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Design optimization of extraction procedure for mercury (II) using Chelex 100 resin

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ABSTRACT

In this paper, an extraction procedure carried out in a closed reactor, for mercury (II) present in an aqueous media using Chelex 100 resin, was applied to develop a mathematical model and optimize process parameters for Hg(II) removal. The optimization process was carried out using 2^4 factorial designs. The individual and combined effect of four process parameters, i.e. initial pH of the solution (1.6 and 7.0), initial Hg(II) concentration (10^{-5} and 10^{-3} mol/L), mass of sodium chloride (0.10 and 73 mg), and resin mass on Hg adsorption (0.05 and 0.2 g), were studied. Analysis of variance showed the relative importance of process parameters in the removal procedure. The optimal conditions to remove Hg from the aqueous solution, at the constant temperature of 20°C and stirring speed of 1,000 rpm were found to be: contact time = 3 h, pH 7, initial Hg(II) concentration = 1 mmol/L, ionic strength = 10^{-4} g, and resin dosage = 0.2 g. Under these conditions, high removal efficiency (98.0%) was achieved. Student's *t*-test on the results of the 2^4 factorial designs, with 16 runs for mercury extraction, showed that the factor "ionic strength value" in the studied levels is statistically significant. The authors plan additional tests using the extraction on column.

Keywords: Mercury (II); Chelex 100; Optimization; Extraction; Factorial design

1. Introduction

Mercury is a carcinogenic heavy metal which poses a potential threat to human health even at very low concentrations. It has been well-documented that mercury may cause brain damage, dysfunction of liver, kidney, gastrointestinal tract, and central nervous system, as well as induce cellular toxicity when bound to intracellular sulfhydryl groups. Inorganic mercury is the most prevalent form of mercury in aquatic ecosystems [1]. On the other hand, mercury recovery is important from the economical point of view because it has a wide range of applications, such as in dental amalgams, anti-fouling paints, electrodes for some types of electrolysis, batteries, fluorescent lamps, catalysts, etc. [2]. Several methods can be applied to remove mercury from aqueous solutions, i.e. ion exchange [3], carbon adsorption [4], sequential injection extraction [5], liquid–liquid extraction [2,6], solid phase extraction [1,7–25], and membrane process [26,27].

Chelating resins have seen considerable application in speciation studies, particularly the commercially available Chelex 100 resin, which is a polystyrene

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divinylbenzene copolymer incorporating iminodiacetate chelating groups. The iminodiacetate groups coordinate metals by means of oxygen and nitrogen bonds and the resins have a particularly strong affinity for trace metals. It was proposed firstly to use Chelex 100 for the preconcentration of total trace metals from seawater. After, she is used to differentiate labile from non-labile fractions of trace metals. Chelex 100 retains free metal ions and loosely bound trace metals [28].

Chelex 100 finds application in many fields, it was effective in binding several metal ions (Cr^{3+}, Ni^{2+}, Zn^{2+}, Tl^{3+}, La^{3+}, and Al^{3+}) [29–31].

Application of multivariate techniques in the optimization of procedures has been encouraged, as these techniques are faster, more economical, and effective, and allow more than one variable to be optimized simultaneously. In chemistry, the factorial design has been widely used in several situations [32].

The objective of our work was to recover mercury (II) using resin Chelex 100 as extractant agent. The influence of operating variables, such as the initial pH of solution, initial Hg(II) concentration, the ionic strength (NaCl mass) value, and the amount of resin (Chelex 100), on the extraction yield was studied. Nowadays, factorial designs have proven their usefulness, and are widely used in statistical planning of experiments to obtain empirical linear models, relating process response to process factors [21]. The effects of the various parameters will be studied using a 2^4 full factorial design.

2. Materials and methods

2.1. Chemicals

Chelex 100 (Bio-Rad Laboratories, CA, USA) is a chelating resin which uses ion exchange to bind transition metal ions. The resin was composed of polystyrene divinylbenzene copolymers containing paired iminodiacetate ions, which act as chelators for polyvalent metal ions [33].

A stock solution of Hg(II) was prepared (100 mL) by dissolving 27.14 mg of analytical grade mercury (II) chloride ($HgCl_2$) obtained from Sigma (St. Louis, MO, USA) in distilled water. The stock solution was further diluted to obtain desired concentration ranging from 10^{-5} to 10^{-3} mol/L.

The 1-(2-pyridylazo) 2-naphthol (PAN) (St. Louis, MO, USA) (≥97.0%) dissolved in a water/ethanol (75/25, v/v) mixture was used for spectrophotometric determination of mercury.

2.2. Batch experiments

Batch experiments were carried out under the following conditions: 0.1 g of Chelex 100, 5 mL of mercury solution, and an agitation speed of 1,000 rpm. The pH, initial Hg(II) concentration, *ionic strength value,* and amount of resin employed are shown in Table 1. Samples were collected after 180 min. Aliquots for analysis were filtered, and 100 μL of the residual mercury concentration was measured by means of the SPECORD 210 plus spectrophotometer at 590 nm using 200 μL PAN at 1 mmol/L and a 700 μL of buffer at pH 13 as reagents. The extraction yield of Hg(II) was calculated using the following equation:

$$Y(\%) = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where C_0 and C_t are the initial concentration and concentration at time "t" of mercury (II) in mol/L, respectively.

2.3. Factorial design of experiments (DOE)

The study results of the extraction of mercury (II) by Chelex 100 resin, at the optimal time of 3 h according to four variables, namely the pH, initial concentration of mercury (II) (mol/L), effect of ionic strength (g), and amount of resin (g), are expressed in terms of the extraction yield by the response Y. These results are subjected to an empirical smoothing. In this method, the experimental values can be used to determine the constants of the polynomial model (Eq. (2)), which are adjusted to the variations of the studied properties [32,34].

In our investigations, a series of 16 attempts was made according to a 2^4 factorial experimental design by varying the pH value (X_1), initial concentration of mercury (X_2), ionic strength (X_3), and amount of Chelex 100 resin (X_4). Two variation levels (−1,+1) for each parameter were considered, as summarized in Table 1.

$$\begin{aligned} Y(\%) &= a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_4X_4 + a_{12}X_1X_2 \\ &+ a_{13}X_1X_3 + a_{14}X_1X_4 + a_{23}X_2X_3 + a_{24}X_2X_4 + a_{34}X_3X_4 \\ &+ a_{123}X_1X_2X_3 + a_{124}X_1X_2X_4 + a_{134}X_1X_3X_4 + a_{234}X_2X_3X_4 \\ &+ a_{1234}X_1X_2X_3X_4 \end{aligned} \quad (2)$$

where Xj (j = 1–4); reduced variable which takes two values: −1 (low level) and +1 (high level); low level = 2 (low value − mean)/range; high level = 2 (high value − mean)/range; mean = (high value + low

Table 1
Factor levels used in the 2^4 factorial experiment designs

Parameter level	Reduced value	X_1	X_2 (M)	X_3 (g)	X_4 (g)
Minimal	−1	1.6	10^{-5}	10^{-4}	0.05
Level 0	0	4.4	5.05×10^{-4}	3.7×10^{-2}	0.125
Maximal	+1	7.0	10^{-3}	7.3×10^{-2}	0.2

value)/ 2; and range = (high value − low value). X_1, X_2, X_3, and X_4 are the reduced variables of pH, initial concentration of mercury (II) (mol/L), effect of ionic strength (g), and amount of resin (g), respectively.

2.4. Validation model

In the case of a design pattern, the model is validated using an appropriate analysis of variance (ANOVA). The model is considered adequate if the variance due to regression is significantly different from the total variance.

Statistica v 9.0 software was used for regression and graphical analyses of the data obtained. The optimum of the studied parameters (pH, initial concentration of Hg^{2+}, value of ionic strength, and amount of Chelex 100 resin) was obtained by analyzing the response contour plots.

3. Results and discussion

The most important parameters which affect the extraction efficiency are the pH, initial concentration of mercury, ion strength, and amount of resin. In order to study the combined effect of these factors, some experiments were performed for different combinations of the physical parameters, using statistically designed experiments. The pH range used was between 1.6 and 7.0. The initial concentration of Hg^{2+} was between 10^{-5} and 10^{-3} mol/L. The ionic strength varied between 0.1 and 73 mg, and the amount of resin between 0.05 and 0.2 g.

The results of the extraction process of mercury are expressed in terms of the extraction yield, which is regarded as the response function in the investigated process. These results are summarized in Table 2.

Preliminary observations show that the extraction yield of Hg(II) is in good agreement with the experimental parameters, and can reach values ranging

Table 2
Experimental design and extraction capacity (%) of Chelex 100

	Factor levels				Extraction capacity (%)	
Experiment No.	X_1	X_2	X_3	X_4	Experimental	Predicted
1	−1	−1	−1	−1	97.74	97.96
2	+1	−1	−1	−1	79.22	80.29
3	−1	+1	−1	−1	98.85	97.35
4	+1	+1	−1	−1	97.94	96.26
5	−1	−1	+1	−1	97.98	95.98
6	+1	−1	+1	−1	79.22	78.07
7	−1	+1	+1	−1	60.29	61.01
8	+1	+1	+1	−1	97.14	97.68
9	−1	−1	−1	+1	97.57	95.74
10	+1	−1	−1	+1	77.61	77.27
11	−1	+1	−1	+1	86.4	87.15
12	+1	+1	−1	+1	97.94	99.22
13	−1	−1	+1	+1	97.80	98.19
14	+1	−1	+1	+1	79.22	81.10
15	−1	+1	+1	+1	72.68	71.21
16	+1	+1	+1	+1	91.66	92.72
17	0	0	0	0	81.74	–
18	0	0	0	0	80.23	–
19	0	0	0	0	79.76	–

Note: Three additional tests at the central point (0, 0, 0) for the calculation of the Student’s *t*-test and Fisher’s tests, using the normal rule of variance.

Table 3
Model coefficients and their corresponding effects upon yield extraction of Hg(II)

Variable	Model		
	Coefficient	Value	Expected effect on the yield extraction
X_0	a_0	88.08	High average extracting capacity of the chelex 100
X_1	a_1	−0.585	Detrimental individual effect of X_1
X_2	a_2	−0.216	Detrimental individual effect of X_2
X_3	a_3	−3.58	Detrimental individual effect of X_3
X_4	a_4	−0.469	Detrimental individual effect of X_4
X_1X_2	a_{12}	8.902	Favorable binary interaction X_1 & X_2
X_1X_3	a_{13}	2.906	Favorable binary interaction X_1 & X_3
X_1X_4	a_{14}	−0.42	Weak detrimental binary interaction of X_1 & X_4
X_2X_3	a_{23}	−3.84	Weak detrimental binary interaction of X_2 & X_3
X_2X_4	a_{24}	−0.223	Weak detrimental binary interaction of X_2 & X_4
X_3X_4	a_{34}	1.31	Favorable binary interaction X_3 & X_4
$X_1X_2X_3$	a_{123}	2.753	Favorable ternary interaction of X_1, X_2, & X_3
$X_1X_2X_4$	a_{124}	−0.26	Weak ternary detrimental interaction of X_1, X_2, & X_4
$X_1X_3X_4$	a_{134}	−1.803	Weak ternary detrimental interaction of X_1, X_3, & X_4
$X_2X_3X_4$	a_{234}	1.11	Favorable ternary interaction of X_2, X_3, & X_4
$X_1X_2X_3X_4$	a_{1234}	−2.00	Weak fourthly detrimental interaction of X_1, X_2, X_3, & X_4

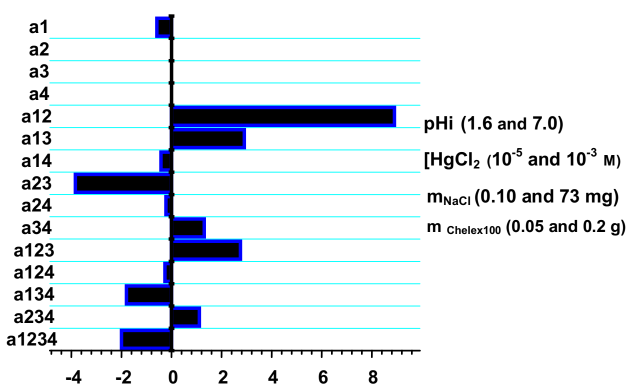

Fig. 1. Graphical study of the coefficients values on the extraction of Hg^{2+} to describe the individual and interaction effects of parameters: a_i [pHi (1.6 and 7.0), $[HgCl_2]$ (10^{-5} and 10^{-3} mol/L), m_{NaCl} (0.10 and 73 mg), and $m_{Chelex100}$ (0.05 and 0.2 g)], a_{ij}, a_{ijk}, and a_{ijkl} are the constants of the polynomial model (Eq. (2)).

between 60.29 and 98.85%, under certain operating conditions.

This correlation allows building the response surface. From Table 2, it already appears that the highest yield extraction value (≈99%) was obtained for minimal pH value, minimal ionic strength and amount of resin, and maximal concentration of mercury (II).

3.1. Analysis of the effects through factorial DOE

Table 3 and Fig. 1 summarizes the coefficient values of the model, supposed to describe the individual effects of parameters, along with their possible interactions.

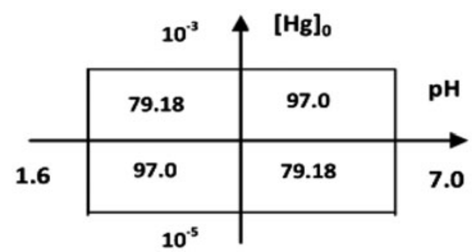

Fig. 2. Factorial interaction between pH and initial concentration of Hg(II) (X_1X_2).

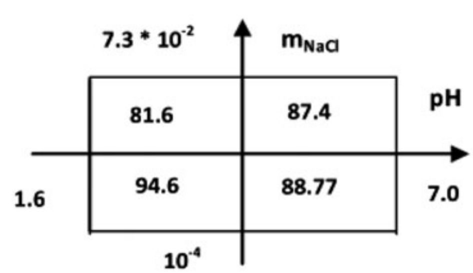

Fig. 3. Factorial interaction between pH and ionic strength (X_1X_3).

The individual effects and interactions of the parameters are discussed on the basis of the sign and

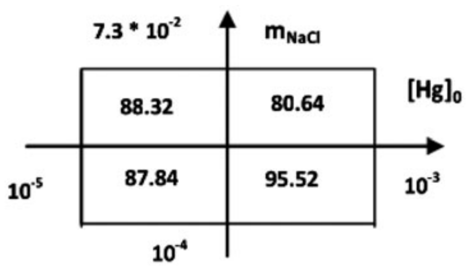

Fig. 4. Factorial interaction between initial concentration of Hg(II) and ionic strength (X_2X_3).

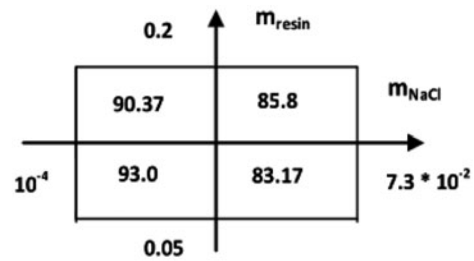

Fig. 5. Factorial interaction between ionic strength and the amount of resin (X_3X_4).

the absolute value of each coefficient. These coefficient features will define the strength of the corresponding effect involved and the way it acts upon the extraction yield (favorable or detrimental).

The first observations from Table 3 already allow making the following statements:

(1) High extracting capacity of Chelex 100 ought to be obtained within the fixed parameter ranges, thereby justifying the suitable choice of the limits.

(2) The individual effect of the initial pH of aqueous phase, initial concentration of mercury, effect of NaCl concentration, and amount of resin have net negative effects on mercury removal.

(3) For the removal efficiency of mercury, the whole binary interaction effect of the variables is found highly significant except for the interaction between the pH and the amount of resin, and that between the initial concentration of mercury with NaCl concentration and the amount of resin Chelex 100.

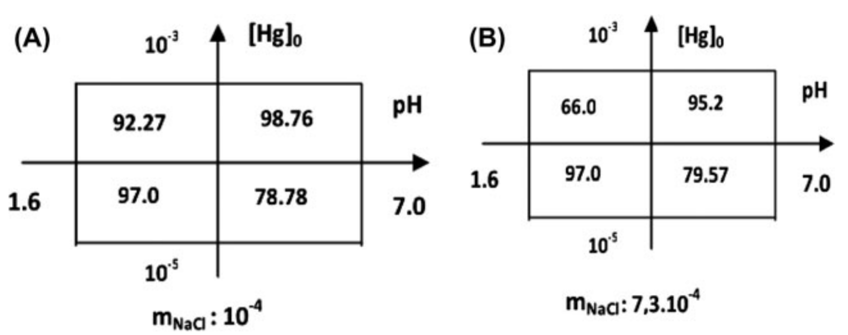

Fig. 6. Factorial interaction between pH, initial concentration of Hg(II), and ionic strength ($X_1X_2X_3$) (A) m_{NaCl}: 10^{-4} g; (B) m_{NaCl}: 7.3×10^{-4} g.

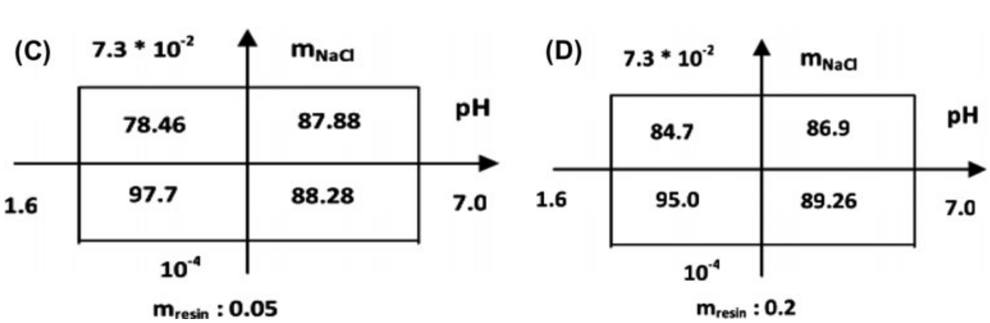

Fig. 7. Factorial interaction between pH, ionic strength, and the amount of resin ($X_1X_3X_4$) (C) m_{resin}: 0.05 g; (D) m_{resin}: 0.2 g.

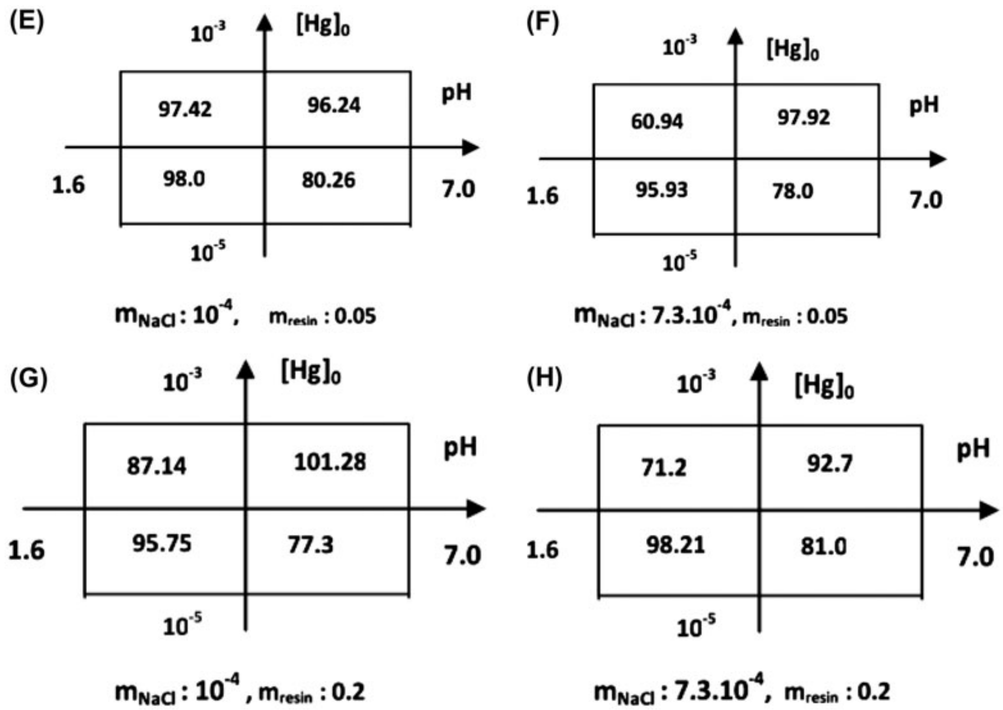

Fig. 8. Factorial interaction between pH, initial concentration of Hg(II), ionic strength and the amount of resin ($X_1X_2X_3X_4$) (E) m_{NaCl}: 10^{-4} g, m_{resin}: 0.05 g; (F) m_{NaCl}: 7.3×10^{-4} g, m_{resin}: 0.05 g; (G) m_{NaCl}: 10^{-4} g, m_{resin}: 0.2 g; (H) m_{NaCl}: 7.3×10^{-4} g, m_{resin}: 0.2 g.

(4) For the ternary interaction, all interactions of variables are considered as very significant factors, except for the interaction: pH—initial concentration of mercury—amount of resin, as well as the interaction: pH—effect of ionic strength—amount of resin.
(5) No synergy must be involved, for the four parameters.

3.2. *The meaning of factorial interaction in the statistics and the application to engineering study*

To explain the relationship between factorial interaction and response, the pH and initial concentration of Hg(II) are regarded as two selected variables. The pH and initial concentration of Hg(II) increase simultaneously. Fig. 2 shows that the factorial interaction is significant, and the responses display a similar tendency as the first variable increases (that is the pH, or the variable along the transverse axis) for the initial concentrations of Hg^{2+}, at 10^{-5} and 10^{-3} mol/L, respectively, as shown in Fig. 2.

Fig. 3 shows the factorial interaction is very significant, and the responses display opposite tendency with the increased variable 2 for the variable (+1) at low level (−1). When X_1 is at the high level (+1), the influence of X_1 over X_3 is insignificant.

When X_2 and X_3 are at the low level (Fig. 4), the effect of X_2 with X_3 is under the limit of interpretation of the measurement error. When X_2 is at the high level, factor X_3 gives rise to decrease the extraction of mercury significatively. However, the slope of X_3 when X_4 is, respectively, at the low and high level (Fig. 5). It means that there is significant slightly between X_3 and X_4.

The pH and initial concentration of Hg(II) increase simultaneously. Fig. 6 shows the factorial interaction is significant. The slope of X_1 and X_2 when X_3 is at the low level is greater than when X_3 is at the high level.

Table 4
Optimum values of the process parameter

Parameter	Optimum value
pH	7
Initial concentration of Hg^{2+} (mol/L)	10^{-3}
Ionic strength (g)	10^{-4}
Amount of resin (g)	0.2

Table 5
Model adequacy tests and variance analysis

Feature	Symbol/equation	Value
Parameter number	P	4
Level number	L	2
Number of experimental attempts	N	16
Number of tests at (0,0,0) point	n	3
Model variance	v	2
Average yield at (0,0,0)	$Y_0 = \sum Y_{0i}/3$	80.60
Random variance	$S^2 = \sum (Y_{0i} - Y_0)^2/v$	1.07
Square root of variance	S	1.034
Risk factor (chosen arbitrary)	α	0.05 (95%)[a]
Student's t-test factor	t_v	4.3[b]
Average error on the coefficient value (trust range)	$\Delta a_i = \pm t_{v,\alpha/2} S/N^{0.5}$	1.111
Number of remaining coefficients	R	9
Model response at (0,0,0)	a_0 (Z_{000})	88.08
Discrepancy on average yield	$d = Z_0 - Z\ (0,0,0) = Z_0 - a_0$	7.50
Error on average yield discrepancy	$\Delta d = \pm t_{v,\alpha/2} S\ (1/N + 1/n)^{0.5}$ with $N = 16$ & $n = 3$	2.80
Average yield for the sixteen attempts	$Z_m = \sum Z_i/16$	88.08
Residual variance	$S_r{}^2 = \sum (Z_i - Z_m)^2/(N - R)$	305.51
Degrees of freedom	v_1	2
Residual degrees of freedom	v_2	8
Observed Fisher's test	$F_{obs} = S_r{}^2/S^2$	285.4
Fisher–Snedecor law	F_{obs}, v_1, v_2	$F(0.95, 2, 8) = 19.37$[c]

[a] $\alpha = 5\%$ was arbitrary chosen. In this case, one regarded that a 95% confidence may be satisfactory.
[b] Student tables with two degrees of freedom at a 95% confidence, t_{crit} (2; 0.05).
[c] See Fisher–Snedecor tables, $F_{crit} = 19.37$.

At low level of X_4, X_3 is extremely significant on the extraction of Hg^{2+} at low level and the performance improves at high level of X_1, respectively. At high level of X_4, the interaction X_1X_3 has a significant slightly effect (Fig. 7).

Fig. 8 shows the relationship between the actual value and the predicted ones, for Hg(II) removal from simulated solutions, using resin. The actual data are the original measurements of mercury concentration in the solution that was determined experimentally using Eq. (1). On the other hand, the predicted values were generated using Eq. (2). A fairly moderate value of the correlation coefficient R^2 (0.984) was obtained between experimental and predicted responses. This could be due to the wide range of coverage of process variables in a limited number of experiments and/or to the contribution of insignificant terms, as shown in Table 3. Therefore, the optimum condition for removal of mercury (II) is given in Table 4. Under these conditions, the values of pH, initial concentration of Hg^{2+}, ionic strength, and amount of resin, for the recovery of mercury in the solution, using Chelex 100, were 7.0×10^{-3} mol/L, 10^{-4} g, and 0.2 g, respectively.

3.3. Analysis of variance (ANOVA)

For the sake of reproducibility, one must check whether this model accurately describes the process investigated by determining which coefficients could be neglected, through Student's t-test and Fisher's test. The model's adequacy strongly depends on the accuracy of the experiment. In the current experiment, the main errors arise from volume and weight measurements. For this purpose, three additional attempts at the central point (0, 0, 0) are required for estimating the average error in the value of each coefficient, on the basis of the random variance [35–37]. The calculations made are summarized in Table 5.

Thus, with a 95% confidence (i.e. $\alpha = 0.05$), and for the two variances (i.e. for three attempts at central point), one assessed the value of $t_{v,1-\alpha/2}$ as being equal to 1.112.

Therefore, at level $(1 - \alpha)$, the confidence range for all the coefficients estimated, using 16 runs ($N = 16$), will be $\Delta a_i = \pm 1.11$ at 95% confidence level. From the Student's t-test, it results that $|a_i| < |\Delta a_i|$ for a_1, a_2, a_4, a_{14}, a_{24}, a_{124}, and a_{234}. Therefore, these coefficients must be removed from the mathematical model because they do not have a significant effect upon the response function, as they are shaded off by their average error. Consequently, the final form of the polynomial model that describes the mercury extraction by Chelex 100 resin is given by the following equation (Eq. (3)):

$$Y(\%) = 88.08 - 3.60X_3 + 8.902X_1X_2 + 2.906X_1X_3 - 3.84X_2X_3 + 1.31X_3X_4 + 2.753X_1X_2X_3 - 1.803X_1X_3X_4 - 2.0\,X_1X_2X_3X_4 \quad (3)$$

This model was supposed to accurately fit the extraction process of mercury investigated herein. Thus, in the vicinity of the expected optimal parameter values, it appears that only the NaCl concentration and interactions between the pH and the initial concentration of analyte parameters have significant effects on mercury removal. Furthermore, adequacy tests were applied to check whether the model utilized is valid within the parameter ranges investigated.

For this purpose, a first method for adequacy calculations [34] showed that the observed value (285.46) of Fisher's test is higher than the critical one (19.37), indicating that the model can be applied within the entire range investigated.

4. Conclusion

The factorial experiment design method is undoubtedly a good technique for studying the influence of major process parameters on response factors, by significantly reducing the number of experiments and henceforth, saving time, energy, and money.

In order to achieve the best conditions for Hg(II) extraction in an aqueous solution by Chelex 100 resin, a full 2^4 factorial design was employed for screening the factors that would influence the overall optimization procedure of batch sorption. This optimization showed that only the NaCl concentration and interactions between pH and initial concentration of analyte parameters have significant effects on the extraction of Hg(II). Other tests using the extraction optimization on column are being achieved and will be communicated in due time.

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