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Investigations of Some Properties of Oxysulphate of Magnesium by Admixing Zinc Oxide as an Additive

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Abstract: ***A French scientist Stanislas Sorel discovered Magnesium oxysulphate cement in 1867. This non hydraulic ecofriendly cement is formed by the reaction between Magnesium oxide and Magnesium sulphate. Dolomite is used as an inert filler to absorb excess heat evolved during the reaction between the two. It is used for grindstone, tiles, artificial stone, cast floors, light weight insulating pannels etc. It's resistance to abrasion is about 1.5 times to that of Portland cement. Incorporation of additives may improve the properties of Magnesia cement by nullifying the harmful effects of the impurities present in the matrix. In this study ZnO is used as an additive and findings are very encouraging. Incorporation of different proportions of ZnO in Magnesia cement gradually decreases the setting periods, improves water tightness and compressive strength. Minor contraction in the linear beams are also noticed with passage of time.***

Keywords: Magnesium oxysulphate cement, Setting periods, Compressive strength, Moisture ingress, Weathering effects, Linear changes.

I. INTRODUCTION

Some materials that produces or promotes cohesion in loosely assembled substances are known as binders .Chemical binders are the substances which can be used directly or indirectly for binding, adhering or fastening substances[1].Glue, gum etc. are organic binders which are known as adhesives and inorganic binders are known as cement[2]. Polymerization tendencies or interlocking crystal habits are actually responsible for their cementing behaviour[3]. Magnesium oxysulphate cement (also known as sorel's cement) was produced by a French scientist Stanislas sorel in 1867[4]. It has many superior properties to that of Portland cement. The resistance of Magnesium oxysulphate cement to abrasion is about 1.5 times to that of Portland cement[5]. It is a promising material for fire proofing and insulation applications[6]. It has no corrosion to steel and effective inhibition of the frost[7]. In USA and Europe the major use of Magnesium oxysulphate cement is in the manufacture of light weight insulating panels[8]. It is also used as binder in flooring materials. It is formed by mixing proper ratios of MgO powder with a concentrated solution of Magnesium sulphate[9]. The reaction between Magnesite powder and Magnesium sulphate solution is exothermic and thus heat evolved during the reaction make cracks and form the product unsound. Inert filler dolomite was mixed in the matrix to absorb the heat[10]. The formation process does not require any type of energy whether heat or light for its setting process making it an ecofriendly cement[11].
Different compositions of oxysulphate cement are known but 5,1,8 form is most commonly found.

$$MgO+H_2O \longrightarrow Mg(OH)_2$$

$$3Mg(OH)_2+MgSO_4.7H_2O \xrightarrow{H_2O} 3Mg(OH)_2.MgSO_4.8H_2O \quad (3,1,8)$$

$$5Mg(OH)_2+MgSO_4.7H_2O \xrightarrow{H_2O} 5Mg(OH)_2.MgSO_4.8H_2O \quad (5,1,8)$$

Additives may play an important role to modify the properties of oxysulphate cement. In this study ZnO is tried as an additive in Magnesium oxysulphate cement to improve its qualities.

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II. MATERIALS

Following three raw materials were used in this study:-

A. *Magnesium Oxide*[12]

Commercial grade Magnesia (lightly calcined) used in this study. The analysis of Magnesite powder is SiO_2=8.51%, CaO=2.80%, MgO=82.70%, Fe_2O_3=0.12%, Al_2O_3=0.98%, LOI=4.40%.

B. *Magnesium sulphate (Epsom salt)*[13]

Technical grade magnesium sulphate used in the formation of Magnesium oxysulphate cement. The chemical composition of Epsom salt is $MgSO_4$=96.80%, Fe_2O_3=0.02%, Al_2O_3=0.07%, CaO=1.40%, Acid insoluble=0.11%, moisture=0.98%.

C. *Dolomite*[14]

Commercial grade dolomite used in this study. 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%.

III. METHOD

Following five experiments were conducted to investigate the influence of ZnO on Magnesium oxysuphate cement.

A. Setting Time Investigation

The effect of ZnO on setting characteristics of Magnesium oxysulphate cement was studied by admixing powdered ZnO in the dry-mix in varying proportion of powdered form of additive was calculated by weight of Magnesia. Wet-mixes were prepared by gauging 1:2 dry mixes (by weight of Magnesia and Dolomite) having different quantities of additive with Magnesium sulphate solution of 25° Be. The volume of gauging solution was kept constant for each lot of dry- mix. Standard procedures were adopted according to IS specification to determine standard consistency, Initial and final setting times using Vicat needle apparatus[15]. Results are summarized in table I.

B. Weathering Effect

Standard blocks prepared for setting time investigation were used in this test. Variation in weights of blocks was measured with passage of time after 24 hrs, 7days, 15 days, 30 days and 45 days using chemical balance. Experimental findings are recorded in table II.

C. Moisture Ingress Test

The effect of ZnO on soundness of the cement was studied by performing steam test[16]. For this all setting time blocks with different amounts of Zinc oxide were first cured for 60 days under identical condition and then were exposed to boiling water for atleast 30 hours in a closed steam bath. Their relative moisture efficacies were the studied as a function of time. Moisture ingress and soundness are inversely proportional. Results are shown in table III.

D. Compressive Strength

To study the effect of ZnO on compressive strength of oxysulphate cement, standard 50cm^2 cubes (70.6mmX70.6mmX70.6mm) were prepared from the standard consistency pastes having ZnO 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 IV.

E. Linear Changes

Standard size blocks (200mmX25mmX25mm) were prepared by wet-mixes having varying quantities of additive in order to study the effect of ZnO on linear changes of Magnesium oxysulphate cement. 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 show the linear change. It the difference in less, more will be the soundness of the product. Results are recorded in table V.

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IV. RESULTS AND DISCUSSIONS

From table I it is clear that incorporation of ZnO in the matrix decreases initial setting times and increases final setting periods. Impurities like active lime etc. present in the matrix promotes the setting process so initial setting time decreases. With the passage of time ZnO converts in $ZnSO_4$ after reacting with Magnesium sulphate (eq. 2) and then these sulphate salt ($ZnSO_4$ & $MgSO_4$) converts active lime into inactive form (eq.3 & eq.5) and along this simultaneous formation of Zinc oxysulphate takes place (eq. 10) which is a slower process. These two factors increase the final setting periods. With passage of time formation of interlocking / interlacing crystals of Zinc oxysulphate and Magnesium oxysulphate takes place. When percentage of additive increases, the formation of interlocking / interlacing crystals of Zinc oxysulphate and Magnesium oxysulphate also increases therefore final setting time decreases.

From table II it is clear that incorporation of ZnO reduces the weight of the trial blocks with time upto 30 days of the observation periods. This is attributable mainly moisture present in the matrix which slowly evaporates with time causing decrease in the weights.

It is represented from table III that the incorporation of additive increases watertightness of the product. Inactivation of harmful impurities like active lime (eq.3and eq.5) and simultaneous formation of strength giving zinc oxysulphate (eq.10) are actually responsible for the soundness (water tightness) of the product.

It is clear from table IV that Strength is increased gradually with increasing quantities of ZnO in the matrix. Simultaneous formation of intercrossing/interlacing crystals of Magnesium oxysulphate and Zinc oxysulphate takes place. Such a crosslinking often results in the betterment of physical properties of the cement as per the principles of polymer science hence strength increases.

Table V shows the effect of ZnO on linear changes of Magnesium oxysulphate cement. Almost insignificant contractions in the trial beams were observed when ZnO is used as an additive. Sound structure formed as a result of interlacing crystals of zinc oxysulphate and sorel's cement leaves little ground for volume changes. Minor shrinkage effects observed may be explained on account of gradual loss of moisture with lapse of time.

The above discussion can be interpreted on the basis of the following chemical changes :-

$$CaO/MgO+H_2O \longrightarrow Ca/Mg(OH)_2 \quad \text{----------(1)}$$

$$ZnO+MgSO_4 \longrightarrow ZnSO_4.MgO \quad \text{----------(2)}$$

$$ZnSO_4+CaO+3H_2O \longrightarrow CaSO_4.2H_2O+Zn(OH)_2 \quad \text{---------- (3)}$$

Active → Inactive

$$ZnSO_4+MgO+H_2O \longrightarrow MgSO_4+Zn(OH)_4 \quad \text{----------(4)}$$

$$MgSO_4+CaO+3H_2O \longrightarrow CaSO_4.2H_2O+Mg(OH)_2 \quad \text{---------- (5)}$$

Active → Inactive

$$ZnO+H_2O \longrightarrow Zn(OH)_2 \quad \text{----------(6)}$$

$$ZnO+MgSO_4+2H_2O \longrightarrow Zn(OH)_2+H_2SO_4+MgO \quad \text{----------(7)}$$

$$MgO+H_2SO_4 \longrightarrow MgSO_4+H_2O \quad \text{----------(8)}$$

$$Mg(OH)_2+ZnSO_4 \longrightarrow Zn(OH)_2+MgSO_4 \quad \text{----------(9)}$$

$$xZn(OH)_2+yZnSO_4+zH_2O \longrightarrow xZn(OH)_2.yZn(SO_4)_2.zH_2O \quad \text{---------(10)}$$

Zinc oxysulphate

$$5Mg(OH)_2+MgSO_4+8H_2O \longrightarrow 5Mg(OH)_2.MgSO_4.8H_2O \quad \text{----------(11)}$$

Magnesium Oxysulphate cement strength giving composition

Table I. Effect of ZnO on setting characteristics of oxysulphate cement.

Conc. Of gauging solution=25°Be	Dry-Mix Composition ..1:2
Vol. of g.s.=74.0ml	a) One part by weight of magnesia
Temparature =30± 2°c	b) Two part by weight of dolomite + additive
Relative humidity ….above 90%	Quantity of dry-mix…..200gm.

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S.NO.	DRY-MIX COMPOSITION (% ADDITIVE)	SETTING TIME	
		INITIAL (MIN.)	FINAL (MIN.)
1	0%	70	210
2	5%	69	263
3	10%	65	248
4	15%	58	242
5	20%	57	240

g.s. = gauging solution

Table II. Effect of ZnO on weathering characteristics of oxysulphate cement.

Conc. Of gauging solution=25ºBe
Vol. of g.s.=74.0ml
Temparature =30± 2ºc
Relative humidity ….above 90%

Dry-Mix Composition …..1:2
(a) One part by weight of magnesia
(b) Two part by weight of dolomite + additive
Quantity of dry-mix…..200gm.

S. NO.	DRY-MIX COMPOSITION (% ADDITIVE)	WEIGHT OF BLOCKS IN GM AFTER				
		1 DAY	7DAYS	15DAYS	30DAYS	45 DAYS
1	0%	259.05	255.39	253.11	252.87	252.07
2	5%	272.95	262.67	260.29	259.15	257.93
3	10%	259.97	250.43	247.57	246.63	245.48
4	15%	262.88	253.43	251.20	250.24	249.09
5	20%	265.14	255.40	253.09	252.14	250.92

Table III. Effect of ZnO on moisture ingress characteristics of oxysulphate cement.

Conc. Of gauging solution=25ºBe
Vol. of g.s.=74.0ml
Temparature =30± 2ºc
Relative humidity ….above 90%

Dry-Mix Composition …..1:2
(a) One part by weight of magnesia
(b) Two part by weight of dolomite + additive
Quantity of dry-mix…..200gm.

S.NO.	DRY-MIX COMPOSITION (% ADDITIVE)	TRIAL BLOCKS KEPT IN BOILING WATER FOR					
		10-5 HRS	5-10 HRS	10-15 HRS	15-20 HRS	20-25 HRS	25-30 HRS
1	0%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
2	5%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
3	10%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
4	15%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.
5	20%	N.E.	N.E.	N.E.	N.E.	N.E.	N.E.

g.s.=gauging solution, N.E.= No Effect

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Table IV. Effect of ZnO on compressive strength of oxysulphate cement.

Conc. Of gauging solution=25°Be
Vol. of g.s.=74.0ml
Temparature =30± 2°c
Relative humidity ….above 90%

Dry-Mix Composition …..1:2
(a) One part by weight of magnesia
(b) Two part by weight of dolomite + additive
Quantity of dry-mix…..565gm.

S. No.	Dry-mix composition (% additive)	Compressive Strength in Kg/cm^2
1	0%	260.53
2	5%	382.77
3	10%	418.84
4	15%	456.91
5	20%	492.99

Table V. Effect of ZnO on linear changes of oxysulphate cement.

Conc. Of gauging solution=25°Be
Vol. of g.s.=74.0ml
Temparature =30± 2°c
Relative humidity ….above 90%

Dry-Mix Composition …..1:2
(a) One part by weight of magnesia
(b) Two part by weight of dolomite + additive
Quantity of dry-mix…..200gm.

S.NO.	DRY-MIX COMPOSITION (% ADDITIVE)	LENGTH OF BEAM (MM)		CHANGE LENGTH IN (MM)
		INITIAL	FINAL	
1	0%	200.00	199.98	0.020
2	5%	200.00	199.966	0.034
3	10%	200.00	199.960	0.040
4	15%	200.00	199.962	0.038
5	20%	200.00	200.042	0.042

V. CONCLUSION

A. ZnO decreases initial setting periods but increases final setting periods.
B. Incorporation of ZnO improves watertightness and strength of the cement.
C. It is a good additive for Magnesium oxysulphate cement.

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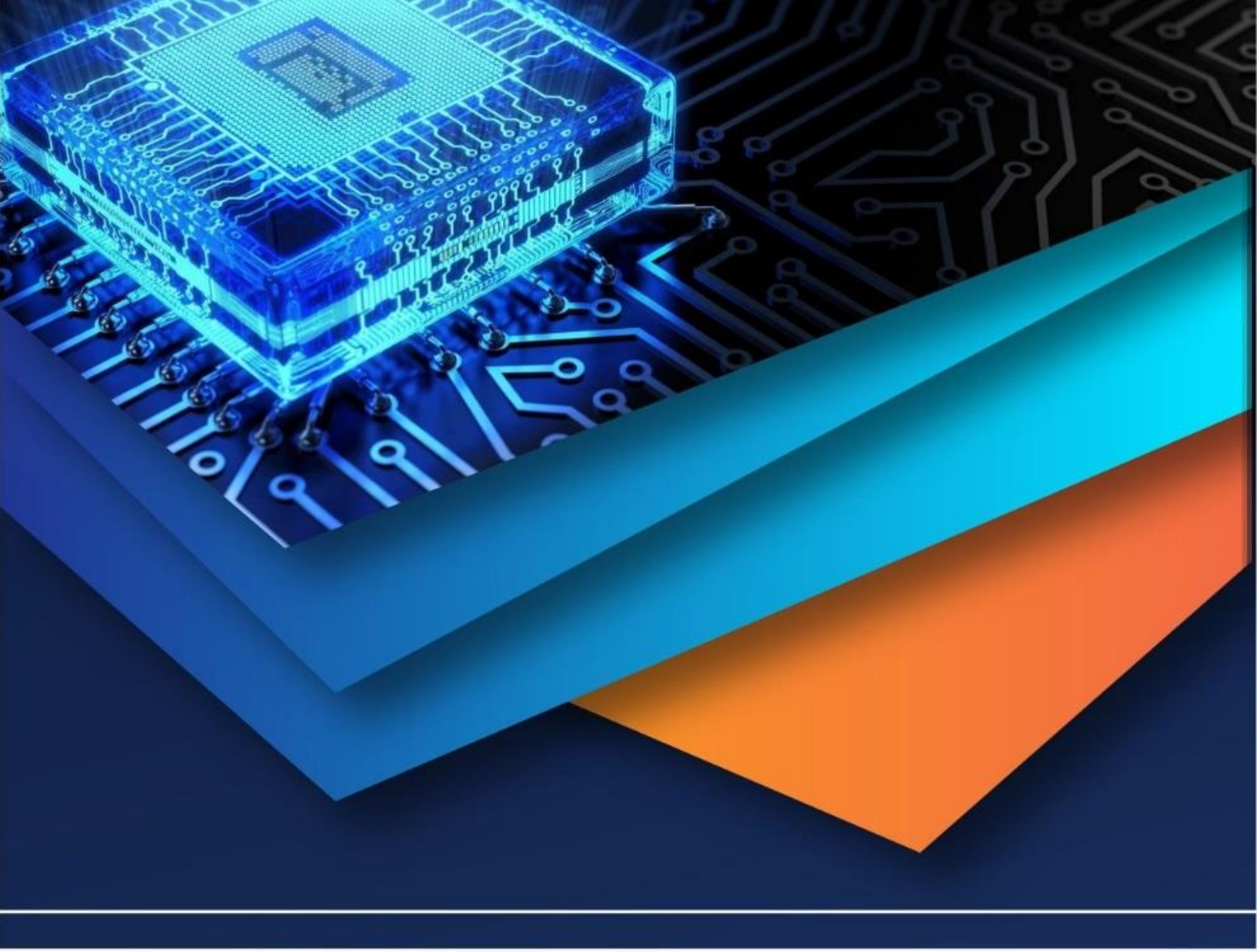

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