

Article

Restraining Sodium Volatilization in the Ferric Bauxite Direct Reduction System

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Academic Editor: William Skinner

Received: 27 December 2015; Accepted: 21 March 2016; Published: 31 March 2016

Abstract: Direct reduction is an emerging utilization technology of ferric bauxite. However, it requires much more sodium carbonate than ordinary bauxite does. The volatilization is one of the most significant parts of sodium carbonate consumption, as reported in previous studies. Based on the new direct reduction method for utilization of ferric bauxite, this paper has systematically investigated factors including heating temperature, heating time, and sodium carbonate dosage influencing sodium volatilization. For the purpose of reducing sodium volatilization, the Box–Behnken design was employed, and the possibility of separating iron and sodium after direct reduction was also investigated.

Keywords: ferric bauxite; alumina leaching; sodium volatilization; pyrolysis; direct reduction

1. Introduction

Ferric bauxite is a highly valuable refractory bauxite that is widely distributed in China [1,2], Laos [3], and Tanzania [3], but is improperly used because it contains much more iron than ordinary bauxite. The possible utilization technologies of ferric bauxite include the extraction of alumina before iron [4,5], extraction of iron before alumina [6,7], pretreatment before Bayer processing [8], and biological treatment [9,10]. Another technology that has recently become a popular topic of research is direct reduction [11–13]. The addition of sodium carbonate facilitates the conversion of diasporite and boehmite into sodium aluminate (water soluble), which is then separated from the solution and used for alumina production. Hematite and goethite can be reduced to iron powder by carbothermal reaction and then separated from the magnetic concentrate. Iron powder may then be used for steel and casting production after agglomeration. However, the direct reduction technology of ferric bauxite at normal atmospheric pressure requires much more sodium carbonate than ordinary bauxite, mainly because ferric bauxite contains more iron than ordinary bauxite, which in turn increases the generated amorphous sodium ferrite and renders sodium volatile [14,15]. Hence, volatilization is one of the most significant parts of sodium carbonate consumption [14]. Our previous research [14] indicated that heating temperature, heating time, and sodium carbonate dosage are significant factors that influence sodium volatilization; however, the values of their effects have not yet been quantified. Box–Behnken design is an independent, rotatable quadratic design with no embedded factorial or fractional factorial points where the variable combinations are at the midpoints of the edges of the variable space and at the center, and is typically adopted to conduct quantitative research by determining the regression

model. In this study, the Box–Behnken design is used to investigate the effects of experimental factors on the sodium volatilization ratio (R) for the purpose of reducing R . However, sodium is a harmful element in iron powder; a decrease in R increases the remaining sodium in the solid phase. Hence, the possibility of separating iron and sodium must necessarily be investigated to ensure that the restraining process can be conducted harmlessly. X-ray diffraction (XRD) [16] and scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS) [17] analysis are performed to determine the mineral phase of sodium in iron powder and tailing. The findings can aid in reducing R and in the subsequent sodium separation after direct reduction and may be beneficial in reducing the sodium carbonate consumption of current alumina production processes.

2. Experimental

2.1. Experimental Materials

The ore used was supplied by the Guangxi Zhuang Autonomous Region of China. Its chemical composition, XRD pattern, and SEM-EDS images are shown in Table 1, Figures 1 and 2 respectively. Table 1 indicates the alumina-silica ratio (A/S) of the ferric bauxite is only 2.7, an extremely low value. Figure 1 shows five main crystalline mineral phases in raw ferric bauxite, namely, hematite, boehmite, diaspore, kaolinite, and goethite. Figure 2 reveals that the minerals in ferric bauxite are finely disseminated and symbiotic with one another. Suggested from the EDS, Figure 2b is diaspore or boehmite, Figure 2c is hematite, Figure 2d is goethite symbiotic with kaolinite, Figure 2e is goethite (darker than hematite), Figure 2f is kaolinite symbiotic with goethite, and Figure 2g is diaspore symbiotic with goethite.

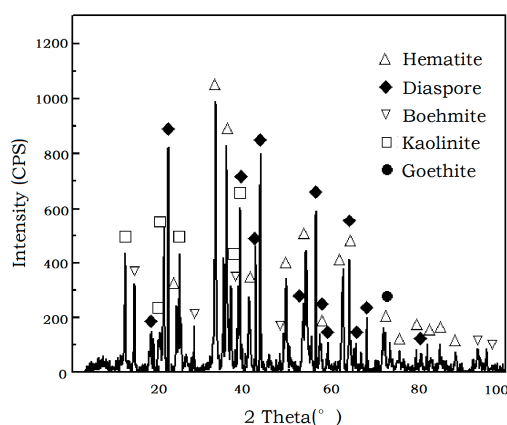


Figure 1. XRD patterns of ferric bauxite.

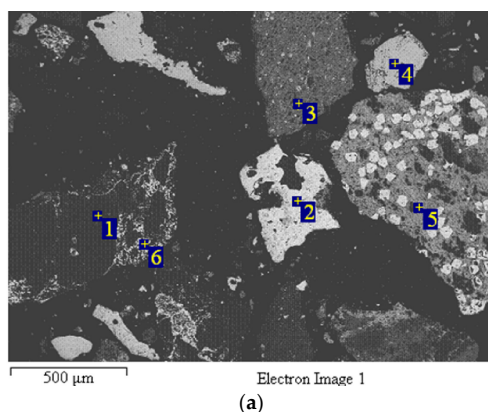


Figure 2. Cont.

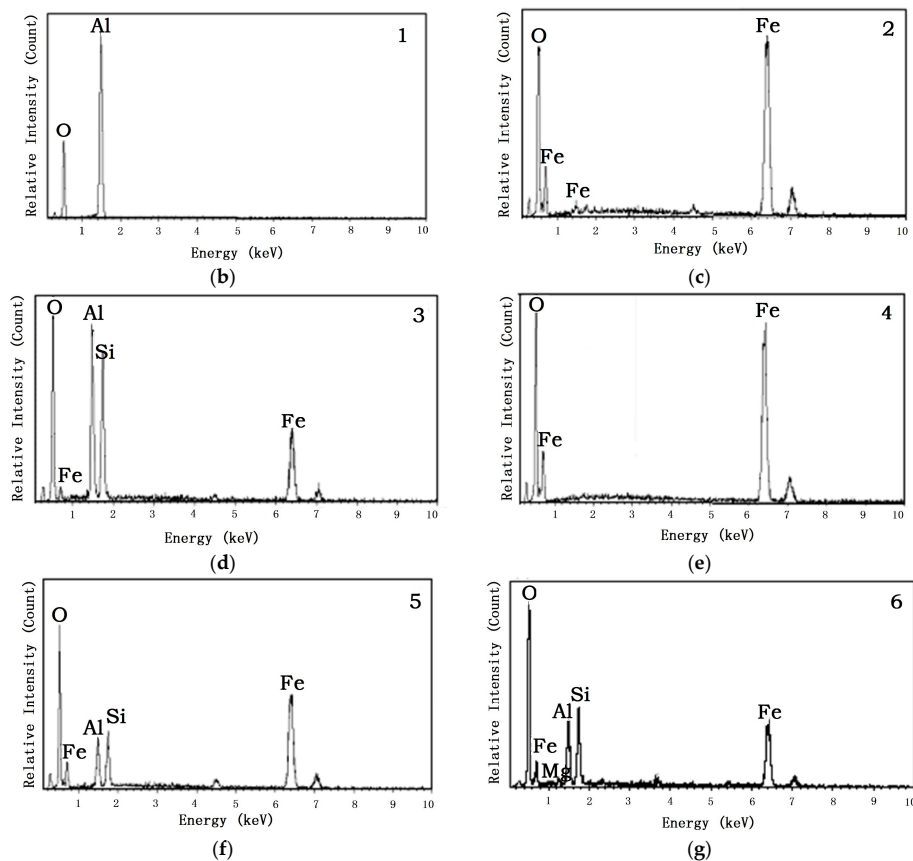


Figure 2. (a) SEM image of ferric bauxite; (b) EDS image of point 1; (c) EDS image of point 2; (d) EDS image of point 3; (e) EDS image of point 4; (f) EDS image of point 5 and (g) EDS image of point 6.

Table 1. Chemical composition of ferric bauxite.

Composition	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	TiO ₂	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	LOI
Content/%	41.13	33.02	12.22	1.49	0.68	0.63	0.32	0.06	0.04	8.97

The proximate analysis is utilized to determine the weight percent moisture, volatile matter, fixed carbon, and ash content of the coal adopted. The proximate analysis results and the chemical composition of coal ash are shown in Tables 2 and 3 respectively.

Table 2. Proximate analysis of the coal used.

Component	Total Moisture (M _t)	Volatile Matter (V _{ad})	Ash (A _{ad})	Fixed Carbon (FC _{ad})
Content/%	9.16	39.42	5.07	46.35

Table 3. Chemical composition of coal ash.

Component	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	K ₂ O	TiO ₂	Na ₂ O	P ₂ O ₅
Content/%	38.00	36.19	21.37	7.15	1.90	1.38	0.84	0.43	0.41

2.2. Experimental Instruments

The direct reduction, grinding, and leaching experiments were performed with a muffle furnace (SX2-10-13, INCH, Beijing, China), a rod mill (XMB-70, Hengcheng, Ganzhou, China), and a stirrer

(JJ-3, Guohua, Changzhou, China) with 1000 mL beaker, respectively. The other instruments used in the experiment include a balance (AR1140, Mettler, Columbus, OH, USA), a filter (XTLZ, Hengcheng, Tangshan, China), a magnetic tube (CXG-99, Yihao, Tangshan, China), and a drying oven (PH050, Shuangxu, Shanghai, China).

The chemical composition of the samples was analyzed using an atomic absorption spectrophotometer (UV-9600, Rayleigh, Beijing, China). The mineral composition of ore was determined by XRD analysis (TTRIII, Rigaku, Tokyo, Japan). The morphology and microzone chemical composition of the sample were examined using an electron microscope (EVO 18, ZEISS, Jena, Germany) equipped with an energy-dispersive spectrometer.

2.3. Experimental Methods

The experiment flow chart is shown in Figure 3.

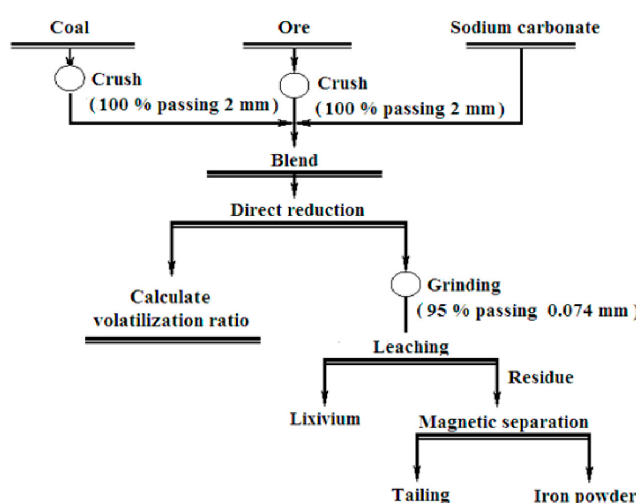


Figure 3. Experiment flow chat.

In each experiment, ore, sodium carbonate, and coal used were crushed to 100% passing 2 mm and were mixed. The mixed ratio of ore and coal was 1:0.25, while the amount of sodium carbonate changed. The amount of sodium carbonate was presented in the form of its relative mass percentage to ore. In each unit experiment, 41 g of the mixed materials was transferred to a lidded 100 mL graphite clay crucible. The crucible was then placed in a muffle furnace with an uncontrolled gaseous atmosphere at 1100 to 1150 °C. A distance of more than 30 mm was placed between the crucible lid and the powder mixture in the crucible to prevent them from making contact with each other. After cooling, the material was ground to 95% passing 0.074 mm at a grinding density of 50% and was then leached in a 1000 mL beaker at a water–solid ratio of 15:1, stirring speed of 360 r/min, leaching temperature of 75 °C, and leaching time of 30 min. The leaching yield of alumina (η_A) was then calculated from Equation (1):

$$\eta_A = \frac{c \cdot V_{ol}}{M \cdot \omega} \quad (1)$$

η_A , the leaching yield of alumina, %;

c , the aluminum oxide concentration of pregnant leach solution, g/L;

V_{ol} , the volume of pregnant leach solution, L;

M , the mass of ferric bauxite adopted in each unit experiment, g;

ω , the mass fraction of Al_2O_3 in ferric bauxite, %.

The leaching residues were transferred to a magnetic separator, and the recycle of iron powder from tailing was conducted with a magnetic separation of 0.14 T.

The sodium content in the crucible and the material mixture were measured before and after heating, and the sodium content difference was attributed to sodium volatilization. R was then calculated from Equation (2):

$$R = \frac{Q_1 \cdot (S_1 + S_2)}{Q \cdot S_2} \quad (2)$$

R , the sodium volatilization ratio, %;

Q_1 , the evaporated sodium infiltrated into crucible, g;

Q , the sodium content in material mixture, g;

S_1 , the area of crucible inside surfaces contacted with the material, cm^2 ;

S_2 , the area of crucible inside surfaces non-direct contacted with the material, cm^2 .

The form of sodium in iron powder and tailing was determined by SEM-EDS. The mineral composition of iron powder and tailing were examined by XRD.

3. Factors Affecting Sodium Volatilization

Previous research [14] indicates that the four significant factors that affect sodium volatilization are heating temperature, heating time, coal dosage, and sodium carbonate dosage. Coal dosage is mainly determined by the hematite and goethite content of ferric bauxite. Hence, it cannot be changed to reduce sodium carbonate consumption. Thus, the effects of heating temperature, heating time, and sodium carbonate dosage on sodium volatilization were measured in this study with a constant coal dosage. The parameter scales were given according to the optimum conditions obtained by the previous research [18] because carbonate consumption reduction must be achieved on the premise of synthetically recovering iron and aluminum. In the Box–Behnken design, the fluctuation range of each parameter scale is 10%. The experimental results are shown in Table 4, the variance analysis of the regression models is shown in Table 5.

Table 4. Box–Behnken design and experimental results.

No.	Factors			R (%)
	Heating Temperature ($^{\circ}\text{C}$)	Heating Time (min)	Sodium Carbonate Dosage (%)	
1	1100	50	85	7.63
2	1100	40	85	10.44
3	1100	45	90	9.44
4	1100	45	80	11.17
5	1150	50	80	7.17
6	1150	50	90	9.25
7	1150	40	80	8.05
8	1150	40	90	10.03
9	1150	45	85	7.61
10	1150	45	85	7.59
11	1150	45	85	7.53
12	1150	45	85	7.64
13	1150	45	85	7.55
14	1200	45	80	10.34
15	1200	45	90	10.06
16	1200	40	85	12.28
17	1200	50	85	13.01

Table 5. Variance analysis of regression models.

Source	Sum of Squares	df	Mean Squares	F	p -Value Prob > F	Result
Linear	44.75	9	4.97	2511.20	0.0001	-
2F1	41.09	6	6.85	3458.63	0.0001	-
Quadratic	11.53	3	3.84	1940.79	0.0001	suggested
Cubic	0.00	0	-	-	-	-
Pure Error	7.92×10^{-3}	4.00	1.98×10^{-3}	-	-	-

The confidence analysis of the quadratic model is shown in Table 6.

Table 6. Confidence analysis of the quadratic model.

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	7.58	1	0.57	6.23	8.94	-
A-Temperature	0.88	1	0.45	−0.20	1.95	1.00
B-Time	−0.47	1	0.45	−1.54	0.61	1.00
C-Sodium Carbonate	0.26	1	0.45	−0.82	1.33	1.00
AB	0.89	1	0.64	−0.63	2.4	1.00
AC	0.36	1	0.64	−1.16	1.88	1.00
BC	0.025	1	0.64	−1.49	1.54	1.00
A^2	2.44	1	0.63	0.96	3.92	1.01
B^2	0.81	1	0.63	−0.67	2.29	1.01
C^2	0.23	1	0.63	−1.25	1.71	1.01

Where, df is the degree of freedom (a non-dimensional number); F is the homogeneity test of variance (a non-dimensional number); P is the probability of obtaining a result that is at least as extreme as the actually observed result, given that the null hypothesis is true (a non-dimensional number); VIF is the variance inflation factor (a non-dimensional number).

The variance analysis of the regression models (Table 5) shows that the quadratic model has a satisfactory fitting effect. The confidence analysis (Table 6) shows that the quadratic model should be the chosen model. The response surfaces of the time–sodium carbonate dosage plot and their corresponding contours are shown in Figure 4.

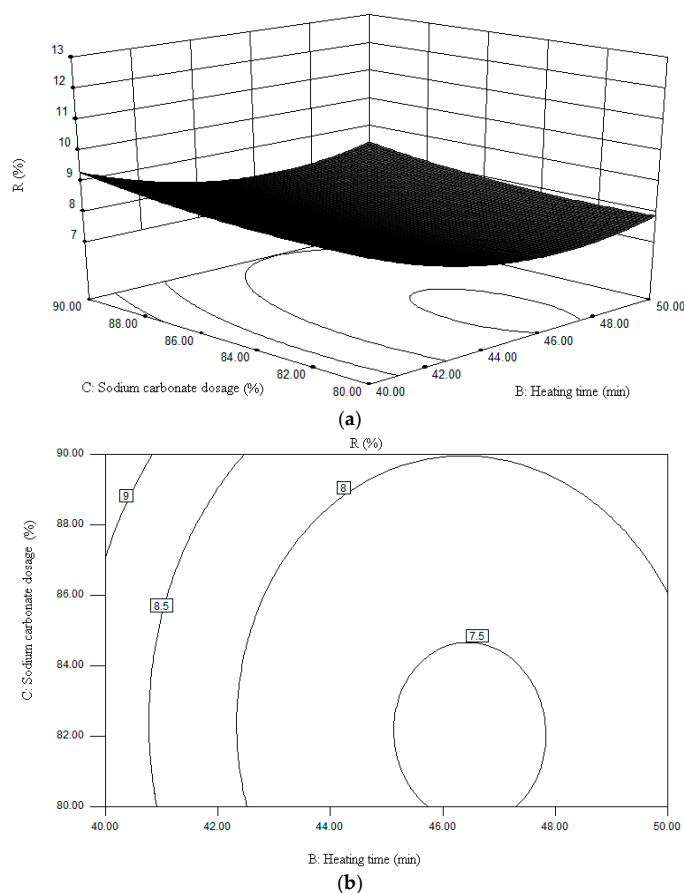


Figure 4. (a) Response surfaces of heating time–sodium carbonate dosage plot and (b) contour of heating time–sodium carbonate dosage plot.

The response surfaces of the temperature–sodium carbonate dosage plot and their corresponding contours are shown in Figure 5.

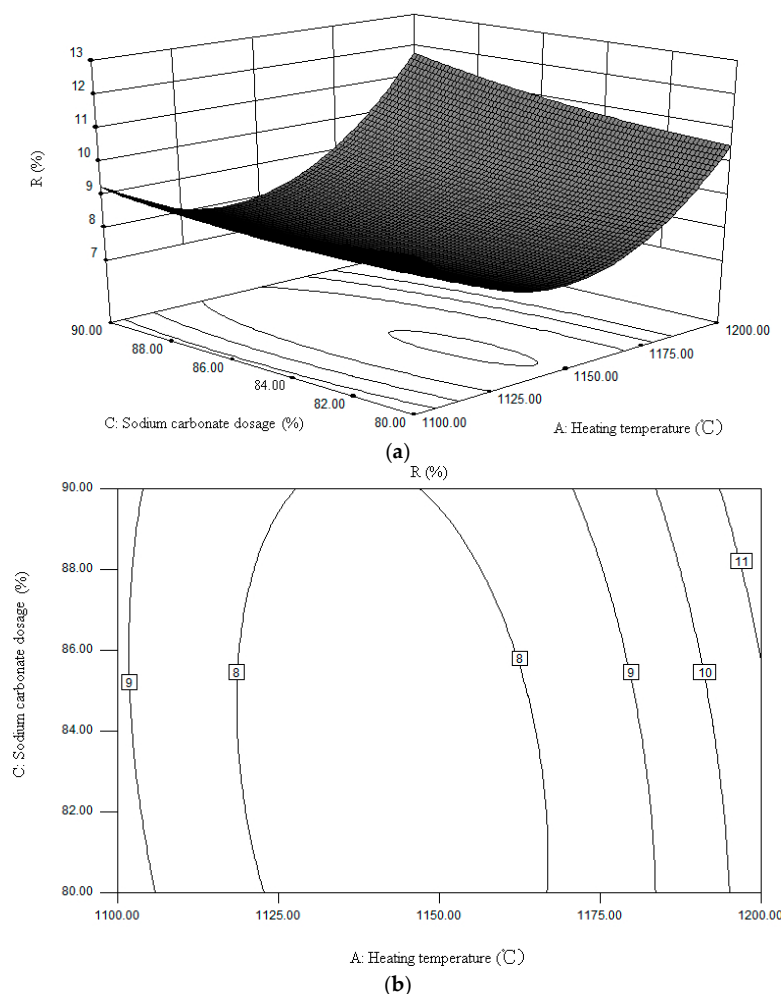


Figure 5. (a) Response surfaces of heating temperature–sodium carbonate dosage and (b) contour of heating temperature–sodium carbonate dosage plot.

The response surfaces of the temperature–time plot and their corresponding contours are shown in Figure 6.

Figure 4 shows that the effect of heating time on sodium volatilization is more significant than that of sodium carbonate dosage. Figure 5 shows that the effect of heating temperature is more significant than that of sodium carbonate dosage. Figure 6 shows that the effect of heating temperature on sodium volatilization is more significant than that of heating time. The descending order of their magnitude is as follows: heating temperature > heating time > sodium carbonate dosage.

Figure 6 shows that R initially decreases and then increases with an increase in heating temperature and that the minimum R is reached at 1150 °C. Some verification experiments were conducted to determine the cause of this minimum R .

Under the reaction conditions explained in Section 2.3, the curve between η_A and heating temperature is shown in Figure 7. η_A initially decreases and then increases with an increase in heating temperature and reaches its peak at 1100 °C. After reacting with an aluminum-bearing mineral, most sodium carbonate molecules are converted into crystal sodium aluminate. These crystals crystallize better and become more stable at higher temperature, hence the minimum R is reached at 1150 °C instead of 1100 °C.

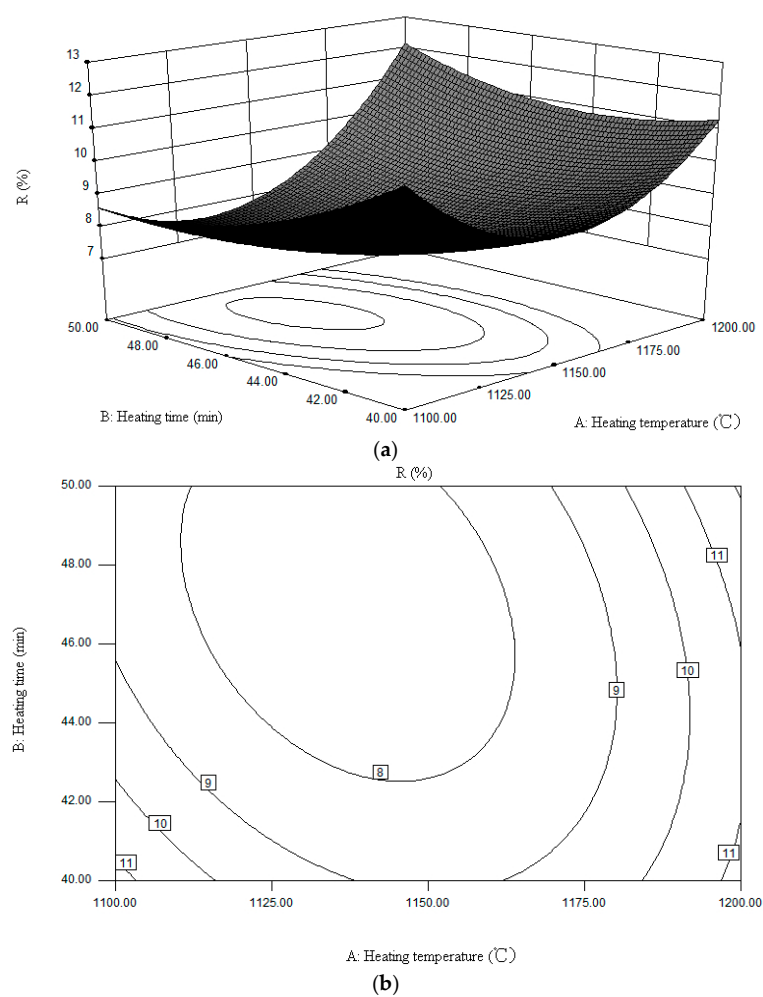


Figure 6. (a) Response surfaces of heating temperature–time and (b) contour of heating temperature–time plot.

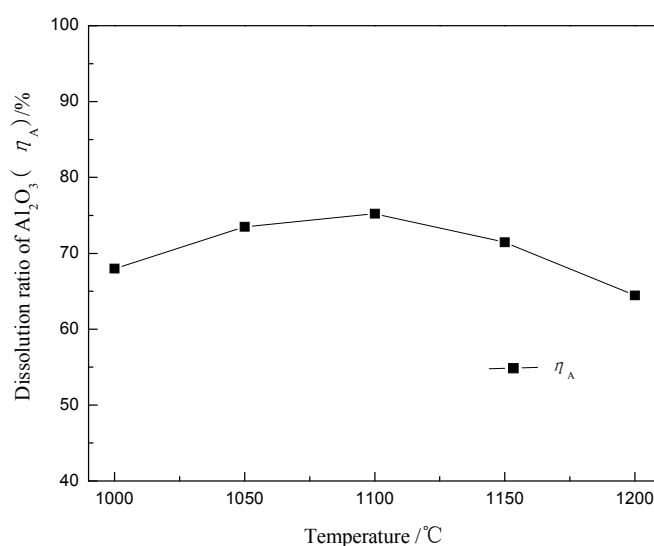


Figure 7. Plot between η_A and temperature.

Table 6 shows that the quadratic model of the effect of heating temperature, heating time, and sodium carbonate dosage on R (sodium volatilization ratio) can be described by Equation (3).

$$R = 2.44A^2 + 0.81B^2 + 0.23C^2 + 0.89AB + 0.36AC + 0.025BC + 0.88A - 0.47B + 0.26C + 7.58 \quad (3)$$

The changing rate of R with A , B , and C can be determined by calculating the partial derivative of R with respect to heating temperature (A), heating time (B), and sodium carbonate dosage (C) as described by Equations (4)–(9).

$$\frac{\partial R}{\partial A} = 4.88A + 0.89B + 0.36C + 0.88 \quad (4)$$

$$\frac{\partial^2 R}{\partial A^2} = 4.88 \quad (5)$$

$$\frac{\partial R}{\partial B} = 1.62B + 0.89A + 0.025C - 0.47 \quad (6)$$

$$\frac{\partial^2 R}{\partial B^2} = 1.62 \quad (7)$$

$$\frac{\partial R}{\partial C} = 0.46C + 0.36A + 0.025B + 0.26 \quad (8)$$

$$\frac{\partial^2 R}{\partial C^2} = 0.46 \quad (9)$$

Analysis shows that $\frac{\partial^2 R}{\partial A^2} = 4.88 > \frac{\partial^2 R}{\partial B^2} = 1.62 > \frac{\partial^2 R}{\partial C^2} = 0.46 > 0$, which means that all these curves are concave and have no flex point. Hence, the larger the second derivative becomes, the more rapidly R changes. Given that the second derivative of heating temperature is 3.01 and 10.61 times larger than that of heating time and sodium carbonate dosage, respectively, heating temperature has a much stronger dependence on R than the other two factors, or its effect is more significant than that of the other two factors. Furthermore, the reaction between bauxite and sodium carbonate accelerates as temperature increases and is conducted more thoroughly as heating time increases. The interactions between A , B , and C are also presented in Table 6.

Our previous research [14,15] indicates that η_A and R increase with an increase in sodium carbonate dosage. However, given that the effect of heating temperature is larger than that of sodium carbonate dosage, the effect of sodium carbonate dosage on R can be ignored in some cases. Hence, at a low temperature and high sodium carbonate dosage, a high η_A and a low R may be simultaneously achieved.

4. The Verification Test and the Possibility of Separation of Iron

4.1. Alumina Leaching and Sodium Volatilization

To verify the effects of restraining method on η_A and the total sodium volatilization amount per unit mass ferric bauxite (V), some verification tests were conducted as shown in Table 7.

Table 7. Verification of restraining method on sodium volatilization and alumina leaching.

Items	Sodium Carbonate Dosage/%	Temperature/°C	Time/min	η_A /%	R /%	V /(g/kg Ferric Bauxite)
Optimum conditions	85	1150	45	75.92	7.59	64.50
Verification tests	100	1100	35	73.53	7.44	74.50
	110	1100	35	74.91	6.38	70.00
	120	1100	35	76.02	5.22	62.50

Under the conditions of heating temperature of 1100 °C, heating time of 35 min, sodium carbonate dosage of 120%, the obtained η_A is 76.02%, which is higher than 75.92%, the value obtained under previous optimum conditions. However, when sodium carbonate dosage is 100% or 110%, η_A would be only 73.53% or 74.91%, respectively. That means η_A will not increase until sodium carbonate dosage rise enough (e.g., from 85% to 120%). Analogously, V would reduce only when the sodium carbonate dosage achieve 120%. If not, it would be increased.

4.2. Tailing

Tailing is the residue of magnetic separation. The XRD pattern of tailing obtained with the restraining method is shown in Figure 8, and the SEM-EDS images are shown in Figure 9.

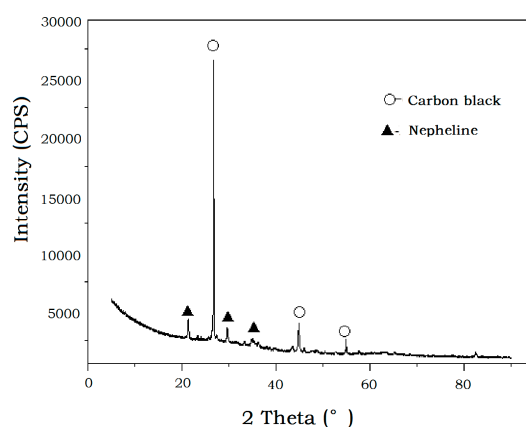


Figure 8. XRD pattern of tailing.

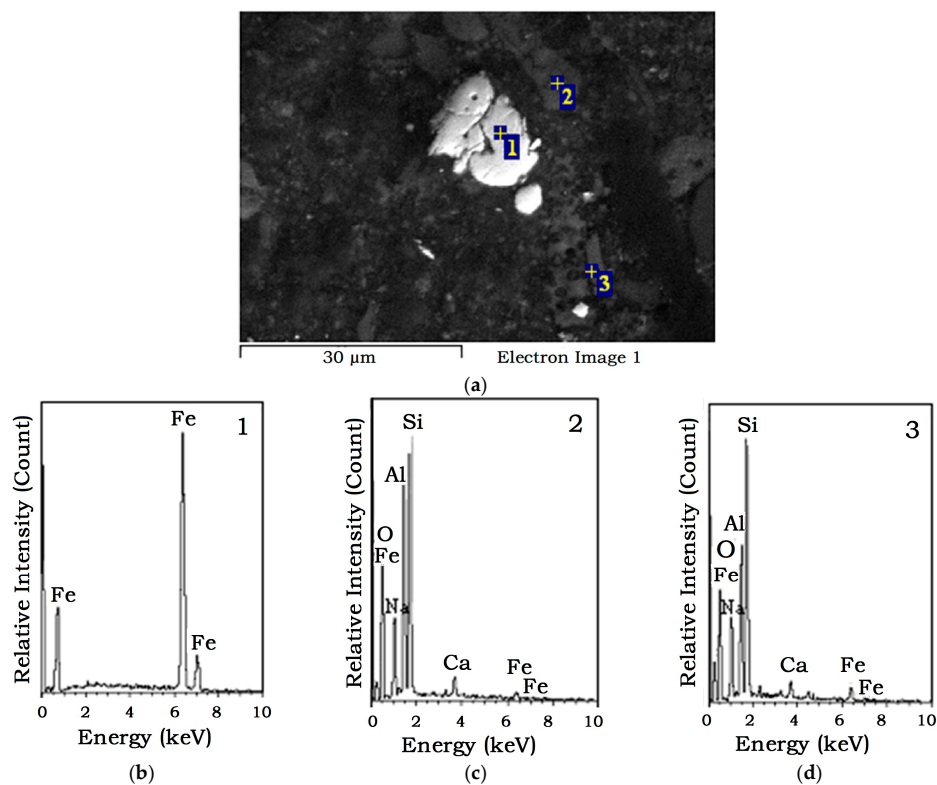


Figure 9. (a) SEM image of tailing; (b) EDS image of point 1; (c) EDS image of point 2 and (d) EDS image of point 3.

Figure 8 shows that nepheline is the main sodium-bearing mineral in tailing. Combined with XRD pattern, Figure 9b is the iron powders lost in tailing, and Figure 9c,d are the nephelines and Figure 9 shows that the sodium in tailing is mainly symbiotic with aluminum and silicon. A comparison of Figures 8 and 9 proves that the gangue granules in the SEM images are mainly nepheline. Given that the A/S of the ferric bauxite adopted is only 2.7, the generation a finite amount of nepheline is acceptable. Kaolinite is the main silicon-bearing mineral in ferric bauxite ore, and sodium carbonate is the main composition of the material. Hence, nepheline is inevitably generated from the reaction between kaolinite and sodium carbonate. The carbon black shown in Figure 8 is the residue of coal.

4.3. Iron Powder

Iron powder is the concentrate of magnetic separation. The XRD and SEM-EDS patterns of iron powder obtained with the restraining method are shown in Figures 10 and 11 respectively.

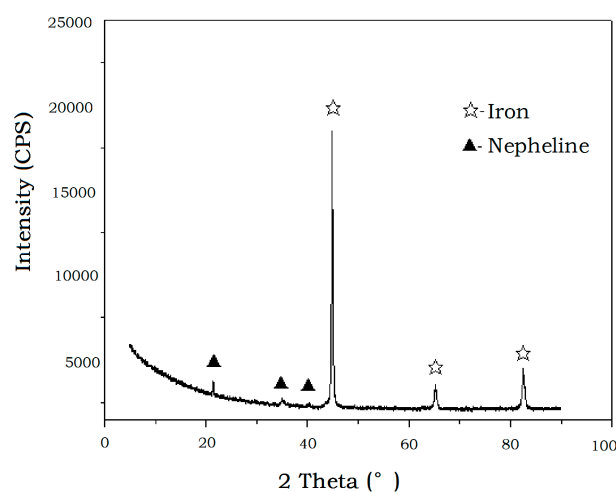


Figure 10. XRD pattern of iron powder.

Figure 10 shows that nepheline is the main gangue mineral in iron powder. Combined with XRD pattern, Figure 11b,c are nepheline and iron powder, respectively. Figure 11b,c show that most of the granules in iron powder are large enough and achieve monomeric liberation, which supposedly indicate the removing possibility of nepheline. If nepheline can be removed by beneficiation, then the quality of iron powder is not affected. Otherwise, the remaining sodium inevitably becomes a harmful component.

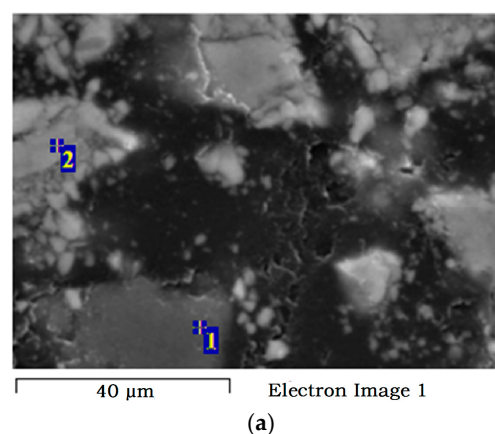


Figure 11. Cont.

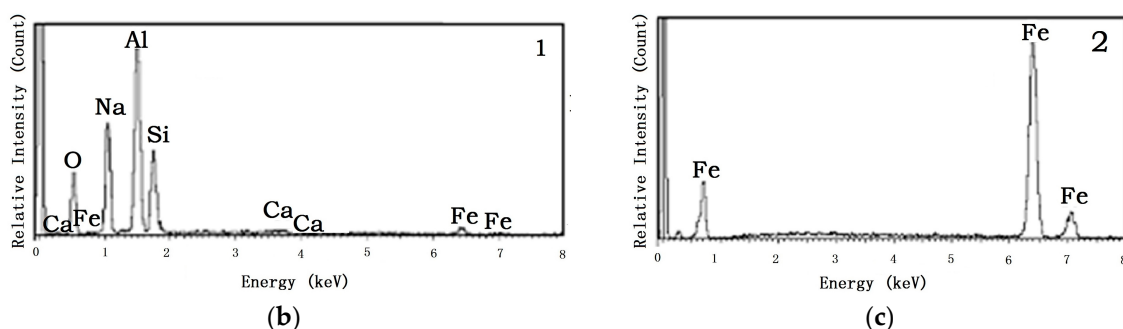


Figure 11. (a) SEM image of iron powder; (b) EDS image of point 1 and (c) EDS image of point 2.

5. Conclusions

The effects of heating temperature, heating time, and sodium carbonate dosage on sodium volatilization were investigated. Results show that the descending order of their magnitude is as follows: heating temperature > heating time > sodium carbonate dosage. Thus, R can be reduced by setting the heating temperature to the minimum possible value, and η_A can be increased by appropriately increasing the sodium carbonate dosage. In other words, a low R and a high η_A can be simultaneously achieved at a low heating temperature and high sodium carbonate dosage. Actually, it is verified that η_A would increase only when sodium carbonate dosage rise enough (e.g., from 85% to 120%).

With sodium volatilization restrained, more sodium is distributed in the solid phase. Nepheline is the main sodium-containing mineral in iron powder, and almost all of its granules are large enough and achieve monomeric liberation, which indicates the removing possibility of nepheline. The restraining process can theoretically be conducted harmlessly.

Acknowledgments: This study was supported by the National Natural Science Foundation of China by a grant number 51304012, the Found of State Key Laboratory of Mineral Processing by a grant number BGRIMM-KJSKL-2015-08, and the Open Foundation of the State Key Laboratory of Advanced Metallurgy (USTB) by a grant number KF 13-04 and 13-05.

Author Contributions: Wentao Hu and Chuanyao Sun conceived and designed the experiments; Xinwei Liu and Xuqin Duan performed the experiments; Huajun Wang contributed materials and analysis tools; Wentao Hu wrote the paper; Huajun Wang made the final modification.

Conflicts of Interest: The authors declare no conflict of interest.

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