MAGNESIUM OXYSULPHATE CEMENT: CHANGE IN PROPERTIES ON ADMIXING SODIUM BICARBONATE AS AN ADDITIVE

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ABSTRACT

Sodium bicarbonate plays an important role, after admixing in Magnesium Oxysulphate Cement (Sorel's Cement), on strength and durability of the product. It's incorporation in the matrix as additive in powerded form as well as saturated solution is studied in detail. Powdered form of $NaHCO_3$ increases the compressive strength and watertightness of the product, whereas its incorporation as saturated solution does not contribute to strength and durability of Sorel's Cement. setting periods, weathering effects and linear changes were also studied for both the forms of the additive.

Keywords : *Magnesium Oxysulphate Cement, Sorel's Cement, Compressive Strength, Moisture ingress, Setting periods, Weathering effects, Linear changes.*

INTRODUCTION

Chemical binders are the substances, which can be used directly or indirectly for binding, adhering or fastening substances. Organic binders are normally known as adhesives while inorganic binders are often regarded as cements. Polymerization tendencies or interlocking crystal habits are actually responsible for cementing behaviour[1]. Sorel S.T., a French scientist, discovered Magnesium Oxysulphate Cement in 1867[2-5]. Oxysulphate cement is formed by the interaction of Epsamite with magnesia in aqueous solution to give a gelatinous composition of Magnesium Oxysulphate (possible compositions are 3,1,8 and 5,1,8). But $5Mg(OH)_2.MgSO_4.8H_2O$ composition is the most commonly found chemical phase[6-9].

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

$$3Mg(OH)_2 + MgSO_4.7H_2O + H_2O \rightarrow 3Mg(OH)_2.MgSO_4.8H_2O$$

$$5Mg(OH)_2 + MgSO_4.7H_2O + H_2O \rightarrow 5Mg(OH)_2.MgSO_4.8H_2O$$

Magnisium Omysulphate Cement has versatile cementing properties, superior to those of Portland Cement [10,11]. The strength and durability of Sorel's cement can be improved by using high grade raw materials, proper proportions, workmanship, conditions of curing (low temperature / high humidity), post curing measures etc[12-15]. Suitable admixtures and their optimum proportions in the matrix nullify the harmful effects of the impurities present in the raw materials and hence contribute to the soundness of the cement. In this study, sodium bicarbonate is tried as an additive in Magnesium Oxysulphate cement to improve its quality.

The chemical composition of commercial grade sodium bicarbonate is $NaHCO_3$[16,17] . In this study it has been used as an additive for magnesia cement in powdered form as well as saturated solution. The anionic part of sodium bicarbonate (HCO_3^-ion) reacts with active lime and other harmful impurities and forms an inactive insoluble phase[18]. Thus the harmful effects of these impurities on the quality of Magnesium Oxysulphate cement are minimized.

EXPERIMENTAL

Raw Materials:

The raw materials required in the present study are magnesia (commercial grade) , magnesium sulphate (Epsom salt) and inert filler (dolomite) [19].

(a) *Magnesia* : Commercial grade magnesia, used in the study is of Salem origin with following characteristics [20] :92% MgO, CaO < 1.9%, Si_2O_3 < 8.2% , Al_2O_3 0.2 – 0.4%, Fe_2O_3 < 1.4%, Ignition loss 2.5% ± 0.5% at 110 ^{0}C, Bulk density 0.85 kg /I, 95% passing through 75 micron IS sieve.

(b) *Magnesium Sulphate (Epsom Salt) $MgSO_4.7H_2O$* : Technical grade Epsom salt used in the formation of Magnesium Oxysulphate cement is strictly according to IS specifications of under given grading [21] : magnesium sulphate > 9.8%, Chloride (as Cl) 1% by mass, Lead (as Pb) in traces, Arsenic As_2O_3 in traces, Iron (as Fe) 70 ppm, Zinc and matter insoluble in water 0.20% by mass.

(c) *Inert filler (Dolomite)* : Incorporation of dolomite in the matrix absorbs the heat evolved during the exothermic formation of Magnesium Oxysulphate cement and hence reduces thermal shocks in the cement. Dololmite of following grading was used – 100% passing through 150 micron IS sieve, 50% retained on 75 micron IS sieve, 28.7% CaO, 20.8% MgO, insoluble and other sesquioxide impurities < 1% and loss on ignition 50%[22].

Procedure:

Experiments were conducted to investigate the way in which incorporation of sodium bicarbonate influences setting characteristics, weathering effects, watertightness, compressive strength and linear changes of oxysulphate cement[23,27,28].

(i)Setting Characteristics **:** The effect of sodium bicarbonate on setting characterstics of the cement was studied by admixing powdered sodium bicarbonate in the dry mix as well as by adding saturated solutions of sodium bicarbonate in the gauging solution in varying proportions (0%, 5%, 10%, 15%, 20%) (separately in two series of trials). The quantity of powdered form of additive was calculated by weight of magnesia and that of solution by volume of gauging solution. Wet mixes were prepared by gauging 1:2 dry mixes (by weight of magnesia and dolomite) having different quantitites of additive with magnesium sulphate solutions of 25^0Be. The volume of gauging solutions 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[23] .Results are tabulated in Tables 1 and 2.

(ii) Weathering Effects **:** Investigations were also made pertaining to the variation in weights of the setting time blocks (with varying % of additive) with passage of time after 24 hrs, 7 days, 30 days and 45 days using chemical balance. Weight of the blocks may decrease or increase with time due to different weathering effects promoted by the additive. Experimental findings are recorded in Tables 3 and 4.

***(iii) Steam Test (Moisture Ingress)* :** The effect of sodium bicarbonate on the soundness of the cement is studied by performing steam test. For this all the setting time blocks with different amounts of sodium bicarbonate (powdered and saturated solution) were first cured for 60 days under identical conditions and then were exposed to boiling water for atleast 30 hours in a closed steam bath. These investigations are desirable in order to evaluate the moisture sealing efficacies of the trial blocks. Relative water vapour transmission has been expressed as a function of time[24,25] . The results are summarized in Tables 5 and 6.

***(iv) Compressive Strength* :** To study the effect of sodium bicarbonate (powder and saturated solution) on compressive strength of oxysulphate cement, it was added in different proportions in the dry mix (by weight of magnesia in case of powdered additive) and in the gauging solution (by volume of gauging solution in case of saturated solution of additive) respectively (separately in two series of trials). Wet mixes prepared by gauging 1:2 dry mixes (by weight of magnesia and dolomite) having varying quantities of additive with constant volume of magnesium sulphate of 25^{o}Be. Standard sized trial blocks (70.6 mm × 70.6mm × 70.6 mm) were prepared from these wet mixes. These moulds were allowed to cure under identical conditions for 30 days and then subjected to compressions (on compressive strength testing machine) just sufficient for their rupture as per standard procedure [15,23]. Results are recorded in Tables 7 and 8.

***(v) Linear Changes* :** Standard sized moulds (200 mm × 25 mm × 25mm) were filled by wet mixes having varying quantities of additive (powdered and saturated solutions) in order to study the effect of sodium bicarbonate on linear changes of oxysulphate cement [26]. Trial beams were kept under 90% relative humidity and 30 ± 2^{0}C temperature for 24 hrs. Initial lengths of the beams were measured after 24 hrs 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. If the difference is less, more will be the soundness of the product. Results are enumerated in Table 9 and 10.

RESULTS AND DISCUSSION

Tables 1 and 2 summarize the effect of increasing quantities of sodium bicarbonate (powder and solution) on setting characteristics of Oxysulphate Cement respectively. Trends in setting process in both the cases are found different . In case of powdered additive, initial as well as final setting periods of Magnesium Oxysulphate cement increase with the increasing quantities of the additive. This is due to the decrease in availability of magnesium oxide in the matrix, because bicarbonate ions react with di and trivalent cations (Ca^{+2}, Mg^{+2}, Al^{+3} etc) which results in the formation of insoluble and inactive phases of their carbonates (Eq1-6 & 11). This decreases the active quantities of strength giving component i.e. magnesium oxide available in the matrix. Such a situation retards the setting process. It is also gathered that when sodium bicarbonate saturated solution is used as an additive (Table 2) , initial setting period decreases with increasing quantities of the additive while final setting period increases remarkably with increasing quantities of the additive. Decreasing initial setting periods, when solutions of additive is used, can be explained on the basis of the prompt reactivity of bicarbonate anions with the magnesia and its ingredients. Such reaction results in the formation of insoluble solid phases of metal carbonates (Eq 1-6, 8,11) . Their formation resists the initial setting needle touching the base of Vicat mould. Hence, their formation in increasing quantities is witnessed by a gradual decrease in initial setting periods. However these solid phases are not cementing or strength giving compositions. Hence increasing final setting periods with increasing quantities of sodium bicarbonate solution or decreasing quantities of magnesium sulphate in the gauging solution are expected on account of parallely decreasing chances of the formation of strength giving compositions.

The effect of sodium bicarbonate (powder and saturated solution) as an additive on weathering characterstics of Oxysulpahte cement is shown in Tables 3 and 4. It is noted that incorporation of sodium bicarbonate in any form reduces the weights of the trial blocks with time up to about 30 days of the observation period. This is attributable mainly to the uncombined free moisture present in the matrix which slowly evaporates with time causing decrease in the weights. Observations recorded on the 45th day reveal almost insignificant decrease or increase in weights. This shows that major reactions involving cement formation are almost over by that period. Minor variations in weights may be either due to decomposition of bicarbonates (decrease in weight or due to rehydration or carbonation (increase in weight) of the matrix (Eq 8,9)

Tables 5 and 6 reveal the effect of sodium bicarbonate (powder and solution) as an additive on moisture ingress characterstics of Oxysulphate cement. It is noted that by the incorporation of powdered additive, watertightness of the product increases. Inactivation of harmful impurities like active lime and simultaneous formation of inert phase like basic magnesium carbonate etc contribute to watertightness (Eq 1-6). However in case when sodium bicarbonate solution is used as an additive (Table 6), 5% and 10% additive show very poor watertightness, whereas watertightness is improved by increasing proportions of additive (15% & 20%). Plausible explanation for this behaviour is that in case of 5% and 10% additive, decomposition of sodium bicarbonate in presence of the gauging solution, which is slightly acidic (salt of strong acid and week base), occurs and carbondioxide is evolved internally in the matrix (Eq 10). Evolved gas remains entrapped in the form of microbubbles. When such a product is exposed to steam / boiling water, these microbubbles expand fast posing internal strains in the structure and reduce watertightness of the product. Incorporation of saturated sodium bicarbonate solutions in higher quantities improves watertightness of the product on account of increasing chances of the formation of water insoluble carbonates as solid phases in the matrix.

Tables 7 and 8 show the effect of sodium bicarbonate (powder and solution) on compressive strength of Oxysulphate Cement. Compressive strength is improved by the incorporation of additive in case of its powdered form. It is noted that the strength decreases with increasing proportions of the powdered additive under dry conditions, small proportions of the additive inactivates harmful impurities like active lime etc (Eq 1-4). In such a situation compressive strength of the trial blocks is found to increase initially. With subsequent additions of the additive, compressive strength is found to decrease sharply due to the porous structure of the trial blocks on account of the evolving carbondioxide. Incorporation of saturated solution of sodium bicarbonate lowers the compressive strength of the product due to decreasing quantities of magnesium sulphate (strength giving component) in the wet-mix.

The above discussion is further substantiated by the linear change investigations shown in Tables 9 and 10. It is noted that incorporation of sodium bicarbonate causes contraction in the linear beams to some extent. This is accountable on the basis of the conversion of expansive or bulky materials (oxides and hydroxides) into compact and crystalline phases (carbonates).

The above discussion can be explained on the basis of the following chemical reactions –

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad \text{--- 1}$$

$$MgO + H_2O \rightarrow Mg(OH)_2 \quad \text{--- 2}$$

$$\underset{\text{Active}}{Ca(OH)_2} + 2\,NaHCO_3 \rightarrow \underset{\text{Inactive}}{Ca(HCO_3)_2} + 2NaOH \quad \text{--- 3}$$

$$Ca(HCO_3)_2 \rightarrow \underset{\text{Inactive/Insoluble phase}}{CaCO_3} + H_2O + CO_2\uparrow \quad \text{--- 4}$$

$$Mg(OH)_2 + 2\,NaHCO_3 \rightarrow Mg(HCO_3)_2 + 2NaOH \quad \text{--- 5}$$

$$Mg(HCO_3)_2 \rightarrow \underset{\text{Inactive/Insoluble phase}}{MgCO_3} + H_2O + CO_2\uparrow \quad \text{--- 6}$$

$$MgSO_4 + 2NaOH \rightarrow Mg(OH)_2 + Na_2SO_4 \quad \text{--- 7}$$

$$2\,MHCO_3 \rightarrow \underset{\text{Decrease in weight}}{M_2CO_3 + CO_2\uparrow} + H_2O \quad \text{--- 8}$$

$$MO + H_2O / CO_3 \rightarrow \underset{\text{Increase in weight}}{M(OH)_2 / CO_3} \quad \text{--- 9}$$

$$2\,NaHCO_3 + MgSO_4 \rightarrow Na_2SO_4 + MgCO_3 + H_2O + CO_2\uparrow \quad \text{---10}$$

$$X\,Mg(OH)_2 + Y\,Mg(HCO_3)_2 + Z\,H_2O \rightarrow \underset{\text{Basic magnesium carbonate (Solid phase non-setting)}}{X\,Mg(OH)_2.YMgCO_3.Z\,H_2O} + YH_2O + YCO_2\uparrow \quad \text{-- 11}$$

$$5Mg(OH)_2 + MgSO_4.7H_2O + H_2O \rightarrow \underset{\text{Magnesium Oxysulphate Cement (Strength giving compositon)}}{5Mg(OH)_2.MgSO_4.8H_2O} \quad \text{--- 12}$$

Table-1 : Effect of Sodium Bicarbonate (Powder) on Setting Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite + additive
Quantity of dry mix … 200 gm

S.No	Dry Mix Composition (% additive)	Setting Time	
		Initial (Min)	Final (Min)
1	0%	30	133
2	5%	35	295
3	10%	38	318
4	15%	40	340
5	20%	50	350

g.s. = gauging solution

Table -2 : Effect of Sodium Bicarbonate (Saturated Solution) on Setting Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
[g.s. of 25^{O} Be + satd. soln. of addtv.]
Temperature … 30 ± 2^{O}C
Relative humidity …above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b)Two parts by weight of dolomite
Quantity of dry mix …200 gm
Sodium bicarbonate solution…saturated (13^{O} Be)

S.No	Dry Mix Composition (% additive)	Setting Time	
		Initial (Min)	Final (Min)
1	0%	30	133
2	5%	23	165
3	10%	19	184
4	15%	13	193
5	20%	8	203

g.s. = gauging solution

Table-3 : Effect of Sodium Bicarbonate (Powder)on Weathering Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite + additive
Quantity of dry mix … 200 gm

S.No	Dry Mix Composition (% additive)	Weight of Blocks in gm after			
		24 hrs	7 days	30 days	45 days
1	0%	258.870	251.163	243.322	241.440
2	5%	252.680	240.400	234.500	234.600
3	10%	242.400	231.540	226.700	226.950
4	15%	243.500	232.760	228.300	229.250
5	20%	251.220	240.060	234.800	235.180

g.s. = gauging solution

Table-4 : Effect of Sodium Bicarbonate (Saturated Solution) on Weathering Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
[g.s. of 25^{O} Be + satd. soln. of addtv.]
Temperature … 30 ± 2^{O}C
Relative humidity …above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b)Two parts by weight of dolomite
Quantity of dry mix …200 gm
Sodium bicarbonate solution…saturated (13^{O} Be)

S.No	Dry Mix Composition (% additive)	Weight of Blocks in gm after			
		24 hrs	7 days	30 days	45 days
1	0%	258.870	251.163	243.322	241.440
2	5%	254.500	241.930	235.750	235.100
3	10%	238.840	226.420	215.230	215.150
4	15%	255.480	242.880	236.430	236.710
5	20%	240.750	226.250	220.570	220.900

Table -5 : Effect of Sodium Bicarbonate (Powder) on Moisturing Ingress Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite + additive
Quantity of dry mix … 200 gm

S.N	Dry Mix Composition (% additive)	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
1	0%	N.E.	N.E.	N.E.	N.E.	C	----
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, C = Cracked

Table-6 : Effect of Sodium Bicarbonate (Saturated Solution) on Moisturing Ingress Characterstics of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 70 ml
[g.s. of 25^{O} Be + satd. soln. of addtv.]
Temperature … 30 ± 2^{O}C
Relative humidity …above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b)Two parts by weight of dolomite
Quantity of dry mix …200 gm
Sodium bicarbonate solution…saturated (13 ^{O} Be)

S.N	Dry Mix Composition (% additive)	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
1	0%	N.E.	N.E.	N.E.	N.E.	C	----
2	5%	C	----	----	----	----	----
3	10%	C	----	----	----	----	----
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, C = Cracked

Table-7 : Effect of Sodium Bicarbonate (Powder) on Compressive Strength of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 200 ml
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite + additive
Quantity of dry mix … 565 gm

Dry Mix Composition	0%	5%	10%	15%	20%
Compressive Strength (kg/cm^2)	275	375	350	330	286

g.s. = gauging solution

Table 8 : Effect of Sodium Bicarbonate (Saturated Solution) on Compressive Strength of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 200 ml
[g.s. of 25^{O} Be +satd.soln. of addtv.]
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite
Quantity of dry mix … 565 gm
Sodium bicarbonate solution…saturated (13^{O} Be)

Dry Mix Composition	0%	5%	10%	15%	20%
Compressive Strength (kg/cm^2)	275	225	185	210	170

g.s. = gauging solution

Table-9 : Effect of Sodium Bicarbonate (Powder) on Linear Changes of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 105 ml
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2
(a) One part by weight of magnesia
(b) Two parts by weight of dolomite + additive
Quantity of dry mix … 300 gm

S.No	**Dry Mix Composition (% additive)**	**Length of Beams (mm)**		**Change in Length (mm)**
		Initial	**Final**	
1	0%	200.00	200.02	0.02
2	5%	200.00	199.79	0.21
3	10%	200.00	199.81	0.19
4	15%	200.00	199.75	0.25
5	20%	200.00	199.60	0.40

Table-10 : Effect of Sodium Bicarbonate (Saturated Solution) on Linear Changes of Oxysulphate Cement

Conc. Of g.s = 25^{O} Be
Volume of g.s. = 105 ml
[g.s. of 25^{O} Be +satd.soln. of addtv.]
Temperature … 30 ± 2^{O}C
Relative humidity…above 90%

Dry –mix composition …… 1:2

(a) One part by weight of magnesia
(b) Two parts by weight of dolomite
Quantity of dry mix … 300 gm
Sodium bicarbonate solution…saturated (13^{O} Be)

S.No	Dry Mix Composition (% additive)	Length of Beams (mm)		Change in Length (mm)
		Initial	Final	
1	0%	200.00	200.02	0.02
2	5%	200.00	199.50	0.50
3	10%	200.00	199.34	0.66
4	15%	200.00	199.44	0.56
5	20%	200.00	199.53	0.47

g.s. = gauging solution

CONCLUSIONS

1. Sodium bicarbonate increases setting periods in all proportions within the experimental limits except initial setting periods in case of sodium bicarbonate saturated solution.
2. Incorporation of sodium bicarbonate (powder) improves the watertightness and strength of the cement (optimum amount is 5%) . Hence in powdered form, it is a good additive.
3. Sodium bicarbonate added in the form of saturated solution is not a good additive.

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