

# EFFECT OF PARTICLE SIZE OF CAC ON STRENGTH OF CASTABLES AT 110°C AND 800°C

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## ABSTRACT

The particle size of calcium aluminate cements (CAC) has a close relationship with the morphology and distribution of hydration products. In this work, two types of cements with and without grinding were used and the influence of particle size distribution of CAC on strength of castables heat treat at 110°C and 800°C was investigated. Matrixes containing different particle size distribution of CAC have been analysed by XRD to investigate the relationship between distribution of hydration products and strength of castables at 110°C and 800°C.

## INTRODUCTION

Calcium aluminate cement (CAC) is widely used in refractory castables as hydraulic binder<sup>[1,2]</sup>. There are many factors which could influence the hydration of CAC in the castables, such as particle size of CAC<sup>[3]</sup>, curing time, curing temperature, micro powder<sup>[4]</sup> and additives<sup>[5]</sup>. It is worth noting that the particle size of CAC not only influences the hydration behavior of CAC<sup>[6]</sup>, but also affects the setting and hardening processes of castables<sup>[7]</sup>.

It is reported that the mechanical strength of CAC bonded castables usually decays at intermediate temperatures ranging from 300°C to 1000°C, which is associated with the decomposition of hydrates at the temperatures<sup>[8]</sup>. It has been also reported that addition of CAC with reduced sizes increased the strength of CAC bonded castable after drying at 110 °C, because smaller-sized CAC could accelerate its hydration rate to increase the hydrates in the castables<sup>[9]</sup>. These hydrates would dehydrate to be small-sized CaO·Al<sub>2</sub>O<sub>3</sub> (CA) and CaO·2Al<sub>2</sub>O<sub>3</sub> (CA<sub>2</sub>) at intermediate temperatures<sup>[10]</sup>. However, the effect of CAC with different particle size on the strength of castables in medium temperature was not reported.

In this work, a commercial CAC was ground to obtain cements with different particle size. The strength of castables containing CAC with different particle size after firing at 110°C and 800°C was tested. The phase composition of the matrix was analyzed by XRD to investigate the relationship between distribution of hydration products and strength of castables at 110°C and 800°C.

## EXPERIMENTAL PROCEDURE

The hydraulic binder used in this work is a commercial CAC. Tab. 1 and Tab. 2 respectively show the chemical composition and the major phase composition of CAC. The cement was dry ground for one hour to decrease the particle sizes. For each batch, 5 kg of the commercial cement was fed into a laboratory mill (500 mm in length and 500 mm in diameter). A combination of 100 kg cylindrical steel rods and spherical steel balls was used as the grinding media with a cement/grinding media weight ratio of 1/20. The particle size distribution of CAC with grinding and without grinding was shown in Tab. 3.

Tab. 1: Chemical composition of calcium alumina cements (wt.%).

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	TiO <sub>2</sub>	K <sub>2</sub> O	Na <sub>2</sub> O
0.22	69.83	0.16	29.5	0.23	0.02	0.01	0.28

Tab. 2: Major phase composition of calcium alumina cements (wt.%).

CA/CA <sub>2</sub>	CA	CA <sub>2</sub>	C <sub>2</sub> AS	α-Al <sub>2</sub> O <sub>3</sub>
1.61	58.44	36.26	0.76	1.62

Tab.3: Particle size distribution and the specific surface area of CAC with and without grinding.

CAC	SSA m <sup>2</sup> /kg	Particle size distribution (μm)				
		D10	D25	D50	D75	D90
Without grinding	403	1.83	3.39	12.41	32.12	47.18
Grinding for 1 h	530	1.06	1.64	3.23	5.71	8.71

The CAC bonded castables were composed of tabular alumina aggregate (Al<sub>2</sub>O<sub>3</sub>> 99 wt%, 6-3 mm, 3-1 mm and 1-0 mm), tabular alumina powder (Al<sub>2</sub>O<sub>3</sub>> 99 wt%) reactive alumina (Al<sub>2</sub>O<sub>3</sub>> 99 wt%) and CAC with different particle size distribution, FS10 was used as dispersants. The mixtures were dry-mixed for 1 min, followed by 2 min wet mixing were carried out in a paddle mixer. After that, the castables were cast in moulds of 160mm×40mm×40mm with vibration, cured for 24h in room temperature. Then, the samples were de-molded

and dried for 24h at 110°C, and fired at 800°C for 3h. Cold modulus of rupture (CMOR) and cold crushing strength (CCS) of the samples were tested after drying and firing. XRD was used to characterize the phase composition of the matrix of the castables.

## RESULTS AND DISCUSSION

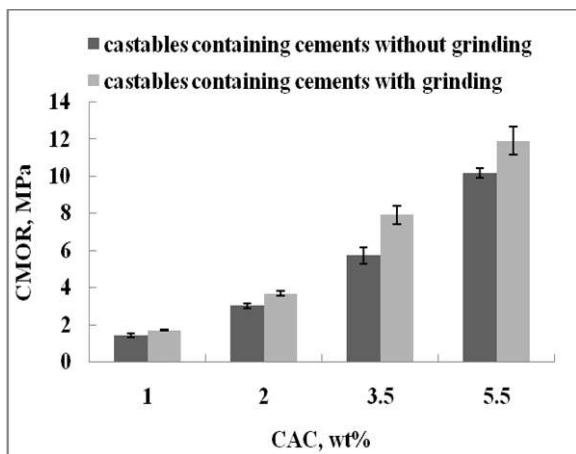


Fig. 1: CMOR of the castables containing cements with and without grinding dried at 110°C for 24h.

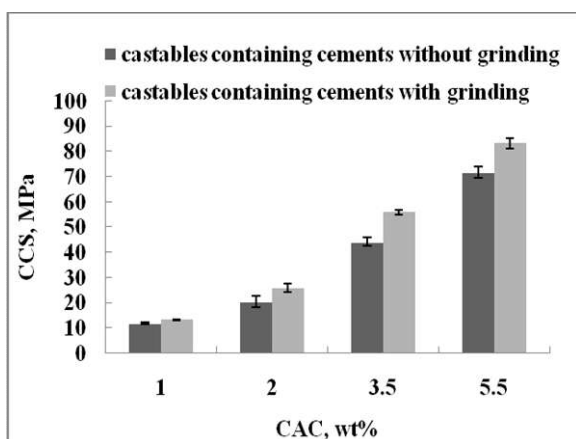


Fig. 2: CCS of the castables containing cements with and without grinding dried at 110°C for 24h.

Figs. 1 and 2 show the CMOR and CCS of the castables containing cements with and without grinding dried at 110°C for 24h. It can be seen that the strength of castables containing CAC with grinding is higher than the castables with CAC as-received. For example, The CMOR and CCS of the castables containing 5.5wt.% CAC with grinding are 11.9MPa and 83.35MPa, respectively. In contrast, the CMOR and CCS of the castables containing 5.5wt.% CAC without grinding are 10.16MPa and 71.61MPa, respectively. The result suggests that the cold strengths of the CAC bonded castables increases with the decrease of particle size of CAC.

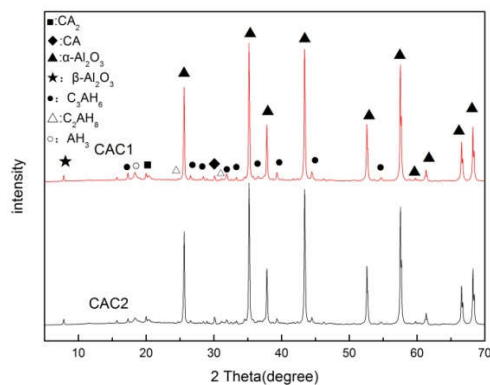


Fig. 3 XRD patterns of the matrix of the castables containing 5.5wt% cements after drying at 110°C for 24h.

To understand why smaller particle size of CAC addition raised the strength of the castable after drying at 110°C for 24h, the castable matrixes containing cements with and without grinding was investigated by XRD. Fig. 3 shows the XRD patterns of the matrix of the castables containing 5.5wt.% cements after drying at 110°C for 24h. As seen in Fig. 3, the phase composition of the castable matrix containing CAC with grinding is same as the sample containing CAC as-received. However, the peak intensities in the two castable matrixes are varied. It can be seen that the peak heights of CA, CA<sub>2</sub> and C<sub>2</sub>AH<sub>8</sub> are lower in the sample containing ground CAC compared with the sample containing CAC as-received, whereas intensities of C<sub>3</sub>AH<sub>6</sub> is higher in the sample with CAC with grinding than with CAC without grinding. This indicates that the content of C<sub>2</sub>AH<sub>8</sub> of the sample containing CAC with reduced sizes is less than the sample containing CAC as-received, and the content of C<sub>3</sub>AH<sub>6</sub> of the sample containing smaller sized CAC is higher. The results show that the decrease of particle size of CAC promotes the hydration reaction of cements. That is, the smaller particle size of CAC is beneficial to convert metastable C<sub>2</sub>AH<sub>8</sub> to stable C<sub>3</sub>AH<sub>6</sub>.

Three distinct processes are involved during hydration of CAC, viz. dissolution, nucleation and precipitation. Actually, during the dissolution period, CAC with a larger particle size would not completely dissolve. After dissolution and nucleation duration, hydrate products are generated on the surface of the unhydrated CAC. So it is hard for water to penetrate through the hydrate products, the hydration reaction cannot take place without sufficient water. Hence, the strength of castables containing CAC with reduced sizes after drying at 110°C is lower on account of lower hydrate products content. In contrast, CAC with a smaller particle size is fully in contact with water for the high specific surface area. More hydrate products are generated with a smaller grain size. Thereby, the castables

containing smaller sized cements exhibit high strengths.

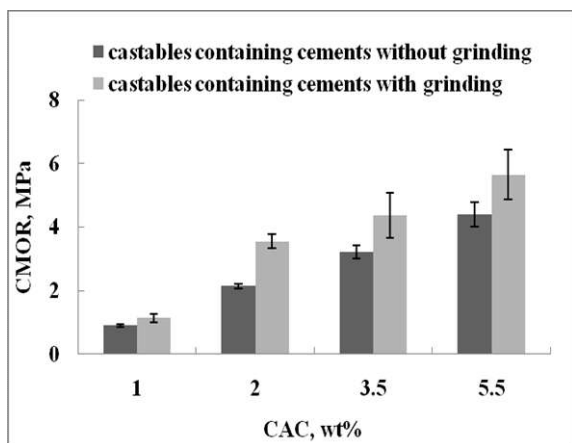


Fig. 4: CMOR of the castables containing cements with and without grinding fired at 800°C for 3h.

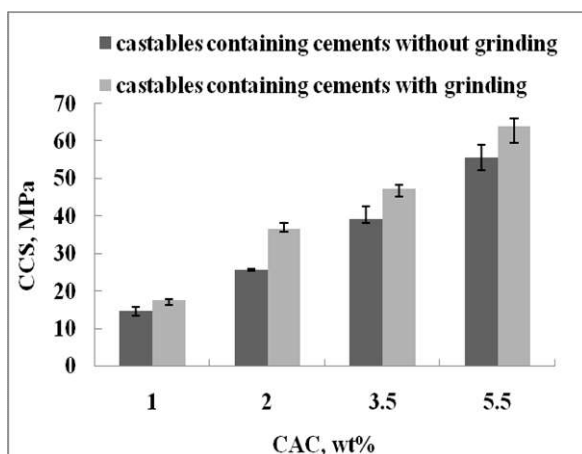


Fig. 5: CCS of the castables containing cements with and without grinding fired at 800°C for 3h.

Fig. 4 and Fig. 5 show the CMOR and CCS of the castables containing cements with and without grinding fired at 800°C for 3h. It shows that the CMOR and CCS of castables with ground CAC after firing at 800°C is higher than the castables containing CAC as-received. For example, The CMOR and CCS of the castables containing 3.5wt.% CAC with grinding are 4.37MPa and 47.37MPa, while the CMOR and CCS of the castables containing 3.5wt.% CAC without grinding are 3.22MPa and 39.29MPa. The result also suggests that the cold strengths of the CAC bonded castables at intermediate temperature increase with the decrease of particle size of CAC. That is because the hydrate products produced by CAC with grinding are smaller and the particle size distribution is uniform, so the defects generated by the decomposition of hydrate products fired at 800°C are also small, the structure of castables is more homogeneous.

## CONCLUSIONS

Two types of CAC with different particle size were used in this work. The cold strength of the CAC bonded castables drying at 110°C for 24h increases with the particle size of CAC

decreases. The XRD patterns of the matrix also show that compare to the samples containing CAC as-received, the contents of CA, CA<sub>2</sub> and C<sub>2</sub>AH<sub>8</sub> of the samples with ground CAC is lower, and the contents of C<sub>3</sub>AH<sub>6</sub> is higher. It is due to CAC with larger particle size would not completely dissolve, unhydrated CAC is covered with hydrate products, it is hard for water to penetrate through the barrier, the hydration reaction cannot take place without sufficient water. Therefore, the decrease of particle size of CAC promotes the hydration reaction of cements.

It is also found that the castables with cements with grinding exhibit high strength at 800°C for 3h. This is because the defects produced by the decomposition of the small hydrate products fired at 800°C are also small and uniform. So the structure is beneficial to the strength of the castables.

## ACKNOWLEDGEMENTS

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## REFERENCES

- [1] Golshan NH, Sarpoolaky H, Souri A, Nourbakhsh A. Influence of colloidal silica on microstructure and properties of forsterite bonded magnesite castables refractory. Proceeding of 51<sup>st</sup> International Colloquium on Refractories; 2008. p. 147-51.
- [2] Ewais EMM, Khalil NM, Amin MS, Ahmed YMZ, Barakat MA. Utilization of aluminum sludge and aluminum slag (dross) for the manufacture of calcium aluminate cement. *Ceram Int.* 2009;35(8): 3381-8.
- [3] Zhang C, Wang Y, Guo B, Shang X, Wang Q, Ye G Effect of Particle Size Distribution on the Hydration Behavior of Calcium Aluminate Cements. *Refractories Worldforum.* 2016;8 (1):75-8.
- [4] Shang X, Ye G, Zhang Y, Li H, Hou D. Effect of micro-sized alumina powder on the hydration products of calcium aluminate cement at 40°C. *Ceram Int.* 2016;42(13):14391-4.
- [5] Shang X, Zhang C, Li H, Zhou W, Wang Q, Ye G. Effect of Sodium Tripolyphosphate on Hydration and Setting Behavior of Different Calcium Aluminate Cements. Proceeding of UNITECR` 15; 2015 Sep 15-18;Vienna, Austria. 2015.
- [6] Ai Qin W, Chengzhi Z, Ningsheng Z. The theoretic analysis of the influence of the particle size distribution of cement system on the property of cement. *Cem Concr Res.* 1999;29(11):1721-6.
- [7] Guan B, Ye Q, Wu Z, Lou W, Yang L. Analysis of the relationship between particle size distribution of  $\alpha$ -calcium sulfate hemihydrate and compressive strength of set plaster-using grey model. *Powder Technology.*

2010;200(3):136-43.

[8] Ismael MR, Salomão R, Pandolfelli VC. Refractory castables based on colloidal silica and hydratable alumina. *Am Ceram Soc Bull.* 2007;86(9):58-61.

[9] Zhang C. Effect of boron oxide and particle size on hydration of calcium aluminate cement and properties of cement bonded castables. Doctoral dissertation, Zhengzhou University. 2017.

[10] Shang X, Yin Y, Li H. The influence of alumina powders on the volume stability of corundum-based castables. *Proceeding of 7<sup>th</sup> International Symposium on Refractory*; 2016 Sep 20-22; Xi'an, China. 2016.