



# Evaluation of an Electrocoagulation Process Modified by Fenton Reagent

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## ABSTRACT

This article is oriented to the degradation of nickel in an ionic state at laboratory level from synthetic water made with nickel sulfate, using the electrocoagulation process with aluminum cathodes and modifying this process by the addition of the Fenton reagent, which results from the combination of hydrogen peroxide ( $H_2O_2$ ) and ferrous sulfate ( $FeSO_4$ ) being this reagent a catalyst and oxo-coagulant agent. The efficiency of this reagent will be compared with the typical treatment with aluminum sulfate, which is a typical process based on ion exchange/coagulation at the same percentage concentrations as the Fenton reagent. For this purpose, the optimum conditions of the advanced electrocoagulation process were determined, which consisted of determining the concentrations of Fenton's reagent at concentrations of 150 ppm, 300 ppm, and 450 ppm, in addition to the operating variables such as pH of 8 and 10, voltage of 17.5 V and 19 V and their reaction time, which were compared with aluminum sulfate at 300 ppm, 600 ppm, and 900 ppm. The results obtained with respect to the typical treatment were 0% nickel degradation. However, with the advanced oxidation treatment, an average reduction of 97.5% was found at the conditions of 19 V, pH 10, and Fenton 150 ppm in a time of 30 min.

## INTRODUCTION

Water is an essential element for the life of all living beings on the planet, and it is also a fundamental human right (Rickert et al. 2016). 97.2% of the water on planet Earth is saline, and only 2.5% corresponds to fresh water; of that 2.5%, 30% is a subway, 68% is in glaciers and other snow layers, and only 1.2% is superficial and found in rivers, lakes and other forms of surface water (Gómez-Duarte 2018), so taking care of it is vital for human development.

Water pollution is a problem that all of us face nowadays since it not only occurs in industrialized or less industrialized countries, but it affects every sector of the population. In addition, many think that this resource that is very indispensable for us will always be there, but the truth is that it is not so since we do not become aware of the acts or activities that we perform with this resource (Guadarrama-Tejas et al. 2016).

Among the main factors that affect ecosystems are heavy metals (HM), which are found in human activities where

mining and smelting operations stand out as the majority among other industrial and urban activities (Malik & Saha 2003), where the water pollution rate can be around 200 million cubic meters per day (Lehmann et al. 1999). This fact leads to a large number of problems both in plant life as these metals end up deposited in soils transported to them by rivers, such as reduced growth or yellowing of leaves (chlorosis) (Kalavathy et al. 2010), as well as in human life where the effects can be skin rashes, stomach upset and ulcers, respiratory problems, weakening of the immune system, kidney and liver damage, lung cancer, heart, bone, testicular, central and peripheral nervous system disorders, cancer or death (Ahmad et al. 2010, Tiemi-Muranaka 2010, Ramírez-Franco et al. 2013).

This growing problem calls for rigorous pollution control and increasingly demanding legislation. In response to this, and given the inability of conventional methods to effectively remove many of the existing pollutants, in recent years, there has been an intense search for new and efficient water treatment technologies (López-Ramírez et al. 2021).

In the last decades, advanced oxidation technologies have been consolidated as an efficient alternative in the destruction of toxic substances, including organic, inorganic, metals, or pathogens. In general, in water treatment, advanced oxidation technologies are used when contaminated effluents have high chemical stability or low biodegradability (López-Ramírez et al. 2021).

Currently, studies are focused on the search for optimum values for the different parameters that affect the oxidation process: pH, temperature, reactor design, nature, and concentration of the pollutant and oxidizing agents that can improve the reaction. In addition, the reaction kinetics, which are generally first order, are studied in detail, among which those shown in Fig.1 stand out:

Among the processes mentioned in Fig. 1, it is observed that heterogeneous processes, with electrical energy, using as central the use of current, is the electro-Fenton, which is a process that is divided into electrochemical oxidation and electrocoagulation that applies the principles of coagulation-flocculation in an electrolytic reactor. This is a vessel equipped with a current source and several electrodes in charge of providing the destabilizing ions of

colloidal particles that replace the functions of the chemical compounds used in conventional treatment, inducing an electric current in the water through metal plates (Caviedes-Rubio et al. 2015).

This electrochemical process has been used for the removal of HM without modification and has obtained remarkable results, which are mentioned in Table 1:

As can be seen, this technique is used for the treatment of wastewater contaminated by HM, mostly from the metallurgical or automotive industries. Since conventional treatments are not very efficient, new technologies must be developed (Alomá-Vicente et al. 2013).

HM is found naturally in the environment, usually in trace amounts. These concentrations are not very harmful to biota in general. Because they are deposited in soils by the decomposition of different minerals (erosion process), they coexist to maintain biogeochemical cycles in both soil and water. However, there is also an anthropogenic enrichment of HM (due to mining, industrial, and agricultural activities, among others), and this process that saturates the natural balance is known as contamination since such concentrations are higher than the geochemical composition of the place.

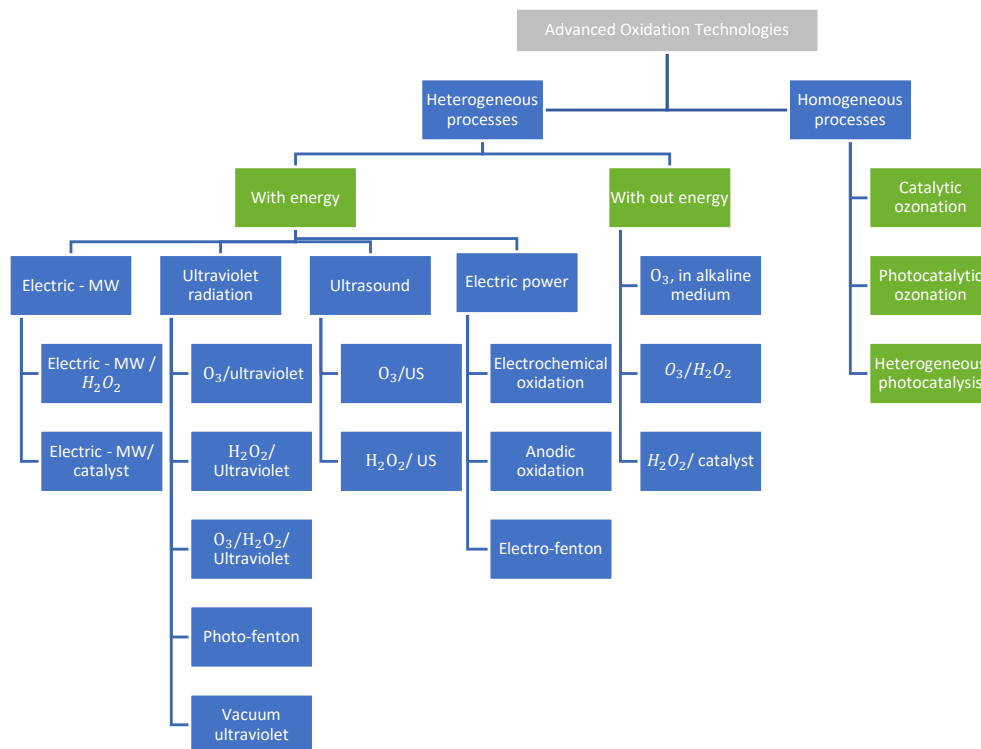


Fig. 1: Classification of advanced oxidation technologies. (Source: Sanz et al. 2013)

Table 1: State of the art of electrochemical processes.

Authors	Year	Results
Abdel-Shafy et al.	2022	They designed an experimental sequence whose purpose was to reduce the amount of Ni, Cu, and Zn in electroplating industry waters.
El-Karamany et al.	2021	Chromium and cadmium solutions from stock concentrations of 560 mg L <sup>-1</sup> chromium and 800 mg L <sup>-1</sup> cadmium were subjected to electrocoagulation.
Chegeni et al.	2021	They planned to perform electrocoagulation in three circumstances. The removal of cyanide and lead, as well as the set of both synthetic and natural water from a gold processing plant.
Sharma et al.	2019	They determined the efficiency of the electrocoagulation process in the removal of Cr (VI) and Pb from the wastewater of an electroplating plant.
Ilhan et al.	2019	The objective was to remove chromium, copper, zinc, nickel, and chemical oxygen demand (COD) from wastewater from the steel industry.
Brahmi et al.	2019	They used aqueous-phase cadmium electrocoagulation first in artificial wastewater and then in real wastewater.

HM such as lead, cadmium, chromium, zinc, mercury, and others are released into aquatic ecosystems as well as soils mainly due to various anthropogenic activities and pose a serious threat to plants, animals, and even humans due to their persistence, bioaccumulation, non-biodegradable property, and toxicity even at low concentrations (Trivedi & Axe 2000, Ahmed et al. 2013). The fact that these metals are found in various ecosystems is of concern since many living things depend on the proper balance in their feeding or living place. How such metals reach the various ecosystems varies depending on the type of activity carried out by man. For example, chromium is a widely used compound in the industry in areas such as plastic plating, metal electroplating for corrosion resistance, leather tanning and finishing, pigments, and wood preservatives (Mohan et al. 2005), other types of metals such as cadmium are used in industry for the manufacture of nickel-cadmium batteries, anti-corrosion agents and pigments (Hamouz et al. 2016). In the case of mercury, six sources of contamination have been identified, which are atmospheric deposition, erosion, human discharges, agricultural materials, mining, and industrial and combustion discharges, as such in natural groundwater and surface water, the content of this metal is below 0.5 µg.L<sup>-1</sup>, however, in wastewater discharged near local mineral deposits and other contaminated sites the concentration of mercury is much higher. Lead is also released into the environment due to the mining industry and the burning of fossil fuels. It is involved in the manufacture of batteries, ammunition, metal products, and devices for X-ray protection (Mohammad et al. 2017).

As can be seen, the greatest contribution to water pollution by HM is due to wastewater discharges resulting from various anthropogenic activities, mainly of an industrial nature. The degree of contamination of water sources is increasing every day due to the constant use of these metals in industry, which increases the concentration of these metals in water and, therefore, increases the risk to humans

and other living organisms by increasing the probability of being exposed to these metals (Cañizares-Villanueva 2000), as shown in Fig. 2 (García & Moreno 2002).

For wastewater, the HM found in wastewater are arsenic with a generation of 12 842 t.y<sup>-1</sup>, cadmium 24 319 t.y<sup>-1</sup>, chromium 61 290 t.y<sup>-1</sup>, copper with a total of 40 905 t.y<sup>-1</sup>, mercury with 498 t.y<sup>-1</sup>, with respect to nickel 60 710 t.y<sup>-1</sup>, lead approximately 16 470 t.y<sup>-1</sup>, selenium a total of 5 904 t.y<sup>-1</sup>, vanadium in wastewater 676 t.y<sup>-1</sup> and of zinc 103 715 t.y<sup>-1</sup> (Pabón et al. 2020).

The toxicity of HM depends on their mobility in the environment, which, in turn, depends on their chemical speciation, persistence, and tendency to accumulate or bioaccumulate (Kumar et al. 2012).

Within HM, nickel is one of the metals used for metal alloys, electroplating, batteries, and electronics, being one of the most widely used and causing diseases such as dermatitis, nausea, chronic asthma, cough, and carcinogenic effects.

The above and the need to develop new technologies for the treatment of wastewater contaminated with HM, nickel being the object of study.

## MATERIALS AND METHODS

The methodology of the present investigation is experimental and quantitative, as shown in Fig. 3, and is centered on 2 experimental stages, the first with a typical process with aluminum sulfate and the second with the electrocoagulation process adding an oxo-coagulant reagent (Fenton). In both experiments, they will be carried out under optimum conditions, and in the case of the reagents, the same quantities will be added. At the same time, each of the experiments will be carried out in triplicate.

### Preparation of Solutions

The solutions at 60 ppm of nickel in 500 mL of distilled water

were prepared with 137 mg of nickel sulfate hexahydrate ( $\text{NiSO}_4 + 6\text{H}_2\text{O}$ ) derived from this chemical compound, which has a purity of 98% and a molecular weight of 263 a.m.u. of which nickel weights 58.69 a.m.u.

The procedure for calculating the nickel sulfate required is shown below, starting by determining the percentage of nickel in the compound as expressed in Equations 1 and 2:

$$\frac{RNS}{N} = \frac{Ni_{a.m.u.}}{SN_{a.m.u.}} * \frac{PurE}{100} \quad \dots(1)$$

RNS/N= Ratio of nickel sulfate and nickel

$Ni_{a.m.u.}$ = Molecular weight of nickel

$SN_{a.m.u.}$ = Molecular weight of nickel sulfate

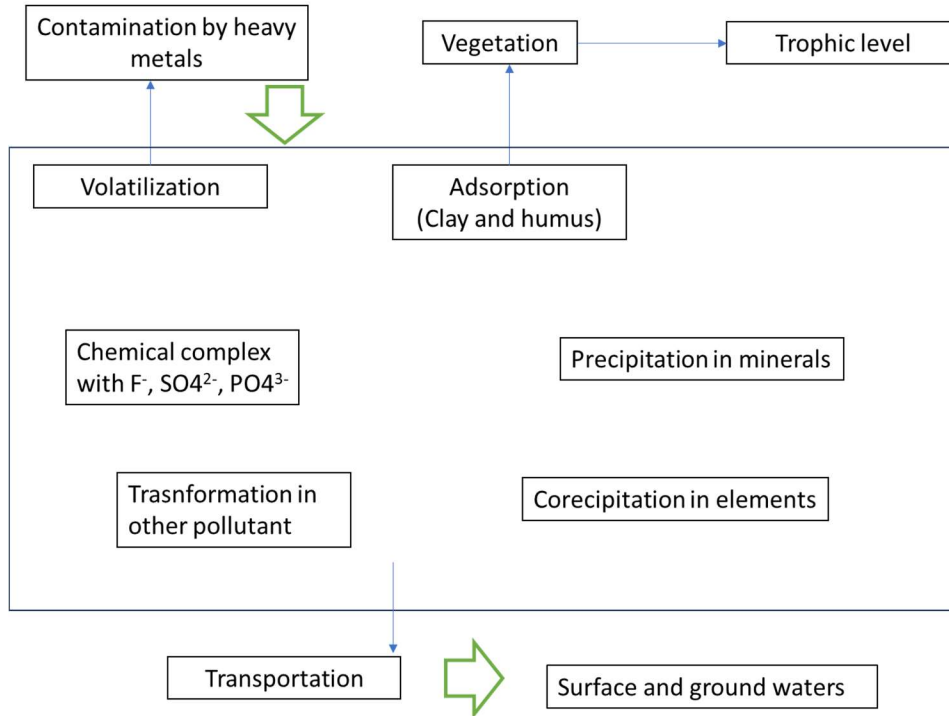


Fig. 2: Dynamics in the heavy metal system. (Source: García et al. 2002)

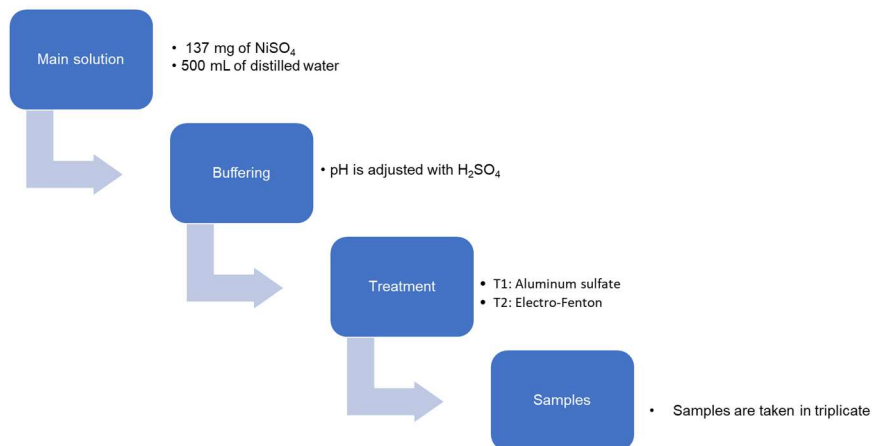


Fig. 3: Process experimentation.

$$\frac{RNS}{N} = \frac{58.69}{263} * \frac{98}{100} = 0.219 \quad \dots(2)$$

Once the ratio of nickel and nickel sulfate is found, we proceed to calculate the necessary amount, remembering that for 500 mL, 30 mg of nickel is needed, so we proceed to calculate the necessary sulfate with equations 3 and 4:

$$Ni = \frac{30 \text{ mg}}{RNS/N} \quad \dots(3)$$

$$Ni = \frac{30 \text{ mg}}{0.219} = 137 \text{ mg} \quad \dots(4)$$

## Treatments

**Aluminum sulfate treatment:** For the titration of aluminum sulfate, the ratios proposed by López-Ramírez et al. (2019) were proposed, which correspond to 30%, 60%, and 90%, which guarantee 3 scenarios, unsaturated, saturated, and supersaturated scenarios, which correspond to 300 ppm, 600 ppm and 900 ppm in 500 mL solutions with an acid pH of 2, 3 and 4 pH.

**Electro-Fenton treatment:** For the electro-Fenton process, the same methodology of Lopez et al. (2019) is used, which indicates that the Fenton process should be evaluated under the following conditions in Table 2:

According to the electrochemical conditions, Jumpatong (2002) is taken as a reference, which indicates that for electrocoagulation, the following is required (Table 3):

**Experimentation and validation of results:** For the validation of the data obtained, the Hanna spectrophotometer was used, following the following procedure:

Standard:

1. The low-range nickel method is selected in the spectrophotometer.

Table 2: Fenton reagent concentrations.

Concentration	Compound	Amount	Unit
Fenton 30%	Hierro	75	mg
	Peróxido	0.22	mL
Fenton 60%	Hierro	150	mg
	Peróxido	0.22	mL
Fenton 90%	Hierro	225	mg
	Peróxido	0.22	mL

Table 3: Requirements for electrocoagulation.

Parameter	Compound	Unit
Voltage	17.5 y 19	V
Electrode	Aluminio	-
pH	8 y 10	-

2. A beaker is filled with 25 mL of distilled water, and a packet of reagent A (HI 93740A-0) is added.
3. Add 1 mL of reagent B (HI 93740B-0) and shake to mix.
4. The solution is allowed to stand for 15 min.
5. Reagent C (HI 93740C-0) is added immediately, stirred, and dissolved.
6. The cuvette is filled with 10 mL and this will be the blank, which will be our nickel-free reference value.
7. The cuvette is placed in the cuvette, the hatch is closed, and zero is pressed.
8. At this point, our Hanna equipment is calibrated and ready for samples.

Sample:

1. 1 mL of sample is obtained, and the solution is diluted 1:10, derived that the range of the spectrophotometer covers 0.000 to 1.000 ppm with a minimum resolution of 0.001 ppm, and 2.5 mL of the sample is poured into 22.5 mL of distilled water.
2. A packet of reagent A (HI 93740A-0) is added and shaken until dissolved. It is important to dissolve all the powder since, during the Fenton reaction, Fe<sup>3+</sup> is produced, and this element in the ionic state can cause interferences.
3. 1 mL of reagent B (HI 93740B-0) is added, and it is shaken to mix.
4. The solution is allowed to stand for 15 min.
5. Reagent C (HI 93740C-0) is added immediately, stirred, and dissolved.
6. Once dissolved, the cuvette is filled with 10 mL and placed in the Hanna.
7. It is placed in the reader; the hatch is closed, and it is given to read.
8. The reading obtained by our spectrophotometer will be 10 times less than the actual reading, so it should be multiplied by 10. Example: If the Hanna marks a reading of 0.689 ppm, the actual reading will be 6.890 ppm.

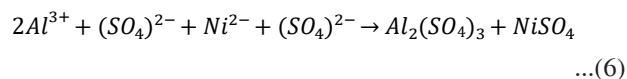
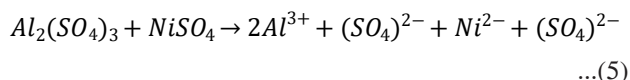
After obtaining the data from the experiments, we proceed to perform the analysis of means by the Tukey method at 95% confidence to have a statistical result that can tell us if there is a difference between the experiments, in addition to the electrocoagulation process, we proceed to perform a full factorial analysis, with 8 runs (24), to visualize the variables that influence the electro-Fenton process, in Minitab 19 (Fig. 4).

## Study Area

The present study was carried out in the facilities of the Instituto Tecnológico de Boca del Río since it was conducted with synthetic water and its treatments were at the laboratory level.

## RESULTS AND DISCUSSION

The results obtained by the chemical precipitation process are shown in Fig. 5, where we can see that despite varying the concentrations of aluminum sulfate, being this compound is the typical coagulant most used in wastewater treatment processes, its nickel reduction results are null. This is because when aluminum sulfate is dissolved in water, it dissociates and creates the sulfate ion, and with respect to nickel, this only associates with it, creating the nickel sulfate complex, the latter being our reagent for synthetic water. This can be seen in equations 5 and 6, so its use is not suitable in the treatment of water contaminated with nickel.



In the advanced electrocoagulation process, 12 experiments were carried out with a treatment time of 30 min and 60 min. These experiments were carried out in triplicate, and removal averages were obtained as shown in Tables 4 and 5:

According to those interactions, which explain graphically how the variables affect us with respect to the processes, it is observed that for the voltage, the optimum value is 19.0 V; according to the pH, the medium should be found in

10 pH at a concentration of 150 ppm of Fenton reagent and the time with the highest removal for most of the processes is 60 min (Fig. 6 & Fig. 7).

However, it can be observed in Table 4 and Table 5 that in the experiments with voltage 19 V, 10 pH with Fenton concentration of 150 ppm and 300 ppm, in the times of 30 min and 60 min, there is no difference in removal, however, for the validation of this assumption a Tukey test will be performed at 95% with respect to the times, this to determine if the electro-Fenton process.

As shown in Fig. 8 and analyzing both optimum processes, it can be observed that the electrocoagulation process with Fenton at conditions of 150 ppm, 10 pH, and 19 V has no statistically significant change, so we can conclude that, at 30 min of the process, the removal of nickel has reached its maximum removal. There will no longer be a notable advance in this process.

With respect to precipitation with aluminum sulfate, Pinillos 2013 indicates that chemical precipitation, filtration by membranes, electrolytic reduction, extraction by solvents, ion exchange, and adsorption have drawbacks such as low efficiency and applicability, which is demonstrated in this research since the treatment of synthetic water yielded zero efficiency because only ion exchange is present. There is no intermediate process, such as the generation of hydroxyl ions or electrocoagulation.

Rojas-Vargas et al. 2020, obtained similar removal results since these researchers obtained a maximum removal efficiency of 95% in a time of 60 min, and Jumpatong (2002) obtained results close to 98% in a time of 180 min; however, can be observed that this research obtained

Resumen del diseño

factores: 3 Diseño de la base: 3,8  
 Corridos: 24 Réplicas: 3  
 Bloques: 1 Puntos centrales (total): 0

Todos los términos están libres de estructuras alias.

Tabla de diseño (aleatorizada)

Corrida	Blq	A	B	C
1	1	+	+	+
2	1	+	+	-
3	1	+	-	+
4	1	+	-	-
5	1	-	+	+
6	1	-	+	-
7	1	-	-	+
8	1	-	-	-
9	1	+	+	+

#	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C15	C16	C17	C18	C19
	OrdenEst	OrdenCorrida	PICentral	Bloques	Voltaje	pH	Fenton	Remoción											
1	7	1	1	1	17,5	10	300	64,67											
2	19	2	1	1	17,5	10	150	54,00											
3	13	3	1	1	17,5	8	300	19,50											
4	15	4	1	1	17,5	10	300	67,50											
5	9	5	1	1	17,5	8	150	52,33											
6	1	6	1	1	17,5	8	150	52,17											
7	11	7	1	1	17,5	10	150	53,33											
8	2	8	1	1	19,0	8	150	98,67											

Fig. 4: Minitab 2019.

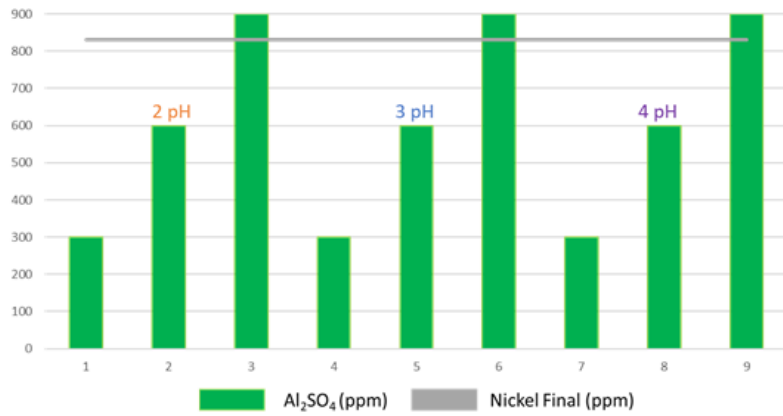


Fig. 5: Results of treatment with aluminum sulfate.

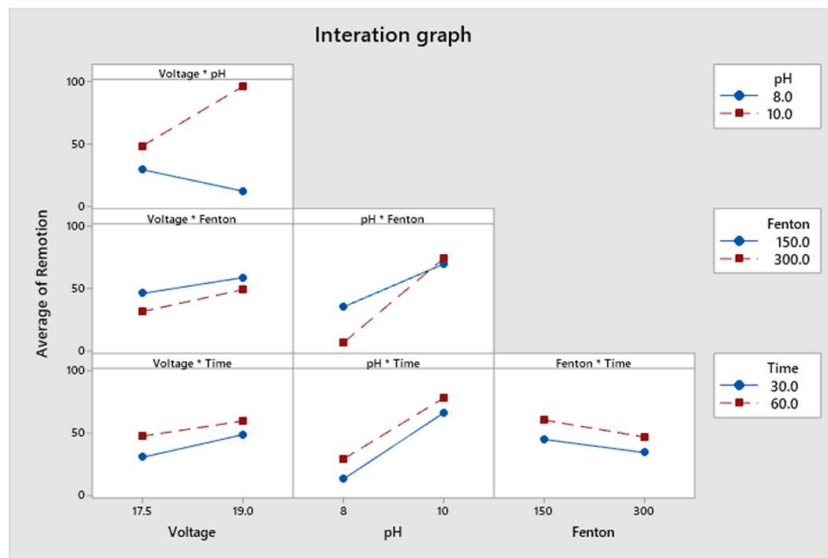


Fig. 6: Interaction graph.

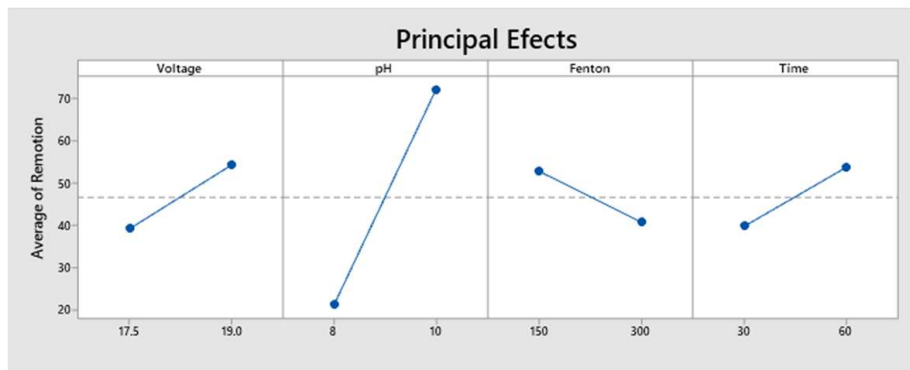


Fig. 7: Effects graph.

Table 4: Average results of advanced electrocoagulation at 30 min.

Voltage [V]	pH	Fenton [ppm]	Average Removal Percentage
17.5	8	150	48.39%
17.5	8	300	0.17%
17.5	8	450	0.61%
17.5	10	150	30.78%
17.5	10	300	42.83%
17.5	10	450	01.22%
19.0	8	150	3.00%
19.0	8	300	0.44%
19.0	8	450	0.39%
19.0	10	150	97.50%
19.0	10	300	93.89%
19.0	10	450	08.00%

Table 5: Average results of advanced electrocoagulation at 60 min.

Voltage [V]	pH	Fenton [ppm]	Average Removal Percentage
17.5	8	150	52.22%
17.5	8	300	17.56%
17.5	8	450	01.22%
17.5	10	150	53.83%
17.5	10	300	66.56%
17.5	10	450	05.06%
19.0	8	150	38.06%
19.0	8	300	08.61%
19.0	8	450	45.94%
19.0	10	150	97.56%
19.0	10	300	94.5%
19.0	10	450	57.17%

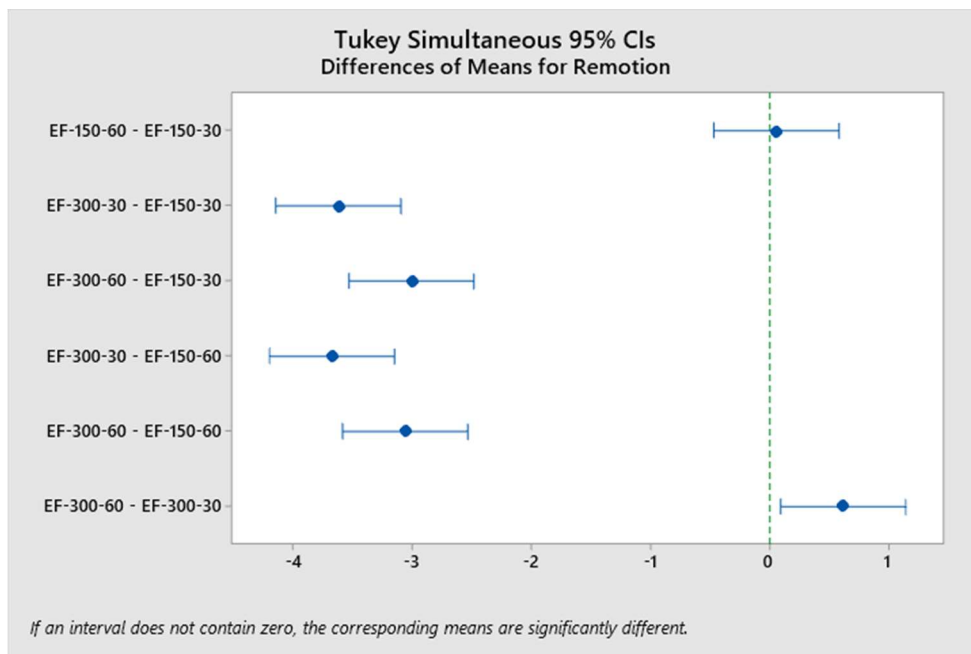


Fig. 8: Test of means with respect to time.

results of 97.50% of nickel removal at 30 min, half the time compared to them.

Martín (2008) mentions that the operating conditions of electrocoagulation depend on the chemistry of the aqueous medium, especially the conductivity, and pH, but also on the particle size, the type of electrodes, the retention time, the spacing between electrodes, and the concentration of the contaminants.

## CONCLUSIONS

When comparing the decrease of nickel (II) in the wastewater samples, we can see that the sample treated with aluminum sulfate  $[Al_2(SO_4)_3]$  did not have any decrease, so the ion exchange treatment for its reduction or elimination is not adequate; however, the advanced oxidation treatments such as electrocoagulation added with Fenton reagent resulted in an average maximum elimination of 97.56%, reducing from 60 ppm to 1,464 ppm of nickel in our water at 19 V, 150 ppm of Fenton reagent, and 10 pH.

Likewise, with the factorial design, it was corroborated



that the optimal variables for the experimentation are 19 V, 10 pH, a concentration of 150 ppm, and a time of 30 min, as corroborated by a Tukey analysis at 95% confidence.

With respect to time, it is observed that the addition of the Fenton reagent to the electrocoagulation process does affect, since authors such as Roja-Vargas et al. and Jumpatong have obtained results of 95% and 98%, respectively, in times from 60 min to 180 min, being the removal results similar to those presented in the present investigation. However, the times are reduced from 50% to 66% without compromising the percentage of nickel removal in the process.

As a conclusion, the advanced electrocoagulation process (electro-Fenton) is more efficient than traditional methods and even than typical electrocoagulation processes since the same efficiencies are achieved in a shorter time, thus reducing the use of energy. In addition, This process is eco-friendly with the environment since the chemicals discharged into the water to be treated are mostly iron, necessary in the geochemical cycle, and the peroxide, when volatilized and consumed, generates a considerable amount of water, so it is recommended from the environmental point of view.

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