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Mitigation of Drying and Carbonation Shrinkage of Cement Paste using Magnesia

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Abstract

In this work, the effect of magnesia (MgO) on the hydrated phase assemblage, microstructure, as well as drying and carbonation shrinkage of hardened Portland cement paste is studied. By means of X-ray diffraction, scanning electron microscopy (SEM/EDS), and thermodynamic simulation, it shows that MgO hydration does not substantially alter the phase assemblage and microstructure of hardened cement paste, except the formation of brucite. The 5% MgO addition reduces the drying shrinkage of cement by about half, which cannot be simply explained by the molar volumetric increase due to MgO to brucite conversion. The results suggest that besides generating early-age expansion offsetting subsequent drying shrinkage, the formed brucite at early stages can enhance the matrix stiffness probably due to crystal formation with reinforcing potentials. In addition, the MgO addition improves the carbonation resistance of hardened cement due to the formation of magnesium-containing calcium carbonates. However, the effectiveness of MgO in mitigating carbonation shrinkage depends on the drying and carbonation sequence.

1. Introduction

Shrinkage mitigation through the use of expansive agents (e.g., sulfoaluminate-type, CaO-type, or MgO-type) has been a practical way of controlling the shrinkage cracking in restrained concrete elements (Bentz and Jensen 2004; Jin et al. 2014; Polat et al. 2015). The primary working mechanism of most expansive agents is that they promote the formation of expansive products (e.g., AFt, portlandite, or Mg(OH)2) at the early stage of cement hydration, which offsets the concrete shrinkage occurred at the later stage (Bentz et al. 2002; Kovler and Zhutovsky 2006; Ye and Radlińska 2017). The amount and type of the expansive products and the kinetics of the associated expansive reactions considerably affect the effectiveness of shrinkage compensation. Due to the concerns that long-period hydration of expansive agents after cement hardening can cause disruptive expansion, normal practice limits the quantities of the expansive agent to small quantities. However, in reality, the expansive reaction in concrete is often difficult to regulate and control, due to the various reactivity of the expansive components and the highly localized expansive products formation (Bentz and Jensen 2004).

Although using sulfoaluminate-type expansive additive is the most common method to generate expansion in concrete, the potential use of MgO-based additive has gained significant attention in recent years. In comparison to the conventional sulfoaluminate-type additive, the MgO-based additive has several advantages, including less water consumption for expansive reactions and higher stability of the expansive products (i.e., Mg(OH)2) (Mo et al. 2010). In fact, the use of MgO in the field dated back to the construction of Baishan arch-gravity dam in China in the 1970s. The 40 years’ industrial experience in China in implementing MgO-based shrinkage compensating additive shows that it can be a more efficient and economical measure than conventional ones (Gao et al. 2008; Mo et al. 2014). Extensive studies have been conducted in China regarding the manufacture of MgO-based expansive additive, the hydration and expansion of MgO in cementitious materials, and the performance of MgO-modified concrete (Mo et al. 2014).

According to the previous study, the shrinkage compensation of MgO-based additive in concrete is through the formation of brucite (Mg(OH)2), which results in 118% volume increase. However, this molar volume increase due to MgO to brucite conversion cannot fully explain the amount of expansion in cementitious solids. As such, the crystal growth theory in confined regions has been proposed (Chatterji 1995), suggesting that the transformation of MgO to Mg(OH)2 may not occur in situ; instead, it involves a series of dissolution and precipitation processes. Therefore, the presence of aqueous Mg2+ due to MgO hydration may have an influence on the hydrated phase assemblage and microstructure of hardened cement. However, little study has ever exclusively investigated the effect of the MgO-based additive on phase assemblage and microstructure of hardened cement paste (HCP).

On the other hand, the previous studies mainly focused on the effect of MgO on the autogenous and thermal shrinkage of HCP (probably due to dam-oriented application) (Qing et al. 2004; Mo et al. 2012). Little information is available regarding the effect of MgO on the development of drying and carbonation
shrinkage. In addition, the presence of aqueous magnesium in HCP may have an impact on the formation of calcium carbonate under natural carbonation. Nevertheless, the effect of a small quantity of MgO addition on the carbonation resistance and phase modification of HCP is scarcely reported.

To fill the aforementioned research gaps, this work investigates the effect of MgO-based additive on the hydrated phase assemblage, microstructure, as well as drying and carbonation shrinkage of HCP. New insights regarding the mechanisms of MgO-based additive in controlling shrinkage and carbonation are provided. This work contributes to a better understanding of the microstructure and properties of MgO-modified cement, which would, in turn, benefit the design of shrinkage and carbonation-resistant concrete.

2. Materials and methods

The ordinary Portland cement (OPC) used was ASTM Type I/II with the chemical composition and physical properties listed in Table 1. A commercial MgO-based additive was used, with its X-ray diffraction analysis shown in Fig. 1. Three types of cement paste with various amounts of MgO-based additive were prepared, i.e., 0% MgO (i.e., plain system), 2.5% MgO, and 5.0% MgO by mass of cement. All of these three mixtures had the same water-to-cement (w/c) ratio of 0.40.

The paste was cast into a non-standardized prism mold with dimensions of 12.7 × 12.7 × 139.7 mm. The small specimen geometry enabled them to reach drying equilibrium with the environment in a much shorter time and prevented the potential cracking due to drying gradient, in comparison to the conventional methods (e.g., ASTM C596). All specimens were cured in a moist room (100% RH, 23 ± 0.5°C) for 24 h before de-molding and then continued to be cured in the moist room.

After 7 d moist curing, the specimens were placed in two environmental chambers with different types of gaseous conditions, to study the effect of carbonation on the shrinkage development of MgO-modified HCP during drying: (i) Nitrogen gas condition: The environmental chamber was programmed at 50% RH and 23 ± 0.5°C, with the dry nitrogen (N\textsubscript{2}) being purged continuously. The CO\textsubscript{2} concentration was periodically checked to be consistently 0 ppm. (ii) Atmospheric condition: The environmental chamber was maintained at 50 ± 4% RH and 23 ± 0.5°C, with dry air being purged. The CO\textsubscript{2} concentration was measured as about 400 ppm.

After being dried for 46 d, the specimens initially dried in nitrogen gas condition was moved to the atmospheric condition, to study the effect of drying and carbonation sequence on the length and mass change of MgO-modified HCP. The changes in length and mass were recorded utilizing a digital comparator with a measuring precision of 0.001 mm and a balance with a measuring precision of 0.01 g. The measurement was terminated at 75 d, in which the length change was less than 1.0% over the one-week period.

The paste was also cast in a series of sealed plastic vials to investigate the effect of MgO addition on the phase evolution in HCP at early ages, namely, 1 d and 7 d. In addition, to study the effect of natural carbonation on the phase evolution of MgO-modified HCP, the paste specimens after drying in nitrogen and atmospheric conditions for 46 d, were collected for X-ray diffraction (XRD) analysis. For the one dried in nitrogen, the entire bulk of paste samples was collected, while for the one dried in the atmosphere, only the surface layer with a thickness of 1mm was collected. This consideration was because the extent of carbonation, as well as the phase assemblage, for carbonated samples, was depth-dependent.

The collected specimens were crushed and milled for about 5 - 10 minutes and front-filled into a zero-background plate. XRD data were collected using a PANalytical Empyrean diffractometer in a conventional Bragg-Brentano θ - 2θ configuration. CuKα X-ray (\(λ=1.5418\) Å) was generated using 40 mA and 45 kV operating conditions. The samples were scanned continuously between 5° and 45° 2θ.

![Fig.1 XRD pattern of the used MgO-based additive.](image-url)
The specimens after drying in nitrogen and atmospheric conditions for 46 d were collected for scanning electron microscopy (SEM/EDS) analysis using FEI Quanta 200 equipped with backscattered electron (BSE) and X-ray detectors. The small pieces of specimens were immersed in isopropyl alcohol for at least two weeks and then vacuum dried for 48h. Afterwards, they were epoxy-impregnated, polished down to 1µm, and carbon coated prior to SEM/EDS acquisition.

3. Thermodynamic modeling

Thermodynamic modeling was performed to study the effect of MgO addition on the phase assemblage of HCP. It was calculated based on the Gibbs energy minimization software GEM-Selektor v.3, with the CEMDATA 14.01 and PSI-Nagra databases (Table 2). For the C-S-H phase, the CSHQ model was used (Kulik 2011). The formation of siliceous hydrogarnet was suppressed due to its slow formation kinetics (Lothenbach 2010). The potential formation of magnesium silicate hydrate (M-S-H) phase was not considered in this study due to the relatively small dosage of MgO added and the absence of highly reactive silica in the system. In addition, the activity coefficients were calculated using the extended Debye–Hückel equation with the ionic size and extended term parameter chosen for KOH. The simulation adopted the composition shown in Table 1 and assumed complete hydration of raw cement and MgO. The simulation was performed using 100 g cement with a varying amount of MgO addition (0 to 15% by mass) in a nitrogen atmosphere with the temperature and pressure of 25°C and 1 bar.

4. Results and discussion

4.1 Phase assemblage

Figure 2 shows the XRD patterns of raw OPC and hardened cement paste (HCP) with various amounts of MgO addition. The raw OPC used in this study inherently contains some quantities of MgO, which is typically the dead-burnt MgO (periclase) formed during clinkering. The main hydrated phases in HCP include semi-crystalline calcium-silicate-hydrate (C-S-H), portlandite, AFt (ettringite), AFm (monocarbonate, hemicarbonate), and brucite. It can be seen that besides increasing the intensity of the peaks corresponding to unhydrated MgO, the addition of MgO-based addi-

| Phase                      | Log $K_{SO}$ |
|----------------------------|--------------|
| Portlandite (CH)           | -5.2         |
| Brucite (MH)               | -16.84       |
| Monocarbonate (C$_2$AcH$_{11}$) | -31.47     |
| Ettringite (C$_6$As$_3$H$_{32}$) | -44.9      |
| Hydrotalcite (M$_2$AH$_{10}$) | -56.02      |
| C-S-H (quaternary solid solution) |
| TobH (C$_{2.5}$SH$_{1.5}$) | -6.19       |
| TobD (C$_{4.5}$S$_{2.5}$H$_{1.33}$) | -6.90     |
| JennH (C$_{1.33}$SH$_{2.17}$) | -10.96     |
| JennD (C$_{1.5}$S$_{0.67}$H$_{2.5}$) | -10.47     |

Note a: In cement chemistry notation: C = CaO, S =SiO$_2$, A = Al$_2$O$_3$, M =MgO, s=SO$_3$, c=CO$_2$, H=H$_2$O, C-S-H=Calcium-silicate-hydrate, AFt = A group of calcium sulfoaluminate hydrates, AFm= A group of tetracalcium aluminates-ferrite hydrates.

Fig. 2 XRD patterns of HCP with various amounts of MgO addition.
tive does not substantially change the solid phase assemblage in HCP. According to the previous study (Vandeperre et al. 2008), the hydration of MgO contributes to the formation of brucite and hydroxylcrite; however, the brucite is in a minor quantity for all the mixtures in the present study, while hydroxylcrite, if exists, is likely in a semi-crystalline structure and hard to be identified by XRD.

Figure 3 shows the compositional plots of the hydrated solid phases in plain and MgO-modified HCP based on SEM/EDS microanalysis. Due to the large interaction volume between electrons and specimen, each SEM/EDS microanalysis point usually contains a mix of various phases. In order to qualitatively identify the mixed phases presented and to deal with the statistical variations of composition, molar relationship plots are typically implemented (Scrivener 2004). Based on the Si/Ca ratio versus Al/Ca ratio plot, C-S-H, portlandite, Aft, and AFm are clearly identified, which is in agreement with the XRD results. However, the S/Ca ratio versus Al/Ca ratio plot also indicates the presence of monosulfate phase, which is however not unambiguously detected in XRD. It may be attributed to the relative minor quantities of monosulfate, in comparison to the carbonate-containing AFm phases such as monocarbonate. The formation of monocarbonate in the present HCP is attributed to the presence of limestone in raw OPC (Lothenbach et al. 2008), as also predicted by thermodynamic modeling (see Fig. 4). The positive correlation between (Na + K)/Ca ratio versus Si/Ca ratio may be an indication of alkali binding in the C-S-H (Ye et al. 2017b). The Mg/Ca ratio versus Al/Ca ratio does not show a clear pattern; however, when both Mg/Ca and Al/Ca ratios are smaller than 0.06, a linear compositional relation can be established, which may be an indication of hydroxylcrite formation (Haha et al. 2011; Ye and Rادلینسka 2016a). Nevertheless, it is clear that the hydroxylcrite is not the sole phase containing both Mg and Al in HCP. Overall, the SEM/EDS analysis suggests that, besides some points showing Mg-rich phases (unhydrated MgO or brucite), there is no substantial difference in the types of formed solid phases between plain and MgO-modified HCP. This observation is in agreement with the previous finding that MgO has little effect on the phase assemblage of HCP and the hydration of MgO and OPC are almost independent (Vandeperre et al. 2008). It is suggested that due to the high OH⁻ concentration in cement pore solution, the
Mg$^{2+}$ diffused from MgO hydration can combine with the surrounding OH$^-$ to form a supersaturated solution with respect to Mg(OH)$_2$ precipitates (Mo et al. 2012). As such, Mg(OH)$_2$ forms at and near the sites of MgO grains, limiting the further dissolution of MgO and thus the other interactions of Mg$^{2+}$ with other hydrated phases.

Figure 4 shows the results from thermodynamic modeling, which displays that the MgO addition first enhances hydrotalcite formation, then brucite when the amount of added MgO reaches about 3.5% by mass. The thermodynamic modeling indicates that the carbonate-containing AFm and AFt phases tend to be gradually decomposed with the progressing of MgO hydration, as the MgO can take Al from AFm and/or AFt to form hydrotalcite. However, a previous study on the phase assemblage of HCP with high MgO content (up to 50% by weight) shows that the brucite is the major hydration product, with hydrotalcite as a minor component (Vandeperre et al. 2008). The discrepancy can be explained by the fast formation of Mg(OH)$_2$ surrounding hydrating MgO grains that shortens the diffusion distance of Mg$^{2+}$ and prevents the occurrence of localized aqueous environments with simultaneous high concentrations of Mg$^{2+}$ and Al$^{3+}$; in contrast, the thermodynamic modeling assumes equilibrium states of MgO with the entire chemistry system.

4.2 Microstructure

Figure 5 shows the BSE images of polished plain and 5% MgO-added HCP, and Fig. 6 shows a representative low-magnification BSE image of 5% MgO-added HCP and its corresponding Mg EDS mapping. It can be seen that Mg-rich solid particles up to about 50 µm, which is the unhydrated MgO or formed Mg(OH)$_2$ or a mix of both, are well distributed in the matrix. According to the previous study, the MgO hydrates locally, and thus the formed Mg(OH)$_2$ mainly locates in the space that originally occupied by the MgO particles (Mo et al. 2012). As such, it was believed that the self-expansion of MgO is solely caused by the transformation of MgO into Mg(OH)$_2$. However, based on the present study, the MgO is not completely hydrated and the observed dark Mg-rich particles in BSE images could be assigned to
the unhydrated MgO or, more likely, a mix of MgO and Mg(OH)₂. It is because that at 46 d, the peak intensity corresponding to MgO in XRD is still stronger in MgO-modified HCP than that in the plain system. If one assumes that these dark particles are all MgO grains, about 50% of MgO was unhydrated at 46 d based on the Mg mapping image analysis. The hydrated MgO may remain in the original sites, forming Mg(OH)₂, as well as interact with the other hydrated phases in the cement matrix, e.g., forming hydrotalcite or Mg²⁺-exchanged portlandite. Nevertheless, the later reaction seems to be limited and depends on the surrounding aqueous environment of hydrating MgO.

4.3 Drying shrinkage

Figures 7(a) and 7(b) show the time-dependent changes of length and mass of plain and MgO-modified HCP dried at both nitrogen and atmospheric conditions. It can be seen that as a result of MgO addition, the HCP exhibits considerable expansion at early ages during moist
curing, which successfully offsets the shrinkage occurred during subsequent drying. While the expansion strain during moist curing due to MgO addition is about hundreds of micron strain, it reduces the drying shrinkage at 46 d by up to 2000 micron strain. It implies that either the hydration of MgO in HCP continues even during drying, or the MgO modifies the mechanical response of solid skeleton to drying-induced stresses. The addition of 2.5% and 5.0% MgO reduces the drying shrinkage of HCP at 46 d by 25% and 45%, respectively. It is somehow surprising that the minor quantities of Mg(OH)₂ formation is highly effective in controlling the drying shrinkage of HCP. It should be noted that even in the case without moist curing (i.e., sealed condition), MgO can also generate early-age expansion and mitigate autogenous shrinkage (Mo et al. 2012).

In addition, the incorporation of MgO barely affects the moisture loss of HCP, which may imply that the pore structure of HCP keeps relatively unchanged due to MgO addition. According to the basic principle of capillary pressure theory, the amount of moisture loss in drying cementitious materials is mainly controlled by the drying relative humidity, water activity and surface tension of pore solution, as well as pore size distribution (Ye and Radlińska 2016b). Given that the properties of pore solution in cementitious materials merely change considerably with organic compound incorporation such as surfactants, they are believed to remain relatively constant due to MgO addition (also supported by the thermodynamic modeling). As such, this study suggested that MgO, at least less than 5% addition, does not substantially change the pore structure of cementitious solids. This is reasonable since the pore structure of HCP is primarily controlled by the formation of porous solids, mainly C-S-H, which is however unaltered by the MgO addition.

Figure 7(c) shows the correlation between drying shrinkage and mass loss of plain and MgO-modified HCP. It can be seen that, regardless of MgO addition, there is a linear correlation between drying shrinkage and moisture loss, as previously reported (Hansen 1987; Ye and Radlińska 2016c; Ye et al. 2017a; Ye et al. 2017b). However, the slope of drying shrinkage with respect to mass loss is dependent on the amount of MgO addition. In particular, it can be seen that the slope tends to decline with the increase of MgO content. Since the amount of moisture loss is directly related to the degree of saturation in a given porous media which is an important factor influencing the magnitude of effective capillary stress (Coussy et al. 2004; Vlahinić et al. 2009), it suggests that the MgO addition can enhance the stiffness (e.g., modulus of elasticity) of the HCP, probably due to Mg(OH)₂ crystal inclusion reinforcement. It should be noted that this argument is based on the assumption that drying-induced capillary stress is the dominant volume change driving force, while the crystallization stress is negligible.

4.4 Carbonation shrinkage

In Fig. 7(d), it can be seen that, in comparison to the one dried in nitrogen, carbonation results in shrinkage in HCP regardless of MgO addition, i.e., carbonation shrinkage (Ye et al. 2017b). The mechanism of carbonation shrinkage in HCP can be attributed to the dissolution of portlandite (Powers 1900; Ye et al. 2017b). In addition, drying in the air results in much less mass loss, in comparison to that dried in the nitrogen. This observation is consistent with the previous finding and could be explained by the mass gain due to carbonation-induced phase and microstructural changes (Ye et al. 2017b). Furthermore, it can be seen that after the specimens reach drying equilibrium, the following carbonation does induce additional volume change. However, in contrast to the dramatic evolution of mass change, the carbonation-induced length change is marginal. By comparing the carbonation shrinkage of MgO-modified HCP after drying equilibrium (see Fig. 7(d)), it can be seen that the MgO addition reduces the carbonation shrinkage by about 40%. It should be noted that the magnitude of carbonation shrinkage is dependent on the carbonation depth and type of formed carbonate products.

In Fig. 8, the carbonated HCP can be roughly categorized into three zones, namely, carbonated zone, par-
tially-carbonated zone, and non-carbonated zone. The carbonated zone is the outermost zone, which is highly depleted in calcium and shows a significant increase in porosity. The significant increase in porosity and darkest level in BSE images can be related to the extensive dissolution of portlandite and decalcification of C-S-H (Johannesson and Utgenannt 2001; Kutchko et al. 2007). The partially-carbonated zone shows a slight reduction in porosity, due to the precipitation of calcium carbonates in the pores (Kutchko et al. 2007). It can be seen that, in comparison to plain HCP, the one with 5% MgO addition shows a thinner carbonation depth and hence a slower carbonation kinetics. The XRD analysis, shown in Fig. 9, indicates that the carbonation of HCP results in extensive decomposition of AFt and portlandite, but the formation of various types of calcium calcites (i.e., calcite, aragonite, vaterite) and alkali carbonates. The precipitated alkali carbonates are likely the result of carbonation of pore solution. For the HCP with 5% MgO addition, magnesium-containing calcium carbonates are also detected. As discussed before, the hydration of MgO involves the liberation of Mg$^{2+}$ in the pore solution. As such, the presence of Mg$^{2+}$ in the pore solution can potentially increase the carbonate-uptaking capacity of cement paste, through the formation of Mg carbonates and/or Ca-Mg carbonates. The reduction of carbonation depth in MgO-modified HCP can thus be related to the enhanced carbonate-uptaking capacity of the Mg-enriched cementitious matrix, under the condition that the pore structure keeps relatively unchanged due to MgO incorporation.

5. Conclusions

In this paper, the effect of the MgO-based additive on the phase assemblage, microstructure, and drying and carbonation shrinkage of HCP is reported. Based on this study, the following conclusions can be drawn:

1. The MgO-based additive does not substantially change the hydrated phase assemblage in HCP, except the formation of brucite. The observation of brucite, rather than hydrotalcite (as predicted by the thermodynamic equilibrium calculation) indicates the highly localized brucite formation at and near the original MgO sites.

2. The 5% MgO addition reduces the shrinkage magnitude of HCP by about half, which can be attributed to the mechanisms of both early-age expansion and stiffness enhancement.

3. The MgO addition reduces the carbonation depth of HCP, which is attributed to the presence of Mg$^{2+}$ in pore solution and formation of magnesium-containing calcium carbonates.

4. The MgO-based additive reduces the carbonation shrinkage after the HCP being dried to equilibrium. However, this effect is not conspicuously observed under the circumstance of simultaneous drying and carbonation.

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