

Reactivity and Function of Magnesium Oxide in Sorel Cement

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Abstract: The reactivity, or rate of consumption, of magnesium oxide (MgO) powder used in forming magnesium oxychloride (MOC) cement is largely affected by its thermal history and particle size. That, in turn, influences both reaction rate and properties of the reaction products of MOC cement. It is believed that the traditional measurements of properties of MgO powder itself are not able to reflect the actual consumption rate of MgO during chemical reaction, which is the key information needed for the appropriate proportions of starting materials to ensure the formation of Phase 5 crystals for better strength and physical properties. The experimental results show that X-ray diffractograms and phase diagram of a ternary MOC system can be utilized to evaluate the reactivity of MgO, and to optimize the formulation of MOC cement as well. Consequently, the apparent molar ratios of MgO over magnesium chloride (MgCl_2) are adopted and recommended. The equivalent weight ratios between MgO and MgCl_2 are also given for the convenience of normal applications.

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Introduction

Magnesium oxychloride (MOC) cement, also known as Sorel cement (Sorel 1867), was developed shortly after the invention of Portland cement. As one of the main representatives of chemically bonded cement, MOC cement has many properties superior to the ordinary Portland cement (Bensted and Barnes 2002). It does not need wet curing; has high fire resistance, low thermal conductivity, and good resistance to abrasion as well as chemicals. The rapid setting and hardening properties as well as the excellent bonding ability to large amounts of different fillers such as gravel, sand, marble flour, asbestos, wood, and expanded clays, make MOC an attractive inorganic cement. Moreover, the lower alkalinity of MOC makes it suitable for use of glass fiber without an aging problem. The major commercial applications of MOC cement are industrial flooring, fire protection, and grinding wheels. By virtue of its acoustic and elastic properties, and attractive marble-like appearance, MOC cement is also used for rendering wall insulation panels, for interior plasters and exterior stuccos, and for decorative panels (de Henau and Dupas 1976).

Sorel cement is formed by mixing powdered magnesium oxide (MgO) with a concentrated solution of magnesium chloride (MgCl_2). The setting and hardening of magnesium oxychloride cement take place in a thorough solution reaction (Bilinski et al.

1984; Menetrier-Sorrentino et al. 1986; Deng and Zhang 1999). The four main crystalline phases in the ternary MOC system are $2\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 4\text{H}_2\text{O}$ (Phase 2), $3\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (Phase 3), $5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (Phase 5), and $9\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 5\text{H}_2\text{O}$ (Phase 9) (Demediuk et al. 1955). Of these, Phases 3 and 5 may exist at ambient temperature, whereas Phases 2 and 9 are stable only at temperatures above 100°C (Cole and Demediuk 1955). The other possible reaction product is the binary phase of magnesium hydroxide or brucite, $\text{Mg}(\text{OH})_2$. The well-crystallized needle-shaped Phase 5 of MOC cement has been described as scroll-tubular whiskers (Tooper and Cartz 1966). The mechanical interlocking and dense microstructure resulting from the intergrowth of these whiskers is a major source for strength development of MOC cement (Matkovic and Young 1973). Therefore, the mechanical properties of MOC depend largely on the phase(s) formation and subsequently on the appropriate proportions of the starting materials.

The reactivity, or the consumption rate, of the magnesium oxide powder used in forming MOC cement is largely affected by its thermal history (calcination temperature and duration) (Harper 1967; Matkovic et al. 1977) and particle size (Eubank 1951). This in turn influences both reaction rate and physical properties of the reaction products of MOC cement. The effects of microstructure of MgO powder, including crystallite size and microporosity, on the hydration of magnesia were also studied (Coleman and Ford 1964). It is believed that the traditional measurement of the properties of MgO powder itself is not able to reflect the actual rate of consumption of MgO, which is the key information needed for the correct proportions of starting materials to ensure the formation of Phase 5 crystals for a better strength development.

It is found that X-ray diffraction measurement and phase diagram analysis of the system $\text{MgO}-\text{MgCl}_2-\text{H}_2\text{O}$ (Sorrell and Armstrong 1976; Urwongse and Sorrell 1980) can be utilized to evaluate the reactivity of the starting materials. On one hand, for a specific MOC mixture design, the phase assemblage of the mixture identified by X-ray diffraction analysis should match the phase compatibility of the composition point in the phase diagram. On the other hand, the calculation of the composition of the

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Table 1. Chemical Compositions of Magnesium Oxide

Component	Mass fraction (%)
MgO	96.58
SiO ₂	0.766
SO ₄	0.162
CaO	1.408
MnO	0.137
Fe ₂ O ₃	0.774
Others	0.173

mixture starts from the weight ratios among the starting materials, and can be altered by adjusting the reactivity of the materials. By doing so, once the composition point of the mixture fits in the appropriate area inside the correct compatibility triangle in the phase diagram, the reactivity or the actual consumption rate of the starting materials can be, to a certain extent, approximated.

Experimental Program

Magnesium oxide, magnesium chloride, and water were the three starting materials of MOC cement. The magnesium oxide used in this study was calcined magnesite powder with a purity of 96% from Jinan, Shandong Province, China. The magnesium chloride employed was hygroscopic hexahydrate (MgCl₂·6H₂O) crystal with a purity of 98% from Kin Shing Ind. Material Co. Ltd., Hong Kong. The chemical compositions of the MgO powder and the hexahydrate analyzed by X-ray fluorescence spectrometer (JEOL JSX-3201Z) are listed in Tables 1 and 2, respectively. The magnesium chloride hexahydrate was first dissolved into water before mixing with magnesium oxide powder to form MOC cement paste.

For the ternary system MgO–MgCl₂–H₂O at ambient temperature, normally the two molar ratios of MgO/MgCl₂(M) and H₂O/MgCl₂(H) are used to describe the system. The specimen matrix of MOC cement with wide ranges and combinations of the two molar ratios is shown in Table 3. The molar ratio of MgO/MgCl₂ starts from 7, which is higher than the theoretical value of 5 for Phase 5, as excess MgO is required to favor the Phase 5 formation. The highest molar ratio of MgO/MgCl₂ reaches 17 as a sufficient amount of the reaction product of magnesium hydroxide is desired for different assemblages of MOC phases. The choice of the molar ratio of H₂O/MgCl₂, however, is limited by the concentration of MgCl₂ solution, fluidity, and cohesiveness of the mixture. Those are also the problems to be faced in the normal application of MOC cement, may be even with more strict requirements to meet.

To facilitate discussions, the following abbreviation is used to specify the different combinations of molar ratios of each mix-

Table 2. Chemical Comparisons of Magnesium Hexahydrate

Component	Mass fraction (%)
MgCl ₂	45.8
H ₂ O	52.0
CaCl ₂	1.3
NaCl	0.6
KCl	0.3

Table 3. Specimen Matrix of MOC Cements with Different Molar Ratios

H ₂ O/MgCl ₂ (H)	MgO/MgCl ₂ (M)					
	07	09	11	13	15	17
10	+	+				
12	+	+	+	+		
14	+	+	+	+	+	
16	+	+	+	+	+	+
18			+	+	+	+
20			+	+	+	+

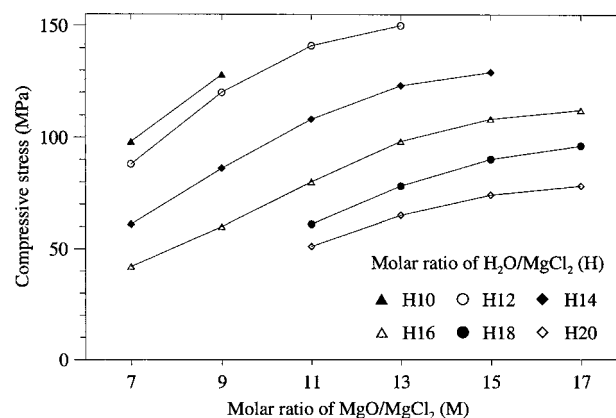
Note: “+”=mixture with a combination of the two molar ratios has been evaluated.

ture. For a mixture with a molar ratio of MgO/MgCl₂ of *xx* and a molar ratio of H₂O/MgCl₂ of *yy*, it is specified as M_{xx}/H_{yy}. For example, the mixture at the top-left corner in Table 3 is specified as M07/H10, and the mixture at the bottom-right corner in Table 3 is specified as M17/H20.

For each mixture assigned in Table 3, cubic specimens with a size of 40 mm × 40 mm × 40 mm were cast in steel molds with vibration compaction. The compressive strength development of the mixtures were recorded (ELE compression machine) at 3, 7, and 14 days after air curing at a temperature of 23 ± 1 °C and under a relative humidity of 60 ± 5% in the curing room. A powder sample was collected from the specimens after 14-day air curing for X-ray diffractogram measurement (Philips PW 1830) to identify the phase assemblage of the corresponding mixtures. A phase diagram of the ternary system MgO–MgCl₂–H₂O at room temperature (22–28 °C) was plotted to locate the composition points of the mixtures. The X-ray diffractograms were then compared with the composition points in the phase diagram to check the compatibility of phase assemblage of the corresponding mixtures.

Results and Discussions

The compressive strengths of the different mixtures after air curing of 7 days are plotted and compared in Fig. 1. It shows that with a fixed molar ratio of MgO/MgCl₂, the strength of the mixtures increases with the decrease of the molar ratio of H₂O/MgCl₂. Besides, with a fixed molar ratio of H₂O/MgCl₂, compressive strengths of the mixtures change with the change of

**Fig. 1.** Compressive strength of the different MOC pastes at the age of 7 days

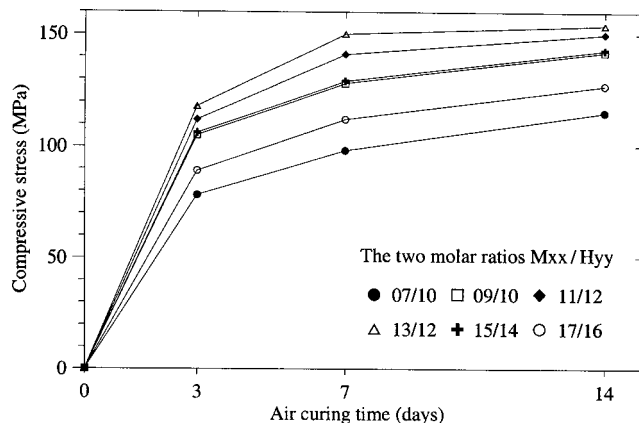


Fig. 2. Growth of compressive strength of the representative MOC pastes

molar ratios of MgO/MgCl_2 . The higher the MgO/MgCl_2 molar ratio, the higher the strength. The highest strength is 150 MPa for the Mixture M13/H12, while the lowest strength is 42 MPa for the Mixture M07/H16. Such a big difference between the maximum and minimum strength among the mixtures may be due to the different microstructures and intrinsic properties of the mixtures with different assemblages of crystalline phases.

The mixtures having the highest 7-day compressive strength corresponding to each molar ratio of MgO/MgCl_2 are M07/H10, M09/H10, M11/H12, M13/H12, M15/H14, and M17/H16, respectively. The strength developments of these mixtures in the first 2 weeks after casting are shown in Fig. 2. As shown in Fig. 2, M13/H12 has the maximum strengths at all ages, while M07/H10 has the minimum strengths. It also shows that Mixture M13/H12 has the fastest strength development compared with the other mixtures. It implies that Mixture M13/H12 may have the best combination of the molar ratios of MgO/MgCl_2 and $\text{H}_2\text{O}/\text{MgCl}_2$ for obtaining Phase 5 crystals and physical strength.

Phase Assemblages

The composition points of some selected mixtures are labeled in the phase diagram of the ternary system $\text{MgO}-\text{MgCl}_2-\text{H}_2\text{O}$ as shown in Fig. 3. It shows that M07/H10 (Point a) lies in the compatibility triangle MgO -Phase 3-hexahydrate, while the mixtures with a molar ratio of $\text{H}_2\text{O}/\text{MgCl}_2$ of 12 (Points b, e, g, and h) lying in the compatibility triangle MgO -Phase 5-Phase 3. According to the phase, compatibility and mass balance in Phase Diagram M07/H10 should have a phase assemblage of Phase 3, MgO , and hexahydrate, while M07/H12, M09/H12, M11/H12, and M13/H12 (Points b, e, g, and h) should have a phase assemblage of Phase 5, Phase 3, and MgO . From Fig. 3, it can also be seen that M13/H14 and M13/H16 (Points i & j) lie in the compatibility triangle $\text{MgO}-\text{Mg}(\text{OH})_2$ -Phase 5, and should have a phase assemblage of Phase 5, $\text{Mg}(\text{OH})_2$, and MgO . The corresponding X-ray diffractograms of the mixtures labeled in Fig. 3 are compared in Fig. 4 (for Points a, b, and e), and Fig. 5 (for the Points g, h, i, and j). Fig. 4 shows that Mixture M07/H10 contains largely Phase 3 with excess MgO , while Mixtures M07/H12 and M09/H12 have both Phases 3 and 5 coexistence as well as MgO residuals.

Moreover, Fig. 5 shows that Mixtures M11/H12, M13/H12, M13/H14, and M13/H16 contain mainly Phase 5 with excess MgO . By comparing the strength measurements and phase iden-

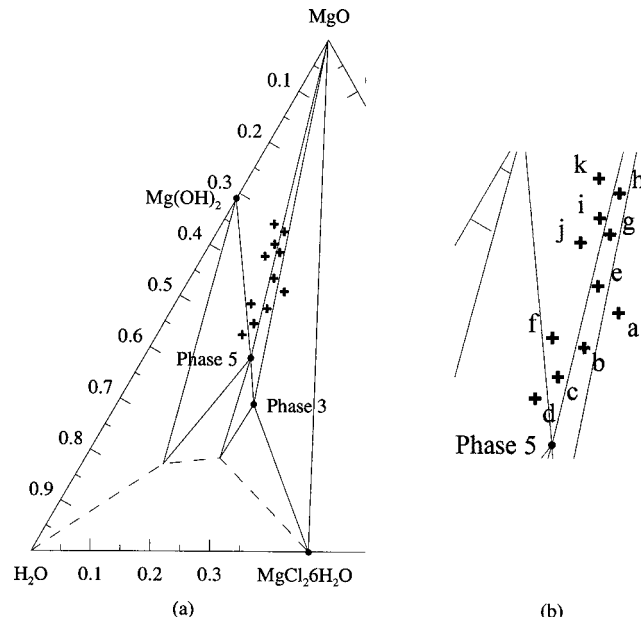


Fig. 3. Phase diagram of the ternary system $\text{MgO}-\text{MgCl}_2-\text{H}_2\text{O}$: (a) isothermal section at room temperature; and (b) magnified portion of (a) with some selected mixtures labeled: a.M07/H10, b.M07/H12, c.M07/H14, d.M07/H16, e.M09/H12, f.M09/H16, g.M11/H12, h.M13/H12, i.M13/H14, j.M13/H16, and k.M17/H16

tification of the mixtures, it reveals that M13/H12 would be the best combination of the molar ratios of MgO/MgCl_2 and $\text{H}_2\text{O}/\text{MgCl}_2$ for obtaining Phase 5 crystals as well as good mechanical properties for MOC cement. The question remaining is why such a high apparent molar ratio of MgO/MgCl_2 (13 for the mixture of M13/H12), which is more than two times the theoretical one of 5, is required for forming MOC phases. The overestimated reactivity, i.e., a 100% of consumption rate, of MgO powder in the calculation of molar ratios would be one of the most possible reasons.

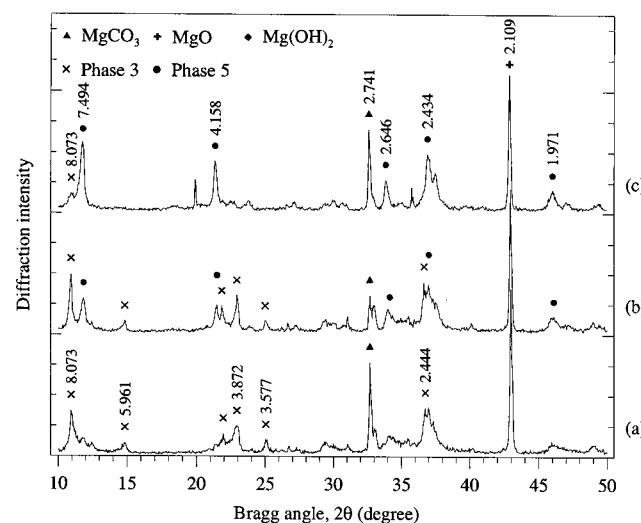


Fig. 4. X-ray diffractograms of the mixtures: (a) M07/H10 (Point a in Fig. 3); (b) M07/H12 (Point b in Fig. 3); and (c) M09/H12 (Point e in Fig. 3)

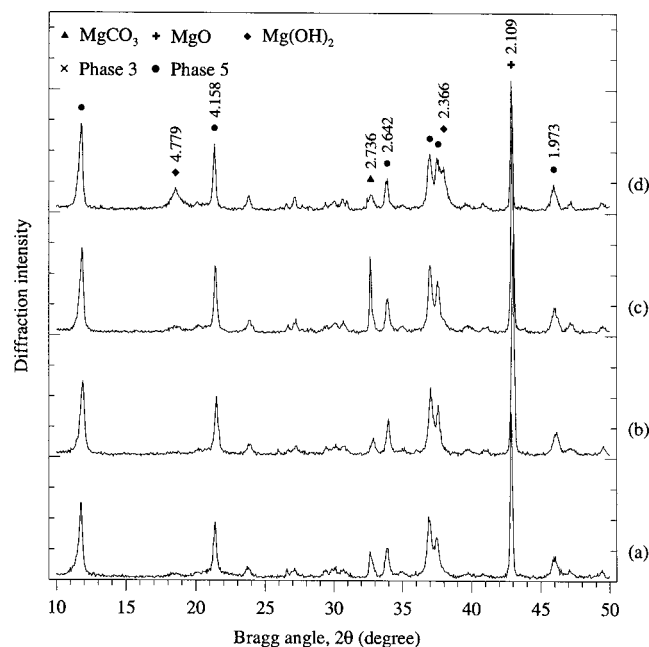


Fig. 5. X-ray diffractograms of the mixtures: (a) M11/H12 (Point g in Fig. 3); (b) M13/H12 (Point h in Fig. 3); (c) M13/H14 (Point i in Fig. 3); and (d) M13/H16 (Point j in Fig. 3)

The X-ray diffractograms of M07/H14, M07/H16, M09/H16, and M17/H16 are compared in Fig. 6 for further information. As shown in Fig. 6, those four mixtures all have Phase 5 crystals as the dominant reaction product. So far, the phase assemblages identified by X-ray diffractograms agree quite well with the phase diagram except one thing: the $\text{Mg}(\text{OH})_2$ content predicted by phase compatibility in the phase diagram is different to that estimated by X-ray diffractogram (XRD). From the phase diagram as

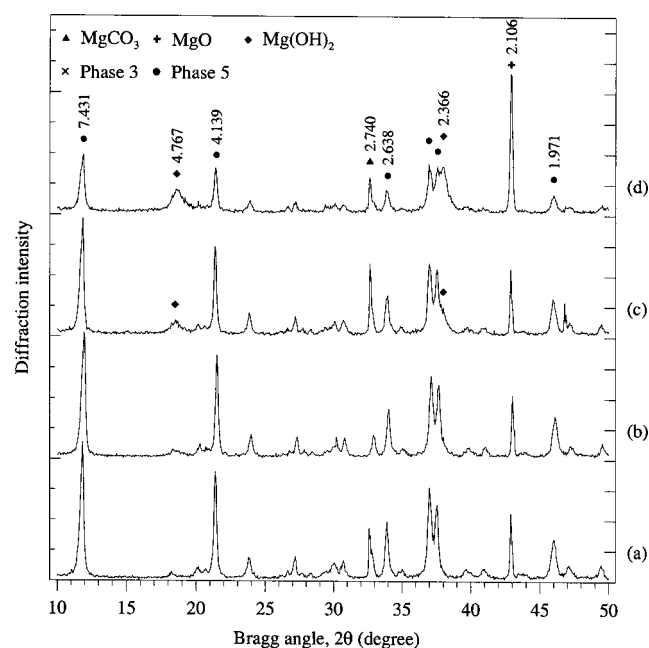


Fig. 6. X-ray diffractograms of the mixtures: (a) M07/H14 (Point c in Fig. 3); (b) M07/H16 (Point d in Fig. 3); (c) M09/H16 (Point f in Fig. 3); and (d) M17/H16 (Point k in Fig. 3)

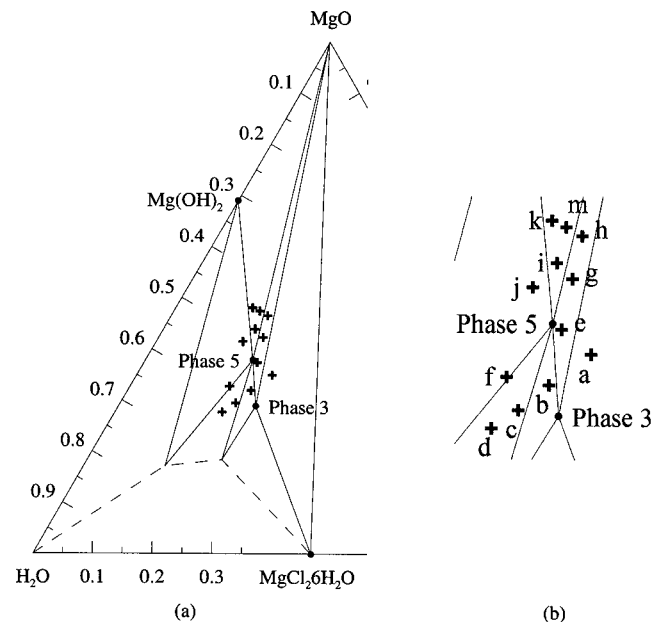


Fig. 7. Adjusted composition points of the mixtures (Fig. 3) with approximated MgO reactivity: (a) isothermal section at room temperature; (b) magnified portion of (a)

shown in Fig. 3, Mixtures M07/H14, M07/H16, M09/H16, M13/H14, M13/H16, and M17/H16 (Points c, d, f, i, j, and k) should all have certain amount of $\text{Mg}(\text{OH})_2$. The $\text{Mg}(\text{OH})_2$ phase is, however, only identified in Mixtures M13/H16 [Fig. 5(d)], M09/H16, and M17/H16 [Figs. 6(c and d)]. Therefore, the apparent molar ratios should be recalculated that the reallocated composition points to be matching with the XRD measurements, as follows.

Rate of Consumption of MgO

Except the chemical purity, the molar ratios given in Table 3 were calculated based on the assumption that 100% of the starting materials would be consumed in the chemical reactions. The experimental results show that such assumption is not appropriate at least for one of the three starting materials. From Fig. 3, it shows that any meaningful reduction of the reactivity, or rate of consumption, of MgCl_2 or water would move the composition points of M07/H12 and M09/H12 (Points b and e) out of the narrow compatibility triangle MgO –Phase 5–Phase 3. The X-ray diffractograms, however, confirm that those mixtures have both Phases 3 and 5 among their reaction products [Figs. 4(b and c)], and the corresponding composition points should be lying in that compatibility triangle. Therefore, the reactivity, or rate of consumption, of MgO should be taken into account to reallocate the composition points of the corresponding mixtures in the phase diagram.

The reduction of the reactivity of MgO will shift the composition points towards the side-line H_2O – MgCl_2 of the phase diagram. In that way, the composition points of M07/H14 and M07/H16 (Points c and d in Fig. 3) will be pulled down and out of the compatibility triangles containing the $\text{Mg}(\text{OH})_2$ phase to match with the X-ray diffractograms. By a trial, it can be done with the reactivity of MgO reduced to about 52%, and the new composition points in the phase diagram are shown in Fig. 7. Fig. 7 shows that M07/H14 and M07/H16 (Points c and d) would not have any $\text{Mg}(\text{OH})_2$ content, while M09/H16 (Point f) would have a little bit, and M13/H16 and M17/H16 (Points j and k) more $\text{Mg}(\text{OH})_2$.

Table 4. Recommended Usage of the Starting Materials of MOC Cement

MgO quality (%) ^a	MgO/MgCl ₂ (ratio)		Water usage
	Molar	Weight	
80	8.5	3.6 p	Depends on the workability requirement
70	10	4.2 p	Depends on the workability requirement
60	11.5	4.8 p	Depends on the workability requirement
50	13.5	5.8 p	Depends on the workability requirement
40	17	7.2 p	Depends on the workability requirement

Note: p =reciprocal of chemical purity of MgO powder.

^aExpected MgO reactivity, or rate of consumption.

These predictions match well with the X-ray diffractograms of the corresponding mixtures as shown in the Figs. 5 and 6.

Furthermore, Point b (M07/H12) is more closer to the composition point of Phase 3 than Point e (M09/H12) as shown in Fig. 7. Besides, Point b lies in the compatibility triangle Phase 5–gel–Phase 3, while Point e lying in the compatibility triangle MgO–Phase 5–Phase 3. That means M07/H12 would have amorphous gel phase and more Phase 3 crystals than M09/H12, leading to a lower mechanical strength. These observations from the phase diagram also agree well with the experimental results of strength measurement and phase identification. Finally, Mixture M13/H12 (Point h) has Phase 5 and MgO as the dominant components in the phase assemblage [Fig. 5(b)], and the degree of crystallization of Phase 3 may be diminished.

Recommendations for Practical Usage

With the approximated consumption rate of the MgO powder, the new composition points match well with the X-ray diffractograms and strength measurements. The recalculated molar ratio of MgO/MgCl₂ of Mixture M13/H12 is decreased from 13 to 6.8, which is still higher than the theoretical value of 5. It shows that the mechanical properties of MOC cement rely not only on the formation of interlaced needle-like Phase 5 crystals, but also the water content and pore structure of the final product. The mixtures with a compressive strength higher than 120 MPa, within 80% of the maximum strength obtained, are M11/H12, M13/H12, M13/H14, and M15/H14 (Points g, h, i, and m in Fig. 7). It demonstrates that the optimum MOC system, in terms of obtaining Phase 5 and having good mechanical properties, is located near and above the composition point of pure Phase 5 in the phase diagram.

It is believed that the excess or unconsumed MgO powder acts as a filler and filling up voids leading to a more compact microstructure associated with high mechanical properties. Normally, the consumption rate of MgO would be ranged from 40% up to 80% depending on the thermal history and storage condition. The apparent molar ratio of MgO/MgCl₂ for a normal practice with different reactivity of MgO powder are recommended in Table 4. The equivalent weight ratios between MgO and MgCl₂ are also given for the convenience of end users. The water content varies much depending on the reactivity of MgO powder as well as the requirement of the workability of the MOC paste. A 40% of water to powder ratio would be a good starting point followed by further readjustment according to the practical situation.

Conclusions

A parametric study of magnesium oxychloride cement with a wide range of molar ratios of MgO/MgCl₂ and H₂O/MgCl₂ is

conducted. By considering the strength measurement and phase identification of the mixtures, it shows that the best combination of the molar ratios of MgO/MgCl₂ and H₂O/MgCl₂ for obtaining Phase 5 crystals as well as good mechanical properties of MOC cement are 13 and 12. The question remaining is why such a high apparent molar ratio of MgO/MgCl₂, more than twice of the theoretical one, is required. The overestimated reactivity of the MgO powder employed in MOC cement is proved one of the most possible reasons. It is also found that the phase assemblages of MOC cements estimated by X-ray diffractogram could not agree well with the prediction from phase diagram analysis without a proper consideration of reactivity, or consumption rate, of starting materials.

To solve the problem, the composition points of the mixtures in the phase diagram have to be reallocated by modifying the reactivity of MgO content to match the phase assemblages identified by X-ray diffractograms. By doing so, the reactivity of the MgO powder employed in MOC cement can be approximated when the experimental measurements match the predictions from the phase diagram. Moreover, it is found that the optimum MOC system, in terms of obtaining Phase 5 and having sound mechanical properties, is located above and close to the composition point of pure Phase 5 in the phase diagram. That may be due to the excess or unconsumed MgO powder acting as a filler and filling up the voids, leading to a more compact microstructure associated with a high mechanical strength of MOC cement.

Finally, the apparent molar ratios of MgO/MgCl₂ for normal practice with different reactivities of MgO powder are recommended. The equivalent weight ratios between MgO and MgCl₂ are also given for the convenience of end users.

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