

Simultaneous effect of pH, deposition time, deposition potential, and step potential on the stripping peak current of lead and cadmium by response surface methodology

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Abstract

In this paper, a simple voltammetric method has been reported for the lead, and cadmium determination using platinum nanoflowers modified glassy carbon electrode (PtNFs/GCE). The effects of pH, deposition time, deposition potential, step potential were investigated on the stripping peak current of lead, and cadmium based on response surface methodology (RSM). The results of RSM analysis and analysis of variance (ANOVA) have shown that the experimental data could be well described by quadratic regression equations with determination coefficients (R^2) of 0.935, and 0.972 for the stripping peak current of lead, and cadmium, respectively. Results of the statistical analysis showed that the fit of the model was good in all cases. The maximum stripping peak current of the lead, and cadmium of 5.54 μ A, and 2.81 μ A, respectively were obtained at the optimum levels of process variables (pH (4.72), deposition potential (-1.14V), deposition time (120s), step potential (7mV)). Testing the model to analyze lead, and cadmium on the PtNFs/GC electrode using differential pulse anodic stripping voltammetry (DPASV) and obtained with the stripping peak current of the lead, and cadmium of 5.43 μ A, and 2.75 μ A, respectively.

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Pb²⁺, Cd²⁺, PtNFs/GCE,
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1 Introduction

Nowadays, the contamination of water by heavy metal ions has become one of the main environmental problems[1]. The wastewaters released from industries such as mining, milling, plating, oil refining, metallurgy, storage batteries, fertilizer production, textile dyeing, and alloy industries contain many heavy metal ions, which widely enter the environment without adequate treatment processes[2]. Heavy metals at higher concentrations can be dangerous and can accumulate in living tissues, causing various diseases[3]. Lead and cadmium pollution is an urgent environmental problem because of the complexity of their mechanisms of biological toxicity and stability in contaminated sites. Lead and cadmium accumulated in the body once absorbed and

endanger the health of humans[4]. A number of popular methods, including isotope dilution, inductively coupled plasma mass spectrometry (ID ICP-MS)[5], and flame atomic absorption spectrometry (FAAS)[6], have been used for the determination of lead and cadmium in different aqueous solution. Most of the reported methods are the high cost of equipment and maintenance, complicated operation, time-consuming and require special sample preparation. For these reasons, the rapid, simple and accurate method is expected to be established.

Among of different analytical methods, electrochemical methods are commonly used for the determination of heavy metal ions, because of their ease of operation, low cost, high sensitivity, and the ability to analyze elemental speciation.



Particularly, modification of electrode surfaces is one of the important developments in recent years because modification of the electrode surfaces significantly increases the sensitivity along with a considerable decrease in detection limit and interfering effects. The use of nanoelectrodes in the field of electrochemical sensors has become an interesting trend in electrochemical research because of their advantages such as increased mass transport, rapid electron transfer and high surface-to-volume ratio[7,8]. The catalytic activity of platinum nanoparticles in the electrochemical analysis was investigated by Yoon et al.[9] by blending Pt nanoparticles with carbon powder and organic binder for electrode manufacture. This modified electrode improved the copper peak current which is three times higher than that measured on the non-modified electrode.

Hence, we studied to develop a new, simple and sensitive platinum nanoflowers modified glassy carbon electrode for the determination of lead, and cadmium. Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving, and optimizing processes[10]. Response surface methodology was used to obtain optimum experimental conditions such as pH, deposition time, deposition potential, step potential.

2 Material and methods

2.1 Material

2.1.1 Reagents

$\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ (Merck); H_2SO_4 (Merck); CH_3COOH (Merck); CH_3COONa (Merck); Lead, and Cadmium stock solution (1000 ppm), purchased from Merck was used for dilution. All chemicals were of analytical grade and distilled water was used for preparing all of the solutions.

2.1.2 Apparatus

Electrochemical measurements were performed using an Autolab CPA-HH5 (Vietnam Academy of Science and Technology) and three-electrode system with platinum nanoflowers modified glassy carbon electrode (PtNFs/GCE) as working electrode, an Ag/AgCl reference electrode and a platinum wire counter electrode were used to perform

electrochemical measurements. Field-emission scanning electron microscope (FE-SEM, S-4800, Hitachi Company, Japan) was employed to evaluate the morphologies of the PtNFs/GCE.

2.2 Method

The electrodeposition of platinum nanoparticles on the bare glassy carbon electrode was carried out in 0.1 M H_2SO_4 solution containing 1.0 mM H_2PtCl_6 at a constant potential of -0.2V.

Following that, the PtNFs/GCE was gently cleaned with distilled water before use. Detection of Pb^{2+} ($10\mu\text{g.L}^{-1}$) and Cd^{2+} ($10\mu\text{g.L}^{-1}$) were performed by different pulse anodic stripping voltammetry (DPASV) in an acetate buffer solution 0.1M. The potential was scanned from -1.2V to +0.2V with pulse amplitude 0.060V; pulse time 0.050 s; step time 0.03 s. In order to enhance the measurement sensitivity, the parameters influencing the stripping peak current were optimized to achieve the required sensitivity. pH, deposition time, deposition potential, step potential were optimized and used in the recommended procedure. All experiments described in this section were performed at room temperature ($25 \pm 1^\circ\text{C}$).

The statistical software MODDE 12.1 trial (Umetrics, Sweden) was used to create the experimental design, statistical analyses, and regression model. RSM based on quadratic and cubic models with central composite circumscribed design (CCC) is composed of full factorial design and star points (star distance: $\alpha = 2$). It has been used to study the simultaneous effects of independent variables (pH, deposition time, deposition potential, step potential) on response functions.

The four independent variables pH, deposition time (s), deposition potential (V), step potential (mV) (were coded with X_1 , X_2 , X_3 , and X_4 , respectively, and each independent variable had five levels (Table 1)). The real value of the variable was related to the coded variable by the formula (1):

$$\text{Coded variable} = \frac{X - X_0}{\lambda} \quad (1)$$

Where X_0 is the real value of variables at the central level, and λ is the step change of the variable. The experiments with coded and real values of the variables are shown in Table 1.

Table 1 Experimental range and levels of the independent variables

Symbol	Variable	Coded variable and Independent variables				
		$-\alpha$	-1	0	1	$+\alpha$
X_1	pH	3.5	4.0	4.5	5.0	5.5
X_2	tdep (s)	60	90	120	150	180
X_3	E (V)	-1.3	-1.2	-1.1	-1.0	-0.9
X_4	U (mV)	4	6	8	10	12

The response functions (Y_1 , Y_2) are the stripping peak current of lead, and cadmium, respectively. The relationship between the response functions and the coded variables is presented by a second-degree polynomial (2):

$$Y = \beta_0 + \beta_i \sum X_i + \beta_{ii} \sum X_i^2 + \beta_{ij} \sum X_i X_j \quad (2)$$

Where Y is a response function; X_i and X_j are independent variables; β_0 is a constant; β_i , β_{ii} , β_{ij} are linear, quadric, and interactive coefficients, respectively. Thirty-one combinations along with 7 replicates of the central point were formed, corresponding to 24 experiments.

3 Result and discussion

3.1 Surface Morphology of PtNFs/GCE

The surface morphology of PtNFs/GCE was investigated by microscopic imaging analysis. Figure 1 shows the typical SEM image of Pt layer electrodeposited on GCE at -0.2 V of potential and 150 s of deposition duration. As can be seen in the SEM image that Pt was formed separately on the GCE (lighter areas) in nanoflowers shape with size varies in the range (50 – 400 nm).

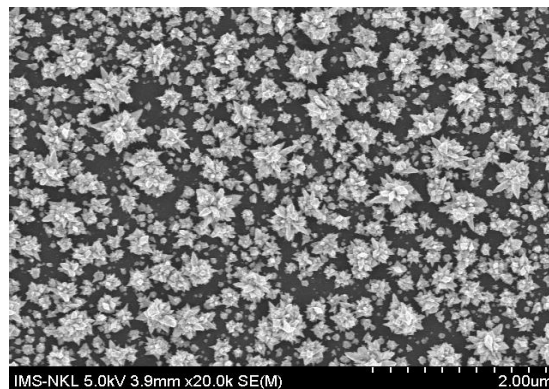


Fig. 1 SEM image of PtNFs/GCE deposited at a potential of -0.2 V for 150 s

3.2 Fitting the model

The 4-factors CCC matrix predicted values and experimental results for the stripping peak current response of lead and cadmium were presented in Table 2. These results were used for statistical analysis and to predict the regression equation with the software MODDE 12.1 trial.

Table 2 The RSM experiment design matrix and experimental results

Exp	Run order	Coded variable				Stripping peak current of lead (I_{Pb}) (μA)		Stripping peak current of cadmium (I_{Cd}) (μA)	
		X_1	X_2	X_3	X_4	Experiment	Predicted	Experiment	Predicted
1	14	-1	-1	-1	-1	3.14	3.30	1.73	1.74
2	9	1	-1	-1	-1	3.86	3.96	1.98	2.04
3	21	-1	1	-1	-1	5.79	5.87	2.74	2.87
4	29	1	1	-1	-1	6.56	6.53	3.08	3.17
5	3	-1	-1	1	-1	2.08	2.40	1.56	1.46
6	18	1	-1	1	-1	2.79	3.07	1.75	1.76
7	16	-1	1	1	-1	4.68	4.97	2.49	2.58
8	23	1	1	1	-1	5.36	5.63	2.87	2.88
9	10	-1	-1	-1	1	1.84	2.45	1.57	1.58
10	17	1	-1	-1	1	2.66	3.11	1.8	1.88
11	1	-1	1	-1	1	4.54	5.01	2.58	2.70
12	13	1	1	-1	1	5.39	5.67	2.91	3.00
13	22	-1	-1	1	1	1.69	1.55	1.41	1.30
14	15	1	-1	1	1	2.15	2.21	1.61	1.60
15	31	-1	1	1	1	3.87	4.11	2.31	2.42
16	8	1	1	1	1	4.18	4.77	2.69	2.72
17	2	-2	0	0	0	3.81	3.30	1.84	1.79
18	20	2	0	0	0	5.12	4.62	2.49	2.39
19	25	0	-2	0	0	2.62	2.21	1.28	1.38
20	27	0	2	0	0	7.93	7.34	3.88	3.62
21	28	0	0	-2	0	5.21	4.65	2.49	2.26
22	30	0	0	2	0	3.31	2.86	1.62	1.69
23	11	0	0	0	-2	4.76	4.53	2.59	2.52
24	4	0	0	0	2	3.59	2.81	2.28	2.20
25	7	0	0	0	0	5.22	5.51	2.74	2.74
26	26	0	0	0	0	5.58	5.51	2.60	2.74
27	24	0	0	0	0	5.41	5.51	2.78	2.74

28	6	0	0	0	0	5.82	5.51	2.71	2.74
29	5	0	0	0	0	5.01	5.51	2.68	2.74
30	12	0	0	0	0	5.64	5.51	2.84	2.74
31	19	0	0	0	0	5.90	5.51	2.82	2.74

3.3 Develop model and statistic analysis

These results were used for statistical analysis and to predict the regression equation with the software MODDE 12.1 trial. The regression coefficient values for the coded variables of the polynomial functions are shown in Table 3. The

statistical Student's (t-test) was used to evaluate the significance of the regression coefficients. The quadratic regression equation of response functions for the stripping peak current of lead (Eq.3), and cadmium (Eq.4) were obtained after removing insignificant regression coefficients.

Table 3 Regression coefficients values (coded variables) of the polynomial model of responses for the stripping peak current of lead, and cadmium

	For the stripping peak current of lead (μA)				For the stripping peak current of cadmium (μA)			
	Coeff.	Std.Err.	t-test	p-value	Coeff.	Std. Err.	t-test	p-value
β_0	5.511	0.190	45.589	2.90E-15 ^a	2.739	0.050	86.530	1.24E-19 ^a
β_1	0.331	0.103	5.065	0.0053 ^a	0.150	0.027	8.771	4.36E-05 ^a
β_2	1.283	0.103	19.631	1.14E-09 ^a	0.561	0.027	32.792	5.36E-13 ^a
β_3	-0.449	0.103	6.875	0.00047 ^a	-0.143	0.027	8.381	7.09E-05 ^a
β_4	-0.428	0.103	6.557	0.00072 ^a	-0.081	0.027	4.726	0.0086 ^a
β_{11}	-0.387	0.094	6.480	0.00079 ^a	-0.163	0.025	10.388	6.36E-06 ^a
β_{22}	-0.185	0.094	3.097	0.046 ^a	-0.059	0.025	3.767	0.030 ^a
β_{33}	-0.439	0.094	7.337	0.00026 ^a	-0.190	0.025	12.143	9.23E-07 ^a
β_{44}	-0.460	0.094	7.692	0.00016 ^a	-0.095	0.025	6.080	0.0014 ^a
β_{12}	0.0063	0.126	0.078	0.97 ^{ins}	0.035	0.033	1.673	0.31 ^{ins}
β_{13}	-0.063	0.126	0.782	0.63 ^{ins}	0.000	0.033	9.75E-06	1 ^{ins}
β_{14}	-0.028	0.126	0.344	0.83 ^{ins}	-0.001	0.033	0.060	0.97 ^{ins}
β_{23}	-0.088	0.126	1.095	0.50 ^{ins}	-0.012	0.033	0.597	0.71 ^{ins}
β_{24}	-0.055	0.126	0.688	0.667 ^{ins}	-0.004	0.033	0.179	0.91 ^{ins}
β_{34}	0.119	0.126	1.486	0.359 ^{ins}	0.001	0.033	0.060	0.97 ^{ins}

Note: "Std. Err" standard error; ^asignificant at $p < 0.05$; ^{ins}insignificant

Table 4 Analysis of variance (ANOVA) test for the response function

The stripping peak current of lead, and cadmium (μA)	Source	df	SS	MS	Fvalue	Fcritical	P-value
For Lead	Regression	8	64.616	8.077	39.368	1.994	<0.001
	Residual	22	4.514	0.205			
	Pure error	6	0.615	0.102			
	Lack of Fit	16	3.899	0.244	2.381	3.938	0.145
	Total cor.	30	69.129	2.304			
For Cadmium	Regression	8	10.483	1.310	95.293	1.994	0.000
	Residual	22	0.303	0.014			
	Pure error	6	0.0421	0.007			
	Lack of Fit	16	0.260	0.016	2.321	3.938	0.152
	Total cor.	30	10.785	0.360			

Note: "df" degree of freedom; "SS" sum of squares; "MS" means of squares; "Fvalue" fisher distribution; "Fcritical" fisher critical; "Total cor" Total corrected

The result Table 3 indicates four variable independents (X_1 , X_2 , X_3 , X_4) signification (P -value < 0.05) but noticed that X_2 variables greater than X_1 , X_3 , X_4 about statistical signification (P -value < 0.05). Besides interaction quadratic X_1^2 , X_2^2 , X_3^2 , X_4^2 also signification of the statistics. The response for the second-order polynomial is given as follows:

$$Y_1 = 5.511 + 0.331X_1 + 1.283X_2 - 0.449X_3 - 0.428X_4 - 0.388X_1^2 - 0.185X_2^2 - 0.439X_3^2 - 0.460X_4^2 \quad (3)$$

$$Y_2 = 2.739 + 0.150X_1 + 0.561X_2 - 0.143X_3 - 0.081X_4 - 0.163X_1^2 - 0.059X_2^2 - 0.190X_3^2 - 0.0953X_4^2 \quad (4)$$

The evaluation of the coefficient signs provides rapid analysis of the parametrical effects of the model variables on the responses. Negative coefficients point out unfavorable effects for the stripping peak current the components X_3 , X_4 , X_1^2 , X_2^2 , X_3^2 , X_4^2 in polynomial Y_1 , Y_2 . Positive coefficients refer to favorable effects on the stripping peak current for the components X_1 , X_2 . However, it is recommended to further evaluate the statistical significance of the predictors and developed models using the ANOVA at level 95% confidence intervals for the stripping peak current (μA) as shown in Table 4.

3.4 Analysis of variance (ANOVA)

Analysis of variance was used to evaluate the suitability of the regression equation with empirical. The result ANOVA for Y model was indicated in Table 4.

The statistical significance of the model is confirmed by the determination coefficient (R^2), the adjusted determination coefficient (R^2_{adj}), and the Fisher distribution (Fvalue). The results clearly illustrate the high value of the coefficient of determination ($R^2 = 0.935$ and $R^2_{adj} = 0.911$ for the stripping peak current of lead). This means that 93.5% of the variability for lead analysis process can be clarified by the independent variables. And $R^2 = 0.972$ and $R^2_{adj} = 0.962$ for the stripping peak current of cadmium, this means that 97.2% of the variability for cadmium analysis process can be clarified by the independent variables. The lack of fit was also calculated to measure how the model fits the data. Thus, the P-values of the lack of fit for the stripping peak current of lead, and cadmium were 0.145, and 0.152, respectively.

An insignificant lack of fit ($P > 0.05$) at level 95% confidence is a desirable property because it suggests the model fits the data well. The calculated Fvalue for the full quadratic regression equations of the stripping peak current response of lead, and cadmium are 2.381, and 2.321, respectively ($< F(0.95, 16, 6) = 3.938$), indicating that the model fits well with experimental data. The results of ANOVA show that the quadratic regression equation models for the stripping peak current response of lead, and cadmium have good statistical validation for predicting experiments with a valid concentration region.

3.5 Response surface 3D, optimization conditions

To study the influence of experimental variables (pH, deposition time, deposition potential, and step potential) on the stripping peak current of lead, and cadmium, three dimensional (3D) surfaces for the predicted responses were drawn and accessible in Fig. 2.

Based on Eq. (2), the response surface plots were developed and illustrated in Fig. 2. The effect of deposition time variable on the response function is indicated Fig 2a, 2d, 2f. The stripping peak current increased with increasing deposition time. As increasing the deposition time, more Pb^{2+} , and Cd^{2+} will be deposited and adsorbed onto the surface of the PtNFs/GCE, then stripping signal will increase. With increasing pH, the stripping peak increased initially and then decreased at higher pH, as shown in Fig 2a, 2b, 2c. Possibly because in the low pH, metal exists as a weak link (labile form) so it can be reduced and accumulated easily on the electrode surface. In the high pH, metal can be in the form of strong bonds (bound form) so is difficult to be eliminated. As a result, less efficient enrichment happens, leading to the lower stripping peak current. Particularly, the sensitivities obtained from the pH values of 4.5 and 4.75 show almost a similar current. Figure 2b, 2d, 2e showed that the effect of deposition potential variable on the response function. It was observed that the stripping peak current increased as deposition potential was changed from -1.0 V to -1.15 V and then it decreased. The stripping peak current increase as the step potential increases up to 7 mV and then the peak current declined slightly as shown in Fig 2c, 2e, 2f.

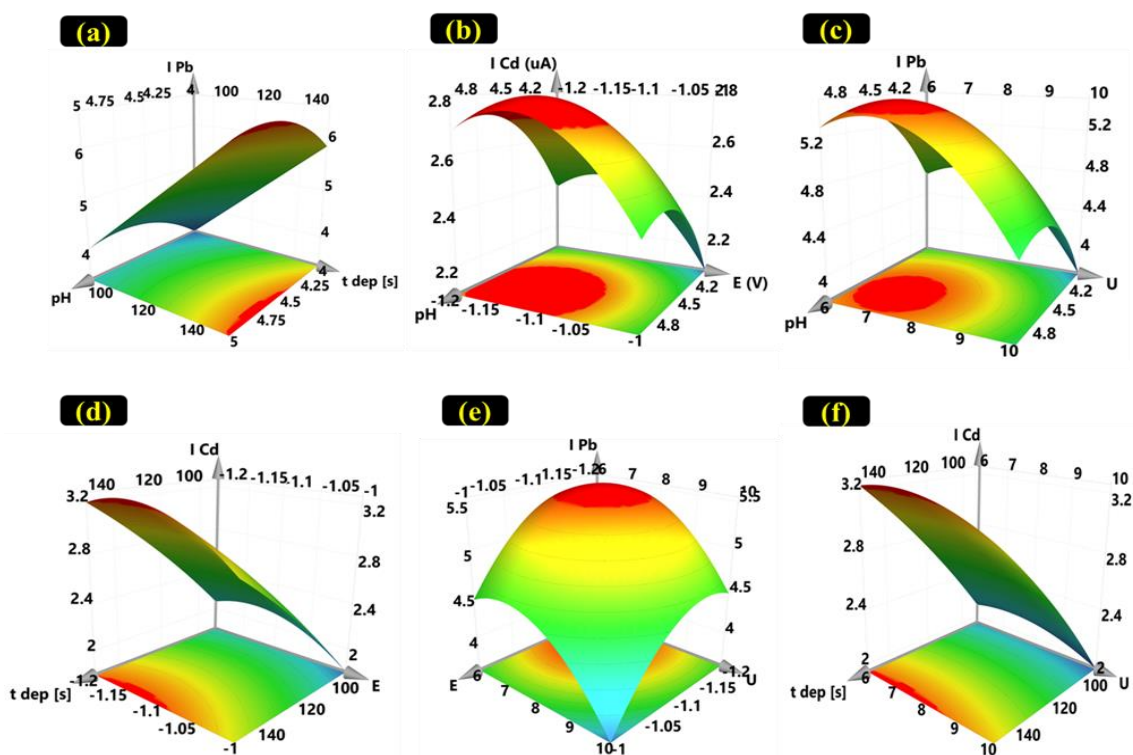


Fig. 2 Response surface showing electrochemical response as a function of two independent variables: (a) pH (X_1) and deposition time (X_2); (b) pH (X_1) and deposition potential (X_3); (c) pH (X_1) and step potential (X_4); (d) deposition time (X_2) and deposition potential (X_3); (e) deposition potential (X_3) and step potential (X_4); (f) deposition time (X_2) and step potential (X_4)

The experimental results were optimized by software MODDE 12.1 trial using the approximating functions of stripping peak current response in Eq. (2). Using highest stripping peak current and lowest relative standard deviation as the optimization target, the optimized condition was obtained as follow: pH of 4.72, deposition potential (-1.14 V), deposition time (120 s), step potential (7 mV) and its application in the determination of lead and cadmium in real water samples. In addition, the interference of some metal ions, surfactants on the stripping peak current will be investigated in the further studies.

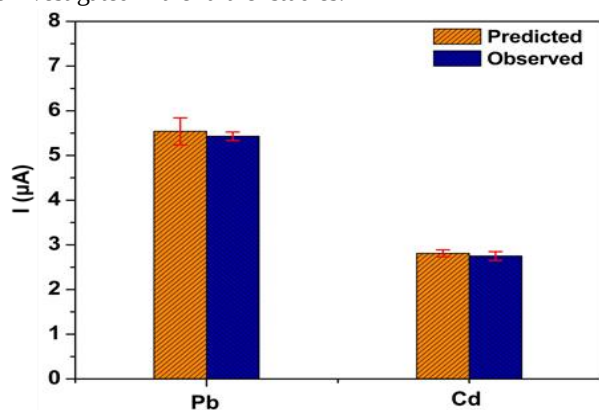


Fig. 3 Comparison modeling and experiment

The comparison in the optimal conditions between predicted and observed values of response was investigated (Fig.3).

Observed values were the measured data from the experiment and the predicted values were calculated by using Eq. (3), and Eq. (4). In this run, the stripping peak current of lead, and cadmium were 5.54 (μA), and 2.81 (μA), respectively as observed value, while predicted value were 5.43 (μA), and 2.75 (μA), respectively. The results confirmed that the predicted value was in good agreement with the observed value and the model performance was validated.

4 Conclusion

Response surface methodology (RSM) was applied for determining and optimizing the variables (pH, deposition time, deposition potential, step potential). The simultaneous effects of four above parameters on the stripping peak current of lead and cadmium are represented in a quadratic regression equation with statistical significance in the experimental region. The model was extracted with R^2 of 0.935, and 0.972 for lead and cadmium, respectively. Analysis variance was used to evaluate the suitability of the regression equation with empirical. ANOVA result shows the model has good data. Finally, results from this method indicated that pH of 4.72, deposition potential (-1.14 V), deposition time (120 s), and step potential (7 mV) results in the maximum current.

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Nghiên cứu ảnh hưởng đồng thời của pH, thời gian điện phân, thế điện phân làm giàu và bước nhảy thế đến cường độ dòng đỉnh hòa tan của chì và cadimi bằng phương pháp đáp ứng bề mặt

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Tóm tắt Trong bài báo này, một phương pháp Von - ampe hòa tan đơn giản đã được báo cáo cho xác định chì và cadimi sử dụng điện cực là các bông hoa nano platin biến tính trên nền cacbon thủy tinh (PtNFs/GCE). Ảnh hưởng của pH, thời gian điện phân, thế điện phân làm giàu và bước nhảy thế đến cường độ dòng đỉnh hòa tan của chì và cadimi đã được nghiên cứu bằng phương pháp đáp ứng bề mặt (RSM). Kết quả phân tích RSM và phân tích phương sai ANOVA đã chỉ ra rằng dữ liệu thực nghiệm có thể được mô tả bằng phương trình hồi quy bậc hai với hệ số xác định (R^2) là 0,935, 0,972 cho xác định chì và cadimi tương ứng. Cường độ dòng đỉnh hòa tan của chì, cadimi đạt cực đại theo mô hình là 5.54 μ A, and 2.81 μ A tại giá trị tối ưu của các yếu tố pH (4,72), thế điện phân làm giàu (-1,14 V), thời gian điện phân (120 s) và bước nhảy thế (7 m V). Chúng tôi thử nghiệm phân tích Pb(II), Cd(II) trên điện cực PtNFs/GCE bằng phương pháp Von – ampe hòa tan anot xung vi phân với điều kiện của mô hình và kết quả thu được cường độ dòng đỉnh hòa tan của chì, cadimi là 5.43 μ A, and 2.75 μ A.

Từ khóa Pb²⁺, Cd²⁺, PtNFs/GCE, phương pháp đáp ứng bề mặt, DPASV.