Y. L. CONG, Z. J. HE, J. H. ZHANG, Q. H, PANG

ISSN 0543-5846
METABK 57(1-2) 75-78 (2018)
UDC – UDK 669.71:535-21:628.336:534.37:66.094.1-546.26:538.11-041=111

EXPERIMENTAL STUDY ON IRON RECOVERY BY MICROWAVE CARBON HEAT REDUCTION-MAGNETIC SEPARATION FROM RED MUD

Received – Primljeno: 2017-09-08
Accepted – Prihvaćeno: 2017-11-10
Original Scientific Paper – Izvorni znanstveni rad

To explore new ways to recycle red mud, this paper study on iron recovery by microwave carbon heat reduction-magnetic separation from red mud. Using orthogonal experimental method， study the effect of carbon to oxygen ratio, microwave power, heating time and slag former ratio on recovery of iron. The results show that microwave power is the main factor affecting iron recovery. After optimized by orthogonal experimental results, in the case when carbon to oxygen ratio is 1,5, microwave power is 3 000 W, heating time is 45 min, slag former ratio is 5 %， the grade and recovery of iron reach to 64,58 % and 90,64 % respectively. Analyzed comprehensivly by X-ray diffraction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometer (EDS), Fe_2O_3 in red mud is reduced to Fe_3O_4 and Fe by microwave carbon heat reduction.

Key words: red mud, microwave, carbon heat reduction, magnetic separation

INTRODUCTION

Red mud is the polluting waste residue of alumina plant when producing alumina. According to the difference between technics and technological level of alumina production, the average yield of 1 ton of alumina is produced with 1,0 ~ 2,0 tons of red mud [1]. Because of the high basicity and production and never being solved in an effective way, it is bringing serious pollution to the surrounding water, soil and atmosphere. In recent years, with the increasing attention to the environmental hazards caused by the piling-up of red mud, as well as the strict requirements of the state environmental protection, the emission of red mud has been greatly improved. But piling-up of red mud is still a serious problem. If it can not being solved in time and efficiently, it will bring great harm to the environment. Therefore, it is of great significance to study the comprehensive utilization and recovery of red mud [2-4].

For the resolution and recovery of red mud, domestic and foreign researchers have conducted relevant researches and testing. For example, valuable metals can be extracted from red mud [5,6], making adsorbent and catalyst[7,8], also it can be used to make cement, ceramics and insulation materials[9-11]. Microwave energy is an efficient and clean energy source. In this paper, microwave carbothermal reduction technique is adopted to carry out direct reduction of red mud with carbon, investigating the recovery effect of iron, and providing a new method of recycling and industrial production of red mud

Y. L. Cong, Z. J. He, J. H. Zhang, Q. H. Pang. School of Materials and Metallurgy, University of Science and Technology Liaoning, China Corresponding author: Z. J. He, doctor, professor; E-mail: hzhj2002@126.com.

EXPERIMENT

Experimental material

The main chemical compositions of red mud for experimental are shown in Table 1, the chemical compositions of coal and slag former in the carbothermal reduction are shown in Tables 2 and 3.

Table 1 **Chemical compositions of red mud / wt, %**

Fe_2O_3	Al_2O_3	SiO_2	CaO	MgO	Na_2O
40,23	16,95	19,53	2,13	0,19	3,73
K_2O	TiO_2	S	P	Burning loss	
0,19	3,48	0,22	0,07	13,28	

Table 2 **Chemical compositionsf coal / wt, %**

Elements	FC_d	V_d	A_d
Content	79,31	14,25	6,44

Table 3 **Chemical compositions of slag former / wt, %**

Elements	SiO_2	CaO	Others
Content	2,95	80,01	17,04

Table 4 **The factors and levels**

Factor	Level		
C/O	1,2	1,5	1,8
Microwave power / W	1 000	2 000	3 000
Heating time / min	25	35	45
Slag former ratio / %	0	5	10

Table 5 **Orthogonal experimental design**

Experimental scheme	C/O	Microwave power / W	Heating time / min	Slag former ratio / %
1	1,2	1 000	25	0
2	1,2	2 000	35	5
3	1,2	3 000	45	10
4	1,5	1 000	35	10
5	1,5	2 000	45	0
6	1,5	3 000	25	5
7	1,8	1 000	45	5
8	1,8	2 000	25	10
9	1,8	3 000	35	0

Experiment method

The orthogonal experimental method was adopted in this paper, mainly studied C/O, microwave power, heating time and slag former ratio on recovery iron effect. The table of factors and levels is shown in Table 4.

According to the information of Table 4, nine experiments can be designed as shown in Table 5.

RESULTS AND ANALYSIS

Effect of experimental factors on iron recovery

After the reactants broken and grinded，passing the magnetic tube under the magnetizing current of 2,5 A. Magnetic separated two times to ensure that the concentrate is not lost. The grade and recovery of iron is shown in Table 6.

As Table 6 shown, the grade of iron is 52,20 % ~ 63,65 % and the recovery of iron is 65,16 % ~ 88,17 % after magnetic separation. The iron grade and recovery of Experiment 1 are the lowest in all experiments, and the grade of iron and recovery are the highest in Experiment 5.

In order to research the effect of each factor on iron recovery in depth, extreme difference analysis method is introduced into analyze the data in this paper. That is to say, the importance of a factor to the experiment is judged according to the range. According to the experimental results obtained in Table 6, calculating the average of each factor at each level, extreme difference is obtained by subtracting the maximum from the minimum.The effect of each factor on iron recovery was shown in Table 7.

The greater the range, and the greater the effect of the corresponding factor in the experiment. So, it can be concluded that the effect of iron recovery from big to small is microwave power, heating time, C/O and slag former ratio. Thus, microwave power is the main factor affecting the recovery of iron.

Optimization experiments and results

In order to verify that microwave power is the main factor affecting the recovery of iron, two optimized experimental schemes are designed as Table 8 shown.

The optimized experiments are carried out according to the same experimental method, and the same magnetic separation and testing, the experimental results are shown in Table 9.

As can be seen from Table 9 that the iron recovery of the two optimized experiments are 89,33 % and 90,64 % respectively, compared with the original orthogonal experiments, the iron recovery has been improved. Scheme 2 compare with scheme 1, only microwave power is increased by 1 000 W, but the grade and recovery of iron increase by about 1 % respectively. It verifies that microwave power is the main important factor affecting the experimental results.

XRD analysis

The XRD spectra of red mud and optimizing experimental scheme 2 are shown in Figure 1.

Refering to Figure 1(a), red mud was complicated and had many kinds of elements. The Fe in red mud mainly existed in the form of Fe_2O_3, and a small amount of Fe existed in minerals such as spinel ($MgFe_2O_4$). According to Figure 1(b), Fe_2O_3 is almost completely reduced to Fe, Fe_3O_4 and FeO after the carbothermal reduction. Some other mineral compositions also vary slightly.

Table 6 **Grade and recovery of iron after magnetic separation / wt, %**

Result	1	2	3	4	5	6	7	8	9
Grade	52,20	59,71	60,08	54,67	63,65	61,01	56,33	57,18	62,47
Recovery	65,16	81,95	83,23	78,85	88,17	82,62	80,07	80,83	86,04

Table 7 **Effect of each factor on iron recovery**

Level	C/O	Microwave power / W	Heating time / min	Slag former ratio / %
Average 1	76,78	74,69	76,20	79,79
Average 2	83,21	83,65	82,28	81,55
Average 3	82,31	83,96	83,82	80,97
Range	6,43	9,27	7,62	1,76

Table 8 **Optimizing experimental schemes**

Scheme	C/O	Microwave power / W	Heating time / min	Slag former ratio / %
1	1,5	2 000	45	5
2	1,5	3 000	45	5

Table 9 **Optimizing experimental results**

Scheme	Grade	Recovery
1	63,79	89,33
2	64,58	90,64

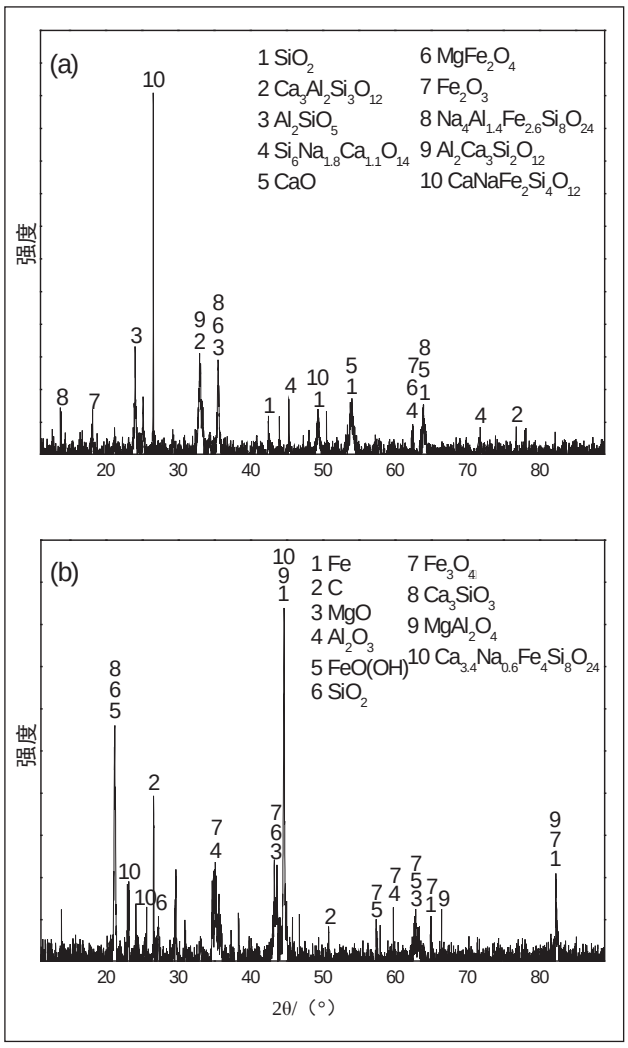


Figure 1 The XRD spectra of red mud(a) and optimizing experimental scheme(b)

SEM and EDS analysis

The product morphology of optimizing experimental scheme 2 is shown in Figure 2

From the whole of the reduction product, it is mostly black materials, and there are silver white bead iron particles on the surface. In order to study the organization structure of the reduction product, it should be polished and observed under SEM, the pictures are shown in Figure 3.

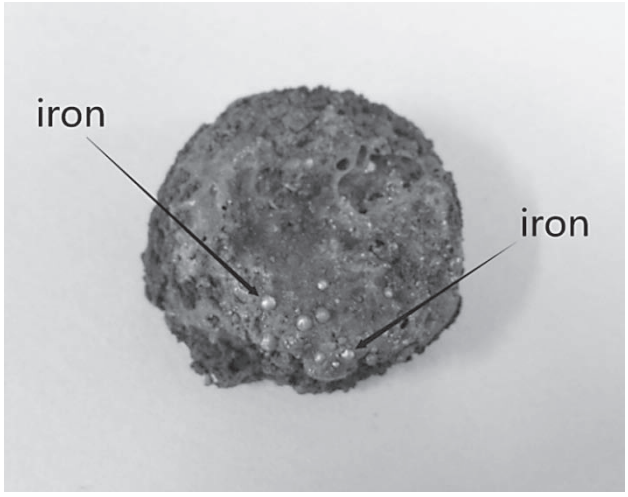


Figure 2 The reduction product

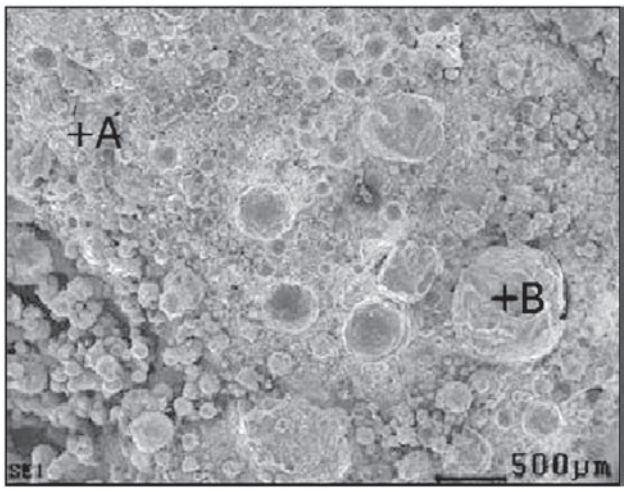
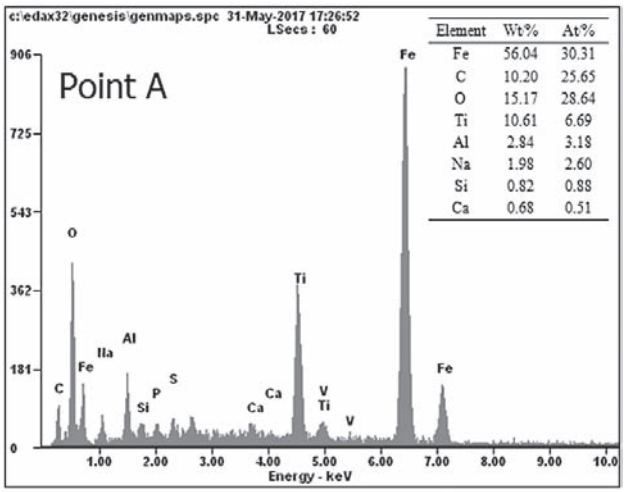


Figure 3 SEM of the reduction product



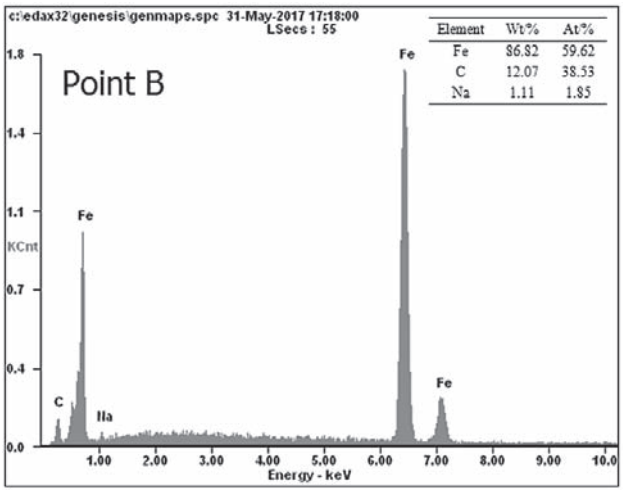


Figure 4 EDS of point A and B

From Figure 3, it has been found that surface structure of the product is compact and rough, and which is accompanied by many spherical substances in different sizes. There are a lot of spherical substances, and the lower left corner is similar to the flocculent slag phase. EDS analysis carried out at point A and point B is shown in Figure 4.

EDS analysis presents that there are three main elements of C, O and Fe at point A, and there are two elements in point B, Fe and C respectively. With the analysis of XRD spectrum, the elements of Fe and O at point A mainly form the oxide Fe_3O_4, while the element C at point A and point B is the residue after reaction, and other elements are elements in the slag system. The energy spectrum of point B shows that the content of Fe is the highest and relatively simple. According to its micro-morphology and XRD analysis, it can be concluded that the near spherical substance under the scanning electron microscope is the metal pellets composed by element Fe.

CONCLUSIONS

Red mud reduced by microwave carbon heating, the iron oxide is reduced and minor amount of iron beads generate. Microwave power is the main factor affecting the grade and recovery of iron.

According to the optimizing experimental results, when C/O is 1,5, microwave power is 3 000 W, heating time is 45 min, slag former ratio is 5%, the grade and recovery of iron reached 64,58 % and 90,64 %. Through the comprehensive analysis of XRD, SEM and EDS, it has been found that the reduced iron is mainly Fe_3O_4, and little amount of iron form.

Acknowledgements

This work is financially supported by the National Science and Foundation of China (No. 51474124 & 51504131) and Science and Foundation of Liaoning (NO.2015020192 & 20170540455) as well as Anshan technological program (NO. 4104).

REFERENCES

[1] Y. Cao, W. D.Li, Y. G.Liu. Properties of red mud and current situation of its utilization[J]. Bulletion of the Chinese Ceramic Society, 26(2007)1, 143-145.

[2] Brunori C, Cremisini C, Massanisso P. et al. Reuse of a treated red mud bauxite waste: studies on environmental compatibility[J]. Journal of Hazardous Materials, 117(2005)1, 55.

[3] X. L.Nan, T. A. Zhang, Y. Liu. Analysis of comprehensive utilization of red mud in China[J]. The Chinese Joural of Process Engineering, 10(2010)s1, 264-270.

[4] Y. Liang, J. N.Chen. Hazards and utilization of red mud[J]. Popular Science and Technology, 16(2014)7, 33-34.

[5] Smirnov D I,Molchanova T V.The investigation of sulphuric acid sorption recovery of scandium and uranium from the red mud of alumina production[J]. Hydrometallurgy, 45（1997）3, 249-259

[6] Z. K. Lu, J. Y. Gao. Process test of valuable metals extracting in high iron red mud[J]. Energy saving of nonferrous metallurgy, 31(2015)3, 17-19.

[7] Wang S, Ang H M,Tadé M O.Novel applications of red mud as coagulant, adsorbent and catalyst for environmentally benign processes[J]. Chemosphere, 72(2008)11, 1621.

[8] Oliveira A A S, Teixeira I F, Christofani T, et al. Biphasic oxidation reactions promoted by amphiphilic catalysts based on red mud residue[J]. Applied Catalysis B Environmental, 144(2014)2, 144-151.

[9] G. K. Ren. Production of cement using modified red mud as raw material[J]. Environmental Protection of Chemical Industry, 28(2008)6, 68-72.

[10] W. Liu, J. Yang. B. Xiao. Application of Bayer red mud for iron recovery and building material production from alumosilicate residues[J]. Journal of Hazardous Materials, 161(2009)1, 474-478.

[11] L. Shi, N. Wang, C. Pang. Research progress of utilizing red mud as a resource of building material[J]. New Building Material, 36(2009)1, 20-24.

Note: The responsible translator for language English is doctor W.L. Zhan - Univerisity of Science and Technology Liaoning, China.