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SEM Study of Magnesium Oxychloride Cement

KEYWORDS: SEM, Magnesium oxychloride cement (MOC), Gauging solution

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ABSTRACT *Magnesium oxychloride cement (MOC) was invented by S.T. Sorel in 1867. It has many properties superior to those of Portland cement. Two main bonding crystalline phases; phase F3 (3Mg(OH)2.MgCl2.8H2O) and F5 (5Mg(OH)2.MgCl2.8H2O) are detected in X-ray diffraction patterns of magnesia cement. Dolomite power was used as inert filler in present study. Dry-mixes were prepared by mixing magnesia and dolomite (inert filler) in the ratio of 1:1 by their weights respectively. Dry-composition was gauged with 20oBe 24oBe, 28oBe, 32oBe and 35oBe gauging solution MgCl2 at 30o C temperature. The SEM images were taken by applying EHT 20,000 KV voltage. The crystallographic, topography and microstructure of experimental blocks of MOC was examined using SEM investigation. The needle shaped MOC crystals were identified in MOC F5 phase.*

1. Introduction:

Magnesia cement has been attracted attention for many years due to their properties and potential applications. It is also known as Sorel cement [1-7]. It is a non-hydraulic cement and formed by mixing powdered magnesium oxide (MgO) gauged with magnesium chloride ($MgCl_2.6H_2O$).

It has many superior properties compared to ordinary Portland cement. It has high fire resistance, low thermal conductivity and good resistance to abrasion and is unaffected by oil, grease and paints [8-13]. It is also distinguished by a high bonding, quick setting time and does not require humid curing [14-18]. It is energy saving and environmental protection cement.Lightly burnt MgO used in magnesium oxychloride cement requires much lower calcination temperatures compared to that for Portland cement. This reduces vast amount of energy consumption [19-22].

The major commercial and industrial applications of MOC cement are industrial flooring, fire protection, grinding wheel and light weight wall panels and also used for rendering wall insulation panels, interior plaster and decorative panels [23-26].

The setting and hardening of the MOC cement takes place in a through-solution reaction. [27]. Four main reaction phases in the ternary system are found; $2Mg(OH)_2.MgCl_2.4H_2O$ (phase 2), $3Mg(OH)_2.MgCl_2.8H_2O$ (phase 3), $5Mg(OH)_2.MgCl_2.8H_2O$ (phase 5) and $9Mg(OH)_2.MgCl_2.5H_2O$ (phase 9) out of which $3Mg(OH)_2.MgCl_2.8H_2O$ (phase 3) and $5Mg(OH)_2.MgCl_2.8H_2O$ (phase 5) is more prominent [28-29]. These phases exist as reinforced components in the ternary system at ambient temperature.

Although Magnesium oxychloride cement has many good engineering and mechanical properties, but it has a drawback that it becomes eroded when exposed to water for a long period of time thereby limiting its outdoor application. Consequently, many investigations on the water resistance of MOC cement have been carried out over the years [30-32]. Scanning electron microscopy (SEM) was carried out to analyzes the surface of materials. SEM measures and evaluates surface pitting, failure analysis, characterization of dust, deposits, contaminants, particles, filter residues, and other.

2. Materials and Methods

Calcined magnesite: Commercial grade magnesia used in this study is of Salem origin having the following characteristics displayed in Table 1:

Table: 1 Chemical composition of calcined magnesite (in mass percentage)

MgO	SiO_2	CaO	Fe_2O_3	Al_2O_3	L O I
71.80%	10.18%	6.72%	0.19%	0.75%	9.82%

Magnesium chloride ($MgCl_2$.6H20): Magnesium chloride ($MgCl_2.6H_20$) used in the study is IS grade 3 of IS:254-1973 with following characteristics: (i) colorless, crystalline, hygroscopic crystals (ii) highly soluble in water (iii) magnesium chloride minimum 94% (iv) magnesium sulphate, calcium sulphate and alkali chloride content < 5%.

Inert filler (dolomite): Dolomite dust was used as inert filler with following grading: (i) 100 % passing through 125 micron IS Sieve (ii) 50% retained on 250 micron IS Sieve. Its chemical composition is listed in Table 2. $CaCO_3$ - 55.50% ; $MgCO_3$ - 42.21%

Table: 2 Chemical composition of dolomite (in mass percentage)

SiO_2	CaO	MgO	Fe_2O_3	Al_2O_3	L O I
0.75 %	31.08 %	20.10 %	0.85 %	0.22 %	46.50%

Preparation of gauging solution: Flakes of magnesium chloride were transferred into plastic containers to which

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potable water was added to prepare concentrated solution. This solution was allowed to stand overnight so that insoluble impurities settle at the bottom. The supernatant concentrated solution was taken out in other plastic containers and well stirred after each dilution before determining the specific gravity. Density of the solution is expressed in terms of specific gravity on Baume scale (°Be).

Preparation of dry-mix composition: Dry-mixes were prepared by mixing lightly calcined magnesite (magnesia) and dolomite (inert filler) in the ratio of 1:1 by their weight.

3. Experimental

3.1 SEM image studies

The crystallographic, topography and microstructure of experimental blocks of MOC was examined using SEM investigation. SEM measures and evaluates surface pitting, failure analysis, characterization of dust, deposits, contaminants, particles, filter residues, and other.

Wet-mixes were prepared by gauging of 1:1 dry-mix composition with different densities (20°Be, 24°Be, 28°Be & 32°Be) of $MgCl_2$ solution at 30°C temperature. Setting time blocks samples of each concentration of magnesium chloride solution were analyzed for SEM investigation. Finding images of different trial blocks are shown in the Figure 1 to 5.

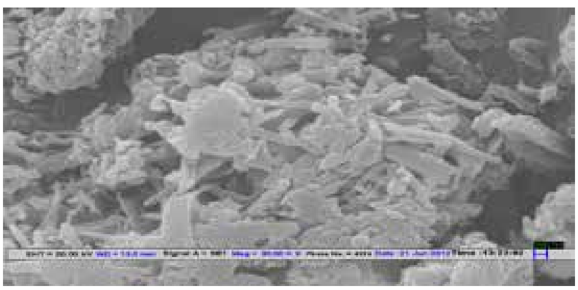

Figure: 1 SEM image of MOC blocks prepared with 20°Be concentration of gauging solution (30°C) and 1:1dry-mix composition

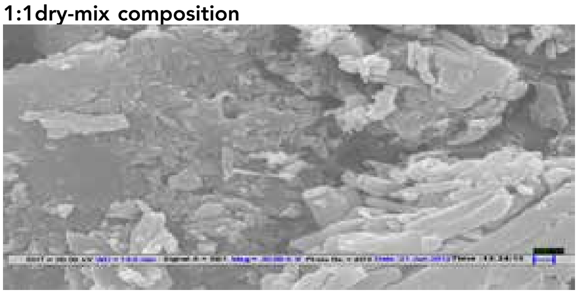

Figure: 2 SEM image of MOC blocks prepared with 24°Be concentration of gauging solution (30°C) and 1:1 dry-mix composition

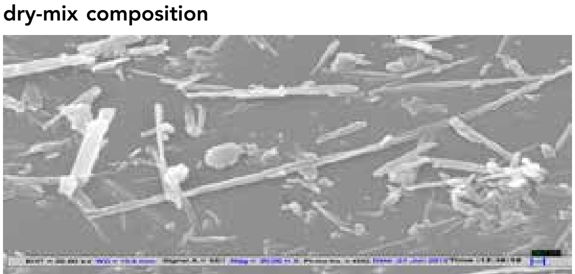

Figure: 3 SEM image of MOC blocks prepared with 28°Be concentration of gauging solution (30°C) and 1:1dry-mix composition

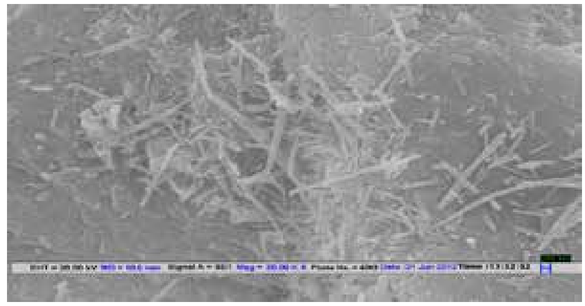

Figure: 4 SEM image of MOC blocks prepared with 32°Be concentration of gauging solution (30°C) and 1:1dry-mix composition

Figure: 1.5 SEM image of MOC blocks prepared with 35°Be concentration of gauging solution (30°C) and 1:1dry-mix composition

4. Result

Scanning electron microscope (SEM) images were used for surface analysis of trial blocks prepared by different concentrations of gauging solution. The SEM images were taken by applying EHT 20,000 KV voltage. Figures 1 to 5 shown SEM micrographs of the MOC prepared at 20°Be, 24°Be, 28°Be, 32°Be & 35°Be concentration of gauging solution at 30°C temperature after setting of one month. These figures demonstrate the fibrous superficial structure of powder surface of different wet-mixes gauged with each concentration. SEM investigations have revealed that powders of concentrations 28°Be and 35°Be have a porous structure. Micro porosities dispersed in the structure of paste 35°Be are apparent as seen in the Figure:5. Nevertheless, paste density 24°Be and 32°Be exhibited the dense and compact structure, as indicated in figure 2 and 4 respectively. The findings of microstructure examinations are in good agreement with the compressive strength measurement and water resisting efficiencies of cement. Density is related to porosity.[33-35] The compressive strength for 1:1 molar ratio of dry-mixes varies with concentrations of gauging solution. Consistent results given in the literature,[36-39] crystalline phase development in neat MOC paste was generally in needle shape. The needle shaped crystals were visible mostly in micro porosities of 32°Be as shown in the Figure:4. MOC has series of characteristics such as fast hardening, high early strength, light weight and good adhesion and hydration phase primarily is $5Mg(OH)_2.MgCl_2.8H_2O$ (5-1-8 phase) and $3Mg(OH)_2.MgCl_2.8H_2O$ (3-1-8 phase). The needle shaped MOC crystals were identified as MOC F5 phase. Porous structure of paste 35°Be is apparent in figure 5 which spread in loose form resulted reduction in the compressive strength. The SEM of paste 35°Be showed the presence of long irregular shaped crystals. The drastic change of the particle shapes was indication of a presence of a new solid amorphous state of MOC due to remaining unused magnesium chloride in the matrix.

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