Performance of Portland Cement Mortar incorporated with Reactive Magnesium Oxide

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Abstract: The present paper aimed at studying the effect of reactive magnesium oxide (MgO) on the physico-mechanical properties of cement mortar (CM). Reactive magnesium oxide was prepared by calcination of hydromagnesite at 550ºC, namely MgO550. 2.5, 5, 10 and 15 wt., % MgO550 have been added to CM. Different experimental methods such as linear expansion, water absorption, and compressive strength, were carried out to evaluate the physico-mechanical properties of CM-MgO blends. The characterization of hydration products formed a long cement matrix was done by using x-ray diffractograms (XRD), Thermogravimetric (TG/DTG) and scanning electron microscope (SEM) techniques. The results showed the CM watery consistency increased with the increase of MgO550 content. The water absorption decreases with MgO550 wt., % up to 5 wt., %. The addition of MgO550 beyond 5 wt., % leads to increase the water absorption. Compressive strength enhancement was observed in case of CM having 2.5 and 5 wt., % MgO550. The CM containing 10 and 15 wt., % MgO550 showed the lowest compressive strength. The linear expansion of CM increases with the increase of MgO550 content.

Key words: Reactive MgO, Cement mortar, Compressive strength, linear expansion, Magnesium hydroxide.

1. Introduction
Cement is obtained by pulverizing clinkers formed by calcinations of raw materials primarily consisting of lime CaO, Silicate SiO2, Alumina Al2O3 and Iron Oxide Fe2O2. The basic raw materials used in the manufacture of Portland cement are Argillaceous and calcareous substances. Calcareous materials are Limestone, Chalk, Marine shells and argillaceous materials are Shale and Clay, Blast furnace slag. Raw materials supply the basic oxides of calcium, Silica, Alumina and Iron Oxide. The ordinary Portland cement (OPC) presents highest mechanical properties [1]-[5]. Meanwhile, it has low resistivity against acids and aggressive media [6]-[8].There are different supplementary materials were added to improve the mechanical properties and resistivity of Portland cement to aggressive media [9]-[17]. The workability of (OPC) was enhanced using different plasticizer [18]-[20]. In this study, different experimental methods were carried out to evaluate the performance of cement mortar incorporated with reactive MgO.

2. Experimental
2.1. Materials
The materials used in this investigation were ordinary Portland cement (OPC), reactive magnesium oxide (MgO) and sand. OPC is produced by Suez Company, Suez governorate, Egypt. MgO produced by calcination of hydromagnesite (HM), which purchased from El-Arabiya Chemical Company. Sand was supplied from El-Wasta City, Beni Suif Governorate, Egypt.

The chemical compositions of starting materials were given in Table (1). The mineralogical composition of Hydromagnesite and MgO is shown in XRD-pattern (Fig. 1). Fig. (2) shows particle distribution of OPC and HM. It is clear that the main HM particle size is 10 µm. In contrast, the mode size of OPC is 60 µm. The details of mix composition are given in Table (2).

2.2. Procedures
2.2.1. Preparation of reactive MgO
Hydromagnesite was calcined at 550 and to produce MgO, namely MgO550. The starting calcination temperature (550°C) was chosen according to differential thermal analysis (DTA) (Fig. 3). It is shown that the endothermic peak located in the range of 60-280 °C is related to the dehydration and dihydroxylation. The carbonate is decomposed at 425-486°C (Frost et al. 2008).

2.2.2. Preparation of mortar cement mixes
The MgO was first dry-mixed with cement mortar for 2 min in a bench-top mixer to achieve homogeneity then adding mixing water. The water to cement ratio (W/C) increases with the increase of MgO550 and MgO1250 content. A crater was done in the center and water was poured into the crater. The mixing operation was then completed by continuous
and vigorous mixing using ordinary gauging trowel for about three min., then cast into the cubic molds (25×25×25mm) or prism (25×25×285mm) in two layers and manually-vibrated to eliminate air voids. The samples were then covered to avoid moisture loss. After 24h of curing in 99±1 relative humidity (RH), the specimens were carefully demoulded and cured in Tap water up to 180 days.

2.2.3 Methods of investigation

The water absorption measurements were done by weighting the saturated specimens (W1) and dried specimens in oven at 80 °C for 24 hours (W2) at curing times of 3, 7, 28, 90 and 180 days. The water absorption is calculated from the following equation:

Water absorption % = [(W1-W2)/ W1] x100

Thermogravimetric analysis (TGA) was carried out by heating the sample in nitrogen atmosphere up to 1000°C with a heating rate of 20°C/min using a DT-50 Thermal Analyzer (Schimadzu Co-Kyoto, Japan). The scanning electron microphotographs were obtained with Inspect S (FEI Company, Holland) equipped with an energy dispersive X-ray analyzer (EDXA).

3. Results and Discussion

3.1. X-ray diffractograms

Figure (4) shows the X-ray-diffractograms of control cement pastes without addition of MgO550 at 3, 28 and 180 days. The XRD patterns exhibit different crystalline peaks related to portlandites Ca(OH)2 (CH), CSH, calcium carbonate (CC) and Ettringite (Af). It is obvious that, the intensity of crystalline peaks related to CH and CSH increase with time up to 180 days of curing. This is related to the interaction between sulfate ions present in cement and CH, forming gypsum and in the presence of C3A, the Af has been formed.

Figure (5) represents the X-ray diffractograms of cement pastes containing 10 wt., % MgO550 (as addition) at 3, 28 and 180 days of hydration. It is shown that, the peak characteristics for MgO at 20 of 42.9° are clearly observable at 3 days of curing. With the increase of curing time, the intensity of this peak decreases and disappeared at 180 days. This confirms the consumption of MgO as results of Mg(OH)2 and Mg-SH formation. Mg(OH)2 and Mg-SH are difficult to be detected by XRD, due to their amorphiticity characters.

Figure (6) illustrates the X-ray diffractograms of cement pastes containing 0, 5, 10 and 15 wt., % MgO550 (as addition) at 180 days of curing. The intensity of crystalline peak related to C3S and C2S decrease with the increase of MgO550 content. This is due to the cationic exchange between Mg2+ and Ca2+ in these phases forming M-S-H and CH. This can be observed from the increase of the CH peak intensity with the increase of MgO550 content, especially in case of M5-550/A mix. The MgO peaks does not observed in case of M5-550/A and M10-550/A mixes, indicating that the MgO550 totally consumed up to 180 days of curing. Meanwhile, this peak is observed in case of M15-550/A mix, indicating that there is unreacted MgO still retained in cement matrix.

3.2. TG/DTG-thermograms

Figure 7 (a and b) represents the TG and DTG-thermograms of control cement pastes at 3, 28 and 180 days of curing. It is obvious that, the weight loss of cement pastes increase with curing time. This is due to continuous hydration and formation of excessive hydration products content. This can be proved by the increase of CSH, CH and CC peaks intensity with time. The increase of CSH peak intensity reflects the development of compressive strength with the increase of curing time.

Figure 8 (a and b) shows the TG and DTG thermograms of M10-550/A mix at 3, 28 and 180 days of curing. The TG curves show that the weight loss of cement paste increases with curing time, suggesting, the successive hydration product formation with the time. The DTG curves exhibit four different endothermic peaks related to CSH, M-S-H, Mg(OH)2, CH and CC. The increase of Mg(OH)2 intensity peak with the time proves the continuous hydration of MgO550. The increase in CC peak intensity with time is mainly due to the atmospheric carbonation of MH and CH.

Figure 9 (a and b) shows the TG and DTG-thermograms of M0, M5-550/A, M10-550/A and M15-550/A at 180 days of hydration. It is clear that there is a marginal increase in weight loss of cement paste with the increase of MgO550 addition. The DTG curves showed that, Mg(OH)2 peaks intensity increase with the increase of MgO550 addition. The CSH peak intensity decreases with the increase of MgO550, especially in M10-550/A and M15-550/A mixes. This is due to the dilution of cement content.

3.3. Linear Expansion

Figure (10) represents the linear expansion of CM containing 0, 5, 10 and 15 wt., % MgO550 up to 28
days of curing in Tapwater at 23±2°C. Evidently, the linear expansion of CM increases with MgO content. This is attributed to the continuous MgO hydration.

3.4. Water absorption

Figure (11) shows the water absorption of CM containing 0 (M0), 2.5 (M2.5-550/A), 5 (M5-550/A), 10 (M10-550/A) and 15 (M15-550/A) wt., % MgO as addition. Obviously, the water absorption values of CM containing 2.5 and 5 wt., % MgO are lower than those of control CM, suggesting the densification occurred by finely powdered MgO which fills open pores. Increasing MgO addition leads to increase the water absorption of CM up to 180 days. This may be due to the dilution of cement content, leading to the formation of little hydration products content.

3.5. Compressive strength

Figure (12) illustrate the compressive strength values of M0, M2.5-550/A, M5-550/A, M10-550/A and M15-550/A mixes. Clearly, the addition of 2.5 and 5 wt., % MgO leads to increase the compressive strength of CM. This may be explained by the densification of CM matrix caused by MgO with high fineness. A significant reduction in compressive strength was observed in case of M10-550/A and M15-550/A mixes. This should be explained by the dilution of cement content as well as the formation of high magnesium silicate hydrate (MSH) with low binding capacity compared to calcium silicate hydrate (CSH). Moreover, the high MgO550 addition leads to the formation of high Mg(OH)2 content which acts as destructive agent for the binding capacity of CM.

3.6. SEM photographs.

Figure (13) displays the SEM-photographs of M0 and M5-550/A at 3 and 180 days as well as M10-550/A and M15-550/A at 180 days of curing. The SEM-photos exhibits different microstructures. The SEM of M0 at 3 days seems to be less compact, containing small aggregates distributing along the surface of microstructure. With time increasing, the microstructure becomes denser. This proves the continuous hydration and formation of hydrated products with time. The SEM of M5-550/A mix seems to be more compact microstructure comparing with M0 mix at 3 and 180 days. This confirms the compressive strength results. As the amount of MgO increases the amount of unreacted MgO increases, confirming the XRD results. In addition, M10-550/A and M15-550/A microstructures show the lowest compaction, confirming the fact that the increases of MgO with 10 and 15 wt., % leads to the dilution of cement content.

Table (1): Chemical compositions of OPC and hydromagnesite.

<table>
<thead>
<tr>
<th>Oxide, %</th>
<th>SiO2</th>
<th>Al2O3</th>
<th>Fe2O3</th>
<th>CaO</th>
<th>MgO</th>
<th>Na2O</th>
<th>K2O</th>
<th>SO3</th>
<th>TiO2</th>
<th>P2O5</th>
<th>L.O.I</th>
<th>Total</th>
</tr>
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<tbody>
<tr>
<td>OPC</td>
<td>20.5</td>
<td>4.86</td>
<td>3.72</td>
<td>62.8</td>
<td>2.35</td>
<td>0.12</td>
<td>0.08</td>
<td>2.14</td>
<td>0.04</td>
<td>0.05</td>
<td>2.35</td>
<td>99.92</td>
</tr>
<tr>
<td>Hydromagnesite</td>
<td>0.09</td>
<td>0.08</td>
<td>0.01</td>
<td>0.4</td>
<td>42.95</td>
<td>-</td>
<td>0.18</td>
<td>0.04</td>
<td>-</td>
<td>0.03</td>
<td>56.19</td>
<td>99.94</td>
</tr>
</tbody>
</table>

Table (2): Mix composition of cement mortar with MgO550.

<table>
<thead>
<tr>
<th>Mix symbol</th>
<th>MgO wt. %</th>
<th>Cement, wt. %</th>
<th>Water/ Cement (W/c) ratio</th>
<th>Calcination temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>M0</td>
<td>0</td>
<td>100</td>
<td>0.40</td>
<td>-</td>
</tr>
<tr>
<td>M5-550</td>
<td>5</td>
<td>100</td>
<td>0.40</td>
<td>550</td>
</tr>
<tr>
<td>M10-550</td>
<td>10</td>
<td>100</td>
<td>0.40</td>
<td>550</td>
</tr>
<tr>
<td>M15-550</td>
<td>15</td>
<td>100</td>
<td>0.40</td>
<td>550</td>
</tr>
</tbody>
</table>

Fig. (1): XRD-pattern of hydromagnesite (MH) and MgO.
Fig. (2): Particle size distribution of OPC, HM, MgO$_{550}$ and MgO$_{1250}$

Fig. (3): DTA-thermogram of hydromagnesite (MH)

Fig. (4): XRD-patterns of cement pastes without addition of MgO$_{550}$ at 3, 28 and 180 days
Fig. (5): XRD-patterns of cement pastes containing 10 wt. % MgO as addition at 3, 28 and 180 days.

Fig. (6): XRD patterns of cement pastes containing 0, 5, 10 and 15 % wt. % MgO (as addition) at 180 days

Fig. (7): (a) TG and (b) DTG-thermograms of M0 mix at 3, 28 and 180 days
Fig. (8): (a) TG and (b) DTG-thermograms of M10-550/A at 3, 28 and 180 days

Fig. (9): (a) TG and (b) DTG-thermograms of M0, M5-550/A, M10-550/A and M15-550/A mixes at 180 days

Fig. (10): Linear expansion of M0, M5-550/A, M10-550/A and M15-550/A mixes up to 28 day
Fig. (11): Water absorption of CM with or without MgO550 (as addition)

Fig. (12): Compressive strength of CM with or without MgO550 (as addition)

Fig. (13): SEM-photographs of M0 and M5-550/A at 3 and 180 days as well as M10-550/A and M15-550/A at 180 days
4. Conclusion
Several findings can be concluded as follows:
Compressive strength enhancement and water absorption reduction were observed in CM containing 2.5 and 5 wt. % MgO550. The addition of MgO550 beyond 5 wt. % leads to increase of water absorption as well as decrease compressive strength. The linear expansion of CM increase with MgO550 content.

The XRD-patterns and TG/DTG- thermograms proved that the rate of MgO550 hydration increase with time, forming excessive Mg(OH)2 content. The SEM photographs confirmed that the densification effect of MgO550 with 2.5 and 5 wt. % which fills open pores of CM, yielding high compact microstructure.

References
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