Digestion Efficiency Improvement of Gibbsite-Boehmite Bauxite

Fengqin Liu, Zegang Wu, Songqing Gu, and Michael Ren

Abstract

Bayer digestion performance of gibbsite-boehmite bauxite is studied in this paper. The chemical and mineral compositions of a gibbsite-boehmite bauxite from Australia are analyzed and tested by XRF, XRD, and SEM. Test results show that the bauxite contains > 50% of alumina (gibbsite and boehmite) and 9% of silica (kaolinite and quartz). Its digestion efficiency is studied in detail with various digestion conditions such as temperatures, holding times and additives. A medium temperature digestion technology has been developed based on the study results and industrial tests in Chinese refineries: boehmite in the bauxite will be totally digested in just a few minutes with bits of lime addition for a high alumina recovery efficiency and most of the unreactive silica minerals in the bauxite are at a stable state with caustic consumption reduced by over 10%, alumina recovery efficiency increased by 2-3% compared with traditional digestion technologies. There is a great application prospect with this medium temperature digestion technology for the Australian bauxite.

Keywords

Gibbsite-boehmite bauxite • Bayer digestion • Digestionefficiency • Caustic consumption • Medium temperature• Holding time • Additive

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Introduction

Chinese alumina output reached 77.45 million tons in 2021, which exceeded half of the global total output by about 54.4%. Imported bauxite has become the major raw material for Chinese alumina refineries, in which the imported Guinea, Australia, and Indonesia bauxites are the main resource for Chinese alumina industry. One of the largest reserves of bauxite resource in the world is from Australia and its bauxite export has been relatively stable [1–4].

Most of the imported Australia bauxite is from north-east Australia with both gibbsite and boehmite as the major alumina minerals. Australian bauxite usually has a complex mineral composition. A study on digestion behaviors of gibbsite-boehmite bauxite has been carried out by some researchers. Qi [5] studied the digestion performances of boehmite in the bauxite at 180-240 °C for 30 min and the mineral compositions of the bauxite residues formed at 143 °C and 245 °C were also discussed. The study results show that boehmite and quartz cannot react at 143 °C. Both boehmite and quartz were completely reacted at 245 °C for 30 min. Jiang [6] studied the bauxite digestion at 270 °C and 280 °C and showed that the best digestion rate of the bauxite could be obtained at 270 °C for 10 min or 280 °C for 5 min. In addition, the relationship between the reaction rate of quartz and the recovery rate of alumina was also studied. Zhang [7] found that the total available alumina in a bauxite at 150 °C and 250 °C were 36.25% and 43.33% respectively, which showed that the high-temperature digestion process for the bauxite should be more suitable by the comparison between alumina lost by the quartz reaction and alumina recovery from the boehmite at high digestion temperature. It is well known that alumina loss can be caused by the reaction of quartz in the high-temperature digestion process. However, a detailed study should be carried out on what kind of digestion process including digestion temperatures, holding times and additives will be beneficial to

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obtain the best alumina recovery for the gibbsite-boehmite bauxite?

First, it is necessary to study the reaction behavior of different minerals in the gibbsite-boehmite bauxite during the high-temperature digestion process for its best Bayer digestion performance, which is of significance for the Chinese refineries to obtain better production benefit.

A study on the digestion efficiency of the Australia gibbsite-boehmite bauxite has been carried out and a medium temperature digestion technology has successfully been developed based on the study results.

Gibbsite-Boehmite Bauxite from Australia

Common Description of Gibbsite-Boehmite Bauxite Digestion

Based on the test results the north-east Australian bauxite is a type of gibbsite-boehmite bauxite, in which the major effective aluminum-bearing minerals are gibbsite and boehmite and the main silica-containing minerals are kaolinite and quartz. Gibbsite can react rapidly with caustic liquor at a lower temperature of 145 °C [8–10], while the higher digestion temperatures of more than 220 °C are needed for boehmite digestion [11, 12]. To ensure maximum dissolution of boehmite in the bauxite the higher temperatures above 220 °C are needed in the digestion process. But at the relatively high digestion temperatures and long holding times, more quartz dissolves, which consumes alumina and caustic in the liquor. Therefore, the key to the study on the digestion process of gibbsite-boehmite bauxite is to ensure the complete digestion of boehmite and to minimize the reaction of quartz [13–16].

The Australian bauxites from different origins have quite different chemical and mineral compositions due to the different mineralization characteristics. The chemical and mineral compositions and digestion performances of multiple bauxite samples from different origins were investigated in this paper. The effects of different digestion temperatures, holding times and additives on alumina to silica ratio (A/S) and caustic to silica ratio (N/S) in the bauxite residues were studied, as well as the bauxite and caustic consumption factors in the digestion process.

Chemical Compositions

The chemical composition of multiple samples of gibbsiteboehmite bauxite from the Australian origins A and B were analyzed by X-ray fluorescence spectroscopy (XRF) and chemical titration analysis. The results are shown in Table 1.

Mineral Compositions

The mineral compositions of the bauxite A1# and B1# were analyzed by X-ray diffraction analyzer (XRD) and the results are shown in Fig. 1.

It can be seen in Fig. 1 that there is almost no obvious difference in the mineral types in the different bauxite samples and only the difference in the individual mineral content in the bauxites. In the gibbsite-boehmite bauxite samples the major mineral compositions are basically the same, such as gibbsite, boehmite, kaolinite, quartz, hematite, anatase, etc. However, the peak intensity of hematite in the B1# sample is much higher than that in the A1# and the peak intensity of quartz is weaker. It can be expected that the reaction performances of the different samples may have certain differences in the digestion process.

Test Procedures and Analysis Instruments

Bauxite Grinding

The bauxite samples used in the experiments were ground by using vibrating milling. The test process had the following characteristics such as very short grinding time each, multiple sieving, and re-grinding of large particles. This grinding method is mainly to ensure that the particle size of the bauxite samples for digestion tests is less than 20 mesh, and the particles larger than 250 microns are more than 50%. This is just for a size distribution of the samples to be courser and more homogeneous.

Bomb Digestion

The bomb digestion equipment was used for bauxite digestion tests, which consists of three parts: molten salt heating furnace, bomb digesters, and operating control system. 6 separate bomb digesters with 150 ml each are configured in and heated by the molten salt. This equipment can ensure not only the same reaction temperature for the different bomb digesters but also independent operation for

 Table 1
 The chemical

 compositions of multiple samples
 of gibbsite-boehmite bauxite

Name	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	A/S	CaO	TiO2
A1#	9.33	9.72	51.83	5.56	0.07	3.13
A2#	9.25	10.77	51.68	5.59	0.08	2.97
A3#	8.63	10.39	52.09	6.01	0.07	3.09
B1#	7.92	13.97	51.16	6.46	0.09	2.38
B2#	8.28	15.02	51.11	6.17	0.06	2.61





them. The bomb rotating device and precise temperature control system is installed for the uniform stirring of the bomb digesters and accurate digestion temperature (± 1 °C).

Materials Characterization

The chemical compositions of the bauxite and residue samples were determined by XRF (mAX, AXIOS, Alamelo, The Netherlands). The X-ray diffractometer (PW1710, Philips, Amsterdam, The Netherlands) was used with Cu K α -radiation at 40.0 kV and 30.0 mA for the mineralogical study. The XRD tests were conducted within the 2θ range of 10-60° using a scanning speed of 0.1° ·min⁻¹. The samples for XRF and XRD tests were ground to pass through a 200-mesh sieve and dried in an oven at 105 °C for 2 h. The micrographs of the samples were observed using a scanning electron microscope (Regulus 8100, Hitachi, Japan) at an accelerating voltage of 15.0 kV. An EDS (ultra-DLD, Shimadzu, Kyoto, Japan) connected with SEM was used to perform elemental analysis on the sample particles. The elements with the content of $3\% \sim 20$ wt.% have the accuracy of relative error < 10%. And the elements with content greater than 20 wt.% have the higher accuracy of relative error <5%.

Test Program and Procedure

Test Procedure

The caustic liquor used in the digestion experiments was prepared with industrial caustic liquor adjusted by the analytically pure sodium hydroxide. The chemical compositions of the caustic liquor and bauxite samples were analyzed and the liquid-to-solid ratio is calculated according to their compositions.

The formula for calculating the dosage mass of bauxite (M) required per 100 mL of digestion liquor is as follows:

$$1.4 = \frac{N_k M \times S_B \times (N/S)_M \times 10}{M \times A_B \times 10 - M \times S_B \times (A/S)_M \times 10 + AO}$$

where N_K is the caustic concentration in the digestion liquor, (g/L); M is the dosage mass of bauxite required for per 100 mL of digestion liquor, (g); S_B is the SiO₂ mass fractions in the bauxite, $(N/S)_M$ is the theoretical caustic to silica ratio in the bauxite residue, A_B is the Al₂O₃ mass fractions in the bauxite, $(A/S)_M$ is the theoretical alumina to silica ratio in bauxite residue, and AO is the alumina concentration in the digestion liquor (g/L), 1.4 is the expected caustic ratio of the liquor after digestion. According to the calculation results a quantitative amount of bauxite samples, lime additives and caustic liquor for the tests were added to the bomb digesters respectively. When the molten salt heating furnace reached the preset temperature, the bomb digesters installed on the rotating frame will be put into the molten salts to preheat, turn on the rotary stirring system and start timing after the preheating stage of just a few minutes.

The bomb digester can be taken out from the molten salts when the digestion time is reached and is put immediately into cold water for cooling. Then the bomb digester is opened and the bauxite slurry is filtered for liquor analysis by chemical titration and the digestion residue is further filtered, washed, and dried for chemical composition analysis by XRF.

Test Program

In this paper, the digestion tests were carried out for the bauxite samples A and B. The digestion test program is shown in Table 2.

Test Targets

In order to study the digestion performances of the bauxite samples, the alumina to silica ratio (A/S) and caustic to silica ratio (N/S) in the bauxite residues were tested by XRF and the chemical analysis of the caustic liquor was analyzed by titration analysis. The A/S is the mass ratio of alumina and silica in the bauxite residues, and the N/S is the mass ratio of sodium oxide and silica in the bauxite residues.

According to the chemical compositions of bauxite samples and their digestion residues, from which the alumina digestion rate, bauxite and caustic consumption in the digestion process can be calculated. The calculation formula is as follows.

$$\eta_{\rm AI} = \left[1 - \frac{(A/S)_{\rm M}}{(A/S)_{\rm B}} \right] \times 100\% \ (\%) \tag{1}$$

where η_{AI} is the alumina digestion rate, $(A/S)_M$ is the mass ratio of Al₂O₃ and SiO₂ in the bauxite residue and $(A/S)_B$ is the mass ratio of Al₂O₃ and SiO₂ in the bauxite sample.

$$MC = \frac{1}{A_{B} - S_{B} \times (A/S)_{M}}, (t/t - Al_{2}O_{3})$$
$$CC = \frac{(N/S)_{M}}{(A/S)_{B} - (A/S)_{M}} \times 1.2903 \times 1000, (kg/t - Al_{2}O_{3})$$

where A_B and S_B are the mass fractions of Al_2O_3 and SiO_2 in the bauxite sample respectively, $(N/S)_M$ is the mass ratio of Na₂O and SiO₂ in the bauxite residue, 1.2903 is the mass conversion factor of Na₂O converted to NaOH, MC and CC are the bauxite and caustic consumptions respectively.

In order to study the digestion behavior of the bauxite samples during the digestion process the mineral compositions of bauxite samples and their residues were analyzed by XRD. In addition, SEM observation was carried out on the morphology of the different minerals in the bauxite samples and their residues.

Test Results and Discussion

A/S and N/S of the Bauxite Residues vs Digestion Temperatures at Different Times

The digestion performances of A1# bauxite at different temperatures were studied. The A/S and N/S of the bauxite residues at the different digestion times at 230–250 °C have been shown in Fig. 2.

The A/S (A/S = 1.11) in the bauxite residues digested at 230 °C for 5 min was very high and the digestion rate of alumina is only 78.95%. The main reason is that the boehmite in the bauxite fails to react completely at 230 °C. With the longer digestion time the A/S in the bauxite residues decreased and the highest digestion rate of alumina is 80.57% at 230 °C, which means the boehmite still remained in the residue. The A/S within 10 min at 240 °C and 250 °C is lower (<0.98), but with longer digestion time the digestion rate of alumina gradually decreased. It seems that the

No	Bauxite	Temperature	Time	Lime additives
1	A1#	230–250 °C	5 min–45 min	2%
2	A2#	240 °C	5 min-60 min	2%
3	B1#	240 °C	5 min-60 min	2%
4	B1#	240 °C	10 min, 60 min	0–2.5%
5	A2#	240 °C	10 min	0%, 1%, 2%

Table 2The digestionconditions of digestionexperiments





boehmite can almost be completely reacted at temperatures above 240 °C for 10 min.

N/S in the bauxite residues is mainly determined by the mineral compositions in the bauxite residues and the attack content of quartz. N/S in the bauxite residues will be obviously increased as the quartz is attacked in large quantities. It can be seen in Fig. 2 that N/S in the bauxite residues is increased with the higher digestion temperature and longer digestion time. N/S in the bauxite residues digested at 240 $^{\circ}$ C for 10 min is still kept at a lower level (less than 0.46).

Therefore, it can be seen by the test results that when the gibbsite-boehmite bauxite is digested for a short time at 240 °C, a better digestion rate of alumina can be obtained than longer time and N/S in the bauxite residues can be kept lower at the same time.

The tests were also carried out for A2# and B1# bauxite at 240 °Cfor the different digestion times. It can be seen in Fig. 3 that both A/S and N/S in the bauxite residues show lower values within 10 min. This means that the bauxite digestion is carried out at the medium temperatures for a relatively short time is more suitable for the gibbsite-boehmite bauxite.

A/S and N/S of the Residues vs Lime Additives

The effects of lime addition on the digestion results have been studied for the gibbsite-boehmite bauxite samples. Figure 4 shows the influences of the different lime additions on A/S and N/S in the bauxite residues at the medium digestion temperature for a short time.

It can be seen in Fig. 4 that with the increase of lime addition the A/S and N/S in the residues decrease at the digestion temperature of 240 °C for 10 min. When the lime addition exceeds 2% the digestion rate of alumina will decrease and N/S in the bauxite residues will increase.

Moreover, A/S and N/S in the bauxite residues can be reduced by the definite addition of lime in the different bauxite digestion processes. It can be found from the mentioned above that the lime addition generally has two effects. One is to eliminate the negative influence of titanium dioxide on the bauxite digestion to improve alumina digestion rate; the second is to change the DSP mineral composition and to replace a part of the sodium oxide in DSP for reducing N/S in the bauxite residues.









It can also be found that the alumina digestion rate can be improved by lime addition during short-time digestion and N/S in the bauxite residues can be reduced as well. The alumina in the bauxite can be completely digested for a long digestion time without adding lime. It is suggested [17–21] that N/S in the bauxite residues decreases with the addition of lime. However, hydrogarnet will be produced instead of common sodium aluminosilicate hydrate when lime is added in excess during the digestion process, resulting in the loss of alumina and caustic. Therefore, the optimum lime addition in the digestion process of gibbsite-boehmite bauxite should be determined by tests.

Mineral Composition Changes During Digestion

The mineral compositions in the bauxite residues at 240 °C for different digestion times are shown in Fig. 5.

It can be seen in Fig. 5 that the bauxite residues are basically the same in terms of mineral types and the differences between the bauxite residues at the different digestion conditions are the content of quartz and boehmite in the residues. It can be found combined with the results in Fig. 2 that boehmite in the bauxite does not completely react for 5 min at 240 $^{\circ}$ C so its A/S is relatively high. But when

digestion time is extended to 10 min the boehmite is digested completely to reduce A/S in the bauxite residues.

It is shown by comparison of the peak intensity of quartz in the bauxite residues in Fig. 5 that the quartz remained in the residues is gradually reduced with the time extension. The reaction equation for quartz can be described as follows:

$$SiO_2 + 2NaOH = Na_2SiO_3 + H_2O$$

 $xNa_2SiO_3 + 2NaAI(OH)_4 + (x + n - 4)H_2O = Na_2O$ $\cdot Al_2O_3 \cdot xSiO_2nH_2O + 2xNaOH$

It is seen from the equation that the reaction of quartz in caustic liquor will cause alumina and caustic loss to form sodium aluminosilicate hydrate in bauxite residues. At medium temperatures the major reaction carried out in 10 min is the rapid digestion of the boehmite and the caustic attack to quartz is relatively slow. After 10 min. digestion the boehmite digestion will be completed and only quartz continue to react with caustic liquor consuming Al_2O_3 and Na_2O in the liquor continuously. Therefore, both A/S and N/S in the bauxite residues will be increased with the time extension after 10 min digestion.

It is concluded that the alumina recovery and caustic consumption during the bauxite digestion process can be changed by the different digestion conditions due to the digestion behaviors of boehmite and quartz.

Fig. 5 The mineral compositions in the bauxite residues for the different digestion times (a) A1#, 5 min; (b) A1#, 10 min; (c) A1#, 20 min







SEM Test Results and Discussion

The quartz morphology and its mosaic situation in the bauxite were observed by SEM to find the reason why there will be the different attack extent for the quartz in the residues at the different digestion conditions.

The SEM images of quartz in gibbsite-boehmite bauxite are shown in Fig. 6.

It can be seen in Fig. 6 that the two kinds of quartz particles present a relatively regular polyhedral shape and the particle size is larger than 10 microns. It seems that there is a thick mineral coating layer on the surface of the quartz particles.

EDS analysis was performed on the position where the coating was thicker, and the elemental composition of the coating material was determined to be Al and O. It can be determined that the encapsulated materials on the quartz surface are gibbsite and boehmite respectively [22]. The existence of this coating layer containing boehmite is beneficial to reducing caustic attack to quartz. In the digestion process, the caustic attack to the quartz can be stopped or delayed to reduce the alumina and caustic loss due to the wrapping of boehmite at the beginning of digestion until the boehmite is completely digested.

Influences of Digestion Parameters on the Consumption of Bauxite and Caustic

The bauxite consumption and caustic consumption refer to the mass of bauxite and the mass of sodium hydroxide required for each 1 ton of alumina produced in the digestion process. The bauxite and caustic consumptions are the most important factors to influence the alumina production cost, which is mainly related to the bauxite grade, A/S and N/S of the bauxite residues. The only way to reduce alumina production costs is to develop the new processes to reduce A/S and N/S in the bauxite residues since bauxite grade is an uncontrollable factor.

It can be seen from the study results mentioned above that the bauxite and caustic consumptions can be changed by the digestion parameters such as temperatures, holding times and additives.

The new digestion processes have been studied in the Chinese alumina refineries using the gibbsite-boehmite bauxite from Australia. Table 3 shows the differences in the digestion results and bauxite and caustic consumptions for the different digestion conditions in the Chinese alumina refinery.

It can be found in Table 3 that both bauxite and caustic consumptions has been reduced by changing the digestion conditions. The bauxite consumption is reduced by 50 kg per ton of alumina and caustic consumption is reduced by 34 kg per ton of alumina. Total alumina production cost is decreased by more than RMB 100.

Conclusions

The gibbsite-boehmite bauxite from Australia contains about 50% of alumina and 9% of silica with alumina to silica ratio of more than 5.

Gibbsite and boehmite are the major alumina minerals and kaolinite and quartz are the main silica minerals existing in the gibbsite-boehmite bauxite.

Table 3 The differentconsumptions for the differentdigestion conditions

Different digestion conditions	A/S in residues	N/S in residues	Bauxite consumption	Caustic consumption
250 °C, 60 min, no additive	1.07	0.66	2.42 t/t	185 kg/t
235 °C, 15 min, 2% of lime	0.98	0.55	2.37 t/t	151 kg/t

It can be seen that A/S and N/S of the bauxite residue will be less than 1 and 0.55 respectively after digestion at the medium temperatures of 230–250 °C for a few min. with a little lime addition. And the boehmite in the bauxite can be digested and most of the quartz remains in the bauxite residue at the digestion conditions mentioned above.

A Bayer digestion technology has been developed based on the test results with a medium digestion temperature of 230-250 °C, a short holding time of a few minutes, and bits of lime addition.

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