 cm), discarding the graphite material and using iron as cathode.

Table 17 also shows the results related to the loss of aluminum during the experiments. It can be seen that the graphite material in all cases leads to less dissolution of sacrificial anode (aluminum), reflecting the low COD removal efficiencies, but not in the removal of phosphorus, which is required for 90 $g.(2L)^{-1}$ 4.71 % removal.

Table 17. Experimental conditions for confirmatory experiments and response aluminum solution

					Aluminum dissolution $g.(2L)^{-1}$	
	CONTROL VARIABLES				NOISE	
	A	B	C	D	IRON	GRAPHITE
A3-B3-C2-D2	8	4.67	57.6	1.5	4.84	3.12
A3-B3-C2-D1	8	4.67	57.6	1	6.86	6.21
A3-B3-C1-D2	8	4.67	39.6	1.5	5.83	4.24
A3-B3-C1-D1	8	4.67	39.6	1	5.96	4.71

Another of the premises is to obtain maximum removal efficiency with less dissolution of the anode in order to reduce process costs EC, however not fulfilled as required to obtain 6.86 g Al/2L 63% efficiency COD removal and 83% removal of phosphorus. It is possible under this scheme having high dissolution with high efficiencies, as to achieve good removal of COD requires greater amount of ionic aluminum. This behavior can be observed for the case of iron where efficiency for 42, 49, 60 and 63% dissolved 4.84, 5.96, 5.83 and 6.86 g Al^{3+}/2L whey.

Conclusion

Experiments 25, 26 and 17 of the experimental design matrix showed the best removals of COD with 49, 47 and 44% respectively. These experiments differed in the factor A (EC process time) as taken levels A2 and A3, giving the options between 6 and 8 hours, the two coincide at the maximum voltage level factor (B3) but not to the flow (C2) and (C1) and distance (D2) and (D1). Using iron and graphite cathodes. With Ti|RuO_2 cathode the COD removal efficiencies are lower than 40% (≅ 34%). Among other things, this allows to assume that this material is discarded and which in turn is more costly.

From the statistical analysis it was found that under A3, B3, C2, D2, R2 must be one that achieves COD removals at or above 49%. This has to be using the conditions of 8h, 4.67 V, 57.6 flow $L.h^{-1}$ and a distance of 1.5 cm with a graphite material.

For most experiments phosphorus removal of 26, 16 and 17 have 90, 83 and 80% respectively. These experiments differ in factor A, and taking the level 2 and 3 again, giving the option (A2) and (A3), both agree on the top level of factor B (B3) but not for the flow (C2) and (C1) and distance (D2) and (D1). Using well as iron and graphite cathodes. Experiments 17 and 26 coincide for both responses to optimize and objectives, simultaneously.

With the statistical analysis applied to the arrangement found A3, B3, C1, D1, R2 must be equal or greater than 90% found through experimental design matrix on the efficiency of phosphorus removal. Using the conditions of 8h, 4.67 V, 57.6 flow $L.h^{-1}$ and a distance of 1 cm with a graphite material. This experiment was run 9 corresponds to the array of experimental design and experiment 26.

After confirmatory experiments found that the best arrangement for electrocoagulation corresponds to aluminum materials (anode) and iron (cathode). This is stated on the basis of finding the best combination with the best answer: A3-B3-C2-D1 (8h, 4.67V, 57.6$L.h^{-1}$ y 1 cm), because it meets both parameters set, greater efficiency COD removal (63%), although not the most efficient phosphorus removal, but if the best with this material (83%).

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