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EFFECT OF TEMPERATURE OF GAUGING SOLUTION ON SETTING CHARACTERISTICS AND MOISTURE INGRESS OF MAGNESIUM OXYCHLORIDE CEMENT – AN ECO-FRIENDLY CEMENT

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ABSTRACT

Magnesium oxychloride cement (MOC, Magnesia / Sorel's Cement), discovered by Sorel S.T. in 1867 and has versatile cementing characteristics. Investigation pertaining to the effect of temperature of gauging solution on setting characteristics and moisture ingress (steam test) of Magnesium oxychloride cement (MOC) has been observed by authors in this paper. The experimental works have been carried out on different dry-mix compositions 1 : 0, 1 : 1, 1 : 2 & 1 : 3 and on different temperatures 30^{o}C, 35^{o}C, 40^{o}C & 45^{o}C at 28^o Be density of gauging solution (g.s.). It has been recorded that initial and final setting time of cement blocks decreased with increasing temperature of gauging solution in the each dry-mix composition. The increasing trends of both setting time with increasing ratio of inert filler (dolomite) have also been noticed at a particular temperature. Results shown that both setting times of cement blocks are directly proportional to the ratio of inert filler in the dry-mix composition but inversely to temperature of gauging solution. The temperature of magnesium chloride solution (gauging solution) shown the great effect on the moisture ingress of MOC.

Key words: MOC, Gauging solution, Setting time, Moisture ingress, Inert filler.

INTRODUCTION

In 1867, Sorel announced the discovery of excellent cement formed from the combination of magnesium oxide and magnesium chloride solution. This cement type is known by many different names, such as Sorel, magnesite, magnesia, oxychloride, chemoxy and magnesium oxychloride cement. This cement has many superior properties to Portland cement[1-7]. It is a high strength, high bonding and quick setting cement with high early strength. It does not need wet curing, has high fire resistance, low thermal conductivity, good resistance to abrasion. It also has high transverse and compressive strengths, 7,000-10,000 psi are not uncommon. It is a tough, stone like fire proof compound that can be used for light or heavy floorings[4,8-9]. Magnesium oxychloride also bonds very well to a variety of inorganic and organic aggregates, such as, fly ash, saw dust, wood floor, marble floor, sand and gravel, giving a cement that has high early strength, insecticidal properties and is unaffected by oil, grease and paints. It is eco-friendly cement and does not require any heat, light or energy source for its setting. The preparation process of MOC cements can not only save a lot of energy but also emit no carbon dioxide [10-11].

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The major commercial applications of magnesium oxychloride cement, are industrial flooring, fire protection, grinding wheels, and because of its resemblance to marble, have been used for rendering wall insulation panels and for stuccos.

The main bonding phases found in hardened cement pastes are $Mg(OH)_2$, 3 $Mg(OH)_2.MgCl_2.8H_2O$ (3-form) and 5 $Mg(OH)_2.MgCl_2.8H_2O$ (5-form)[12-14]. 5-form is the phase with superior mechanical properties and is formed using a molar ratio of MgO : $MgCl_2$: H_2O = 5 : 1 : 13 with a slight excess of MgO and the amount of water as close as possible to theoretical required for formation of the 5-form and hydration of the excess MgO to form $Mg(OH)_2$[15-16]. Effect of some additive on setting, strength and moisture resistance on MOC has been studied by various researchers[17-21].

Despite many merits associated with this cement, its poor resistance to excessive exposure to water has restricted its outdoor applications. There are only a few reports available worldwide on MOC cement concrete. Therefore, there is exigent need to develop data through laboratory work for evolving the standards and codes of practice for MOC cement so that the excellent bonding properties of this cement can be fully and effectively utilized in areas where such materials are urgently needed.

Hence, the present study was undertaken to investigate the effect of temperature of $MgCl_2$ solution on setting characteristics and moisture ingress (steam test) of magnesium oxychloride cement. Various compositions with dry-mixes containing inert fillers (dolomite) in different proportions (in the ratio of 1 : 0, 1 : 1, 1 : 2, 1 : 3) and $MgCl_2$ solution of density 28^oBe were prepared for the study. The direct application of the test results of different compositions obtained from the study have been discussed in detail in this paper.

EXPERIMENTAL

Materials and methods

The raw materials used in the study were calcined magnesite (magnesia), magnesium chloride and dolomite powder.

Calcined magnesite

Magnesia used in the this study was of Salem (Chennai) having following characteristics – (i) Bulk density 0.85 Kg/I (ii) 95% passing through 75 micron (200 IS sieve) (iii) minimum magnesium oxide 90% (iv) Ca0 < 1.5% (v) Ignition loss at $100^oC \sim 2.5 \pm 0.5\%$.

Magnesium chloride ($MgCl_2.6H_20$)

Magnesium chloride used in the study was Indian Standard Grade 3 of Indian Standard: 254 – 1973 with following characteristics: (i) Colorless, crystalline, hygroscopic crystals. (ii) Highly soluble in water. (iii) Magnesium chloride hexahydrate minimum 95% (iv) Magnesium sulphate, calcium sulphate and alkali chlorides (NaC1) contents were less than 4%.

Inert filler (dolomite)

Dolomite dust with following grading was used as an inert filler : (i) 100% passing through 250 micron Indian Standard Sieve (ii) 50% retained on 125 micron IS Sieve (iii) Ca0 ~ 28.7% (iv) Mg0 ~ 20.8% (v) Insoluble and other sesquioxide contents were less than 1.0%

Preparation of magnesium chloride solution

Magnesium chloride solution was prepared in water. Flakes of magnesium chloride were transferred into plastic containers to which 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. Concentration of the solution is expressed in terms of specific gravity on Baume scale (^{o}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 : 0, 1 : 1, 1 : 2, and 1 : 3 by their weight.

Determination of Standard consistency and setting times of cement pastes

Wet mixes were prepared by gauging magnesium oxide powder with magnesium chloride solution of known concentrations. The standard consistency, initial setting and final setting times were determined as per IS 10132-1982 using Vicat apparatusa[22-24]. The observed results are summarized in the Table 1- 4.

Determination of Moisture ingress test (Steam tests)

To find out the effect of temperature of gauging solution on moisture ingress of magnesia cement, standards setting time blocks were used. These were subjected to steam tests after one month air curing under identical conditions to estimate their relative moisture sealing efficiencies according to the standard procedure[25-28]. The observed results are summarized in the Table 5-8.

RESULTS AND DISCUSSION

(a) IS Consistency of gauging solution

Table 1 shows the volume of the gauging solution required for IS consistency is found to increase slightly with increasing temperature of gauging solution from 30^{o}C to 45^{o}C respectively of the dry-mix composition 1 : 0 at 28^{o}Be density of gauging solution. This may be attributed to high temperature of gauging solution to increasing the exothermic reaction between MgO and gauging solution. Thus increase in amounts of the gauging solution for IS consistency. Same trend for IS consistency is found in the dry-mixes composition 1 : 1 (Table 7). Slightly change in volume of gauging solution required for IS consistency may be seen in dry-mixes composition 1 : 2 & 1 : 3.

(b) Setting characteristics of MOC blocks

Table 1 reveals that Initial setting time decreases as the temperature of gauging solution increases from 30^{o}C to 45^{o}C respectively of dry-mix composition 1 : 0. Similar trends are also observed in the final setting time of the MOC but rate of decreasing in setting time is more in final setting than initial setting.

$$MgO + MgCl_2.6H_2 \xrightarrow{30\text{–}45^oC} 5\,Mg(OH)_2.MgCl_2.8H_2O/3Mg(OH)_2.MgCl_2.8H_2O + \text{Exothermic}$$

(Magnesia cement)

Table 1: Effect of temperature of magnesium chloride solution (G.S.) on standard consistency and setting times of MOC cement

Concentration of Gauging Solution (G.S.) - 28^{o}Be **1 : 0 (dry mix)** Humidity: 85 ± 5 %

Temperature of $MgCl_2$ solution (^{o}C)	Volume of gauging solution (mL)	Setting times (min.)	
		Initial	Final
30^{o}C	111	61	172
35^{o}C	111	53	136
40^{o}C	111.5	44	96
45^{o}C	111.5	40	78

Table 2 reveals that Both initial and final setting time of the MOC of dry-mix composition 1 : 1 decreases as the temperature of gauging solution increases from 30°C to 45°C respectively.

$$MgO + MgCl_2.6H_2O + Mg/CaCO_3 \xrightarrow{30\text{–}45^oC} 5\,Mg\,(OH)_2.MgCl_2.8H_2O \,/\, 3Mg\,(OH)_2.\,MgCl_2.8H_2O$$
$$\text{(Magnesia cement)} + CaO + CO_2 \uparrow + \text{Exothermic}$$

Table 2: Effect of temperature of magnesium chloride solution (G.S.) on standard consistency and setting times of MOC cement

Concentration of Gauging Solution - 28°Be **1 : 1 (dry mix)*** Humidity: 85 ± 5 %

Temperature of $MgCl_2$ solution (°C)	Volume of gauging solution (mL)	Setting times (min.)	
		Initial	Final
30°C	83	94	317
35°C	83	85	198
40°C	84.5	58	121
45°C	84.5	50	110

*One part of magnesia & one part of dolomite

Same trends of decreasing in initial and final setting times with increasing temperature from 30°C to 45°C respectively are observed in the dry-mixes composition 1 : 2 & 1 : 3 (Table 3 & 4).

Table 3: Effect of temperature of magnesium chloride solution (G.S.) on standard consistency and setting times of MOC cement

Concentration of Gauging Solution - 28°Be **1 : 2 (dry mix)*** Humidity: 85 ± 5 %

Temperature of $MgCl_2$ solution (°C)	Volume of gauging solution (mL)	Setting times (min.)	
		Initial	Final
30°C	73	104	425
35°C	72.5	89	213
40°C	73	66	151
45°C	71	70	142

*One part of magnesia & two part of dolomite

Table 4: Effect of temperature of magnesium chloride solution (G.S.) on standard consistency and setting times of MOC cement

Concentration of Gauging Solution - 28°Be **1 : 3 (dry mix)*** Humidity: 85 ± 5 %

Temperature of $MgCl_2$ solution (°C)	Volume of gauging solution (mL)	Setting times (min.)	
		Initial	Final
30°C	66	138	457
35°C	69	99	206
40°C	69	76	179
45°C	66.5	80	155

*One part of magnesia & three part of dolomite

These decreasing trends of setting time of different dry-mix composition cement blocks may be explained with the help of the property of crystalline phases present in the cement paste of varying temperature of gauging solution. It is due to higher temperature of gauging solution, exothermic reaction proceeds very fast and the setting process of cement composition increases. Hence, the both setting time decreases.

It is further firm footing from above discussion, dry–mix composition 1 : 1, 1 : 2 & 1 : 3 takes more setting time than 1 : 0 composition for every temperature of gauging solution. It is fact that dolomite powder used as filler in these dry-mix composition which have calcium and magnesium carbonate content. Dolomite filler increases the setting process due to its decarbonation during the setting processes.

$$Ca/MgCO_3 \longrightarrow CaO + MgO + CO_2 \uparrow$$

(c) Moisture ingress (Steam test) of the blocks

Investigation pertaining to the effect of temperature of gauging solution on moisture ingress of MOC cement blocks is shown in Table 5-8. The temperature of gauging solution has great effect on the moisture ingress of MOC.

Table 5: Effect of temperature of magnesium chloride solution (G.S.) on moisture ingress (steam test) in the trial blocks of MOC

Concentration of Gauging Solution -28^{o}Be **dry mix: (1 : 0)*** Humidity: 85 ± 5 %

S. No.	Temperature of gauging solution	Observations					
		2 hr	4 hr	6 hr	8 hr	10 hr	12 hr
1	30^{o}C	NC	NC	NC	NC	PC	C
2	35^{o}C	NC	NC	PC	PC	C	-
3	40^{o}C	NC	PC	PC	C	-	-
4	45^{o}C	C	-	-	-	-	-

*One part by weight of magnesia and no parts by weight of dolomite

N.C: Not Cracked, P.C: Partially Cracked, C: Cracked

Table 6: Effect of temperature of magnesium chloride solution (G.S.) on moisture ingress (steam test) in the trial blocks of MOC

Concentration of Gauging Solution -28 ^{o}Be **dry mix: (1 : 1)*** Humidity: 85 ± 5 %

S. No.	Temperature of Gauging Solution	Observations					
		2 hr	4 hr	6 hr	8 hr	10 hr	12 hr
1	30^{o}C	NC	NC	NC	NC	NC	PC
2	35^{o}C	NC	NC	NC	NC	PC	PC
3	40^{o}C	NC	NC	NC	PC	PC	C
4	45^{o}C	NC	NC	PC	C	-	-

*One part by weight of magnesia and one parts by weight of dolomite

N.C: Not Cracked, P.C: Partially Cracked, C: Cracked

Table 7: Effect of temperature of magnesium chloride solution (G.S.) on moisture ingress (steam test) in the trial blocks of MOC

Concentration of Gauging Solution -28°Be **dry mix: (1 : 2)*** Humidity: 85 ± 5 %

S. No.	Temperature of gauging solution	Observations					
		2 hr	4 hr	6 hr	8 hr	10 hr	12 hr
1	30°C	NC	NC	PC	PC	C	-
2	35°C	NC	PC	PC	C	-	-
3	40°C	NC	NC	PC	C	-	-
4	45°C	NC	NC	PC	C	-	-

*One part by weight of magnesia and two parts by weight of dolomite

N.C: Not Cracked, P.C: Partially Cracked, C: Cracked

Table 8: Effect of temperature of gauging solution on moisture ingress (steam test) in the trial blocks of MOC

Concentration of Gauging Sol -28°Be **dry mix: (1 : 3)*** Humidity: 85 ± 5 %

S. No.	Temperature of Gauging Solution	Observations					
		2 hr	4 hr	6 hr	8 hr	10 hr	12 hr
1	30°C	NC	NC	PC	PC	C	-
2	35°C	NC	NC	NC	PC	C	-
3	40°C	NC	PC	C	-	-	-
4	45°C	NC	PC	C	-	-	-

*One part by weight of magnesia and three parts by weight of dolomite

N.C: Not Cracked, P.C: Partially Cracked, C: Cracked

Table 5 reveals that lower temperature of gauging solution has great tendency of moisture resisting efficiency up to 8 hrs. After this period trial blocks are either partially cracked or completely cracked. Same trend may be seen in dry–mix composition 1 : 2 (Table 7) & 1 : 3 (Table 8). This is due to high exothermic reaction proceeding in trial blocks. But in case of dry–mix composition 1 : 1 (Table 6), the effect of temperature of gauging solution on moisture ingress are shown very small up to 6 hrs. This may be attributed to that equal amount of (one part of magnesia and one part of dolomite) is used for trial blocks preparation. This is due to interlocking chain forming tendency between magnesia and dolomite. It is interesting to note that the formation of interlocking crystal of calcium and magnesium along with MOC contribute to the water tightness of the product up to a certain limits. Accordingly, it is noted that equal amount of dolomite improves water tightness of the product.

CONCLUSION

As a result of the effect of the temperature of gauging solution on dry-mix composition & their setting process, following important conclusions have been drawn -

(i) Setting times of cement blocks are inversely proportional to temperature of gauging solution but directly proportional to the ratio of inert filler in the dry-mix composition.

(ii) The magnesium oxychloride cement composition with its good cementing characteristics are formed when dry-mix have 1 : 1 composition.

(iii) It has found that dry-mix composition 1 : 0 takes very less setting time and dry-mix composition 1 : 2 & 1 : 3 takes more setting time. Hence, very low & very high setting times are not suitable for development the cementing properties in the wet mix composition.

(iv) Moisture resisting efficiency is found better in dry-mix composition 1 : 1. Therefore, 1 : 1 dry-mix composition is good composition for cementing characteristics**.**

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