



## Application of Response Surface Methodology for Optimization of Platinum (IV) Adsorption Using Magnetic Cellulose Nanoparticles Modified with Ethylenediamine

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### ABSTRACT

The current study adsorption characteristics of platinum(IV) onto the ethylenediamine-modified magnetic cellulose nanoparticles (MCNGE) have been investigated. The prepared adsorbent were characterized using Fourier transform spectroscopy (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM) techniques. Optimization the experimental parameters namely Pt(IV) concentration (15-35 mg/l), temperature (34–50 °C), pH of solution (2–5), and particles dose (0.03-0.06 g) were performed using a means of central composite design (CCD) and response surface methodology (RSM). Analysis of variance (ANOVA) was conducted to evaluation the model, the main of the independent variables and their interactions for adsorption of Pt(IV) from aqueous solution. The results of the quadratic model indicated that the model was highly significant with F-value ( $F_{\text{model}} = 55.09$ ) and value of  $\text{prob} > F (< 0.0001)$ . The optimum adsorption conditions were determined as initial pH 2.5, temperature 46°C, adsorbent dosage 0.05 g and initial platinum(IV) ion concentration 22mg/l. The maximum capacity of MCNGE for Pt(IV) was found to be 19.45 mg/g. The magnetic cellulose nanoparticle is an environmental friendly product with low energy costs in adsorption of heavy metals from aqueous phase.

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### INTRODUCTION

Platinum is a precious metal and extensively used in various industries because of specific chemical and physical properties. Precious metals like platinum is not balanced by supply due to the limited resource, for this reason various preconcentration approaches have been documented in previous books and reviews: liquid–liquid extraction (LLE), precipitation, coprecipitation, and adsorption etc [1–3]. There is a requirement to develop a system for recovering precious metals from aqueous solution which should be environmental friendly product with low cost.

Magnetic chelating resins are widely used in adsorption technology for removing heavy metal ions and concentration of noble metals because they have some advantages, including precipitated rapidly, low energy consumption process, cost-effectiveness and easily collected [4,5].

Cellulose is the most abundant and renewable polymer in nature and along with its derivatives has been

widely used in earth [6-9]. Cellulose itself has low adsorption as well as variable physical stability. The properties of cellulose may be modified with changing both chemical and physical structure. The modification of cellulose can be performed to achieve suitable structural and satisfactorily properties for adsorption process. The main processes for chemical modification of cellulose include oxidation, halogenations, esterification and etherification. Valuable properties can also be imparted to cellulose with grafting a second polymer on the cellulose polymer backbone [10].

In this study, magnetic cellulose nanoparticles have been prepared, and then modified by grafting of glycidylmethacrylate and reaction with amino groups (ethylenediamine). The resulting modified magnetic cellulose nanoparticles were characterized by Fourier transform spectroscopy (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM). Thus, the MCNGE was used for the adsorption of Pt(IV) from aqueous solution. Influence of adsorption parameters such as pH, initial Pt(IV), adsorbent dose and

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temperature on percent adsorption have been investigated. A central composite design (CCD) was combined with response surface methodology (RSM) and was used for optimization of experimental parameters.

## MATERIAL AND METHODS

### Synthesis of magnetic cellulose nanoparticles

A known weight of cellulose was dispersed in 7 wt% NaOH+12 wt% urea aqueous solution precooled (-12 °C) to obtain a transparent 4 wt% of cellulose solution [11]. An aqueous solution of Fe ions with a molar ratio of Fe(III)/Fe(II) ~ 2 was prepared by dissolving the required amount of 5.46 g FeCl<sub>3</sub>.6 H<sub>2</sub>O, and 2 g FeCl<sub>2</sub>.4H<sub>2</sub>O in 50 ml distilled water with 10 ml of 1M HCl, then solution of NaOH (30%, w/v) was added drop-wise under constant stirring at 40 °C at a controlled pH10–11. The suspension was heated at 90 °C for 1 h under continuous stirring and nitrogen atmosphere. The quantity of suspension was added with constant stirring to cellulose solution. A homogeneous mixture was formed, which was then decantation and washed several times with water and dried at 70 °C in vacuum, which was designated as MCNGE.

### Preparation of amino-modified magnetic cellulose nanoparticles

Amino-modified magnetic cellulose nanoparticles were prepared through grafting of GMA [12] followed by reaction with EDA [13]. 2 g of MCNGE was dispersed in 30 ml of distilled water and heated at 40 °C under nitrogen atmosphere for 1 h. Then 5.92 ml of GMA was added, and the mixture was kept stirring for 15 min. Then 10 ml of a 0.1 mol/l CAN (dissolved in 1 mol/l HNO<sub>3</sub>) solution was added. Grafting was allowed to proceed for 2 h at 40 °C with continuous stirring and under nitrogen atmosphere. The product obtained was filtered off and then washed by distilled water several times, finally dried at room temperature, which was designated as MCNGEG. 1 g of product obtained in the previous step was suspended in 3 ml of ethylenediamine dissolved in 12 ml DMF. Then mixture was refluxed at 75–80 °C for 72 h on an oil bath, which was designated as MCNGE.

### Characterization methods

The FT-IR spectra displayed in this study were recorded on a spectrophotometer model 8400S (SHIMADZU, Japan) between 4000 and 500 cm<sup>-1</sup>, with a resolution of 2 Cm<sup>-1</sup>. XRD model XPertPROMPD (PAN alytical, Netherland) was used to investigate the crystal structure of the samples obtained. Scanning electron microscopy (SEM) was used to investigate the surface morphology of the developed bimetallic particles using the SEM model

EM3200 (KYKY, China). The residual concentration of Pt(IV) measurements were made on a Shimadzu ICP–OES Sequential plasma spectrometer model ICPS-7000 (Japan).

### Process variables and experimental design

Four process variables, viz. initial Pt(IV) concentration (C<sub>0</sub>), initial pH of the solution (pH), the operating temperature (T) and dose of adsorption (D) were selected to investigate their influence on adsorption of the Pt(IV) from aqueous solution by the modified magnetic cellulose nanoparticles.

Response surface methodology (RSM) contains a group of empirical techniques devoted to the valuation of relationship existing between controlled experimental factors and searching optimum conditions of variables to predict responses. RSM has been applied successfully in research areas and scientific such as chemistry, physics, biology, biochemistry, etc. [14].

In one variable at a time (OVAT) response is optimized by varying a single factor whereas keeping all other factors constant at a specific set of condition. This method is time consuming and does not provide the correct consideration of quantitative interactions between various factors. The main advantages of RSM are employed to know interaction among various factors and the reduced number of experiments.

The CCD is an effective design used for sequential experimentation and provides suitable amount of information for testing the goodness of fit and does not need unusually large number of design points there by reducing the overall cost associated with the experiment [15].

The behavior of system is described by the mathematical second degree polynomial model Eq. (1) [16-20]:

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1}^n \sum_{j=1}^n \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where  $Y$  is the predicted response  $x_i, x_j, \dots, x_n$  are the input variables, which affect the response  $Y$ ,  $x_i^2, x_j^2, \dots, x_n^2$  are the square effects,  $x_i x_j, x_i x_n$  and  $x_j x_n$  are the interaction effects,  $\beta_0$  is the intercept term,  $\beta_i (i=1, 2, \dots, n)$  is the linear effect,  $\beta_{ii} (i=1, 2, \dots, n)$  is the squared effect,  $\beta_{ij} (i=1, 2, \dots, n; j=1, 2, \dots, n)$  is the interaction effect and  $\varepsilon$  is a random error.

The central composite design (CCD) is the most frequently used approach of RSM in the experimental design. In the basis of the half-fraction factorial design 22 experiments were designed.

range of variables was decided on the basis of literature reports for adsorption of platinum [21-23].

The selected variables with their limits, notations and units are given in Table 1. The experimental design matrix that is contained 22 sets of experimental

conditions and the related responses (% adsorption) is shown in Table 1.

**TABLE 1.** Experimental factors and levels in the central composite design.

Factors	levels			star point	
	low (-1)	Central(0)	High (+1)	- $\alpha$	+ $\alpha$
(X <sub>1</sub> ) pH	2	3.5	5	0.5	6.5
(X <sub>2</sub> ) adsorbent dosage(g)	0.03	0.04	0.06	0.01	0.07
(X <sub>3</sub> ) concentration (ppm)	15	25	35	5	45
(X <sub>4</sub> ) Temperature(°C)	34	42	50	26	58

Runs	X1	X2	X3	X4	Adsorption (%)
1	2	0.06	35	50	88.33
2	5	0.03	15	50	86.86
3	2	0.06	15	50	90.41
4	3.5	0.04	25	42	93.2
5	5	0.06	15	34	86.3
6	5	0.03	35	50	85.34
7	5	0.06	35	34	85.9
8	2	0.03	35	34	91.1
9	3.5	0.04	25	42	93.52
10	3.5	0.04	25	42	93.9
11	3.5	0.04	25	42	92.16
12	2	0.03	15	34	92.2
13	0.5	0.04	25	42	85.9
14	3.5	0.04	25	26	88.9
15	3.5	0.04	25	58	93.9
16	3.5	0.04	5	42	91.83
17	3.5	0.04	25	42	93.29
18	3.5	0.07	25	42	94.14
19	3.5	0.01	25	42	90.3
20	3.5	0.04	45	42	89.5
21	3.5	0.04	25	42	93.84
22	6.5	0.04	25	42	80.07

### Batch adsorption

Batch adsorption were directed to study the effect of the four independent variables on adsorption of the Pt(IV) from aqueous solution by MCNGE and means of central composite design (CCD). For adsorption experiments, a known amount of the MCNGE (0.03-0.06 g) was added to 25 ml of solution initially containing (15-35 mg/l) of Pt(IV). Stock solution of Pt (IV) (1000 ppm) was prepared by dissolving hexachloroplatinic acid hexahydrate in distilled water and the pH was adjusted

(2-5) precisely using dilute hydrochloric acid or sodium hydroxide. The contents were agitated at 150 rpm in a water bath shaker for 60 min at different temperatures (34-50°C) to attain equilibrium. Based on our preliminary studies and experiments, the contact time and other conditions were selected. The equilibrated samples were taken out and the aqueous phase was separated from adsorbent (MCNGE). The residual concentration of Pt(IV) in the solution phase was then determined simultaneously by ICP-OES. Removal efficiency

expressed as percent adsorption of Pt(IV) ion was determined using the following equation [24].

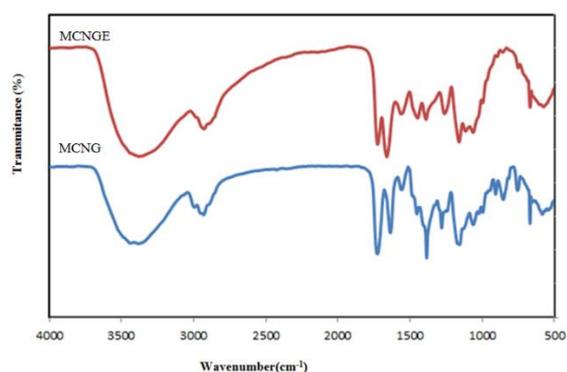
$$\text{Adsorption\%} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

where  $C_0$  and  $C_e$  (both in mg/l) are the initial and the equilibrium concentrations of Pt(IV) respectively.

## RESULTS AND DISCUSSION

### Characterization of the adsorbent

Figure 1 shows the FT-IR spectra of MCNGEG and MCNGE. The peaks at 660 corresponds to Fe-O binding. The spectrum of MCNGEG shows peaks at 1726  $\text{cm}^{-1}$  (strong, C=O ester stretching), and at 852  $\text{cm}^{-1}$  and 906  $\text{cm}^{-1}$  (C-O-C epoxide ring stretching). The spectrum of MCNGE due to reaction of group epoxide with ethylenediamine disappearance of the epoxide peaks. The stretching vibrations of the  $\text{NH}_2$  groups appear at 3250–3450  $\text{cm}^{-1}$  but where,  $\nu_{\text{NH}}$  can not be detected due to its overlapping with the  $\nu_{\text{OH}}$  band [25-27].



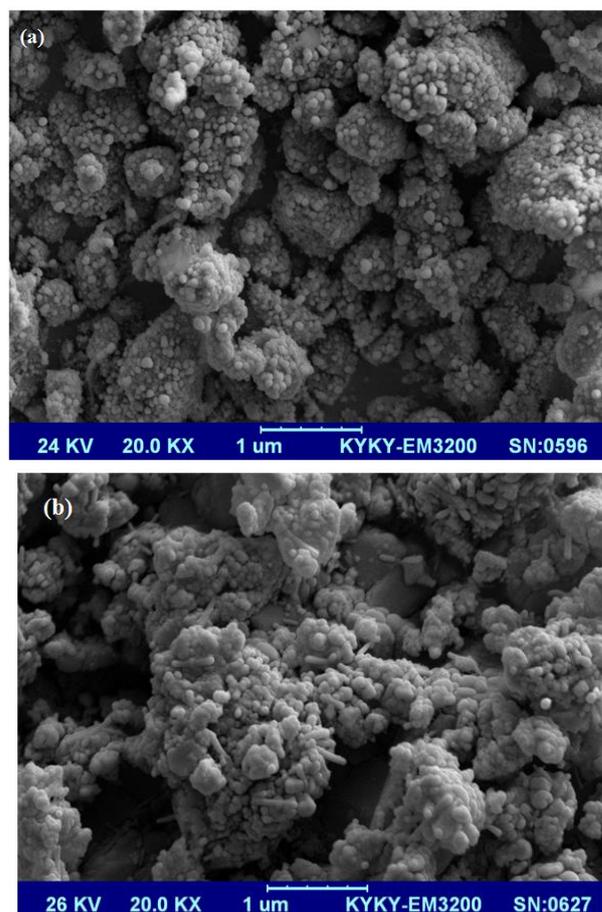
**Figure 1.** FT-IR of GMA-functionalized magnetic cellulose nanoparticles before (MCNGEG) and after (MCNGE) reaction with ethylenediamine

The SEM micrograph of magnetic nanoparticles  $\text{Fe}_3\text{O}_4$  and MCNGE displayed in Figure 2. The SEM analysis of the products provides information on the morphology and size of them. It can be seen from Figure 2a that the magnetic particles have a particle shape with diameter distribution from 36 nm. The  $\text{Fe}_3\text{O}_4$  particles are adequately coated and have maintained their spherical morphology (Figure 2b).

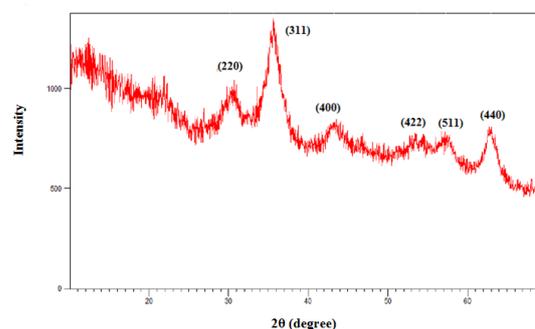
Figure 3 shows the XRD patterns for MCNGE. Six characteristic peaks for  $\text{Fe}_3\text{O}_4$  marked by their indices ((220), (311), (400), (422), (511), (440)) were observed in MCNGE. The observed broadness and lower intensity of that line in compare with pure  $\text{Fe}_3\text{O}_4$  indicates the lower degree of crystallinity of imbedded  $\text{Fe}_3\text{O}_4$  particles in cellulose.

### Data analysis by response surface methodology

Effect of process variables namely adsorbent dose, initial concentration, pH and temperature and was investigated using central composite design (CCD). Regression



**Figure 2.** Micrographs of scanning electron microscope, of  $\text{Fe}_3\text{O}_4$  (a), MCNGE (b)



**Figure 3.** XRD patterns for MCNGE

analysis in coded terms of the experimental parameters yielded the following regression equation for % adsorption of platinum.

$$\begin{aligned}
Y = & +88.08315 - 1.68795 (\text{pH}) - 308.99653 \\
& (\text{Dose}) + 0.44751 (\text{concentration}) + 0.35387 (\text{T}) \\
& + 80.88889 (\text{pH} \times \text{Dose}) + 0.010500 (\text{pH} \times \\
& \text{concentration}) + 0.12750 (\text{pH} \times \text{T}) + 0.11667 (\text{Dose} \times \\
& \text{concentration}) + 6.22917 (\text{Dose} \times \text{T}) - 3.28125\text{E-}003 \\
& (\text{concentration} \times \text{T}) - 1.22017(\text{pH})^2 - 1940.62500 (\text{Dose})^2 - 8.25391\text{E-}003 \\
& (\text{concentration})^2 - 0.010026 (\text{T})^2
\end{aligned} \quad (3)$$

The statistical significance of the response surface model in the form of analysis of variance (ANOVA) is often performed to evaluate the importance of the model [28] showed in (Table 2). In order Model Eq. (3) was used to evaluate the influence of the process variables on the adsorption of Pt(IV) by magnetic cellulose nanoparticles modified with ethylenediamine. The results of the quadratic regression model indicated that the quadratic model was highly significant with F-value ( $F_{\text{model}} = 55.09$ ) and value of  $\text{prob} > F (< 0.0001)$ . The quality of the fit model was checked by the coefficient value ( $R^2$ , adequate precision and adjusted- $R^2$ ). In this case  $R^2 = 0.9923$  indicated that only 0.67% of the total variable was not explained by the model. Adequate precision is the signal to noise ratio that compares the range of the predicted values at the design points to the average prediction error and a ratio greater than 4 is desirable [29]. Here, it is equal to 26.94. Adjusted- $R^2 = 0.9743$  is also high, displaying a high significance of the model.

The influence of four different variables on the response factor, percent adsorption of Pt(IV) is shown in the 3D response surface plots (Figure 4a–f).

The interactive behavior of two independent variables was shown in three-dimensional (3D) response surface plots. Since, the quadratic model in this work has four independent variables, two independent variables varying within their experimental ranges and two variables were held constant at their center level for each plot.

Figure 4a depicts the simultaneous effect of solution pH and temperature at constant initial concentration of Pt (29 mg/l) and adsorbent dosage (0.05g) on the percent adsorption of Pt(IV) from aqueous solution by MCNGE. The percent adsorption of Pt(IV) in solution declines with increasing pH within their respective experimental ranges. At low pH, platinum is usually present in solution

in its most stable form, i.e., Pt(IV) and can stable complexes form especially with amino group chelation sites of MCNGE, due to its characteristics as soft acid [30].

Figure 4b depicts percent adsorption of Pt(IV) by MCNGE as a function of the of pH and initial concentration of Pt(IV) in solution at constant temperature (45.5°C) and adsorbent dosage (0.05g). It was observed that percent adsorption of Pt(IV) declines with increasing pH and initial Pt-concentration. With raising the concentration Pt(IV), unsaturated sites become saturated and there will be very few unsaturated sites available on surface of the MCNGE. Thus some adsorbate was not able to be adsorbed.

Figure 4c The connection between initial solution pH and adsorbent dosage shows that with decrease of adsorbent dosage percent adsorption of Pt(IV) in aqueous solution by the MCNGE declines. At higher adsorbent dosage, the number of active sites was increased.

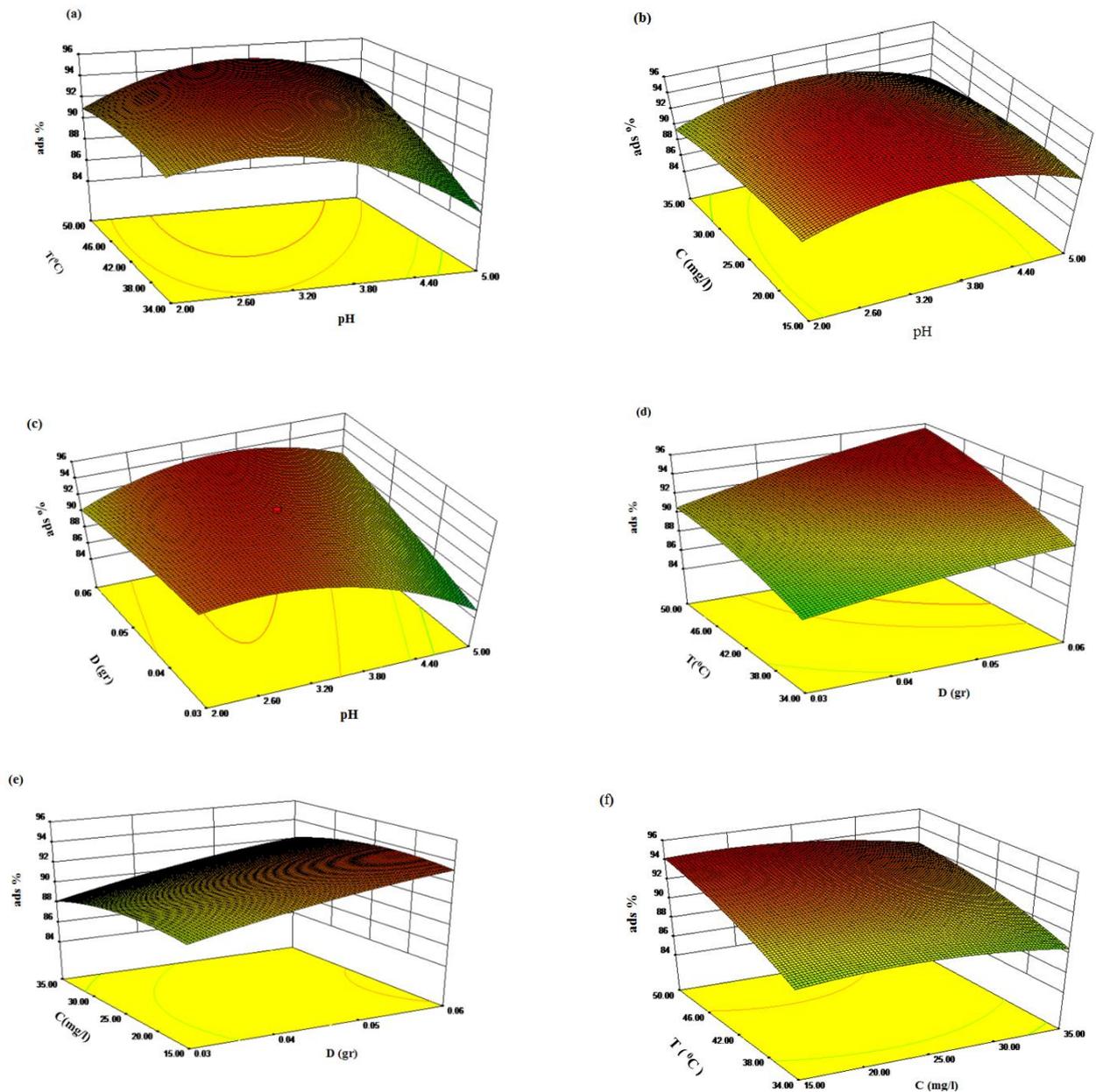
the simultaneous effect of temperature and adsorbent dosage on percent adsorption of Pt(IV) at constant pH (3.5) and initial Pt-ion concentration (29mg/l) in Figure 4d depicts that the percent adsorption of Pt(IV) increases with temperature and dose of the adsorbent.

The effect of the adsorbent dosage and Pt concentration on percent adsorption process is shown in Figure 4e It may be seen that the percent adsorption of ion Pt(IV) increases with dose of the adsorbent, while declines with increase in initial Pt-concentration.

The interactive effect of temperature and initial concentration of Pt-ions in solution on the percent adsorption process by MCNGE at constant solution pH (3.5) and adsorbent dosage (0.05 g) is shown in Figure 4f

### Optimization of adsorption of Pt(IV)

Optimization of the independent process variables to maximize the adsorption of Pt(IV) in aqueous solution by ethylenediamine-modified magnetic cellulose nanoparticles was performed using the quadratic model within the studied experimental range. The optimization modeling suggested the optimum amounts of the selected four independent process variables as, the initial Pt(IV) concentration 22 mg/l, temperature 46°C, initial solution pH 2.5, and the adsorbent dose 0.05g, to achieve the maximum adsorption capacity (19.45 mg/g) under the optimum conditions.



**Figure 4.** The response surface plot showing effect of temperature and pH (a), Pt(IV) concentration and pH (b), adsorbent dosage and pH (c), temperature and adsorbent dosage (d), Pt(IV) concentration and adsorbent dosage (e), temperature and Pt(IV) concentration (d)

## CONCLUSION

The magnetic cellulose nanoparticles have been prepared, and then modified by grafting of glycidylmethacrylate and reaction with amino groups (ethylenediamine) and is used for the adsorption of Pt(IV) from aqueous solution. Percent adsorption of Pt(IV) by MCNGE was studied as a

function of temperature, solution pH, initial concentration of Pt(IV) and dose of adsorbent. The central composite design (CCD) was used for optimization of adsorption Pt(IV) onto the MCNGE. The optimum values of the variables was determined namely

**Table 2.** Analysis of variance (ANOVA) for CCD

Source of variation	Sum of square	Degree of freedom	Mean square	F-value	P-value	
Block	0.29	1	0.29			
Model	289.24	14	20.66	55.09	<0.0001	significant
X <sub>1</sub>	16.99	1	16.99	45.32	0.0005	
X <sub>2</sub>	7.37	1	7.37	19.66	0.0044	
X <sub>3</sub>	5.95	1	5.95	15.88	0.0072	
X <sub>4</sub>	12.5	1	12.5	33.33	0.0012	
X <sub>1</sub> X <sub>2</sub>	13.25	1	13.25	35.33	0.001	
X <sub>1</sub> X <sub>3</sub>	0.2	1	0.2	0.35	0.4943	
X <sub>1</sub> X <sub>4</sub>	9.36	1	9.36	24.97	0.0025	
X <sub>2</sub> X <sub>3</sub>	0.03	1	0.03	6.53E-0.03	0.9382	
X <sub>2</sub> X <sub>4</sub>	2.24	1	2.24	5.96	0.0504	
X <sub>3</sub> X <sub>4</sub>	0.55	1	0.55	1.47	0.2709	
X <sub>1</sub> <sup>2</sup>	197.9	1	197.9	527.72	<0.0001	
X <sub>2</sub> <sup>2</sup>	5.01	1	5.01	13.35	0.0107	
X <sub>3</sub> <sup>2</sup>	17.89	1	17.89	47.7	0.0005	
X <sub>4</sub> <sup>2</sup>	10.81	1	10.81	28.83	0.0017	
Lack of fit	0.42	2	0.21	0.47	0.658	not significant
Pure error	1.83	4	0.46			

2.5 for initial pH of the solution, 22mg/l for the initial Pt(IV) concentration, 46°C for reactor temperature and 0.05 g for the adsorbent dose. The statistical data indicate that the Langmuir isotherm equations is the best fit one. The maximum adsorption capacity under the optimum conditions of temperature, pH and dose adsorbent found to be 19.45 mg/g.

The present developed adsorbent (MCNGE) is suitable for adsorption of Pt(IV) because of large surface area, high surface reactivity, selective adsorption with separation ease of phase and have good adsorbent efficiency for repeated use. The product is considered as green chemistry with perfect positive environmental impact. The adsorption is known an energy saving process

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**Persian Abstract**

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چکیده

در این مقاله ویژگی‌های جذب پلاتین بر روی نانو ذرات مغناطیسی سلولز اصلاح شده با اتیلن دی آمین مورد بررسی قرار گرفت. ویژگی‌های جاذب سنتز شده توسط تکنیک‌های FT-IR، XRD و SEM مورد بررسی قرار گرفت. بهینه سازی پارامترهای مؤثر بر آزمایش از جمله: غلظت اولیه پلاتین (۳۵-۱۵ mg/l)، دما (۳۴-۵۰ °C)، pH محلول (۲-۵) و میزان جاذب (۰.۰۳-۰.۰۶ gr) توسط طراحی مرکب مرکزی (CCD) و روش سطح پاسخ انجام شد. جهت ارزیابی از مدل اهمیت متغیرهای مستقل و اثرهای متقابل آنها بر روی هم در فرایند جذب پلاتین در محلول آبی از جدول آنالیز واریانس استفاده شد. نتایجی از مدل نشان داد که مدل در نظر گرفته شده با میزانی از (F-value = ۵۵.۰۹) و ( $\text{prob} > F < 0.001$ ) اهمیت می‌باشد. شرایط بهینه جذب عبارت است از: ۲۵ pH، دما برابر با ۴۶ °C، میزان جاذب برابر با ۰/۰۵ گرم، غلظت اولیه پلاتین برابر با ۲۲ mg/l. بیشترین ظرفیت جذبی از جاذب برای جذب Pt(IV) برابر با ۱۹/۴۵ mg/g بدست آمد.

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