



Removal of copper and lead by electrocoagulation process: effects of experimental parameters and optimization with full factorial designs

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Received 06 Jan 2023,

Revised 31 Jan 2023,

Accepted 01 Feb 2023

Keywords:

- ✓ metal ion;
- ✓ electrocoagulation
- ✓ monopolar;
- ✓ full factorial design;

Citation: Ano J., Koné H., Yapo N. S., Drogui P., Yao K. B., Adouby K. (2023) Removal of copper and lead by electrocoagulation process: effects of experimental parameters and optimization with full factorial designs, *J. Mater. Environ. Sci.*, 14(2), 173-183.

Abstract:

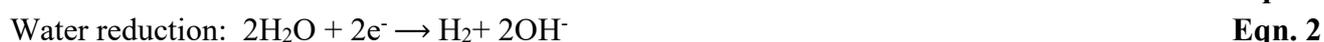
The comparison of metal ions (Cu^{2+} and Pb^{2+}) removal by electrocoagulation (EC) process has been investigated. The effluents were treated in a batch reactor containing ten iron electrodes connected in monopolar configuration. The effects of factors such as initial metal ion concentration ($45 - 85 \text{ mg L}^{-1}$), current intensity ($0.3-1 \text{ A}$) and electrolysis time ($20 - 60 \text{ min}$) were quantified using full factorial design (FFD). The respective contributions of the main factors on the metal removal rates are 30.24%, 12.9% and 35.21% for Pb^{2+} and 3.05%, 36.43% and 39.07% for Cu^{2+} . The signs of the coefficients indicate that concentration has a negative effect on the treatment efficiency of both cations, while current and time have positive effects. Optimization of the process resulted in the removal of 95.15% of Pb^{2+} and 80.22% of Cu^{2+} from the water. Electrocoagulation is therefore a very effective process for removing metal cations from water.

1. Introduction

Water pollution by heavy metals has become a global problem threatening the environment, health and food security (Lu *et al.*, 2023; Siddiqui *et al.*, 2023). Heavy metals come mainly from the wastewaters of industrial, agricultural and mining activities (Bazrafshan *et al.*, 2015). When heavy metal wastewater is not treated properly and is released into the environment, they are found in soils as well as in surface and groundwater (Yobouet *et al.*, 2016). As they are non-biodegradable, they can persist in these receiving environments and in living organisms for long periods of time, creating serious ecological and public health problems (Siddiqui *et al.*, 2023; Martins *et al.*, 2012). Copper and lead are among the most dangerous metals for humans (Bouguerra *et al.*, 2015). They cause severe damage to vital organs (kidney, nervous system, liver and brain) and can lead to infertility, abortion, stillbirth and neonatal death (Bouguerra *et al.*, 2015). To date, various treatment methods are used to reduce heavy metal pollution in water including lead and copper. These methods include chemical precipitation, ultrafiltration, reverse osmosis, activated carbon adsorption, ion exchange and electrochemical

methods (Adhoum *et al.*, 2004; Sadoon and M-Ridha, 2019 ; Siddiqui *et al.*, 2023). The adsorption of heavy metals by porous substances and active functional groups is efficient (Siddiqui *et al.*, 2023) but moves them from one phase to another thus constituting secondary waste. In recent years, electrocoagulation (EC) has received considerable attention because it is simple to use, less costly, requires fewer chemicals and is easily automated (Shokri and Fard, 2022). It is an electrochemical process that generates *in situ* different types of coagulants by redox reactions at the electrodes and hydrolysis reactions in the effluent (Boinpally *et al.*, 2023; Othmani *et al.*, 2022). With iron electrodes, the main reactions that take place in the EC process are illustrated by **Eqns. 1- 5** (Adou *et al.*, 2022; Hakizimana *et al.*, 2017; Bako *et al.*, 2018 ; Manikandan and Saraswathi, 2023; Moussa *et al.*, 2017)

At the electrodes



Within the solution



The $\text{Fe}(\text{OH})_2$ and $\text{Fe}(\text{OH})_3$ species, with their large specific surface areas, are beneficial for a rapid adsorption of pollutants (Ano *et al.*, 2020; Drogui *et al.*, 2007). The removal of metal ions from water results from precipitation, co-precipitation and especially adsorption on iron hydroxides (Meunier *et al.*, 2006). In the literature, studies on the treatment of heavy metals by EC have been described (AlJaberi and Hawaas, 2023 ; Bhagawan *et al.*, 2014; Gatsios *et al.*, 2015; Meunier *et al.*, 2006). Generally, the work consists of simultaneously removing the metals in aqueous solution. A comparative study on metal removal by EC, taken separately in solutions, does not exist in the literature. Moreover, the classical method (One factor at a time) is used. This method requires a large number of tests with many other disadvantages (Bezerra *et al.*, 2008). It is increasingly replaced by experimental design methodology (EDM) as it enables to easily study the main and interaction effects of experimental parameters (factors), to model and to optimize processes with a very reduced number of trials (Allé *et al.*, 2020). Among the many designs that exist, two-level full factorial designs (FFD) are the most popular because of their simplicity and relatively low cost (Tarley *et al.*, 2009).

The overall objective of this study is to conduct a comparative study of the treatment of two metal cations (Cu^{2+} and Pb^{2+}) separately in synthetic solutions using the FFD. The specific objectives are to (i) quantify the main and interaction effects of parameters (the initial concentration of metal, the current intensity and the electrolysis time) on the removal efficiency and (ii) to determine the optimal conditions.

2. Methodology

2.1 Preparation of metal solutions

For EC tests, desired concentration of copper and lead were prepared from stock solutions highly concentrated in lead and copper (1000 mg L⁻¹ for each solution). Stock solutions of lead and copper were prepared from the copper nitrate ($\text{Cu}(\text{NO}_3)_2$) and lead nitrate ($\text{Pb}(\text{NO}_3)_2$) salt (Panreac, 99 % purity), respectively.

2.2 Electrochemical set-up and experimental procedure

The EC experiments are carried out in acrylic electrolytic cell with a capacity of 1.7-L in batch mode (**Figure 1**). Ten (10) electrodes made of iron, each of flat and rectangular shape (11.5 cm x 10 cm), are placed parallel to each other in monopolar configuration with a spacing between them of 1 cm. The electrical current is generated by an AL781D elc generator and measured using an ALDA DT-830D ammeter. The effluent is constantly stirred at 700rpm with magnetic stirrer (AGIMATIC –N type).

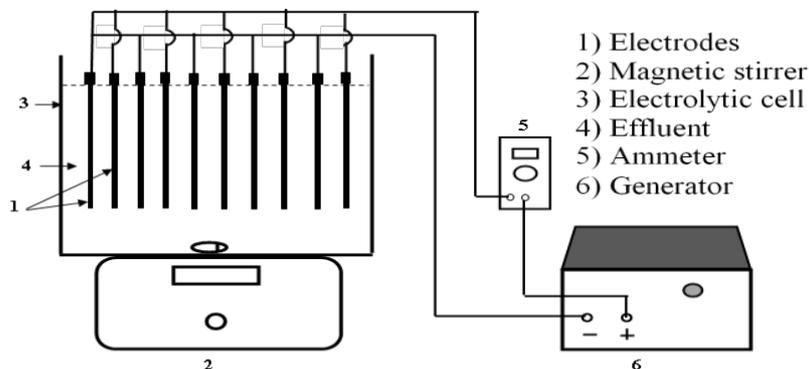


Figure 1. Schematic of the experimental setup

The experimental procedure consists of introducing a metal solution into the reactor. 0.34 g of sodium sulphate Na_2SO_4 (Prolabo, purity 99 %) was added in the solution to improve its conductivity. The treatment is initiated after defining operating conditions. At the end of each test, the mixture is transferred to 2-L graduated cylinder for natural settling for 24 hours. After settling, part of the supernatant is filtered under vacuum using a glass microfiber filter Whatman (circles diameter 47mm). The filtrate is collected to determine the metal residual concentration. The removal efficiency was calculated using **Eqn. 6**:

$$\text{Removal rate (\%)} = (C_0 - C_r)/C_0 \times 100 \quad \text{Eqn. 6}$$

Where C_0 and C_r are the initial and residual metal ion concentration.

The concentrations of metal ion (copper and lead) were determined by flame atomic absorption spectrometry (air-acetylene) using a VARIAN AA20 spectrometer.

2.3 Experimental design methodology (EDM)

In this study, three factors have been investigated using a two-level FFD (2^3): initial metal concentration (X_1), current intensity (X_2) and electrolysis time (X_3). This design was employed to firstly investigate the main and interaction effects of the factors on the removal of metals (Cu^{2+} and Pb^{2+}) and, subsequently, to optimize the treatment process. The selected factors and their levels are presented in **Table 1**.

Table 1. Factors and levels of FFD

Factors	Real variables	Coded Variables	Level -1	Level +1
Concentration of ion (mg L^{-1})	U_1	X_1	45	85
Current intensity (A)	U_2	X_2	0.3	1
Time (min)	U_3	X_3	20	60

The linear polynomial model, associated to a 2³ factorial design taking into account the second order interactions, is written according to [Eqn. 7](#):

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \quad \text{Eqn. 7}$$

Where Y represents the experimental responses (metal removal efficiency); b_0 represents the mean value of the observed responses; X_i represents the coded variable (-1 or 1); b_i represents the main effect of factor i on the response and b_{ij} represents the interaction effect between factors i and j.

A positive sign of factor's coefficients indicates a synergistic effect on the response, while a negative sign indicates an antagonistic effect ([Briton et al., 2018](#)). The b_i , b_{ij} , the standard deviation and the coefficient of determination values as well as the ANOVA analysis were obtained using the NEMROD-W software, version 9901. For the optimization, the Design Expert software (version 11) was used.

The conversion of the real variables U_i to the coded variables X_i , is given by the following formula ([Eqn. 8](#)) ([Kiari et al., 2022](#)):

$$X_i = \frac{U_i - U_i^0}{\Delta U_i} \quad \text{Eqn. 8}$$

Where X_i , U_i , $U_{i,0} = (U_{i,max} + U_{i,min})/2$ et $\Delta U_i = (U_{i,max} - U_{i,min})/2$ represent the coded value, the actual value, the actual value at the center of the experimental domain and the step size of factor i, respectively. $U_{i,max}$ and $U_{i,min}$ represent the maximum and minimum values of the real variable U_i , respectively.

3. Results and Discussion

3.1 Estimation of model coefficients and statistical analysis of data

The combination of the levels of the three factors makes it possible to build an experimental protocol of 8 assays. The performance of the 8 tests gives the results summarized in [Table 2](#)

Table 2. FFD test results

Test N°	Experimental matrix			Experimental plan			Lead	Copper
							Y ₁ (%)	Y ₂ (%)
1	-1	-1	-1	45	0.3	20	97,420	57.990
2	+1	-1	-1	85	0.3	20	90,490	41.730
3	-1	+1	-1	45	1	20	100.000	95.970
4	+1	+1	-1	85	1	20	95.000	89.000
5	-1	-1	+1	45	0.3	60	100.000	96.000
6	+1	-1	+1	85	0.3	60	97,970	90.720
7	-1	+1	+1	45	1	60	100.000	100.000
8	+1	+1	+1	85	1	60	100.000	100.000

An analysis of the table shows a strong dispersion of responses according to the experimental conditions, which means that the chosen variables are important ([Yobouet et al., 2016](#)). The removal rates of metals vary from 90.49 % to 100 % for Pb²⁺ and from 41.73 % to 100 for Cu²⁺. A high current intensity of 1A and a long duration of 60 minutes are required to completely remove the metal ions regardless of their initial concentration. To understand the effects of the factors on metals removal rates, it is necessary to calculate the coefficients of the mathematical models. The values of the coefficients and their statistical significance are given in [Table 3](#). The average values of the abatement

rates of the 8 assays are given by the coefficient b_0 . 97.61% of Pb^{2+} and 83.926% of Cu^{2+} have been removed. The coefficients of the main and interaction effects of the independent variables are statistically significant if the critical probability of each is less than 5% ($p\text{-value} < 5\%$) (Ouattara *et al.*, 2021). Thus, the removal rates are significantly influenced by the three main factors (initial concentration (X_1), current intensity (X_2) and electrolysis time (X_3)) for Pb^{2+} and by the factors X_2 and X_3 for Cu^{2+} . Regarding the interactions, all forms of interaction influence the removal efficiency of Cu^{2+} whereas for Pb^{2+} , it is only the X_2X_3 interaction that influences the process.

Table 3. Average, main and interaction coefficients of the different factors

Coefficients	Pb^{2+}	Signif. %	Cu^{2+}	Signif. %
b_0	97.610	< 0.01 ***	83.926	0.380 **
b_1	-1.745	0.456 **	-3.564	8.9
b_2	1.140	0.698 **	12.316	2.59 *
b_3	1.883	0.423 **	12.754	2.50 *
b_{12}	0.495	1.61 *	1.821	17.1
b_{13}	1.238	0.643 **	2.244	14.0
b_{23}	-0.633	1.26 *	-8.996	3.54 *

*** p-value << 0.1%; ** p-value << 1% ; * p-value << 5 %

The coefficients of determination of the models ($R^2 = 0.999$; $R_{adj}^2 = 0.996$ for Pb^{2+} removal rate and $R^2 = 1$; $R_{adj}^2 = 1$ for Cu^{2+}) are very close to 1, which indicates that the regression models correlates well with the experimental response (Bao *et al.*, 2022). The ANOVA analysis of the models gives the F-values and p-values of the postulated models. The calculated F-value of Pb^{2+} model is 276.17 with a p-value of 4.60%. For the Cu^{2+} , the F-value and p-value are 10735.9133 and 0.739%, respectively. These F values, above the critical value in the Fisher-Snedecor table ($F_c = 5.99$) with $p\text{-value} < 5\%$, show that the models are valid and robust (Briton *et al.*, 2018).

3.2 Comparative study of the contributions of the main factors and their interactions

The significance test of the coefficients makes it possible to know the factors that most influence the process studied. However, a classification of these influencing factors is important. The Pareto chart is the perfect tool for this approach as it allows to estimate the relative weight of each of the parameters in relation to the whole by calculating the relative contributions P_i (Eqn. 9) (Ano *et al.*, 2019):

$$P_i = \left(\frac{b_i^2}{\sum b_i^2} \right) \times 100 \quad \text{Eqn. 9}$$

Where b_i represents the main effect of factor i .

Figure 2 shows the Pareto chart of the main effects of the factors and their interactions on the removal rate of metal cations. For Pb^{2+} removal rates, the contributions are 30.24%, 12.9% and 35.21%, for initial concentration (X_1), current intensity (X_2) and electrolysis time (X_3), respectively. These same main factors, taken in the same order as above, contribute 3.05%, 36.43% and 39.07% for the Cu^{2+} removal rate. The three main factors X_1 , X_2 and X_3 have strong contributions to the removal rates. However, for a given factor, the contributions vary from one type of metal to another. The factor X_1

influences the removal of Pb^{2+} more than Cu^{2+} . On the other hand, the main factors X_2 and X_3 contribute more to the Cu^{2+} removal rate.

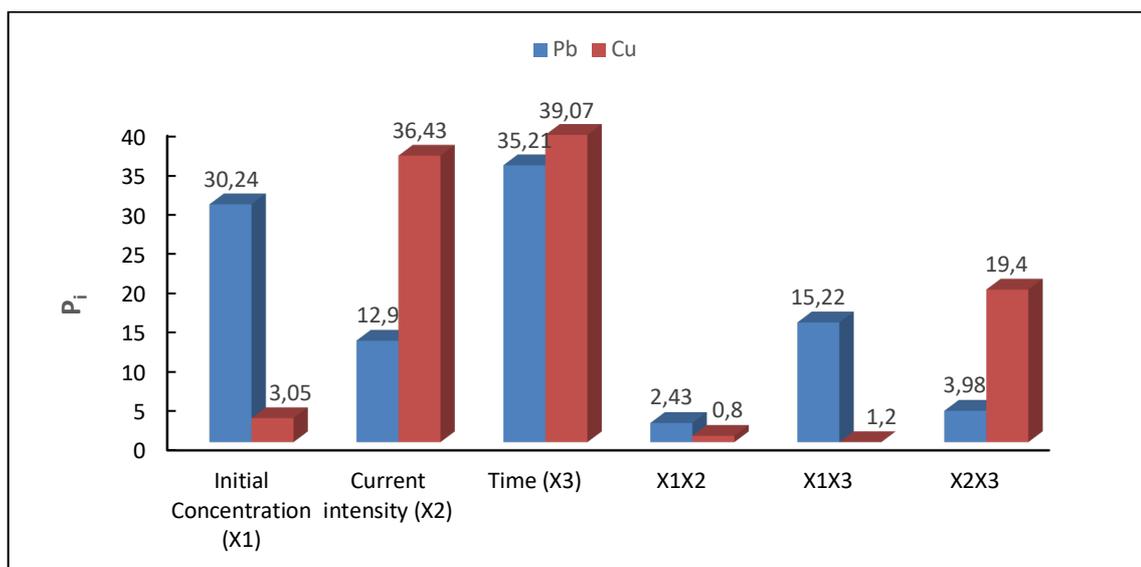


Figure 2. Contributions of the different factors and their interactions

Focusing on the significant interactions terms, it can be seen that the interactions X_1X_2 , X_1X_3 and X_2X_3 contribute 2.43%, 15.22% and 3.98%, respectively, for Pb^{2+} removal rate. On the other hand, for Cu^{2+} removal rate, the significant interaction (X_2X_3) contributes 19.4%. The significant effects have a cumulative contribution of 99.98% and 97.59% for Pb^{2+} and Cu^{2+} removal efficiencies, respectively. This means that a small contribution is therefore attributed to non-significant effects, around 0.02% and 4.41% for Pb^{2+} and Cu^{2+} , respectively.

3.3 Main effects of experimental parameters on metal ions removal

The negative coefficient of initial lead concentration ($b_1 = -1.7455$) means that the increase of initial lead concentration from 45 to 85 $mg L^{-1}$ contributes to decrease the removal rate of Pb^{2+} to 3.491% (2×1.7455). Working under different conditions, this result was also obtained by [Burboa-Charis et al \(2019\)](#). These authors observed a decrease in the removal efficiencies when Cd^{2+} and Zn^{2+} ions concentrations vary from 20 to 60 $mg L^{-1}$. According to these authors, this is possibly due to the insufficiency of coagulants to adsorb this increasing concentration of metal ions. For Cu^{2+} , the coefficient is insignificant to the removal efficiency. This result was also observed by [Gatsio et al. \(2015\)](#). According to this work, by increasing initial copper concentration from 10 to 90 $mg L^{-1}$, the percentage of removal is always maintained in the same order of magnitude (100%). The positive values of the coefficients ($b_2 = 1.140$ for Pb^{2+} and $b_2 = 12.316$ for Cu^{2+}) show that metals removal efficiency was improved by increasing the current intensity from 0.5 to 1 A. The removal rate of Pb^{2+} increases on average to 2.28% (2×1.14) and to 24.632% (2×12.316) for Cu^{2+} . Results obtained with the FFD are consistent with results of others researchers ([Akbal and Camci, 2010](#); [Gatsios et al., 2015](#)). By increasing current intensity, large amounts of hydroxide ions (OH^-), metal ions (Fe^{2+} and Fe^{3+}) as well as the dihydrogen bubbles are produced according to Faraday's law ([Al Aji et al., 2012](#); [Beiramzadeh et al., 2022](#)). This large quantity of hydroxide ions would directly precipitate a large number of Cu^{2+} and Pb^{2+} into $Cu(OH)_2$ and $Pb(OH)_2$ or could form more iron hydroxides ($Fe(OH)_2$ and $Fe(OH)_3$) which remove large quantity of Cu^{2+} and Pb^{2+} by adsorption, complexation and co-precipitation ([Drogui et al., 2011](#); [Kessentini et al., 2019](#); [Meunier et al., 2006](#)). Furthermore, the large number of gas bubbles (H_2)

with small sizes, by flotation effect, drags the metals trapped in the flocs to the free surface of the reactor (Burboa-Charis *et al.*, 2019).

The removal efficiency of pollutants is also dependent on the electrolysis time. It is the most important factor with a strong contribution and a positive effect ($b_3 = 1.883$ for Pb^{2+} and $b_3 = 12.754$ for Cu^{2+}). Increasing electrolysis time, from 20 to 60 minutes, contributes to improve the removal rate on average to 3.766% (2×1.883) for Pb^{2+} and 25.508% (2×12.754) for Cu^{2+} . These results are in agreement with the work of Bhagawan *et al.* (2015) and Aljaberi and Hawaas (2023). The result could be explained by the amount of metal hydroxides generated which increases with electrolysis time (Faraday's law) and so the adsorption sites of Cu^{2+} and Pb^{2+} . The time also defines the contact time between the adsorbents (metal hydroxides) and the cations Pb^{2+} and Cu^{2+} . The longer the contact time, the more metal cations are adsorbed on the surface of the metal hydroxides.

3.4 Effects of interactions of experimental parameters on metal ion removal

One of the advantages of the EDM over the traditional method is that it highlights the effects of interactions between factors. These interactions are likely to influence the response. With Pb^{2+} response, every 3 interactions X_1X_2 , X_1X_3 and X_2X_3 have non-negligible effects ($b_{12} = 0.495$, $b_{13} = 1.238$, $b_{23} = -0.633$). Although the coefficients of the interactions enable to quantify the associated effects, they do not explain how they influence the response. Interpretations of the interactions can be facilitated by the interaction graphs (Figure 3).

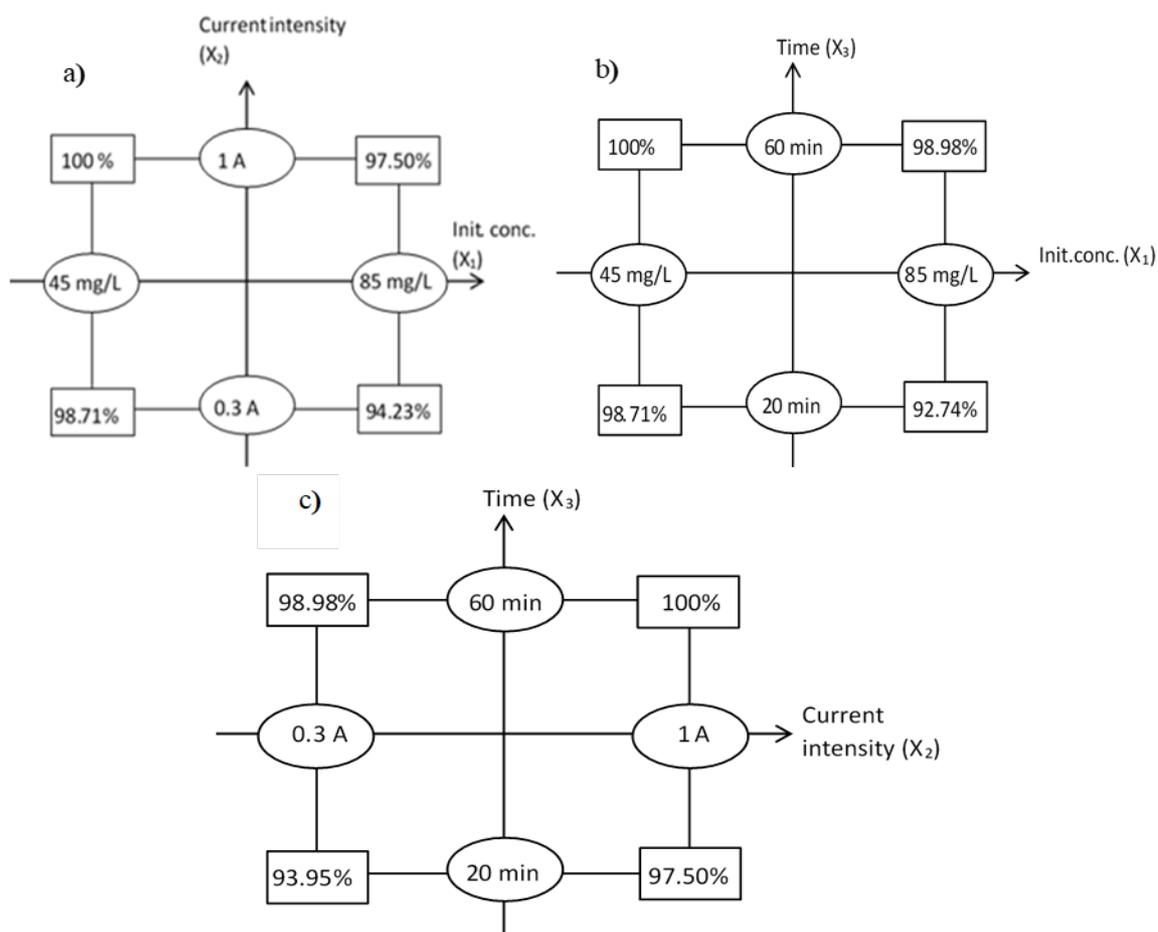


Figure 3. Interaction X_1X_2 (a), X_1X_3 (b) and X_2X_3 (c) on Pb^{2+} removal rates

According to Figure 3a, when the initial concentration (X_2) is set at the lowest level (45 mg L^{-1}), the current intensity, evolving from 0.3 to 1 A, leads to an increase in Pb^{2+} removal rate from 98.71 to

100 % (a gain of 1.29%). On the other hand, for water containing a higher content (85 mg L^{-1}), the yield increases from 94.23 to 97.5% (a gain of 3.27%). For X_1X_3 interaction (Figure 3b), the removal rate increases from 98.71 to 100% (a gain of 1.29%) when the treatment time evolves from 20 to 60 min for an initial concentration set at 45 mg L^{-1} . For the high initial concentration (85 mg L^{-1}), the removal rate increases from 92.74 to 98.98% (a gain of 6.24%). The last influential interaction is X_2X_3 (Figure 3c). For a current fixed at 0.3 A, the removal rate evolves from 93.95 to 98.98% (a gain of 6.24%) and from 97.5 to 100% (a gain of 2.5%) when current is fixed at 1 A with a treatment duration increasing from 20 to 60 min. The analysis of these graphs have shown that the effect of each factor on Pb^{2+} removal efficiency is a function of the low and high levels of the other factors.

With Cu^{2+} , the significant interaction (X_2X_3) is shown in Figure 4. According to the figure, when the current intensity (X_2) is set at the lowest level (0.3 A), increasing the duration (from 20 to 30 min) leads to a strong increase in the Cu^{2+} removal rate from 49.86 to 93.36% (a gain of 43.5%). However, for a current set at the high level (1 A), the rate increased from 92.48 to 100% (a gain of 7.52%). The effect of the treatment time is not constant but depends on the level of current intensity.

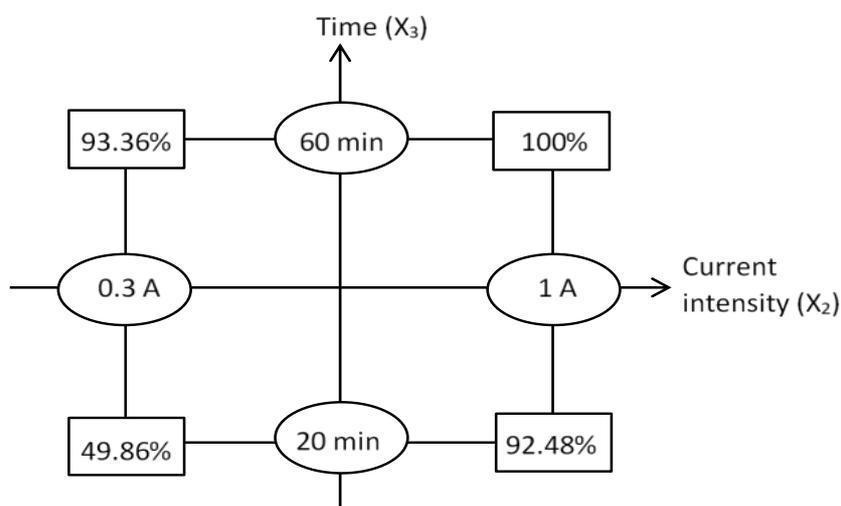


Figure 4. X_2X_3 interaction on Cu^{2+} removal rates

3.5 Process optimization

The Design Expert software (version 11) was used to obtain the optimal conditions. In order to minimize the energy consumed and to treat waste water heavily loaded with heavy metals, the criteria selected for the optimization condition for metals removal are as follows: current intensity and electrolysis time have been minimized with lesser importance (3/5 weighting factor); ii) the initial concentration was maximized with lesser importance (3/5 weighting factor) and iii) metal ions removal efficiency was maximized with high importance (5/5 weighting factor). The proposed solutions and desirabilities are presented in Table 4.

Table 4. Conditions obtained with the Design expert software

Solutions	U_1 (mg L^{-1})	U_2 (A)	U_3 (min)	Removal rate (%)	Desirability (%)
Pb^{2+}	79.9	0.42	42	97.61	0.75
Cu^{2+}	74.85	0.401	54	84%	0.724

The responses estimated by the models (Y_{pred}) were confirmed by additional experiments performed under the optimal conditions. The experimental values (Y_{exp}) obtained ($\text{Pb}^{2+} = 95.15 \%$ and

Cu²⁺ = 80.22%) are compared to the predicted values using the coefficients of variation (CV) (Eqn. 10):

$$CV = \frac{Y_{\text{exp}} - Y_{\text{cal}}}{Y_{\text{exp}}} \times 100 \quad \text{Eqn. 10}$$

These low CV values (2.58% and 4.7% for Pb²⁺ and Cu²⁺, respectively), below 10%, reflect the reliability and reproducibility of the experiment (Ntakiyiruta *et al.*, 2022).

Conclusion

From this study, it was found that electrocoagulation is a very effective technique for the removal of heavy metals (Pb²⁺, Cu²⁺) from wastewater. With a reduced number of experiments using the full factorial design, the results showed that the removal rates of copper and lead are affected by factors such as initial concentration of metal ion, current intensity and electrolysis time. An increase in current intensity and electrolysis time results in higher treatment efficiency of metal ions. In contrast, the initial concentration has a negative effect on the removal of Pb²⁺ and a negligible effect on Cu²⁺. Maximum removal rates of 95.15% for Pb²⁺ and 80.22% for Cu²⁺ have been obtained for minimum current, minimum duration and high metal ion loading.

Conflict of interest: All the authors declare that there is no conflict of interest.

Acknowledgement: The authors would like to thank anonymous reviewers who provided helpful comments. This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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(2023) ; <http://www.jmaterenvirosci.com>