



Investigation on the formation of tobermorite in calcium silicate board and its influence factors under autoclaved curing



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HIGHLIGHTS

- The synthesis condition is not ambiguous for complicated cement system in calcium silicate board.
- This paper is focus on the formation of tobermorite in calcium silicate board.
- The research of influence factors for calcium silicate board has a guiding function in practical application.

ARTICLE INFO

Article history:

Received 20 December 2016
Received in revised form 7 March 2017
Accepted 16 March 2017
Available online 24 March 2017

Keywords:

Tobermorite
Autoclaved steaming time
Autoclaved steaming temperature
Ca/Si ratio

ABSTRACT

Compared with the traditional partition board, the superior properties of calcium silicate board are attributed to the generation of tobermorite, which can be influenced by many preparation conditions. The purpose of this study is to investigate the effect of Ca/Si ratio, and some others factors on the preparation and formation of tobermorite in calcium silicate board. The results reveal that the optimal cement content is 72%, at this time, Ca/Si ratio in calcium silicate board reaches 1.22. The tobermorite will gradually fade away when Ca/Si ratio continues to increase. XRD and mercury intrusion porosimetry after compression molding demonstrate that the molding pressure has mainly no effect on the hydration production and only has improvement on the structure of specimens. Due to the transformation of hydration production, autoclaved curing time and curing temperature should be controlled in specific range of 4–8 h and 190–195 °C, respectively. Additionally, the strength tests in different preparation factors present that the synthesis conditions confirmed in this paper are very promising to be applied in calcium silicate board.

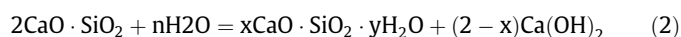
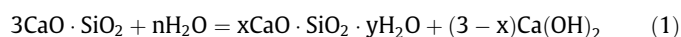
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1. Introduction

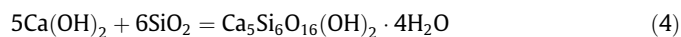
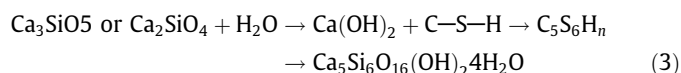
Recently, calcium silicate board has been applied extensively in china due to its better properties with sound insulation, lightweight, flame-retardant and moisture resistance [1–4]. Compared with the traditional partition board, the superior properties of calcium silicate board are attributed to the generation of tobermorite ($\text{Ca}_5\text{Si}_6\text{O}_{17}\cdot 5\text{H}_2\text{O}$), which is a type of crystalline calcium silicate hydrate synthesized by the reaction in the CaO-SiO₂-H₂O system [5]. It presents the characteristics of low density and better thermal insulation property, which can be widely used in the field of building materials or chemical industry [6–9].

Calcium silicate board is prepared by using OPC as calcium material, reactive quartz sand as siliceous material, wood fiber or others fiber as reinforcing materials [10]. The calcium silicate

board is commonly prepared by the method of compression molding after raw materials is stirred evenly, then the specimens begin to pretreatment for 4–12 h in room temperature. The chemical equation of this chemical reaction process has been proposed to be:



Then, specimens are cured for 4–24 h in vapor of 0.8–1.2 MPa at 180–220 °C for the formation of tobermorite according to the following reaction [11–13]:



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Under normal conditions of temperature and pressure, the hydration of C₃S paste yields to the poorly crystalline and non-stoichiometric C–S–H and portlandite [14,15]. At higher temperatures, it has been observed that C–S–H is no longer stable and replaced by more stable crystalline products [16]. According to Eq. (3), in the absence of reactive quartz sand, the hydration reaction of specimens can generate high crystallinity phases, C₂SH gel [Ca₂(HSiO₄)OH], hillebrandite [Ca₂SiO₃(OH)₂] or C₃SH [Ca₆Si₂O₇(OH)₆], considerably with increasing curing temperature from 85 to 200 °C [17], which are considered to be deleterious because they result in low mechanical properties. It is known that low crystallinity C–S–H at high temperatures leads to the formation of highly crystalline phases in a higher Ca/Si ratio [18]. The reduction of Ca/Si ratio can prevent the formation of high crystallinity phases by the addition of reactive quartz sand. Under the condition of low Ca/Si ratio, low crystallinity C–S–H gel begins to change when the temperature above 120 °C and C₅S₆H₅ (tobermorite) generates gradually with the extension of time. In addition, xonotlite will form due to the increasing temperature of 165 °C to 385 °C and curing time is long enough. These evolved crystal texture mainly controls the mechanical and thermal properties of the product, like high pressure resistance and low thermal conductivity [19]. According to Eq. (4), quartz sand reacts with Ca(OH)₂ originated the hydration of silicate mineral to generate tobermorite, which is conducive to prevent the formation of high crystallinity phases. There remain many different efforts made to synthesize tobermorite by the similar preparation process. Huang et al. used the blend of lime and active silica (kaolin) to synthesize the tobermorite [20] in vapor of 1.5 MPa and 200 °C for 5–20 h. Youssef et al. used the inorganic admixtures in the experiment to improve greatly the formation of tobermorite in the steel slag–quartz–lime system [21]. Ríos adopted the method of different cement-based materials to synthesize the tobermorite in the system CaO–SiO₂–Al₂O₃–H₂O under hydrothermal conditions at 175 °C for a monitoring time of 24 h [22].

Although the preparation technology of calcium silicate board has been matured, the synthesis condition is also ambiguous for complicated cement system. At present, researchers mostly pay attention to the study of pure tobermorite and almost no research involves the practical application. In this paper, the effects of Ca/Si ratio, compression molding, autoclaved steaming temperature and steaming time were investigated, which was focus on the formation of tobermorite in calcium silicate board.

2. Experimental

2.1. Preparation

Portland cement clinker was used as main raw material to investigate the formation mechanism of tobermorite, the quartz (SiO₂ content >95%) was used as high temperature stability agent, which can improve the mechanical strength through the reaction with cement clinker. The detailed chemical components of clinker and quartz were shown in Table 1. From Table 1, it can be seen that clinker could provide calcareous and siliceous material, while quartz could provide siliceous material. The theoretical Ca/Si ratio of tobermorite was 0.83, which was corresponding to the clinker content of 60%. In this experiment, some extension points were

designed. The relationship between Ca/Si ratio and clinker percent of total mass for clinker and quartz is presented in Table 2.

The calcium silicate board was prepared as follow. First, cement clinker was mixed with quartz sand and stirred evenly for a given water-cement ratio of 0.2. Then, the specimens were prepared by the method of compression molding under different pressure of 8, 16, 24, 32, 40 and 48 MPa. Thereafter, the specimens began to pretreatment for 12 h in room temperature. After that, it was cured at 175, 180, 185, 190, 195 °C for 2, 4, 6, 8, 10 h, respectively. When the reaction time arrived, the specimens were quickly cooled down to about 50 °C by using the fan. At last, the calcium silicate board was obtained after the specimens were dried at 40 °C for 12 h in dryer. The entire technological process diagram was shown in Fig. 1.

2.2. Testing methods

Flexural strength of calcium silicate board was tested by using a universal testing machine (CDT1305-2), whose capacities were 300 KN. The specimens were carried out in testing machine until it was damaged at a loading speed of 0.3 KN/s. SEM images were acquired to observe the morphology of tobermorite in calcium silicate board by Quanta FEG 250 field emission scanning electron microscope produced in FEI (American) whose resolution is smaller than 1 nm. X-ray diffractometry (Bruker D8 Advance, Germany) was used to qualitatively trace mineralogical phases through comparing the standard diffraction patterns. The differential scanning calorimetry (DSC) and thermo-gravimetric (TG) curves were collected by a TGA/1600HT simultaneous thermal analysis instrument produced by Merrier in Switzerland, at heating rate of 10 °C/min in flowing argon at a rate of 50 ml/min. The porosity of specimens were determined using mercury intrusion porosimetry (MIP) Micromeritics, AutoProe 9500 IV produced in America by Quantachrome.

3. Results and discussion

3.1. Clinker content (Ca/Si ratio)

In this paper, the formation of tobermorite in calcium silicate board originated from the hydration of cement clinker and the reaction of Ca(OH)₂ and reactive quartz sand. Thus the presence of clinker, or the Ca/Si ratio would have significant influence on the mineralogical phases of calcium silicate board. XRD patterns of all the specimens with different clinker content were shown in Fig. 2, in which mineralogical phases were distinguished by the comparison with ICDD PDF database. The molding pressure, autoclaved curing time and temperature were set as 40 MPa, 6 h and 195 °C, respectively. It presented that the variation of mineralogical phases for the specimen was closely related with the content of clinker.

In detail, the diffraction peaks of tobermorite in XRD pattern strengthened gradually as the Ca/Si ratio increased in the range of 50% to 70%. It showed that the increasing of calcareous material was conducive to the forming of tobermorite. Obviously, the diffraction peaks of SiO₂ were predominant, which indicated that the content of quartz sand was rich at this time. Sufficient SiO₂ implied the lack of clinker. Dramatically, the diffraction peaks of

Table 1
Chemical component of raw materials.

Raw material	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Others
Cement clinker	67.61%	20.71%	4.67%	3.19%	1.69%	2.13%
Quartz	1.02%	95.67%	2.71%	–	0.30%	0.3%

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