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ANALYZED

STRENGTH DEVELOPMENT IN MAGNESIUM OXYCHLORIDE AND OTHER CEMENTS

CANADA INSTITUTE FOR S.T.I.

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OTTAWA

INSTITUT CANADIEN DE L'I.S.T.

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STRENGTH DEVELOPMENT IN MAGNESIUM OXYCHLORIDE AND OTHER CEMENTS

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ABSTRACT

Measurements of compressive strength, modulus of elasticity, and microhardness were made on magnesium oxychloride cement systems having a wide range of porosity. Analysis of the plots of log mechanical property versus porosity gave expressions interrelating mechanical properties which were independent of porosity. Comparison of these results with those of the portland cement system show that magnesium oxychloride cement paste has a higher value of modulus of elasticity and microhardness over the porosity range studied. Microhardness of magnesium oxychloride cement paste is also greater than that for all gypsum plaster preparations in the porosity range studied. Mechanical behaviour of magnesium oxychloride cement compacts is improved when heated in water at 85 °C. It appears that this heat treatment results in formation of a porous body consisting mainly of $Mg(OH)_2$. Among the systems magnesium oxychloride, portland cement, gypsum and magnesium hydroxide, it appears that magnesium hydroxide forms the strongest bodies.

On mesure la résistance à la compression, le module d'élasticité et la microdureté de ciments d'oxychlorure de magnésium de porosité très variée. Une analyse des propriétés mécaniques logarithmiques et des diagrammes de porosité produit des expressions qui relient entre elles des propriétés mécaniques indépendantes de la porosité. Une comparaison de ces résultats et de ceux du ciment portland montre que la pâte de ciment d'oxychlorure de magnésium a un module d'élasticité et une microdureté plus élevés pour les variations de porosité étudiées. La microdureté de la pâte de ciment d'oxychlorure de magnésium est également plus élevée que celle de toutes les préparations de plâtre de gypse pour les variations de porosité étudiées. Lorsque des comprimés de ciment d'oxychlorure de magnésium sont chauffés dans de l'eau à 85°C, leur comportement mécanique est amélioré. Il semble que ce traitement thermique produise un corps poreux comportant surtout du $Mg(OH)_2$. Il semble que l'hydroxyde de magnésium produise des corps plus résistants que l'oxychlorure de magnésium, le ciment portland ou le gypse.

Magnesium oxychloride cement, also known as Sorel cement, was discovered by Sorel in 1867 (1). This cement is formed by mixing finely divided magnesium oxide with an aqueous solution of magnesium chloride. Magnesium oxychloride cement possesses many properties superior to those of portland cement. It has high fire resistance, low thermal conductivity, high resistance to abrasion, and high compressive and flexural strengths. Many organic and inorganic aggregates, which may not be suitable for making portland cement concrete, can be used in combination with the oxychloride cement.

Depending on the conditions of preparation, four types of oxychloride complexes are known to form in the $MgO - MgCl_2 - H_2O$ system. They are $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (5-form)*, $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (3-form)*, $2Mg(OH)_2 \cdot MgCl_2 \cdot 5H_2O$ (2-form)* and $9Mg(OH)_2 \cdot MgCl_2 \cdot 6H_2O$ (9-form)* (2). Continuous exposure of the oxychloride to air results in the formation of magnesium chlorocarbonate of formula $Mg(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$.

The use of oxychloride cement is not so widespread as it might be because it loses strength on prolonged exposure to water. The stabilization of oxychloride against attack by water is therefore a matter of importance and an understanding of the mechanism of strength development for the oxychloride system is necessary. The use of compacted systems has led to some success in understanding the bonding mechanism in portland cement and gypsum pastes (3, 4, 5, 6). This technique was applied to oxychloride cements to investigate the probable bonding mechanism that occurs and to compare it with the bonding mechanisms of other cementitious systems as portland cement and gypsum pastes.

Experimental

Materials

The following materials were used:

1. Magnesium Oxide. The MgO powder was supplied by Basic Chemicals, Cleveland, Ohio. It had the following characteristics: N_2 surface area, $20m^2/g$; active CaO , 1.5%; ignition loss 4%; fraction passing through 200 mesh, 98%. The material satisfied the requirements of A.S.T.M. C-275-61.
2. Magnesium Chloride Solution. Aqueous solution of specific gravity 1.18 was prepared by mixing $MgCl_2 \cdot 6H_2O$ in a dry form with distilled water.

Measurements

Helium pycnometer was used to measure the solid volume of the cement specimens of known geometry. Porosity was determined from the apparent volume and the solid volume. This avoids the problem of dissolution that arises when water is used as the displacement medium. Samples were conditioned at 11% relative humidity for each measurement. Application of this technique to other inorganic cement systems is described elsewhere (7). Porosity was also determined using methanol displacement employing Archimedes principle.

* abbreviated designation

Modulus of elasticity was measured on discs with a diameter of 3.2 cm and a thickness of 1.3 mm. This procedure involves measuring the deflection of a specimen when it is loaded at its centre and supported at three points located on the circumference of a circle 2.5 cm in diameter (8). Each value represents an average obtained for three specimens. The same discs used for measuring the modulus of elasticity were conditioned at 50% relative humidity for microhardness determination. Microhardness was measured with a Leitz microhardness testing machine with a Vickers indenter. Each microhardness value recorded herein is an average of three discs; five microhardness measurements were made on each disc. Compressive strength of magnesium oxychloride paste samples was measured for 5.1 cm cubes.

Differential thermograms were obtained using the 990 Dupont thermal analysis system. Twenty mg of the material were used for each run. The rate of heating was 20 °C/min ($T = 150$ sec/in) and the sensitivity was $\Delta T = 5$ m cal/sec/in.

The microstructure of some of the samples was examined using a Cambridge Stereoscan Mark 2A. The examination was done on fractured surfaces.

X-Ray examinations were carried out with a Debye Scherrer Camera. Microdensitometer traces were made from the X-ray films. Surface area was obtained by means of a numinco-Orr surface area-pore volume analyser with N_2 as the adsorbate. Powdered samples were dried for 3 hours at 110 °C prior to measurements.

Chloride estimation was done using a chloride ion selective electrode according to a method described by Berman (9).

Preparation of Specimens

System I

Cement paste samples were prepared at $MgCl_2 \cdot 6H_2O/MgO$ solution-solid ratios of 0.59, 0.64, 0.71, 0.77 and 0.86 by weight. Mixes were cast in cube molds and cured at 50% relative humidity for periods of up to ten months; this was to ensure that the pastes prepared at different solution-solid ratios were almost completely hydrated.

Discs 3.2 cm in diameter, 1.3 mm thick were cut for measurements of microhardness and modulus of elasticity from 3.2 cm cores from cube samples.

System II

The cement paste with a solution-solid ratio of 0.59 (hydrated 10 months) was finely ground and used for fabrication of compacts. Compacts 3.2 cm in diameter, 1.3 mm thick were made at compaction pressures of 133 MPa to 1665 MPa, giving a wide range of porosities.

System III

System II compacts were immersed in water at 22 °C and were slowly heated for 30 min to 85 °C and maintained there for 5 hours. This treatment would dissolve $MgCl_2$ from the system.

Other Cement Systems

The preparation of portland cement pastes and hydrated plaster systems have been described in earlier works (3, 4, 5, 6).

Results

Porosity - Mechanical Property Relationships

The logarithms of modulus of elasticity, microhardness and compressive strength were plotted against porosity; the plots for all systems indicate linear relationships (Figs. 1, 2, 3).

Modulus of elasticity, microhardness and compressive strength for all magnesium oxychloride preparations studied obeyed the general relationship, $E, H, S = (E_o, H_o, S_o) \exp [(-b_{E,H,S})p]$ where E, H, S refer to modulus of elasticity, microhardness and compressive strength respectively and p is porosity. An analysis of linear regression for magnesium oxychloride and other systems is recorded in Table I. The slope of the line representing

TABLE I
Regression Analysis of Modulus of Elasticity, Microhardness and Compressive Strength Data

Preparation	$E_o^* \quad b_E$		$H_o^* \quad b_H$		$S_o^* \quad b_S$		$S_{yx}(\pm)^{**}$			r^+			$CL(\pm)^{++}$		
	E_o	b_E	H_o	b_H	S_o	b_S	E	H	S	E	H	S	E	H	S
Magnesium Oxychloride Paste (System I)	45.39	0.035	317.0	0.089	238.0	0.047	1.088	1.068	1.020	83.06	98.23	98.22	1.98	1.95	2.150
Magnesium Oxychloride Compacts (System II)	46.56	0.096	130.8	0.132	-	-	1.092	1.159	-	97.12	97.08	-	1.94	2.06	-
Portland Cement Paste	29.30	0.030	90.0	0.046	170.0	0.048	-	-	-	-	-	-	-	-	-
Portland Cement Compacts	29.30	0.030	90.0	0.046	-	-	-	-	-	-	-	-	-	-	-
Compacts of Bottle Hydrated Plaster	63.3	0.114	108.9	0.106	-	-	-	-	-	-	-	-	-	-	-
Hydrated Plaster Compacts	52.4	0.030	121.3	0.069	-	-	-	-	-	-	-	-	-	-	-
Magnesium Hydroxide Compacts	-	-	800	-	-	-	-	1.067	-	-	98.0	-	-	2.26	-

* Units for E, H, S are $\text{MPa} \times 10^{-3}$, $\text{MPa} \times 10^{-1}$ and MPa

** Standard error of estimate

† Correlation coefficient

++ 90% confidence limits

log modulus of elasticity versus porosity for the compacted System II is about three times greater than the slope of the corresponding line for the in situ hydrated paste of System I (Fig. 1). The data for System III compacts (heat treated in water at 85 °C) were included in the data population for System I. A summary of the mechanical properties for System III compacts is recorded in Table II. Heat treatment of System II compacts in water resulted in a porosity increase of 63 to 189%, microhardness increase of 18 to 64%, and modulus of elasticity increase of 64%. Length

TABLE II

Mechanical Properties of System III Compacts Treated at 85 °C in H₂O

Initial Porosity, P _i , %	Final Porosity, P _f , %	$\frac{P_f}{P_i}$	H _i (MPa x 10 ⁻¹)	H _f (MPa x 10 ⁻¹)	$\frac{H_f}{H_i}$	E _i (MPa x 10 ⁻³)	E _f (MPa x 10 ⁻³)	$\frac{E_f}{E_i}$	$\frac{\Delta L}{L}$, %
7.3	21.1	2.89	40.2	47.1	1.18	20.01	19.75	0.98	0.90
11.6	23.9	2.06	28.6	41.5	1.45	12.52	20.66	1.64	0.84
13.4	25.8	1.92	27.2	30.5	1.12	12.00	15.37	1.28	0.68
15.0	26.3	1.75	18.0	29.5	1.64	11.40	17.00	1.49	-
17.8	29.1	1.63	19.2	26.5	1.38	7.82	12.62	1.62	0.66

change due to heat treatment was between 0.66 and 0.90%. Surface area increased from 4.95 to 8.99 m²/gm.

The heat treated compacts of system III have a narrow porosity range (21.1 to 29.1%). Extrapolated data for systems I and III may not be collinear with each other (Fig. 1) over the entire porosity range. The line representing the extrapolated modulus of elasticity for system II compacts intersects that of system I at approximately zero porosity. The modulus of elasticity for in situ hydrated magnesium oxychloride paste (system I) is approximately 30 to 50% greater than that for hydrated portland cement paste over the porosity range studied. It should be noted that values of modulus of elasticity for both compacted portland

FIG. 1
Modulus of elasticity vs porosity for magnesium oxychloride and other inorganic cement systems

hydrated plaster

hardness and
plots for all systems

pressive strength for
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, S refer to modulus
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pressive Strength Data

r [†]		CL(±) ^{††}		
H	S	E	H	S
98.23	98.22	1.98	1.95	2.150
97.08	-	1.94	2.06	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
98.0	-	-	2.26	-

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cement paste and in situ hydrated portland cement paste lie on the same line (8). The line for portland cement paste intersects the line for compacted magnesium oxychloride (system II) at about 7% porosity. The line for modulus of elasticity of compacts of bottle hydrated plaster is nearly collinear with the line for compacted magnesium oxychloride (system II) and intersects the portland cement paste line at approximately 9% porosity.

The line for in situ hydrated plaster compacts (compacted first, then hydrated) lies slightly above the line for in situ hydrated magnesium oxychloride paste (system I).

Microhardness data for the magnesium oxychloride systems studied show the same trend as modulus of elasticity data (Fig. 2). Microhardness values for compacted magnesium oxychloride (system II) gradually approach those for in situ hydrated paste (system I) as porosity decreases. The slope of the line for log microhardness versus porosity for system II is nearly 50% greater than that for system I. The extrapolated lines for systems I and II do not intersect. The line for portland cement paste intersects the line for compacted magnesium oxychloride (system II) at approximately 5% porosity.

FIG. 2

Microhardness versus porosity for magnesium oxychloride and other inorganic cement systems

The line representing log microhardness versus porosity for compacts of bottle hydrated plaster intersects the extrapolated line for system II compacts at about 7% porosity. Modulus of elasticity at a given porosity is greater for hydrated plaster compacts than hydrated magnesium oxychloride paste (system I); the converse is true for microhardness.

The plot of log compressive strength versus porosity for in situ hydrated magnesium oxychloride cement paste (system I) results in a straight line (Fig. 3). Values of compressive strength for oxychloride paste are approximately 30% greater than for hydrated portland cement paste of equal porosity.

Figure 4 is the plot of microhardness versus modulus of elasticity for in situ hydrated magnesium

$$E = E_o \left(\frac{S}{S_o} \right)^{b_E/b_S} \quad (2)$$

$$H = H_o \left(\frac{S}{S_o} \right)^{b_H/b_S} \quad (3)$$

where b_E/b_H , b_E/b_S , and b_H/b_S are the ratios of the mean slope of the log mechanical property versus porosity relationships. For hydrated portland cement paste at room temperature and a variety of autoclaved cement-silica preparations, b_H/b_S approaches unity resulting in a linear relationship between microhardness and compressive strength (10).

Micrographs of the various magnesium oxychloride preparations were made and a few typical features are shown in Figs. 7a, b, c. Figure 7a illustrates the typical microstructure for all system I pastes studied. A needle-shaped microstructure is observed in isolated regions, usually in large voids. In low porosity regions the crystals appear to form a more monolithic structure as observed by Matkovic and Young (11). Unlike the needle-shaped crystals observed in system I pastes, the fractured surfaces of system II compacts consist of discrete particles of various sizes with well defined boundaries (Fig. 7b). These particles are bonded together by a more monolithic structured material than observed in system I. Figure 7c illustrates the typical heat treated system III compacts and shows an amorphous-like microstructure with a glassy and compact appearance. Experience with other cementitious systems (5, 12) indicates that this type of microstructure may give a better bond than the less compact mass in Fig. 7b.

Figure 8 gives the thermograms for systems II and III and magnesium hydroxide. The thermogram for system I is similar to that of system II

FIG. 4

Microhardness versus modulus of elasticity for magnesium oxychloride cement paste

(2)

(3)

the mean slope of the

For hydrated portland
cured cement-silica
linear relationship

side preparations were
b, c. Figure 7a illus-
trates studied. A needle-

FIG. 4

versus modulus of
for magnesium
cement paste

and is not presented. There is an indication of an endothermic effect in system II at 120 °C and medium sized endothermic peaks at about 165 °C and 225 °C. Large endothermal effects are present at about 365 °C and 420 °C. An additional endothermal effect is indicated at about 460 °C. This is clearer in the DTA trace. The peaks may be interpreted as representing the presence of magnesium hydroxide, the 3-form or 5-form or both forms of oxychloride. According to Cole and Demediuk (13) the 5-form gives a moderate endothermal effect in the range of 450 to 500 °C. The endothermal effect at about 460 °C is usually attributed to the presence of the 5-form or $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ or both. The pastes were all prepared at a high $\text{Mg}(\text{OH})_2/\text{MgCl}_2$ ratio and hence the 5-form is expected to be present. The system III compacts show only a broad valley in the range of about 75 °C to 250 °C and a large endothermic peak at about 425 °C.

FIG. 5

Modulus of elasticity versus compressive
strength for magnesium oxychloride
cement paste

Heat treated system III compacts had X-ray diffraction patterns indicating an increased amount of $\text{Mg}(\text{OH})_2$ as compared to untreated system II compacts (Fig. 9). A strong peak at $d = 7.5 \text{ \AA}$ present in system II compacts is absent in system III compacts. The $7.5 \text{ \AA}$ peak can be ascribed to the presence of the 5-form of magnesium oxychloride complex. These results indicate that heat treatment of system II results in the decomposition of the oxychloride complex.

Portland cement paste has the same mechanical property versus porosity relationship for compacted as for in situ hydrated specimens. Unlike portland cement, magnesium oxychloride cement, compacted and in situ hydrated, has separate and distinct mechanical property versus porosity curves (Figs. 1, 2). This suggests that, at any particular porosity value, the interparticle bonds formed during the hydration of oxychloride cement

cannot be remade during compaction. This is also true for various gypsum systems (3). Hydrated portland cement appears to be unique among the inorganic cement systems studied in that interparticle bonds can be remade upon compaction.

In the oxychloride cement system, increased compaction appears to enhance formation of interparticle bonds. This is obvious from the mechanical behaviour of the compacted system which approaches that of the in situ hydrated system as porosity decreases (Figs. 1, 2).

The curves for log modulus of elasticity versus porosity for the oxychloride cement and gypsum systems (in situ and compacted systems) almost converge at zero porosity. The oxychloride cement system and, to a lesser extent, the gypsum system, however, do not show this property for microhardness. It is not easy to explain this lack of convergence of microhardness at zero porosity for compacts and paste hydrated samples. Microhardness measurement is a failure process in that it involves plastic deformation as opposed to one involving elastic deformation. The presence of flaws and irregular pore geometry is therefore likely to have a greater influence on microcrack propagation during hardness measurement than during measurement of modulus of elasticity.

It should also be noted that the in situ hydrated magnesium oxychloride cement paste is a multicomponent composite probably consisting of 3- and 5-form hydrates, $Mg(OH)_2$, unhydrated MgO , $MgCl_2 \cdot 6H_2O$ and possibly other hydrates of $MgCl_2$. The degree of dispersion of the different products and the extent to which they participate in bonding is not easily understood and is difficult to explain in terms of different crystalline modifications which are not distinguishable from the micrographs. Figure 7b, however, shows the presence of discrete 2 - 5 μm particles for a compact having 18.86% porosity. The paste in Fig. 7a has approximately 18% porosity, some needle-shaped morphology, and generally seems to have a more compact microstructure. The

FIG. 6

Microhardness versus compressive strength for magnesium oxychloride cement paste

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FIG. 7a

Magnesium oxychloride
cement paste - system I

FIG. 7b

Magnesium oxychloride
compact - system II

FIG. 7c

Magnesium oxychloride
compact - system III

latter may account for the increased strength of system I at a given porosity. Microhardness in system III is higher than that in system II; the microstructure of system III presents a more compact matrix.

It was thought that treatment in water would enhance the strength development in system III due to a recrystallization effect as in gypsum (14). DSC investigations revealed that the endothermal dip for system III at about

425 °C was due only to $\text{Mg}(\text{OH})_2$ (Fig. 8). A comparison of this curve with pure $\text{Mg}(\text{OH})_2$ under similar conditions confirmed this. X-ray diffraction results of system III show that lines due to oxychloride complex were absent and that peaks due to the presence of $\text{Mg}(\text{OH})_2$ at $d = 1.80$, 2.37 and 4.75 Å all increased in intensity. Chloride estimation of system II and system III were also compared and it was seen that system III contained only about 10% of the total chloride that was contained in system II. The true density of system III was found to be 2.31 g/cc which is closer to the density of pure $\text{Mg}(\text{OH})_2$.

As shown previously, this material contained mainly $\text{Mg}(\text{OH})_2$; it would appear that the strength of system III was mainly due to the $\text{Mg}(\text{OH})_2$ component which was left after the oxychloride complex became unstable in hot water. To verify that pure $\text{Mg}(\text{OH})_2$ had similar strengths, some compacts of different porosities were made from it. Their microhardness was found to be slightly above the line representing system III (Fig. 2). Although systems I and III are of different composition, they are collinear with respect to the plot of log mechanical property versus porosity. This coincidence in the behaviour of the two systems appears to be fortuitous.

FIG. 8

Differential scanning calorimetry traces

Conclusions

For a particular porosity, in situ hydrated oxychloride cement has a mechanical behaviour superior to that of hydrated portland cement paste. Interparticle bonds are not remade during compaction of magnesium oxychloride cement which seems to have a bonding mechanism similar to that of the gypsum system. The mechanical behaviour of magnesium oxychloride cement compacts improves when treated in water at 85 °C and the surface area increases from 4.95 to 8.99 m²/g. Among the gypsum, magnesium oxychloride, portland cement, and magnesium hydroxide systems, it appears that magnesium hydroxide forms the strongest bonds in the porosity range studied.

A porosity as low as 21% is obtained in a sample of the magnesium hydroxide system by preparing a compact of powdered magnesium oxychloride paste at 550 MPa and heating in water at 85 °C. A compact with a porosity of 32% resulted when a pressure of 550 MPa was applied to a powder of $\text{Mg}(\text{OH})_2$. This would indicate that indirect methods may be successfully used if a low porosity system cannot be obtained by direct compaction. It appears that the system containing essentially $\text{Mg}(\text{OH})_2$ (system III), formed by leaching of

calorimetry traces

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FIG. 9

Densitometer traces of X-ray films

oxychloride if ground and remade into compacts, would have the same mechanical property versus porosity relation as compacts of pure Mg(OH)₂. In this respect it would be similar to hydrated portland cement.

Modulus of elasticity, microhardness, and compressive strength of magnesium oxychloride cement pastes and compacts were found to be a simple exponential function of porosity and are interrelated in the following manner:

$$E = E_0 \left(\frac{H}{H_0} \right)^{b_E/b_H}$$

$$S = S_0 \left(\frac{E}{E_0} \right)^{b_S/b_E}$$

$$H = H_0 \left(\frac{S}{S_0} \right)^{b_H/b_S}$$

The constants have physical significance in that E_0 , S_0 , and H_0 are modulus of elasticity, compressive strength and microhardness at zero porosity respectively; b_E/b_H , b_S/b_E , b_H/b_S are ratios of the slopes of the log mechanical property versus porosity function. Mechanical properties expressed in terms of porosity provide a good basis for comparison of cementitious systems.

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