



Effect of Epsom Salt Concentration and Dry-Mix Composition on Bonding Properties of Magnesium Oxysulfate

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Non-hydraulic magnesium oxysulfate cement has many superior properties than ordinary portland cement and it draws much research interest due to energy saving and environment protection considerations. It is formed by the exothermic reaction between lightly calcined MgO and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (epsom salt) solution. This heat is responsible for cracking and makes the product unsound. The incorporation of inert filler can overcome this problem by absorbing the excess heat. In present work, a parametric study was conducted to investigate the effect of dry-mix composition (MgO: inert filler) and gauging solution epsom ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) concentration on cementing properties of magnesium oxysulfate cement. The results show that a 1:1 dry-mix composition of MgO and dolomite with 25% concentration of magnesium sulfate works best in the cementing action of magnesium oxysulfate.

Keywords: Magnesium oxysulfate, Magnesia, Epsom salt, Setting properties.

INTRODUCTION

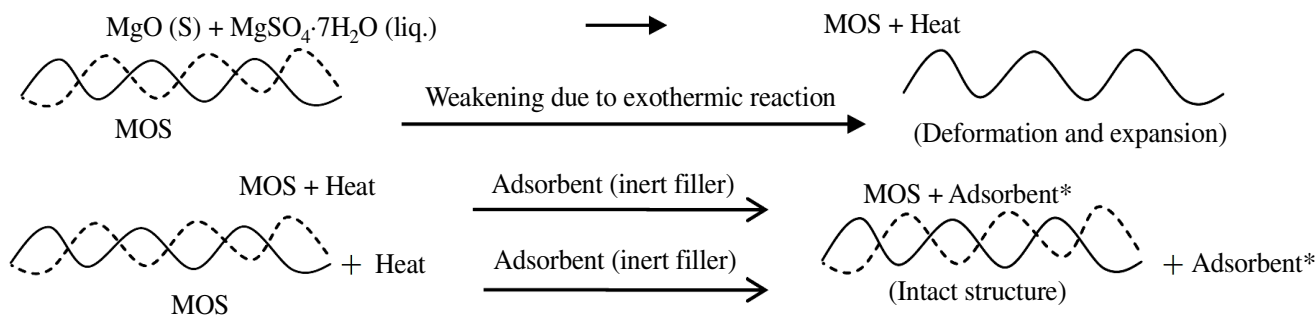
In 1867, French engineer Stanislas Sorel discovered non-hydraulic magnesia cement [1,2]. It is distinguished by hardness, high bonding, quick setting time and does not require humid curing. Magnesium oxychloride and magnesium oxysulfate are main two types of magnesia cement.

Magnesium oxysulfate is an air-hardening cementing material and possesses some excellent properties compared with ordinary portland cement, such as light weight, low thermal conductivity, fire protection, high mechanical strength, good volume stability, high cohesiveness in light-weight panels [3-6]. For many years, magnesium oxysulfate cement has been used in the commercial production of decorative materials, light-weight thermal insulating materials and fire-proof materials [7]. Consistent with magnesium oxychloride (MOC) cement, magnesium oxysulfate (MOS) cement also shows the merits of rapid setting, early strength and high acid solubility, which is in contrast to portland cement [8]. It arouses great attention to the less hygroscopic nature of magnesium sulfate as opposed to magnesium chloride. Thereby enabling easier shipping and a longer shelf life for bagged cements [9]. Magnesium oxysulfate cement is less damaging to steel reinforcement and it has superior

resistance to weather compared with magnesium oxychloride cement [10,11]. The production of lightly burnt magnesium oxide used in magnesia cement requires much lower calcination temperature than used for portland cement, hence magnesium oxysulfate cement is a green and environmental friendly civil engineering material with 50-60% reduction in carbon dioxide emissions compared to ordinary portland cement [12].

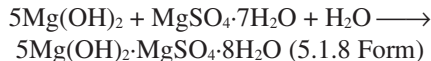
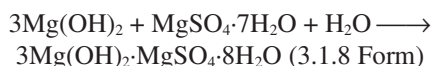
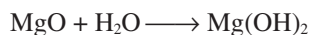
Magnesium oxysulfate is formed by lightly calcining MgO with a concentrated solution of magnesium sulfate [13]. This reaction is exothermic, hence heat is produced, which causes cracks in the cement and decreases its strength & moisture resistance and makes the product unsound. To overcome this problem, inert filler is used in the matrix. Inert filler does not participate in the cementing reaction but absorbs heat through a three-body collision mechanism. In present work, dolomite powder was used as an inert filler to reduce thermal shocks in the cement.

The setting and hardening properties of magnesium oxysulfate cement depend on the ternary hydration phases and microstructures [14,15]. The compressive strength is primarily determined by the type, relative content and microstructure of hydration products ($x\text{Mg}(\text{OH})_2 \cdot y\text{MgSO}_4 \cdot z\text{H}_2\text{O}$ phases), as significantly impacted by the molar ratio of MgO, MgSO_4 and H_2O ,



MOS = Magnesium oxysulfate cement

as well as temperature. At temperatures ranging from 30 to 120 °C, following magnesium oxysulfate phases existing in the magnesium oxide-magnesium sulfate-water (MgO-MgSO₄-H₂O) system were 3Mg(OH)₂·MgSO₄·8H₂O (3·1·8 phase), 5Mg(OH)₂·MgSO₄·8H₂O (5·1·8 phase) 5Mg(OH)₂·MgSO₄·3H₂O (5·1·3 phase) or 5Mg(OH)₂·MgSO₄·2H₂O (5·1·2 phase), Mg(OH)₂·2MgSO₄·3H₂O (1·2·3 phase) and Mg(OH)₂·MgSO₄·5H₂O (1·1·5 phase) [16-18]. The 3·1·8 and 5·1·8 are the main strength phases formed in magnesium oxysulfate cement [19-21]. The reactions of formation of 3·1·8 and 5·1·8 form of magnesium oxysulfate cement are given below:



Magnesium oxysulfate cement has many beneficial engineering and mechanical properties, but its large-scale commercial application is limited due to its poor water resistance [22-24], resulting in a significantly decreased strength of the hardened paste in water [25]. The weakness of magnesium oxysulfate cement comprises poor water resistance, which leads to high moisture absorption, which tends to high deformability in structure. Hence, its large scale commercial use is limited.

Present study is focused on the effect of different compositions of MgO and dolomite (dry-mix composition) and gauging solution (MgSO₄) concentration on cementing properties of magnesium oxysulfate cement to find out the best cementing composition of magnesium oxysulfate cement.

EXPERIMENTAL

The raw materials used in the study were calcined magnesite (magnesia), technical grade magnesium sulfate heptahydrate and dolomite powder (inert filler) were procured from the commercial sources.

Magnesium oxide (MgO): Commercial grade magnesia used in the study. It was collected from Suksha export, Bagru, Jaipur, Rajasthan. The chemical composition of MgO was MgO = 82.70%, SiO₂ = 8.51%, CaO = 2.80%, Fe₂O₃ = 0.12%, Al₂O₃ = 0.07%, Loss on ignition (LOI) = 4.40%.

Magnesium sulfate (MgSO₄·7H₂O): The epsom salt used was of Indian Standard technical grade [26] with the following

characteristics: colourless, crystalline, hygroscopic crystals; highly soluble in water. It was collected from Divi globals, Jaipur city, India. Its chemical composition was MgSO₄ = 96.80%, Fe₂O₃ = 0.02%, Al₂O₃ = 0.07%, CaO = 1.40%, moisture = 0.98%, acid insoluble = 0.11%.

Dolomite: Waste material of dolomite mines (dolomite dust produced during cutting, shaping, etc.) was used as an inert filler [27]. It was collected from Matasya Industrial Area, Alwar city, India. The chemical composition of dolomite was SiO₂ = 5.06%, CaO = 29.40%, MgO = 19.50%, Fe₂O₃ = 0.82%, Al₂O₃ = 0.23%, LOI = 44.50%, CaCO₃ = 52.50%, MgCO₃ = 40.95%, brightness = 93.00%, whiteness = 95.30%.

Methods

Preparation of dry-mixes: Dry-mixes were prepared by mixing calcined magnesium oxide and dolomite in the ratio of 1:0, 1:1, 1:2 and 1:3 by weight.

Preparation of gauging solution: Magnesium sulfate (technical grade) was dissolved in luke warm water to make a saturated solution and allowed to stand overnight so that insoluble impurities settled down at the bottom of the container. The supernatant saturated solution was filtered with the help of vacuum pump and as known as gauging solution for oxysulfate cement. The concentration of the gauging solution was determined in terms of degree on the Baume scale; the higher the concentration, higher the density of the gauging solution.

Preparation of wet-mix: Gauging solution was added in the dry-mix to form a wet-mix of workable consistency. All the experiments were performed under the identical conditions of temperature (about 30 °C) and humidity (> 90%). Following experiments were conducted to investigate the effect of epsom salt concentration and dry-mix composition on bonding properties of magnesium oxysulfate cement. Every experimental method was repeated three times and the average was reported.

Setting time investigations: Different dry-mix proportions were taken and then these thoroughly mixed compositions were gauged with different concentrations of gauging solution (0° Be to 31° Be) to obtain wet-mixes of Indian Standard consistency. The wet-mixes so prepared were filled into Vicat moulds and initial and final setting times of these wet-mixes were determined with the help of Vicat needle apparatus as per standard procedure [28].

Weathering effect investigations: Setting time blocks were cured under identical conditions of temperature and humidity for 24 h. These blocks were then weighed on a chemical

balance at different time intervals. Trends in the change in weights of these blocks with passage of time give a clear idea about weathering effects.

Moisture ingress investigations: Setting time blocks prepared from different dry-mix compositions were exposed to steam/boiling water in a steam bath after 60 days of curing according to the standard procedure. This is unavoidable before evaluating their respective moisture sealing efficacies.

Compressive strength investigations: To investigate the compressive strength of magnesium oxysulfate cement standard sized 50 cm² (70.6 mm × 70.6 mm × 70.6 mm) cubes of different compositions were prepared from gauging different dry-mixes with varying concentrations of magnesium sulfate solution. These wet-mixes were kept in moulds for 24 h. These blocks were cured for 28 days under identical conditions of temperature and humidity. Then the compressive strength of different trial blocks was determined by a compressive strength testing machine.

Linear change investigations: To determine the change in the length of magnesium oxysulfate cement, different trial beams (200 mm × 25 mm × 25 mm) were prepared from the different dry mix compositions of magnesium oxide and dolomite (1:0, 1:1, 1:2, 1:3) and gauging magnesium sulfate solution of varying concentrations. The length of the beams was determined with the help of Vernier callipers after 24 h and 28 days of curing under identical conditions of temperature and humidity.

RESULTS AND DISCUSSION

Effect on setting properties: Table-1 represents the setting characteristics of different compositions of magnesia cement (with reference to varying concentrations of gauging solution). It is observed that the volume of gauging solution required to obtain IS consistency is maximum for all 1:0 dry-mix compositions and this amount is reduced remarkably for 1:1, 1:2 and 1:3 dry-mix compositions. Magnesia is the main reactant and absorbent of the cement hence incorporation of filler reduces the relative amount of magnesia in the dry-mix therefore reducing amounts of the gauging solution with increasing proportions of the fillers are expected. For any specific concentration of gauging solution initial and final setting time increases with the increasing quantities of filler in the matrix *i.e.* setting time is minimum for 1:0 dry-mix compositions and maximum for 1:3 dry-mix compositions. It is observed that initial and final setting times decrease from 0° Be to 25° Be and then increase up to 31° Be concentration of gauging solution. These trends in setting behavior suggest that at concentrations of about 25° Be, relative proportion of magnesium sulfate and water in the gauging solution are just sufficient for the cementing reaction. Hence at this concentration, the rate of formation of cement is maximum. Accordingly, the setting periods were found to be minimum at this concentration. For other concentrations of gauging solutions either magnesium sulfate is in excess (> 25° Be) or water is in excess (< 25° Be), both these situations hamper cementing process. Accordingly, setting periods are found to increase with increasing dilutions. Increasing setting periods with increasing proportions of the filler for a given concentration of the gauging solution are

TABLE-1
EFFECT OF DRY-MIX COMPOSITION AND GAUGING SOLUTION CONCENTRATION ON SETTING PROPERTIES OF MAGNESIUM OXYSULFATE CEMENT

Concentration of gauging solution	Dry-mix composition (MgO: dolomite)	Volume of gauging solution (mL)	Setting time (min)	
			Initial	Final
0° Be	1:0	100.0	82	420
	1:1	65.0	104	480
	1:2	53.0	120	520
	1:3	51.0	150	560
4° Be	1:0	100.0	79	410
	1:1	64.5	92	430
	1:2	53.5	100	440
	1:3	52.0	130	510
8° Be	1:0	102.0	70	230
	1:1	71.0	81	242
	1:2	59.0	90	265
	1:3	57.5	100	360
12° Be	1:0	105.0	60	218
	1:1	74.0	73	222
	1:2	62.0	86	240
	1:3	60.5	90	300
16° Be	1:0	106.0	58	207
	1:1	77.0	70	215
	1:2	65.0	83	229
	1:3	62.5	87	245
20° Be	1:0	106.0	54	198
	1:1	80.0	65	210
	1:2	69.0	79	225
	1:3	64.0	82	235
22° Be	1:0	107.0	53	192
	1:1	82.0	63	207
	1:2	71.0	74	220
	1:3	65.0	75	226
23° Be	1:0	110.0	52	190
	1:1	85.0	63	204
	1:2	72.0	74	213
	1:3	67.0	75	220
24° Be	1:0	105.5	52	189
	1:1	86.5	63	202
	1:2	75.5	72	211
	1:3	67.5	75	216
25° Be	1:0	108.0	51	187
	1:1	87.0	62	200
	1:2	74.0	70	210
	1:3	68.0	74	214
26° Be	1:0	109.0	53	190
	1:1	87.0	67	208
	1:2	76.0	72	215
	1:3	68.0	76	220
28° Be	1:0	109.0	55	192
	1:1	87.0	68	212
	1:2	75.0	73	219
	1:3	69.0	76	223
31° Be	1:0	112.0	57	194
	1:1	88.0	69	219
	1:2	76.0	74	225
	1:3	69.0	77	330

explicable on the basis of the relatively decreasing proportions of magnesia in the matrix. Such a situation also retards setting process. It is notable that variations are more in case of final setting periods than that in case of initial setting periods with

every trial of dry-mixes. This is attributable to the mechanism of setting. Initial setting involves processes like hydration, gelation, *etc.* which are relatively faster, whereas super saturation, formation of stereospecific interlacing crystals, *etc.* which are relatively slower processes, involved in final setting periods. Hence, the initial setting periods were affected relatively less whereas final setting periods were affected considerably. At 0° Be concentration of magnesium sulfate (absence of magnesium sulfate) cement forming component is absent, therefore factors responsible for initial setting were not affected much (because hydration is possible even with water), but formation of strength-giving compositions responsible for final setting periods is not possible. Accordingly quite high final setting periods were recorded with simple water as gauging solution.

Effect of weathering: Weathering characteristics of different compositions of magnesium oxysulfate cement are summarized in Table-2. Water left in the matrix after final setting period gradually evaporates with lapse of time which is the reason of decrease in weights of the trial blocks. When the amount of magnesium sulfate was far less than the optimum amount, vapourization was fast and free magnesia was also there. This free magnesia has tendency to absorb the

atmospheric carbon dioxide. Accordingly, it was observed that at very low concentrations of gauging solution, weights become constant speedily and may also show a slight increase in weights at later stages on account of carbonation.

Effect of moisture: Table-3 represents the effect of moisture ingress characteristics of different compositions of magnesium oxysulfate cement. All the trial blocks with 1:0 dry-mix composition and varying concentrations of gauging solution (0° Be to 31° Be) were cracked during curing and with the incorporation of dolomite (inert filler) there was no cracking. The reason is that reaction between magnesium oxide and magnesium sulfate was exothermic and fillers absorbs excess heat. It is clear from Table-3 that water tightness of the trial blocks with any specific concentration of gauging solution increases with increasing amount of filler. Also, it may be observed that water tightness of the trial blocks increases with increasing concentrations of gauging solution. This is attributable to the fact that amount of magnesia available freely becomes less and less with increasing amounts of magnesium sulfate in the matrix. Hence, chances of expansion or formation of 'cracks' are reduced proportionately.

Effect on compressive strength: It is revealed from Table-4 that change in dry-mix compositions (MgO:dolomite) and

TABLE-2
EFFECT OF DRY-MIX COMPOSITION AND GAUGING SOLUTION CONCENTRATION
ON WEATHERING EFFECTS OF MAGNESIUM OXYSULFATE CEMENT

Concentration of gauging solution	Dry-mix composition (MgO:dolomite)	Weight of trial blocks in g after			
		1 day	7 days	30 days	45 days
0° Be	1:0	202.432	179.191	177.700	176.520
	1:1	224.430	206.000	205.280	205.115
	1:2	221.600	211.300	204.100	204.110
	1:3	202.500	196.700	192.010	192.000
4° Be	1:0	217.171	192.420	189.930	190.100
	1:1	236.450	217.210	215.912	217.220
	1:2	220.200	204.340	202.560	201.190
	1:3	215.880	200.470	201.430	201.000
8° Be	1:0	225.670	203.660	197.000	195.820
	1:1	237.220	221.150	214.560	215.210
	1:2	230.100	212.840	209.460	208.300
	1:3	235.230	219.560	216.820	214.910
12° Be	1:0	217.800	207.340	197.220	193.770
	1:1	249.190	241.280	230.024	228.021
	1:2	234.610	227.930	215.240	214.480
	1:3	241.230	232.340	221.950	221.000
16° Be	1:0	243.210	238.880	229.650	225.980
	1:1	252.000	248.440	239.170	234.390
	1:2	250.720	245.000	235.250	230.910
	1:3	238.470	233.320	220.890	216.880
20° Be	1:0	246.180	235.920	226.230	225.180
	1:1	248.205	238.310	229.080	226.500
	1:2	243.920	232.770	224.000	221.545
	1:3	227.830	215.360	208.635	207.550
22° Be	1:0	232.880	218.450	209.860	208.120
	1:1	261.410	248.640	238.960	236.800
	1:2	244.020	229.750	217.910	217.200
	1:3	245.960	230.950	220.330	219.320
23° Be	1:0	237.100	224.510	213.000	210.720
	1:1	262.420	248.610	238.400	236.320
	1:2	246.240	231.020	222.480	221.200
	1:3	246.760	231.820	221.960	220.740

25° Be	1:0			Cracked during curing			
	1:1	No effect	No effect	No effect	No effect	No effect	No effect
	1:2	No effect	No effect	No effect	No effect	No effect	No effect
	1:3	No effect	No effect	No effect	No effect	No effect	No effect
26° Be	1:0			Cracked during curing			
	1:1	No effect	No effect	No effect	No effect	No effect	No effect
	1:2	No effect	No effect	No effect	No effect	No effect	No effect
	1:3	No effect	No effect	No effect	No effect	No effect	No effect
28° Be	1:0			Cracked during curing			
	1:1	No effect	No effect	No effect	No effect	No effect	No effect
	1:2	No effect	No effect	No effect	No effect	No effect	No effect
	1:3	No effect	No effect	No effect	No effect	No effect	No effect
31° Be	1:0			Cracked during curing			
	1:1	No effect	No effect	No effect	No effect	No effect	No effect
	1:2	No effect	No effect	No effect	No effect	No effect	No effect
	1:3	No effect	No effect	No effect	No effect	No effect	No effect

TABLE-4
EFFECT OF DRY-MIX COMPOSITION AND GAUGING SOLUTION CONCENTRATION ON COMPRESSIVE STRENGTH OF MAGNESIUM OXYSULFATE CEMENT

Concentration of gauging solution	Dry-mix composition (MgO:dolomite)	Compressive strength (Kg/cm ²)
0° Be	1:0	C
	1:1	Below 10
	1:2	Below 10
	1:3	Below 10
4° Be	1:0	C
	1:1	Below 10
	1:2	Below 10
	1:3	Below 10
8° Be	1:0	C
	1:1	Below 10
	1:2	Below 10
	1:3	Below 10
12° Be	1:0	C
	1:1	118.11
	1:2	87.10
	1:3	69.44
16° Be	1:0	C
	1:1	135.20
	1:2	110.00
	1:3	96.58
20° Be	1:0	C
	1:1	155.50
	1:2	135.20
	1:3	120.25
22° Be	1:0	C
	1:1	190.12
	1:2	160.32
	1:3	151.98
23° Be	1:0	C
	1:1	220.30
	1:2	180.00
	1:3	162.79
24° Be	1:0	C
	1:1	250.22
	1:2	230.00
	1:3	221.12
25° Be	1:0	C
	1:1	260.53
	1:2	250.45
	1:3	244.22

26° Be	1:0	C
	1:1	258.22
	1:2	250.32
	1:3	242.24
28° Be	1:0	C
	1:1	254.21
	1:2	240.98
	1:3	235.12
31° Be	1:0	C
	1:1	251.33
	1:2	230.00
	1:3	222.01

C = Sample cracked during curing

concentrations of magnesium sulfate solution (0° Be to 31° Be) affect the compressive strength of the cement considerably. Cement sample cubes prepared with only magnesia (1:0 dry-mix composition) and magnesium sulfate solution cracked during curing due to highly exothermic reaction and availability of free magnesia. As the fillers can absorb excess heat and reduce the thermal shocks in the bulk, their incorporation increases the strength of the cement. It was observed that trial blocks with concentrations of gauging solution 0° Be, 4° Be and 8° Be have insignificant compressive strength. It was also revealed that for any concentration of gauging solution, compressive strength of trial blocks with 1:1 dry-mix composition is found to be maximum. Compressive strength is less for 1:2 and 1:3 dry-mix composition due to reduced proportion of magnesia and excess amount of inert filler dolomite in the reaction mixture. Also, compressive strength of trial blocks increases from 0° Be to 25° Be (conc. of gauging solution) and then decreases up to 31° Be *i.e.* compressive strength is maximum for trial blocks of 1:1 dry mix composition and 25° Be concentration of gauging solution. Plausible cause of this fact appears to be the presence of water and magnesium sulfate in optimum ratio in 25° Be gauging solution as required for the strength-giving compositions. More concentrated or diluted gauging solutions have either magnesium sulfate in excess or moisture in excess in the matrix. The former is not the strength-giving factor and the later promotes exothermic hydration reaction responsible for thermal shocks. Hence, in either case, decreasing strengths were expected.

Effect on linear changes: Table-5 summarizes the effect of linear changes of magnesium oxysulfate cement. It is observed that for all the 1:0 dry mix compositions linear change is maximum. Since the reaction between magnesium oxide and magnesium sulfate is exothermic, the greatest linear change

occurs at low concentrations. When dolomite is incorporated in the reaction mixture, it absorbs the excess heat evolved during the progress of the reaction hence change in length decreases. It is also observed that minimum change in length was found at 25° Be concentration of gauging solution.

TABLE-5
EFFECT OF DRY-MIX COMPOSITION AND GAUGING SOLUTION CONCENTRATION
ON LINEAR CHANGE OF MAGNESIUM OXYSULFATE CEMENT

Concentration of gauging solution	Dry-mix composition (MgO:dolomite)	Initial length (mm)	Final length(mm)	Change in length (mm)
0° Be	1:0	200	199.704	0.296
	1:1	200	199.712	0.288
	1:2	200	199.717	0.283
	1:3	200	199.725	0.275
4° Be	1:0	200	199.709	0.291
	1:1	200	199.719	0.281
	1:2	200	199.729	0.271
	1:3	200	199.732	0.268
8° Be	1:0	200	199.739	0.261
	1:1	200	199.748	0.252
	1:2	200	199.760	0.240
	1:3	200	199.770	0.230
12° Be	1:0	200	199.760	0.240
	1:1	200	199.780	0.220
	1:2	200	199.769	0.231
	1:3	200	199.798	0.202
16° Be	1:0	200	199.802	0.198
	1:1	200	199.825	0.175
	1:2	200	199.835	0.165
	1:3	200	199.845	0.155
20° Be	1:0	200	199.831	0.169
	1:1	200	199.850	0.150
	1:2	200	199.870	0.130
	1:3	200	199.880	0.120
22° Be	1:0	200	199.865	0.135
	1:1	200	199.879	0.121
	1:2	200	199.889	0.111
	1:3	200	199.898	0.102
23° Be	1:0	200	199.901	0.099
	1:1	200	199.918	0.082
	1:2	200	199.928	0.072
	1:3	200	199.938	0.062
24° Be	1:0	200	199.898	0.102
	1:1	200	199.902	0.098
	1:2	200	199.905	0.095
	1:3	200	199.907	0.093
25° Be	1:0	200	199.950	0.050
	1:1	200	199.970	0.030
	1:2	200	199.980	0.021
	1:3	200	199.797	0.020
26° Be	1:0	200	199.944	0.056
	1:1	200	199.951	0.049
	1:2	200	199.971	0.029
	1:3	200	199.978	0.022
28° Be	1:0	200	199.944	0.056
	1:1	200	199.951	0.049
	1:2	200	199.972	0.028
	1:3	200	199.979	0.021
31° Be	1:0	200	199.944	0.056
	1:1	200	199.952	0.048
	1:2	200	199.973	0.027
	1:3	200	199.979	0.021

Conclusion

The negative effect of exothermic reaction between MgO and MgSO₄ solution can be removed by incorporating dolomite in the reaction mixture. And magnesium oxysulfate cement can be made cheaper without affecting the quality of product by the incorporation of cheap inert fillers in the matrix. The setting-time investigations revealed that the minimum setting time was observed for 25° Be concentration of gauging solution. The water tightness of the blocks increases with increasing amounts of filler in the matrix and also with higher concentrations of gauging solution. The ideal composition for the maximum strength and durability was obtained by using gauging solution of 25° Be and 1:1 dry-mix composition.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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