Effect of grain size of MgO powder on the physico-mechanical properties of magnesium oxysulfate cement paste

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Abstract

The effect of MgO powder on the water demand, bulk density, water absorption and compressive strength of magnesium oxysulfate cement paste as a function of MgO grain sizes has been investigated. In this study three mixes were used. They are composed from an Egyptian MgO powder of a grain size < 90 µm with different contents of $MgSO_4$ (16.66, 21.10 and 28.60 m%). The results of compressive strength of cement pastes cured in air at room temperature for 7 days noted that the mix containing 16.66 m% of $MgSO_4$ gives the best compressive strength. The other three mixes (M1, M2 and M3) were prepared with a constant percentage of $MgSO_4$ (16.66 m%) and three different grain sizes of MgO; normal (as received), < 300 µm and < 150 µm. The water demand, bulk density, water absorption and compressive strength of hardened magnesium oxysulfate cement pastes cured in air at room temperature for 3, 7, and 28 days were studied. Compressive strength retention coefficient was calculated. The result showed that as the grain size decreases the compressive strength of hardened magnesium oxysulfate cement pastes increases.

Keywords: grain size, oxysulfate cement, magnesium cement

1. Introduction

Magnesium oxide, or calcined magnesia, is normally obtained by calcination of magnesite ($MgCO_3$) at a temperature around 750 °C. The quality or reactivity of the formed magnesium oxide powder is largely affected by its thermal history (calcination temperature and firing duration) and particle size [1].

Cements made from magnesia have been developed many years ago, such as magnesia oxychloride cement (MOC), magnesia oxysulphate cement (MOS), and magnesia phosphate cement (MPC) [2].

A number of cements based on magnesia have been previously studied. If a salt such as magnesium chloride or sulfate is added to reactive magnesia and the mixture is allowed to react, hydrated magnesium oxychlorides and magnesium oxysulfates are formed. These hydrates can be very strong but are not sufficiently weatherproof as well as corrosive. Magnesium oxysulfates were discovered by *Olmer* and *Delyon* in 1934 [2].

Zinc, calcium, copper and other elements also form similar compounds. Magnesium oxychlorides (*Sorel* cements) achieve higher compressive strengths than magnesium oxysulfates. The main problem with *Sorel* cements is that both magnesium oxychlorides and magnesium oxysulfates tend to break down in water and particularly in acids. Corrosion of steel reinforcement also occurs [3].

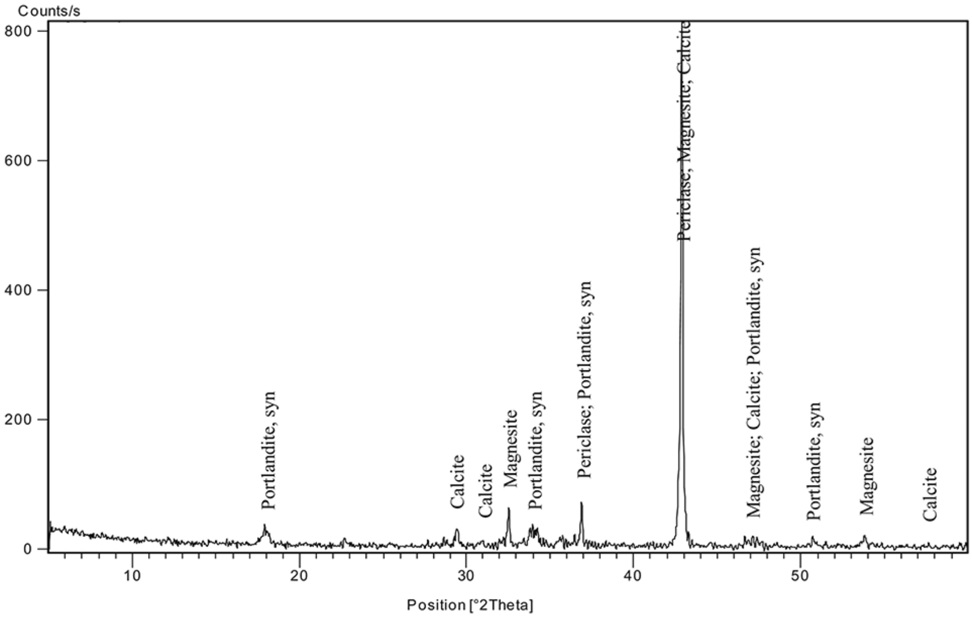
Magnesia (MgO) is mostly produced by calcination of magnesite ($MgCO_3$). Depending on the thermal conditions, caustic magnesia or dead-burned magnesia is obtained. Caustic magnesia, a comparatively more porous and reactive material obtained at relatively low temperatures, has many industrial applications, such as in agriculture, cattle feed, environmental control, manufacture of special cements and in other special uses. Dead-burned magnesia (MgO), produced at high temperatures, is mainly utilized in the refractory industry. The rate and extent of hydration are factors that affect the functional performance of both types of magnesia [4].

This work aims to study the effect of grain sizes of MgO powder on the physico-mechanical properties such as bulk density, water absorption and compressive strength of hardened magnesium oxysulfate cement pastes as a function of curing time up to 28 days.

2. Materials and methods

Magnesite as a raw material is extracted from the southern Eastern Desert of Egypt. The calcination was done by a traditional method of firing in a mazut furnace at the industrial area at Helwan Governate. The raw material was fired for about 24 h and left inside the furnace until reaching ambient temperature then ground to a certain grain size. In this work it was considered that the produced grain size of MgO powder after grinding and collected in special sacks is the normal grain size. The MgO was characterized with respect to the mineralogical composition by using X-ray diffraction technique, see *Fig. 1*. The MgO sample composes mainly of 56% periclase, 40% magnesite and traces of $CaCO_3$ and $Ca(OH)_2$ each 2%. Particle size analysis of the MgO was carried out by using dry sieve analysis following the Egyptian standards (ESS) [5] as illustrated in *Fig. 2*.

Three mixes (M-I, M-II and M-III) were prepared by mixing MgO powder with grain size less than 90 µm and three percentages of $MgSO_4$ 16.66, 21.10 and 28.60 m%. Compressive strength results of the hardened pastes of the three mixes cured in air for 7 days are plotted in *Fig. 3*. The results indicated that the mix which containing 16.66 m% $MgSO_4$ (M-I) gives higher value of compressive strength than the other two mixes (M-II and M-III). This means that the ratio of 16.66 m% for $MgSO_4$ is



Chemical Formula	Mineral Name	Semi Quant [%]
MgO	Periclase	56
$MgCO_3$	Magnesite	40
$CaCO_3$	Calcite	2
$Ca(OH)_2$	Portlandite	2

Fig. 1. X-ray diffraction pattern of MgO powder
1. ábra MgO por röntgendiffraktogramja

the most practical one. As the amount of $MgSO_4$ increases the residual or un-reacted $MgSO_4$ increases which decreases the compressive strength. Therefore, the amount of magnesium oxysulfate decreases due to the increase of $MgSO_4$ or the cement paste content decreases which affects the mechanical properties or compressive strength.

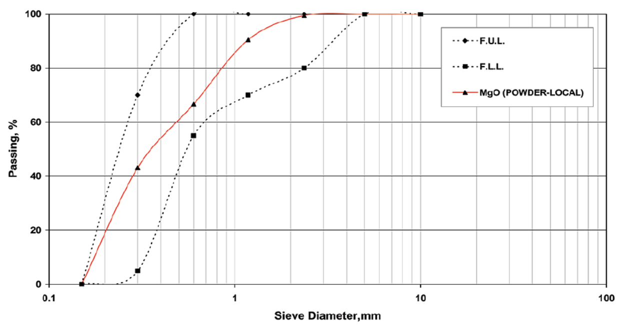


Fig. 2. Grain size distribution (dry sieve analysis) of normal MgO powder
2. ábra Szemcseméret-eloszlás (száraz szitavizsgálat) eredménye normál MgO porra vonatkozóan

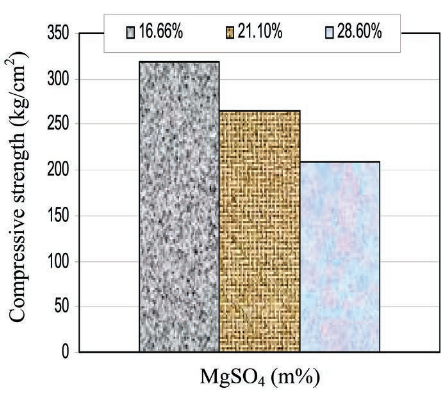


Fig. 3. Dry compressive strength of mixes M-I, M-II and M-III after curing in air for 7 days
3. ábra Száraz nyomószilárdság értékek az M-I, M-II és M-III keverékekre, levegőn történő 7 napos tárolás után

Continuing the work processing, another three mixes (M1, M2 and M3) were prepared using $MgSO_4$ at the above recommended value (16.66 m%) and MgO powder (83.34 m%) with three different grain sizes: 1) grain size less than 3 mm and larger than 500 µm, 2) grain size less than 300 µm and 3) grain size less than 150 µm. The two latter grain sizes of MgO powder were prepared by using sieve diameters of 300 and 150 µm to produce two samples of MgO powder with grain size less than 300 µm and less than 150 µm, respectively. The suggested three mixes are presented in *Table 1*.

Mixture No.	Grain size of MgO powder	Composition (m%)	
		MgO powder	$MgSO_4$
M1	Less than 3 mm and larger than 500 µm	83.34	16.66
M2	less than 300 µm	83.34	16.66
M3	less than 150 µm	83.34	16.66

Table 1. Suggested mixtures of the material used
1. táblázat Vizsgált keverékek összetétele

The grain size analysis of the normal MgO powder is graphically represented in *Fig. 2*. It is noted that the MgO powder has grain size larger than 500 µm and less than 3 mm. Cubic specimens of 5 cm side length were cast in steel moulds using vibration compaction. The physical and mechanical properties were determined after curing of hardened magnesium oxysulfate cement pastes at room temperature in air for 3, 7 and 28 days. The water demand of each cement paste was determined according to ASTM [6] and bulk density [7] as well as dry compressive strength and wet compressive strength cured under tap water for 24 hours [8] of blended cement pastes up to 28 days were investigated.

3. Results and discussion

3.1 Water demand

The water demands for the three mixes (M1, M2, and M3) are 25, 26, and 27.5 m%, respectively. The results show that as the grain size decreases the water demand increases. This may be related to that the grain size of MgO powder decreases the specific surface area of MgO powder and consequently the water demand increases. Each mix was mixed with the water demand for studying the physico-mechanical properties such as compressive strength, water absorption and bulk density of cement pastes up to 28 days.

3.2 Bulk density

The bulk density of hardened magnesium oxysulfate cement pastes (M1, M2, and M3) cured in air at room temperature for 28 days was found to be 1.8, 1.86 and 2.0, respectively. The results show that as the grain size decreases the bulk density increases. This may be due the decrease of grain size of MgO powder leads to more compact structure with lower porosity and increase the rate of hydration. The larger specific surface area of any material accelerates the rate of reaction.

3.3 Water absorption

The water absorption of the magnesium oxysulfate cement pastes (M1, M2, and M3) cured in air at room temperature for 28 days was 6.8, 6.6 and 3.96 m%, respectively. The results show that as the grain size decreases the water absorption decreases. This may be due to that as grain size of MgO powder decreases a more compact structure with low porosity is produced due to the increase of the reaction forming hydration products which deposit in the open pores of the cement pastes.

3.4 Dry compressive strength

The results of dry compressive strength of the cured specimens of the different mixes (M1, M2, and M3) after 3, 7 and 28 days at room temperature in fresh air are plotted in *Fig. 4*. It is clear to observe that the hardened magnesium oxysulfate cement paste (M1) possesses the lowest compressive strength value. The mix M3 which contains the MgO powder with grain size less than 150 μm has compressive strength values higher than Mixes M1 (normal grain size of MgO) and M2 (MgO powder of grain size less than 300 μm) at all curing times. As the grain size decreases the compressive strength increases. This is in a good agreement with the results of bulk density as well as water absorption. Also, the decrease of grain size of MgO powder tends to increase the rate of hydration as well as the formation of magnesium oxysulfate. This is the cement matrix which gives the physico-mechanical properties. Also, the results show that a gradual increase of compressive strength values with curing time is visible for all samples. This is due to the higher degree of formation of hydration products which are the main binding agent of the cement paste. In other words, the reaction goes as a function of curing time. The results indicated that the compressive strength increases as grain size decreases. This may be due to the increase in specific surface area as grain size of MgO powder decreases, which leads to an increase of the amount of active crystalline products, consequently their accumulation closes the available pores and forms a more compact structure that results an increase in the compressive strength of hardened magnesium oxysulfate cement pastes.

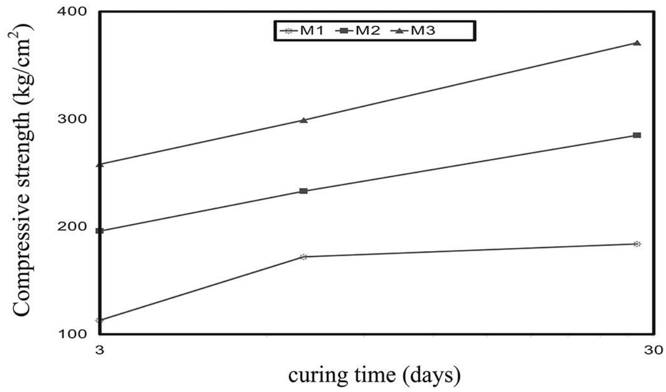


Fig. 4. *Dry compressive strength of mixes M1, M2, and M3 after curing in air at room temperature for 3, 7 and 28 days*
4. ábra *Száraz nyomószilárdság értékek az M1, M2 és M3 keverékekre, levegőn, szobahőmérsékleten történő 3, 7 és 28 napos tárolás után*

3.5 Wet compressive strength

The results of wet compressive strength of cured specimens of the different mixes (M1, M2, and M3) immersed in tap water for 24 hours after 3, 7 and 28 days at room temperature are plotted in *Fig. 5*. It is clear that the mix M1 which contains the MgO powder of the control grain size has the lowest wet compressive strength. The Mix M3 with the MgO powder of grain size less than 150 μm has higher wet compressive strength values than mixes M1 (normal grain size of MgO) and M2 (MgO powder with grain size less than 300 μm) at all curing times. This is due to the increase of water absorption as the grain size decreases. As it was indicated above, the water absorption was 6.8, 6.6 and 3.96 m% for mixes M1, M2, and M3, respectively. This result shows that as the grain size of MgO powder decreases the water absorption decreases and consequently the wet compressive strength increases. Generally, the dry compressive strength is higher than the wet compressive strength.

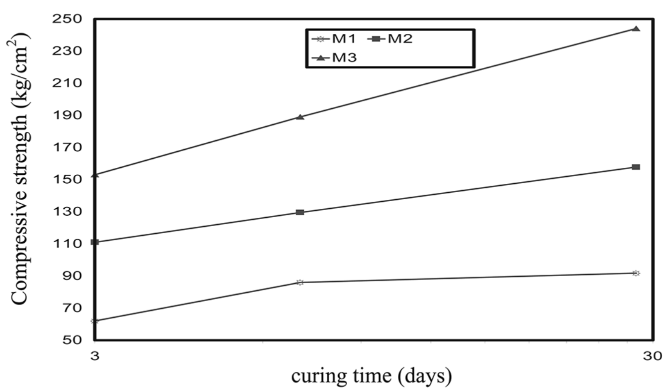


Fig. 5. *Wet compressive strength of mixes M1, M2 and M3 after curing in air at room temperature for 3, 7 and 28 days*
5. ábra *Nedves nyomószilárdság értékek az M1, M2 és M3 keverékekre, levegőn, szobahőmérsékleten történő 3, 7 és 28 napos tárolás után*

From the result of dry and wet compressive strength of the three mixes M1, M2, and M3, the strength retention coefficients

(= wet compressive strength / dry compressive strength) of specimens of magnesium oxysulfate cement was calculated [9] and graphically represented in *Fig. 5*. It is clear that the value of the strength retention coefficients of mixes increases as the grain size of MgO powder decreases which completely in agreement with the results obtained from the dry and wet compressive strength properties.

This may be due to that more active products proceed as a result of decreasing the grain size of MgO powder and their accumulation closes the available pores volume forming a more compact structure that decreases the water absorption and consequently decreases partial the solubility as well as increases the water resistance of magnesium oxysulfate cement.

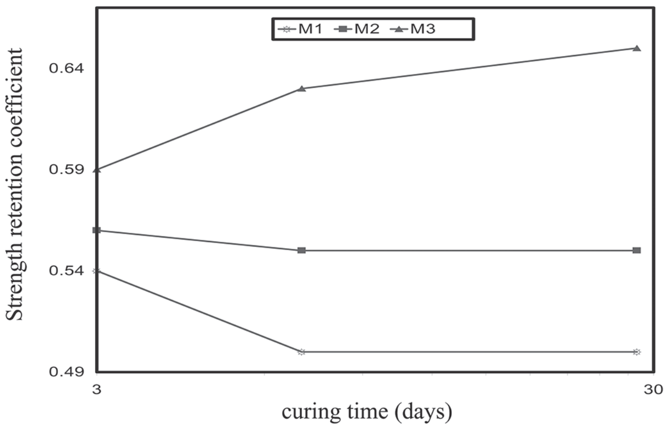


Fig. 6. Strength retention coefficients (%) of mixes M1, M2 and M3 at 3, 7, 28 days
6. ábra Vízlágyulási tényezők (%) az M1, M2 és M3 keverékekhez 3, 7, 28 napos korban

4. Conclusions

The main conclusions of this work can be drawn as follows:

1. As the grain size of MgO decreases the water demand increases.
2. As grain size of MgO decreases the dry and wet compressive strength of hardened magnesium oxysulfate cement paste increases by curing time.
3. The decrease of grain size of MgO powder produces high values of strength retention coefficients and improves the water resistance of magnesium oxysulfate cement paste.

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MgO por szemcseméretének hatása oxiszulfát cement pépek fizikai, mechanikai jellemzőire

A MgO szemnagyságának a hatását mutatja be a cikk az oxiszulfát cementek vízigényére, halmazsűrűségére, vízfelvételére és nyomószilárdságára, különböző mennyiségű magnéziumszulfát adagolás mellett. A nyomószilárdság vizsgálatok eredményei szerint a 7 napig nedves utókezeléssel tárolt cementhabarcs minták közül a 16.66 m% magnéziumszulfátot tartalmazónak a legkedvezőbbek a tulajdonságai. A vizsgálatok eredményei szerint a MgO szemnagyságának csökkentésével a megszilárdult cementpépek nyomószilárdsága nő.
Kulcsszavak: szemcseméret, oxiszulfát cement, magnézia cement