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RESEARCH ARTICLE

EFFECT OF SOAPSTONE POWDER ON CEMENTING PROPERTIES OF MAGNESIUM OXYSULFATE CEMENT

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ABSTRACT

Magnesium oxysulfate cement is a type of Sorel's cement and it does not require any type of energy whether heat or light, making it an eco-friendly binding substance. Non- hydraulic Magnesium oxysulfate cement has many superior properties to that of ordinary Portland cement. Magnesium oxysulfate cement draws much research interest due to energy saving and environmental protection consideration. In spite of a variety of advantages, its commercial use is limited due to its low early strength and poor water resistance. In this study, the effect of soapstone powder on setting times, weathering effect, compressive strength, moisture ingress, and linear changes has been comprehensively investigated. The results show that strength and water resistance increases while setting time decreases by adding soapstone powder in different percentages.

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INTRODUCTION

Non-hydraulic Magnesia cement was discovered in 1867 by French engineer Stanislas Sorel[1,2]. It stands out for its hardness, good bonding, short setting time, and lack of a humid curing environment. The two main forms of Magnesium cement are Magnesium oxychloride and Magnesium oxysulfate. In comparison to regular Portland cement, Magnesium oxysulfate is an air-hardening cementing substance that has some excellent qualities, including light weight, low thermal conductivity, fire protection, high mechanical strength, good volume stability, and high cohesiveness in light-weight panels[3-6]. Decorative materials, light-weight thermal insulating materials, and fire-resistant materials are all commercially produced using Magnesium oxysulfate cement. In contrast to Portland cement, Magnesium oxysulfate (MOS) cement has the benefits of rapid setting, early strength, and high acid solubility, which is consistent with magnesium oxychloride (MOC) cement[7-8]. The fact that Magnesium sulfate is less hygroscopic than Magnesium chloride attracts a lot of attention. Consequently, bagged cements may be shipped more easily and have a longer shelf life[9]. Compared to Magnesium oxychloride cement, Magnesium oxysulfate cement has better weather resistance and is less harmful to steel reinforcement[10,11]. Magnesium oxysulfate (MOS) cement is a green and environmentally friendly civil engineering material with 50–60% lower Carbon dioxide emissions compared to regular Portland cement because the production of lightly burned Magnesium oxide used in Magnesium cement requires much lower calcination temperatures than used for 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 the present research work dolomite powder was used as an inert filler to reduce thermal shocks in the cement (Fig. 1: Schematic presentation)[14]. 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 (xMg(OH)$_2$·yMgSO$_4$·zH$_2$O phases), as significantly impacted by the molar ratio of MgO, $MgSO_4$ and H_2O, as well as temperature. At temperatures from 30 to 120°C, following Magnesium oxysulfate phases existing in the Magnesium oxide–Magnesium sulfate–water (MgO–$MgSO_4$–H_2O) system are $3Mg(OH)_2·MgSO_4·8H_2O$ (3·1·8phase), $5Mg(OH)_2·MgSO_4·8H_2O$ (5.1.8 Phase) $5Mg(OH)·MgSO_4·3H_2O$ (5·1·3phase) or $5Mg(OH)·MgSO_4·2H_2O$ (5·1·2phase), $Mg(OH)_2·2MgSO_4·3H_2O$ (1·2·3 phase) and $Mg(OH)_2·MgSO_4·5H_2O$ (1·1·5 phase)[16-18]. 3.1.8 & 5.1.8 are the main strength phases formed in Magnesium oxysulfate cement[20-22].

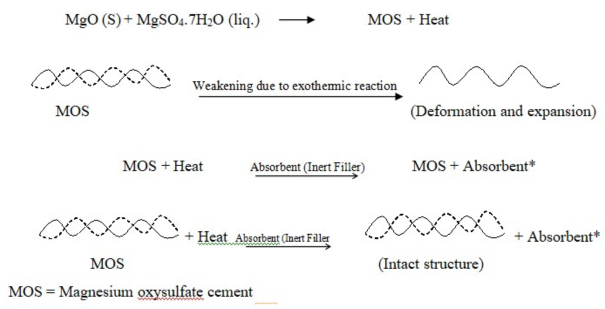

Fig. 1. Schematic presentation

The reactions of formation of 3.1.8 and 5.1.8 form of Magnesium oxysulfate cement are given below:

$$MgO + H_2O \longrightarrow Mg(OH)_2$$
$$3Mg(OH)_2 + MgSO_4 + 8H_2O \longrightarrow 3Mg(OH)_2.MgSO_4.8H_2O$$
$$5Mg(OH)_2 + MgSO_4 + 8H_2O \longrightarrow 5Mg(OH)_2.MgSO_4.8H_2O$$

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. Harmful impurities present in the raw materials responsible for degradation of the product. Active lime (CaO) is always present in the form of impurity in the raw materials, which is harmful for Magnesium oxysulfate cement. Because Calcium oxide readily react with water and forms Calcium hydroxide. During this process, a large amount of heat is evolved which converts water into steam. Therefore cracks produce in the cement and degrade its water resistance and compressive strength.

The incorporation of additive is considered to be one of the most useful method to improve the performance of cementitious materials. Suitable admixtures and their optimum proportions in the matrix can overcome these problems either by nullify the harmful effects of the impurities present in the raw materials or by forming additional bond in the matrix. Soapstone industry is one of the leading stone industries in India. Soapstone is a type of metamorphic rock. It is a soft, dense (more dense than marble, slate, lime stone & graphite), non porous, very easy to carve, non absorbent, heat resistant, high specific heat capacity compound and it is resistant to acids and alkalis. It is impenetrable and will not stain. A lot of waste (solid and slurry form) produced during mining and cutting of Soapstone. Due to excellent chemical and physical properties of Soapstone, the powder waste of Soapstone industry can useful for increasing moisture sensitivity and compressive strength of Magnesium oxysulfate cement.

MATERIALS AND METHODS

The raw materials used in the study were lightly calcined Magnesite (Magnesia), technical grade Magnesium sulfate heptahydrate and Dolomite powder (inert filler).

- ***Magnesium oxide* (MgO):** Commercial grade Magnesia used in the study. It was collected from Suksha export, Bagru, jaipur, Rajasthan. The chemical composition of MgO is: MgO = 82.70%, SiO_2 = 8.51%, CaO = 2.80%, Fe_2O_3 0.12%, Al_2O_3 = 0.07%, Loss on Ignition (LOI) = 4.40%.
- ***Magnesium sulfate* ($MgSO_4.7H_2O$):** 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, Rajasthan. Its chemical composition is: $MgSO_4$ = 96.80%, Fe_2O_3 = 0.02%, Al_2O_3 = 0.07%, CaO = 1.40%, Moisture= 0.98%, Acid Insoluble = 0.11%.
- ***Dolomite*:** Waste material of Dolomite mines (Dolomite dust produced during cutting and shaping etc.) was used as an inertfiller[28]. It was collected from Matasya Industrial Area, Alwar, Rajasthan. The chemical composition of Dolomite is : SiO_2 = 5.06%, CaO = 29.40%, MgO =19.50%, Fe_2O_3 = 0.82%, Al_2O_3 = 0.23%, LOI = 44.50%, $CaCO_3$ = 52.50%, $MgCO_3$ = 40.95%, Brightness = 93.00%, Whiteness = 95.30%.
- ***Soapstone:*** The chemical composition of Soapstone powder is SiO_2 = 62.20%, CaO = 1.12%, MgO = 30.2%, Fe_2O_3 = 0.94%, Al_2O_3 = 0.30%, Loss on Ignition = 4.82%.

Pre experimental planning

- ***Preparation of dry-mixes:*** Dry-mixes were prepared by mixing equal amounts of lightly calcined Magnesium oxide and Dolomite in the ratio of 1:1 by their weights.
- ***Preparation of gauging solution:*** Technical grade Magnesium sulfate was dissolved in luke warm water to make a saturated solution. This solution is allowed to stand over night so that insoluble impurities settled down at the bottom of the container. The supernatant saturated solution is filtered with help of vacuum pump. This solution is known as gauging solution for oxysulfate cement. The concentration of gauging solution is determined in terms of degree on Baume scale with the help of Baume hydrometer. The higher the degree on Baume scale, the higher will be density and concentration of the gauging solution.
- ***Preparation of the wet-mix:*** Gauging solution was added in the dry-mix (with different percentages of soapstone powder) to form a wet-mix of workable consistency according to Indian Standard.
- All the experiments were performed on the best composition of Magnesium oxysulfate cement (MgO : Dolomite was in 1:1 proportion and density of gauging solution was 25^0Be) under identical conditions of temperature (30^0C) and humidity (above 90%). Following experiments were conducted to investigate the influence of soapstone powder on Magnesium oxysulfate cement. All the experiments repeated in three times and then average is reported.

- **Setting (Hardening) time investigation:** The effect of soapstone powder on setting characteristics of Magnesium oxysulfate cement was studied by admixing soapstone powder in the dry-mix in varying proportion. The amount of powdered form of additive was calculated by weight of Magnesia. Wet-mixes were prepared by gauging 1:1 dry mixes (by weight of Magnesia and Dolomite) having different quantities of additive (5%, 10%, 15% & 20%) with Magnesium sulfate solution of 25^o Be. Standard procedures were adopted according to Indian Standard specification to determine standard consistency, Initial and final setting times using Vicat needle apparatus[28]. Results are summarized in Table 1.

- ***Weathering effect:*** Standard blocks prepared for setting time investigation were used to determine the effect of weather. Variation in weights of blocks was measured with passage of time after 24 hrs, 7days, 30 days and 45 days using chemical balance. Experimental findings are recorded in Table 2.
- ***Moisture ingress test***: The effect of soapstone powder on soundness of the product was studied by performing steam test. For this all setting time blocks with different amounts of soapstone powder were first cured for 60 days under identical condition and then were exposed to boiling water for at least 30 hours in a closed steam bath. Their relative moisture efficacies were studied as a function of time. Moisture ingress and soundness are inversely proportional. Results are shown in Table 3.
- ***Compressive strength:*** To study the effect of Soapstone on compressive strength of Magnesium oxysulfate, standard 50cm^2 cubes (70.6mmX70.6mmX70.6mm) were prepared from the standard consistency pastes having Soapstone powder in

different amounts. These cubes were tested after 30 days of curing under identical conditions with the help of compressive strength testing machine as per standard procedure. Results are recorded in Table 4.

- ***Linear Changes:*** Standard size blocks (200mmX25mmX25mm) were prepared by wet-mixes having varying quantities of additive in order to study the effect of soapstone on linear changes of Magnesium oxysulfate. Trial beams were measured after 24 hours using micrometer scale. After 28 days of curing, under identical conditions, final lengths of the beams were determined. The difference of the two readings shows the linear change (expansion/contraction). If the difference in less, more will be the soundness of the product. Results are summarized in Table 5.

RESULTS AND DISCUSSION

Table 1. Effect of Soapstone on Setting characteristics of Magnesium oxysulfate cement

% of Soapstone in dry-mix Composition	Setting time	
	Initial (min)	Final (min)
0%	70	210
5%	39	142
10%	38	130
15%	35	122
20%	32	117

Incorporation of soapstone powder in the matrix decreases initial & final setting times (Table1). As the amount of the additive increases, time of setting (initial & final) continuously decreases. Active lime present in the raw materials, forms calcium hydroxide and produces heat (the amount of the heat is so high that incorporated dolomite not fully absorbed this heat, if amount of dolomite is increased in the dry-mix then amount of MgO reduces which is one of the essential component of Magnesium oxysulfate cement, hence it is not worthful. This heat accelerates the initial as well as final setting. Active silica present in the soapstone reacts with active lime and form Calcium silicate, which is inactive. As the amount of soapstone, deactivation of active lime also increases, therefore decrease in initial & final setting period is observed.

Table 2. Effect of Soapstone on weathering characteristics of Magnesium oxysulfate cement

% of Soapstone in dry-mix Composition	Weight of blocks in gm after			
	24 hrs	7 days	30 days	45 days
0%	259.05	256.39	252.87	250.07
5%	257.43	253.82	250.57	248.80
10%	252.53	245.04	241.55	239.92
15%	251.88	244.45	240.10	239.60
20%	248.27	242.70	239.42	237.93

Incorporation of soapstone powder reduces the weights of the trial blocks with time as noticed up to 45 days of the observation periods. This is attributable mainly due to the moisture present in the matrix which slowly evaporates with time causing decrease in the weights (Table 2).

Table 3. Effect of Soapstone on water resistance property of Magnesium oxysulfate cement

% of Soapstone in dry-mix composition	**Trial blocks kept in boiling water for**					
	0-5 hrs	**5-10 hrs**	**10-15 hrs**	**15-20 hrs**	**20-25 hrs**	**25-30 hrs**
0%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
5%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
10%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
15%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
20%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.

N.E. = No Effect

Table 4. Effect of Soapstone on compressive strength of Magnesium oxysulfate cement

% of Soapstone in dry-mix composition	Compressive strength (kg/cm^2)
0%	260.530
5%	276.553
10%	296.593
15%	300.605
20%	306.533

It is represented in Table 3 that the incorporation of soapstone increases water tightness of the product. Incorporation of the additive deactivates active lime and this positively affects the water tightness of the product. The superior qualities of soapstone powder (dense, non porous, non absorbent, impenetrable, etc.) in the matrix, responsible for decrease in moisture sensitivity of Magnesium oxysulfate cement. Table 4 represents the effect of soapstone on compressive strength of the product. When the amount of the additive increases, deactivation of active lime increases, hence mechanical strength of the product also increases. Ca^{++} and Mg^{++} are bivalent II A group elements. Ca^{++} can replace some Mg^{++} in Magnesium oxysulfate. Mixed Mg-Ca oxysulfate is big in size (Ca^{++} ions are big). Also Calcium oxysulfate is water soluble. These factors lead to the weakening of the structure. It is made inert (out of phase) by the incorporation of silica of soapstone, hence the chances of mixed crystal formation are eliminated and the strength remains intact. Further active lime in its inert form will absorb heat of reaction by way of three body collision mechanism and hence will contribute to the soundness of the structure, hence almost insignificant linear changes were observed (Table 5).

Table 5. Effect of Soapstone on linear changes of Magnesium oxysulfate cement

% of Soapstone in dry-mix composition	Lengths of Beams		Change in Length (mm)
	Initial	Final	
0%	200.0	199.980	0.020
5%	200.0	199.982	0.018
10%	200.0	199.985	0.015
15%	200.0	199.990	0.010
20%	200.0	200.093	0.007

The above discussion can be explained by following chemical changes:

$$CaO + H_2O \longrightarrow Ca(OH)_2 \text{---------------------------(i)}$$

$$MgO + H_2O \longrightarrow Mg(OH)_2 \text{--------------------------(ii)}$$

$$CaO + SiO_2 \longrightarrow CaSiO_3 \text{----------------------------(iii)}$$

Active Inactive

$$3Mg(OH)_2 + MgSO_4 + 8\ H_2O \longrightarrow 3Mg(OH)_2.MgSO_4.8H_2O \text{-----(iv)}$$

(3.1.8 Form)

$$5Mg(OH)_2 + MgSO_4 + 8H_2O \longrightarrow 5Mg(OH)_2.MgSO_4.8H_2O \text{-----(v)}$$

(5.1.8 Form)

(Strength giving composition)

CONCLUSION

Experimental finding concluded that Soapstone powder decreases setting time (both initial and final) and improves water resistance property of Magnesium oxysulfate cement. Soapstone powder increases compressive strength of the Magnesium oxysulfate cement. Almost insignificant linear changes were observed after adding soapstone powder in the matrix.

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