Experimental Study on Strength and Frost Return of Red Mud Unburned Brick

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Abstract. The comprehensive utilization of red mud had become a common problem in alumina industry. This paper presented a method for preparing red mud non burning cementitious material without frost return. The red mud from an aluminum oxide plant in Guizhou was used as the research object, and the effects of excitation time on compressive and flexural strength were investigated. The better compressive and flexural strength were obtained when the excitation time was 6h. The compressive strength were 15.62Mpa, 18.55Mpa and 15.6Mpa respectively, and the flexural strength were 2.86Mpa, 3.58Mpa and 3.54Mpa respectively when they were maintained for 7 days, 28 days and immersion for 24 hours, which met the strength requirements of MU15. The sodium in red mud was transformed into stable sodium aluminum hydroxysilicate Al₃Na₄(OH)(SiO₄)₃ phase finally, and the concentration of Na⁺ in the leaching solution extracted by horizontal shaking method was about 370mg/L, so it would not cause frost return to the non burning cementitious material. This study would open up a new way for the mass consumption of red mud.

Key words: Red mud, Unburned brick, Strength, Sodium, Frost return

1 Introduction
Red mud was a leaching residue produced in alumina production process. Its alkalinity was high. Generally, its pH was about 12. Its main chemical components were alumina, iron oxide, sodium oxide, potassium oxide and calcium oxide. It looked reddish brown because it contained a certain amount of iron oxide [1]. According to different ore compositions, the production of 1t alumina would lead to the production of 0.7-2 tons of red mud [2-3]. The output of red mud in China was increasing by 100 million tons per year. At present, the cumulative stockpile stock had exceeded 600 million tons [4]. In recent years, the following utilization approaches had been studied: recycling of valuable metals [5-6], using as adsorption materials [7-8], using as cement mixtures [9-10], production of ceramics [11], preparation of new functional materials [12], preparation of building materials [13-14], etc. However, due to their small red mud consumption, the current development and utilization rate of red mud was less than 5% [15], and stockpiling was still the main treatment method. On one hand, the storage of red mud occupied a lot of land, on other hand, the alkali in it would enter the soil with the leaching of rainwater, resulting in alkalinization of soil and pollution of groundwater [16]. At the same time, red mud was easy to form dust, causing air pollution and serious damage to the environment. The outline of the national 14th five year plan clearly put forward that the comprehensive utilization rate of red mud must reach more than 60%. Therefore, the task of comprehensive utilization of red mud resources
was very urgent. In order to realize the mass consumption of red mud, it must be applied to the field of
building materials or transportation. The high alkali content of red mud would lead to the serious frost
back problem of red mud cementitious materials. Many studies had avoided this problem because
there was no good method to solve it. However, the author believed that this was an important obstacle
to the advancement of the technology of preparing cementitious materials from red mud. Based on the
above, this paper explored a method to prepare the non burning cementitious material with red mud
and they had no frost return. The effects of excitation time on compressive strength and frost return
were investigated to provide reference for mass consumption of red mud.

2. Experiment

2.1. Experimental Raw Materials
Red mud was taken from an alumina plant in Guizhou, and cement for binder was taken from an
enterprise in Guiyang City, Guizhou Province and it was 425# cement. The formulation of activator
was produced by the laboratory, and its main components were lime, MgCl$_2$ and C$_{12}$H$_{10}$NSO$_3$Na. The
chemical composition of red mud was shown in table 1.

| Component | Content (wt%) |
|-----------|---------------|
| Al$_2$O$_3$ | 23.24         |
| SiO$_2$   | 23.72         |
| Fe$_2$O$_3$ | 3.90         |
| CaO       | 27.92         |
| TiO$_2$   | 3.29          |
| MgO       | 1.89          |
| Na$_2$O   | 3.68          |
| K$_2$O    | 0.20          |
| S         | 0.80          |
| Loss      | 11.36         |

From table 1, the main chemical components of the red mud were alumina, silica, calcium oxide,
iron oxide, titanium dioxide and sodium oxide.

2.2. Experimental Process
Firstly, 500g red mud, 100ml activator and 500ml water were put into a 2000ml beaker, it was stirred
for 20 min to mix evenly. After the excitation reaction was carried out for 120 min, 240 min, 360 min,
480 min and 600 min, the solution was filtered. Secondly, the red mud after excitation treatment was
dried in a 100°C oven for 6h. The dried red mud was evenly mixed with cement (the mass ratio of
dried red mud to cement was 5:1), then an appropriate amount of water was added for uniform mixing.
Thirdly, it was pressed into a cuboid blank with height of 30mm, length of 80mm and width of 40mm
on a universal pressure testing machine. After natural drying for 24 hours, it could be put into the
curing box for 28 days to obtain unburned bricks. Finally, the compressive strength, flexural strength
and frost return of these unburned bricks were studied when they were mainted for 7 days, 28 days and
water immersion for 24h.

2.3. Performance characterization
The compressive strength, flexural strength and frost return of the sample would be tested in
accordance with the standard test method of GB/T2542-2012. The phase identification was carried out
by XRD analyzer(D8) of Brooke company in Germany. The microstructure was analyzed with su8020
SEM analyzer of Hitachi, Japan. The leaching solution was extracted according to the horizontal
oscillation method (GB5085.3-2007), and the Na$^+$ concentration was determined using ICP-MS 7800
of Agilent.

3. Results and Discussion

3.1. Compressive and Flexural Strength
The non fired brick was prepared from red mud treated with different excitation time. The compressive
strength and flexural strength of the bricks after 7 days, 28 days and 24 hours of re leaching were
studied. The results were shown in figure1 and figure 2.
With the extension of excitation time, the compressive strength increased first and then decreased according to Fig. 1. When the excitation time was 6h, the strength after 7 days, 28 days and 24 hours of water immersion was large, which were 15.62Mpa, 18.55Mpa and 15.6Mpa respectively, reaching the MU15 level required by the national standard GB50574-2016. The flexural strength gradually increased with the extension of excitation time according to Fig. 2. When the excitation time was greater than 6h, the effect of time extension on flexural strength decreased. The flexural strength tended to be stable when the excitation time was 6h, which were 2.86Mpa, 3.58Mpa and 3.54Mpa respectively. It could also be seen that after 28 days of maintenance and 24 hours of water immersion, the compressive and flexural strength of unburned bricks decreased only a little. When the excitation time was 6 hours, they decreased by 16% and 1% respectively, meeting the requirements of building materials.
3.2. Frost Return
Non fired bricks were prepared from red mud treated with different excitation times, and they were maintained for 28 days for frost return tests. The results were shown in figure 3.

![Figure 3](image)

**Figure 3.** Effect of excitation time on frost return (a--0h,b--2h,c--4h,d--6h,e--8h).

The non fired bricks prepared from red mud treated at different excitation times did not return according to Fig.3. Then C sample was broken and analyzed by XRD, the result was shown in figure 4. At the same time, after five samples being crushed, the Na$^+$ concentration in solution were determined according to the horizontal shaking method. The results were shown in table 2.

![Figure 4](image)

**Figure 4.** XRD pattern of Unburned brick.

| Excitation/h | 0   | 2   | 4    | 6   | 8   |
|--------------|-----|-----|------|-----|-----|
| Na$^+$/mg/L  | 384.55 | 379.42 | 375.23 | 377.44 | 368.55 |

It was generally believed that high alkali content in red mud would lead to frost return, in which the alkali existed in the form of simple Na$_2$O. It could be seen from figure 4 that sodium in red mud existed in stable sodium aluminum hydroxysilicate Al$_3$Na$_4$(OH)(SiO$_4$)$_3$ phase, not in simple Na$_2$O phase, so there was no frost return phenomenon. It could be seen from table 2 that after crushing the unburned brick, the sodium ion concentration in the leaching solution measured by the horizontal shaking method was only about 370mg/L, which was very low compared with 5-10g/L in the leachate of the red mud yard, which also explained that the above samples would not return frost.
4. Conclusion
The red mud from an aluminum oxide plant in Guizhou was excited by a compound activator. The effects of excitation time on the compressive strength and flexural strength after 7 days, 28 days and 24 hours of immersion were investigated. When the excitation time was 6h, the compressive strength were 15.62Mpa, 18.55Mpa and 15.6Mpa respectively, and the flexural strength were 2.86Mpa, 3.58Mpa and 3.54Mpa respectively, meeting the MU15 required by the national standard GB50574-2016. The sodium in the red mud finally existed in the stable sodium Hydroxylaluminosilicate Al$_3$Na$_4$(OH)(SiO$_4$)$_3$ phase, and the Na$^+$ concentration in the leaching solution extracted by the horizontal shaking method was 370mg/L. therefore, there was no frost return phenomenon in the prepared unburned brick samples.

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