

IMPROVING THERMO-MECHANICAL PROPERTIES OF TABULAR ALUMINA CASTABLES VIA USING NANO STRUCTURED COLLOIDAL SILICA

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ABSTRACT

Great attempts were made to reduce the amount of calcium aluminate cement (CAC) content in refractory castables to improve their hot strength. Using more than 2-3 wt% CAC may cause low melting phases formation in the refractory matrix leading to weak thermo-mechanical properties of the castables. Colloidal Silica can affect the structure of refractory castables to achieve superior thermo mechanical properties. Replacing calcium aluminate cement (CAC) by colloidal silica as a water base binder, speeds up drying, reduces the amount of liquid phase at high temperatures and may lead to mullite formation, which will increase the hot strength of the refractory castables. In this research, the influence of colloidal silica addition on bulk density, apparent porosity and HMOR of a tabular alumina based refractory castable containing have been studied. The results showed that samples containing colloidal silica have higher hot strength compared to those containing only CAC as binder due to the better compaction, less liquid phase formation at high temperature.

Keywords: Nano Silica, Alumina Castable, Cement, HMOR

INTRODUCTION

After more than 20 years of development and testing, low and ultra low-cement castables have proven their suitability for many applications in different industries particularly in iron and steel production. The main difference between the traditional and the castables with reduced cement content can be stated as fraction of fine (a few microns) and ultrafine (sub-micron) material addition.

These particles aid packing and increase thereby the compactness of the castables. Consequently, the cement and so the water needed for casting is lowered.[1] Using less water leads to less porosity when heated, so the castable loses less strength upon firing and will be less prone to gas and slag attack (*Fig. 1*). [2, 3]

Moreover, lowering cement content reduces CaO in alumina castables, decreases the amount of low melting phases at around 1200°C[2] which in turn improves the corrosion resistance and creep strength in service.[4]

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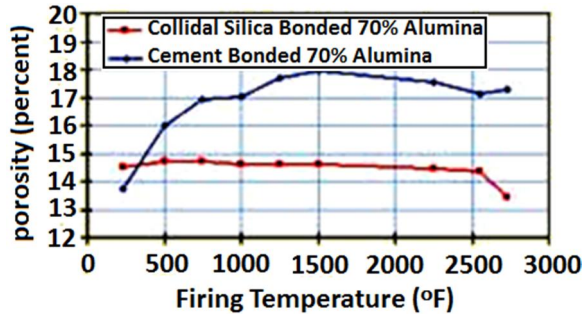


Fig. 1 – A comparison of porosity for cement bonded and colloidal silica bonded (a sub-micron agent) refractories[3].

Colloidal Silica is a sole with ultra fine particles of silica (*Fig. 2*) which can affect the structure of refractory castables due to its superior thermo mechanical properties. Replacing calcium aluminate cement (CAC) by colloidal silica as a binder, provides all the advantages of the low and ultra low cement castables, while eliminating most of their disadvantages. [5]. Although the colloidal silica bonded products show a lower initial strength than cement bonded ones due to decreased hydraulic bond, they show a good progression in strength development similar to the cement bonded products at moderate temperatures. At elevated temperatures, the strength differences become more dramatic.

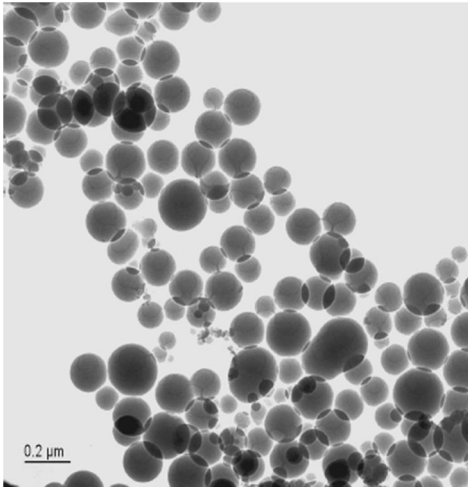


Fig. 2 – Colloidal Silica under TEM [6]

Low-melting-temperature $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ phases associated with CAC castables are responsible for liquid phase formation which resulting lower hot strengths. Because the colloidal silica bonded materials are CaO-free, they do not generate these low melting phases and typically exhibit higher hot strengths which results in better in-service erosion resistance for these materials. [3] Also Silica fumes with extremely small particle size (average size is 0.15 μm) replace water by

attaching themselves to cement particles because of their opposite charge; so, lower water requirement reduces the porosity while increases the density and

strength. Moreover, since the cement is partially hydrated, there is no loss of strength for the colloidal silica containing castable at intermediate temperatures.[7] Another factor which increases the strength of colloidal silica bonded castables at high temperatures is the mullite bond formation. Mullite formation in refractory castables has been found dependant on several factors[8] such as using nano-sized silica which promotes it and causes mullite formation at lower temperatures. [9,10] Mullite is a very much desirable phase in castables developing improved hot strength, creep resistance, good thermal stability as well as slag penetration resistance.[11] In the present study, apparent porosity, bulk density, hot modulus of rupture and phase analysis of a cement-free colloidal silica containing castable have been compared to those of the normal LCC one.

EXPERIMENTAL

1. Raw Materials

The chemical analyses of the raw materials used in this study are listed in *Table 1*.

Table 1- Chemical analyses of the raw materials

	<i>Tabular Alumina, (wt%)</i>	<i>Reactive Alumina, (wt%)</i>	<i>Calcium Aluminate Cement(wt%)</i>	<i>Colloidal Silica, (wt%)</i>
Al ₂ O ₃	99.4	99.5	69.8-72.2	0
CaO	0.03	0.03	26.8-29.2	0
SiO ₂	0.03	0.04	0.2-0.6	40.2
Fe ₂ O ₃	0.02	0.02	0.1-0.3	0
Na ₂ O	0.35	0.04	< 0.5	0
K ₂ O	0	0		0

The recipes of the studied castables (sample C, which is a low cement castable, LCC, containing 5 wt% cement and sample N, a no-cement castable containing colloidal silica) are provided in *Table 2*.

Table 2 – Composition of castables

<i>Castable Composition (wt%)</i>	<i>C</i>	<i>N</i>
Tabular Alumina	85	87
Reactive Alumina	10	10
Cement	5	0
Colloidal silica	0	7.5
Dispersant	0.1	0.1
water	5	4.5

2. Castables Preparation

For each sample, a 3kg batch mixture using the formulation in *Table 2* was prepared by dry mixing for 4 minutes at slow speed, using a Hobart mixer with 5 liter capacity mixing bowl. Then wet mixing was carried out by addition

of water with further 4 minutes mixing at medium speed. Test samples were prepared by casting the mixture into the stainless steel molds for 1 minute, with the dimensions of 150*25*25 mm by vibrating at 50HZ, The castables were cured at 20°C and a relative humidity of 95% for 24 hours in the mold. After curing time, the samples were allowed to be dried at the temperature of 110°C, and then fired at 1400°C for 5 hours (by the rate of 300°C per hour).

3. Testing

Bulk density and apparent porosity were determined by ASTM standard methods. The given values of these properties in result section are the average of three samples for each formulation. Hot modulus of rupture, HMOR, was carried out according to DIN 51048 using the Netzsch 442D/3 model. These values are also the average of three measures.

RESULT AND DISCUSSION

1. Bulk density and apparent porosity

Bulk density and apparent porosity of the samples are given in Fig 3 and

Fig 4.

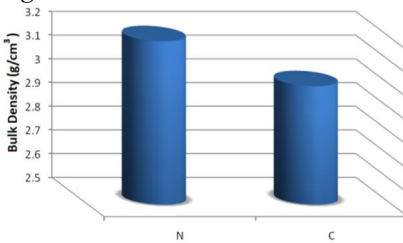


Fig. 3 – Bulk density of the samples fired at 1400°C for 5 hrs

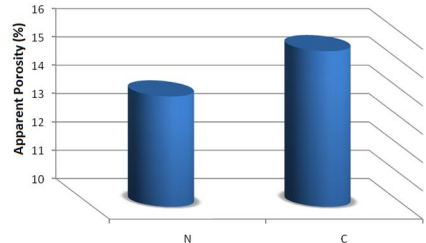


Fig.4 – Apparent porosity of the samples fired at 1400°C for 5 hrs

In LCC castables (Sample C), the cement content requires water for placement. This leads to high porosity when heated, the castable loses strength and becomes vulnerable to infiltration e.g. by slags. Besides, due to dehydration of the hydrate phases before the ceramic bond is formed, the castables also show a strength lowering at intermediate temperatures.[2] Colloidal silica particles, due to their nano sizes (~15 nm), could behave as a liquid[12] and so less water was needed in preparation of the sample N, which led to less amount of apparent porosity upon drying and firing.

Colloidal silica, due to its high specific area, anticipates sintering and so the sample N shows higher bulk density than sample C.

2. HMOR

The HMOR amounts of the samples are shown in Fig. 5. Using fine and reactive powders such as reactive alumina and colloidal silica in the bond phase of sample N can precipitate mullite needles from 1300°C in which the mullite needle-like particles grow out of a the matrix promote a good reinforcement

according to Schumacherr's report. [2] In *Fig. 5*, It can be seen that the sample containing colloidal silica (Sample N) shows higher amount of HMOR, better compaction, lower apparent porosity and higher bulk density due to presence of colloidal silica and consequently, formation and growth of long, needle-shaped mullite crystals.

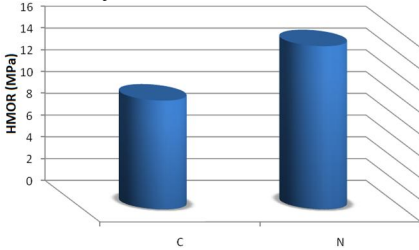


Fig.5 – Hot Modulus of Rupture (HMOR) of the samples fired at 1400°C for 5 hrs

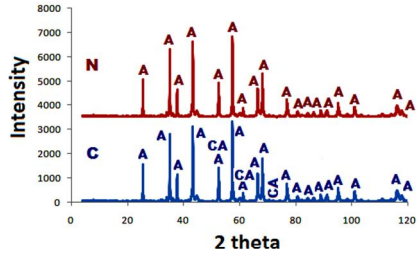


Fig. 6 – XRD patterns of the samples fired at 1400°C for 5 hrs. A represents alumina and CA represents calcium-hexaluminate($6\text{Al}_2\text{O}_3 \cdot \text{CaO}$)

3. Phase Analysis

The XRD patterns of the samples can be seen in *Fig. 6*.

In the XRD pattern of sample C, calcium aluminium oxide can be detected besides alumina, which is a cement phase and has a lower melting point than alumina and acts as a flux, so can lower the HMOR of the sample.

Phase analysis of the sample N reveals mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) peaks, although they are weak due to low amount in the total phase constituents of the castable, so mullite was assumed as a trace phase.

CONCLUSIONS

Colloidal silica sol could behave as a liquid and so the castables containing colloidal silica need less water to be prepared, which leads to lower their apparent porosity than that of LCC ones. Colloidal silica, due to its high specific area, anticipates sintering and so leads to better compaction. Comparing the traditional low calcium aluminate cement castables, colloidal silica containing refractory castables attains better mechanical strength at high temperatures.

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