Research on the Microstructure and Reaction Properties of Ferrous Mineral in Laos Bauxite

Xiang-Hui Cang
Professor
Zhengzhou Research Institute of China Aluminum Corporation, Zhengzhou China
Corresponding author: zyy_cangxh@rilm.com.cn

Abstract

This article mainly studies the reaction properties of Laos' laterite bauxite. Research on the microstructure of hematite, goethite, ilmenite and distribution characteristics utilized existing analytical methods such as Scanning Electron Microscope (SEM), Energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction analysis (XRD). More goethite can affect red mud settling velocity, high temperature digestion can improve the dissolution of the Laos bauxite significantly and moreover, the hematite ore cannot react to bauxite components and sodium-aluminate solution then transfer to the red mud.

Keywords: Microstructure; Ferrous mineral; Reaction properties.

1. Introduction

The primary use of bauxite ore is for the extraction of metallic aluminum in the aluminum industry, as refractory materials and abrasive materials, and as raw materials for high aluminum cement [1]. At present, the related research of various minerals in bauxite is mainly based on X-ray diffraction (XRD), DTA-TG thermal analysis [2], infrared spectroscopy (IR) [3] and other characterization methods. It is difficult however, to reflect the influence of the existing state and distribution characteristics of minerals on the digestion performance of bauxite. In this article, the microstructure and composition of bauxite were characterized by scanning electron microscope (SEM), energy disperse spectroscopy (EDS).

Laos's bauxite was chosen as a typical representative iron-rich bauxite and is of great significance for solving the problem of the current shortage of high-quality bauxite in China. A Laos laterite bauxite was studied, its main ore phases beinggibbsite, hematite, goethite, with secondary minerals of kaolinite, quartz, and ilmenite. In addition, there are scattered minerals such as phosphosiderite, chlorite, and zircon. Since different kinds of iron minerals directly affect the settling performance of red mud slurry [4], the technological test conditions of laterization bauxite were obtained through settling performance tests of digestion slurry and the selection of the type and dosage of flocculant.

2. Sample Preparation and Research Methods

The following process was followed to prepare samples for analysis:

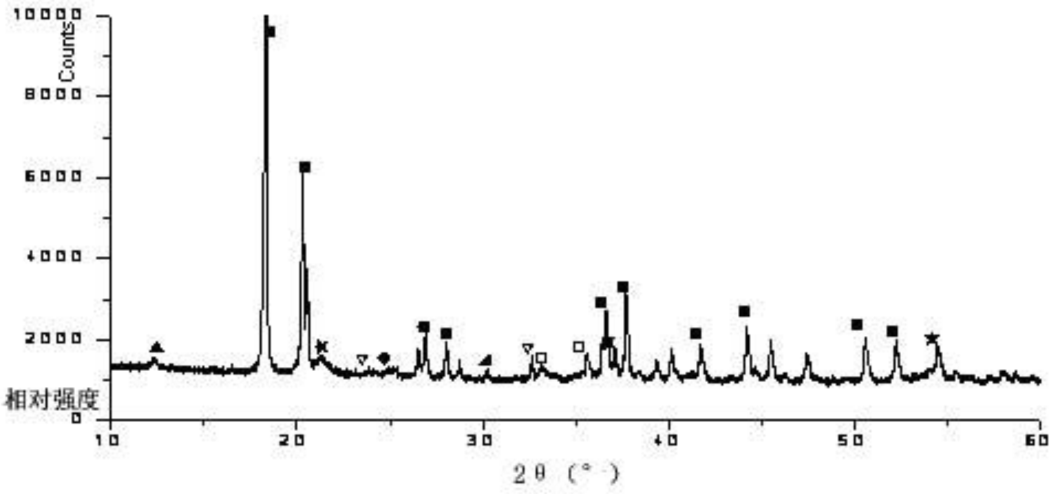
1. Red-brown and yellow-white block samples were selected from the raw ore and crushed to flakes to obtain a fresh section of the ore
2. The sample was smoothed back with sandpaper and placed in a beaker filled with absolute ethanol
3. Ultrasonic cleaning was undertaken for 8 minutes to remove debris attached to the surface
4. After the ethanol was volatilized, the sample was fixed and sprayed onto carbon to obtain a sample for SEM and EDS.

In order to compare the results, diffraction and polarization samples were also prepared for typical iron-bearing ores. The ore phase was measured on ORTHOLUXIIPOL-BK polarizing microscope from German Lerzt Company. Ore composition was performed on X'pert pro X-ray diffractometer from Netherlands PANalytical Company. Component analysis was conducted on JEOL JSM-6360LV scanning electron microscope and Oxford energy spectrum.

3. Results and Discussion

3.1 Analysis of Ore Phase

The phase of the reddish-brown raw ore was analyzed by XRD. As shown in Figure1, the sample is mainly composed of gibbsite and alumogoethite, and contains hematite, ilmenite, magnetite, kaolinite, quartz and other minerals. The main iron minerals are alumogoethite, hematite, ilmenite and magnetite.



■ Gibbsite ; ★Alumogeothite ; ○ Hematite ; ▽ Ilmenite ;
◆ Anatase ; ▲ Kaolinite ; ● Quartz ; ◢Magnetite

Figure 1. XRD of Laos's bauxite.

3.1.1 Alumogoethite

Alumogoethite is a mineral produced by in-phase replacement of aluminum. It is common for iron to be replaced by aluminum in goethite of gibbsite to form alumogoethite. Due to the different amount of substitution, the morphology of the formed alumogoethite is not very similar and is mainly present as heterogeneous and amorphous aggregates.

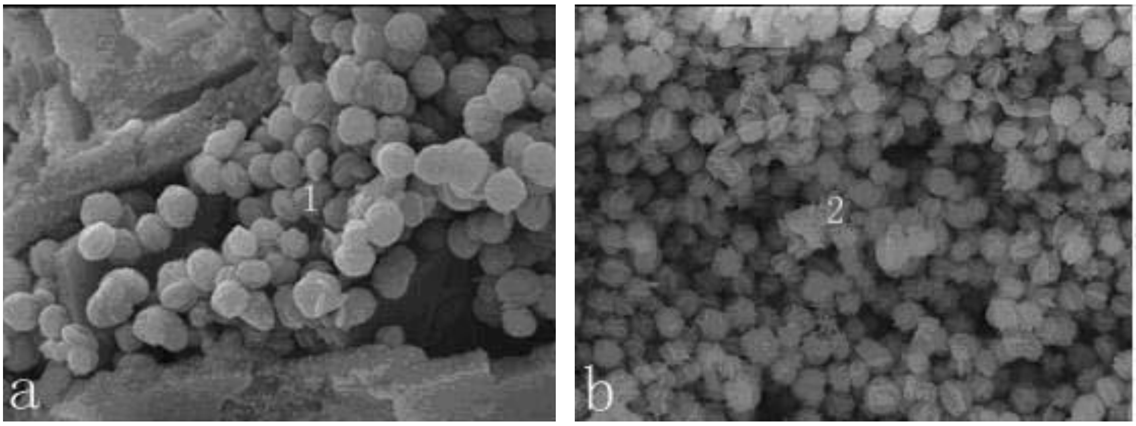
The replacement rate of aluminum can be determined by:

- Alkali treatment, washing, filtering, and then acid treatment, washing, filtering, drying of a weighed amount of sample to produce a certain amount of residue.
- Analysis of the chemical composition of the residue, to obtain the d value of the diffraction peaks of the (111) and (110) crystal planes of the goethite by XRD [5]
- Calculation of the displacement rate of aluminum in the alumogoethite: Al mol % = 21, and the molecular formula of alumogoethite can be determined as $Al_{0.21}Fe_{0.79}OOH$.

3.1.2 Hematite

Hematite crystal is normally flat with a kidney-shape surface as shown in Figure 2a. Hematite in Laos bauxite however, is typically fine oolitic particles with a rose-shape as shown in Figure 2b.

It is one of the two homogeneous multiphase variants: γ-Fe2O3, belonging to isometric system, the particles are relatively uniform in size, and gather into spherical aggregates in the gaps of the gibbsite, also called iron roses. The semi-quantitative analysis of the above carried out with EDS, shows the main elements are Fe and O, which also verify the existence of hematite.

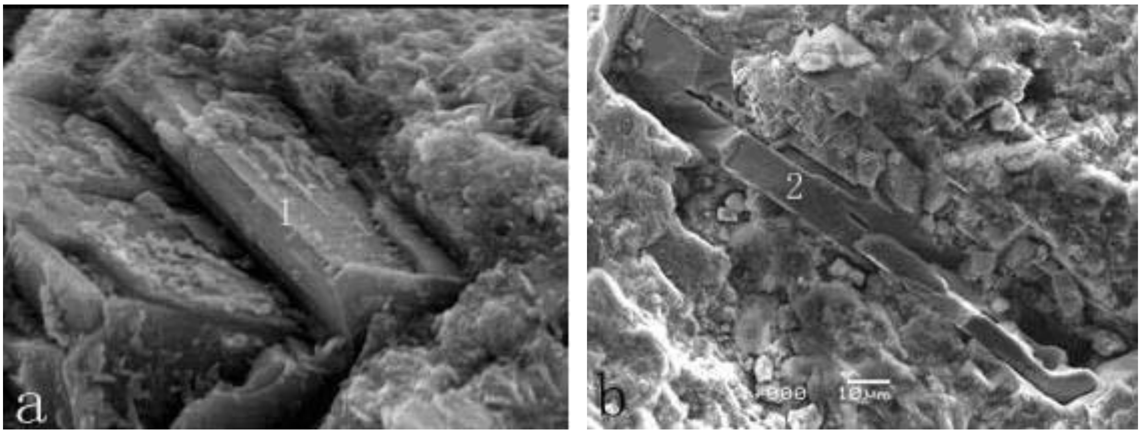


1.Flat and kidney-shape hematite 2. Rose-shape hematite

Figure 2. Hematite microstructure of Laos bauxite.

3.1.3 Ilmenite

Ilmenite belongs to the trigonal system, usually in irregular granular, scaly or thick plate shapes. Ilmenite is rare in gibbsite, mostly appearing as euhedral or other-shaped crystal grains scattered among other mineral particles. The degree of crystallinity in Laos bauxite is better, and under the SEM can be seen to be mostly regular thick plates as shown in Figure 3a and flakes as shown in Figure 3b, which are easy to distinguish. The semi-quantitative analysis of the above-carried out with EDS shows the main elements are Ti, Fe, O, and a small amount of Al, which determine the existence of ilmenite.

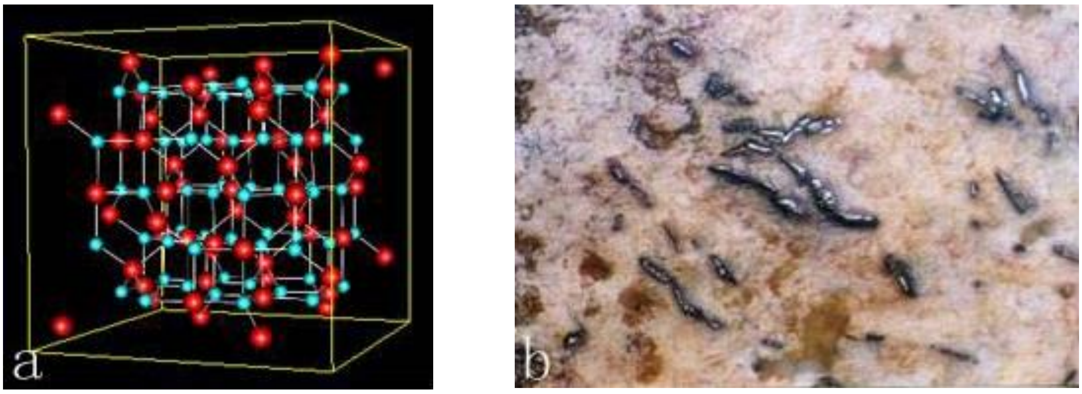


1. Thick-ban ilmenite 2.Sheet ilmenite

Figure 3. Ilmenite microstructure of Laos bauxite.

3.1.4 Magnetite

Magnetite is not common in laterization bauxite, and it is relatively small in Laos bauxite with a total amount not ing 5 %. Magnetite belongs to an isometric system, and the aggregates are dense granular blocks, usually octahedral crystal, black, or streak black. The crystal structure is shown in Figure 4a. It is difficult to separate magnetite from other minerals such as hematite and limonite by SEM and EDS. Polarizing microscope reveals that magnetite is distributed in a slender strip shape among other fine iron minerals as shown in Figure 4b.



a. Crystal structure of magnetite
O: bigger ball, Fe: small ball

b. Black-strip magnetite

Figure 4. Crystal structure and structure of Magnetite in Laos bauxite.

3.1.5 Goethite

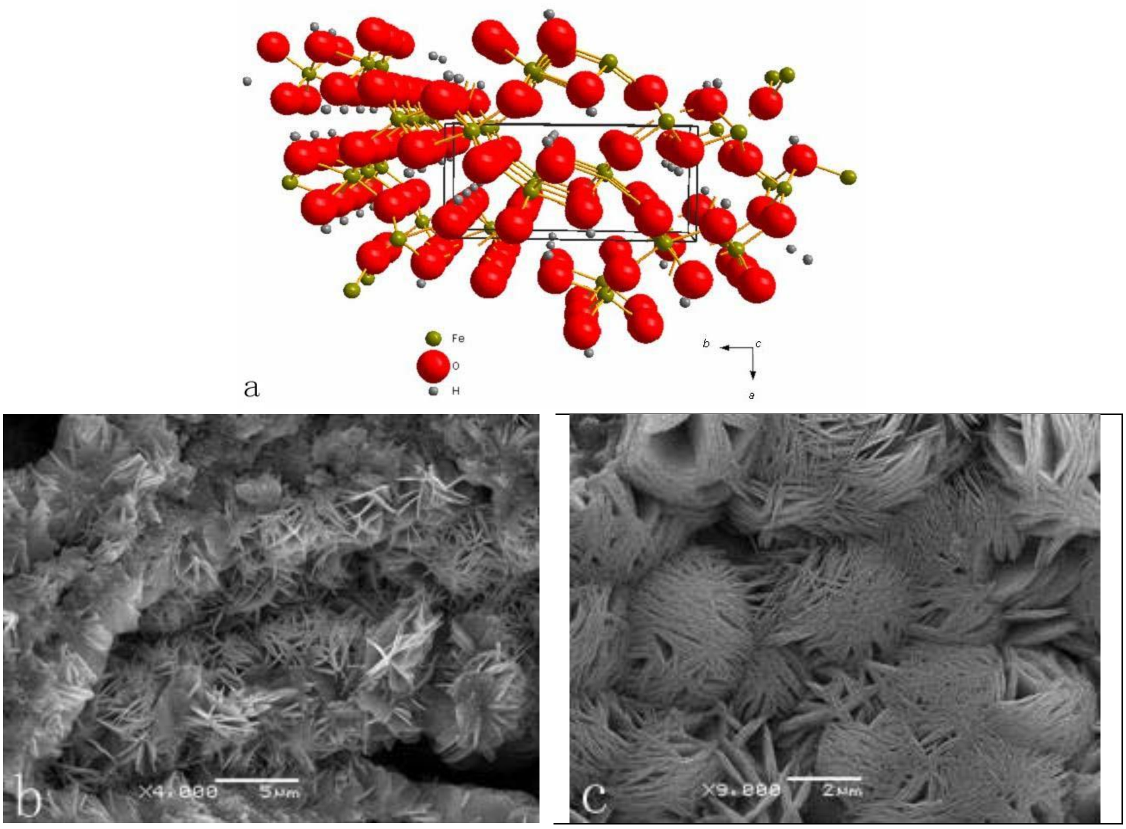


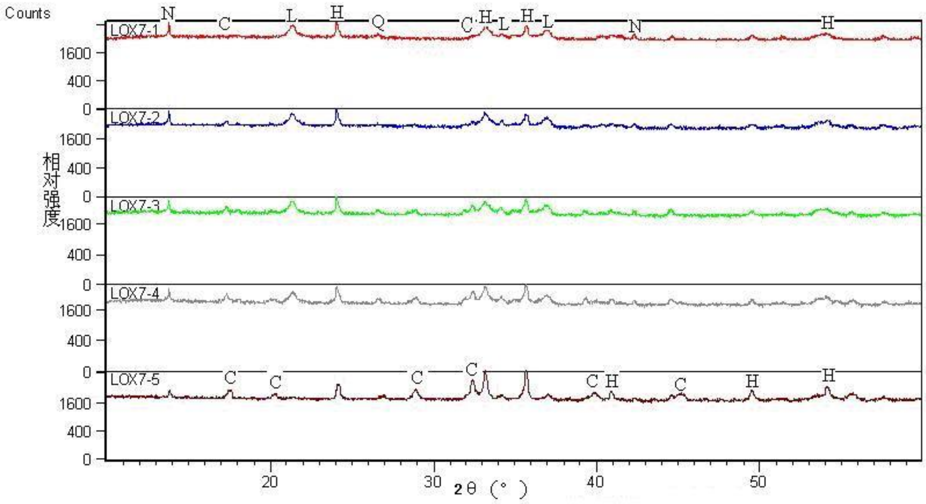
Figure 5. Crystal structure and microstructure of goethite in Laos bauxite

The crystal structure of goethite is an orthorhombic system, with orthorhombic bipyramidal crystals as shown in Figure 5a above, with the crystals parallel to the c-axis being needle-shaped, columnar and having longitudinal stripes. The aggregates of goethite crystals are generally spherical, stalactitic or blocky, with concentric layers and radial fiber structures, while goethite is mostly in the form of fine needle-like microcrystalline clusters and needle-like radial aggregates under the SEM [6]. The goethite in Laos bauxite is usually gathered in needles and clusters and embedded in long and narrow mineral fissures as shown in Figure 5b and 5c.

3.2 Reaction Properties of Iron Minerals

3.2.1 Dissolution Properties of Iron Minerals

In the Bayer process of digestion, hematite does not react with the bauxite components and the sodium-aluminate solution then transfer to the red mud. The results of XRD analysis of the dissolved red mud in Laos bauxite also reveal that the hematite could not react as shown in Figure 6.



N. Sodium aluminosilicate ; C. Garnet hydrate ; H. Hematite ; D. Ilmenite ;
L. Alumogoethite ; T. Perovskite ; Q. Quartz.

Figure 6. XRD of red mud after reaction.

Digestion experiments with different lime dosage were performed at digestion temperature of 145 °C, 260 °C, digestion time of 30 min, and Nk (caustic concentration of the liquor as Na_2O) of 160 g/L. When lime was added, alumogoethite could transform into hematite, while hematite could not react with lime, and the sodium-aluminate solution then directly transferred into the red mud. Ilmenite and magnetite were found to not participate in the reaction during high temperature digestion.

The test results are shown in Figure 7. It can be concluded that the combination of lime addition of 10% or more, with high temperature digestion, can significantly improve the digestion performance of Laos bauxite.

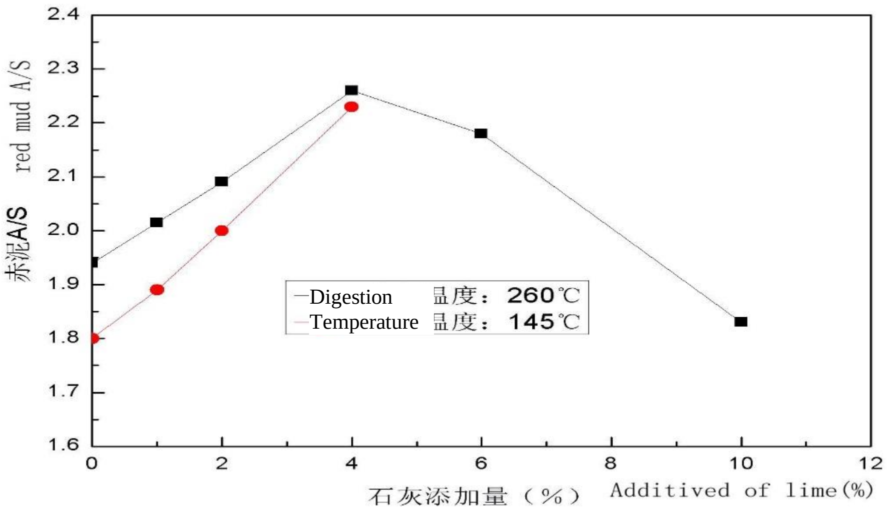


Figure 7. The influence on the A/S from different digestion temperature and joined capacity.

3.2.2 The Influence of Iron Minerals on the Settling Performance of Red Mud

Under the SEM, most of the iron minerals are seen to be wrapped and disseminated on the crystal face of gibbsite, and the crystal structure can't be observed [7]. This study mainly focuses on the cross section of iron minerals to know the existing state of minerals. Laos bauxite consists of alumogoethite (> 20% w/w) and less other iron minerals (< 5% w/w), while Vietnam bauxite is mainly hematite. Nalco 85232 flocculant was used to compare settling performance of the red mud from the two bauxites. The results in Table 1 show the settling velocity of Vietnam bauxite dominated by hematite, has little change, while the settling velocity of Laos bauxite, which is primarily alumogoethite, decreases significantly under the same conditions. A good settling effect can be obtained when the flocculant dosage is greater than 20 g/t dried red mud.

Table 1. Comparer of settling performance to the different bauxite red mud.

Bauxite	**Dosage (g/t-dried red mud）**	**Settling rate (m/h)**	**Content of supernatant solids (g/L)**
Laos bauxite	40	38.10	0.41
	25	21.95	0.32
	19	9.88	0.21
	11	5.22	0.14
Vietnam bauxite	40	49.02	0.67
	25	56.60	0.38
	19	33.65	0.55
	11	24.40	0.42

4. Conclusion

Based on XRD and SEM, the microstructure and existing state of iron minerals in Laos bauxite have been studied in detail. Combining knowledge of the crystal structure of various minerals and the influence of iron minerals on the dissolution conditions of bauxite assists in understanding their possible interaction in industrial production. The results reveal that higher levels of goethite can affect red mud settling velocity, and that high temperature digestion can significantly improve the dissolution of the Laos bauxite, confirming that the bauxite can meet the requirements of industrial production when processed under a certain alkali concentration and digestion temperature, followed by the addition a certain amount of flocculant when red mud is separated.

5. References

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