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Optimization by response surface method of dissolution of metallic zinc obtained from waste Zinc-Carbon batteries in nitric acid solutions

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ABSTRACT: In this study, the interactive effects of the process variables containing the concentration of nitric acid, the amount of zinc, and the reaction time on the efficiency of the dissolution of metallic zinc in nitric acid solutions were investigated by applying response surface methodology (RSM). It was found that the dissolution efficiency increased with an increase in the concentration of nitric acid, and the reaction time, and with a decrease in the amount of zinc. The multiple regression analysis to the experimental data was applied to observe the interactive effects of the experimental parameters. The second-order polynomial equation was obtained. The optimal experimental conditions were determined by using the optimization module in Design-Expert software, and different solution points were found.

KEYWORDS: Dissolution; Metallic zinc; Nitric acid; Response surface method; Waste battery

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RESUMEN: *Optimización mediante el método de superficie de respuesta de una disolución de zinc metálico obtenido a partir de la disolución en ácido nítrico de baterías de zinc-carbono al final de su ciclo de vida.* En este estudio, se investigaron los efectos interactivos de las variables del proceso: concentración de ácido nítrico, la cantidad de zinc disuelta en la batería y el tiempo de reacción sobre la eficiencia de la disolución de zinc metálico en soluciones de ácido nítrico aplicando la metodología de superficie de respuesta (RSM). Se encontró que la eficiencia de disolución aumentaba al aumentar la concentración de ácido nítrico y el tiempo de reacción y disminuía al hacerlo la cantidad de zinc. Se aplicó el análisis de regresión múltiple a los datos experimentales para observar los efectos interactivos de los parámetros experimentales. Se obtuvo la ecuación polinomial de segundo orden. Las condiciones experimentales óptimas se determinaron mediante el uso del módulo de optimización en el software Design-Expert, y se encontraron diferentes puntos de solución.

PALABRAS CLAVE: Ácido nítrico; Batería al final de ciclo de vida; Disolución; Método de superficie de respuesta; Zinc metálico

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1. INTRODUCTION

Zinc is one of the most important non-ferrous metals used in industry, and approximately 20-30% of total zinc production around the world is supplied from secondary sources containing zinc, such as zinc ash, zinc dross, brass smelting and waste batteries (Rabah and El-Sayed, 1995; Sahu *et al.*, 2004; Tsakiridis *et al.*, 2010). Because waste zinc carbon and alkaline zinc manganese dioxide batteries include substantially zinc and manganese, they can be utilized as secondary sources in the production of these metals. In the literature, various studies on the recovery of zinc and manganese in waste battery powders have been performed by applying hydrometallurgical route. The aqueous solutions of sulfuric acid (Shin *et al.*, 2007; Biswas *et al.*, 2016; Turhan Özdemir and Demirkıran, 2019a), hydrochloric acid (Baba *et al.*, 2009), ammonia (Senanayake *et al.*, 2010), ammonium chloride (Nogueira and Margarido, 2015), ammonium carbonate (Shin *et al.*, 2008), and sodium hydroxide (Shin *et al.*, 2009; Turhan Özdemir and Demirkıran, 2019a; Demirkıran and Turhan Özdemir, 2019) have been used as the leaching agents for the recovery of zinc and manganese from waste zinc carbon and alkaline zinc manganese dioxide batteries.

In the production of a zinc carbon battery, the cylindrical zinc can be used as the anode material. It is oxidized to zinc oxide via the oxidation-reduction reaction occurring during the use of battery. However, the entire metallic zinc is not oxidized to zinc oxide, and a waste zinc carbon battery may contain a significant amount of metallic zinc. As stated above, the most of studies in the literature are focused on the recovery of metals in waste battery powders. There is no information on the recovery of metallic zinc in waste zinc carbon batteries. Thus, the present study concerns about the evaluation of zinc metal in waste zinc carbon batteries by means of the statistical experimental design.

In the statistical experimental design, because the experimental parameters can be simultaneously varied, more information about the process can be obtained with minimum number of trials. Hence, the experimental design is a useful tool to see the interactions between two or more variables by reducing number of trials (Abazarpoor *et al.*, 2013; Chollom *et al.*, 2020). In the present study, response surface methodology (RSM) was used to optimize the dissolution efficiency of metallic zinc.

RSM is a combination of the statistical and mathematical methods that are useful for designing experiments, modelling, analyzing the effects of variables, and the optimization of engineering problems. In this technique, the main objective is to optimize the response surfaces influenced by various process parameters (Niaki *et al.*, 2015). This methodology has been widely adopted in the industries, such as drug and food industry, chemical and biological processes for the purpose of either producing high quality products or operating the process in a more economical manner and ensuring the process in a more stable and reliable way (Ghosh *et al.*, 2012; Sudamalla *et al.*, 2012; Ohale *et al.*, 2017; Yolmeh and Jafari, 2017). RSM has been also applied to optimize the recovery efficiency of metal values from waste battery powders (Ijadi Bajestani *et al.*, 2014; Shalchian *et al.*, 2015; Niu *et al.*, 2016; Tanong *et al.*, 2017; Turhan Özdemir and Demirkıran, 2019b).

In this study, the concentration of nitric acid, reaction time, and zinc amount were selected as the independent variables. The interactive effects of these variables on the dissolution efficiency of zinc were investigated, and a statistical model representing the relationship between the dissolved zinc and the independent variables was constructed.

2. MATERIALS AND METHODS

2.1. Material

After waste zinc-carbon batteries were collected, they were manually dismantled by using screwdriver, plier and metal-cutting scissors and the cylindrical metallic zinc can was separated from the other components of battery. The surface of zinc plate created from the cylindrical can was washed with distilled water and cleaned mechanically with abrasive paper to eliminate the contaminants originating from battery powder. Afterwards, zinc plate was chemically cleaned by diluted nitric acid solution and rinsed with distilled water, and it was dried at room temperature. The phase and chemical analyses of the cleaned zinc plate were performed by X-ray diffractometer (Rigaku RadB-DMAX II) and XRF spectrometer (Spectro Xcpus), respectively. The result of the chemical analysis presented that zinc plate is composed of 98.84% Zn, 0.63% Pb, and 0.53% other. The result of the XRD pattern concerning zinc plate is given in Fig. 1. After the cleaned zinc plate was cut by metal-cutting scissors and brought into square pieces with a size of 0.5x0.5 cm, these pieces were utilized as material in the dissolution experiments.

2.2. Method

The dissolution experiments of metallic zinc pieces were performed in a jacketed glass reactor. After 500 mL of nitric acid solution having known concentration was poured into the reactor and supplied a constant reaction temperature of 25 °C by circulatory water bath, a given amount of zinc pieces was added to this solution, and the reactor content was

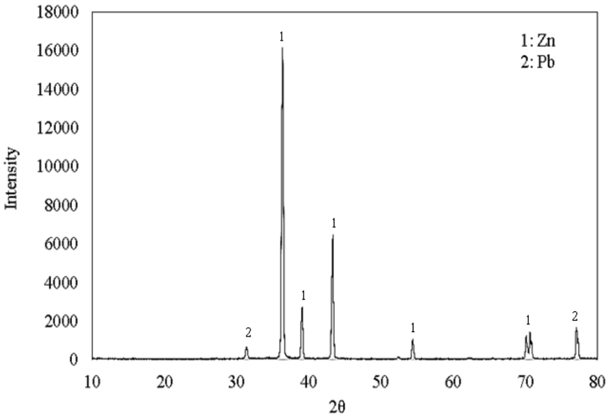

Figure 1. XRD analysis of the cleaned zinc plate obtained from waste zinc carbon battery.

mixed at stirring speed of 400 rpm by a mechanical stirrer during the dissolution process. At the end of the reaction time, the amounts of zinc ions in a sample of 3 mL taken form the solution in the reactor were detected complexometric titration. Titriplex III solution and puffer tablet were used as titrant solution and indicator, respectively. The amount of the dissolved zinc in the solution was calculated in percentage term as given in Eq. (1).

$$\% \text{ Dissolved zinc} = \left[\frac{\text{Zinc amount in the solution}}{\text{Zinc amount in metal pieces}}\right] \text{x } 100 \tag{1}$$

In the present work, CCD, which is one of the most commonly used methods of RSM, was employed to determine the effects of the independent variables on the response and to obtain the optimal response. Nitric acid concentration, zinc amount, and reaction time were selected as the independent variables each at two levels. The level of the independent variables and their experimental ranges are given in Table 1. The total number of experiments was determined according to the expression given in Eq. (2).

$$N = 2^n + 2n + m \tag{2}$$

In Eq. (2), N is the total number of experiments, n is the number of the independent variables, and m is the number of replicates. Since three different independent variables are selected, a 2^3 full factorial CCD with 6 axial points and 6 repetitions at the center point was utilized for RSM. Design-Expert Version 11.0.1.0 trial software was used to determine the total number of experiments, to analyze the experimental data, to estimate the regression equation, and to optimize the experimental conditions. The empirical relationship between the response and the independent variables was determined by applying the regression equation in Eq. (3), which includes the linear and cross effect of the variables.

Table 1. The independent variables and their levels

Independent variables	Sign	Level		
		-1	0	+1
Nitric acid concentration, M	X_1	0.15	0.325	0.50
Zinc amount, g	X_2	0.80	1.90	3.00
Reaction time, min	X_3	10	20	30

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{11}X_1^2 + a_{22}X_2^2 + a_{33}X_3^2 \tag{3}$$

In Eq. (3), Y denotes the response; a_0 is the model constant; a_1, a_2, and a_3 are the linear coefficients; a_{12}, a_{13}, and a_{23} are the interactive coefficients; a_{11}, a_{22}, and a_{33} are the quadratic coefficients. The analysis of variance (ANOVA) was applied to evaluate the lack of fit, the coefficient of determination (R^2), and the adequacy of the model. The interaction between the process variables was evaluated by using the three dimensional surface plots and the respective contour plots.

3. RESULTS AND DISCUSSION

Nitric acid is one of the strong mineral acids and is a strong oxidizing agent. Thus, it is a good dissolving agent for most of metals (Ahn *et al.*, 2011; Kurushkin, 2015). The chemical reaction for the dissolution of metallic zinc in nitric acid solutions can be simply written as in Eq. (4) or in Eq. (5).

$$Zn_{(s)} + 2H^+_{(aq)} \rightarrow Zn^{2+}_{(aq)} + H_{2(g)} \tag{4}$$

$$Zn_{(s)} + 2HNO_{3(aq)} \rightarrow Zn(NO_3)_{2(aq)} + H_{2(g)} \tag{5}$$

However, it has been expressed in the literature that the dissolution of metallic zinc in nitric acid solutions is quite complicated. Various researchers have stated that different reaction products may form at the end of the reaction occurred between nitric acid and zinc metal depending on the concentration of nitric acid. The following reactions for the dissolution process among zinc and nitric acid have been proposed in the literature (Khalil and EI-Manguch, 1987; Mihit *et al.*, 2007; Ahn *et al.*, 2011; Kurushkin, 2015).

$$4Zn_{(s)} + 10HNO_{3(aq)} \rightarrow 4Zn(NO_3)_{2(aq)} + NH_4NO_{3(aq)} + 3H_2O_{(l)} \tag{6}$$

$$4Zn_{(s)} + 10HNO_{3(aq)} \rightarrow 4Zn(NO_3)_{2(aq)} + N_2O_{(g)} + 5H_2O_{(l)} \tag{7}$$

$$3Zn_{(s)} + 8HNO_{3(aq)} \rightarrow 3Zn(NO_3)_{2(aq)} + 2NO_{(g)} + 4H_2O_{(l)} \tag{8}$$

The experimental program proposed by CCD and the experimental responses obtained from the tests are shown in Table 2. The experimental responses seen in this table display that the dissolution yields vary from 26% to 99.99%.

A second-order polynomial model given in Eq. (9) was obtained by applying multiple regression analysis to the experimental data. This model equation gives the empirical relationship between the

Table 2. The experimental program purposed by CCD and the responses obtained the tests

Exp. run	X_1	X_2	X_3	Experimental response (Zn%)	Predicted response by model (Zn%)
1	0.325	1.9	3.18	55.00	59.55
2	0.031	1.9	20	26.00	33.48
3	0.325	1.9	20	96.64	96.12
4	0.325	1.9	20	96.20	96.12
5	0.500	0.8	10	96.72	92.37
6	0.325	1.9	20	96.22	96.12
7	0.325	0.005	20	99.99	99.99
8	0.500	3.0	10	90.27	93.66
9	0.500	0.8	30	98.43	99.99
10	0.150	3.0	30	64.34	65.27
11	0.325	1.9	20	96.15	96.12
12	0.325	1.9	20	96.17	96.12
13	0.325	1.9	36.82	96.81	97.09
14	0.325	1.9	20	96.19	96.12
15	0.500	3.0	30	99.90	99.99
16	0.619	1.9	20	98.18	95.53
17	0.150	0.8	30	98.43	91.62
18	0.150	0.8	10	57.00	53.39
19	0.150	3.0	10	35.00	29.13
20	0.325	3.75	20	85.44	84.60

dissolved zinc (response) and the independent variables. The quadratic model in Eq. (9) shows that it involves one constant term, three linear terms, three quadratic terms, and three two-factor interactions.

$$Y = 96.12 + 18.45X_1 - 6.26X_2 + 11.16X_3 + 6.39X_1X_2 - 7.43X_1X_3 - 0.5212X_2X_3 - 11.18X_1^2 - 0.3503X_2^2 - 6.29X_3^2 \quad (9)$$

To test the statistical significance and adequacy of the model in Eq. (9), the analysis of variance (ANOVA) and *F*-test were conducted by using the Design Expert Software. The results obtained are listed in Table 3. The probability values (*P*-values) observed in Table 3 can be used as a tool to check the significance of each variable and their interactions.

An *F*-value of 44.19 for the model implies that the model is significant enough for the regression analysis between the response and the independent variables. A value of Prob > F less than 0.050 indicates that the related independent variable in the model has a significance on the dissolution efficiency of zinc. In Table 3, Prob > F value for the model represented the dissolution process indicates that the model is significant. Besides, it is seen from Table 3 that Prob > F values for the coded terms of X_1, X_2, X_3, X_1X_2, X_1X_3, X_1^2, and X_3^2 are less than 0.05. Thus, it can be expressed that the coded terms of X_1, X_2, X_3, X_1X_2, X_1X_3, X_1^2, and X_3^2 in Eq. (9) are statistically significant terms. The R^2 value for the response model in Eq. (9) is 0.987. This R^2 value indicates that there is a good agreement between the experimental responses and the predicted responses by model.

To see the agreement between the experimental and the predicted responses by the empirical model, a graph of the predicted responses versus the experimental responses was constructed in Fig. 2. It can be seen from Fig. 2 that there is a reasonable agreement between the experimental and the predicted responses.

The three dimensional response surfaces and the contour plots obtained from the quadratic model for zinc dissolution yield were drawn using the Design Expert Software. Figures 3-5 display the relationships between the responses and the experimental

Table 3. ANOVA result of the quadratic model for zinc response

Source	Coefficients	Sum of squares	DF	Mean square	F-Value	Prob.>F
a_0	96.12					
X_1	18.45	4647.83	1	4647.83	187.29	<0.0001
X_2	-6.26	535.78	1	535.78	21.59	0.0009
X_3	11.16	1701.24	1	1701.24	68.55	<0.0001
X_1X_2	6.39	326.53	1	326.53	13.16	0.0046
X_1X_3	-7.43	441.49	1	441.49	17.79	0.0018
X_2X_3	-0.5212	2.17	1	2.17	0.0876	0.7733
X_1^2	-11.18	1800.61	1	1800.61	72.56	<0.0001
X_2^2	-0.3503	1.77	1	1.77	0.0713	0.7949
X_3^2	-6.29	570.81	1	570.81	23.00	0.0007
Model		9868.87	9	1096.54	44.19	<0.0001
Residual		248.17	10	24.82		
Lack of fit		247.99	5	49.60	1419.67	<0.0001
Pure error		0.1747	5	0.0349		
Total		10117.03	19			

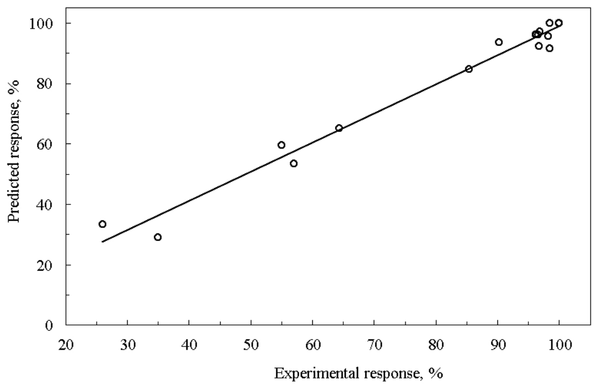

Figure 2. The graph of the experimental versus the predicted responses.

levels for each variable. These plots can be utilized to discover the effects of any of the two variables, while the other variable is kept constant at its center level. In the response surfaces, the clear peaks illustrate that the optimal conditions are exactly inside the design boundary and the optimum values drawn from these figures are in close agreement with those obtained by optimizing the regression equation. An analysis of Figs. 3-5 shows that the dissolution yield increases with the increasing nitric acid concentration and reaction time, and with the decreasing zinc amount.

The interactive effect of nitric acid concentration and zinc amount on the dissolution yield is shown in Fig. 3. It can be seen from this figure that the dissolution yield increases with the increasing nitric acid concentration and with the decreasing zinc amount.

Figure 4 displays the interactive relationship between the reaction time and nitric acid concentration on the dissolution of zinc. Figure. 4 indicates that the dissolution yield of zinc increases with an increase in reaction time and nitric acid concentration.

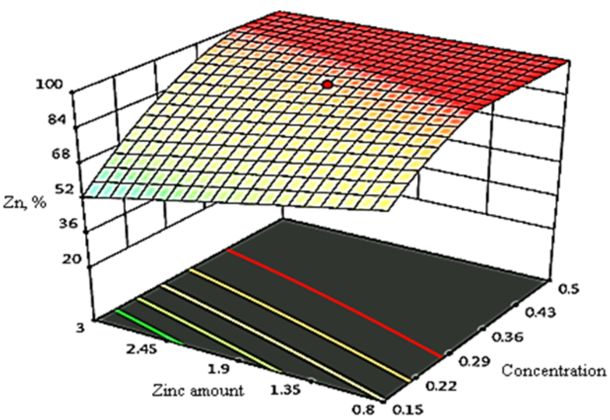

Figure 3. The interactive effect of the concentration of nitric acid and the amount of zinc on the dissolution yield.

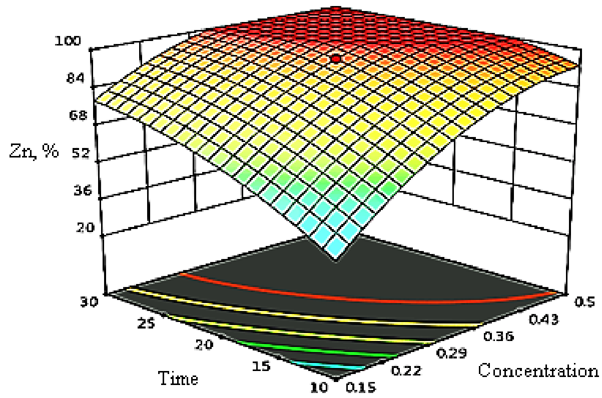

Figure 4. The interactive effect of the concentration of nitric acid and the reaction time on the dissolution yield.

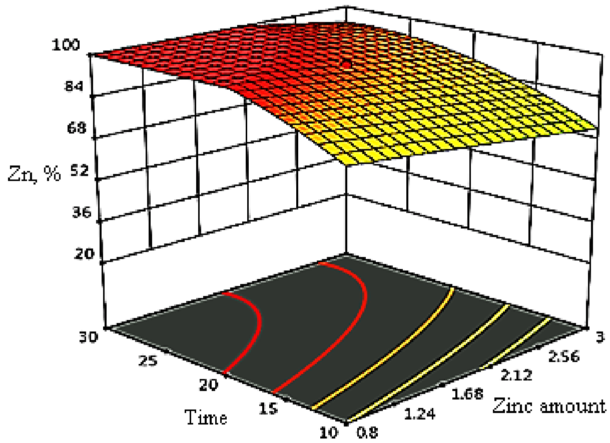

Figure 5. The interactive effect of the amount of zinc and the reaction time on the dissolution yield.

The interactive relationship given in Fig. 5 illustrates the effect of the reaction time and zinc amount on the dissolution of zinc. This figure indicates that the dissolution yield of zinc increases with an increase in reaction time and with a decrease in zinc amount.

The optimal experimental conditions were determined by using the optimization module in Design-Expert software, and different solution points were found. Among these optimal conditions, while the optimal values of nitric acid concentration, zinc amount, and reaction time were at 0.382 M, 2.468 g, and 13.6 min, respectively, the experimental dissolution efficiency of zinc was found to be 86%. The dissolution efficiency value predicted by model was 90.8% at the same experimental conditions. While the values of these three variables were at maximum values (0.5 M, 3 g, and 30 min), both the experimental dissolution efficiency and the dissolution efficiency predicted by model were determined to be 99.9%:

4. CONCLUSIONS

- In this work, the optimal dissolution conditions of metallic zinc obtained from waste zinc carbon batteries was examined in nitric acid solutions. The concentration of nitric acid, the reaction time and the amount of zinc were selected as the independent variables, and RSM was employed to optimize the values of these parameters. To see the interactive effects of the process variables, the multiple regression analysis to the experimental findings was performed, and a representative statistical model showing the relationship between the dissolved zinc and the independent variables was derived. The findings obtained showed that the dissolution process was positively affected with an increase in the concentration of nitric acid and the reaction time, and with a decrease in the amount of zinc. It was observed that the empirical model represented finely agreement between the experimental and the predicted values.
- The optimal experimental conditions were determined by using the optimization module in Design-Expert software, and different solution points were found. Among these optimal conditions, while the optimal values of the concentration of nitric acid, the amount of zinc, and the reaction time were at 0.382 M, 2.468 g, and 13.6 min, respectively, the experimental dissolution efficiency of zinc was found to be 86%.
- The dissolution efficiency value predicted by model was 90.8% at the same experimental conditions. While the values of these three variables were at maximum values (0.5 M, 3 g, and 30 min), both the experimental dissolution efficiency and the dissolution efficiency predicted by model were determined to be 99.9%.

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