

Effect of Nano-SiO₂ Content on the Setting Time and Mechanical Properties of Tricalcium Silicate

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ABSTRACT

Using analytically pure calcium carbonate and silica as raw materials, high purity tricalcium silicate (C₃S) is prepared by solid-state reaction. The tricalcium silicate was sintered at a temperature of 1550°C with the heat preservation time of 6 h. The final sample needs three times of calcination circles. Ultrasonic cleaning machine was used to disperse the water and different amount of nano-SiO₂ (0, 1, 2, and 3 wt%) for 5 min, then mixed with C₃S, and formed into standard blocks. The setting time and compressive strength of each age (1, 3, 7, and 28 days) was measured to investigate the effect of nano-SiO₂ on the setting time and mechanical properties of C₃S. The results show that the content of free calcium oxide (*f*-CaO) in C₃S sample is lower than 0.8% tested by glycol ethanol method sensitive, C₃S crystal type is triclinic system T₁ by X ray diffraction (XRD). The nano-SiO₂ cut down the setting time of C₃S and the setting time of C₃S is decreasing with the increasing content of nano-SiO₂. The initial setting time of C₃S with different content of nano-SiO₂ (0, 1, 2, and 3 wt%) are 187, 129, 105, and 85 min, respectively, the final setting time are 219, 194, 158, and 130 min. The addition of nano-SiO₂ can significantly improve the early compressive strength of C₃S, but the effect of nano-SiO₂ on improving compressive strength of C₃S is gradually weakened with the hydration of C₃S.

1. INTRODUCTION

Nanomaterials have many special properties, such as volume effect, surface effect, size effect, and macroscopic quantum tunnel effect because of its small particle size and large specific surface area (Zhongtai, Yuanhua, Zilong, & Junying, 2000). In this new century, the technology of nanomaterials is developing at an astonishing speed and will be applied extensively with many materials. Although cement is a common building material, its main hydrate C–S–H gel is a natural nanomaterial (Björnström, Martinelli, Matic, Börjesson, & Panas, 2004; Qing, Zenan, Deyu, & Rongshen, 2007; Richardson, 2000), and it is significantly important to the study of nanomaterial-modified cement-based materials.

The influence of nano-SiO₂ addition on properties of hardened cement paste as compared with silica fume has been studied by Qing et al. (2007). Jo, Kim, Tae, and Park (2007) investigated the properties of cement mortars with nano-SiO₂. Besides, Li, Xiao, Yuan, and Ou (2004) have studied cement mortars with nano-SiO₂ or nano-Fe₂O₃ to explore their super mechanical and smart (temperature and strain sensing) potentials. Up to now, the main research focuses on the influence of nanomaterials

on cement; however, there are a few studies investigated the effect of nanomaterials on cement clinker single mine C₃S.

In view of the above-mentioned, C₃S mixed with nano-SiO₂ have been studied to explore changes in setting time and mechanical potentials. It was found that the nano-SiO₂ cut down the setting time of C₃S and the setting time of C₃S is decreased with the increasing content of nano-SiO₂; the compressive strengths of the C₃S with nano-SiO₂ were higher than those of a plain C₃S.

2. EXPERIMENTAL

2.1 Preparation and characterization of C₃S

The CaCO₃ and SiO₂ in a 3:1 molar ratio were mixed in a planetary ball mill for 20 min at a speed of 500 r/min and then were dried at 100°C for 4 h. The tricalcium silicate was sintered at a temperature of 1550°C with the heat preservation time of 6 h, quenched at room temperature, and milled to obtain a sample; glycol–ethanol method was used to determine *f*-CaO content and then calcined again until *f*-CaO content is less than 1%.

Figure 1 shows that pure tricalcium silicate obtained after three times calcination and its *f*-CaO content lower than 0.8%.

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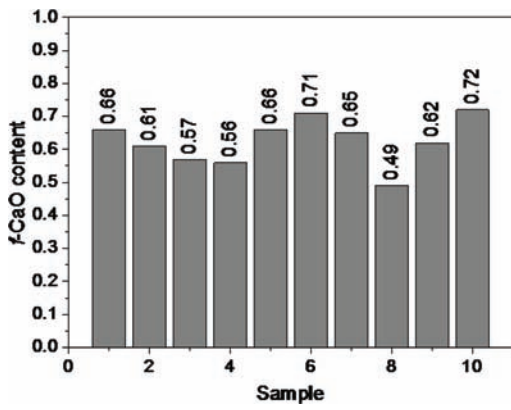


Figure 1. f-CaO content in C₃S after third calcination.

C₃S crystal forms include trigonal system: R; monoclinic: M₁, M₂, and M₃; and triclinic system: T₁, T₂, and T₃. In the quenching process of the C₃S, its crystal form changes from R-type to M-type and T-type, the symmetry is decreased at the same time. Symmetry reduction primarily presented the split of XRD diffraction peaks, which can be used to distinguish C₃S crystal forms. X ray powder diffraction to determine the different crystal forms of C₃S is mainly based on the differences of two fingerprint region diffraction peak shape of samples in 31–33° and 51–52.5° (Stephan & Wistuba, 2006; Wang, Li, He, Pan, & Shen, 2014).

Figure 2 is C₃S XRD pattern after three times calcination at 1550°C; Figure 3 is obtained by zooming in Figure 2 at 31–33° and 51–52.5°; it has four characteristic peaks at 31.5–33° and three characteristic peaks at 51–52.5°; this is consistent with the results reported by Wang et al. (2014); therefore, the crystal form of C₃S is triclinic system T₁-type.

Figure 4 is the particle diameter distribution of C₃S. The figure shows that the average particle size of the C₃S is X_{av} = 20.872 μm, and the median particle size is X₅₀ = 17.850 μm. What is more, the density of C₃S is 3.15 g/cm³.

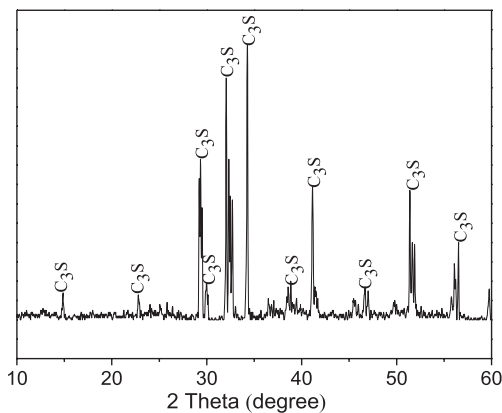


Figure 2. C₃S XRD pattern after three times calcination at 1550°C for 6 h.

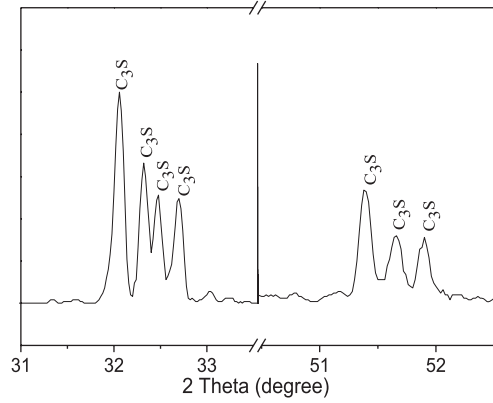


Figure 3. C₃S XRD pattern at 31–33° and 51–52.5°.

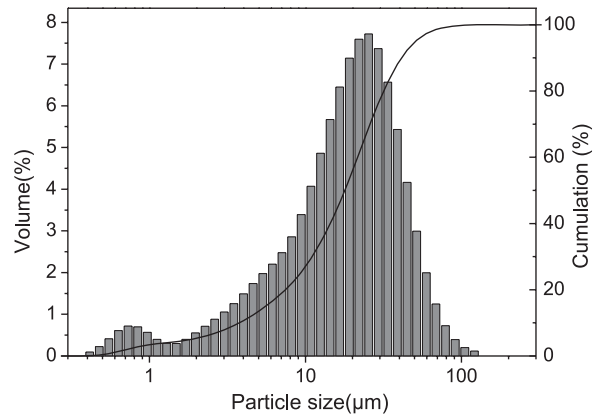


Figure 4. Particle diameter distribution of C₃S.

2.2 Sample preparation

Table 1 is the proportion of C₃S–SiO₂–H₂O system. Nano-SiO₂ mixed with water was dispersed in an ultrasonic cleaner (operating frequency is 40 kHz and ultrasonic electric power is 50 W) for 5 min, then mixed with C₃S, and formed into standard blocks. Compressive strength was measured when the samples cured to the prescribed age.

Table 1. Proportion of C₃S–SiO₂–H₂O system.

Sample	m(C ₃ S)/g	m(SiO ₂)/g	m(H ₂ O)/g	W/C
NS0	100.0	0	40	0.4
NS1	99.0	1.0	40	0.4
NS2	98.0	2.0	40	0.4
NS3	97.0	3.0	40	0.4

3. RESULTS AND DISCUSSION

3.1 Effect of nano-SiO₂ content on the setting time of cement clinker mineral C₃S

The effects of nano-SiO₂ content on setting time are presented in Figure 5. The figure shows that the initial setting time of C₃S with different content of nano-SiO₂

(0, 1, 2, and 3 wt%) are 187, 129, 105, and 85 min, respectively, the final setting time are 219, 194, 158, and 130 min. This indicates that adding nano-SiO₂ can shorten C₃S initial setting time and final setting time; moreover, the initial setting time and final setting time of C₃S are shortened with the increase of nano-SiO₂ content.

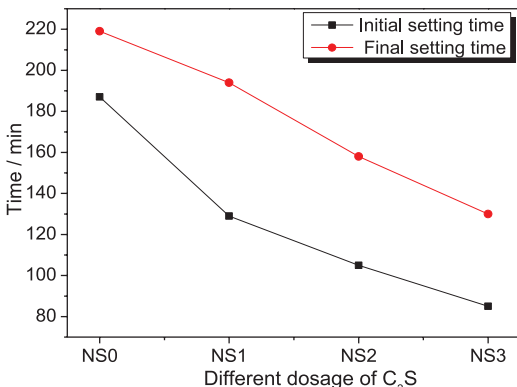


Figure 5. Effect of nano-SiO₂ content on the setting time of C₃S.

3.2 Effect of nano-SiO₂ content on the strength of cement clinker mineral C₃S

The effect of nano-SiO₂ content on the compressive strength of C₃S can be seen in Figure 6. Figure 7 presents the compressive strength growth rate of C₃S with different dosage of nano-SiO₂ compared with pure C₃S. The figures show that the compressive strength of C₃S with 3 wt% nano-SiO₂ increased by 288.97, 103.77, 34, and 14% in 1, 3, 7, and 28 days, respectively, compared with pure C₃S; at the same age, the compressive strength of C₃S increased with the augment of nano-SiO₂ content, but the growth rate of compressive strength decreased with the increase of age. It can be concluded that the addition of nano-SiO₂ can significantly improve the early compressive strength (1 and 3 days) of C₃S, but the effect of nano-SiO₂ on improving compressive strength of C₃S is gradually weakened with the hydration of C₃S.

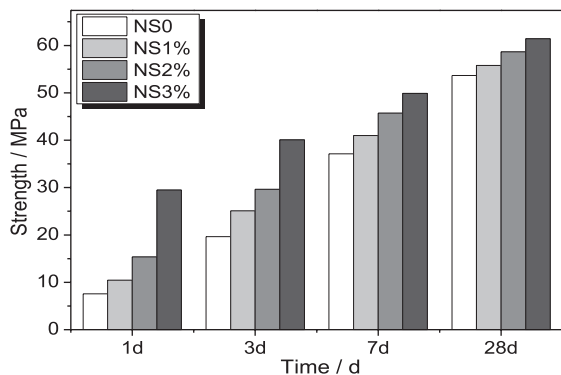


Figure 6. Effect of nano-SiO₂ content on the compressive strength of C₃S.

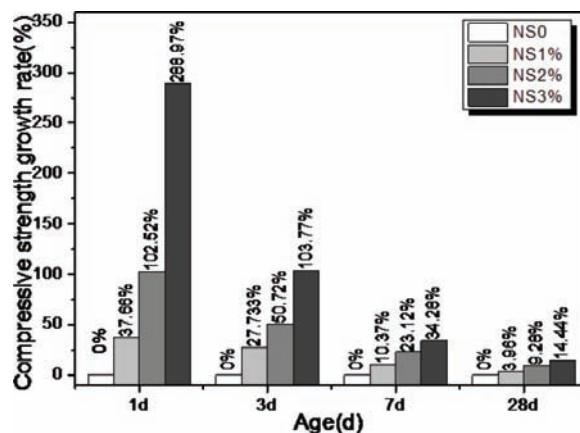


Figure 7. The compressive strength growth rate of C₃S with different dosage of nano-SiO₂ compared with pure C₃S.

4. CONCLUSION

Adding a little amount of nano-SiO₂ to tricalcium silicate (C₃S) can significantly accelerate the early hydration of C₃S. Nano-SiO₂ can shorten the initial setting time and the final setting time of C₃S, moreover, both setting times decrease with the increasing content of nano-SiO₂.

Nano-SiO₂ can significantly improve the early compressive strength (1 and 3 days) of C₃S, but the effect of nano-SiO₂ on improving compressive strength of C₃S is gradually weakened with the hydration of C₃S.

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