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Modelling and Optimizing the Solvent Extraction of Cadmium from Phosphoric Acid using Experimental Design

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The removal of cadmium from phosphoric acid was carried out using the solvent extraction process, taking into account factors such as pH, concentration of the extracting agent [EA], organic phase/aqueous phase (O/A) ratio, stirring time and stirring rate. In order to study the effect of these involved factors and their interactions on the extraction percentage of cadmium, a composite central design (CCD) of 24 experiments was adopted. An empirical model was developed and validated by applying ANOVA analysis. The graphical representation of this model in the variable space allowed to determine the optimal conditions of these factors. The extraction of cadmium from phosphoric acid reached a percentage of the order of 98%, under the following conditions: pH = 3, [EA] = 10^{-2} M, O/A = 1.1, stirring time 90 min, stirring rate 800 rpm.

Keywords: Solvent extraction, Cadmium, Experimental design, Central composite design, Response surface methodology.

INTRODUCTION

The removal of cadmium is one of the major problems faced by manufacturers of phosphoric acid and/or fertilizers. Several methods of purifying phosphoric acid from cadmium are reported, including membrane technologies such as electrodialysis (ED), reverse osmosis and nanofiltration [1], by adsorption on activated carbon [2] and by solvent extraction [3-7].

The solvent extraction process used several types of the commercial extracting agents, notably for the extraction of cadmium from phosphoric acid, where di-2-ethyl hexyl phosphoric acid (D2EHPA) remains the best cadmium extractant from phosphoric acid as compared to other tributylphosphate (TBP), trioctyl phosphine oxide (TOPO), triphenyl phosphine oxide (TPPO) or diphenylamine used individually or in a mixture [6]. The extractant (D2HEPA) was also applied in the diluted form in kerosene as reported by Mellah & Benachour [7].

Recently, our group [8] reported the solvent extraction of cadmium from phosphoric acid using $C_{11}H_{18}N_2O$ (hexahydroquinazolin-2-ones) as extracting agent (EA) and benzene as solvent. Different factors that affect the efficiency of cadmium extraction from phosphoric acid were also taken into the consideration [7]. In order to achieve high yields from this process, various factors must be studied and optimized. Hence the need to carry out conventional multifactorial experiments, in a classical way, by varying one factor and keeping the other conditions fixed. This method does not allow studying the effect of the interactions between the factors involved on the one hand, and on the other hand requires a large number of experiments, which generates a higher cost and a lot of time required for the process [9].

In order to optimize all the influencing parameters and therefore to eliminate these limitations of this classical method, a statistical design of experiment such as the response surface method (RSM) must be carried out. RSM represents a set of mathematical and statistical techniques useful for developing, improving and optimizing the process and can be used to assess the relative importance of several influencing factors, even in the presence of complex interactions. The main objective of RSM is to determine the optimal operational conditions for the system or to determine an area that meets the operational specifications [10,11]. The application of this statistical experimental design technique in the development of solvent extraction processes can lead to improved yields, while taking into account the effect of each factor individually and also the effect of their interactions, as well as a reduction in development time and overall costs.

The objective of this study is to model and optimize the removal of cadmium from phosphoric acid by the solvent extraction operation, while searching for a possible optimum of the response surface area between the cadmium extraction percentage and the five factors involved, namely, pH, concentration of the extracting agent [EA], organic phase/aqueous phase (O/A) ratio, duration stirring and stirring rate. This study is modelled for all the experiments using a central composite design. Statistical calculations are performed using the software JMP [12,13].

EXPERIMENTAL

For all the experiments, the aqueous phase consisted of 2.5 mol phosphoric acid solution containing 10^{-3} mol/L of cadmium. The organic phase was prepared by dissolving three different concentration of synthesized hexahydroquinazolin-2-ones ($C_{11}H_{18}N_2O$, 0.5 × 10^{-2} M, 10^{-2} M, 1.5 × 10^{-2} M) as extracting agent in benzene. All reagents were of analytical grade and used without further purification. The pH of the solutions was adjusted to the desired value by adding a small amount of NaOH. The pH was measured using a pH meter (model JENWAY 3520 pH Meter).

Extraction procedure: The various extraction experiments were carried out in a batch system (comprising 5 mL of the aqueous phase and 5 mL of the organic phase), magnetically stirred (model VWR incubating Mini Shaker) at 20 ºC (room temperature).

After stirring the aqueous and organic phases, this mixture underwent gravity settling and then separated the two phases. Cadmium concentrations were determined in the aqueous phase by inductively coupled plasma spectrometer (ICP-OES Perkin-Elmer Optima 8000). Cadmium concentrations in the organic phase were calculated from the difference between cadmium concentrations in the aqueous phase before and after extraction.

Statistical analysis: The extraction of cadmium from phosphoric acid was modelled and optimized using the response surface methodology (RSM). The composite central design was chosen to study the relationship between the percentage extraction of cadmium and the various factors involved.

Central composite design: Among the several existing experimental designs in experimental research methodology, preference was given to central composite designs which have optimal qualities in predicting the calculated response at any point in the domain (Fig. 1).

Choice of factors and experimental field: Based on the various works carried out [3-5,14], The factors that influence the response extraction of cadmium from phosphoric acid are pH, concentration of the extracting agent [EA], ratio (organic phase/aqueous phase (O/A)), duration of stirring and rate of stirring. The experimental field chosen to study the influence of these five factors are presented in Table-1.

The variables are coded according to the following equation:

$$X_i = \frac{x_i - x_i^o}{\Delta x_i} \quad (1)$$

with: X_i: Coded variable, x_i: Natural variables, x_i^o: Value of the i^{th} variable of the center of interest, Δx_i: Step of the variation of the real variable.

Mathematical model: The equation of the theoretical model is therefore written:

$$Y = \beta_0 + \sum_{j=1}^{5}\beta_j X_j + \sum_{j=1}^{5}\sum_{j'=1\, j'\neq j}^{5}\beta_{jj'}X_j X_{j'} + \sum_{j=1}^{3}\beta_{jj}X_j^2 \quad (2)$$

where: Y: Theoretical response function, X_j: Coded variables of the system, β_0: constant term of the model, β_j: coefficients of the model of variable X_j, β_{jj}: coefficients of the model of square variable X_j^2, $\beta_{jj'}$: coefficients of the interaction model between X_j and $X_{j'}$,

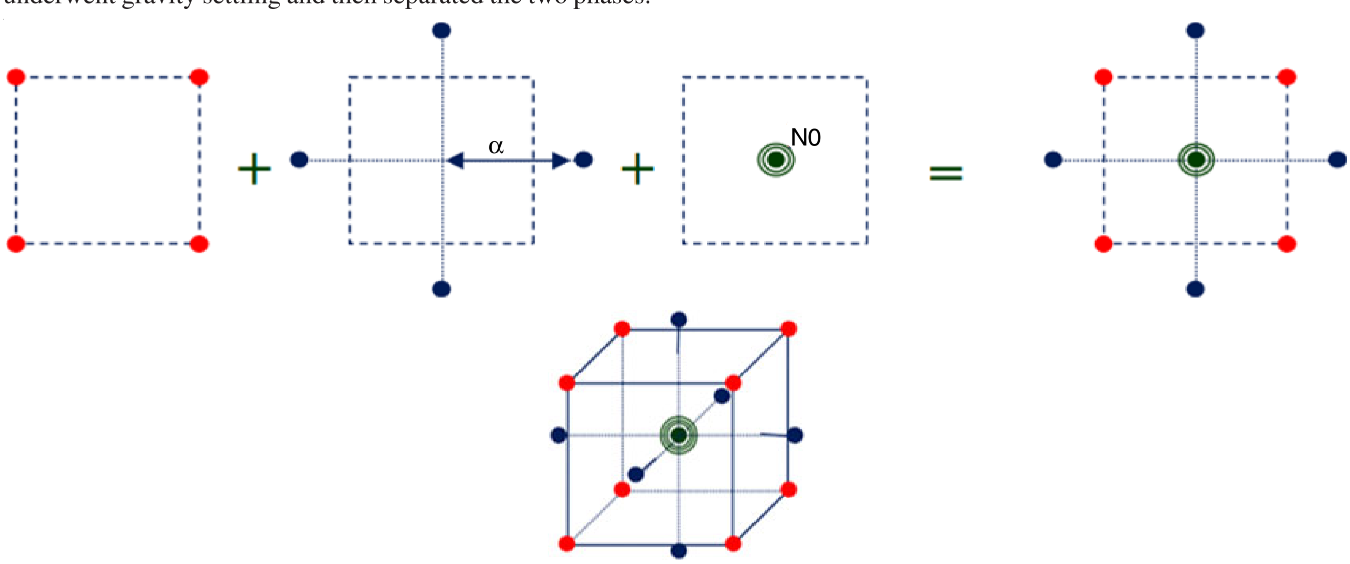


Fig. 1. A central composite design

TABLE-1
NATURAL AND CODED VARIABLES

Coded variables	X_1, X_2, X_3, X_4, X_5*	Unit	Levels				
			-1.3408	-1	0	1	1.3408
Natural variables (x_i)	x_1 = pH	–	1.66	2	3	4	4.34
	x_2 = [EA]	10^{-2} M	0.33	0.5	1	1.5	1.67
	x_3 = O/A	–	0.3	0.4	0.7	1	1.1
	x_4 = Duration	min	–	30	–	90	–
	x_5 = Stirring rate	rpm	–	400	–	800	–

$X_1 = (x_1 - 3)/1$; $X_2 = (x_2 - 1)/0.5$; $X_3 = (x_3 - 0.7)/0.3$; $X_4 = (x_4 - 600)/200$; $X_5 = (x_5 - 75)/15$

This model contains 19 terms: Constant term = 1, Linear terms = 5, Square terms = 3, Rectangular terms = 10.

In order to calculate the b_u coefficient, we can use the least squares method:

$$b_u = \frac{Y_u}{\sum_{i=1}^{n} X_{iu}^2} \quad \text{where: } Y_u = \sum_{i=1}^{n} Y_i \qquad (3)$$

The values X_{iu} and y_i correspond to the experiment i, the contrast is represented by the term Y_u.

Matrix of experiences: For the three variables at three levels, the solution is therefore the central composite design whose number of trials, for P variables with an experiment at the center is:

$$2^P + 2P + 1 = N \qquad (4)$$

Either N = 15 for P = 3, the odd number does not allow for a balanced contrast between the two levels of the other variables X_4 and X_5. The trick is to add a test in the center that doesn't disturb in any way, *i.e.* N = 16 which is a power of 2. Unfortunately, this number is less than the number of coefficients in the model which are 19. To complete this plan, it is sufficient to double the number of trials, *i.e.* 32 experiments But, this number of trials can be reduced: indeed, the term 2^P of eqn. 4 becomes a plane 2^4 where X_4 or X_5 is naturally introduced or by defining the last variable through the intermediary of the four first ones: $X_5 = X_1X_2X_3X_4$ or $X_4 = X_1X_2X_3X_5$. These 4 interactions are all concomitant with triple interactions that are neglected. The first 16 trials are therefore those of the fractional factorial design 2^{5-1}.

As for the 8 trials of the part of the star plane of the central composite plane corresponding to the axial points + number of experiments in the center, *i.e.* (2 × 3) + 2 = 8 of the first 3 variables, it is easy to add the ordinary contrasts of the complete plane 2^2, repeated twice in order to obtain the desired orthogonality. There is therefore no need to repeat the star part.

Table-2 lists the set of de 16 + 8 = 24 trials, defining the matrix of experiments. The nth row of this matrix defines the experimental conditions of the nth experiment.

The calculation of the distance ± α of the axial points is determined by solving the ordinary two-square equation:

TABLE-2
CENTRAL COMPOSITE DESIGN PRESENTED ACCORDING TO THE STANDARD ORDER

	Order		Coded variables values				
	Logical order	Random order	X_1	X_2	X_3	X_4	X_5
Factorial points	1	5	–	–	–	–	+
	2	1	–	–	–	+	–
	3	6	–	–	+	–	–
	4	11	–	–	+	+	+
	5	23	–	+	–	–	–
	6	15	–	+	–	+	+
	7	20	–	+	+	–	+
	8	18	–	+	+	+	–
	9	2	+	–	–	–	–
	10	7	+	–	–	+	+
	11	14	+	–	+	–	+
	12	22	+	–	+	+	–
	13	9	+	+	–	–	+
	14	8	+	+	–	+	–
	15	12	+	+	+	–	–
	16	13	+	+	+	+	+
Axial points	17	24	-1.3408	0	0	–	–
	18	21	+1.3408	0	0	–	–
	19	16	0	-1.3408	0	–	+
	20	4	0	+1.3408	0	–	+
	21	17	0	0	-1.3408	+	–
	22	3	0	0	+1.3408	+	–
Centre points	23	19	0	0	0	+	+
	24	10	0	0	0	+	+

$$16-\left(\frac{(16+2\alpha^2)^2}{24}\right)=0 \quad (5)$$

The result is α = 1.3408.

RESULTS AND DISCUSSION

In order to make an overall study of the effect of each factor and their interactions on the percentage extraction of cadmium from phosphoric acid, a statistical analysis was carried out by the JMP software [12,13]. The experimental (y_i) and theoretically predicted ($\hat{y}_i$) results are presented in Table-3. These estimated values ($\hat{y}_i$) and the corresponding residue ($e_i = y_i - \hat{y}_i$) are calculated from the following model:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{15}X_1X_5 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{25}X_2X_5 + b_{34}X_3X_4 + b_{35}X_3X_5 + b_{45}X_4X_5 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (6)$$

The analysis of variance of the experimental error (ANOVA) is shown in Table-5. This variance is obtained by dividing the sum of the squares Σe_i^2 of the residual by the number of the degree of freedom ν (number of degree of freedom = number of experiments – number of model coefficients). The estimation of the variance of the experimental error (S_R^2) is obtained:

$$S_R^2=\frac{26.8335}{13}=2.06412 \quad (7)$$

These analyses are performed using Fisher's Snedecor 'F' test. This Fisher's Snedecor test is used to determine the significance of each of the interactions between the variables, which in turn can indicate trends in the interactions between the variables.

The experimental Snedecor factor is obtained by dividing the mean square (CM_u) by the variance of the experimental error (S_R^2) [15,16]:

$$F_{exp}=\frac{CM_u}{S_R^2} \quad (8)$$

The estimate of the individual mean square (CM_u) is obtained by dividing the sum of the squares of each coefficient (SS_u) by its degree of liberty ($\nu_u = 1$):

$$CM_u=\frac{SS_u}{\nu_u} \quad (9)$$

The estimate of the sum of the squares of the coefficients (SS_u) is obtained by multiplying the square of the coefficient (b_u) by the sum of the squares of the values of X_u:

$$SS_u=b_u^2\sum X_{iu}^2 \quad (10)$$

In general, the larger the amplitude of F, the smaller the value of P, the more significant the corresponding coefficient term.

Tests of effects and estimation of coefficients: The coefficients of the cadmium extraction percentage response model for the different factors and their meanings are grouped in Table-4. It is found that (i) for a confidence level of 99 %, the most significant factors are the ratio of the organic phase to the aqueous phase (O/A) (P = 0.0002), the pH (P = 0.0009)

TABLE-3
EXPERIMENTAL DATA AND THEORETICALLY PREDICTED OF THE CADMIUM EXTRACTION PERCENTAGE OF PHOSPHORIC ACID ACCORDING TO THE COMPOSITE CENTRAL PLAN

Order		Coded variables values					Reponse % Cd		
Logical	Random	X_1	X_2	X_3	X_4	X_5	Experimental (y_i)	Predicted ($\hat{y}_i$)	Standard error ($e_i = y_i - \hat{y}_i$)
1	5	–	–	–	–	+	88.81	88.90013	–0.09013
2	1	–	–	–	+	–	81.47	80.37431	1.09570
3	6	–	–	+	–	–	85.1	84.32860	0.77140
4	11	–	–	+	+	+	98.2	99.20851	–1.00851
5	23	–	+	–	–	–	83.85	82.57652	1.27348
6	15	–	+	–	+	+	83.37	83.87642	–0.50642
7	20	–	+	+	–	+	92.26	90.94287	1.31713
8	18	–	+	+	+	–	98.46	97.96539	0.49461
9	2	+	–	–	–	–	68.78	68.66116	0.11884
10	7	+	–	–	+	+	72.45	73.17499	–0.72499
11	14	+	–	+	–	+	89.57	89.84465	–0.27465
12	22	+	–	+	+	–	85.38	84.66682	0.71318
13	9	+	+	–	–	+	79.12	78.08639	1.03361
14	8	+	+	–	+	–	79.98	79.57091	0.40909
15	12	+	+	+	–	–	86.29	85.76807	0.52193
16	13	+	+	+	+	+	87.98	88.30190	–0.32190
17	24	–1.3408	0	0	–	–	78.6	80.64139	–2.04139
18	21	+1.3408	0	0	–	–	72.99	73.63426	–0.64426
19	16	0	–1.3408	0	–	+	93.31	93.11475	0.19525
20	4	0	+1.3408	0	–	+	86.24	88.42120	–2.18120
21	17	0	0	–1.3408	+	–	79.23	80.73479	–1.50479
22	3	0	0	+1.3408	+	–	96.79	97.99779	–1.20779
23	19	0	0	0	+	+	91.86	89.85909	2.00091
24	10	0	0	0	+	+	92.42	89.85909	0.56091

TABLE-4
TESTS OF EFFECTS AND ESTIMATION OF COEFFICIENTS

F_{exp}	Prob. > |F|	Sum of squares (SC_{bu})			Significance
Constante	77.228957	-	-	<.0001	
pH	-3.749434	259.75558	48.4014	0.0009	***
[EA]	2.2455055	23.5908	4.3958	0.0901	*
O/A	17.86224	530.08733	98.7734	0.0002	***
Duration	0.0376797	22.23375	4.1429	0.0974	*
Stirring rate	0.5577249	55.23995	10.2931	0.0238	**
(pH)*(pH)	-3.850745	70.7474	13.1827	0.015	**
(pH)*([EA])	1.60375	10.28806	1.917	0.2248	NS
([EA])*([EA])	0.5210327	0.09533	0.0178	0.8992	NS
(pH)*(O/A)	2.5770833	9.56356	1.782	0.2395	NS
([EA])*(O/A)	-3.3625	4.07031	0.7584	0.4237	NS
(O/A)*(O/A)	0.020593	1.63E-05	0	0.9987	NS
(pH)*(Duration)	-0.023588	9.25307	1.7242	0.2462	NS
([EA])*(Duration)	0.0277112	3.22409	0.6008	0.4733	NS
(O/A)*(Duration)	0.1422639	30.26511	5.6394	0.0636	*
(pH)*(Stirring rate)	-0.137741	3.47869	0.6482	0.4573	NS
([EA])*(Stirring rate)	-1.498837	103.98805	19.3765	0.007	***
(O/A)*(Stirring rate)	0.1794974	0.53119	0.099	0.7658	NS
(Duration)*(Stirring rate)	-0.016174	40.73373	7.5901	0.0401	**

***Significant to 1 % ($F_{0.01}(1,5) = 16.26$); ** Significant to 5 % ($F_{0.05}(1,5) = 6.61$); * Significant to 10 % ($F_{0.10}(1,5) = 4.06$) [15,16]
NS = Non-significative

and the interaction of the extracting agent concentration [EA]* stirring rate (P = 0.007); (ii) for a confidence level of 95%, the significant factors are the stirring rate (P = 0.0238), the quadratic pH*pH interaction (P = 0.015) and the duration of stirring*stirring rate (P = 0.0401); and (iii) for a confidence level of 90%, the significant factors are the concentration of extracting agent [EA] (P = 0.0901), the duration of agitation (P = 0.0974), the O/A* duration of stirring interaction (P = 0.0636).

However, the effects of these interactions *viz.* pH*[EA], [EA]*[EA], pH*(O/A), [EA]*(O/A), (O/A)*(O/A), pH*Duration, [EA]*Duration, pH*Stirring rate, (O/A)* Stirring rate, are not significant (P = 0.2248, P = 0.8992, P = 0.2395, P = 0.4237, P = 0.9987,P = 0.2462, P = 0.4733, P = 0.4573, P = 0.7658, respectively).

Therefore, the model (eqn. 6) is developed as follows for a 90% significance level:

$$Y = 77.228957 - 3.749434X_1 + 2.2455055X_2 + 17.86224X_3 + 0.0376797X_4 + 0.5577249X_5 - 3.850745X_1^2 + 0.1422639X_3X_4 - 1.498837X_2X_5 - 0.016174X_4X_5 \quad (11)$$

The values in parentheses and below each coefficient in the model (Tables 4 and 5) represent the standard deviations S_{bu}, where S^2_{bu} is the estimated variance of the b_u coefficients. It is calculated by applying the formula:

$$S^2_{bu} = \frac{S_4^2}{\sum X_{iu}^2} \quad (13)$$

The result is:

$$S_{bj} = \sqrt{0.00609832} = 0.07809176$$

$$S_{bjj'} = \sqrt{0.9105001} = 1.8210002$$

$$S_{bjj} = \sqrt{0.13920168} = 0.37309742$$

In present case, the ANOVA of percentage of cadmium extraction response indicates that the model is very significant where F_{exp} (18,5) = 14.7423) is greater than the theoretical ($F_{0.01}(18,5) = 4.59$) at $\nu_1 = 18$ and $\nu_1 = 5$ degrees of liberty, for a confidence level of 99%, with the P value 0.0037 < 0.01.

Effects of different factors and their interactions on the cadmium extraction percentage: The Pareto diagram represents the classification of the orthogonal estimation of the coefficients from highly significant to non-significant. Table-6 shows that the effect of the ratio of organic phase to aqueous phase (O/A) and pH are the most significant on the percentage extraction of cadmium from phosphoric acid relative to other factors. It is also found that the effect of the interaction concentration of the extracting agent [EA]* stirring rate and the quadratic effect of pH are more significant compared to the other interactions.

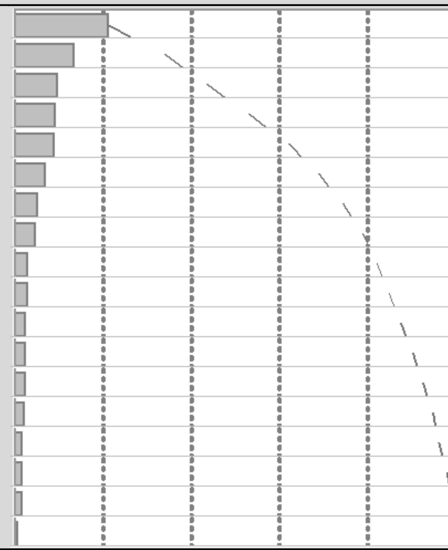
TABLE-5
ANALYSIS OF REGRESSION VARIANCE FOR THE CADMIUM EXTRACTION PERCENTAGE RESPONSE

Source	Degrees of liberty	Sum of squares	Mean square	F_{exp}	P-value	Signification
Model	18	1424.1159	79.1175	14.7423	0,0037	***
Residues	5	26.8335	5.3667	-		
Total	23	1450.9494				

F_{exp}: Experimental Snedecor factor; ***: significant to 99% ($F_{0.01}(18,5) = 4.59$) [15].

TABLE-6
ESTIMATED REGRESSION COEFFICIENTS SORTED IN ASCENDING ORDER OF THE CADMIUM EXTRACTION PERCENTAGE

Term	Estimation orthogonal
O/A	5.023210
pH	-3.204571
Stirring rate	2.356150
pH*pH	-2.279569
[EA]*Stirring rate	-2.099693
Duration	1.699710
Duration*Stirring rate	-1.302781
O/A*Duration	1.113330
[EA]*Duration	0.744442
pH*Duration	-0.688207
pH*[EA]	0.654728
pH*O/A	0.631254
[EA]	0.556225
O/A*O/A	0.467526
[EA]*O/A	-0.411820
pH*Stirring rate	-0.380717
[EA]*[EA]	0.366454
O/A*Stirring rate	0.148771



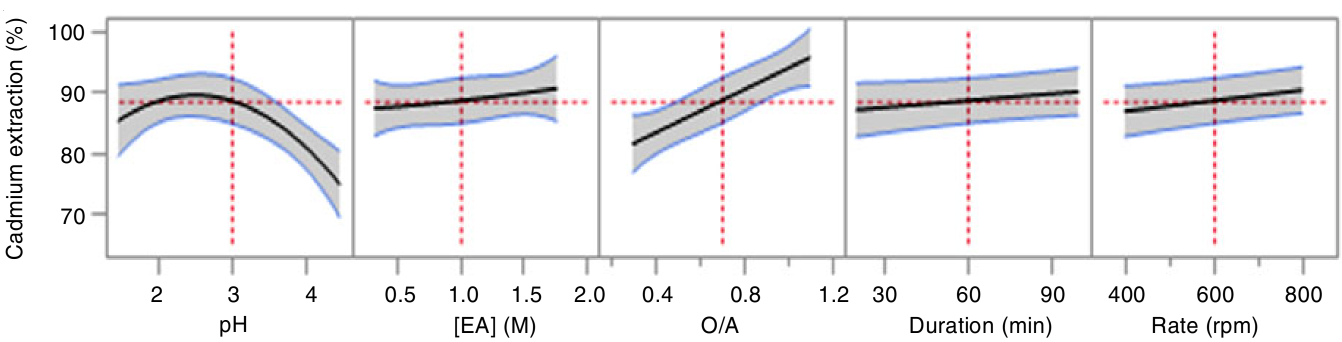


Fig. 2. Effect of the five factors on the percentage extraction of cadmium

Estimation of the coefficients of present response (Fig. 2) shows that the pH has a positive effect for a range between 1.5 and 3 on the percentage of cadmium extraction and above the value 3 the effect of the pH restores a negative effect. On the other hand, the concentration of the extracting agent [EA] × 10^{-2} M, the ratio of the organic phase/aqueous phase (O/A), the rate and duration of stirring have a positive effect on present response.

Validation of model: Fig. 3 illustrates the correlation between predicted and experimental values. From this curve, it can be seen that the model represents well the response (extraction percentage of cadmium) as a function of the five factors, with a high correlation coefficient $R^2 = 0.98$ and a low value of P = 0.0037. Similarly, the difference between the predicted and experimental values is very small.

Optimization: In order to predict an optimal response, a graphical representation (Fig. 4) of the model in the variable space is carried out by simulating the response surface and by contour plotting. Fig. 4a shows the representation of the percentage cadmium extraction response of phosphoric acid in responses surfaces. The projection of this response in the O/A = f(pH) diagram gives an outline plot (Fig. 4b-c). These plots show the optimal conditions for a high cadmium extraction

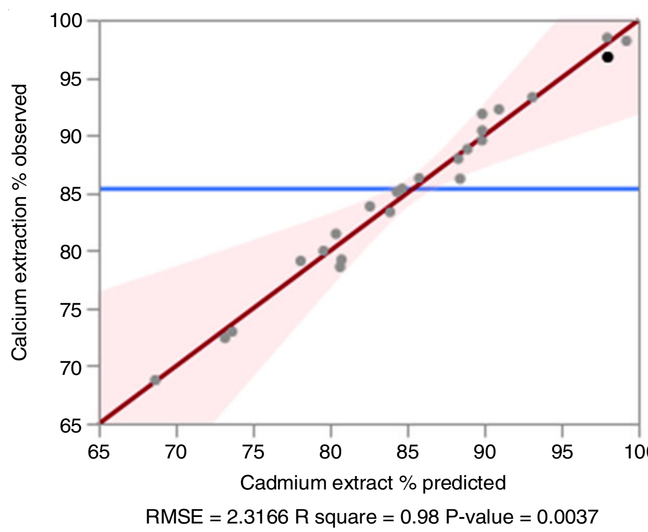


Fig. 3. Correlation between observed values and predicted values of the cadmium extraction percentage

percentage; the pH for a range from 2 to 3.5, the ratio of the organic phase/aqueous phase (O/A) for a range from 0.9 to 1.2, the concentration of the extracting agent which is of the order of 1 × 10^{-2} M, the duration and stirring rate which corres-

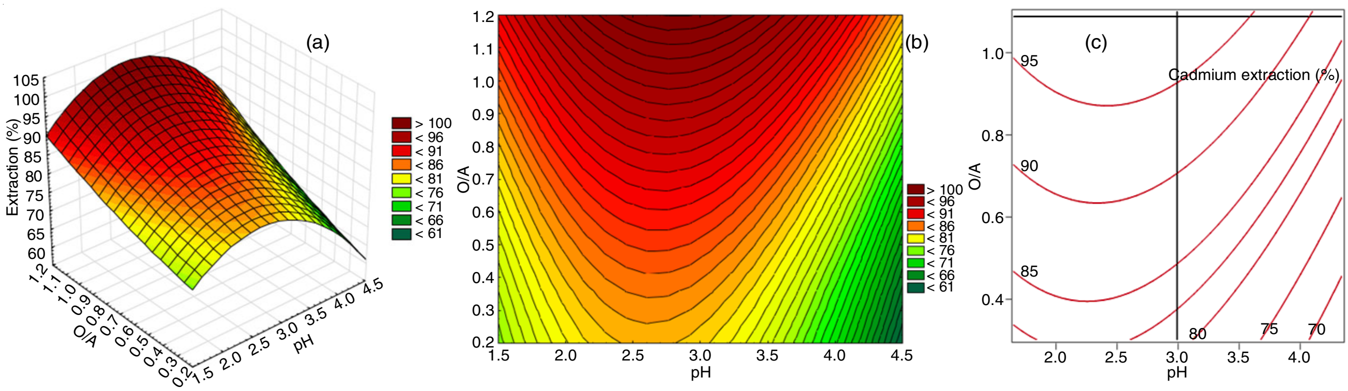


Fig. 4. Response surface and contour plot of the cadmium extraction percentage

pond to 90 min and 800 rpm, respectively. These different conditions allow us to obtain cadmium extraction percentages higher than 96%. Therefore, the optimal point of our response, which was of the order of 98.68%, corresponds to the following conditions: pH = 3, [EA] = 1.10^{-2} M, O/A = 1.1, stirring duration 90 min, stirring rate 800 rpm. This predicted value (98.68%) of the response was experimentally confirmed under these conditions, where a percentage of the order of 98% was obtained.

The optimization of the various parameters can influence the cadmium extraction process, such as concentration of the extracting agent, pH, phase ratio (O/A), duration and stirring rate, resulted in a high cadmium extraction percentage of 98%. The use of hexahydroquinazolin-2-ones ($C_{11}H_{18}N_2O$) as an extracting agent showed impressive results when compared to other extracting agents reported in the literature. The use of diluted di-2-ethyl hexyl phosphoric acid (D2EHPA) as an extracting agent in benzene and kerosene resulted in a cadmium extraction percentage of 56% [6] and 65.46% [7], respectively. In another work, another extractant 7-(4-ethyl-1-methyloctyl)-8-hydroxyquinoline diluted in kerosene resulted in an extraction percentage of 71% [17].

Conclusion

The extraction of cadmium from phosphoric acid by the solvent extraction process was modelled and optimized using the surface response method. The selected composite central design clearly demonstrated successful experimental design and analysis of the results. The optimal conditions for the five factors involved were pH = 3, concentration of the extracting agent [EA]= 1.10^{-2} M, organic phase/aqueous phase (O/A) ratio = 1.1, stirring duration = 90 min and stirring rate 800 rpm. The effect of the ratio of organic phase to aqueous phase (O/A) and pH are the most significant on the percentage extraction of cadmium from phosphoric acid relative to other factors. Also, the effect of the extractant interaction concentration [EA]* and the squared effect of pH are more important compared to the other interactions.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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