

## Experimental Study on Preparation of Un-fired Bricks by Bauxite Residue and Other Industrial Solid Wastes

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### Abstract

Utilization of bauxite residue is a worldwide problem. Bauxite residue with high desulfurization activity, can be used as a desulfurization agent for wet flue gas desulfurization, and the by-product of desulfurization is desulfurized bauxite residue. In this paper, the un-fired brick was prepared by adding desulfurization bauxite residue (red mud), fly ash and boiler slag as the main raw materials. The effects of different proportions of bauxite residue and desulfurized bauxite residue and the addition of slag, lime and cement on the properties of un-fired bricks were studied. Under the optimum ratio, the compressive strength of un-fired bricks cured at room temperature 28d was 22.5MPa, and the bulk density was 1.57g/cm<sup>3</sup>. Its technical indicators met the MU20 grade requirements of “Un-fired rubbish gangue brick” (JC/T 422-2007). Using this process to prepare un-fired bricks, a variety of industrial solid wastes can be used in a large amount, with low cost, energy conservation and environmental protection.

**Keywords:** Bauxite Residue, Desulfurized Bauxite Residue, Un-fired Bricks.

### 1. Introduction

Bauxite residue is a strong alkaline waste generated from the production of alumina using bauxite as the raw material, which is difficult to utilize [1]. At present, more than 90% of alumina production in China adopts the Bayer process. Due to the difference in production process and bauxite grade, the production of 1t alumina produces about 1~2t bauxite residue [2]. The annual generation of bauxite residue in China is close to 100 million tons, and the cumulative stockpile stock is more than 1.2 billion tons, with a utilization rate of less than 5%. In a large number of cases, the bauxite residue deposits are stacked, which not only occupies a lot of land, but also poses great environmental risks.

The utilization of bauxite residue is a worldwide problem, especially Bayer bauxite residue. Bauxite residue has the characteristics of fine particles, large specific surface area, strong alkalinity, a high content of effective sulfur fixing components, and has strong adsorption capacity and reactivity to SO<sub>2</sub> gas [3,4]. Scholars have carried out a lot of research on bauxite residue for flue gas desulfurization [5-9], and achieved promising technical results. The application of Bayer bauxite residue in industrial flue gas wet desulfurization is the interaction of two harmful substances, which can not only reduce the emission of SO<sub>2</sub> in flue gas, but also realize the dealkalization of bauxite residue. The byproduct of bauxite residue used for flue gas desulfurization is desulfurized bauxite residue, whose Na<sub>2</sub>O content can be reduced to less than 1.5%. It has the potential to be used in building materials after activation treatment.

In this paper, in order to find a way for the comprehensive utilization of desulfurized bauxite residue and bauxite residue, and the collaborative utilization of fly ash and boiler slag industrial solid waste. The desulfurized bauxite residue, bauxite residue, fly ash and boiler slag are used as the main raw materials and lime and cement are added to prepare un-fired brick. By compounding the two kinds of bauxite residues, the activity of fly ash is stimulated synergistically, and the strength of un-fired brick is improved, which has more advantages than the traditional preparation of un-fired brick by adding bauxite residue alone.

## 2. Materials

### 2.1 Bauxite Residue

In this study, Bayer bauxite residue from an aluminium oxide plant in Guizhou was selected as the raw material. The bauxite residue is reddish brown paste in appearance, with pH value of 11.75 and true density of 2.9g/cm<sup>3</sup>. Table 1 showed the main chemical composition of the bauxite residue (shown as the metal oxide). The main chemical composition of the bauxite residue is Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and CaO, accounting for about 75.30% of the whole component, followed by Na<sub>2</sub>O, MgO and K<sub>2</sub>O.

**Table 1. The main chemical composition of the bauxite residue.**

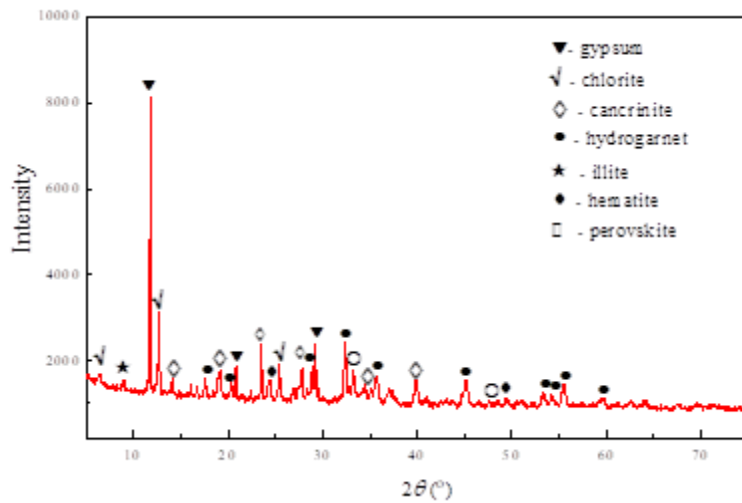
Element	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	MgO	K <sub>2</sub> O	TiO <sub>2</sub>
Content/%	22.20	16.43	21.40	4.37	15.27	1.50	1.04	4.55

The mineral composition of bauxite residue is analyzed by X-ray diffraction analyzer. The main mineral constituents of bauxite residue are shown in Table 2 and the XRD diffraction pattern of bauxite residue is shown in Figure 1. The mineral composition of bauxite residue is chlorite, cancrinite, hydrogarnet, calcite, illite, hematite, perovskite and diaspore.

**Table 2. The phase analysis results of bauxite residue\*.**

Mineral	chlorite	cancrinite	hydrogarnet	calcite	illite	hematite	perovskite	diaspore
Content/%	18.3	18.0	12.5	7.5	6.7	18.5	7.4	5.8

\*:XRD semi-quantitative analysis results



**Figure 1. XRD diffraction pattern of bauxite residue.**

Scanning electron microscopy is used to observe the micro morphology of bauxite residue, as shown in Figure 2. When magnified 4000 times, it can be seen that bauxite residue particles are irregular aggregate structures with uneven size, and particles are agglomerated. When magnified 10000 times, it is obvious that bauxite residue is a combination of plate shape or blocky small particles, and the particle surface is rough and porous.

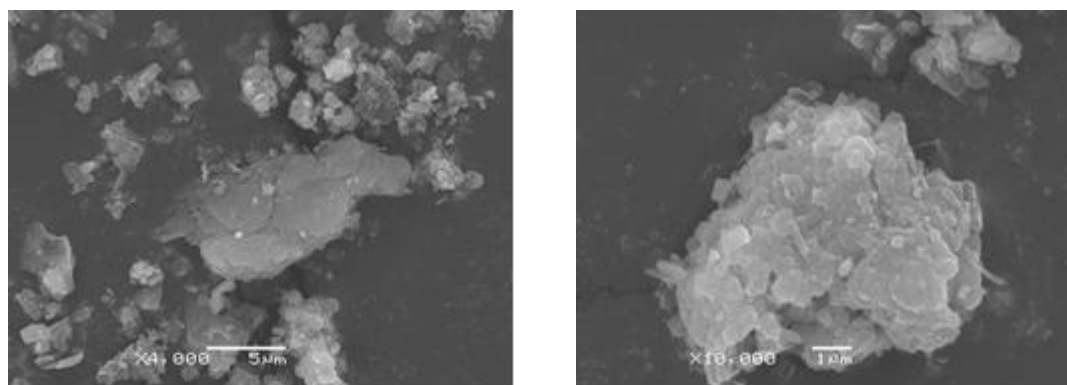


Figure 2. SEM of bauxite residue.

## 2.2 Desulfurized Bauxite Residue

Using bauxite residue for flue gas wet desulfurization is to use the effective sulfur fixing component in bauxite residue to react with SO<sub>2</sub> in flue gas. The alkali and combined alkali of bauxite residue react with SO<sub>2</sub> to produce sulfite and sulfate. After desulfurization, the Na<sub>2</sub>O level of the bauxite residue is reduced to 0.83%. The calcium component in the bauxite residue reacts with SO<sub>2</sub> to produce calcium sulfite. After oxidation treatment, the calcium sulfite phase is transformed into a gypsum phase. The main chemical constituents of desulfurized bauxite residue, as determined by XRF, are shown in Table 3.

Table 3. The main chemical composition of desulfurized bauxite residue

Element	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	MgO	TiO <sub>2</sub>	SO <sub>2</sub>
Content/%	21.24	16.05	20.12	0.83	14.58	1.23	4.28	3.17

The mineral constituents of desulfurized bauxite residue was analyzed by X-ray diffraction analyzer and the main mineral constituents of desulfurized bauxite residue are shown in Table 4. The XRD diffraction pattern of desulfurized bauxite residue is shown in Figure 3. The diffraction peak of dihydrate gypsum in desulfurized bauxite residue is relatively strong, and calcium containing phases such as hydrated garnet and calcite are weakened. This is because some calcium containing substances in bauxite residue gradually absorb and react with SO<sub>2</sub> in flue gas to produce calcium sulfite, which is then oxidized to calcium sulfate.

Table 4. The phase analysis results of desulfurized bauxite residue\*.

Mineral	gypsum	chlorite	cancrinite	hydrogarnet	illite	hematite	perovskite
Content/%	9.0	15.8	14.5	12.0	6.5	18.6	7.5

\* XRD semi-quantitative analysis results

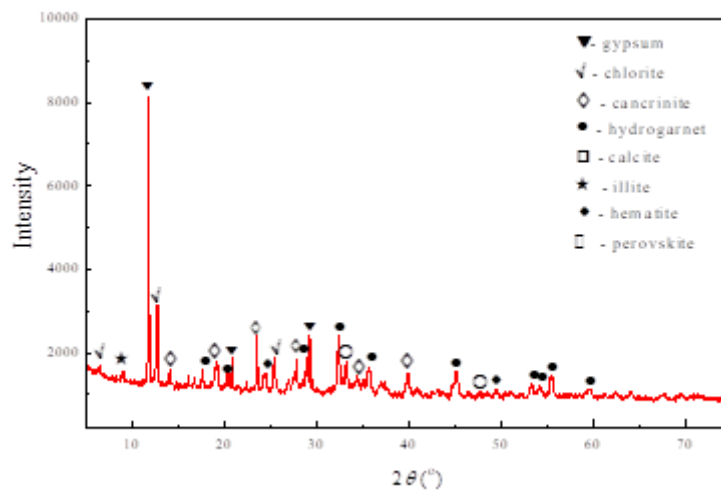


Figure3. XRD diffraction pattern of desulfurized bauxite residue.

### 2.3 Fly Ash

The fly ash is taken from the self-contained thermal power plant in an aluminium plant of Guizhou. Its moisture content is 14.56%, loss on ignition is 8.7%, SiO<sub>2</sub> content is 44.07%, and Al<sub>2</sub>O<sub>3</sub> content is 30.29%. The specific chemical composition, as measured by XRF, is shown in Table 5.

Table 5. The main chemical composition of fly ash.

Element	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	MgO	K <sub>2</sub> O	TiO <sub>2</sub>
Content/%	30.29	44.07	9.83	0.23	2.67	0.48	0.73	1.26

### 2.4 Boiler Slag

The boiler slag is taken from the self-contained thermal power plant in an aluminium plant of Guizhou. Its main component is loose and porous active glass, which is used as aggregate after passing 4-mesh standard sieve. The specific chemical composition is shown in Table 6.

Table 6. The main chemical composition of boiler slag.

Element	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	MgO	K <sub>2</sub> O	TiO <sub>2</sub>
Content/%	31.45	51.41	9.74	0.21	2.41	0.49	0.88	1.01

### 2.5 Other Materials

1. Lime: commercial lime powder, with a calcium oxide content of more than 95%.
2. Cement: ordinary Portland cement with the commercial grade of 42.5.

## 3. Sample Preparation

### 3.1 Equipment

1. Raw material dry mixing equipment: SHR-10A high-speed mixer;
2. Raw material wet mixing equipment: JJ-5 cement mortar mixer;
3. Un-fired brick forming equipment: ZS-5 mortar specimen forming and vibrating table;
4. Un-fired brick forming mold: 40mm×40mm×160mm triple mold;

- Strength testing equipment: YAW-300D-10D automatic compression and bending testing machine.

### 3.2 Preparation Process of Un-fired brick

The process flow of preparing un-fired brick is shown in Figure 4.

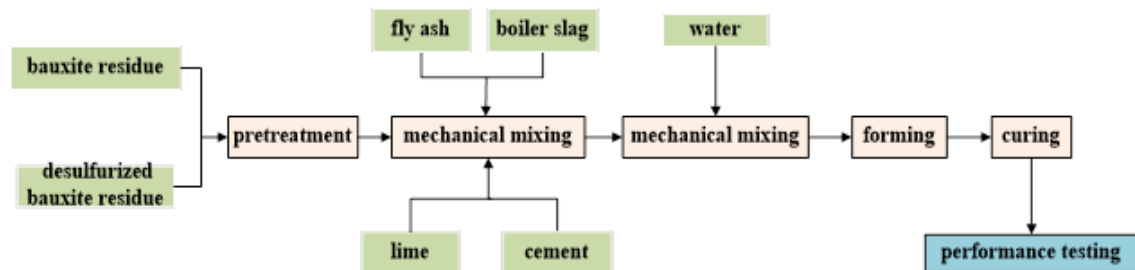


Figure 4. The process flow of preparation of un-fired brick.

The specific operation process is as follows:

- Prior to use, all raw materials were prepared in readiness, including bauxite residue and desulfurization bauxite residue are dried (180°C) and crushed, boiler slag is dried and sieved through a 4 mesh (4.75 mm) sieve, and the fly ash and lime are dried.
- Weigh each raw material according to different proportions and use a SHR-10A high-speed mixer for premixing. The total amount of materials mixed in each batch is 1.2kg.
- Put the mixed materials into JJ-5 cement mortar mixer according to a certain water cement ratio and mix for 5min.
- Take out the mixed materials, put them into the triplet mold, vibrate them 120 times on ZS-5 mortar specimen forming and vibrating table, wrap them with plastic film in order to keep the surface moist, after the surface is scraped, and naturally cure them in the curing box (average temperature 20°C) to the specified age (the age in this study is 7d and 28d).
- The test blocks cured to the required age shall be tested for 7d and 28d flexural and compressive strength with automatic compression and bending testing machine.

## 4. Results and Discussion

### 4.1 Study on Proportioning of Bauxite Residue Un-fired bricks

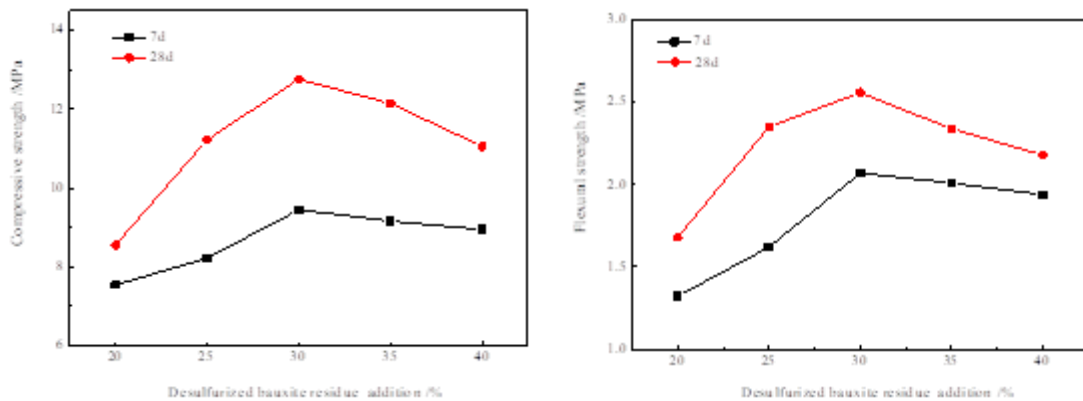
#### 4.1.1. Effect of the Ratio of Desulfurized Bauxite Residue and Bauxite Residue on the Properties of Un-fired Bricks

Na<sub>2</sub>O and K<sub>2</sub>O contained in bauxite residue are alkaline activators of fly ash, which can increase the strength provided by the fly ash in a cementitious system. Desulfurized bauxite residue contains a large amount of gypsum phase, and gypsum and lime can produce synergistic effect, so that lime can promote the reduction of the Ca(OH)<sub>2</sub> crystal size and improve its dispersion during reaction. At the same time, the introduction of a gypsum phase can also promote the hydration of fly ash and improve the early strength of materials [9]. This batch of trials is based on 50% of the total content of desulfurized bauxite residue and bauxite residue. Fly ash, slag, lime and cement are added to prepare un-fired bricks. By changing the relative proportion of desulfurized bauxite residue and bauxite residue, the optimal content of desulfurized bauxite residue and bauxite residue is determined. In order to eliminate the influence of fly ash, slag, lime and cement, the content is fixed at 20% and 17% respectively 10% and 3% (mass fraction), and the test mix proportion is shown in Table 7.

**Table 7. Ratio test of desulfurized bauxite residue and bauxite residue.**

Test No	Amount of raw materials, %					
	desulfurized bauxite residue	bauxite residue	fly ash	boiler slag	lime	cement
R1	20	30	20	17	10	3
R2	25	25	20	17	10	3
R3	30	20	20	17	10	3
R4	35	15	20	17	10	3
R5	40	10	20	17	10	3

The compressive strength and flexural strength of un-fired bricks with different amounts of desulfurized bauxite residue and bauxite residue are shown in Figure 5. From the figure, it can be seen that as the amount of desulfurized bauxite residue increases from 20% to 30%, the 7d and 28d compressive strength and flexural strength of un-fired bricks gradually increase. When the amount of desulfurized bauxite residue exceeds 30%, the compressive strength and flexural strength of un-fired bricks decrease. At the same time, the level of bauxite residue content is the same. Therefore, the ratio of desulfurized bauxite residue and bauxite residue is determined to be 30% and 20%. The analysis shows that the increase of bauxite residue content to a certain extent can increase the pH value of the slurry, stimulate more hydration reactions of fly ash, generate more hydration products, and improve the mechanical strength of the material [10].



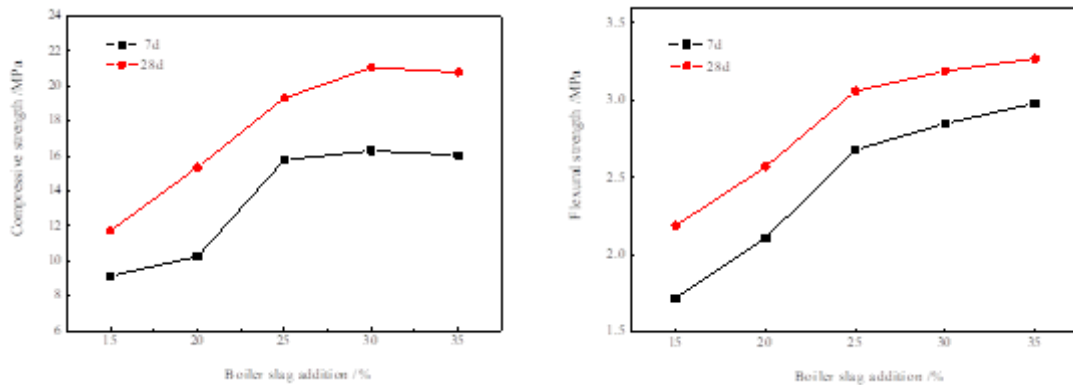
**Figure 5. Test results of different amounts of desulfurized bauxite residue.**

#### 4.1.2. Effect of Boiler Slag Addition on the Performance of Un-fired Bricks

The amount of aggregate has a great impact on the performance of un-fired bricks. Although the aggregate does not directly participate in the reaction, the aggregate with reasonable gradation can enhance the compactness and strength of un-fired bricks. In this study, boiler slag is selected as aggregate, which can not only utilize the solid waste of thermal power plants, but also save costs. This batch of tests studied the influence of boiler slag content on the performance of bauxite residue un-fired bricks. The test mix ratio is shown in Table 8.

**Table 8. Test table of different proportions of boiler slag.**

Test No	Amount of raw materials, %					
	desulfurized bauxite residue	bauxite residue	fly ash	boiler slag	lime	cement
S1	31.0	20.5	20.5	15	10	3
S2	29.0	19.0	19.0	20	10	3
S3	26.0	18.0	18.0	25	10	3
S4	24.0	16.5	16.5	30	10	3
S5	22.0	15.0	15.0	35	10	3



**Figure 6. Test results of different amounts of boiler slag.**

The compressive strength and flexural strength of un-fired bricks with different boiler slag content are shown in Figure 6. It can be seen from the figure that as the slag content increases from 15% to 30%, the 7d and 28d compressive strength of un-fired bricks gradually increases, and then there is a downward trend. When the boiler slag content is 30%, the 7d and 28d compressive strength is the highest, 16.31MPa and 21.06MPa respectively. When the boiler slag content is increased from 15% to 25%, the 7d and 28d flexural strength of un-fired bricks is greatly improved, and the flexural strength is slowly improved when the boiler slag content continues to increase from 25% to 35%. Therefore, it was judged that a boiler slag content at 30% would be an optimum level.

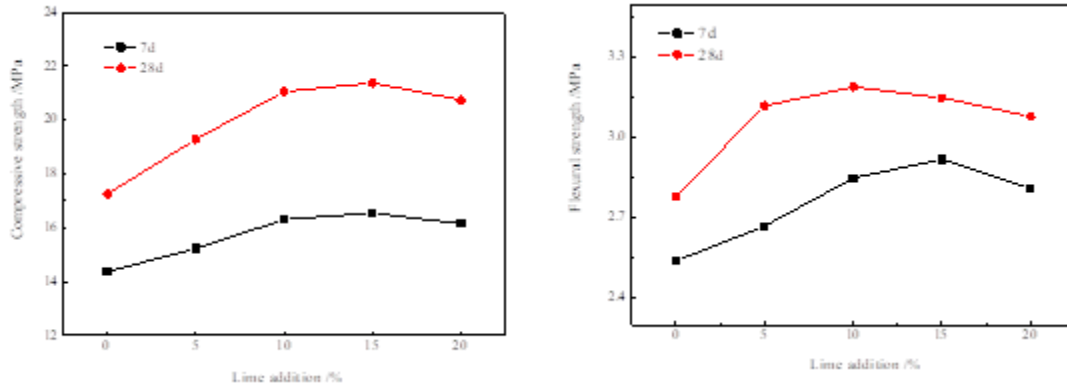
**4.1.3. Effect of Lime addition on the Performance of Un-fired Bricks**

Lime plays a significant role in the formation of the strength of un-fired bricks. This is because a large amount of fly ash is added to the un-fired brick. The calcium hydroxide produced by lime slaking can stimulate the activity of fly ash and react with it to produce hydrated calcium silicate and hydrated calcium aluminate. This group of tests studied the influence of lime content on the performance of bauxite residue un-fired brick. The test mix ratio is shown in Table 9.

**Table 9 Test table of different proportions of lime.**

Test No	Amount of raw materials, %					
	desulfurized bauxite residue	bauxite residue	fly ash	boiler slag	lime	cement
L1	29.00	19.00	19.00	30	0	3
L2	26.00	18.00	18.00	30	5	3
L3	24.00	16.50	16.50	30	10	3

L4	22.00	15.00	15.00	30	15	3
L5	18.00	12.00	12.00	30	20	3



**Figure 7. Test results of different amounts of lime.**

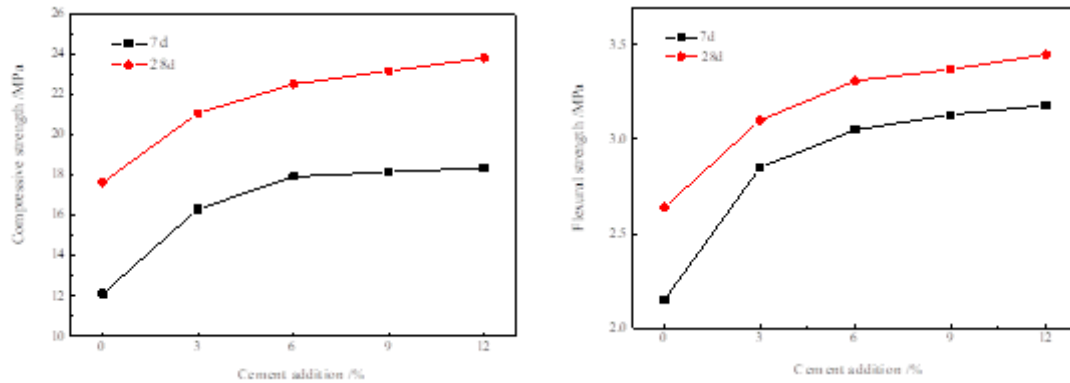
The compressive strength and flexural strength of un-fired bricks with different lime content are shown in Figure 7. From the figure, it can be seen that as the lime content increases from 0% to 20%, the 7d and 28d compressive strength and flexural strength of un-fired bricks gradually increase, and then there is a downward trend. The amount of lime added should not be too small, but considering the cost of un-fired bricks, the amount of lime added is not easy to be too large, so try to reduce the amount of lime when the strength of un-fired bricks is satisfied; a level of 10% lime was therefore judged to be a suitable level.

#### 4.1.4. Effect of Cement Addition on the Performance of Un-fired Bricks

As a commonly used cementitious material, cement can produce hydration reaction after mixing with water, and then produce a solid binder, which can provide strength for un-fired bricks and act as a cementing agent. This group of tests studied the influence of cement content on the performance of un-fired bricks. The test mix ratio is shown in Table 10.

**Table 10. Test table of different proportions of cement.**

Test No	Amount of raw materials, %					
	desulfurized bauxite residue	bauxite residue	fly ash	boiler slag	lime	cement
C1	26.00	17.00	17.00	30	10	0
C2	24.00	16.50	16.50	30	10	3
C3	23.00	15.50	15.50	30	10	6
C4	22.00	14.50	14.50	30	10	9
C5	20.00	14.00	14.00	30	10	12



**Figure 8. Test results of different amounts of cement.**

The compressive strength and flexural strength of un-fired bricks with different cement content are shown in Figure 8. It can be seen from the figure that the 7d and 28d compressive strength and flexural strength of un-fired bricks gradually increase, as the cement content increases from 0% to 12%. When the cement content increases from 0% to 6%, the 7d and 28d compressive strength and flexural strength of un-fired bricks greatly increase, and the strength increases slowly when the cement content continues to increase from 6% to 12%. Therefore, considering the performance and cost of un-fired bricks, the cement content is controlled at 6%. Therefore, the cement content should be controlled at 6%, considering the performance and cost of un-fired bricks.

**4.2 Physical Property Test of Un-fired Bricks**

According to the above research results, the optimal proportion of bauxite residue based un-fired bricks is obtained as follows: desulfurization bauxite residue 23%, bauxite residue 15.5%, fly ash 15.5%, boiler slag 30%, lime 10% and cement 6%. The water absorption, frost resistance, carbonation performance, softening performance and bulk density of the un-fired bricks prepared under the optimal proportioning conditions were tested, and the test results are shown in Table 11. The compressive strength of un-fired bricks is 22.50MPa, the bulk density is 1.57g/cm<sup>3</sup>, the water absorption is 15.43%, the compressive strength after 5 freeze-thaw cycles is 18.77MPa, the compressive strength after carbonation is 18.14MPa, and the average softening coefficient is 0.92. Its technical indicators meet the MU20 grade requirements of “Un-fired rubbish gangue brick” (JC/T 422-2007).

**Table 11. Main technical indexes of un-fired bricks.**

No	Test items	Unit	Require of JC/T 422-2007	Result	Grade
1	Compressive strength	MPa	≥20.0	22.50	MU20
2	Water absorption	%	≤18.0	15.43	MU20
3	Frost resistance (Compressive strength after 5 freeze-thaw cycles)	MPa	≥16.0	18.77	MU20
4	Carbonation property (Compressive strength after carbonation)	MPa	≥16.0	18.14	MU20
5	Softening coefficient		≥0.8	0.92	MU20
6	Bulk density	g/cm <sup>3</sup>		1.57	



Figure 9. Un-fired brick test sample.

### 4.3 Analysis of Strength Formation Mechanism of Un-fired Brick

The mineral composition of un-fired brick was analyzed by X-ray diffraction analyzer. The phase analysis result of un-fired brick is shown in Table 12 and the XRD diffraction pattern is shown in Figure 10. The main mineral composition of un-fired brick is calcite ( $\text{CaCO}_3$ ), quartz ( $\text{SiO}_2$ ), ettringite (Aft), cancrinite ( $\text{Na}_6\text{Ca}_2\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)2.2\text{H}_2\text{O}$ ), calcium hydrated garnet ( $\text{Ca}_3\text{Al}_2(\text{SiO}_4)(\text{OH})_8$ ), hematite ( $\text{Fe}_2\text{O}_3$ ) and perovskite ( $\text{CaTiO}_3$ ).

Table 12. The phase analysis results of un-fired brick\*.

Mineral	calcite	quartz	ettringite	cancrinite	calcium hydrated garnet	hematite	perovskite
Content/%	17.5	22.3	16.2	8.6	10.2	4.6	2.5

\*:XRD semi-quantitative analysis results

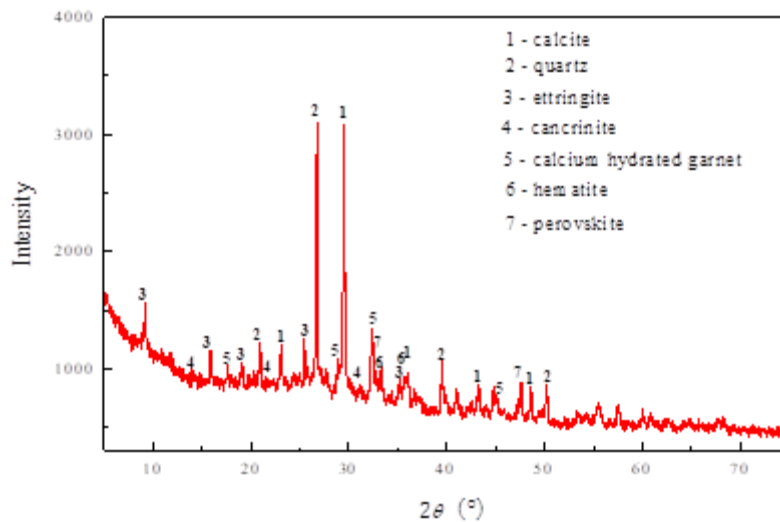


Figure 10. XRD diffraction pattern of un-fired brick.

Based on the XRD analysis of un-fired brick, it is believed that the activity of fly ash is increased by the combination of desulfurized bauxite residue and bauxite residue. NaOH in bauxite residue can form  $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  with  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in fly ash. This mineral exists in the solidified body with high stability new phases such as sodalite or hydrated sodium aluminosilicate, which plays a good cementation role. Desulfurized bauxite residue contains a large amount of gypsum. After heating and activation, the  $\text{SO}_4^{2-}$  released by it has a high excitation effect on active  $\text{SiO}_2$

and Al<sub>2</sub>O<sub>3</sub>, promotes the hydration process, generates more hydration products, and forms ettringite (AFt) with AlO<sub>2</sub><sup>-</sup>, Ca<sup>2+</sup> and OH<sup>-</sup> in the system, which increases the stability of the crystal structure and improves the early strength of un-fired brick [11]. By adding lime and cement, fly ash is hydrated under the excitation of Ca(OH)<sub>2</sub> to produce hydrated calcium silicate, hydrated calcium aluminate and other components, which improves the later strength and durability of un-fired brick. In addition, during the preparation of un-fired brick, some boiler slag is added as aggregate, which is bonded with cementitious materials to play the role of skeleton support. The addition of aggregate boiler slag can reduce the bulk density of un-fired brick and further improve the strength of un-fired brick.

## 5. Conclusions

1. Un-fired bricks were prepared with desulfurized bauxite residue, bauxite residue, fly ash and boiler slag as the main raw materials. The optimum ration was found to be: desulfurized bauxite residue 23%, bauxite residue 15.5%, fly ash 15.5%, boiler slag 30%, lime 10% and cement 6%. The compressive strength of un-fired brick cured at room temperature for 28 days is 22.50MPa; its technical performance met the MU20 grade requirements of “Un-fired rubbish gangue brick” (JC/T 422-2007).
2. The alkaline nature of bauxite residue and gypsum component of desulfurized bauxite residue synergistically stimulate the activity of fly ash. At the same time, boiler slag is used as aggregate to improve the strength and durability of un-fired bricks.
3. In the next phase, the industrial test of using bauxite residue for flue gas desulfurization will be carried out on the in-house boiler of the alumina plant, and the industrial test of preparing un-fired bricks with desulfurized bauxite residue and bauxite residue as raw materials and compounding other industrial solid wastes will be carried out.

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