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# Optimization of recovering cerium from the waste polishing powder using response surface methodology

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**Abstract:** Recovery of rare earth from the waste rare earth polishing powder is of great importance to improve the process economics. The current recovery methods result in the generation of large quantities of polluted discharge necessitating waste treatment systems. The present work attempts to extract cerium from rare earth slurry waste by thiourea reducing and hydrochloric acid leaching, towards which central composite technique of the response surface methodology is adopted to design the experiments and to optimize the process conditions to maximize the recovery rate. The effects of four major parameters such as temperature, duration of leaching, dosage of thiourea, and the concentration of HCl were assessed, and the optimal process conditions were identified. Analysis of variance (ANOVA) was utilized to identify the suitable model and to eliminate the insignificant model parameters. The optimized process conditions for maximizing the recovery rate are identified to be a leaching temperature of 90°C, duration of 150 min, thiourea dosage of 0.2 g/g, and HCl concentration of 3.5 mol/l. A recovery of 91.23% could be achieved and validated through repeat experimental runs at the optimized process conditions. The optimized process samples are characterized utilizing XRD to validate the recovery.

**Keywords:** cerium; leaching; recovery; thiourea; waste rare earth polishing powder.

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## 1 Introduction

Ceria-based polishing powder is one of most important rare earth products. As "the king of polishing powder," it has been widely used in polishing glass and silicon wafers because (i) its ceria particles are relatively soft so they avoid surface scratching and (ii) the slurry reacts chemically with glass, silicon, and silica surfaces dissolving protuberances and smoothing the surfaces [1, 2]. However, an enormous amount of abrasive materials is wasted after being used for polishing glass. The slurry for glass polishing is a mineral containing a high concentration of cerium, which is mixed with elements such as Fe, Al, and Si in the polishing process. The heterogeneous nature and the complexity of slurry waste make it difficult to extract cerium from the waste, but after being extracted at a low cost, cerium of a high purity has great economic benefit [3–5].

At present, the most common methods used for extracting cerium from the polishing powder waste are low-temperature sulfatizing roasting, alkali roasting, and froth flotation. While low-temperature sulfatizing roasting can obtain a good decomposition and recovery result, it needs high concentration sulfuric acid roasting at 1300°C [3, 6], which will be a threat to equipment, environment, and operating safety. Alkali roasting can only get low purity rare earth solid solution with low value, and it also has the disadvantages of tedious process and low decomposition efficiency. There are also some problems existing in froth flotation process: (1) the waste powder is too light to get effective collision with bubble; (2) the high surface area of the particle would consume much flotation reagent, which will destroy the flotation process and lead to the increased cost; and (3) the agglomeration could be caused easily between fine particles and the flotation will be not effective. So, it is very necessary to develop green, environment-friendly and economic process of recovering cerium from the waste polishing powder [7, 8].

Extracting cerium from rare earth polishing powder by thiourea reducing and hydrochloric acid leaching is explored in the present work. In order for this process to

be effective, the yield of cerium is desirable and hence a process optimization exercise to identify the optimum conditions is performed. In our previous studies, single-factor experiments show that the leaching temperature, leaching duration, thiourea dosage, and HCl concentration are major factors of the recovery yield. So, the process variables explore being these four parameters while the response variable, being the yield of cerium. A response surface methodology (RSM) is utilized, as it is one of the relevant multivariate techniques that have the capability to perform multivariant experimental design, statistical modeling, and process optimization. It is the most economical and convenient method for characterizing a complicated experimental process with minimum number of experiments [9–12]. The structural change of the powder due to the extraction process is investigated by means of X-ray diffraction (XRD).

# 2 Materials and methods

## 2.1 Materials

The rare earth polishing powder waster used in this experiment was supplied by a waste recovery company in Henan Province, China. The samples were dried at 100°C for 12 h and subjected to X-ray fluorescence (XRF) and XRD. The results are shown in Table 1 and Figure 1.

**Table 1:** The chemical composition of rare earth polishing powder waste (XRF).

| Elements | Ce | Si | La | Na | Ca | F | Al | Mg |
|---|---|---|---|---|---|---|---|---|
| Content (wt.%) | 29.43 | 17.37 | 4.06 | 5.79 | 5.24 | 3.57 | 2.22 | 1.40 |

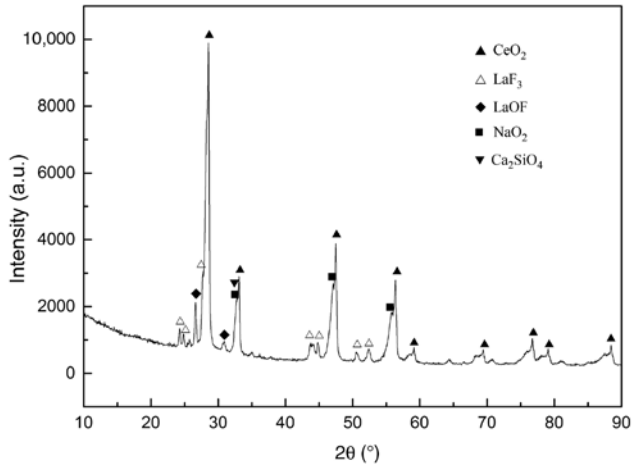


**Figure 1:** X-ray diffraction patterns of the raw materials.

As it seen in Figure 1, the main phases of raw materials are $CeO_2$, $LaF_3$, LaOF, $NaO_2$, and $Ca_2SiO_4$. The Ce content of the dried waste polishing powder was found to be 29.43%, and the XRD (Figure 1) shows the Ce in the form of $CeO_2$, while the content of La was as low as 4.06% and in form of the compound $LaF_3$ and LaOF. Impurity elements are mainly Si, Na, and Ca and in form of $NaO_2$ and $Ca_2SiO_4$ coming from glass substrates in polishing process. The shape of $CeO_2$ peak in Figure 2 shows that raw materials have a high content of $CeO_2$, which agrees with the XRF result.

## 2.2 Experimental methods

In this work, an extracting process was designed, as demonstrated in Figure 2. As cerium was found in waste polishing powder in the form of cerium oxide, which is insoluble in water, common acid (except for concentrated sulfuric acid) and alkali solution, to extract cerium from rare earth polishing powder the Ce(IV), must be reduced to Ce(III) before hydrochloric acid leaching. Thiourea, used as a kind of organic complexing agent, had been selected to be a reductant in this experiment; it can get a strong reaction with oxidant to produce urea, sulfuric acid, and other organic compounds [13–15]. And the oxidation-reduction potential of cerium has relativity with pH value, which decides that Ce(IV) is the powerful oxidant in acid solution. So, Ce(IV) can be extracted by hydrochloric acid leaching due to the reduction action of thiourea.

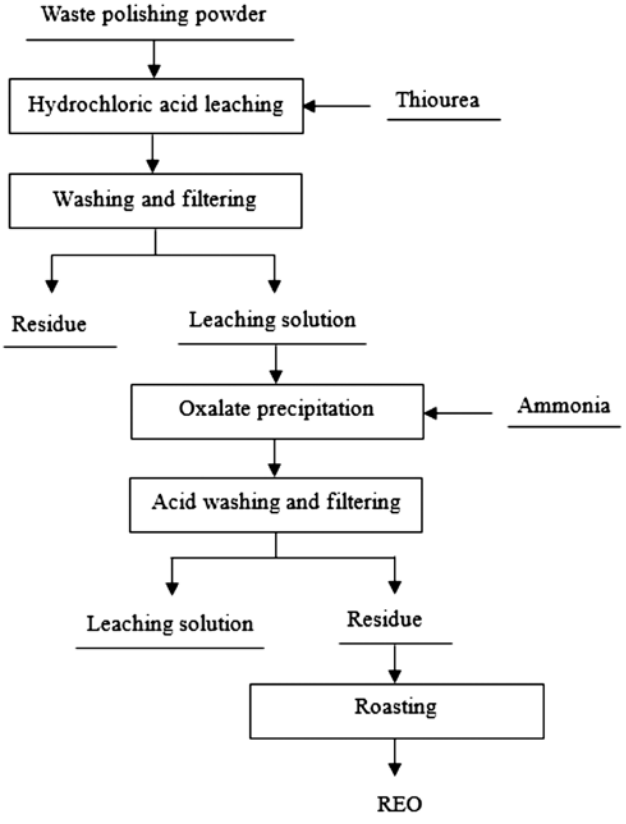


**Figure 2:** Experimental process of extracting cerium from the waste polishing powder.

Cerium was got by using oxalate as precipitant, which is the common precipitant in producing the rare earths and has a high purification rate (in a pH range of 1.8–2.0) to transition metal, Fe, Al, Ca, Si, and so on. In aqueous solution, rare earth can react with oxalate to produce $RE_2(C_2O_4)_3{\cdot}nH_2O$, which is insoluble in water and slight soluble in acid. And the pure cerium oxide can be obtained by oxidative roasting $RE_2(C_2O_4)_3{\cdot}nH_2O$ for 2 h at a temperature of over 800°C [16].

Oxalate was also used as precipitant to determine rare earth content of polishing powder waster by gravimetric method. And the rare earth content of polishing powder waster $CeO_2$% was 40.94% through repeat tests. The recovery rate $Y$% was calculated by the following mass balance equation:

$$Y\% = \frac{M}{M_1 \times \mathrm{CeO_2}\%} \times 100\%, \quad (1)$$

where $M$ is the mass of the finally obtained cerium oxides, $M_1$ is the mass of initial raw materials, and $CeO_2$% is the concentration of rare earth.

### 2.3 Design of experiments

A central composite design (CCD) of RSM was utilized to identify the optimal experimental conditions to maximize the extraction with minimum number of experiments and to identify the significant parameters and interactions between the parameters. A total of 30 experiments including six repeat run at the center point of all the variables were performed. The dependent variables selected were leaching temperature ($\chi_1$), leaching duration ($\chi_2$), thiourea dosage ($\chi_3$), and HCl concentration ($\chi_4$). The range of process parameters investigated in the present work, denoted in the form of coded variables, is shown in Table 2, while the exact experimental conditions are shown in Table 3. The ranges of these variables are selected through rigorous literature analysis as well as based on the results of the preliminary experiments.

The mathematical model relating the independent variable to the dependent variable was developed through the model equation as shown below,

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i \chi_i + \sum_{i=1}^{k} \beta_{ii} \chi_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} \chi_i \chi_j, \quad (2)$$

where $Y$ is the dependent response; $\beta_0$ is the constant coefficient; $\beta_i$ is the linear coefficient; $\beta_{ii}$ is the quadratic coefficients; $\beta_{ij}$ is the interaction coefficients; $k$ is the number of factors studied and optimized in the experiment; $\chi_i$ and $\chi_j$ are the coded values of independent variables; and the terms $\chi_i\chi_j$ and $\chi_i^2$ represent the interaction and quadratic terms, respectively.

**Table 2:** Range of variables utilized in the experimental design.

| Variables | Coded variable level | | | | |
|---|---|---|---|---|---|
| | −1.414 | −1 | 0 | 1 | 1.414 |
| Leaching temperature (°C) | 53.79 | 60 | 75 | 90 | 96.21 |
| Leaching duration (min) | 5.15 | 30 | 90 | 150 | 174.85 |
| Thiourea dosage (g/g) | 0.01 | 0.05 | 0.2 | 0.41 | 153.64 |
| HCl concentration (mol/l) | 0.01 | 1 | 3.5 | 7.04 | 0.16 |

**Table 3:** Central composite design arrangement and results.

| Run | $\chi_1$ (°C) | $\chi_2$ (min) | $\chi_3$ (g/g) | $\chi_4$ (mol/l) | $Y$ (%) |
|---|---|---|---|---|---|
| 1 | 60.00 | 30.00 | 0.20 | 3.50 | 30 |
| 2 | 90.00 | 30.00 | 0.20 | 3.50 | 45 |
| 3 | 60.00 | 150.00 | 0.20 | 3.50 | 39 |
| 4 | 90.00 | 150.00 | 0.20 | 3.50 | 90 |
| 5 | 75.00 | 90.00 | 0.05 | 1.00 | 22 |
| 6 | 75.00 | 90.00 | 0.35 | 1.00 | 39 |
| 7 | 75.00 | 90.00 | 0.05 | 6.00 | 38 |
| 8 | 75.00 | 90.00 | 0.35 | 6.00 | 82 |
| 9 | 60.00 | 90.00 | 0.20 | 1.00 | 29 |
| 10 | 90.00 | 90.00 | 0.20 | 1.00 | 45 |
| 11 | 60.00 | 90.00 | 0.20 | 6.00 | 39 |
| 12 | 90.00 | 90.00 | 0.20 | 6.00 | 90 |
| 13 | 75.00 | 30.00 | 0.05 | 3.50 | 23 |
| 14 | 75.00 | 150.00 | 0.05 | 3.50 | 41 |
| 15 | 75.00 | 30.00 | 0.35 | 3.50 | 45 |
| 16 | 75.00 | 150.00 | 0.35 | 3.50 | 85 |
| 17 | 60.00 | 90.00 | 0.05 | 3.50 | 19 |
| 18 | 90.00 | 90.00 | 0.05 | 3.50 | 44 |
| 19 | 60.00 | 90.00 | 0.35 | 3.50 | 37 |
| 20 | 90.00 | 90.00 | 0.35 | 3.50 | 89 |
| 21 | 75.00 | 30.00 | 0.20 | 1.00 | 23 |
| 22 | 75.00 | 150.00 | 0.20 | 1.00 | 41 |
| 23 | 75.00 | 30.00 | 0.20 | 6.00 | 46 |
| 24 | 75.00 | 150.00 | 0.20 | 6.00 | 84 |
| 25 | 75.00 | 90.00 | 0.20 | 3.50 | 80 |
| 26 | 75.00 | 90.00 | 0.20 | 3.50 | 79 |
| 27 | 75.00 | 90.00 | 0.20 | 3.50 | 78 |
| 28 | 75.00 | 90.00 | 0.20 | 3.50 | 79 |
| 29 | 75.00 | 90.00 | 0.20 | 3.50 | 78 |
| 30 | 75.00 | 90.00 | 0.20 | 3.50 | 79 |

The experimental data were analyzed using statistical software Design Expert version 7.1.5 (STAT-EASE Inc., MN, USA). The optimum conditions were identified by maximizing the objective function through the model equation developed. Three dimensional response surface plots indicating the effect of independent variables on the dependent variables were developed.

## 3 Results and discussion

The experimental conditions along with the results are shown in Table 3. The yield is observed to vary in the range of 51.29%–92.89%. The repeat runs at the center point of the variables (experiment runs 15–20) indicate the uncertainties involved in the experimental system.

### 3.1 Model development

Table 4 presents the results of various models tested with the experimental data. A high $F$ value coupled with the

**Table 4:** Statistical parameters for sequential models.

| Source | Sum of squares | Degree of freedom | Mean square | *F* value | p-Value | $R^2$ |
|---|---|---|---|---|---|---|
| Linear | 11735.33 | 4 | 2933.83 | 13.70 | <0.0001 | 0.6954 |
| 2FI | 1215.75 | 6 | 202.63 | 0.93 | 0.4976 | 0.7675 |
| Quadratic | 3867.93 | 4 | 966.98 | 240.46 | <0.0001 | 0.9967 |
| Cubic | 39.17 | 8 | 4.90 | 1.71 | 0.2637 | 0.9990 |
| Residual | 17.13 | 6 | 2.86 | | | |

low p value indicates the suitability of the model. Based on the results in Table 4, the cubic model is eliminated at a statistical level significance of 0.05. As suggested by the software, the quadratic model is found to be the most suitable model relating dependent and independent variables. Several indicators were used to evaluate the adequacy of the fitted model, and the results are shown in Table 5. The coefficient of determination ($R^2$), the adjusted determination coefficient (adj. $R^2$), coefficients of variation (CV), and model significance (*F* value) are used to judge the adequacy of the model.

As shown in Table 5, the model *F* value of 298.74 implies that the model is significant. There is only a 0.01% chance that a model *F*-value this large could occur due to noise. p-Value <0.0001 shows that model terms are significant. The accuracy and variability of the above model could be evaluated by the coefficient of determination $R^2$. The coefficient of determination ($R^2$) of the model is obtained as 0.9967, which indicates that 99.67% of the variability in the dependent variable could be explained, and only 0.33% of the total variations cannot be explained by the model. Additionally, the value of adjusted determination coefficient (adj. $R^2$) is 0.9933, which suggests good correlations between the dependent and the independent variables. CV is the ratio of the standard error of estimate to the mean value of the observed response, expressed in percentage. A model can be considered reasonably reproducible if the CV is not greater than 10%. A low value of CV (3.73%) shows the high degree of precision and a reliability of the experimental values [17, 18].

Based on the *F* value (Table 5), $\chi_1$ shows the largest *F* value of 913.85, indicating that temperature had the most significant effect on extraction efficiency, compared to $\chi_2$, $\chi_3$, and $\chi_4$. The effect of thiourea dosage on extraction efficiency is more significant than HCl concentration, with *F* values being 748.08 and 671.40, respectively. And the effect of leaching duration is insignificant than other factors, with *F* values being 584.87.

By applying least squares method and multiple regression analysis on the experimental results, the following

**Table 5:** Analysis of variance (ANOVA) for response surface cubic model for dechlorination.

| Source | Sum of squares | Degree of freedom | Mean square | *F* value | p-Value |
|---|---|---|---|---|---|
| Model | 16819.01 | 14 | 1201.36 | 298.74 | <0.0001 |
| $\chi_1$ | 3675.00 | 1 | 3675.00 | 913.85 | <0.0001 |
| $\chi_2$ | 2352.00 | 1 | 2352.00 | 584.87 | <0.0001 |
| $\chi_3$ | 3008.33 | 1 | 3008.33 | 748.08 | <0.0001 |
| $\chi_4$ | 2700.00 | 1 | 2700.00 | 671.40 | <0.0001 |
| $\chi_1\chi_2$ | 324.00 | 1 | 324.00 | 80.57 | <0.0001 |
| $\chi_1\chi_3$ | 182.25 | 1 | 182.25 | 45.32 | <0.0001 |
| $\chi_1\chi_4$ | 306.25 | 1 | 306.25 | 76.15 | <0.0001 |
| $\chi_2\chi_3$ | 121.00 | 1 | 121.00 | 30.09 | <0.0001 |
| $\chi_2\chi_4$ | 100.00 | 1 | 100.00 | 24.87 | 0.0002 |
| $\chi_3\chi_4$ | 182.25 | 1 | 182.25 | 45.32 | <0.0001 |
| $\chi_1^2$ | 1171.97 | 1 | 1171.97 | 291.43 | <0.0001 |
| $\chi_2^2$ | 1260.78 | 1 | 1260.78 | 313.52 | <0.0001 |
| $\chi_3^2$ | 1973.27 | 1 | 1973.27 | 490.69 | <0.0001 |
| $\chi_4^2$ | 1597.16 | 1 | 1597.16 | 397.16 | <0.0001 |
| Residual | 2.80 | 4 | 0.70 | | |
| Cor total | 16875.31 | 28 | | | |

$R^2 = 0.9967$; adj. $R^2 = 0.9933$; CV = 3.73%.

cubic equation is found to relate the dependent variable after eliminating the insignificant parameters as shown in Eq. (3).

$$Y = 78.8 + 17.5\chi_1 + 14\chi_2 + 15.83\chi_3 + 15\chi_4 + 9\chi_1\chi_2 + 6.75\chi_1\chi_3 + 8.75\chi_1\chi_4 + 5.5\chi_2\chi_3 + 5\chi_2\chi_4 + 6.75\chi_3\chi_4 - 13.44\chi_1^2 - 13.94\chi_2^2 - 17.94\chi_3^2, \quad (3)$$

where $\chi_1$, $\chi_2$, $\chi_3$, $\chi_4$, and $Y$ are leaching temperature (°C), leaching duration (min), thiourea dosage (g/g), and HCl concentration (mol/l), respectively. The suitability of model equation is evaluated using the correlation coefficients ($R^2$), which is 0.9815 for Eq. (3). The $R^2$ value of model equation is high, which indicates good agreement between experimental data and the model prediction.

The predicted values of extraction efficiency are calculated using the regression model and compared with experimental data in Figure 3. As can be seen, the predicted values are close to the experimental data, indicating the suitability of the model for optimization of extraction process.

## 3.2 Interactions of the factors

The yield of cerium over different combinations of independent variables can be visualized through three-dimensional view of response surface plots (Figures 4–6). Figure 4 shows the effect of leaching temperature and duration on extracting cerium from waste polishing powder (thiourea dosage and HCl concentration are fixed at 2.3 g/g and 3.5 mol/l, respectively), while Figure 5 shows the effect of thiourea dosage and leaching temperature on

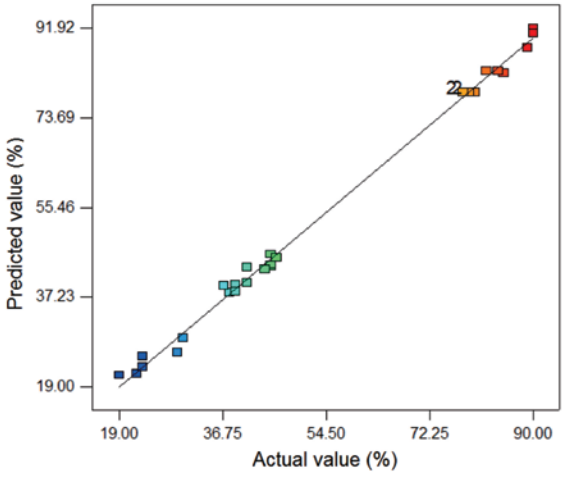


**Figure 3:** Linear correlation between actual and predicted values.

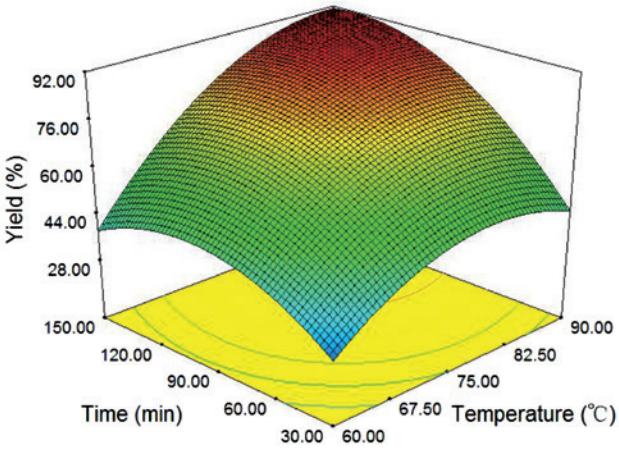


**Figure 4:** Three-dimensional response surface and contour plot of leaching time vs. temperature on $CeO_2$ yield.

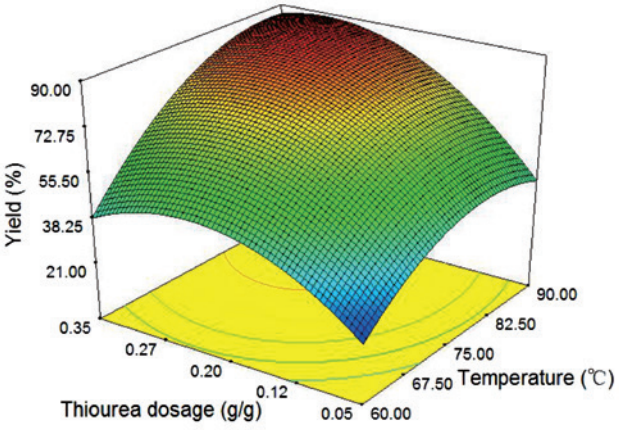


**Figure 5:** Three-dimensional response surface and contour plot of thiourea dosage vs. leaching temperature on $CeO_2$ yield.

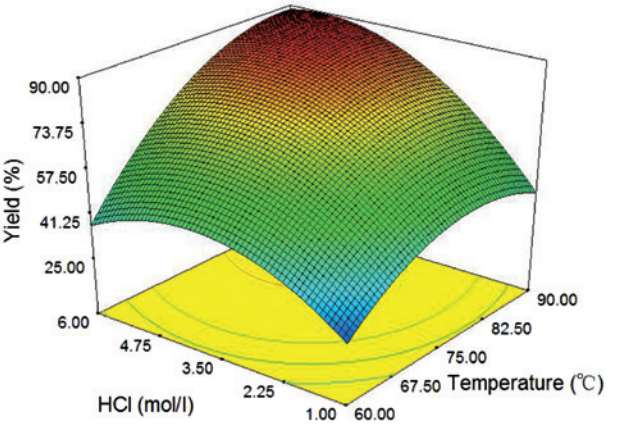


**Figure 6:** Three-dimensional response surface and contour plot of HCl concentration vs. leaching temperature on $CeO_2$ yield.

the yield of cerium (leaching duration and HCl concentration are fixed at 150 min and 3.5 mol/l, respectively). It can be seen from Figures 4 and 5 that yield peaks at a temperature of around 90°C. An increase in temperature and duration necessarily would increase the extraction efficiency, and hence, an increase in the %yield with increase in temperature and duration is observed. When the temperature is under 80°C and time is under 90 min, the %yield increases rapidly with increase in temperature and duration primarily due to the faster decomposition of thiourea at higher temperature reducing Ce(IV) to Ce(III). However, the yield is found to reduce reaching an asymptote at temperatures in excess of 80°C. It should be ascribed to the rapid volatilization of hydrochloric acid and consumption of thiourea at higher temperature [13–15].

Figure 6 shows the effect of HCl concentration and leaching temperature on extraction efficiency of cerium (thiourea dosage and leaching duration are fixed at 2.3 g/g and 150 min). Either an increase in the HCl concentration or temperature increases the recovery yield. The highest recovery yield is observed to correspond to the maximum of HCl concentration and leaching temperature. However, the effects of HCl concentration on the extraction efficiency can be divided into two stages. It was in the first stage that the extraction efficiency is obviously improved by the increasing HCl concentration until 3.5 mol/l. In the second stage, the HCl concentration over 3.5 mol/l did not have a significant impact on the extraction efficiency.

### 3.3 Process optimization and the phase changes

As shown in Table 6, the optimum conditions that maximized recovery yield are obtained utilizing the model equation and the optimizer option available with the Design Expert Software. These optimal conditions are identified to be a leaching temperature of 90°C, duration of 150 min, thiourea dosage of 0.2 g/g, and HCl concentration of 3.5 mol/l. The experiments were repeated at the optimized process conditions to ensure the acceptability of the optimized process conditions. An average value of the repeat experiments at the optimized conditions is 91.23%. The proximity of the model prediction with the results of the repeat runs indicates the success of the optimization exercise.

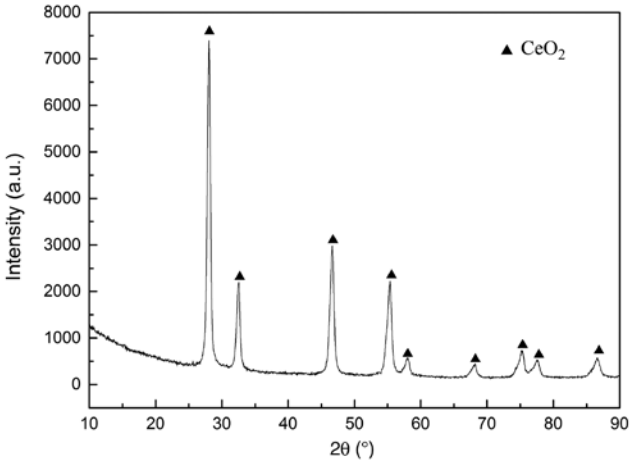


**Figure 7:** XRD pattern of samples after microwave roasting.

The samples generated under the optimized process conditions were subjected to characterization using X-ray diffraction. Figure 7 shows that cerium oxide exists as $CeO_2$, which means that cerium was oxidized to a tetravalent state by oxidative roasting at 800°C for 2 h.

## 4 Conclusions

As a green, environment-friendly and economic process, thiourea reducing and hydrochloric acid leaching is appropriate for extracting cerium from the waste polishing powder. RSM and CCD are successfully used for determining the optimal conditions for extraction process. The mathematical model is established using sets of experimental data and analysis of variance, and the $R^2$ values of all parameters show a good fit of the model with experimental data. Process optimization is carried out, and the optimum conditions for maximizing the recovery yield are identified to be a leaching temperature of 90°C, duration of 150 min, thiourea dosage of 0.2 g/g, and HCl concentration of 3.5 mol/l. Under optimum conditions, the obtained experimental recovery yield of 91.23% is found to agree satisfactorily with the predicted value of 91.92%. The comparison of X-ray diffraction patterns shows that most of

**Table 6:** Predicted and experimental values of the responses at optimum conditions.

| Temperature (°C) | Time (min) | Thiourea dosage (g/g) | HCl concentration (mol/l) | Yield (%) | |
|---|---|---|---|---|---|
| | | | | Predicted value | Experimental value |
| 80 | 150 | 0.2 | 3.5 | 91.92 | 91.23 |

impurities in waste polishing powder has been removed by the extraction process and the fairly pure cerium dioxide is obtain.

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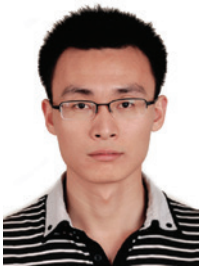
## Bionotes



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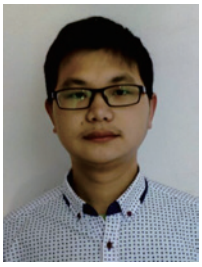
Ganfeng Tu is a PhD supervisor in Northeastern University. He mainly engages in the metallurgy and application of rare earths and the comprehensive utilization of metallurgical resources.

**Xiaoping Zhu**

Xiaoping Zhu is a PhD student at Northeastern University, China. His primary research interests include the metallurgy processing of rare earths, rare earth metal, and alloys.



**Xianghui Kong**

Xianghui Kong is a Master's student at Northeastern University, China. He currently carries out research on recovering cerium from the waste polishing powder.