Effect of Al(OH)$_3$ and La$_2$O$_3$ on the Sintering Behavior of CaO Granules via CaCO$_3$ Decomposition

Bingrong Li, Yaowu Wei*, Nan Li, Tao Zhang, Jinhu Wang
The State Key Laboratory of Refractories and Metallurgy, Wuhan University of Science and Technology, Wuhan 430081, China

Abstract:
Hydration resistance of CaO aggregates is the key to the successful application of lime-based refractories. Granulation technology on limestone with additives and one step calcination method was used in this study to investigate the effects of Al(OH)$_3$ and La$_2$O$_3$ on the sintering behavior and the hydration resistance of CaO granules. The result showed that the hydration resistance of CaO granules was improved significantly by adding Al(OH)$_3$ and La$_2$O$_3$. The shell of sintered CaO granules was relatively dense after calcination and the specific surface area of CaO granules decreased from 7.5×10$^{-2}$ m$^2$/g to 1.2×10$^{-2}$ m$^2$/g. The average pore size of CaO granular was decreased from 3.54 μm to 0.83 μm due to the liquid phase sintering when Al(OH)$_3$ additive was used. La$_2$O$_3$ promoted the densification of CaO compact by the ion substitution and solution with CaO. La$^{3+}$ approached into CaO lattices, which enlarged the vacancy concentration of Ca$^{2+}$ and accelerated the diffusion of Ca$^{2+}$.

Keywords: Limestone; CaO granules; Granulation; Sintering; Hydration resistance.

1. Introduction
Lime-based refractories are often used in secondary steelmaking processes due to its relatively low vapor pressure at high temperatures and numerous high-temperature superior properties [1-3]. Furthermore, with the development of clean steel production, lime-based refractories have been becoming more and more important [4, 5]. CaO is one of the most suitable refractory constituents which can meet with requirements on durability and purifying function. Lime-based refractories are considered as an essential direction for the development of metallurgical industries and other high-temperature fields [6, 7]. However, lime can easily react with moisture in the air, and that damage to lime-based refractories. A lot of works has been done to prepare densified lime-based refractories by adding admixtures [8-12], granulation [13, 14] and surface treatment [15]. For example, secondary calculations at high temperatures [16] to enhance the hydration resistance of lime-based refractories. Different additives can promote the sintering process of lime and the effect of additives on the sintering process of lime can be divided into two mechanisms [17]. One was confirmed that some can enhance solid-state sintering, such as La$_2$O$_3$, MgO and ZrO$_2$. The other including Fe$_2$O$_3$, Al$_2$O$_3$, and CuO that can improve the sintering of lime through liquid phase sintering. An interesting approach is the method combined granulation and calcination technology to improve the hydration resistance of CaO samples was described by Qi Zhang and Yaowu Wei [13], the CaO granules can be used for producing lime-based refractories directly without crushing.

* Corresponding author: weiyaowu@wust.edu.cn (Yaowu Wei)
It was reported that CaO derive directly from the decomposition of CaCO₃ was very reactive and highly sinterable [18,19], but many works so far have focused on secondary calculation method to get densified lime sample because that can destroy CaCO₃ pseudomorph in limestone sintering and promote the densification of the sample. But the secondary calculations technology was high energy consumption. Chen et al. obtained the compact CaO clinkers with good hydration resistance which directly sintered from lightweight CaCO₃ by adding MgO and ZrO₂, and the addition of MgO and ZrO₂ promoted the solid-state sintering, inhibited the secondary recrystallization, and made a homogeneous CaO microstructure [20]. Hence, one step calcination technology with proper additive is hopeful to reduce the preparation cost of CaO granules.

The primary objective of this work is to investigate the effects of Al(OH)₃ and La₂O₃ on the sintering behavior and the hydration resistance of CaO granules, which derived directly from the decomposition of limestone powder to find a new economical way to produce CaO granules that can be used for producing lime-based refractories directly without crushing.

2. Materials and Experimental Procedures

The starting materials used in this work were limestone powder (d₅₀ = 12 μm), Al(OH)₃ powder (AR, Aladdin Industrial Corporation, Shanghai, China) and La₂O₃ powder (AR, Aladdin Industrial Corporation, Shanghai, China). The chemical composition of limestone was shown in Tab. I and the compounding of batches were shown in Tab. II. Batches were prepared by mixing the limestone powder with 0.5, 1.0 and 1.5 wt% additives in a Poly Urethane barrel mill for 2 hours. The batches were granulated by EIRICH mixer (RV02E; Maschinenfabrik Gustav Eirich, Hardheim, Germany), and then CaCO₃ granules were dried at 110°C for 24 hours. Finally, CaCO₃ granules were treated at 1650°C for 3 hours and then CaO granules were obtained. The photos of CaO granules are shown in Fig. 1.

| Sample          | S₀ | Al₀.₅ | Al₁.₀ | Al₁.₅ | La₀.₅ | La₁.₀ | La₁.₅ |
|-----------------|----|-------|-------|-------|-------|-------|-------|
| Limestone power | 100| 99.5  | 99.0  | 98.5  | 99.5  | 99.0  | 98.5  |
| La₂O₃ powder    | -  | 0.5   | 1.0   | 1.5   | -     | -     | -     |
| Y₂O₃ power      | -  | -     | -     | -     | 0.5   | 1.0   | 1.5   |

Fig. 1. Photo of CaO granules: (a) Granules with Al(OH)₃ and (b) Granules with La₂O₃.
Phase analysis was characterized by X-ray diffractometry (XRD, Philips Xpert TMP) with Cu Kα radiation (λ=1.54187 Å) at a scanning speed of 2° per min. The pore size distributions were measured through mercury porosimetry measurement (AutoPore IV 9500, Micromeritics Instrument Corporation). The true density (Automatic true density analyzer, ACCUPYC1330; Micromeritics Instrument Company, USA), bulk density, apparent porosity and hydration resistance of CaO granules were tested. The bulk density and apparent porosity was measured based on the Archimedes' principle using kerosene as the medium. The microstructure of CaO granules was investigated by scanning electron microscopy (JSM 6610, JEOL, Japan) and energy spectrum analysis (QUANTAX 200 30; BRUKER, Berlin, Germany). The average grain size of the CaO granules in SEM was counted via Image-pro plus 6.0 software (Bethesda, MD, USA).

Two methods were used to evaluate the hydration resistance of CaO granules:

1. Hydration resistance of sintered lime samples was measured in the humidity chamber.
   CaO granules with different additive were placed in a humidity chamber at 50 °C and 90 % humidity for 10 hours. The hydration resistance of CaO granules was evaluated by Equation (1).
   \[
   H_c = \frac{W_1 - W_0}{W_0} \times 100\%
   \]  
   (1)
   Where \(W_0\) is the original weight of CaO granules and \(W_1\) is the dry weight of CaO granules after curing in the humidity chamber. \(H_c\) is the weight gain of CaO granules, the lower the \(H_c\), the better the hydration resistance of CaO granules.

2. Hydration resistance of CaO granules in the air
   The hydration resistance of CaO granules was evaluated by measuring the mass gain after soaking the granules in the air. \(M_0\) gram CaO granules with a particle size of 3-1 mm were placed in the air. \(M_x\) is the dry weight of CaO granules after soaking in the air for regular days. The hydration rate was calculated as shown in Equation (2). \(H_a\) is the weight gain of CaO granules, the lower the \(H_a\), the better the hydration resistance of granules.
   \[
   H_a = \frac{M_x - M_0}{M_0} \times 100\%
   \]  
   (2)

The closed porosity (πf) of the sample is the ratio of the total volume of the closed pores in a porous body to its bulk volume, which can be calculated via the Equation (3).
   \[
   \pi_f = \frac{V_2}{V_0 + V_2} \times 100\% = \left(1 - \frac{\rho_b}{\rho_t}\right) \times 100\%
   \]  
   (3)
   Where: \(\pi_f\) - The closed porosity of the sample;  
   \(V_1\) - The true volume of the sample (cm³);  
   \(V_2\) - The volume of the closed pores in the sample (cm³);  
   \(\rho_b\) - The bulk density of the sample (g/ cm³);  
   \(\rho_t\) - The true density of the sample (g/ cm³);

The cylindrical compressive strength test method of CaO particles was measured by taking an appropriate amount of 8-5 mm granules into the pressure cylinder, the corresponding pressure value was recorded when the downward movement distance of the stamping die reached 20 mm. The strength of the cylinder of CaO particles was calculated as equation (4).
\[ P = \frac{F}{s} \]  \hspace{1cm} (4),

where: \( P \) - The cylinder strength of CaO granules (MPa); \( F \) - The pressure value when the downward movement distance of the stamping die reaches 20 mm (N); \( s \) - Pressure area (cm\(^2\)).

3. Results and Discussion

3.1. Phase composition

Fig. 2a shows the XRD patterns of the CaO granules with Al(OH)\(_3\) sintered at 1650°C for 3h. CaO as the main crystalline phase in additive-free CaO granules (sample A\(_0\) and La\(_0\)). C\(_3\)A phase was detected in CaO granules when Al(OH)\(_3\) was used as the additive in the granulation process. Fig. 2b shows the XRD patterns of the CaO granules with La\(_2\)O\(_3\) sintered at 1650 °C for 3h. The major diffraction peaks can be indexed as CaO phase for all the CaO samples no matter how much La\(_2\)O\(_3\) was used. The position of the diffraction peaks of CaO crystallographic was shifted (right side in Fig. 2b) as a result of adding La\(_2\)O\(_3\), suggesting that La\(^{3+}\) entered into the CaO lattice, changing the lattice parameter of CaO.

![Fig. 2. XRD patterns of sintered CaO granules: (a) Al(OH)\(_3\) additive and (b) La\(_2\)O\(_3\) additive.](image1)

3.2. Density and porosity of CaO granules

![Fig. 3. Effects of additive on the density and porosity of CaO granules. (a) Bulk density; (b) Apparent porosity.](image2)

Fig. 3 shows the effect of additives on the bulk density and apparent porosity of CaO granules. With the increment of Al(OH)\(_3\) additive, the bulk density was increased gradually.
from 2.57 g/cm³ to 2.87 g/cm³ and the apparent porosity decreased from 20.7 % to 4.3 %.

Similarly, with the increment of La₂O₃ additive, the bulk density was increased gradually from 2.57 g/cm³ to 2.96 g/cm³ and the apparent porosity was decreased from 20.7 % to 3.7 %.

Fig. 4 shows the relationship between the closed porosity of CaO granules and the additive amount. It can be seen from Fig. 4 that the closed porosity of CaO granules with additives decreased significantly. The closed porosity of additive-free CaO granules was 21.6% and decreased to 9.8-10.4% when Al(OH)₃ or La₂O₃ was introduced.

Fig. 4. Relationship between the closed porosity of CaO granules and additive amount.

3.3. Pore size distribution and microstructure

The pore size distributions of the sintered specimens are shown in Fig. 5. The unimodal pore size distributions are observed in all specimens. In additive-free CaO granules, the peak distribution of pores was in the ranges of 1.5-3 μm and decreased markedly. With the increment of additives, the peak of the pore in the specimens with 1.5 wt% Al(OH)₃ and 1.5 wt%La₂O₃ was almost disappeared. The average pore size of the additive-free CaO granules was 3.54μm, but the average pore size decreased from 3.54 μm to 0.87-0.83μm when the additives were introduced.

Fig. 5. The pore size distribution of CaO granules with additives, (a) Al(OH)₃ additive; (b) La₂O₃ additive.

To investigate the change in the pore size distribution, the microstructure of the CaO specimens with 0 and 1.5 wt% additives are shown in Fig. 6. The microstructures of the surface of the CaO granules were different. The additive-free CaO granules were porous, the
number of intergranular pores and intragranular pores were large, and the neck area between granules was fine. Comparing additive-free CaO granules, the densification of CaO granules with additive increased significantly, the size and number of the intragranular pores were very small. The intergranular pores were dominant pores in the microstructure and the neck area was larger. These findings explained the fact that the pore size distributions of CaO specimens were unimodal and the pore peak of CaO specimen with 1.5 wt% additives were almost disappeared. The microstructures were accordant with the results shown in Fig. 5.

The smaller pore size of CaO granules with additives was attributed to their lower special surface area. For example, the special surface of CaO granules was found to decrease from 7.5×10^{-2} m^2/g to 1.2×10^{-2} m^2/g and 1.3×10^{-2} m^2/g when 1.5 wt% Al(OH)₃ or 1.5 wt% La₂O₃ was introduced (Tab. III). The relationship between the cylindrical compressive strength of CaO granules and the additive amount was shown in Fig. 7. It can be seen from the result that the cylindrical compressive strength of CaO granules with additives was increased obviously.

Tab. III. The special surface of CaO granules, (×10^{-2}) m²/g.

| Amount of additive, wt % | 0   | 0.5 | 1.0 | 1.5 |
|--------------------------|-----|-----|-----|-----|
| Al(OH)₃                  | 7.5 | 5.4 | 4.5 | 1.2 |
| La₂O₃                    | 7.5 | 5.4 | 3.4 | 1.3 |

Fig. 7. Relationship between the cylindrical compressive strength of CaO granules and additive amount.

Fig. 8 shows the SEM image and EDS results of inner CaO granules. It can be seen by comparing the microstructure of CaO granules in Fig. 6 and Fig. 10 that the surface of CaO
granules was much denser than that of interior and many pores were found in the interior of the granules. The densification of CaO granules with additive was increased when comparing the additive-free CaO granules, the large pores were gradually eliminated in CaO granules with additive and a little pore were presented in intergranular. It can be seen from Tab. IV that the average CaO grain size was increased from 28.4 μm to 46.8 μm and 54.2 μm when Al(OH)₃ and La₂O₃ were introduced in the preparation process of CaO granules.

Fig. 8. SEM images of CaO granules: (A) Interior of additive-free CaO granules (B) Interior of CaO granules with 1.5 wt% Al(OH)₃ (C) Interior of CaO granules with 1.5 wt% La₂O₃.

Tab.IV. The average grain size of CaO granules, μm.

| Amount of additive, wt% | 0   | 0.5 | 1.0  | 1.5  |
|------------------------|-----|-----|------|------|
| Al(OH)₃                | 28.4| 41.9| 42.1 | 46.8 |
| La₂O₃                  | 28.4| 34.8| 37.0 | 54.2 |

Fig. 8A shows the microstructure of additive-free CaO granules. It can be seen that CaO grains grew with an increment of irregular grain boundaries, the pores were presented in intergranular positions and usually in grain boundary triple point. Fig. 8B and Fig. 8C shows the microstructure of a specimen with 1.5 wt% Al(OH)₃ and 1.5 wt% La₂O₃, respectively. The number of pores of CaO granules with additives decreased significantly and the remaining pores were present in the grain boundary and intragranular position.

It can be seen from Fig. 8B that CaO grain was surrounded by a light gray phase (C₃A) that identified by EDS (Tab. IV). C₃A was characterized by low melting points that were located in the grain boundary triple point. With the increment of Al(OH)₃ additive, a homogeneous microstructure with low porosity (4.3 %) was formed.

The microstructure analyses of specimens with La₂O₃ are shown in Fig. 8C. A homogeneous microstructure composed mainly of a well-distributed phase. It can be seen that the dark gray phase was identified as CaO (Tab.V). CaO grain was surrounded by a light gray phase, which composed of elements Ca and La. The CaO grain size was increased and the
number of pores was decreased when 1.5 wt% La$_2$O$_3$ was introduced, the homogeneous microstructure with low apparent porosity was formed at the same time.

| Spectrum | Ca   | O    | Mg  | Al  | La  |
|----------|------|------|-----|-----|-----|
| 1        | 68.95| 30.86| 0.21| -   | -   |
| 2        | 46.08| 47.33| 0.76| 5.82| -   |
| 3        | 44.59| 8.91 | 0.54| -   | 41.72|

3.4. Hydration resistance

The hydration resistance of CaO granules in the humidity chamber was evaluated by Equation (1) and the result was shown in Fig. 9. It can be seen from Fig. 9 that both Al(OH)$_3$ and La$_2$O$_3$ were favorable for the hydration resistance of CaO granules. It is observed that the weight gain of CaO granules with additives decreased appreciably. The weight gain of additive-free CaO granules in the humidity chamber was 13.8 % and decreased to 1.6-1.8 % respectively when Al(OH)$_3$ and La$_2$O$_3$ were introduced.

The hydration resistance of CaO granules in the air was evaluated by measuring the mass gain after soaking the CaO granules in the air under a temperature of 20-28 °C and...
relative humidity of 49-87%. The hydration resistance of CaO granules in the air evaluated by Equation (2) and the result was shown in Fig. 10. It can be seen from Fig. 10 that the weight gain of CaO was increased with the increment of soaking days and both Al(OH)₃ and La₂O₃ were favorable for the hydration resistance of CaO granules. The optimal additive amount of Al(OH)₃ and La₂O₃ was 1.5 wt%, which were the same as that in the humidity chamber. It should be noticed that the hydration rate was gradually accelerated with the process of soaking days.

3.5. Discussion

As we know, even a small addition of oxide influences the densification, reducing the sintering temperature, suppressing or promoting the grain growth, modifying the microstructure and mechanical properties [21]. It is the general phenomenon that sintering aids could form a low-viscosity ternary liquid phase to promote densification via liquid-phase sintering, the process of sintering was accelerated by the decomposition product of Al(OH)₃. The reaction and sintering process of CaCO₃ and Al(OH)₃ was described in Fig. 11. At the initial stage, the contacts between the CaCO₃ and Al(OH)₃ were point contacts in the CaCO₃ granules (Fig. 11a). When the heating temperature increased to 300-800 °C, CaCO₃ and Al(OH)₃ decomposed to CaO and Al₂O₃, and then the CaCO₃ pseudomorphs and Al₂O₃ with micro-sized pores were formed [22, 23] (Fig. 11b). The local liquid phase was formed through the reaction among the impurities, CaO and Al₂O₃ when heating temperature rise to 1300 °C (which calculated by FactSage software and shown in Fig. 12a). The CaCO₃ pseudomorphs and Al₂O₃ both dissolved into the liquid phase (Fig. 11c) later, which favorable for wetting and spreading behaviors of the liquid phase. With the increment of heating temperature, the amount of liquid phase increased and then promoted the dissolution of the CaO and Al₂O₃ into the liquid phase (Fig. 11d). During this process, the Ca²⁺ and Al³⁺ inter-diffused between the CaCO₃ pseudomorphs and Al₂O₃ through the liquid phase because of their concentration gradients that resulted in grain growth through solution and precipitation. With the progress of the sintering, CaO grain size increased significantly and densification increased at the same time. Finally, the neck bonds of CaO grain were coarse and the liquid phase and pores were

**Fig. 11.** Schematic representation of the reaction sintering mechanism of CaO granules. (a) Packing of the particles; (b) Decomposition of the CaCO₃ and Al(OH)₃; (c) liquid Formation; (d) Penetration of the liquid and elimination of pores; (e) growth of the CaO grain.
located in grain boundary (Fig. 11e), which was by the microstructure that was shown in Fig. 10.

Fig. 12. Effect of additives on the liquid amount in CaO granules with temperature: (a) Al(OH)₃, (b) La₂O₃.

Due to the appearance of the liquid phase, the surface energy of pores on the surface of CaO granules changed from "solid-vapor" to "solid-liquid" as the dominant form of surface energy, which accelerated sintering by a solution-diffusion-precipitation (SDP) mechanism. Under the action of the capillary force of the liquid phase, the apparent porosity of CaO granules rapidly aggregated to form a dense sintering scarfskin and the grains coarse [24]. Based on the SEM images, due to the improvement of sintering and grain size growth, the densification of CaO granules was increased and formed a uniform structure, resulted in the formation of uniform pores. Pores acted as the penetration channels for water vapor and therefore the higher the pores volume, the lower the hydration resistance of CaO granules.

The porosity of CaO granules with La₂O₃ addition was decreased substantially when the porosity registered in the additive-free CaO granules was taken into consideration (Fig. 3). The improvement of bulk density is due to the higher true density of La₂O₃ (6.51 g/cm³), in comparison to the CaO (3.34 g/cm³). When the amount of La₂O₃ was 1.5 wt%, the bulk density increased appreciably, with a corresponding value of apparent porosity of 3.7 %. It can be seen from Fig. 12b that the liquid amount in CaO granules was very low with the increment of temperature. The densification mechanism was correlated with solid solution reaction; the solid solution resulted in high atomic mobility, encouraged mass transfer and enhanced direct bond formation. The sintering process of CaO granules with La₂O₃ was mainly controlled by the volume diffusion mechanism [25]. The radius of La³⁺ is 0.103 nm, while is similar to the radius of Ca²⁺ (0.092 nm). Thus, La₂O₃ formed a limited solid solution with CaO, and the defect reaction of vacancy formation by incorporating the additives can be expressed as follows:

\[
\text{La}_2\text{O}_3 \rightarrow 2\text{La}^{\text{Ca}} + \text{V}_\text{Ca} + 3\text{O}_0
\]  \hspace{1cm} (5)

Ca²⁺ was substituted by La³⁺ at the high temperature and enhanced CaO grain growth via vacancy formation. This phenomenon led to the growth of grain size was more distinct, the microstructure was modified, solid solution layer formed around CaO grain. The number of pores in intergranular or grain boundary triple point decreased during the sintering process as a result of grain boundary migration, leading to the formation of larger pores on the crystal surface, which was eliminated with the sintering process. The protective layer improved
hydratation resistance of CaO granules by preventing the CaO from contacting water vapor and the reduced initial formation of Ca(OH)$_2$ crystallites.

Although the hydration resistance of CaO granules in this study was not satisfied when compared with those prepared from calcium hydroxide [12-15], this study suggested the introduction of Al(OH)$_3$ and La$_2$O$_3$ improved the properties of CaO granules that prepared by one-step calcination technology. Compared with previous experiments by Ghosh[19], the average grain size of CaO in this study was increased and the weight gain in the humidity chamber was decreased significantly. Moreover, the prepared CaO granules can be directly used in the preparation of lime-based refractories without secondary crushing, which would improve the convenience of preparation of refractories. Moreover, it can be seen from Fig. 10a that the liquid amount of CaO granules was increased significantly with the increment of temperature and Al(OH)$_3$ content. Many studies have shown that too much liquid phase deteriorated high-temperature mechanical properties of refractories [19,20]. On the other hand, an excess amount of La$_2$O$_3$ addition accumulated at grain boundaries and prevented further growth of grains [4,25]. So, the amount of the additive in this study was controlled no more than 1.5wt%.

4. Conclusion

Section text CaO granules were fabricated from limestone via one-step calcination and subsequently fired at 1650 °C for 3 hours in this study, which derived directly from the decomposition of limestone. The introduction of Al(OH)$_3$ and La$_2$O$_3$ were both beneficial to the sintering of CaO granules but the mechanisms were different. The sintering of CaO granules was accelerated by the formation of a liquid phase when Al(OH)$_3$ was introduced in fabrication. The sintering was accelerated by the formation of a solid solution when La$_2$O$_3$ was introduced. Hence, the hydration resistance of the CaO granules with additives was improved significantly. Another important factor that improved the hydration resistance of CaO granules was the pores of CaO granules was decreased dramatically when the additive was introduced and the shell of CaO granules was relatively dense.

5. References

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