TUBE DIGESTION TECHNOLOGY FOR THE MID-QIAN BAUXITE[①]

Liu Zigao, Yang Changshi
Guiyang Aluminium Magnesium Design and Research Institute, Guiyang 550001

ABSTRACT Satisfactory results are obtained when the mid-Qian sedimentary diasporic bauxite is subjected to Bayer digestion at 245~ 280 ℃ with the addition of certain amount of lime. The conditions influencing the digestion process, according to their importance, are as follows: temperature, caustic soda concentration of starting liquor, residence time and burden molar ratio, etc. Conditions for both traditional and tube digestions have been determined. Adding excessive lime and adopting single-stream tube digestion technology are of great advantage for retarding velocity of scale growth on the surface of heat exchangers and for treating and utilizing red mud etc. in the meanwhile.

Key words bauxite lime tube digestion scale

1 INTRODUCTION

The proved reserve of the sedimentary bauxite in the mid-Qian district (i. e. the middle part of Guizhou province) is about 4. 2 hundred million tonnes. Both morphology and structure of this bauxite are not complicated, and its mineralogical composition and chemical component are almost the same, so it is easy to carry out Bayer digestion at 245 ℃ or above.

The laboratorial experiments and pilot test of 1. 7 m^3/h bauxite slurry Bayer digestion with indirect heating have proved, however, that tube digestion process at 270~ 280 ℃ may be better for mid-Qian bauxite.

High-temperature digestion is an essential measure to improve traditional Bayer process for producing alumina. It makes not only circulating efficiency increase and heat consumption of digestion decrease, but also caustic soda concentration of evaporated mother liquor and heat consumption of evaporation lower.

So long as the scaling velocity on the heat exchange surface of bauxite slurry preheater is retarded, and cleaning the scale is considered as part of the design and operation, it will be easy to realize high-temperature Bayer digestion with indirect heating.

The red mud derived from Bayer digestion process can be subjected to denatrium treatment by adding lime milk, and the denatrium efficiency can be 65% or so; but, compared with lime Bayer process, it will increase molar ratio of digested liquor by 0. 05~ 0. 06.

This paper discussed behaviors of silicon and titanium minerals in mid-Qian bauxite during pre-desilication and preheating processes prior to digestion, and studied Bayer digestion conditions and tube digestion technology.

2 MATERIALS AND EQUIPMENTS

2. 1 Materials

Three bauxite samples, provided by Guizhou Aluminium Smelter, were from the first and the second phase projects and the preliminary production respectively, and ground to pass 100 mesh, dried and kept in desiccators.

The lime was taken from Guizhou Aluminium Smelter also, and ground to pass 100 mesh and calcined at 1 000 ℃ for 1 h.

Its chemical components are as follows:

① Received Sep. 6, 1995; accepted Jan. 23, 1996

CaO_{tot} 93.1%, CaO_f (available CaO) 91.7%, MgO 2.5%.

The starting liquor for digestion was made from evaporated mother liquor of Guizhou Aluminium Smelter. The distilled water or NaOH or NaCl was added to the evaporated mother liquor, if necessary, to make its molar ratio be 3.3, and $[Na_2O]_c$ (caustic soda concentration) / Na_2O_{tot} be about 0.9.

2.2 Equipments

Main experimental facilities are:

Multiple autoclave set, t = 300 ℃;

A 5 L autoclave, p = 25 MPa, t = 400 ℃, 6 kW, with electromagnetic reciprocating stirrer;

SN-lux-pox type polarization microscope;

RIGAKU type X-ray diffraction system.

3 RESULTS AND DISCUSSION

3.1 Components of samples of the mid-Qian bauxite

The XRD pattern and IR spectrum of sample No. 1 are shown in Figs. 1 and 2, respectively. The mineralogical composition of sample No. 1 is as follows: diaspore 69%, boehmite 10%, kaolinite 9%, illite 5%, anatase 1%, rutile 1%, etc. The essential chemical components of samples used are given in Table 1. By screen analy-

Fig. 1 The XRD pattern of the bauxite sample No. 1

1 —diaspore; 2 —kaolinite;
3 —boehmite; 4 —illite; 5 —anatase

Fig. 2 The IR spectrum of diaspore in the bauxite sample No. 1

Table 1 The essential chemical components of samples of the mid-Qian bauxite (%)

No.	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	CaO
1	74.33	6.38	0.83	3.58	0.62
2	71.10	8.82	1.66	3.44	0.43
3	66.01	12.64	2.88	3.21	0.20
No.	Na_2O	K_2O	LOI	A/S	
1	–	–	13.91	11.65	
2	–	–	13.67	8.06	
3	0.06	0.13	13.42	5.22	

Notes: LOI —loss on ignition;
A/S —weight ratio of Al_2O_3 to SiO_2.

sis, the grain size distribution is determined: +0.124 mm 3.5%, and +0.088 mm 10%.

3.2 Behaviors of silicon and titanium minerals during pre-desilication and preheating processes prior to digestion

Pre-desilication efficiency of samples can be improved with increasing temperature, holding time and amount of lime addition, but reduced with increasing ratio of liquid to solid.

The desilication process runs throughout digestion process of alumina. At first the velocity of silicon entering into solution is larger than that of silicon precipitating in siliceous sediment, thereafter mass silicon precipitates in siliceous sediment untill desilication reaction tends to equilibrium. Less than 40% of the total SiO_2 in bauxite can be removed by pre-desilication prior to digestion at 90 ℃ for 3~ 4 h.

The desilication reaction can accelerate with increasing temperature, for instance, the time in which the desilication reaction comes up to equilibrium can be: 30 min at 140 ℃, 20 min at 160 ℃, and 10 min at 180~ 200 ℃. It means that the residence time in which bauxite slurry passes through each preheater must be reduced with increasing preheating temperature to prevent scale from depositing on the surface of heat exchange.

In the absence of lime in bauxite slurry, titanium minerals in samples would form sodium titanate ($Na_2O \cdot mTiO_2 \cdot nH_2O$). Owing to the fact that the forming velocity of sodium titanate is about 6~ 9 times greater than that of calcium titanate at a certain temperature, especially above 200 ℃, scaling danger caused by titaniferous sediment can be decreased in the presence of lime in the bauxite slurry. The forming quantity of calcium titanate ($(Ca, Mg)O \cdot TiO_2 \cdot nH_2O$) would be: very little below 180 ℃ in a short time; 1% at 200 ℃ in 10 min; 1.6% at 240 ℃ in 10 min; 2% ~ 3% at 260~ 280 ℃ in 10 min.

3.3 Measures against scaling

Compared with other bauxites, scale growth caused by siliceous and titaniferous sediments is relatively slow and acceptable during preheating and digestion processes for mid-Qian bauxite slurry, because the original chlorite and rutile etc. are still present in the red mud. The measures for retarding and preventing scale from generating on the surface of heat exchange are as follows:

(1) to improve the ratio of alumina to silicon dioxide;

(2) to raise pre-desilication efficiency;

(3) to add siliceous and titaniferous sediments to bauxite slurry as well as lime, making "growth scale" form in the stream of bauxite slurry instead of on the surface of heat exchange;

(4) to add certain amount of MgO to bauxite slurry so as to form porous scale, making clean easy and keeping reasonable heat transfer coefficient;

(5) to adopt a velocity of 2.5 m/s above instead of 1.5 m/s below from the beginning, and be sure to make the velocity of bauxite slurry passing through each preheater increase stage by stage with increasing preheating temperature. Even the velocity of 3~ 3.5 m/s for the last stage of preheater is feasible for retarding scaling, especially "settled scale";

(6) to choose multistage tubular exchanger with 8~ 12 stages, reducing temperature difference of bauxite slurry to retard the velocity of scale generating on the surface of heat exchange.

3.4 Digestion characteristics and digestion conditions

Bayer digestion characteristics of the mid-Qian bauxite are better than those of other diasporic bauxites found in China (Fig. 4).

Digestion conditions influencing alumina extraction efficiency, in the order of importance, are as follows: temperature, caustic soda concentration of starting liquor ($[Na_2O]_c$), residence time (t) and molar ratio of digested liquor (MR), etc. The results of typical Bayer digestion are given in Table 2.

The X-ray diffraction pattern of red mud derived from Bayer digestion for bauxite sample No. 1 is determined (see Fig. 3). Its mineralogical composition is hibschite 47%, cancrinite 32%, hydroxy calcium titanate 14%, perovskite

Fig. 3 The XRD pattern of red mud derived from Bayer digestion for sample No. 1 at 250 ℃

1 —hibschite; 2 —cancrinite; 3 —hematite

Table 2 Typical Bayer digestion results of the mid-Qian bauxite

t / ℃	$[Na_2O]_c$ / g·L^{-1}	Time / min	MR	CaO_f / %	E / %
245	220	60	1.50	6.5	98.40
250	200	90	1.37	4.5	96.14
260	160	15	1.50	12.7	94.83
280	160	15	1.50	5.86	97.61
280	140	15	1.50	12.7	96.60

Notes: CaO_f —available CaO content in lime;
E —avaiable Al_2O_3 extraction efficiency.

2%, rutile 2%, and hematite 3%, etc. The relation between alumina extraction efficiency and temperature is shown in Fig. 4. Bayer digestion reaction approached to equilibrium at 245 ℃, but it would be of greater advantage for Bayer digestion process to carry out at 280 ℃.

Influence of caustic soda concentration of starting liquor on alumina extraction efficiency is shown in Fig. 5. It is evident that $[Na_2O]_c$ of starting liquor is no less than 220 g/L at 245 ℃ for Bayer digestion; while at 280 ℃, according to Table 2, it is advisable to choose $[Na_2O]_c$= 180 g/L.

The relation between alumina extraction efficiency and molar ratio (MR) of Na_2O/Al_2O_3 of digested liquor is shown in Fig. 6. Fig. 6 and Table 2 show that, at 245 and 280 ℃, it is easy to obtain satisfactory digestion results with MR less than 1.5 and E higher than 96.6%, and that the accumulation of sodium chloride in starting liquor seems to be harmful to alumina extraction.

Size distribution of +0.147 mm ≤10%, +0.095 mm ≤20% is satisfactory for Bayer digestion of the mid-Qian bauxite[1]. Comparing alumina extraction efficiency of floatation concentrate whose particle size is −0.074 mm > 95%

Fig. 4 The relation between alumina extraction efficiency and temperature

($[Na_2O]_c$= 200 g/L, time= 90 min, MR= 1.60)
1 —Xiuwen bauxite; 2 —Xin'an bauxite;
3 —Pingguo bauxite

Fig. 6 The relation between alumina extraction efficiency and molar ratio (MR) of Na_2O/Al_2O_3 of digested liquor

(t= 245 ℃, $[Na_2O]_c$= 220 g/L,
time= 90 min, CaO_f= 4.5%)
1 —NaCl 5 g/L; 2 —NaCl 10 g/L

with that whose particle size is − 0.074 mm > 70%, the former surpasses the latter by 0.3%.

It leaves no doubt that there is an influence of lime on alumina extraction efficiency of the mid-Qian bauxite when Bayer digestion is performed at temperatures below 300 ℃. Both activity and addition quantity of lime have influences on digestion velocity of alumina. Alumina extraction efficiency increases with increasing lime addition, but decreases gradually when lime addition is excessively increased. By lime-Bayer process combined soda contained in red mud decreases obviously, and the ferric oxide in it becomes about 5% below; besides, the newborn mineral is essentially hibschite containing various amount of silicon, which makes the red mud favourable for treatment and utilization[2, 3].

As stated above, the feasible Bayer digestion conditions are listed in Table 3.

Table 3 Bayer digestion conditions for the mid-Qian bauxite

Type of digestion	t / ℃	$[Na_2O]_c$ / $g \cdot L^{-1}$	Time / min	MR	CaO_f / %
Traditional	245~250	220~240	60	1.50~1.60	6~9
Tube	270~280	140~180	15	1.50~1.60	6~18

The settling performance of red mud derived from Bayer digestion of mid-Qian bauxite is excellent; it can be improved, moreover, by either increasing temperature or adding excessive lime into bauxite slurry.

4 CONCLUSION

Bayer digestion performance of the mid-Qian sedimentary diasporic bauxite is excellent, 96.6 % of available alumina extraction efficiency can be obtained under the condition of $[Na_2O]_c$ of starting liquor 140~180 g/L and CaO_f of lime additive 12% ~ 18% etc. Single-stream tube digestion at 270~280 ℃ for 15 min would be of great advantage for retarding scaling velocity on the surface of the bauxite slurry heat exchanger, therefore, intensify Bayer digestion process.

Owing to the fact that the essential newborn mineral hibschite is 50% and above and ferric oxide content is about 5% below in red mud, it is of particular advantage to transform red mud into active mixed material of portland cement by using lime calcination process at low temperature.

REFERENCES

1 Yang Chongyu, Wang Yanming, Long Yuanzhi. Process Technology of Alumina Production, (in Chinese). Beijing: Metallurgical Industry Publishing House, 1982.

2 Sichacov V M *et al*. Nonferrous Metals, (in Russian), 1984, (12): 43—46.

3 Malits N S. New Advance in Alumina Production with Series Process, (in Russian). Moscow: Metallurgical Industry Press, 1989.

(Edited by Li Jun)