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Study of using Dolomite as Starting Material Resource to Produce Magnesium Oxychloride Cement

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Abstract

In this paper, the starting materials necessary for producing magnesium oxychloride cement (MOC) were produced from dolomite ore using leaching-carbonation-evaporation-pyrohydrolysis processes. The influences of the molar ratio of MgO/MgCl₂ (Mₚ) and H₂O/MgCl₂ (Hₚ) on the properties of the MOC (cured for 7 days) were determined using physico-mechanical methods. SEM and XRD analyses were conducted to determine the change of phase and microstructure of the selected 7-day MOC depending on the production conduction. The experimental results show that the best mix proportion of 7-day MOC was found to be Mₚ₉₂H₁₄ and its thermal conductivity, flexural strength, and compressive strength values were found to be 1.202 W/mK, 4.22 MPa, and 87.7 MPa, respectively. The water resistance of the MOC was improved by a small amount of H₃PO₄ (4% of MgO by weight). Consequently, if MOC is produced from dolomite, high-purity synthetic aragonite and CO₂ would be obtained as byproducts, which are strongly demanded.

1. Introduction

Cement is one of the most important building materials and has accelerated industrialization and modernization. However, during cement production, CO₂ gas is released into the atmosphere because of the calcination of limestone (CaCO₃). The production of 1 t of cement generates 0.9 t of CO₂ as a byproduct (Hendriks et al. 2004), which represents approximately 6–8% of all anthropogenic carbon emissions. The amount of CO₂ emitted into the atmosphere because of the calcination of limestone (CaCO₃) ore to produce one of the raw materials (MgO) used to prepare MOC (El-Mahllawy et al. 2012; Tan et al. 2015). The suitability of dolomite ore for producing MOC was evaluated by Kacker et al. (1970).}

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They partially calcined dolomite at 750°C for 2 h and then added it to an MgCl₂ solution to prepare MOC. CaCO₃ acted as a filler in the mix proportion of their MOC. The methodology adopted in the present work differs from those of the aforementioned studies in which dolomite was used to produce MOC. In the present work, the raw materials necessary for producing MOC were obtained from dolomite by leaching–precipitation–evaporation–pyrohydrolysis processes. The influences of the MgO/MgCl₂ and H₂O/MgCl₂ molar ratios and the amount of phosphoric acid (H₃PO₄) on the properties of MOC were investigated in detail. The compressive strength, bulk density, and water immersion test were measured for each prepared MOC. In addition, SEM examination was performed to determine the change of selected MOC microstructures. The thermal conductivity, fire strength, flexural strength, and dimensional stability properties of the final MOC product was determined.

2. Experimental procedure

2.1. Raw materials and mix proportion

The starting materials (MgO and MgCl₂·6H₂O) for producing MOC were obtained from dolomite. The chemical composition of the dolomite used for producing raw materials to prepare the MOC was 19.84% MgO, 31.37% CaO, and 48.2% CO₂. The purity of each raw material was quite high (99.9%) because the impurities had been removed after the leaching process (Altiner et al. 2016). During the leaching process, CO₂ gas was stored in a lab-scale gasometer for use in the subsequent carbonation process that was conducted to remove Ca²⁺ ions as synthetic aragonite crystals from the leachate solution (Altiner and Yildirim 2016) and obtain an Mg-rich solution. Afterwards, the solution was crystallized at 130°C using an evaporator (Altiner and Yildirim 2016) to obtain MgCl₂ crystals. Finally, MgO particles were prepared through pyrohydrolysis and calcination processes (temperature: 650°C, reaction time: 270 min). The production of MgO from dolomite will be explained in a future study. The obtained materials were subsequently used to produce MOC.

P5 is known to be the most stable MOC phase at ambient temperature; the MgO/MgCl₂ and H₂O/MgCl₂ molar ratios were determined for this purpose (Table 1). Given the molar ratios in Table 1, MgCl₂·6H₂O was first dissolved in tap water to obtain a magnesium chloride solution. Thereafter, MgO particles were added to the solution, which was stirred gently (80 rpm) by a mechanical stirrer for 15 min to obtain homogeneous MOC pastes. The prepared pastes were poured into Plexiglas molds using a vibration table and cured for 24 h at ambient temperature. The initial and final setting times of the MOC samples were determined via a Vicat apparatus according to standard ISO 9597:2008. After hardening for 24 h, each MOC sample was removed from its mold and cured in air (20°C and 50% relative humidity) for 7 days.

In this paper, the mix proportions of MOC samples given in Table 1 are denoted as MₓHₙ (where M = nMgO/nMgCl₂, H = nH₂O/nMgCl₂ (Li and Chau 2007). For example, the notation M₄.74H₁₄ indicates the MOC with MgO/MgCl₂ and H₂O/MgCl₂ molar ratios of 4.74 and 14, respectively.

2.2. Effects of H₃PO₄ addition on water resistance of MOC

The 7-day MOC (20 × 20 × 20 mm³) samples were immersed in tap water for 10 days to determine their water resistance. By the end of the tenth day, the compressive strength value of the MOC samples was determined. The water resistance coefficient (Wₚ) was calculated using Eq. 1 (Liu et al. 2015).

\[ W_p = \frac{CSI_{im}}{CSI_{7d}} \]

where \( W_p \) is the water resistance coefficient, \( CSI_{im} \) is the compressive strength value of the MOC immersed in tap water for 10 days (MPa), and \( CSI_{7d} \) is the compressive strength value of the MOC sample on day 7 (MPa). The amounts of Cl⁻ ions before and after the immersion tests were measured via the silver nitrate (AgNO₃) titration method (Association 1992). Given all of the experimental results, a small amount of H₃PO₄ (1–4% of H₃PO₄/MgO ratio) was added to the magnesium chloride solution before the addition of MgO to produce the selected mix proportion of MOC. The influences of the H₃PO₄ addition on the water resistance of the selected MOC were then investigated using all of the aforementioned procedures described in this paper.

2.4. Testing and characterization methods

2.4.1. Physico-mechanical properties of MOC

The compressive strength value (CSV) of each 7-day MOC sample (20 × 20 × 20 mm³) was determined according to TS EN 12390-10 (2010). The loading speed was 50 N/s. Each test was repeated two times to reduce the experimental error of the CSVs of the MOC samples.

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Table 1. Mix proportion of MOC with various molar ratios.

| MgO/MgCl₂ (M)* | H₂O/MgCl₂ (H)* |
|---------------|---------------|
|               | 13  | 14  | 15  | 16  | 17  | 18  |
| 4.74          | X   | X   | X   | X   | | |
| 5.52          | X   | X   | X   | X   | X   | |
| 6.06          | X   | X   | X   | X   | X   | |
| 7.97          | X   | X   | X   | X   | X   | X   |

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In addition, the bulk density of 7-day MOC prepared with various M_xH_y molar ratios was determined according to standard TS EN-1097-6 (2015).

2.4.2. Evaluation of microstructures of selected MOC
After the CSV test, four selected samples with molar ratios of M_4.74H_13, M_5.52H_13, M_6.06H_14, and M_7.92H_15 selected from all of the samples were ground using an agate mortar for XRD and SEM examination. The microstructures on the fractured surface of each 7-day MOC were evaluated using a scanning electron microscope (Zeiss 55 and Philips XL 30S FEG) after the samples were coated with gold particles. The crystallographic properties of selected MOC was determined using a Rigaku X-ray diffractometer equipped with Cu Ka radiation in the 2θ range of 15 – 60° with a 0.02 step size.

2.4.3. Determination of final MOC product properties
The thermal conductivity (TC) of the final MOC product was measured through the hot wire method (ASTM 2013). The TC is the property of material to conduct heat, whereas the fire strength test is used to evaluate concrete behavior in terms of heat conduction in the event of a fire outbreak. The rate at which heat is transferred from a hot surface to the cold surface of a concrete component was determined through this method.

In addition, concrete can be classified on the basis of the experimental results of the fire strength tests. Fig. 1 shows the apparatus designed for the fire strength test. The average surface temperature of each MOC product was measured using a laser infrared thermometer with K-type thermocouple functionality (CEM, DT8835). The temperature of the bottom, middle, and upper points of the final MOC product were measured before the test. Afterwards, the fire source (900°C) was exposed to the unexposed surface of the product at a distance of 10 mm for 10 min. To represent the surface temperature of the unexposed side of the product, the temperature of each product was measured at the same points at 30-s time intervals during the fire test. The average temperature of the unexposed side of each MOC product was calculated. The heat transfer rate of the MOC was determined using the following equation (Anonymous 2016). The loss of MOC after each test was further weighted.

\[ R_{\text{rate}} = \lambda \cdot A \cdot (P_{T2} - P_{T1}) / 1000 \]  

where, \( R_{\text{rate}} \) is the heat transfer rate of MOC (J/s), \( \lambda \) is the thermal conductivity value of MOC (W/m·K), \( A \) is the surface area of MOC (m²), \( P_{T2} \) is the measured temperature of exposed surface by fire source (K), \( P_{T1} \) is the measured temperature of unexposed surface of MOC (K), \( P_k \) is the thickness of MOC (m).

The flexural strength value (FSV) of the final MOC product was determined according to TS EN 12390-5 (2010). Furthermore, the dimensional changes in the final MOC product at 70°C after 48 h were determined (TSE EN 1604, 2013). The length, width and thickness of MOC was measured before and after the dimensional change test. The volumetric change shows the dimensional stability of MOC cement. If the volumetric change was small, no structural problems related to cement would be observed.

3. Results and discussion

3.1. Effects of molar ratio of M_xH_y on production of MOC
The possible chemical reactions in the ternary system of MgO – MgCl_2 – H_2O for producing MOC were explained in detail in previous studies (Dehua and Chuanmei 1999; Karimi and Monshi 2011). We therefore do not discuss the chemical reactions that occur during the hardening of MOC here. We determined that the initial setting of each MOC paste began after 30 min, whereas the final setting time of the paste varied between 170 and 195 min depending on its mix proportion.

The presence of Cl ions was an important parameter in the MOC manufacturing process; changing the MgCl_2 concentration in the MOC paste led to the undesired MOC phase, as shown in the ternary phase diagram of the MgO–MgCl_2–H_2O system (Fig. 2). Each mix proportion of MOC was prepared to obtain P5. However, F3 can sometimes be obtained in the case of MOC samples with an M_4.74 ratio, as evident in the phase diagram. Depending on the M_4 ratio, more MgO than needed can be added to the mix proportion of MOC. However, excess MgO is not harmful and may act as a filler (Karimi and Monshi 2011). These findings were confirmed by the XRD analyses given in Fig. 3. Excess MgO and Mg(OH)_2 crystals were determined. Similarly, higher H_2O levels were required for workability of the MOC paste. The concentration of Cl⁻ ions, which can lead to corrosion problems, was kept to a minimum in
the MOC pastes (Sglavo et al. 2011). These ratios strongly affected the microstructure of the resulting MOC when MgO and H2O were added in greater-than-necessary amounts to the MOC paste, as shown in Fig. 4.

To obtain needle-shaped MOC crystals, the MOC molar ratio should be M 6.06H14 (Fig. 4c). With the addition of more MgO to the MOC, the needle-like crystals of MOC disappeared at the molar ratio of M7.92H15 because the pores in the MOC were filled by excess MgO particles (Fig. 4d). The intergrowth of needle-shaped crystals of the MOC led to an increase of its strength value (Karimi and Monshi 2011). The obtained results given in Fig. 5 are consistent with those of previous studies. The highest CSV of the 7-day MOC samples was 87.7 MPa in the case of M 6.06H14, which contains needle-shaped crystals (Fig. 4c). The CSVs of the 7-day MOC samples started to decrease with disappearing needle crystals of the MOC when excess MgO was added. For instance, the CSV of MOC M7.92H15 was found to be 80 MPa. Even though needle-shaped crystals appeared in MOC samples M 4.74H13 and M5.52H13, their CSVs were 42 and 77 MPa, respectively. These lower CSVs are related to the amount of MgO in the MOC samples. The bulk density of MOC prepared in various molar ratios was in the range of 1.71 and 1.98 g/cm3 as shown in Table 2.

### 3.2. Effects of H3PO4/MgO ratio on water resistance of MOC

MOC is not durable in water, although an increase of the MgO/MgCl2 molar ratio (M;) leads to an increase of its water resistance (Dehua and Chuanmei 1999). A possible decomposition reaction of MOC in water is represented as the following equation:

\[ \text{Mg}_5(\text{OH})_3\text{Cl} \cdot 4\text{H}_2\text{O} \rightarrow 2.5\text{Mg(OH)}_2 + 0.5\text{Mg}^{2+} + \text{Cl}^{-} + 4\text{H}_2\text{O} \]  

(3)

To obtain needle-shaped MOC crystals, the MOC molar ratio should be M 6.06H14 (Fig. 4c). With the addition of more MgO to the MOC, the needle-like crystals of MOC disappeared at the molar ratio of M7.92H15 because the pores in the MOC were filled by excess MgO particles (Fig. 4d). The intergrowth of needle-shaped crystals of the MOC led to an increase of its strength value (Karimi and Monshi 2011). The obtained results given in Fig. 5 are consistent with those of previous studies. The highest CSV of the 7-day MOC samples was 87.7 MPa in the case of M 6.06H14, which contains needle-shaped crystals (Fig. 4c). The CSVs of the 7-day MOC samples started to decrease with disappearing needle crystals of the MOC when excess MgO was added. For instance, the CSV of MOC M7.92H15 was found to be 80 MPa. Even though needle-shaped crystals appeared in MOC samples M 4.74H13 and M5.52H13, their CSVs were 42 and 77 MPa, respectively. These lower CSVs are related to the amount of MgO in the MOC samples. The bulk density of MOC prepared in various molar ratios was in the range of 1.71 and 1.98 g/cm3 as shown in Table 2.
XRD analyses shown in Fig. 6 reveal that the phase of selected MOC decomposed after immersion in water for 10 days. The MOC crystals converted to Mg(OH)₂ thereafter. This is in line with the SEM examination. It is clear that the needle-shaped crystals of MOC disappeared after immersion in water for 10 days because the MOC decomposed (Fig. 7). A comparison of SEM images of the MOC before and after immersion in water (for instance, Fig. 4(a) and Fig. 7(a)) clarified the disappearance of the needle-shaped crystals in the MOC. Furthermore, the presence of Cl⁻ ions in the water after the immersion test indicates the decomposition of the MOC. The amounts (by mass) of Cl⁻ ions dissolved from the MOC were 50.7% for M₄.74H₁₃, 56.1% for M₅.₅₂H₁₃, 51.2% for M₆.₀₆H₁₄, and 37.2% for M₇.₉₇H₁₅ after the immersion test.

Unfortunately, the decomposition of the MOC samples led to a decrease of their CSVs. A comparison of the CSV value of an MOC before and after an immersion test is shown in Fig. 8. The CSV of the MOC (M₆.₀₆H₁₄) decreased from 87.7 MPa to 18.05 MPa, which corresponds to a 79% reduction in the CSV. The CSV of the MOC was found to be higher depending on its Mₓ molar ratio after the immersion test. However, the MOC prepared in the molar ratio of M₆.₀₆H₁₄ was scattered as the cracks were formed on the surface of MOC during the immersion test. The water resistance coefficient (Wᵣ) of each MOC is listed in Table 3.

Therefore, H₃PO₄ additives were added at concentrations ranging from 1 to 4% (by weight of MgO) to the mix proportion of the MOC composition (M₆.₀₆H₁₄) with the highest CSV to increase its Wᵣ (Deng 2003; Tan et al. 2014; Gapparova et al. 2015). Thereafter, the CSVs of the MOC samples with various H₃PO₄/MgO ratios were determined; the results are shown in Fig. 9. An increase of the H₃PO₄/MgO ratio in the MOC led to an increase of the Wᵣ of the MOC. After the immersion tests, the CSV of the MOC with 4% H₃PO₄/MgO was found to be 61 MPa. The Wᵣ of this MOC was calculated.
lated to be 0.69 (= 61/87.7).

Lu et al. (1994) have reported that a gelatinous structure formed on the surface of an MOC because of the presence of PO$_4^{3-}$ anions in its composition; they also reported that the pores of the MOC were already coated by the gelatinous layer before the immersion test. Consequently, water could not diffuse inside the MOC, preventing the decomposition of its structure (Tan et al. 2014). These findings were confirmed by the XRD examination shown in Fig. 10. The characteristic peaks belonging to P5 of MOC were determined. MgO and Mg(OH)$_2$ peaks were further observed. No crystal peaks related to phosphor were observed due to an adding small amounts of H$_3$PO$_4$ to the mix proportion of MOC.

In the present work, the structure of the MOC did not decompose after immersion in water, as shown in the SEM images (Fig. 11). Needle-shaped crystals were observed in each SEM image. However, Cl$^-$ ions were detected after the immersion test. The amounts of Cl$^-$ ions dissolved from the MOC varied between 12.2 and 22.65% (by mass) depending on its H$_3$PO$_4$/MgO ratio. With respect to the SEM images in Fig. 11, more pronounced crystals in the MOC were observed when the H$_3$PO$_4$/MgO ratio in the MOC was 4%. The 7-day MOC (M$_{0.06}$H$_{14}$, H$_3$PO$_4$/MgO ratio: 4%) is hereafter referred to as the final product.

3.3. Determination of final MOC product properties

The flexural strength value (FSV) of MOC cement varied between 2 – 14 MPa depending on its production condition (Misra and Mathur 2007; Zhou and Li 2012; Wen et al. 2014). In this paper, the FSV of the final MOC product was found to be 4.22 MPa that was in good agreement with those studies. The increase of curing days leads to increase the FSV of the final product as indicated in the previous study (Wen et al. 2014). Then, the FSV of the final MOC product (thickness of 10 mm) in this paper will increase in an extended curing times. The final MOC product was stable at 70°C. There was no considerable difference determined on the product after 48 h as shown in Table 4.

The TCV of the final MOC product was found to be 1.202 W/(m·K), which is consistent with the results of a previous study (Xu et al. 2016). This value is one of the most important properties of an MOC. The heat source was exposed for 10 min to the surface of the final MOC.

| Parameter | Before | After | Difference (%) |
|-----------|--------|-------|----------------|
| Length (mm) | 71.74  | 71.45 | 0.40           |
| Width (mm)  | 71.12  | 71.04 | 0.08           |
| Thickness (mm) | 9.68   | 9.40  | 2.89           |

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Table 4. Dimensional stability of final MOC product (thickness of 10 mm).

| Parameter | Before | After | Difference (%) |
|-----------|--------|-------|----------------|
| Length (mm) | 71.74  | 71.45 | 0.40           |
| Width (mm)  | 71.12  | 71.04 | 0.08           |
| Thickness (mm) | 9.68   | 9.40  | 2.89           |
product ($M_6.06H_{14}$, H$_3$PO$_4$/MgO: 4%) prepared with a thickness of 10, 25, 50 and 70 mm. The temperature of the unexposed surface of the MOC with a thickness of 10 mm increased from 15°C to 382°C after the test. However, this value tends to decrease with increasing thickness value of MOC. The differences in the temperature of the exposed surface of the MOC were determined before and after the test. Table 5 gives the experimental results of the fire resistance tests. No ignition was observed and that the flame did not advance on the surface of the final MOC product.

When the thickness of that product was $\geq$25 mm, the differences between exposed and unexposed surfaces of the final MOC product were found to be lower 30°C and the weight loss was below 11%. The heat transfer rate of the final MOC product was greatly influenced by its thickness (Fig. 12). When the thickness of MOC increased to 70 mm from 10 mm, the $R_{\text{rate}}$ decreased to 1.17 J/s from 3.82 J/s. Considering these experimental results, the final MOC product was classified as A1 construction product according to the TSE 13501-1+A1 2013 (TSE 2013).

### 4. Conclusions

This paper shows that dolomite ore can be evaluated as starting materials for producing magnesium oxychloride cement (MOC). Leaching–carbonation–evaporation–pyrohydrolysis processes were carried out to prepare the starting materials (MgO and MgCl$_2$). The best experimental results in terms of physico-mechanical properties, XRD and SEM examination were obtained when the mix proportion of MOC was $M_{6.06H_{14}}$. To increase the WR of the MOC, a small amount of H$_3$PO$_4$ (4% of MgO by weight) was added. The WR of the MOC was found to be 0.69, which is an acceptable value for the composite concrete.

Consequently, approximately 2.49 t of dolomite is required to produce 1 t of MOC based on the experimental results obtained in this work. This value would be decreased with adding of filler to produce lightweight MOC product. In a next study, the influences of using expanded perlite and exfoliated vermiculite as fillers on the property of MOC product will be investigated. During the production per 1 t of MOC, 1.38 t of synthetic aragonite (CaCO$_3$) and 0.58 t of CO$_2$ with a high purity would be obtained as byproducts, which are highly demanded in many industrial areas.

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