



Effect of 5-phase seed crystal on the mechanical properties and microstructure of magnesium oxychloride cement



Xu Zhang, Shaojin Ge, Hongning Wang, Ruoyu Chen *

School of Petrochemical Engineering, Changzhou University, Changzhou, Jiangsu, PR China

HIGHLIGHTS

- Synthesized 5-phase crystals were added as seed crystals to the two kinds of magnesium oxychloride cement (MOC) systems.
- Seed crystals can promote the formation and growth of the 5-phase in MOC, thus improving compressive strength.
- Seed crystals can make the microstructure of the MOC more compact, thus improving water resistance.

ARTICLE INFO

Article history:

Received 11 January 2017

Received in revised form 24 May 2017

Accepted 26 May 2017

Keywords:

Magnesium oxychloride cement (MOC)

Seed crystal

Reaction process

Compressive strength

Water resistance

Microstructure

ABSTRACT

In this research, the effects of synthesized 5-phase seed crystals $[5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}]$ on the compressive strength, reaction process, and microstructure of the two kinds of MOC system were investigated. The results show that the addition of 5-phase seed crystals can significantly increase the formation rate of 5-phase in the MOC, which can improve the compressive strength of MOC, especially in its early stage; The compact microstructure not only improves compressive strength but also increases water resistance. Moreover, the effect of seeds in improving compressive strength and water resistance is more noticeable in the MOC system with phosphoric acid.

© 2017 Elsevier Ltd. All rights reserved.

1. Introduction

Magnesium oxychloride cement (MOC) was discovered by Sorel in 1867. As an air-dried gel material, MOC is formed by mixing magnesium oxide powder with magnesium chloride solution [1]. The excellent performance of MOC, including effective resistance to corrosion and abrasion, lower thermal conductivity, and better durability, makes it an attractive inorganic cementitious material [2]. The hydration of MOC takes place in a through-solution reaction [3], and the main phases in MOC are $3\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (3-phase) and $5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (5-phase) at room temperature [4,5]. The maximum formation of 5-phase crystals is desirable on the basis of findings that this phase provides the main mechanical strength [6–8]. During the reaction process of the MOC, the formation rate, relative content, and microstructure feature of the 5-phase will directly affect the mechanical strength of MOC at different curing ages.

Despite numerous advantages, the application of MOC is limited in engineering applications because it reduces in strength rapidly with prolonged exposure to water [9]. The water-resistance of MOC could be improved by modifying the composition and by doping various additives. It is generally accepted that phosphoric acid and soluble phosphate can greatly improve the water-resistance of MOC [10–12]. Deng [13] studied the effect mechanism for soluble phosphates on the water resistance of MOC and found that phosphate radical anions can affect the formation reactions of 5-phase in MOC pastes and that the water-resistance of MOC could be improved significantly; however, the effect of soluble phosphates affects the retardation of setting MOC pastes. Therefore, the earlier mechanical strength of the hardened MOC pastes with a phosphate additive is decreased significantly. Tan et al. [14] studied the effect of phosphoric acid on the properties of magnesium oxychloride cement. Results showed that 5-phase formed much more slowly in MOC pastes with phosphoric acid additive. Thus, the setting speed and the mechanical strength of MOC were reduced greatly. In addition, the microstructure is one factor that affects the mechanical properties of MOC [9]. Chau et al. [15]

* Corresponding author.

E-mail address: cxdcry@163.com (R. Chen).

observed that a large number of interlaced needle-shaped 5-phase crystals appear in the MOC with high mechanical strength and compactness. The compact microstructure not only contributes to the improvement of compressive strength but also slows erosion by external water of the internal structure.

In general, the growth of the 5-phase crystals is critical in the curing process of MOC pastes, and it will directly affect the mechanical strength and microstructure of the cement. It is widely accepted that the growth of a particular crystal can be facilitated by the addition of seed crystal in the crystallization process of inorganic salts [16]. In this research, pure 5-phase crystals $[5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}]$ are synthesized first by the reaction of active magnesium oxide and magnesium chloride solution. After which, synthesized 5-phase crystals are added, as seed crystals, to both the ordinary MOC system and the MOC system with phosphoric acid as a water-resistance agent. The main objective of this research was to investigate the effect of seed crystals on the formation rate of the 5-phase, the mechanical strength, and the water-resistance of MOC. In addition, the effect of 5-phase seed crystals on the internal microstructure of magnesium oxychloride cement was investigated in detail.

2. Experiments

2.1. Raw materials

The three kinds of raw materials for making MOC in the laboratory are magnesia powder, magnesium chloride, and water. The three kinds of raw materials for making seed crystals are magnesium carbonate basic pentahydrate, magnesium chloride, and water. In this study, the magnesia powder was calcined magnesite powder with active magnesium oxide (α -MgO), 63% by weight, with an average particle size of 75 μm (Haicheng, Liaoning Province, China). The content of α -MgO was determined by hydration method, according to the ferrous metallurgy industry standard in the People's Republic of China (YB/T 4019-2006). The chemical compositions of the magnesia powders are given in Table 1. Both the magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ purity = 98 wt %) and the magnesium carbonate basic pentahydrate (MgO purity = 40.0–44.5 wt%) are produced by Sinopharm Chemical Reagent Co. Ltd., Shanghai, China.

2.2. Preparation of 5-phase seed crystals

The high-activity light-burned MgO powder was produced by calcination of magnesium carbonate basic pentahydrate in a furnace at 600 $^{\circ}\text{C}$ for 2 h. Before mixing with the high-activity light-burned MgO powder to produce 5-phase seed crystals, the magnesium chloride hexahydrate was first dissolved in distilled water to form the MgCl_2 solution with the desired concentration. A mixture of high-activity light-burned MgO powder and magnesium chloride solution, with a molar ratio of $\text{MgO}:\text{MgCl}_2:\text{H}_2\text{O} = 0.32:1:13$, was placed in a closed reactor at 80 $^{\circ}\text{C}$ for 3 d. Then, the reactor was taken out, and the mixture was cooled to room temperature. Next, the product was washed three times with absolute ethanol and dried at 50 $^{\circ}\text{C}$ for 48 h to obtain pure 5-phase seed crystal (Fig. 1).

2.3. Preparation MOC specimen

In this study, the initial ratio of raw materials for making magnesium oxychloride cement was determined as $\text{MgO}:\text{MgCl}_2:\text{H}_2\text{O} = 6.5:1:13$ (molar ratio). MgO was calculated as α -MgO. The amount of 5-phase seed crystal or phosphoric acid in the specimens was 1% by weight of magnesia powders. To prepare the specimens of the neat MOC paste, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ was first dissolved in distilled water to form the MgCl_2 solution with the desired molar ratio. Then, the weighted magnesia powder was mixed with the MgCl_2 solution mechanically to form homogenous MOC paste. Before preparation of the MOC paste containing phosphoric acid or seed crystal, the phosphoric acid was mixed with MgCl_2 solution, and the seed crystal was mixed with magnesia powder, respectively. The MOC paste was cast in $40 \times 40 \times 160 \text{ mm}^3$ steel molds and was vibration compacted and cured for 1 d at room temperature and then unmolded and air-cured for 1–28 d before being used in the various experiments. To facilitate the description, the MOC samples are numbered and given in Table 2. Where Samples A1 and A2 are MOC system without adding phosphoric acid as a water-resistance agent, Samples A3 and A4 are MOC system with phosphoric acid as a water-resistance agent.

2.4. Specimen analysis

2.4.1. Compressive strength and strength-retention coefficient

Compression strength was tested by using a fully automatic cement-strength testing machine (AEC-201), produced by Ai Li Kang Equipment Co. Ltd. (Wuxi, China). At the curing ages of 3, 7, 14, 21, and 28 d, the compressive strength was measured at a loading rate of 2.4 kN/s. Six samples were tested for each composition and the average results were reported. To determine the water-resistance performance of the fabricated MOC specimens, the samples after curing at room temperature for 28 d were immersed in tap water for 28 d, and then the specimens were retrieved and tested for compressive strength. In this work, the strength-retention coefficient is calculated by the following formula:

$$\omega = \frac{P}{Q},$$

where ω is strength retention coefficient; P is the compressive strength of the specimen after immersion in water for 28 d; and Q is the compressive strength of the specimen cured for 28 d before immersion in water.

2.4.2. Magnesium chloride reaction degree analysis

Magnesium chloride reaction degree analysis was done at the curing ages of 1, 3, 7, 14, 21 and 28 d. Powder samples were prepared by crushing the specimens and passing the powder through a sieve with a screen aperture of 75 μm at different curing ages. Powder sample (1 g) was weighed accurately and added to the beaker with 20.00 ml anhydrous ethanol. The mixture was stirred at room temperature for 5 h at the same rotational speed, and then the mixture was filtered and washed with absolute ethanol. All the filtrates were titrated with mercurimetric method [17]. Sym-diphenylcarbazone and bromophenol blue were used as the mixed indicator. The reaction degree of the MgCl_2 was calculated from the following formula:

Table 1
The chemical compositions of the magnesia powders.

Components	MgO	SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	Loss on ignition
Mass fraction: %	84.27	6.25	1.68	0.28	0.57	6.95

Download English Version:

<https://daneshyari.com/en/article/4918295>

Download Persian Version:

<https://daneshyari.com/article/4918295>

[Daneshyari.com](https://daneshyari.com)