Desalination and Water Treatment
www.deswater.com

doi:10.5004/dwt.2017.20835

79 (2017) 338–346
June

Application of catalytic nanopolymers for the removal of Bisphenol A from aqueous solutions: assessed by three statistical modeling strategies

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Received 12 October 2016; Accepted 16 April 2017

ABSTRACT

This study reports a new strategy for stabilizing palladized iron (Fe-Pd) nanoparticles with sodium carboxymethyl cellulose (CMC) as a stabilizer for the removal of Bisphenol A (BPA) from aqueous solutions. Transmission electron microscopy (TEM) analyses indicated that the CMC-stabilized nanoparticle with a diameter 20 nm is highly dispersed in water. At an optimum dosage of 0.17 g/l, the Fe-Pd bimetallic nanoparticle was able to remove 94% of BPA (C_0 = 0.75 mg/l) in 70 min, suggesting that presence of Pd could significantly enhance removal of BPA. It was found that the optimum pH, contact time, and BPA concentration for efficient removal of BPA concentrations from aqueous solutions were 7, 70 min, and 0.75 mg/l, respectively. It was concluded that Fe-Pd bimetallic nanoparticle is an efficient adsorbent for BPA removal from aqueous solutions.

Keywords: Bisphenol A (BPA); Catalytic reduction; Carboxymethylcellulose sodium (CMC); Endocrine disrupting chemicals (EDCs); Nanopolymer; Stabilizer

1. Introduction

Bisphenol A (BPA) is a chemical monomer with high-volume production in industries. It is a suspected endocrine disruptor (ED), which is widely applied in the production of epoxy and phenol resins, flame retardants, polyesters, polyacrylates, polysulfone, and polycarbonate (PC) plastics [1]. BPA has been shown to leach from food and beverage containers, some dental sealants and composites under normal conditions of use [2]. Final products included adhesives, protective coatings, powder paints, automotive lenses, protective window glazing, building materials, compact disks, optical lenses, thermal paper etc. [3]. Several studies have revealed that BPA could be released and migrated into the environment and natural water resources from the paper mill, agricultural and industrial effluents, natural degradation of polycarbonate plastics, landfill leachates, storm water, wastewaters of industries producing BPA, etc. [4]. Along with advantages and widespread application of BPA, this compound is very harmful to human health, wildlife, and ecosystem [1]. Numerous toxicological and biochemical studies have confirmed that BPA has estrogenic properties and also agonistic effect toward the estrogenic receptor. Furthermore, due to proven estrogenic effects in human and hormonal effects, it considered as a risk factor for breast cancer [5]. Various methods of chemical and physical treatment processes have been suggested to remove BPA from water resources involving ozonation, adsorption on activated carbon, nanofiltration [6], reverse

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osmosis, photocatalytic decomposition, electrochemicaloxidation, and biological processes [1]. Recently, researchers have shown an increased interest in utilizing nanoparticles for removing of a contaminant in water. In the past decades, zero-valent iron nanoparticle (nZVI) which has been applied to degrade a wide array of environmental contaminants was proven to be an effective reductant [7–10]. Coating the surface of nZVI with a small amount of second metal such as Pd, Pt, and Ni, which possesses catalytical activity, significantly enhances the degradation rate of contaminants relative to nZVI, alone [11].

He and Zhao [12] and He et al. [13] developed a new class of nanoparticles prepared by modifying nZVI with sodium carboxymethylcellulose (CMC), an environmental-friendly and low-cost starch or food-grade cellulose for degradation of chlorinated hydrocarbons in water. Fig. 1 shows the molecular structure of carboxymethyl cellulose (CMC) sodium salt.

According to the undesirable consequences of BPA, trying to find a solution in order to eliminate it from aquatic environments seems rational [1].

Response surface methodology (RSM) is a collection of statistical and mathematical technique to discover the relationship of an interesting response and several factors. RSM can be used to approximate the unknown relationship, estimate and test the effect of factors, and to find the optimum conditions of factors. To achieve these goals, a series of experiments or runs need to perform. Central composite design (CCD) is a popular design to specify levels and combinations of factors in experiments. CCD could capture curvature in the model of response with a small series of experiments. It consists of a full or fractional factorial design in that each factor has two levels, plus some center points, and two axial or star points for each factor. The axial or star points have the distance α from the central points and α chose in a way that design has some favorable properties. In this regard, Orthogonality, the desired property that guarantees estimation of each coefficient in RSM, is not affected by other coefficients [14–17].

Therefore, this study aims to experimentally investigate BPA removal from aqueous solutions using prepared CMC-stabilized Pd/Fe nanoparticles. In addition, the current study involves evaluation of the effects of pH, contact time, BPA initial concentration, and nanopolymer dosage on the BPA removal using response surface methodology with the central composite design. In this study, for the first time the application of a catalytic synthetized nanopolymer was implemented as a new method for treatment of aqueous solutions containing BPA and the optimum parameters were determined according to both experimental analysis and response surface method (RSM).

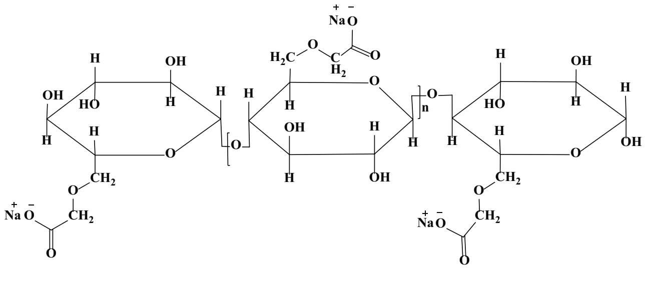


Fig. 1. The molecular structure of carboxymethyl cellulose (CMC) sodium salt [13].

2. Materials and methods

2.1. Materials

The physicochemical properties of the BPA (99% pure) that was supplied from Sigma-Aldrich (St. Louis, MO, USA) are presented in Table 1.

Potassium hexachloropalladate (purity >99%), and sodium carboxymethylcellulose (CMC, M.W. = 90,000) were obtained from Merck chemical company. Ferrous sulfate ($FeSO_4{\cdot}7H_2O$), and sodium borohydride ($NaBH_4$) were purchased from Sigma–Aldrich Co. Methanol (HPLC grade) was also used.

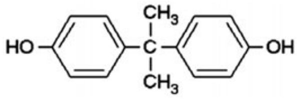
2.2. Preparation of nanopolymer

The nanoscale stabilized particles were prepared in according to Feng He et al. study [13]. Nanoscale stabilized Fe-Pd particles were synthesized by borohydride reduction using CMC as a stabilizer. Firstly, 70 mL of the 0.8 g/l CMC solution in water was purged with purified $N_2$for 15 min under stirring condition. Then, 10 ml of 5.56 g/l $FeSO_4{\cdot}7H_2O$ in water was added to the CMC solution. After this, the mixture was purged with N_2 for 15 min to complete the formation of the Fe-CMC complex. The Fe^{2+} ions were then reduced to Fe^0 by adding a certain amount of 2.2 g/l of sodium borohydride solution to the mixture. When visible gas evolution ceased (after 15 min), bimetallic particles were synthesized by loading trace amount of 60 ppm K_2PdCl_6 solutions onto the wet iron particles under stirring and nitrogen gas. The solution should be kept a few hours under nitrogen and vacuum to give a crystal-like solid.

2.3. Experimental design

To model and determine the optimum conditions toremove BPA, the experimental conditions were designed by

Table 1
The characteristic of BPA [1]

Common name (abbreviation)	Bisphenol A (BPA)
Molecular weight (g/mol)	228.1
Log K_{OW}	3.3
PKa	9.6–10.2
Chemical structure	
Water solubility	120–300 mg/l
Vapor pressure (25°C)	$5:3*10^{-9}$
Made by	Combining acetone and phenol
Chemical formula	2, 2-(4,4-Dihydroxydiphenyl) propane

central composite design (CCD) and analyzed by RSM, while contact time, nanopolymer dose (NPD), BPA concentration, and pH were the independent variables. The central composite design is a full 2 levels factorial by the four mentioned factors (16 points), plus two central points and eight axial points with alpha =1.4826 to achieve orthogonality, that was replicated two times (52 observations). Table 2 shows the coded levels and actual values of the variables.

The relationship between response Y and four independent variables: X_1, X_2, X_3 and X_4 could be approximated by aquadratic polynomial equation with two-way and three-way interactions as follows:

$$\begin{aligned} Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \\ + b_{44}X_4^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 \\ + b_{34}X_3X_4 + b_{123}X_1X_2X_3 + b_{124}X_1X_2X_4 + b_{134}X_1X_3X_4 + b_{234}X_2X_3X_4 \end{aligned} \quad (1)$$

where Y is the predicted response; b_0 is a constant; b_1, b_2, b_3, and b_4 are the linear coefficients; b_{11}, b_{22}, b_{33}, and b_{44} are the quadratic coefficients; and b_{12}, b_{13}, b_{14}, b_{23}, b_{24}, and b_{34} are coefficients of two-way interactions; and b_{123}, b_{124}, b_{134}, and b_{234} are the coefficients of three-way interactions.

In the present study, three following different strategies for modeling were used:

a) Multiple regression with all observations using ordinary least square method;
b) Multiple regression after removing outlier observations using ordinary least square; and
c) Multiple regression with all observations using the robust technique [18].

To find parsimonious and hence more reliable models, the backward variable selection was used in each of the modeling strategies. Then, the numerical optimization was used in the experimental range for proposed models by three modeling strategies and their candidates for optimum conditions estimated. Finally, these candidates for optimum values with one extra point between them were tested in the laboratory to check validations of calculated optimum values and to evaluate modeling strategies and select the final model.

2.4. Experiments

A stock solution was prepared by adding 10 mg BPA into a mixture of Milli-Q water (990 mL) and methanol (10 mL) and was kept in a refrigerator until use. The other diluted solutions were prepared from this stock solution. As can be seen in Table 2, in this study, the parameters carried out in chemical experiments were pH, BPA concentration, nano-polymer dosage (NPD) and contact time. A rotating shaker (200 rpm) was used for shaking the experimental flasks. The final sample solutions were extracted by glass syringes equipped with 0.45 µm syringe filters for analysis. At the same time, control experiments (without the addition of the nanoparticles) were carried out in parallel. H_2SO_4 0.1 M and NaOH 0.1 M were applied for the solution pH adjustment. Removal efficiency (%) of BPA by nanopolymers processes was calculated by the following equation:

$$RE(\%) = \frac{(C_i - C_a)}{C_i} \times 100 \quad (2)$$

where C_i and C_a (mg/l), are initial and the final BPA concentrations, respectively.

Table 2
The range and codification of the independent variables (X_i) used in the experimental design

Variables	Actual values of the coded levels				
	–1.4826	–1	0	1	1.4826
BPA concentration (X_1), mg/l	0.76	1	1.5	2	2.24
Nano-Polymer Dose (NPD) (X_2), g/l	0.03	0.05	0.1	0.15	0.17
pH (X_3)	1.55	3	6	9	10.45
Time (X_4), min	10.35	20	40	60	69.65

2.5. Analytical methods

The amount of BPA in the final samples was determined by a high-performance liquid chromatography system (Waters 515 HPLC pump) equipped with a UV detector (Waters 486 detector). The analysis was performed at 280 nm wavelength using a C_{18} reverse-phase column (Diameter: 5 µm, Length: 25 cm) with 20 µl sample injection. Methanol and deionized water were mixed at 65:35 (v:v) and used as mobile phase at a flow rate of 0.5 mL/min. The retention time of BPA was 13.3 min. The calibration curve equation ($R^2 = 0.9982$) for BPA detection was as follow:

$$\text{Peak area} = 27283\ \text{CBPA} + 1463.2 \quad (3)$$

where CBPA was the BPA concentration (within the range of 0–2.5 mg/l).

3. Results and discussion

3.1. Characterizations of synthesized nanoscale Fe–Pd particles

A transmission electron microscope (TEM) (Philips EM 208 S, 100 KV) was used to observe the particle characterizations: particle shape, size, and composition (Fig. 2). As can be seen in this figure, the stabilized Fe-Pd nanoparticles were highly separated and distinguished from each other in TEM image. This obtained result could be assumed due to the validated reaction between nanoparticle Fe-Pd and CMC. The TEM data indicated that the fresh stabilized nanoparticles have a size of about 20 nm. However, some researchers indicated that the size of CMC-stabilized nZVI could be manipulated by altering CMC molecular weights or CMC–Fe^{2+} molar ratio [19].

3.2. CCD analysis and modeling

Ordinary least square (OLS) with all observations (first strategy) gives the following model using backward variable selection:

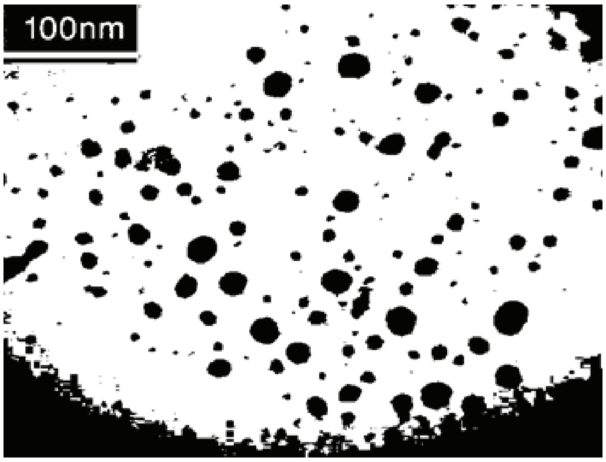


Fig. 2. TEM micrograph of the surface of the CMC-stabilized Fe–Pd bimetallic nanoparticles.

$$\begin{aligned}\mathit{BPA\ removal\ efficiency} = {} & 158 - 160 \times BPA + 52.9 \times BPA^2 \\ & - 0.342 \times pH^2 + 0.00278 \times Time^2 \\ & + 40.5 \times NPD \times pH - 16.7 \times BPA \\ & \times NPD \times pH \end{aligned} \tag{4}$$

After removing four outlier observations with studentized residual (residual divided by an estimate of its standard deviation) larger than 1.96, the ordinary least square with backward variable selection again (second strategy) was used that gives the following model:

$$\begin{aligned}\mathit{BPA\ removal\ efficiency} = {} & 157 - 136 \times BPA - 8.16 \times pH + 37.8 \times BPA^2 \\ & - 1221 \times NPD^2 + 0.0026 \times Time^2 + 230 \times BPA \\ & \times NPD + 3.05 \times BPA \times pH + 72.7 \times NPD \times pH \\ & - 47.6 \times BPA \times NPD \times pH \end{aligned} \tag{5}$$

Finally, the robust regression with all observations (third strategy) was used that gives the following model using backward variable selection:

$$\begin{aligned}\mathit{BPA\ removal\ efficiency} = {} & 153 - 146 \times BPA - 219 \times NPD + 40.1 \times BPA^2 \\ & - 369 \times NPD^2 - 0.737 \times pH^2 + 0.00178 \times Time^2 \\ & + 267 \times BPA \times NPD + 3.58 \times BPA \times pH + 80.9 \\ & \times NPD \times pH - 52.3 \times BPA \times NPD \times pH \end{aligned} \tag{6}$$

A numerical method was used to find optimum conditions for each candidate model. All combinations of predictors assessed in the experimental range and the combination of factors that yield maximum BPA removal efficiency were searched. Optimum nanopolymer dose, contact time and BPA concentration were 0.17 g/l, 70 min, and 0.75 mg/l, respectively. But different candidate models predict different optimum values for pH.

The first statistical modeling (OLS regression with all observations) proposed 7 as optimum values for pH, while, the second strategy (OLS regression after removing four outlier observations) proposed 8.5. However, the third statistical modeling (Robust regression using all observations) proposed 6.6 as the optimum value for pH.

To check the validity of models and to find the best modeling approach, four experiments were conducted using pH = 6.6, pH = 7, pH = 7.5, and pH = 8, when the other conditions were in their optimum values. The observed BPA removal efficiencies were 92%, 98%, 96%, and 94% for mentioned experiments, respectively.

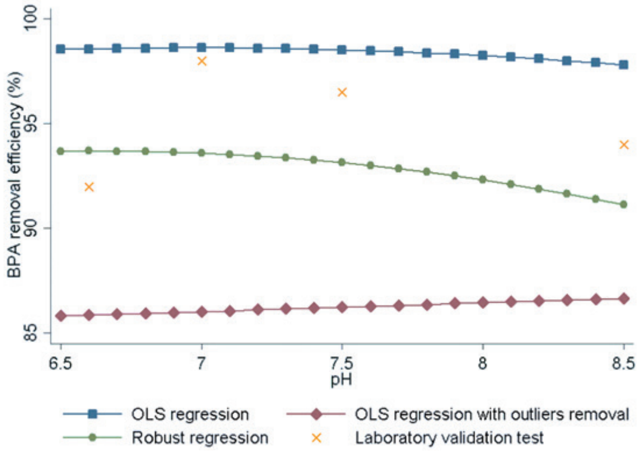


Fig. 3. Comparison of modeling methods and checking models using laboratory validation tests.

Fig. 3 shows these four laboratory validation tests and prediction of the different statistical modeling approach. In order to have formal criteria to compare models, the absolute prediction errors were calculated for these candidate models. The mean (standard deviation) of absolute errors were 3.26 (2.57) for the first modeling strategy, 8.93 (2.67) for the second modeling strategy, and 3.09 (1.13) for the third modeling strategy. Hence, the third modeling strategy (robust regression) identified as the best and used as the final model. The complete specification of the final model presented in Table 3. The Pareto chart and normal probability plot of coefficients in the final model presented in Fig. 4. The three-dimensional surface plot and two-dimensional contour plot of the final model presented in Figs. 5 and 6, respectively. The optimum conditions for BPA removal efficiency that confirmed using laboratory validation experiment show in Table 4.

3.3. Effect of nanopolymer dosage (NPD)

In order to determine optimal NPD, different doses ranging from 0.03 to 0.17 g/l were evaluated. Fig. 7 presents the prediction of BPA removal efficacy vs. nanopolymer dose.

As can be seen, a nanopolymer dosage of 0.17 g/l had maximum efficiency for BPA removal (94%). In a recent study, on the aggregation of agitate-coated nanoparticles, researchers observed that electrostatics was the primary mechanism for particle stabilization/aggregation. Consequently, particle stabilization will be affected by the valence and concentration of cations [13]. Therefore, in this study, an optimum dose of 0.17 g/l of nanopolymer was determined.

3.4. Effect of pH

It was reported that solution pH could affect both syntheses, and thus, the properties of the Fe or Fe-Pd bime-

Table 3
The final regression model for BPA removal efficiency

Terms	Coefficients	Std. Error	T statistic	P-value	95%CI L	95%CI U
(Constant)	153.1768	4.55458	33.63	<0.001	143.9786	162.375
BPA	–145.7613	5.527823	–26.37	<0.001	–156.9249	–134.5976
NPD	–219.2477	46.69783	–4.70	<0.001	–313.5559	–124.9396
BPA^2	40.1166	1.628622	24.63	<0.001	36.82753	43.40566
NPD^2	–369.3859	171.9588	–2.15	0.038	–716.6637	–22.1081
pH^2	–0.7369001	0.0366754	–20.09	<0.001	–0.8109675	–0.6628327
Time^2	0.001778	0.0001691	10.52	<0.001	0.0014366	0.0021194
BPA × NPD	267.2598	22.09599	12.10	<0.001	222.636	311.8835
BPA × pH	3.583142	0.3018095	11.87	<0.001	2.973625	4.192658
NPD × pH	80.88849	4.59137	17.62	<0.001	71.61603	90.16095
BPA × NPD × pH	–52.25237	3.04887	–17.14	<0.001	–58.40969	–46.09506

Lack of fit test was not significant with P-value = 0.152 (Goodness of fit accepted).

R-Squared = 99.37%; Adjusted R-Squared = 99.17%; Predicted R-Squared = 98.74%

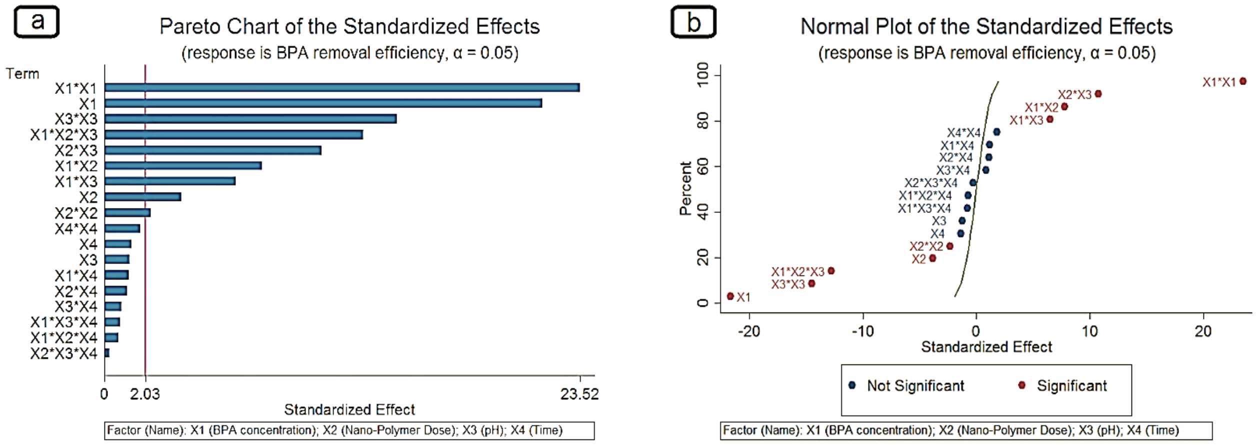


Fig 4. a) Pareto chart and b) normal probability plot; of coefficients in a robust regression model that involve linear, quadratic, two-way, and three-way interactions. The Standardized effects correspond to T-Statistics, so a big absolute value means a statistically significant effect.

tallic nanoparticles and the corrosion rate of the particles [20]. Moreover, the pKa values of BPA arein the range of 9.6–10.5. Therefore, it is implying that the ionization of BPA occurs at around pH 8–10 to form the phenolate and bisphenolate anions [21]. Hence, in this section, the pH impact on the BPA removal from the aqueous solutions was evaluated. Fig. 8 presents the prediction of BPA removal efficiency vs. pH.

The pH ranges of the samples were adjusted from 3 to 9 by using 1 M sodium hydroxide and sulfuric acid. Fig. 8 shows the BPA removal efficiency (RE) at pH 7 (optimum pH) was 94% and it was decreased with growing the pH. This observation is consistent with the results observed for p-NCB reductive dechlorination by non-stabilized Fe–Pd nanoparticles [20]. In the study conducted by Badruddoza, it was shown that the adsorption of BPA remained roughly unchanged at pH below about 7.0. While the BPA absorption increased in pH above about 7.0 and gradually decreased [1].

3.5. Effect of BPA concentration

The influence of initial BPA concentration on its RE was investigated under optimum specified conditions. Fig. 9 includes information about RE at different initial BPA concentrations (0–2.5 mg/l). It can be observed that with increment in the initial BPA concentration, its RE was decreased from about 94% in the initial concentration of 0.75 mg/l to approximately 56% in the BPA concentration of 1.7 mg/l and then RE was increased with increasing the concentration of BPA.

3.6. Effect of contact time

In this section, the contact time impacts were evaluated on the BPA removal from the aqueous solutions. Fig. 10 shows that 94% removal of BPA occurs in 70 min contact time and 85% of BPA removal occurs in 10 min contact time.

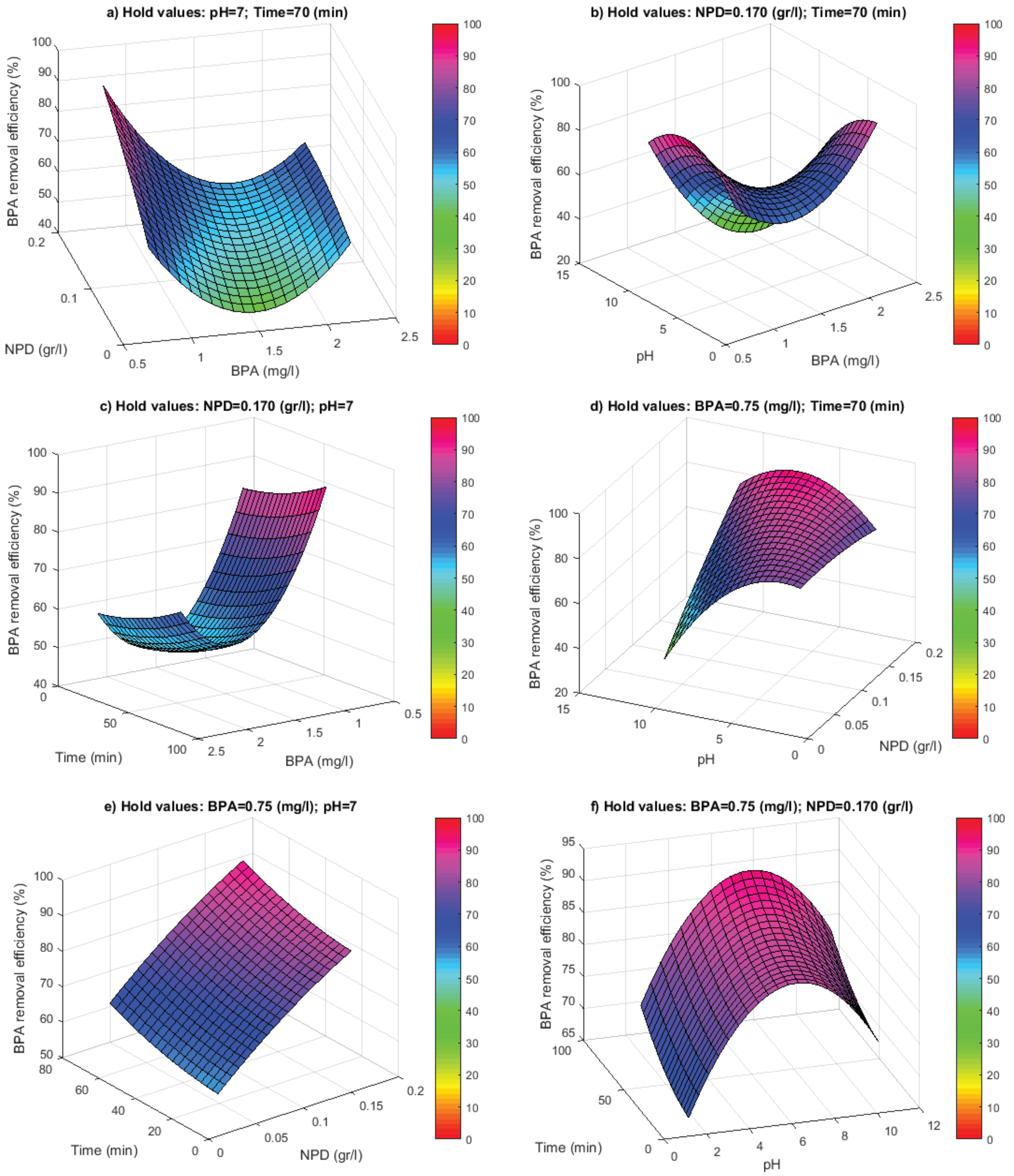


Fig. 5. Three-dimensional surface plot of predicted BPA removal efficiency in the final model vs. a) BPA concentration and nanopolymer dose; b) BPA concentration and pH; c) BPA concentration and time; d) Nanopolymer dose and pH; e) Nanopolymer dose and time; f) pH and time.

3.7. Process kinetic

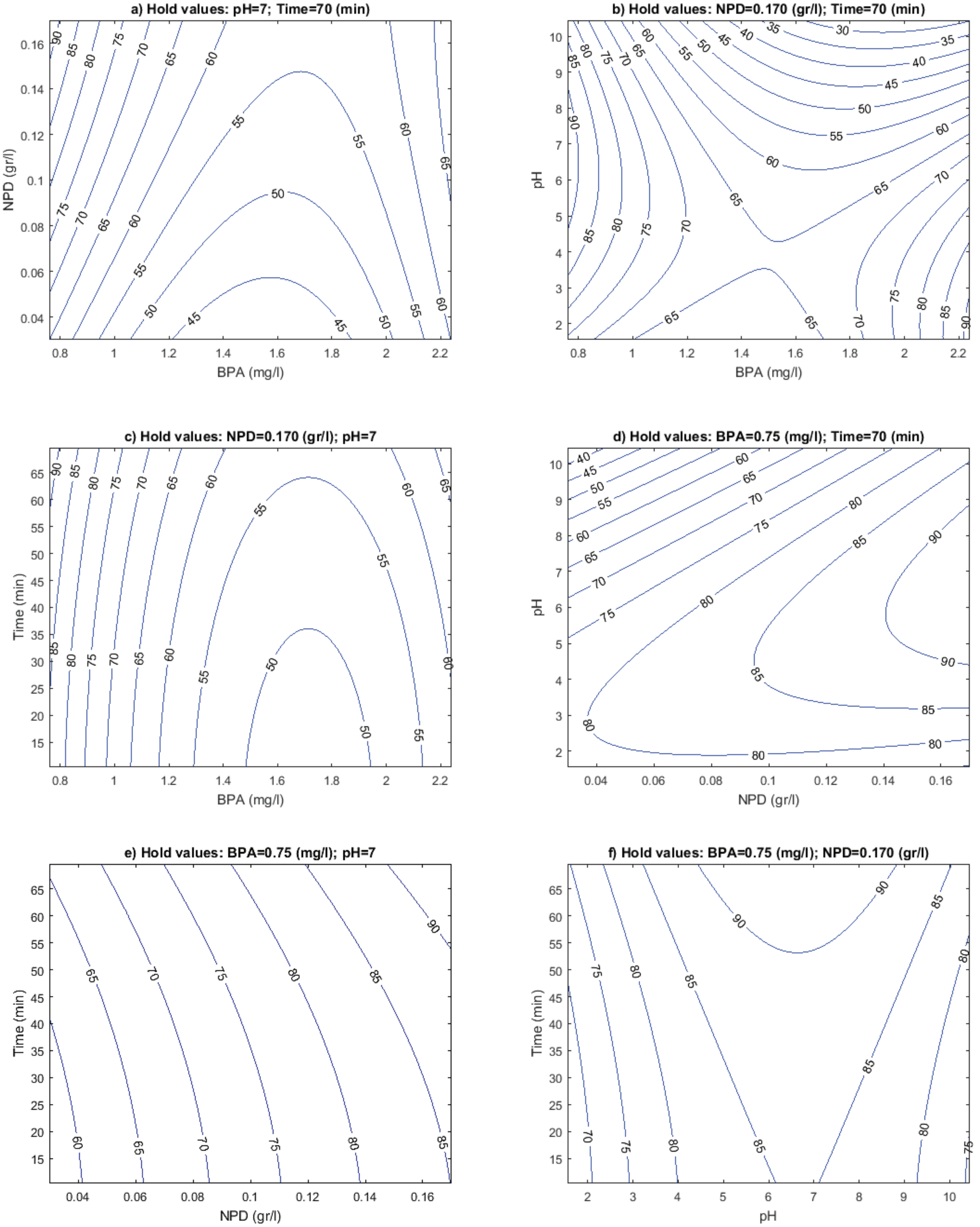
In this study, the kinetic process considering models, which allowed determination of the rate at which BPA removed from aqueous solutions, was investigated.

The used kinetic model is presented in the following equation:

$$R = K(C)^n \tag{7}$$

where R is the initial reaction rate; K is a constant; C is the initial concentration of BPA, and n is the degree of reaction [22].

The reaction kinetics of BPA in optimum concentration 0.75 mg/l was studied to determine the required time to


a) Hold values: pH=7; Time=70 (min)

b) Hold values: NPD=0.170 (gr/l); Time=70 (min)

c) Hold values: NPD=0.170 (gr/l); pH=7

d) Hold values: BPA=0.75 (mg/l); Time=70 (min)

e) Hold values: BPA=0.75 (mg/l); pH=7

f) Hold values: BPA=0.75 (mg/l); NPD=0.170 (gr/l)

Fig. 6. Two-dimensional contour plot of predicted BPA removal efficiency in the final model vs. a) BPA concentration and nanopolymer dose; b) BPA concentration and pH; c) BPA concentration and time; d) Nanopolymer dose and pH; e) Nanopolymer dose and time; f) pH and time.

Table 4
Predicted optimum conditions for BPA removal efficiency that confirmed using laboratory validation experiment

pH	7
Contact time (min)	70
NPD (g/l)	0.17
BPA concentration (mg/l)	0.75

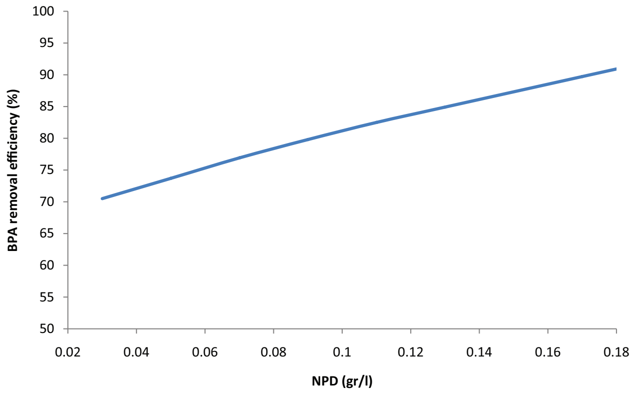


Fig. 7. Predicted BPA removal efficiency using the final model vs. NPD(BPA = 0.75 (mg/l), pH = 7, and contact time = 70 min).

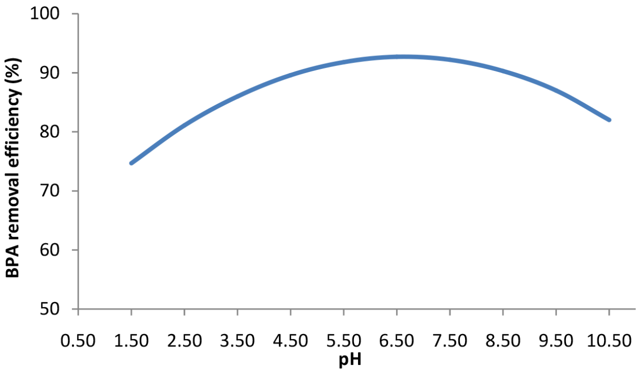


Fig. 8. Predicted BPA removal efficiency using the final model vs. pH(BPA = 0.75 (mg/l), NPD = 0.170 (g/l), and Time = 70 min).

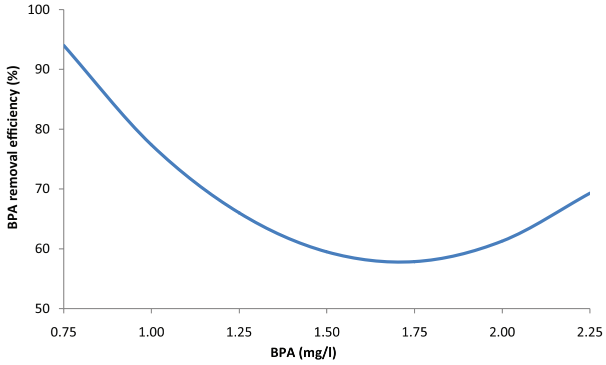


Fig. 9. Predicted BPA removal efficiency using the final model vs. BPA concentration (NPD = 0.170 (g/l), pH = 7, and Time = 70 min).

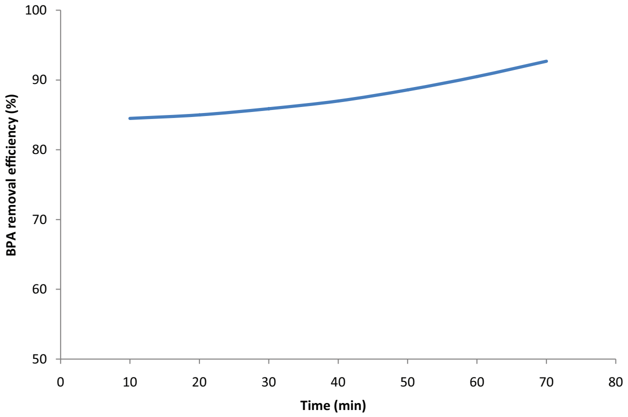


Fig. 10. Predicted BPA removal efficiency using the final model vs. time (BPA = 0.75 (mg/l), NPD = 0.170 (g/l), and pH = 7).

achieve equilibrium. In addition, the zero, first, second and third order kinetic models were studied. According to the obtained results,the best kinetic model for BPA removal from aqueous solutions confirmed of second order equations (k = 3.241 min^{-1}, R^2 = 0.946).

4. Conclusions

The catalytic removal of BPA using the CMC-stabilized Fe–Pd nanoparticles on different conditions was demonstrated in this study. The effects of experimental parameters on BPA degradation by stabilized bimetallic nanoparticles were also examined during the experiments. It was found that the optimum dose of nanopolymer was 0.17 g/l and the optimum pH, contact time, and BPA concentration for efficient removal of BPA were 7, 70 min and 0.75 mg/l, respectively. It is recommended that the CMC-stabilized Pd/Fe nanoparticles can be considered as a vital tool for the remediation of contamination from aqueous solutions. However, it is suggested that CMC-stabilized Pd/Fe nanoparticles would be compared to non-stabilized Pd/Fe nanoparticle ones in the future research to remove the chemical contaminant from the environment.

Acknowledgments

This paper is a result of an MSc approved Thesis (No: 393562). The Environment Research Center andVice-chancellor for Research at Isfahan University of Medical Sciences that financially supported it.

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