Optimization Study on the Leaching of High Iron-Bearing Zinc Calcine After Reduction Roasting

JUNWEI HAN, WEI LIU, WENQING QIN, YONGXING ZHENG, and HONGLIN LUO

The selective leaching of zinc from high iron-bearing zinc calcine after reduction roasting was optimized by Taguchi experimental design method. The experimental parameters and their ranges were 303 to 343 K (30 to 70 °C) for leaching temperature (*T*), 7 to 15 mL/g for liquid/solid ratio (*L*/*S*), 70 to 150 g/L for H_2SO_4 concentration (*C*), 5 to 25 minutes for time (*t*), and 100 to 500 rpm for stirring speed (*R*). The results show that the optimum conditions were 303 K (30 °C), 9 mL/g, 110 g/L, 20 minutes, and 400 rpm, respectively. Under these conditions, about 92.81 pct Zn was extracted and more than 86 pct Fe was reported into the leach residue. *L*/*S* and *C* had significant effects on the extractions of zinc and iron, while *t* and *R* had no significant effects, and T had significant effect on iron extraction but negligible effect on zinc extraction. This indicates that diffusion was not a major control step of the leaching process, and the dissolution of iron was controlled by chemical reaction. The interactive effects of parameters were negligible. The leach residue was mainly composed of Fe_3O_4 and ZnS, and its particle size was very fine.

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I. INTRODUCTION

ZINC is of great importance for various applications in metallurgical, chemical, and textile industries and is mainly produced from zinc sulfide concentrates by conventional hydrometallurgical method.[1] By the process of zinc production, the sphalerite (ZnS) present in the concentrates is firstly converted into soluble oxide structure by an oxidative roasting and, then, the zinc calcine produced is subjected to a neutral and low acid leaching to generate a zinc sulfate solution for purification and subsequent electrolysis.[2–4] In general, approximately 10 pct of iron impurity accompanies with the concentrates, which tends to react with the zinc oxide to form zinc ferrite ($ZnFe_2O_4$) in the oxidative roasting process.[5–7] Zinc ferrite is stable and insoluble in dilute sulfuric acid solution, and thus its formation results in a generation of considerable amount of leach residues in the following leaching process.[8] The residues containing $ZnFe_2O_4$ pose a concern over not only environmental pollution but also economical loss.[9,10] On the other hand, with the consumption of low iron-bearing zinc sulfide concentrates, this problem is becoming more and more serious, because iron content of the concentrates used in zinc production is increasing gradually due to the fact that high iron-bearing zinc ores are abundant in the world, especially in China.

Both pyrometallurgical and hydrometallurgical methods have been developed industrially to overcome the difficulty of zinc extraction from zinc ferrite. The most common pyrometallurgical method is Waelz process,[11,12] in which the residue obtained after low acid leaching is subjected to a reduction roasting using large amounts of coke or coal at above 1273 K (1000 °C) for the transformation of zinc ferrite into metallic zinc. Although 90 to 95 pct of zinc can be recovered from the leach residue, some fatal drawbacks including high energy cost and strict requirement on dust collection system make this technology, as well as other similar methods, being washed out gradually. The most widely used hydrometallurgical approach is to leach the neutral residue using hot and concentrated sulfuric acid instead of dilute sulfuric acid to destroy the ferrite structure and, therefore, increase the extraction of zinc and decrease the amount of leach residue.[13,14] By this method, the extraction of zinc can be improved significantly, but more than 80 pct of iron is also dissolved into the leachate, which must be precipitated as jarosite, goethite, or hematite prior to purification and electrolysis processes.[15–18] Although iron precipitation methods are a well-established technology, its residues have never found any application and require further disposal. Currently, some researchers have paid their attention to the combination of pyrometallurgical and hydrometallurgical processes, which is, mainly through roasting process, such as transformation roasting,[19–21] sulfation roasting,[22] and reduction roasting,[23–25] to decompose $ZnFe_2O_4$ into soluble zinc phases for facilitating the subsequent extraction of zinc by neutral and low acid leaching processes. These methods in reducing energy consumption and environment protection have a

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significant potential, but they are still in the experimental stage and do not attach importance to iron recovery.

For comprehensive recovery of zinc and iron from high iron-bearing zinc concentrates, the conventional hydrometallurgical process was being improved by our work in recent years.[26,27] In our study, a selective reduction roasting at lower temperature was added after the oxidative roasting to make full use of the heat generated in the previous process for converting the $ZnFe_2O_4$ produced into zinc oxide (ZnO) and magnetite (Fe_3O_4). The reactions occurred in the reduction roasting process are given in Eqs. [1] and [2]. The ZnO generated could be extracted in subsequent neutral and low acid leaching, while the Fe_3O_4 was reported into the leach residue and then was recovered by magnetic separation. The improvement not only simplifies zinc production process depending on the cancelation of iron precipitation process, but also reduces the anticorrosion requirement of leach equipments and the quantity of leach residue. The present research focused on selective extraction of zinc from high iron-bearing zinc calcine after the reduction roasting, and correspondingly mineralogical changes during this process were characterized by XRD, VSM, and SEM.

$$3ZnFe_2O_4 + CO(g) = 3ZnO + 2Fe_3O_4 + CO_2(g) \quad [1]$$

$$ZnFe_2O_4 + CO(g) = ZnO + 2FeO + CO_2(g) \quad [2]$$

Taguchi method has been shown to be an effective means for the improvement of the productivity in the stage of research and development, so that high-quality items can be produced quickly at low cost. It has been found lots of applications in a wide range of industrial fields for its universal applicability to all engineering fields.[28–30] Taguchi experimental design method was therefore applied to determine the optimum leach conditions for selectively dissolving zinc in the leachate rejecting iron in the residue. The effects of experimental parameters including leaching temperature (T), liquid/solid ratio (L/S), H_2SO_4 concentration (C), leaching time (t), and stirring speed (R) on extractions of zinc and iron were investigated by an $L_{25}(5^5)$ orthogonal array.

II. EXPERIMENTAL

A. *Materials*

High iron-bearing zinc calcine used in this study was kindly supplied from a zinc hydrometallurgical plant in Inner Mongolia, China. The zinc calcine was firstly subjected to a reduction roasting with coal gas, for the selective decomposition of zinc ferrite within the zinc calcine into ZnO and Fe_3O_4, under the conditions established previously,[31] which were 33 pct flow rate ratio of coal gas to CO_2, 1073 K (800 °C) roasting temperature, and 1 hour time duration. The reduction roasted zinc calcine obtained was used as the raw material for the leaching experiments. The main compositions, crystalline phases, and magnetic hysteresis loops of the zinc calcines before and after the reduction roasting are given in Table I, Figures 1 and 2, respectively. As shown in Table I, the main composition of the zinc calcine is Zn, O, and Fe. After the reduction roasting, the content of Zn and Fe was increased, while the content of O was decreased because of $ZnFe_2O_4$ decomposition. It can be seen from Figure 1 that the zinc calcine is composed of ZnO, $ZnFe_2O_4$, Fe_3O_4, ZnS, and $ZnSi_2O_4$, and the XRD peaks of $ZnFe_2O_4$ were shifted to the position of Fe_3O_4 after the reduction roasting, indicating that the $ZnFe_2O_4$ contained in the zinc calcine was selectively converted into Fe_3O_4 and ZnO. It is seen from Figure 2 that the magnetism of zinc calcine was enhanced significantly after the reduction roasting, which is attributed to the conversion of $ZnFe_2O_4$ to Fe_3O_4, according to Figure 1. This also confirmed the conclusion obtained from Figure 1.

B. *Methods*

The schematic diagram of experimental equipment used in the present study is shown in Figure 3. For each test, 100 g of the reduction roasted zinc calcine was put into a glass beaker containing sulfuric acid solution, which had been prepared in the light of required liquid/solid ratio and H_2SO_4 concentration, heated in a thermostatically controlled water bath at target temperature in advance, and provided with a mechanical stirrer. After leaching, the leachate was separated from the residue by vacuum filtration, whose volume was measured by a measuring cylinder, and analyzed for contents of zinc and iron by chemical methods (EDTA titration for Zn and potassium dichromate titration for Fe). The leach residue was dried, weighed, and preserved in closed vessel for other related analyses and subsequent experiments. Distilled water was used throughout this experiment.

In addition, it is necessary to introduce the analysis techniques used in the whole experiments. X-ray fluorescence spectroscopy (XRF, Rigaku, model ZSX Primus II) and chemical methods were used for determining chemical composition of samples, X-ray diffraction analysis (XRD, Rigaku, TTR-III) was applied to determine crystalline phases, vibration sample magnetometer (VSM, BHV-50HTI) was used to detect magnetism, and scanning electron microscopy (SEM, JEOL, JSM-6490LV) was applied to the morphology study.

In this study, the orthogonal array design (OAD) was adopted to determine experimental plan in order to obtain the optimum leaching conditions effectively and economically. It is well known that the operation parameters including liquid/solid ratio, acid concentration, leaching temperature, time, and stirring speed are major factors and have different effects on extractions of zinc and iron in acid leaching process. Therefore, the five-level orthogonal array (OA) $L_{25}(5^5)$, which denotes five factors each with five levels,[32,33] was chosen to arrange the leaching experiment and is given in Table II. Accordingly, the exact levels of every factor were selected according to our knowledge, preliminary tests, and applied feasibility, and the results are shown in Table III. Additionally, a question needs to be explained that the order of experiments was made random in the

Table I. Main Chemical Compositions of the Zinc Calcine and Leach Residue (Mass Pct)

Element	Zn	O	Fe	S	Pb	Cu	Si	Ca	Mn	As	Ag
Before roasting	56.15	22.9	13.31	2.44	1.27	0.91	0.89	0.57	0.53	0.04	0.014
After roasting	58.74	20.41	14.08	2.25	1.26	1.02	1.13	0.70	0.61	0.05	0.015
Leach residue	18.55	26.80	44.55	4.86	4.42	1.26	0.22	0.16	1.05	0.02	0.046

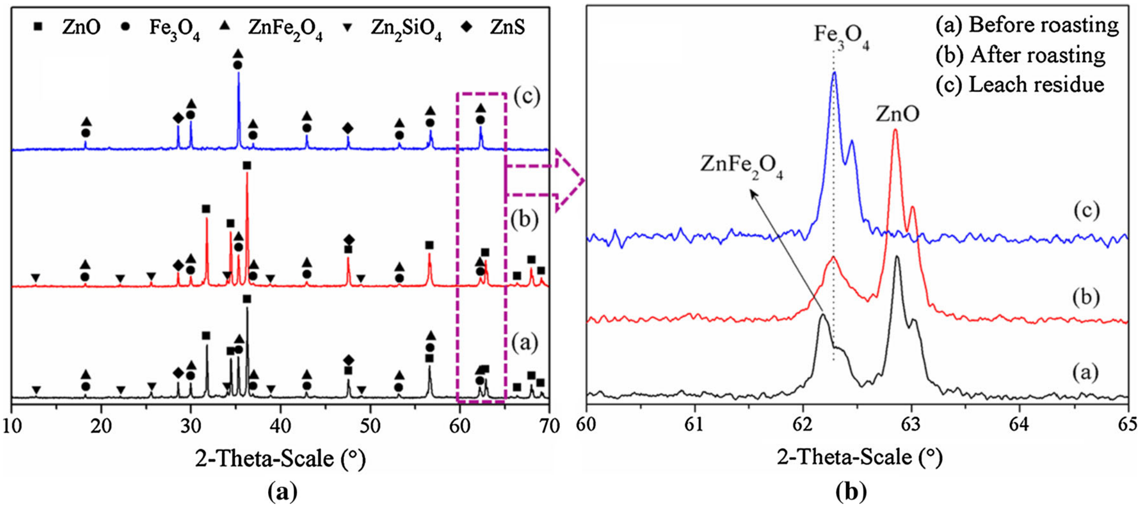

Fig. 1—XRD patterns of the zinc calcine before and after reduction roasting and leach residue: (*a*) within 10 to 70 deg; (*b*) within 60 to 65 deg.

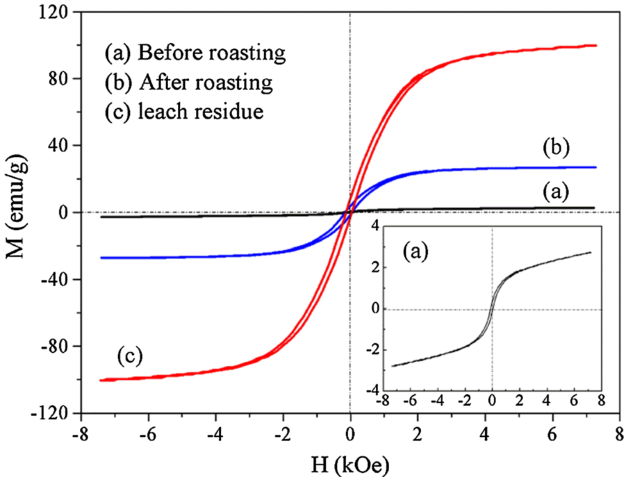

Fig. 2—Magnetic hysteresis loops of the zinc calcine before and after reduction roasting and leach residue.

operating process to avoid noise sources, although the experimental plan was listed according to Table II.

III. RESULTS AND DISCUSSION

A. *Statistical Analysis for Optimizing Conditions*

All tests were strictly performed twice according to the candidate conditions. The leaching efficiencies of zinc and iron were acted as responses, and the mean values of the two experimental results are also displayed in Table II. The obtained data were analyzed using an EXCELL program for evaluation of the effect of each leaching parameter on the optimization criterion. The objective of this study is to selectively dissolve zinc from the zinc calcine into dilute sulfuric acid solution and reject iron to leach residue as magnetite. Therefore, the

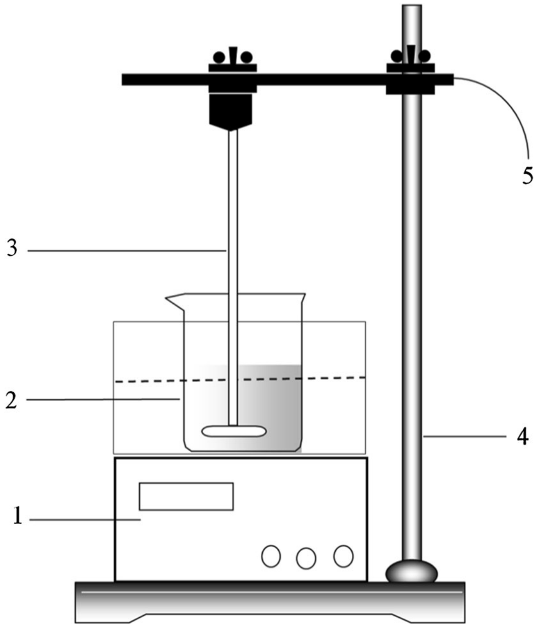

Fig. 3—Schematic diagram of leaching equipment: 1-thermostatically controlled water bath; 2-glass beaker; 3-mechanical stirrer; 4-iron support; 5-joint stirrer controller.

Table II. L_{25} (5^5) Experimental Plan Table and Mean Responses of Zn and Fe

Experiment Number	Parameters and Their Levels					Responses (Pct)	
	A	B	C	D	E	Zn	Fe
1	1	1	1	1	1	46.23	3.21
2	1	2	2	2	2	81.22	7.08
3	1	3	3	3	3	90.84	11.92
4	1	4	4	4	4	91.48	16.82
5	1	5	5	5	5	92.85	26.42
6	2	1	2	3	4	66.44	8.05
7	2	2	3	4	5	90.54	12.09
8	2	3	4	5	1	91.46	31.92
9	2	4	5	1	2	91.59	28.86
10	2	5	1	2	3	86.65	24.17
11	3	1	3	5	2	82.91	19.03
12	3	2	4	1	3	90.8	29.97
13	3	3	5	2	4	93.73	19.31
14	3	4	1	3	5	83.62	21.14
15	3	5	2	4	1	91.79	26.82
16	4	1	4	2	5	86.58	21.78
17	4	2	5	3	1	92.31	34.94
18	4	3	1	4	2	76.42	21.08
19	4	4	2	5	3	90.96	27.98
20	4	5	3	1	4	91.09	27.62
21	5	1	5	4	3	91.43	12.73
22	5	2	1	5	4	73.96	6.39
23	5	3	2	1	5	89.52	14.03
24	5	4	3	2	1	92.23	20.43
25	5	5	4	3	2	93.56	32.58

Table III. Parameters and Their Values Corresponding to Their Levels

Parameters		Levels				
		1	2	3	4	5
A	T [K (°C)]	303 (30)	313 (40)	323 (50)	333 (60)	343 (70)
B	L/S (mL/g)	7	9	11	13	15
C	C (g/L)	70	90	110	130	150
D	t (min)	5	10	15	20	25
E	R (rpm)	100	200	300	400	500

maximum amount of zinc extraction and minimum amount of iron extraction were considered as optimization criterion for the selection of leaching conditions. The Taguchi method recommends that mean response of each factor is analyzed for investigating the changed trend of response with the increase in level,[11] and the results with respect to zinc and iron are shown in Figures 4 and 5, respectively.

In the view of above, the system is optimized when the response for zinc is as large as possible and that for iron is as small as possible, hence we deal with factor levels that maximize the mean response of zinc and minimize that of iron. There are two questions that need to be illustrated in terms of the application of mean response. The first one is about the calculation of mean response,[28,34] which is complex at first glance and will be explained with an example. Considering the parameter B (L/S ratio), the mean response at the level 1 (7 mL/g) is the average of those data obtained from experiments with experiment nos. 1, 6, 11, 16, and 21. Likewise, the mean response of level 2 is the average of those obtained according to all the experiments carried out under the conditions containing 9 mL/g, which are experiment nos. 2, 7, 12, 17, and 22. The second is that these figures, such as Figures 4 and 5, are only used to display the trend of each factor more understandable and cannot be used to predict other values which were not experimented.

As seen from Figure 4, liquid/solid ratio and H_2SO_4 concentration had more effective influences on extraction of zinc than other parameters depending on the difference in change degree of mean response and they were proportional to mean response. The increase in liquid/solid ratio and H_2SO_4 concentration pronouncedly promoted zinc extraction because of the increase of the amount and concentration of H^+ ions provided by H_2SO_4. On the other hand, it can be found from Figure 5 that liquid/solid ratio, H_2SO_4 concentration, and temperature had positive effects on the extraction of iron. It is interesting that the mean response significantly

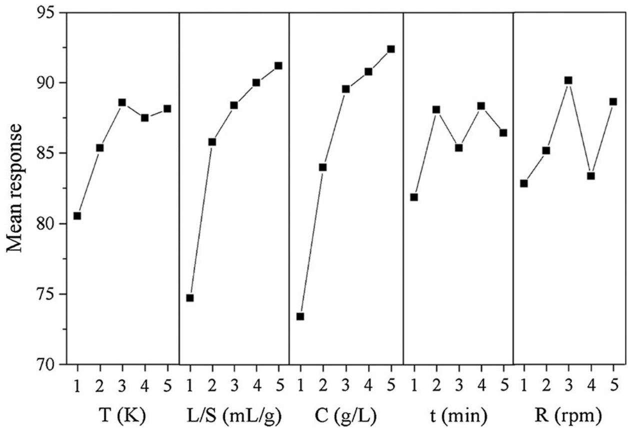

Fig. 4—Effects of controllable factors on mean responses for zinc.

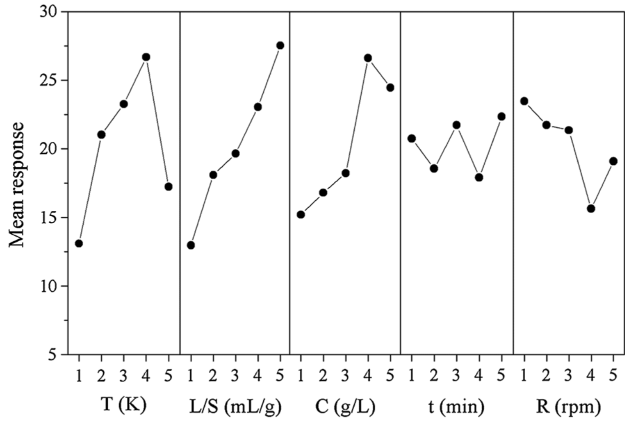

Fig. 5—Effects of controllable factors on mean responses for iron.

decreased as the temperature increased from level 4 to 5, which is attributed to the explanation that the oxidization of Fe^{2+} to Fe^{3+} as in Eq. [7] could be accelerated at higher temperatures and thus the Fe^{3+} ion precipitation by hydrolysis according to Eq. [8] was promoted. This is bad for subsequent filtration and remaining magnetite structure although which is conducive to decreasing the extraction of iron. Compared with zinc, it is seen that stirring speed was conversely proportional to mean response, indicating that increasing the stirring speed could promote the selective extraction of zinc from the calcine. Obvious is the fact that leaching time had little effect on the extractions of iron and zinc and that the required time for the leaching was distinctly short, in comparison with conventional leaching process. The quick dissolution of zinc and iron is accounted by the effect of the reduction roasting. In the leaching process, the reactions occurred probably are listed as follows:

$$ZnO + 2H^{+} = Zn^{2+} + H_2O \qquad [3]$$

$$Zn_2SiO_4 + 4H^{+} = 2Zn^{2+} + 2H_2SiO_4 \qquad [4]$$

$$FeO + 2H^{+} = Fe^{2+} + H_2O \qquad [5]$$

$$Fe_3O_4 + 8H^{+} = Fe^{2+} + 2Fe^{3+} + 4H_2O \qquad [6]$$

$$4Fe^{2+} + O_2 + 4H^{+} = 4Fe^{3+} + 2H_2O \qquad [7]$$

$$Fe^{3+} + 3H_2O = Fe(OH)_3 + 3H^{+}. \qquad [8]$$

Before selecting the optimum conditions, it is better that the effect of each parameter on the extractions of zinc and iron would be investigated further. Statistical analysis of variance (ANOVA) was therefore performed to see which parameters are of statistical significance. *F* test is a tool to check whether, or not, the operating parameters have a significant effect on the extraction efficiency, and accordingly the *F* value for each parameter is a ratio of the squared deviations to the mean of the squared error.[33] The larger *F* value, in general, indicates the greater effect on the extraction value.[35] The results of ANOVA with *F* test for zinc and iron are presented in Tables IV and V, respectively.

As shown in Figure 4, it is found that liquid/solid ratio and H_2SO_4 concentration had significant effects on the extraction of zinc. The *F* values of the two factors were greater than the extracted *F* value from the table for 95 pct confidence level ($F = 6.39$), which indicates that the variances of these factors were significant compared with that of error. However, the *F* values for temperature, time, and stirring speed were smaller than 6.39 and thus these factors had no significant effect on zinc extraction at least within the experimental ranges. For iron extraction, it is found from Table V that the temperature, liquid/solid ratio, and H_2SO_4 concentration had significant effects on the leaching process, while leaching time and stirring speed had no significant effects in the range of the proposed study. It is expected that these results concluded by ANOVA were in accordance with that by mean response. Based on the results obtained from Figures 4 and 5, the leaching conditions, which are $(T)_3$ [323K (50 °C)], $(L/S)_5$ (15 mL/g), $(C)_5$ (150 g/L), $(t)_4$ (20 minutes), and $(R)_3$ (300 rpm), were selected in terms of maximizing the mean response for zinc (case 1). Also in terms of minimizing the mean response for iron, $(T)_1$ [303 K (30 °C)], $(L/S)_1$ (7 mL/g), $(C)_1$ (70 g/L), $(t)_4$ (20 minutes), and $(R)_4$ (400 rpm) were selected (case 2). Obviously, the selected conditions for zinc extraction are inconsistent with those for iron extraction and thus the optimum conditions should be obtained on the basis of the comprehensive consideration. From what has been discussed above, the optimum temperature was chosen as 303 K (30 °C), depending on the conclusion obtained that leaching temperature was a major factor for iron extraction, while it had negligible effect on zinc

Table IV. Result of the Analysis of Variance for Dissolution Values of Zinc

Parameters		Sum of Squares	Degrees of Freedom	Variances	*F*
A	*T* [K (°C)]	218.892	4	54.72	1.78
B	*L*/*S* (mL/g)	878.965	4	219.74	7.15
C	*C* (g/L)	1196.829	4	299.21	9.73
D	*t* (min)	138.144	4	34.55	1.12
E	*R* (rpm)	210.053	4	52.51	1.71
	Error	123.010	4	30.75	

Table V. Result of the Analysis of Variance for Dissolution Values of Iron

Parameters		Sum of Squares	Degrees of Freedom	Mean Squares	*F*
A	*T* [K (°C)]	556.584	4	139.15	13.59
B	*L*/*S* (mL/g)	594.149	4	148.54	14.51
C	*C* (g/L)	499.030	4	124.76	12.19
D	*t* (min)	75.866	4	18.97	1.85
E	*R* (rpm)	181.693	4	45.42	4.44
	Error	40.950	4	10.24	

Table VI. Predicted and Observed Extractions of Zn and Fe and Their Confidence Intervals

		Case 1		Case 2		Case 3		Case 4	
Parameters		Value	Level	Value	Level	Value	Level	Value	Level
A	*T* [K (°C)]	323 (50)	3	303 (30)	1	303 (30)	1	303 (30)	1
B	*L*/*S* (ml/g)	15	5	7	1	9	2	11	3
C	*C* (g/L)	150	5	70	1	110	3	90	2
D	*t* (min)	20	4	20	4	20	4	20	4
E	*R* (rpm)	300	3	400	4	400	4	400	4
Zn	Predicted (pct)	97.56		62.09		89.28		86.37	
	Observed (pct)	93.14		55.39		92.81		92.25	
	Confidence interval (pct)	89.21-99.9		53.74-70.44		80.93-97.63		78.02-94.72	
Fe	Predicted (pct)	34.72		0.74		8.89		9.02	
	Observed (pct)	28.38		1.89		13.79		15.21	
	Confidence interval (pct)	26.88-42.56		0.01-8.58		1.05-16.73		1.19-16.86	

extraction. However, it is difficult to deduce the optimum levels of liquid/solid ratio and H_2SO_4 concentration which had significant effects on the extractions of zinc and iron. Based on our knowledge and previous experiments, the combinations of $(L/S)_2$ $(C)_3$ and $(L/S)_3$ $(C)_2$ were determined as alternatives for optimum liquid/solid ratio and H_2SO_4 concentration. Consequently, the optimum conditions should be chosen as $(T)_1$ [303 K (30 °C)], $(L/S)_2$ (9 mL/g), $(C)_3$ (110 g/L), $(t)_4$ (20 minutes), and $(R)_4$ (400 rpm) (case 3) or $(T)_1$ [303 K (30 °C)], $(L/S)_3$ (11 mL/g), $(C)_2$ (90 g/L), $(t)_4$ (20 minutes), and $(R)_4$ (400 rpm) (case 4). It can be seen that the experiments according to cases 1 to 4 have not been done during the whole period of the experimental stage and their results were predicted using above findings and modeled significant effects by the Taguchi method. In order to test the predicted results, corresponding confirmation experiments were carried out and the confidence intervals for ±5 pct risk were also predicted correspondingly. These results are presented in Table VI. As shown in this table, there is a good agreement between predicted values and experimental values which are within the predicted confidence intervals, indicating that these results are within the 95 pct significance level, the interactions between different factors are probably unimportant, and the additive

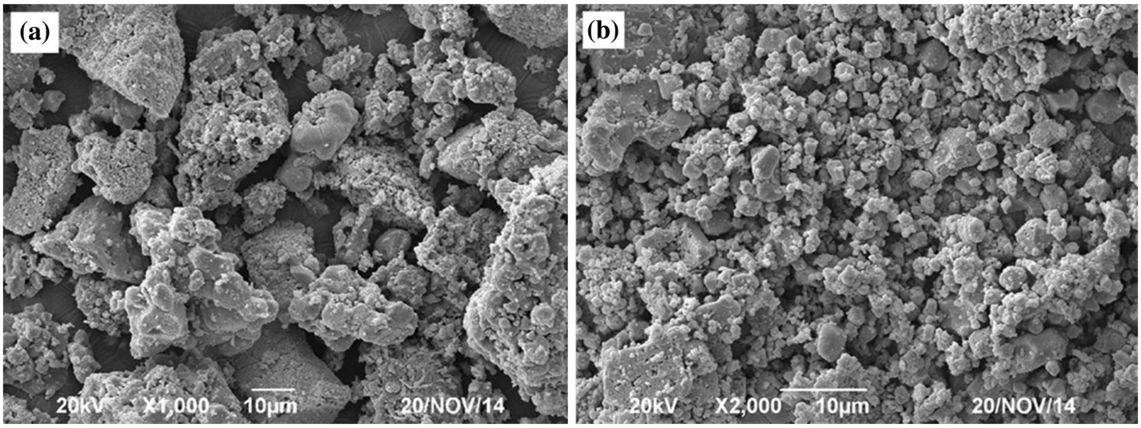

Fig. 6—SEM images of reduction roasted zinc calcine (*a*) and leach residue (*b*).

model is a good approximation. It is also seen that the extraction of zinc obtained in case 3 was larger than that in case 4, while the extraction of iron in case 3 was lesser than that in case 4. This indicates that $(T)_1$ [303 K (30 °C)], $(L/S)_2$ (9 mL/g), $(C)_3$ (110 g/L), $(t)_4$ (20 minutes), and $(R)_4$ (400 rpm) were the ultimate optimum conditions.

B. *Analysis of the Leach Residue*

The main chemical composition, XRD pattern, and magnetic hysteresis loop of the leach residue obtained under the optimum conditions are shown in Table I, Figures 1 and 2, respectively. As shown in Table I, in comparison with the zinc calcines, it is obvious that the iron, as well as other valuable metals including lead and silver, was concentrated in the leach residue, indicating that the objective of this study, which is to dissolve zinc into the leachate while rejecting iron into the residue, was well achieved. It can be found from Figure 1 that the residue was mainly composed of Fe_3O_4, ZnS, and $ZnFe_2O_4$, suggesting that the soluble zinc phases including ZnO and Zn_2SiO_4 contained in the zinc calcine were dissolved completely and, in the mean time, those insoluble zinc phases, such as ZnS and $ZnFe_2O_4$, were reported into the leach residue. It is seen from Figure 2 that magnetism of the leach residue was much stronger than that of the reduction roasted zinc calcine, which confirms that most of the iron in the form of magnetite was concentrated in the leach residue. In order to investigate morphology changes during the leaching process, the SEM images of the reduction roasted zinc calcine and the leach residue are presented in Figure 6. Obviously, the grains of the reduction roasted zinc calcine were fine and porous, which is the main reason for the quick dissolution of zinc and iron. It is also seen from Figure 6 that the particles of the leach residue, most of which were smaller than 10 μm, were much smaller than those of the zinc calcine. This indicates that the crystalline grain size of the magnetite generated during the reduction roasting process was very fine.

IV. CONCLUSIONS

In this research, Taguchi experimental design method was used to optimize the selective leaching of zinc from high iron-bearing zinc calcine after the reduction roasting, under the conditions which were 33 pct flow rate ratio of coal gas to CO_2, 1073 K (800 °C), and 1 hour time. According to the results obtained, liquid/solid ratio and H_2SO_4 concentration had significant effects on the extractions of zinc and iron. Leaching temperature had significant effects on iron extraction but had negligible effect on zinc extraction, which indicates that the dissolution process of iron was controlled by chemical reaction. Leaching time and stirring speed had no significant effects on the extractions of zinc and iron, indicating that diffusion was not a major control step of the leaching process. The optimum conditions were reaction temperature of 303 K (30 °C), liquid/solid ratio of 9 mL/g, H_2SO_4 concentration of 110 g/L, leaching time of 20 minutes, and stirring speed of 400 rpm. Under the conditions, about 92.81 pct of zinc was extracted and more than 86 pct of iron, mainly in the form of magnetite, was reported into the leach residue, suggesting that the objective of this study had been achieved. Confirmation experiments indicated that the extractions of zinc and iron had a good agreement with their predicted valves and were within the predicted confidence intervals with 95 pct significant level. This proves that the interactive effects of parameters were negligible. The mineralogical study of the leach residue shows that the presence of insoluble zinc phases, such as ZnS and $ZnFe_2O_4$, was the main reason for the limitation of zinc extraction, and that the iron, mostly as magnetite, along with other valuable metals including lead and silver was concentrated in the leach residue, whose particles were very fine.

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