Journal of Materials and Environmental Sciences
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2018, Volume 9, Issue 5, Page 1525-1535

https://doi.org/10.26872/jmes.2018.9.5.168

http://www.jmaterenvironsci.com

Removal of zinc and iron from synthetic contaminated solution by electrocoagulation (EC): Comparison between iron and aluminum electrodes

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Received 13 May 2017,
Revised 26 Jul 2017,
Accepted 31 Jul 2017

Keywords

- ✓ electrocoagulation;
- ✓ iron electrode;
- ✓ aluminum electrode;
- ✓ zinc;
- ✓ iron
- ✓ pH;
- ✓ duration.

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Abstract

The present work presents the results obtained during the treatment of a synthetic aqueous solution contaminated by zinc or iron at the laboratory scale by electrocoagulation process. Iron and aluminum electrodes are used separately. The influence of the electrocoagulation parameters as well as the pH of the liquid have been studied. In order to understand the process of EC, natural decanting was done after EC. The treatment with the iron and aluminum plates with a voltage of 1.5V and a current density of 1.5 $A.m^{-2}$ allowed a heavy metal removal after 5 hours of settling. It was found that the treatment duration, the settling time and the pH of the medium play a major role in the treatment by electrocoagulation. The treatment with Aluminum electrode allowed 54.47% and 100% removal of Zinc and iron respectively whereas iron electrode allowed 40.65% and 100% removal of zinc and iron respectively. The treatment with aluminum electrode allows fast removal of the two heavy metals in solution. Treatment with aluminum blades seems to be more effective than iron blades.

1. Introduction

Runoff of rainwater on soils containing heavy metals, atmospheric pollutants [1-2], domestic wastewater and the discharge of hospitals, industries and agriculture are a dangerous sources of water pollution [2].

Agricultural and industrial sources, coming from water overloaded by products from spreading (fertilizers, pesticides) or from industrial processes that use water in the composition, manufacture and cleaning of products become an important source more than the others due to their composition [3, 4]. Industrial activity in the extraction or production of metals generates effluent charged with toxic metallic elements at varying concentrations and sometimes rejected without treatment in the receiving environment [4]. Thus, water pollution by heavy metals is currently of great concern in view of their harmful consequences for living beings. This fact leads to the use of more appropriate criteria for the protection of populations exposed to contamination by these metallic species, so it is important to look for adequate means of purifying contaminated water [5, 6].

Several methods of water treatment have been developed. The present study focuses on electrocoagulation. Electrocoagulation (EC) is a polluted water treatment technique that has proved to be effective in the treatment of certain soluble or colloidal pollutants, such as found in liquid discharges those containing heavy metals, emulsions, suspensions [7-10].

In electrocoagulation, the coagulant is released into the solution in situ by the dissolution of a sacrificial anode. When the electrodes are supplied with current, there is simultaneously a dissolution of the metal of the anode and a disengagement of the hydrogen gas H_2 in the vicinity of the cathode. Metals (aluminum and iron) are generally used as an anode [11-12]. Their dissolution leads to the formation of metal hydroxides, metal oxyhydroxides and polymeric hydroxides. These compounds generally have better coagulation efficiency than chemical coagulants. The compounds formed destabilize the colloids in suspension by adsorbing, neutralizing or precipitating the polluting species dissolved in the liquid [6, 13]. Even many papers have presented some works on EC, the comparative simultaneous removal of zinc and iron by EC hasn't been presented yet and the effect of the type of metal electrode on their removal hasn't been developed.

The objective of this work is to study EC as a process to remove zinc and iron from the synthetic solutions depending on the pH and the treatment time. Then a comparison will be made between the electrodes of iron and aluminum but also of studying the effect of the pH variation during the treatment by electrocoagulation.

2. Material and methods

2.1. The synthetic solution to be treated

The synthetic solution is prepared in the laboratory by dissolving sodium nitrate salt ($NaNO_3$), zinc sulphate ($ZnSO_4$) and Mohr's salt ($Fe(SO_4)2.(NH_4)2$- 6 H_2O) in distilled water. After complete dissolution of the salts, the contaminated zinc and iron solution is submitted to the EC treatment. The electrocoagulation treatment is used to removezinc and iron in the initial proportions shown in the table below:

Table 1: Heavy metals concentration on the solution

	Zn	**Fe**
C1 (mg/L)	0.14	2.14
C2 (mg/L)	0.12	2.00
C3 (mg/L)	0.11	1.85
Average (mg/L)	0.12	1.99

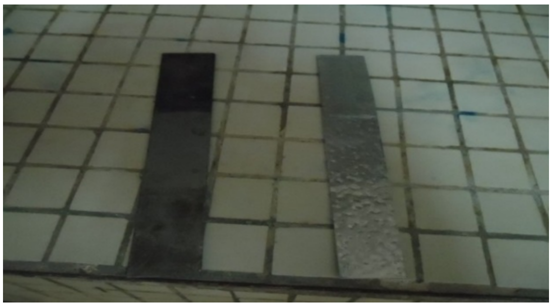
The metals concentration in the solution is measured three times (C1, C2and C3)(Table1) and then averaged. After the treatment, an average of three measurements will also be made.

2.2. The experimental device

The treatment device is an electrochemical reactor composed of a planar iron or aluminum electrode and a cylindrical counter electrode connected in series.

150 mL of the solution is placed in a beaker of 250 mL, a beaker acting as an electrochemical reactor. Throughout the study, this volume is constant. In the beaker,the iron or aluminum electrode is immersed, a counter electrode, a reference electrode and a magnetic bar are also immersed in the beaker. The beaker is then placed on a stirrer set at 200 rpm to allow the synthetic solution to be always homogeneous. The whole cell is connected to a potentiostat which delivers an electrical voltage of 1.5V low to avoid unwanted reactions like water heating. The potentiostat is connected to a computer which allows to set the parameters for the study, to start the treatment proper and to display on the screen characteristic curves of the treatment.

Two electrodes were used separately in the electrochemical reactor for the treatment of the synthetic solution. The iron electrode or the aluminum electrode are separated by a distance of 3 cm from the counter electrode. This low distance is optimized to avoid ohmic drop but not so large to have sufficient reactions between the electrode and the counter electrode. The two electrodes are flat and of the same size and have the same length of 15 cm and a width of 2.5 cm (figure 1). After each treatment, the electrode is cleaned with a sponge and then rinsed and quenched in a beaker containing distilled water.



Electrode of iron Electrode of aluminum

Figure 1: Working electrodes

These electrodes are immersed at a height of 8 cm giving a working surface of 20 cm^2. The geometry allows easy cleaning of the electrodes and easy implementation.

The potentiostat is connected to the power supply of the national electricity company supposed to be 220V. The intensity I is determined by the relation:
$I = Uk/d$(Eq.2-1)
I = the intensity of the delivered current.
U = the voltage delivered by the generator
k = the conductivity of the solution
d = the distance between the working electrode and the counter electrode.
The following figure shows the two electrodes which were used for processing during the treatment.
The current allows a homogeneous dissolution of the metal at the anode and a release of the gas at the cathode.
The dissolution is a function of the time that takes place of treatment of the synthetic solution. Let Me be a given mass of the metal before treatment and let M be the molecular mass of the dissolved metal and Ms the mass of the metal after treatment the following relationship is obtained which gives the material balance at a time $\Delta t = \tau$ as follows:
$Me - Ms = MI \phi\tau / nF$(Eq. 2-2)
M = molecular weight of dissolved metal
I = the intensity of the current flowing through the cell.
N = the number of electrons involved in the electrochemical reaction (2 electrons for the iron electrode and 3 electrons for the aluminum electrode).
F = number of Faraday
ϕ = the faradic yield of dissolution.
From the above, considering only the resistance of the synthetic solution, the voltage at the edge of the cell can be determined by the following relation.
$U = RI$ (Eq. 2-3)
R = resistance of the synthetic solution.
U = Tension de la cellule
I = Intensité du courant

2.3. Analytical techniques

2.3.1. pH measurements of solutions

The pH is measured using a combined meter pH / EC / TDS / Temperature (pH, EC, TDS, Temperature) of the mark MARTINI (Mi 806) Synthetic solution before and after treatment.
The measurements are carried out within the solution in order to avoid the influence of the pH in the vicinity of the electrodes since this is acid close to the anode and basic close to the cathode.

2.3.2. Conductivity measurements

The electrical conductivity of the solutions is also measured by the MARTINI brand instrument (Mi 806) combined meter pH / EC / TDS / Temperature. It is sufficient for the same device to select the conductivity option.

2.3.3. The balance

The various salts, in particular zinc sulphate and mohr's salt are weighed out with a balance of type 88ADAM PV214 Max 210g d = 0.0001 g. The balance is connected to the electric current and is placed in a very secure enclosure and protected against any influence of the external environment that can affect the measurements. The weight of the working electrodes is also measured with the same balance.

2.3.4. The agitator

Two magnetic stirrers are used during the experiments. A Nevdeloh Mr Coot agitator allows stirring, dissolving and homogenizing the salts in distilled water before treatment. The stirring speed is 500 rpm.
The second stirrer set at a speed of 200 rpm is used to homogenize the metallic dissolution in the synthetic solution during the treatment.

2.3.5. Analysis of iron and zinc

2.3.5.1. Decantation

After each treatment, the synthetic solution is collected in a 250 mL beaker and then left for decantation at least 5 hours. During this period the particles are found at the bottom of the beaker constituting the sludge of the treated solution. Then the supernatant is collected in another beaker and the pH of the solution is put to a neutral pH (substantially 7) before measuring.

2.3.5.2. Analyzes

The HACH Color Meter is the device that is used to measure the concentrations of heavy metals in solution. The use of the colorimetric can be divided into four phases:

- Colorimeter Preparation
- Preparation of the sample
- Setting the device zero
- Measurement of prepared sample

The following cells in figure 2 are to measure metals concentrations in the solution before and after treatment.

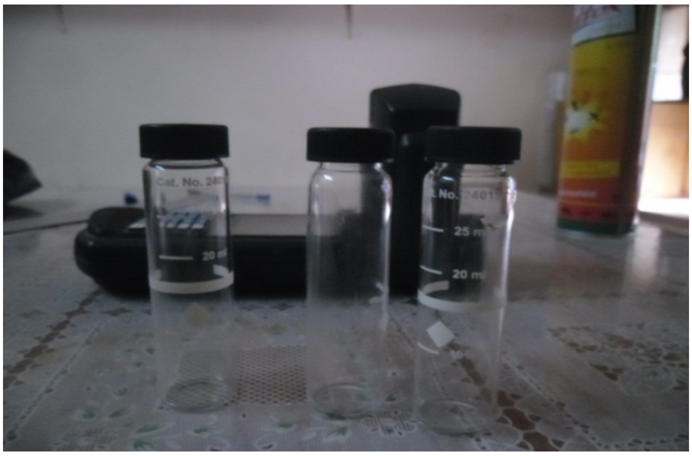


Figure 2: cells of measurement

The colorimeter measures only concentrations in its range of measurements. For our study the concentrations of 0.1mg are chosen to be in the range of the wavelength of the colorimeter. The following table (Table 2) illustrates the measurement intervals of the colorimeter for the metals of our study.

Table 2: The parameters and ranges available for the DR / 850

Parameters	Primary form	Others forms	Measuring rangeof the primary form(Mg / L unless otherwise stated)
Aluminum	Al	Al_2O_3	0 – 0.800
Iron	Fe	-	0 – 3.00
Zinc	Zn	-	0 – 3.00

2.3.6. Abatement determination

The abatement rate is calculated by the following relationship: Mean concentration at t = 0h minus the mean concentration at t = 1h after treatment divided by the concentration at t = 0h, then multiply by 100.

$$\mathrm{Tr} = [(\mathrm{Ci} - \mathrm{Cf}) / \mathrm{Ci}] * 100 \qquad \text{(Eq.2-4)}$$

Tr = Abatement rate
Ci = Initial Concentration
Cf = Final Concentration

3. Results and Discussions

3.1. Determination of the appropriate pH for metals removal

According to many works, pH is considered as a main parameter that has real effect on electrocoagulation process efficiency [4, 14-18]. The objective of this part is to determine an appropriate pH which allows good removal of pollutants (metals).

3.1.1. Effect of pH on zinc elimination after one hour EC treatment with iron electrode

The figure 3 shows two high level diagram, one at pH equal to 4 and the second at pH 7 with 25% reduction of Zinc. The elimination of the zinc in a solution by electrocoagulation with the iron electrode is favorable in the less acidic and neutral media. In basic media, there is a little treatment. The best removal of zinc in acidic and neutral media is explained by the presence of non-soluble form of iron dissolved at the anode and then the formation of iron polymers which adsorbzinc and settle with it [19]. This explanation is also reported by Zewail and al. during chromium treatment [20].

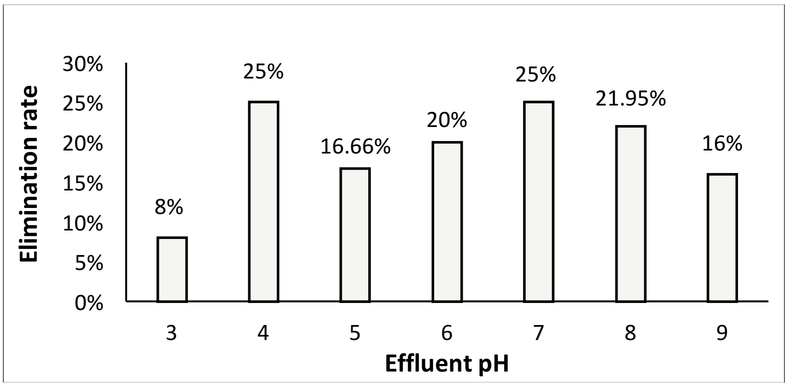


Figure 3: Effect of pH on zinc removal with iron electrode

3.1.2. Effect of pH on iron elimination after one hour EC treatment with iron electrode

The figure 4 represents the effect of pH on iron removal with the iron electrode. The figure shows a high level removal of iron at pH equal to 8.

It should be emphasized that, apart from the very acidic pH where there is no elimination, the abatement rate is clearly high for the rest of the pH studied and is greater than 50% after pH = 5. Similar results have been reported by Mahmad and al. with chromium removal treated by iron electrodes [18].

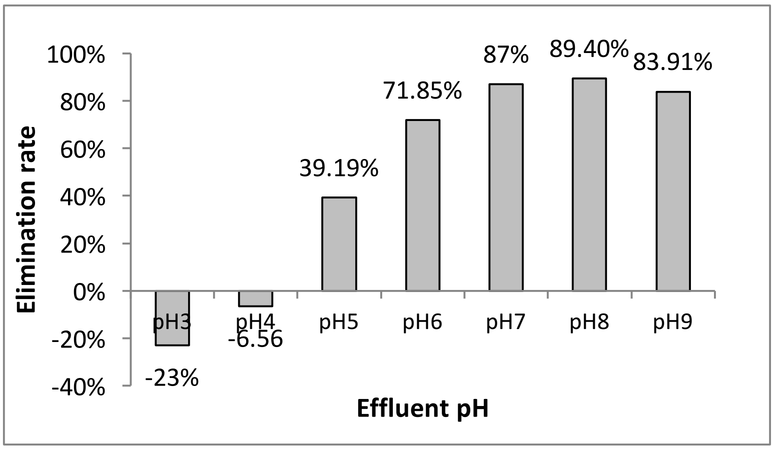


Figure 4: Effect of pH on iron removal with iron electrode

It is observed that in the acidic media there is no removal of iron during one hour treatment. In contrary, there is an increase in the iron concentration. This results from the fact that the iron electrode is attacked in an acid medium. And since we use an iron electrode, for all pH below 5, the electrode is attacked and dissolves in the synthetic solution thereby increasing the iron concentration in the medium. That is also confirmed by the works of Zongo [7].From the results, it can be said that the pH of the synthetic solution plays a decisive role in the formation of precipitates of metal hydroxides which contribute to flocculation during coagulation and electrocoagulation. The pH of the medium is therefore critical in order to obtain a better yield for a much reduced time.

3.1.3. Effect of pH of synthetic solution on zinc elimination after 1 hour EC treatment with aluminum electrode

For the figure 5 showing the elimination of zinc with the aluminum electrode, a slight increase from pH value equals 4 is observed. From pH equal to 7, there is a high elimination ratesof zinc on the solution. The best elimination rate is observed at pH equal to 9 but could be considered as low (25%).The authors [14, 19] have found that at neutral pH, more hydroxyl ions are producedimproving zinc removal when aluminum electrode is used.This confirms our results. Treatment at pH 9 and for a long time would be ideal for the total removal of zinc from the solution.

3.1.4. Effect of pH of synthetic solution on elimination of iron after one hour EC treatment with the aluminum electrode

The EC treatmentwith aluminum electrode after one hour treatment allows 86.27% of iron removal at pH 6 according to the figure 6.

It decreases to pH equal to 7 and grows again to reach a second high levelin basic medium at pH value equals 8 to reach again 86.27% abatement. This elimination rate is confirmed by the research of Bouaouine et al. [1].

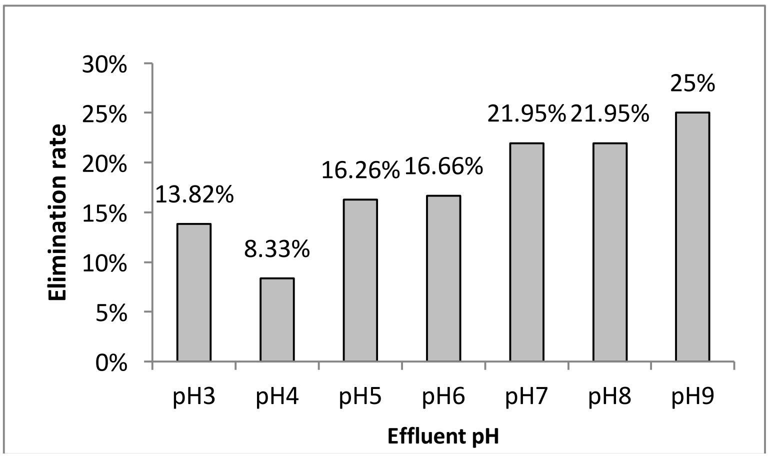


Figure 5: Effect of pH on zinc removal with aluminum electrode

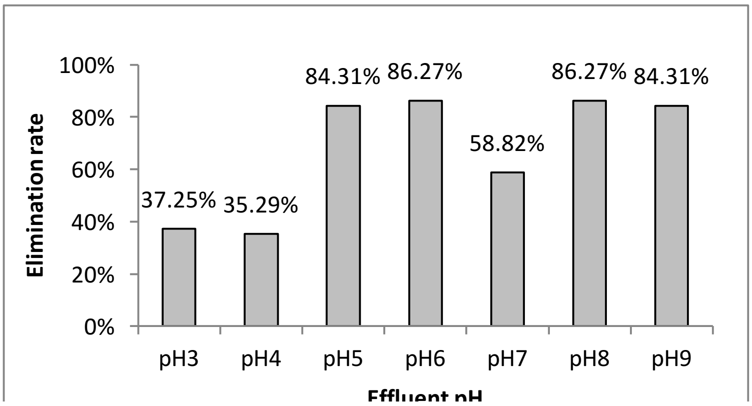


Figure 6: Effect of pH on iron removal with aluminum electrode

The media pH equal to 5 and 6 and themedia equal to 8 and 9 allow an elimination rate of more than 84.31% after one hour EC treatment. These media is considered as the ideal for the treatment of the synthetic solution by electrocoagulation with the aluminum electrode.

The results obtained are confirmed after the treatment, by measuring the solution obtained with the colorimeter and by using the equation (Eq.2-4). Strong acid solutions are highly conductive, due to the presence of H^+ ions. During electrolysis, the voltage is almost constant (1.5 V) due to the effect of NaCl which acts as a support electrolyte and which contains the chloride ions which prevent the passivation of aluminum. During the electrolysis, the solutions appear first clear, then the flocs are formed, which makes them progressively turbid and this is in agreement with Bouaouine et al. [1].

3.2. Effect of time on elimination of iron and zinc with aluminum electrode

The study carried out previously on the removal of heavy metals allows us to understand that the the optimum pH for the removal of iron is 8 with 86.27% elimination rate when aluminum is used as electrode. To study the effect of time on the removal of metals,the pH equals 8 is chosen as an optimal medium of iron from the synthetic solution.

3.2.1. Zinc removal at pH equal to 8 with aluminum electrode

Through the figure 7, there is a slight increase of the elimination of zinc in the solution during the treatment. Between 1 hour and 3 hours of treatment, the elimination is substantially 10% at the interval of 1 hour. The elimination decreases after 3 h of treatment and reaches 54.47% after 5 h of treatment. This is due to the fact that zinc is not treated here at its optimum pH of elimination.

3.2.2. Iron removal at pH = 8 with aluminum electrode

At pH8, we observe that iron removal is function of treatment duration as it is observed in figure 8. After 3 hours, more than 91% of iron are been removed from the synthetic solution. After 5 hours of treatment 100% of iron is removed. This is in agreement with Yildiz et al. [21].

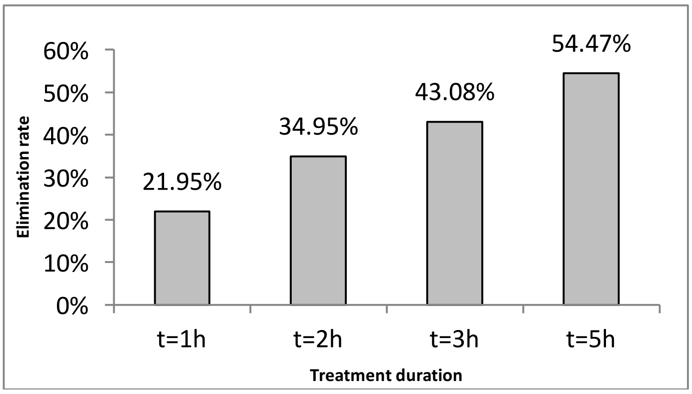


Figure 7: Histogram of zinc removal as a function of time with the aluminum

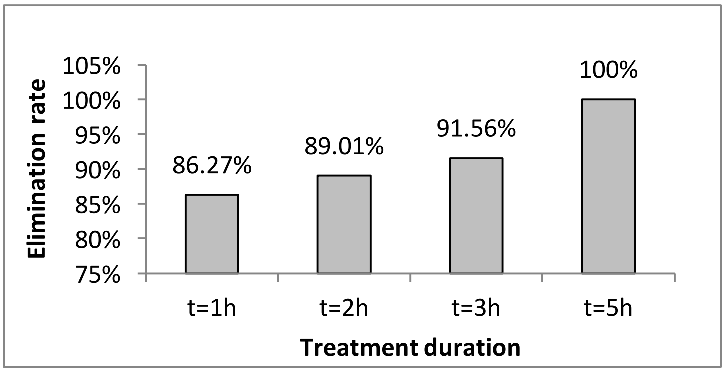


Figure 8: Effect of time on iron elimination with aluminumelectrode

3.3. Effect of time on the elimination of Fe and Zn with the iron electrode.

3.3.1. Zinc removal at pH = 8 with the iron electrode

We observe in the figure 9 that the concentration of zinc in the solution is the same at 2 hours of treatment and 1 hour of treatment. After 5 hours of treatment the rate is still relatively low of 45.65%. This could be explained by the fact that pH 8 is not suitable for the removal of zinc with the iron electrode. We need a relatively longer time if we want to attain the best pH to remove entire zinc from the solution.

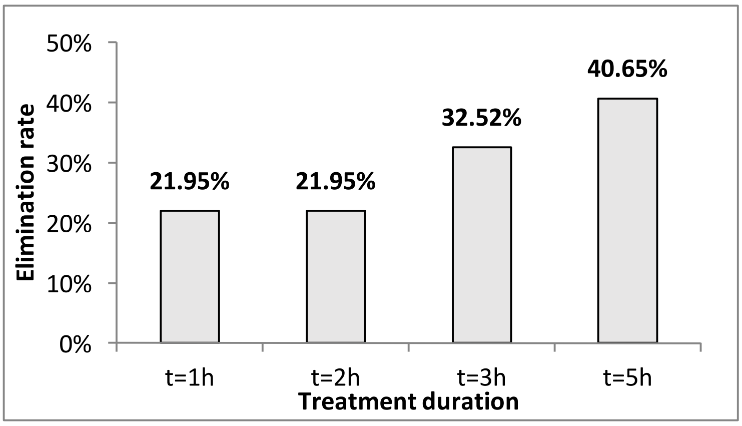


Figure 9: Effect of time on Zinc removal with the iron electrode

3.3.2. Iron removal at pH = 8 with the iron electrode

A slow elimination of the iron with the iron electrode after 1 h, 2 h and 3 h of treatment is observed in the figure 10, respectively from 80.39% to 85.09% and 87.58%. But at 5 hours of treatment the whole amount of iron in the solution is removed. PH 8 is therefore the ideal medium for the removal of iron with the iron electrode in a synthetic solution and this is confirmed by Zewail and al. [20] and Yildiz et al. [21].

3.4. Effect of time on elimination of iron and zinc compared between iron and aluminum electrode

3.4.1. Effect of time on the removal of Zn compared between the iron electrode and the aluminum electrode

By comparing the rate of elimination of the zinc in the solution during the electrocoagulation treatment between the iron electrode and the aluminum electrode, the graphs below are obtained. It can be said in agreement with the work by Tchamango [22] that the aluminum electrode is suitable to eliminate zinc more quickly in asynthetic solution than the iron plate.

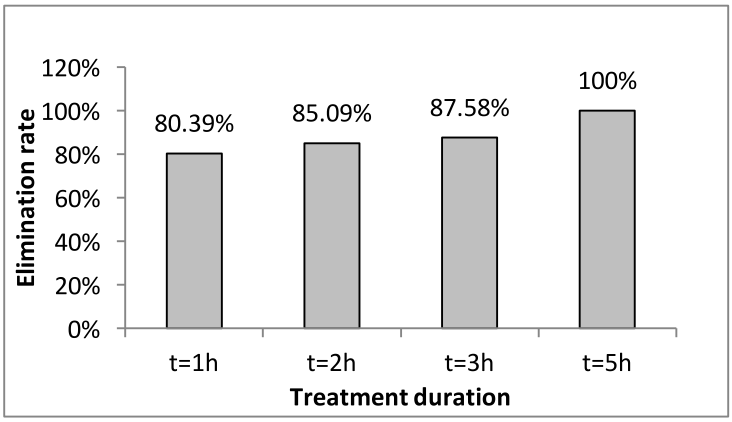
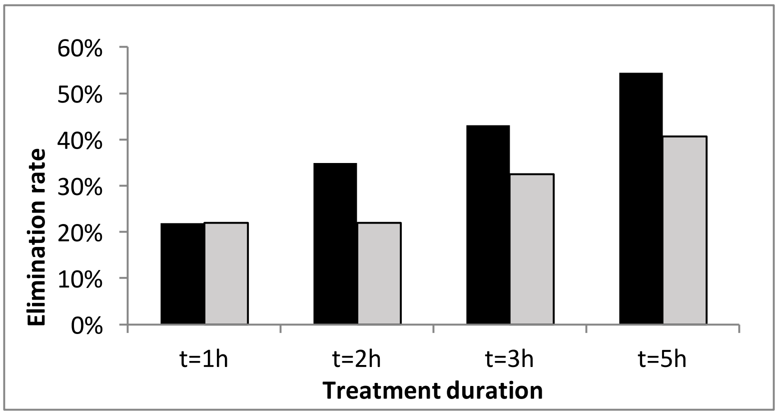


Figure 10: Effect of time on iron removal with the iron

It is observed that the zinc removal rate with the aluminum electrode shown by black diagrams is clearly above that of the iron electrode shown by grey serie throughout the treatment time. The aluminum electrode is more suitable to remove zinc from the synthetic solution than iron electrode by EC.

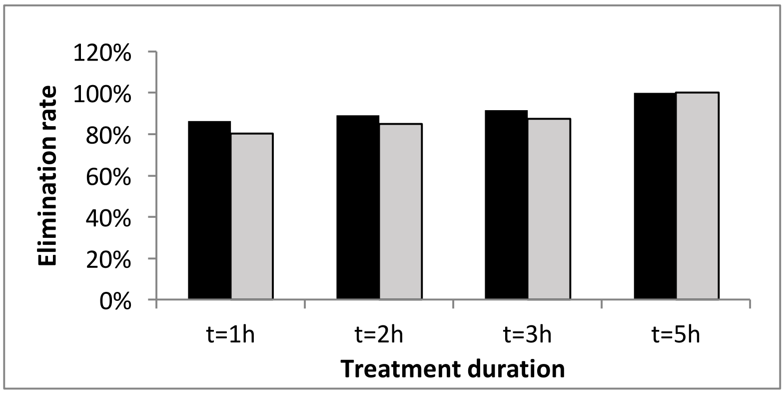
3.4.2. Effect of time on the removal of iron compared between the iron and aluminum electrode

A comparative study of the removal of iron from synthetic solution by the iron and aluminum electrode in figure 11 and 12 show an increasing trend in both situations, which is synonymous with gradual elimination. The aluminum electrode is more suitable to remove iron from the solution than the iron electrode. This results in the fact that over the entire treatment time the quantity of iron eliminated with the aluminum electrode is clearly above that of the iron, which is in agreement with the research of Bouaouine et al.[1]. The appearance of the representative diagram shows a rapid increase from the beginning of treatment to completion.



Serie in black = aluminum electrode Serie in grey = electrode of iron

Figure 11: Effect of time on the removal of zinc compared between the iron and aluminum electrode



Serie in black = aluminum electrode Serie in gray = electrode of iron

Figure 12: Effect of time on the removal of iron compared between the iron and aluminum electrode

Through the results of this analysis it can be said that the use of the aluminum electrode in the treatment of heavy metals, in particular iron with the electrocoagulation method, is the most indicated. With aluminum electrode there is a quick remove of metal compared to iron electrode.

3.5. Evolution of the synthetic solution pH during the treatment with iron and aluminum electrodes

After each treatment, the synthetic solutions are collected in 250 mL beakers and then the pH is measured. A variation in the pH of the solution is observed by the measurements taken during the treatment of the synthetic solution.

3.5.1. Evolution of the pH on the removal of Fe, Zn with the iron electrode

Through the results of and the figure 13 below, it can be seen that the longer the treatment time the more the medium tends to become neutral then acid. From the first hour of treatment, a great drop in the pH of the medium passing from 8.05 to 7.46 is observed. This is justified by the fact that there is more release of H^+ ions into the solution. This is due to the corrosion of the iron anode but also to the dissolution of the metals in solution. After three hours of treatment the medium becomes more and more acid but with very little variation.

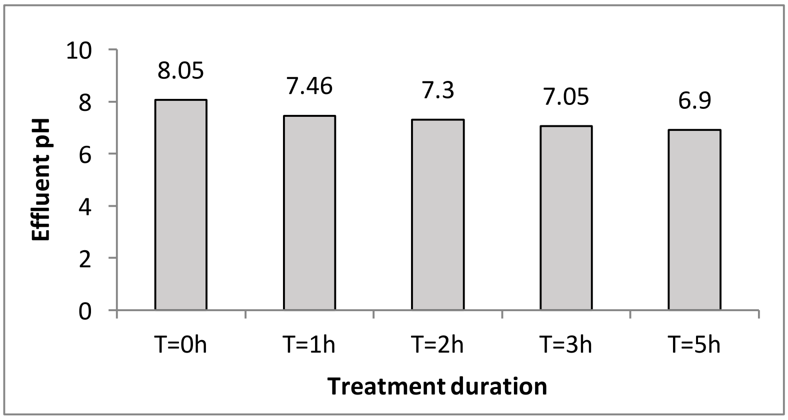


Figure 13: Variation of the pH during the treatment with the iron anode

3.5.2. Evolution of the pH of the synthetic solution during the removal of iron and zinc with the aluminum electro

The results below resulting from the pH measurements after each treatment show a decrease of the pH of the medium ranging from a basic pH to a neutral pH and then acidic pH. However, a slight decrease of pH was observed throughout the treatment time. This decrease can be explained by the fact that the electrolysis which occurs during the electrocoagulation treatment makes it possible to progressively release, as a function of the treatment time, H^+ ions making it possible to make the solution more acidic. The aluminum electrode is oxidized under the action of direct current to give dissolved Al^{3+} ions, which react with water to give polymeric or monomeric species according to the following reaction:

$$x\,Al^{3+} + y\,H_2O \rightarrow Al_x(OH)_y^{(3x-y)+} + y\,H^+ \text{(Eq.3-1)}$$

The H^+ ions produced at the anode create an acid medium on the surface. Dobolyi [23] shows that the pH may become less than 4.5 and this confirms the results.

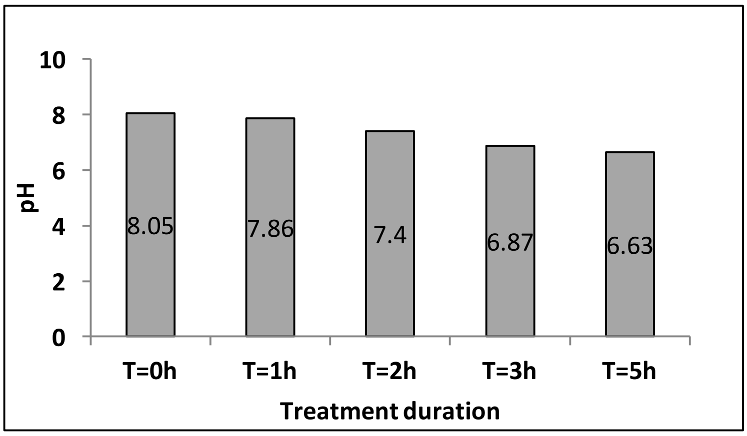


Figure 14: pH variation during treatment with aluminum anode

Some authors have studied the variations of the final pH during electrocoagulation and found that it's function to the initial pH. The results show that the final pH increases in acidic medium and decreases in basic medium [15, 22]. That is confirmed by the results of the two figures 13 and 14 above.

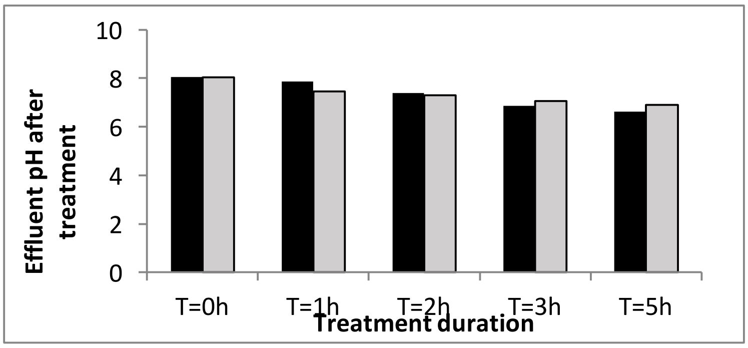
It is also justified by the fact that during electrocoagulation the electrodes are submitted to corrosion. That generally takes place in the presence of a supporting electrolyte (sodium chloride in this case) or acids. In this case, the quantities of metal introduced into solution are higher than those calculated by application of the Faraday law. However, the consequences of this corrosion are not fundamentally different from those expected in an electrolysis and this confirms Tchamango'sworks [22]. The pH variations during electrocoagulation experiment are essentially determined by the nature of the solutions and by the cathodic reactions.

3.6. *Compared effect of the synthetic solution pH on the removal of iron and zinc between aluminum and iron electrodes*

The figure 15 below can be divided into two parts.

The first part lies between t = 0 and t = 2. For this part of the figure, it is noted that the variation of the pH of the synthetic solution treated by aluminum electrode is higher than iron electrode EC. This is due to the fact that during this period there is less H^+ ion generation which can lower the pH of the solution with the aluminum electrode faster than the iron electrode. The iron electrode thus oxidizes very rapidly and allows the release of many positive ions into the solution. This is due to the fact that during electrocoagulation the electrodes are submitted to corrosion. This generally takes place in the presence of a supporting electrolyte (sodium chloride in this case) or acids. In this case, the quantities of metal introduced into solution are greater than those calculated by application of the Faraday law, which is in agreement with the work by Tchamango [22].

The second part starts from t = 3 to t = 5. During this period, the diagram of pH evolution during the electrocoagulation treatment with the iron electrode passes over that of the aluminum electrode. As the pH curve of aluminum treatment continues to decrease, that with the iron electrode tends to stabilize and varies very slightly.



Series in black = aluminum electrode Gray series = iron electrode

Figure 15: Compared variation of the pH during the treatment between the iron electrode and the aluminum electrode

The ions and other chemical species present in solution are responsible for the pH of the synthetic solution by their acid-base properties and their solubilities. These compounds are capable of being replaced by the Al^{3+}, Fe^{2+}, and Fe^{3+} ions, which also have more or less marked acidic properties. For the case of our study, all the solutions are rendered at a pH substantially equal to 8 before any treatment. The measurements after treatment differ depending on whether an iron electrode or an aluminum electrode is used. It is also observed that regardless of the electrode used, the pH after treatment is significantly lower than the pH before treatment and this is in accordance with the research of Lemlikchi [5] and Bennajah [24].

Conclusion

This study allowed us to understand the capacity of the electrocoagulation method to remove heavy metals in a solution. By the various processes and the various working anodes used, in particular iron and aluminum electrode, we obtain complete elimination after five hours of treatment at the optimum pH. The treatment with Aluminum slides is particularly more suitable for iron and zinc than iron slides. This is consistent with the results of several researchers including Bouaouine et al. for the removal of Cr, Zn, Ni and Fe, respectively, 79%, 86%, 89% and 90%, respectively.

We also note that the variation of pH depends on the nature of the anode. For the same values of the ratio [Metal]/[H^+], higher pH values are reached with iron electrode at the end of the electrolysis compared to aluminum electrode. This can be explained by the fact that the iron (II) hydroxide is formed at a higher pH before oxided to form Fe (III) whereas with aluminum, one form of Al (III) can be formed.

It is therefore necessary in order to have a good yield to find the suitable medium pH of elimination and to use the most appropriate anode for each metal.

The future to our study may consist to develop pilot to address real effluent and the theoretical simulation of the phenomena occurred during EC treatment. Solar energy can be also prospected to replace the fossil energy.

Acknowledgments- The authors would like to thank the ANEC / ISP network and its financial partner the International Science Program (ISP) for the financial support in building the scientific and technical capacities of network members through South-South cooperation.

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