



Technical note

Preparation and characterization of one-part magnesium oxychloride cement

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HIGHLIGHTS

- One-part MOC was prepared by the control in hydration process.
- The performance of MOC-powder depends on MgO/MgCl₂ molar ratio and water content.
- The hydration reaction had continued after addition of water to one-part MOC.
- The strength gap between one- and two-part MOC increased with W/MgO ratio.

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ABSTRACT

This work aims at enhancing the commercial viability of magnesium oxychloride cement (MOC) by preparing one-part MOC (just added water). It was synthesized by mixing of magnesium chloride (MgCl₂) with magnesium oxide (MgO) at water to MgO (W/MgO) ratios, making MOC-paste in non-workable form. The MOC-slurry was immediately dried in an oven at 60 °C for 24 h, yielding solidified material which pulverized to produce one-part-MOC powder with a fixed particle size. Two major factors, which affect the performance of one-part MOC, were examined: the first is the increase of MgO/MgCl₂ molar ratio, while the W/MgO ratio is kept maintaining constant. The second includes the increase of W/MgO ratio at constant MgO/MgCl₂ molar ratio. One-part-MOC powder was mixed with water at W/MOC powder ratio of 0.25, and then cured. A traditional two-part MOC containing MgO (solid part) and MgCl₂ solution (liquid part) was used for comparison. The results revealed that the W/MgO ratio and MgO/MgCl₂ molar ratio strongly affect the performance of one-part MOC. The compressive strength development of the hardened cement pastes proved the continuation of hydrate phases formation after mixing one-part-MOC with water. At 14-day of curing, the compressive strength value of an optimal one-part MOC was found to be lower than that of two-part-MOC, containing the same MgO/MgCl₂ molar ratio, by 14%.

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1. Introduction

In 1867, Sorel cement or magnesium oxychloride cement (MOC) was discovered by Frenchman Stanislas Sorel [1]. MOC was prepared by mixing of magnesium oxide (MgO) with magnesium chloride (MgCl₂) [2–5]. The MgO/MgCl₂ and H₂O/MgCl₂ molar ratios are the main parameters, which potentially affect the mechanical properties of MOC [6]. The main hydration products of MOC, which responsible for its hardening and strength, are 5 Mg(OH)₂·MgCl₂·8H₂O (phase 5), 3 Mg(OH)₂·MgCl₂·8H₂O (phase

3) and 2 Mg(OH)₂·MgCl₂·8H₂O (phase 2). The composition of hydrate phases mainly depends on the MgO/MgCl₂ molar ratio [6]. The mechanism of hydrate phases formation includes three steps: the first is the neutralizing process in which MgO powder is neutralized by free H⁺ produced from the dissociation of MgCl₂ crystals in water. The second includes the formation of binuclear, tri-nuclear and poly-nuclear complexes {Mg_x(OH)_y(H₂O)_z}^{2x-y} by the hydrolyzing-bridging reaction. In final step, the condensation of these phases and the adsorption of Cl⁻ (to equalize the positive charge on complexes) have been occurred, leading to form amorphous gel which crystallized in a few days or weeks [7].

MOC characterizes by low, thermal conductivity, high early strength, high firing and a good abrasion resistivity [8,9]. Although advantageous properties, the MOC showed poor water resistivity

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[8], limiting its application for practical engineering projects. Various additives were added to improve the resistivity of MOC to strength regression under water curing [10–16].

Based on the mechanism of MOC formation [7], an innovative experimental method was applied to facilitate the utilization of MOC in different engineering projects by synthesizing one-part-MOC. The preparation process was carried out by the control in the hydrolyzing-bridging and condensation processes responsible for the formation and polymerization of hydrated phases in MOC. This was performed by the control in water added to MgO-MgCl₂ system. Where, water plays an important role on the hydrolyzing-bridging and condensation processes [6]. The appropriate water content enhances the rate of phases formation and polymerization. So, the decrease in water content causes the retardation of nuclear phases condensation, producing material predominantly contains unpolymerized species, which have high capability to polymerize if additional amount of water was added.

Hence, the MgO was mixed with MgCl₂ solution at water to MgO ratio which makes the paste in non-workable form. The hydrolyzing-bridging and condensation processes initiate after addition of MgCl₂ solution to MgO. In order to stop the hydration reaction, the removal of water in the homogeneous slurry has been carried out at 60 °C for 24 h. The solidified material was pulverized to produce one-part MOC-powder with a fixed particle size. The probability of the continuation of hydrate phases formation was examined by the addition of water to one-part-MOC powder.

2. Experimental

The two starting materials are magnesium oxide (MgO) and magnesium chloride hexahydrate (MgCl₂·6H₂O). MgO used in this investigation is calcined magnesite with purity of 97% and average particle size of 25 μm from El-Gomhoria Chemical Company, Egypt. The MgCl₂·6H₂O with purity of 98% was purchased from LOBA Company, India. The chemical compositions of the raw materials analyzed by X-ray fluorescence spectrometer (Xios, PW1400) are listed in Table 1.

For one-part MOC powder preparation, two categories were designed as shown in Table 2. The MOC-1, MOC-2, and MOC-3 mixes were designed by varying of MgO/MgCl₂ molar ratios (19, 14, and 9, respectively) at constant water to MgO (W/MgO) of 0.10. The MOC-2, MOC-4, and MOC-5 mixes were formulated by varying W/MgO ratios (0.10, 0.13, and 0.16, respectively) at constant MgO/MgCl₂ molar ratio of 14. After automatic mixing of MgO powder with MgCl₂ solution for 5 min, the homogeneous slurry was immediately dried in an oven at 60 °C for 24 h. The solidified samples were pulverized to pass from 25 μm sieve, yielding one-part MOC powder.

In order to prepare MOC-paste, the water (at W/MOC powder ratio of 0.25) was added to one-part-MOC powder, followed by

an automatic mixing for 3 min. Two-part-MOC (MOC-6), as counterpart for one-part MOC-2, MOC-4 and MOC-5, was prepared by mixing of MgO with MgCl₂ dissolved in water equivalent to W/MgO ratio of 0.25. The workable pastes were poured into 50*50*50 mm³ stainless steel mold and vibrated in an automatic vibration Table for 1 min., and then the surface was smoothed by a thin edged trowel followed by curing in air at 23 ± 2 °C and 65 ± 5% relative humidity. After 24 h, the hardened-MOC specimens were demolded and cured at the same condition until the time of compressive strength reaches such as 1, 7 and 14 days.

The compressive strength test was carried out according to ASTM C109M-16 specification [17] using five tons German-Bruf-Pressing Machine with a loading rate of 100 kg/min. The compressive strength applied on three cubic samples of each mix at 1, 7 and 14-day of air curing. At 14-day of compressive strength, the broken cubes were immersed in solution composed of acetone: methanol at ratio of 1:1, to remove free water followed dried at 80 °C for 24 h. The part of dried samples was kept for microstructure analysis and the other crushed to a pass from 45 μm for X-ray diffraction (XRD) analysis.

XRD-analysis was recorded on a Philips PW 1050/70 Diffractometer using a Cu-Kα source with a post sample Kα filter. XRD patterns were collected in the range of 5–50 2θ° with a scanning speed of 1sec./step and resolution of 0.05°/step. Silica was used as an internal standard. Data were identified according to the XRD software. Scanning electron microscopy (SEM) were done, using Inspect S (FEI Company, Holland), equipped with an energy dispersive X-ray analyzer (EDXA). The sample was covered by gold in order to enhance its conductivity and the accelerated voltage was set at 10kv.

3. Results and discussion

3.1. Compressive strength

The compressive strength values of the hardened one-part MOC-1, MOC-2 and MOC-3 up to 14-days of air curing are shown in Fig. 1. The MgO/MgCl₂ molar ratio strongly affects the compressive strength of hardened-MOC-pastes. The MOC-2 showed the highest compressive strength at all ages of curing. The higher and lower MgO/MgCl₂ molar ratios (19 and 9, respectively) have a negative impact on the compressive strength development after the addition of water to one-part MOC powder. At MgO/MgCl₂ molar ratio of 19, the MgCl₂ is no sufficient for the formation of hydrated phases, after the addition of water to one-part MOC-1. Meanwhile, the high MgCl₂ concentration (at MgO/MgCl₂ molar ratio of 9) enhances the formation of polymerized poly-nuclear MOC-phases, decreasing the probability of continuation of hydrate phases formation. The MgO/MgCl₂ molar ratio of 14 is the appropriate value to enhance the performance of one-part MOC powder.

Table 1
Chemical compositions of the starting materials used.

MgO	Components	MgO	CaO	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	SO ₃
	Mass fractions	97.18	1.72	0.48	0.25	0.03	0.21
MgCl ₂ ·6H ₂ O	Components	MgCl ₂	H ₂ O	CaCl ₂	KCl	NaCl	–
	Mass fractions	45.7	52.0	1.6	0.2	0.5	–

Table 2
Mix proportions of one-part MOC powder.

Mixes notation	MOC-1	MOC-2	MOC-3	MOC-4	MOC-5
MgO/MgCl ₂ molar ratio	19	14	9	14	14
W/MgO ratio	0.10	0.10	0.10	0.13	0.16

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