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Impact of varying proportions of dolomite powder filler on setting period and compressive strength of magnesia Cement an eco-friendly cement

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ABSTRACT

A parametric study was conducted by authors to investigate the impact of varying proportions of dolomite as filler on cementing properties (standard consistency, setting time and compressive strength) of magnesia cement. For this purpose, dry-mix compositions were prepared by mixing magnesia and dolomite in the ratio 1:0, 1:1, 1:2, 1:3, 1:4, 1:5 and 1:6 by their weight and gauged with 24^{o}Be and 28^{o}Be densities of gauging solution. It was observed that initial and final both setting times were increasing remarkably. It has also been recorded that initial and final setting time of cement blocks increased with increasing concentration of gauging solution (24^{o}Be and 28^{o}Be) in the each dry-mix composition. Results show that both setting time of cement blocks are directly proportional to the ratio of inert filler in the dry-mix composition but compressive strength is decreased with varying dry-mixes ratio.

Key words: *Magnesia cement, Gauging solution, Setting time, Inert filler, Compressive strength*

Introduction

Sorel in 1867 announced the discovery of excellent cement formed from the combination of magnesium oxide and aqueous magnesium chloride solution (Sorel, 1867). This cement type is known by many different names, such as Sorel cement, magnesia cement and magnesium oxychloride cement. This cement has many superior properties to Portland cement as observed by various researchers (Beaudoin and Ramachandran, 1975; Beaudoin *et al.*, 1977; Chandrawat and Yadav, 2000).

The main environmental advantage associated with magnesia cements is that the starting material, magnesia, is readily obtained from the calcining of magnesite. The process of magnesite decomposition occurs at temperatures around 400^{o}C less than that of limestone so it requires considerably less energy to manufacture. It is cheaper and more durable than regular concrete, and it emits less carbon dioxide. Due to its high mechanical qualities, such as compressive strength and bonding strength, low thermal conductivity, and great fire-resistant properties, magnesium oxychloride cement has been used in construction materials, thermal insulation, and packaging.

Despite these advantages, this sustainable material nevertheless faces a significant problem that restricts its economic viability: weak water resistance. Unreacted light-blasted magnesia can dissolve when it is transformed to $Mg(OH)_2$ in humid settings. To address this fundamental problem with the material, a number of approaches have been examined. Researchers have looked into how to modify magne-

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sium oxychloride cement using organic acids, soluble phosphates, inert fillers, and active SCMs. Incorporating waste gypsum, which improves the material's water resistance but drastically lowers its mechanical qualities, has been studied for its potential advantages (Altiner, and Yildirim, 2017).

In recent years, a variety of building, industrial, and agricultural wastes have been investigated for their potential to enhance mechanical and water resistance. These methods are also less harmful to the environment than organic acids and chemicals (Chandrawat *et al.,* 2001; Yadav, 1989; Liska, and Al-Tabbaa, 2008).

Magnesia cement is a type of eco-friendly concrete that uses leftovers or waste products from many sectors during production and uses less energy. It emits less carbon dioxide and is cheaper and more enduring than conventional concrete. Magnesia cement has replaced conventional cement as the preferred alternative in the construction industry since it is recyclable and environmentally beneficial. It only slightly harms the environment. It reduces carbon dioxide emissions, uses less water, and conserves a significant amount of energy (Gupta, Chandrawat, and Yadav, 1994; Liska, and Al-Tabbaa, 2008)

It is a high strength, high holding and fast 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 strength strengths. It is a tough, stone like fire proof compound that can be used for light or heavy floorings (Chandrawat, 1976; Chandrawat, and Yadav, 2000).

Experimental

Materials

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

Calcined magnetite: Magnesia used in the this study was of Salem (Chennai) having following characteristics– (i) Bulk density 0.85 kg/I and 95% passing through 75 micron (200 IS sieve) (ii) Minimum magnesium oxide 86% (iii) CaO < 2.5% (iv) Ignition on loss $CO_2.H_2O$ < 8% (v) Carbon dioxide (CO_2) < 2.5%

Magnesium chloride ($MgCl_2.6H_2O$) : 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 200-micron Indian Standard Sieve (ii) 50% retained on 125 microns IS Sieve (iii) CaO~28.7% (iv) MgO~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 having potable water to prepare concentrated solution. This solution was allowed to stand overnight so that insoluble impurities settle at the bottom. The supernatant concentrated arrangement was taken out in other plastic compartments and very much blended after every weakening prior to deciding the particular gravity. Convergence of the arrangement is communicated as far as unambiguous gravity on Baume scale (°Be).

Determination of Standard consistency: The volumes of the gauging solution required for standard consistency for each dry-mix were determined as per IS 10132-1982 using Vicat apparatus (Chandrawat, 1976; Indian Standard-10132, 1982). The observed results are recorded in Table 1 and 2.

Determination of Setting Time: Wet-mixes prepared for IS consistencies were used for setting times determination. Setting times (initial and final) were determined as per IS-10132-1982 using

Vicat apparatus (Chandrawat, 1976; Indian Standard-10132, 1982). The observed results of setting times are recorded in Table 3 and 4.

Determination of compressive strength: Investigations of the impact of varying proportion of dolomite filler on compressive strength of magnesia cement, cement paste moulds (70.6 mm^3 cubes) were prepared in iron moulds with dry-mixes (1:0 to 1:6) gauged with gauging solution of 24°Be and 28°Be densities for normal consistency. These cubes were tested after 30 days as per standard procedure (ASTM- C109/C109M, 2008). The results are shown in Table 5 and 6.

Results and Discussion

Table 1 and 2 shows the volume of gauging solution

($MgCl_2.6H_2O$) required for preparation of 1:0, 1:1, 1:2, 1:3, 1:4, 1:5 and 1:6 wet-mixes composition decreased in each densities of gauging solution for normal consistency respectively. The viciousness of wet-mixes does not see to vary appreciably with in 24°Be and 28°Be densities of gauging solution. The volume of gauging solution decreases constantly with increasing proportion of filler. This may be due to the corresponding decreasing proportion of magnesia (MgO) in dry-mixes compositions. Under such a situation less and less amount of gauging solution is required to maintain the same consistency. The volume of gauging was maximum for (1:0) dry-mix that is magnesia without filler. This is because, for the inert fillers, solution is only required to wet their surface; they do not undergo any chemical reaction. For dry-mixes with higher ratio of filler such as 1:4 to 1:6 mixes, the volume of gauging solution for each density (24°Be and 28°Be) remains almost the same, as in these mixes the proportion of magnesia is low in respect to dolomite. In these mixes slight excess gauging solution causes excessive of sweating.

Table 3 and 4 revealed the effect of dolomite proportions on the setting characteristics of magnesia cement for dry mixes composition. It was found that initial as well as final setting time increases as 1:0, 1:1, 1:2, 1:3, 1:4, 1:5 and 1:6 dry-mixes composition respectively. From the Table 3 and 4, it can be seen that for each density of gauging solution (24°Be and 28°Be), as the proportion of filler increase the setting time also increase. This is due to formation of 3 and 5-phase more, which required more time for set and

Table 1. Effect of dolomite filler on standard consistency of dry-mix compositions of magnesia cement

	Gauging solution: 24° Be;				Temperature: 32 °C		
	*Dry-mix composition: 1:0 to 1:6; Humidity: 75 ± 5%						
Dry mix compositions	1:0	1:1	1:2	1:3	1:4	1:5	1:6
Volume of gauging solution Required (in ml)	96	75	66	60	55	52	49

* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

Table 2. Effect of dolomite filler on standard consistency of dry-mix compositions of magnesia cement

	Gauging solution: 28° Be;				Temperature: 32 °C		
	*Dry-mix composition: 1:0 to 1:6; Humidity: 75 ± 5%						
Dry mix compositions	1:0	1:1	1:2	1:3	1:4	1:5	1:6
Volume of gauging solution Required (in ml)	96	76	67	61	56	53	50

* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

Table 3. Effect of dolomite filler on setting times of magnesia cement compositions

	Gauging solution: 24° Be;				Temperature: 32 °C		
	*Dry-mix composition: 1:0 to 1:6; Humidity: 75 ± 5%						
Dry mix compositions	1:0	1:1	1:2	1:3	1:4	1:5	1:6
Initial setting time (in minutes)	62	86	108	120	142	153	177
Final setting time (in minutes)	188	220	252	283	312	334	358

* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

Table 4. Effect of dolomite filler on setting times of magnesia cement compositions

	Gauging solution: 28° Be;				Temperature: 32 °C		
	*Dry-mix composition: 1:0 to 1:6; Humidity: 75 ± 5%						
Dry mix compositions	1:0	1:1	1:2	1:3	1:4	1:5	1:6
Initial setting time (in minutes)	77	98	128	145	160	167	187
Final setting time (in minutes)	198	245	273	302	321	344	376

* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

Table 5. Effect of dolomite filler on compressive strength of magnesia cement compositions
Gauging solution: **24° Be**; Temperature: 32 °C
Dry-mix composition: 1:0 to 1:6; Humidity: 75 ± 5%

Dry-mix Compositions	Compressive strength (in MPa)			
	M-1	M-2	M-3	ACS
1:0	76.238	75.837	77.241	76.439
1:1	68.614	69.417	69.015	69.015
1:2	56.978	56.175	58.984	57.379
1:3	47.147	48.150	46.144	47.147
1:4	30.495	30.094	30.896	30.495
1:5	22.470	23.072	23.674	23.072
1:6	15.247	15.648	16.050	15.648

ACS: Average Compressive Strength; M-1: Mould-1; M-2: Mould-2; M-3: Mould-3
* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

Table 6. Effect of dolomite filler on compressive strength of magnesia cement composition
Gauging solution: 28° Be; Temperature: 32 °C
Dry-mix composition : 1:0 to 1:6; Humidity: 75 ± 5%

Dry-mix Compositions	Compressive strength (in MPa)			
	M-1	M-2	M-3	ACS
1:0	82.257	82.658	83.059	82.658
1:1	71.222	71.824	70.620	71.222
1:2	64.200	62.194	63.197	63.197
1:3	53.768	52.965	52.163	52.965
1:4	36.514	36.112	36.313	36.313
1:5	26.482	27.285	26.884	26.884
1:6	18.056	18.457	18.858	18.457

ACS: Average Compressive Strength; M-1: Mould-1; M-2: Mould-2; M-3: Mould-3
* In dry-mix composition, one part by weight of magnesia and remaining part by weight of dolomite.

also due to inert filler absorbs the heat which evolves during the reaction between MgO and $MgCl_2$.

$MgO + MgCl_2.6H_2O \rightarrow 5Mg(OH)_2.MgCl_2.8H_2O/3Mg(OH)_2.MgCl_2.8H_2O$ + Exothermic
(Magnesia cement)

It is further firm footing from above discussion, dry–mix composition 1:1 takes more setting time than 1:0 composition. It is fact that dolomite powder used as filler in the dry-mix 1:1 (One part of MgO and one part of dolomite), which have calcium and magnesium carbonate content. Due to their content, dolomite filler increases the setting process due to its de- carbonation during the setting process.

$Ca/MgCO_3 \rightarrow CaO + MgO + CO_2$

This magnesia still contains small amounts of CO_2 and contaminated with over burnt/dead burnt magnesia & calcium oxide, which is not suitable for magnesia cement. Similar trends have been found for all dry-mixes composition. From Table 3 and 4, it can see that density of gauging solution has played a major effect on setting period of various compositions. Dry-mixes gauged with 28°Be solution have more setting time than 24°Be solution for all proportions of filler. As the density of magnesium chloride increases, the mixes trend to become highly viscous and initial setting time increases. This is due to the formation of 3 and 5, which require more time to set. As already noted, similar trends were also observed in final setting for all proportion of dolomite filler but in case of dry-mix compositions 1:3 setting processes are slightly slow as compared to 1:0, 1:1 & 1:2 dry-mix compositions, due to increasing the calcinations process of dolomite and exothermic reaction. From table 6, it can be seen that the density of the gauging solution shown great effect on compressive strength of various compositions. The dry-mix (1:1) gauged with 24°Be solution has compressive strength 69.015 MPa. If same mix is gauged with 28°Be solution the strength comes out to be 71.222 MPa. Similarly, dry-mix (1:2) has compressive strength 57.379 MPa with 24°Be solution.

When density of solution is increased to 28°Be, the strength of same mix comes out to be 63.197 MPa. When the proportion of filler is increased from 1:4 to 1:6, the compressive strength results are low at 24°Be solution comparatively 28°Be gauging solution. This is because of the small amount of magnesia to form cement for binding at high proportion of filler in these mixes. This owe to correspondingly decreasing proportion of magnesia in dry-mixes and less chances for the formation of strength giving composition (Magnesium Oxychloride Cement).

Conclusion

(i) Setting times of cement blocks are directly proportional to the ratio of inert filler in the dry-mix composition.
(ii) Magnesia cement requires lower calcination temperature than ordinary Portland cement. Hence, it can be replaced by conventional ordinary Portland cement as the preferred alternative in the construction industry. Due to energy saving and environmental protection consideration, it is an eco-friendly cement.

(ii) 1:1 dry-mix cement composition has good cementing characteristics setting time as well as compressive strength.

(iii) It has found that dry-mix composition 1:0 takes very less setting time and dry-mix composition 1:1, 1:2, 1:3, 1:4, 1:5 and 1:6 are takes more setting time. Hence, very low and very high setting times are not suitable for development the cementing properties in the wet-mix composition. Therefore, 1:1 dry-mix composition is good composition for cementing characteristics**.**

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