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Effect of Potassium Oxalate as Admixture on Oxychloride Cement

*1Dr. Vijay Singh Yadav and 2Devdutt Arya

*1, 2Department of Chemistry, RR College, Alwar, Rajasthan, India.

Abstract

Sorel S.T., a French Scientist discovered a non-calcareous Magnesia cement by treated magnesia with aqueous solution of halides and sulphate salts in 1867 which showed cementing characteristics and was even superior than calcareous Portland cement. It is prepared by the combination of magnesium chloride solution with magnesium oxide. Inert filler dolomite is also added in the matrix to absorb the excess heat evolved during the exothermic formation of the cement, which reduces thermal shocks. Additives may alter the properties of the cement. Mixing of Potassium oxalate in Magnesium oxychloride cement (Sorel's cement) as an additive shows very encouraging results. Remarkable increase in strength and watertightness of the cement is noticed with increasing quantities of the additive along with moderation in its setting. Weathering effects also reveal favourable findings. No significant volume changes are noticed on mixing the additive in the matrix.

The effect of admixing of potassium oxalate on oxychloride cement in the matrix has been investigated. It is shown that potassium oxalate decrease the setting process of the cement and improves compressive strength.

Keywords: Setting periods, weathering effects, moisture ingress, compressive strength, linear changes

1. Introduction

Oxychloride cement (magnesia cement) has many superior properties to that of Portland cement (Sorel 1867; Beaudin and Ramachandra 1975; Beaudin *et al* 1977). The chemical composition of the additive or admixture available in commercial grade is (COOK)2.H2O. Little scientific data are available about its effect on oxychloride cement. We have investigated the effect of (COOK) 2.H_2O on oxychloride cement because its anionic part forms inactive insoluble phase with active lime and other harmful impurities. Similarly the cationic part of it is known to be a cementing ingredient with inter-locking chain forming tendencies. This is due to its polyvalent character.

Binding, adhering or fastening processes of the substances can be achieved by chemical binders due to their polymerization tendencies or interlocking crystal habits [1]. Inorganic binders are also known as cements while organic binders are regarded as adhesives. Magnesium oxychloride cement was discovered by Sorel S.T, a French scientist, in 1867 and hence also known as Sorel's cement [2-5]. It is prepared by the interaction of magnesium chloride with magnesia in aqueous solution. Possible compositions of the cement are 3,1,8 and 5,1,8 but the later composition is the most commonly found chemical phase [6-8].

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

$3Mg(OH)_2 + MgCl_2.7H_2O + H_2O \rightarrow 3Mg(OH)_2.MgCl_2.8H_2O$

$5Mg(OH)_2 + MgCl_2.7H_2O + H_2O \rightarrow 5Mg(OH)_2.MgCl_2.8H_2O$

Non calcareous Sorel's cement has versatile cementing properties superior to those of calcareous Portland cement [9, 10]. High grade raw materials, proper workmanship, conditions of curing (low temperature and high humidity) and post curing measures may improve the strength and durability of the cement [11-14]. Some additives may play an important role in further improvement of the soundness of the cement by way of nullifying the harmful effects of the impurities present in the matrix. In this study potassium oxalate has been tried as an additive to improve the quality of Magnesium oxychloride Cement.

2. Materials

The raw materials used in the preparation of Magnesium oxychloride cement are magnesium oxide, magnesium chloride and dolomite (inert filler) having the following characteristics [16].

a) **Magnesia:** Magnesia used in the study was of Salem origin. Magnesium oxide content was more that 90%, loss on ignition was 2 ± 1% and traces of active lime were present. Silica and oxides of III group (Iron and Aluminum) were found to be present in meager amounts [17].
b) **Magnesium Chloride $MgCl_2.7H_2O$:** Technical grade magnesium chloride has been found to be suitable for oxychloride flooring composition according to IS specifications. Magnesium chloride content was found to be more than 98%, sulphate (as SO_4), Lead (as Pb),

***Corresponding Author:** Dr. Vijay Singh Yadav

Arsenic (as As_2O_3), Iron (as Fe), Zinc and matter insoluble in water were found to be present in traces [18].

c) **Dolomite (Inert Filler):** In the preparation of oxychloride cement, locally available unclaimed dolomite powder was used as inert filler passing through 150 micron sieve and retaining 50% on 75 micron sieve. Reactive impurities like active lime were checked before use. Dolomite absorbs the heat evolved during the exothermic formation of Sorel's cement and thus reduces thermal shocks in the structure. It contains 28.7% CaO, 20.8% MgO, insoluble and other sesquioxide contents < 1.0%, loss on ignition about 50% [19].

3. Experimental

Effect of potassium oxalate on some properties of oxychloride cement has been studied by incorporating it in different amounts in wet-mix.

i). *Setting Periods*

Effect of potassium oxalate on setting characteristics of magnesia cement (oxychloride cement) has been studied by incorporating it in different amounts [0%, 5%, 10%, 15% & 20%]. Setting periods of the wet-mixes prepared by gauging 1: 2 dry-mixes of diverse compositions separately with the gauging solution were determined adopting the standard procedure (Indian Standard Institution 1982). Setting period were determined by adopting standard procedures as per IS specification using Vicat needle apparatus [20]. Observed results are summarized in table 1.

Table 1: Effect of potassium oxalate on setting characteristics of oxychloride cement.

Sl. No.	Observation	Composition of dry mix (% additive)				
Gauging solution: 22□ Be; temperature: 31□□Dry-mix composition: 1 : 2*; and humidity: above 75%		0%	5%	10%	15%	20%
1.	Volume of gauging solution (ml)	54	54	63	57	57
2.	Initial setting time (min)	140	200	230	240	300
3.	Final setting time (min)	225	300	305	315	335

*One part by weight of magnesia and two parts by weight of dolomite.

ii). Weathering Investigation

Variation in weights of the setting time blocks (taken from vicat moulds) for 24 h, 7 and 30 days is recorded. Weights of test blocks increase or decrease with time due to the different weathering effects promoted by the admixture. Experimental findings are recorded in table 2.

Table 2: Effect of potassium oxalate on weathering of oxychloride cement.

Sl. No.	Observation	Composition of dry mix (% additive)				
Gauging solution: 22□ Be; temperature: 31□□Dry-mix composition: 1 : 2*; and humidity: above 75%		0%	5%	10%	15%	20%
1.	Weight after 24 h (g)	256.52	267.82	272.84	277.04	278.02
2.	Weight after 7 days (g)	252.20	261.85	267.85	268.48	268.00
3.	Weight after 30 days (g)	249.70	259.36	265.42	265.09	264.02

*One part by weight of magnesia and two parts by weight of dolomite.

iii). Moisture Ingress Test

Standard setting time blocks were used to find out the effect of potassium oxalate on moisture ingress in oxychloride cement. These were subjected to moisture treatments (steam test) to estimate their relative moisture sealing efficiencies according to the standard procedure (Yadav 1989; Gupta *et al* 1990, 1994). [21, 22].Observed results are summarized in table 3.

Table 3: Effect of potassium oxalate on moisture ingress (steam test) in the trial blocks.

Sl. No.	Observation	Composition of dry mix (% additive)				
Gauging solution: 22□ Be; temperature: 31□□Dry-mix composition: 1 : 2*; and humidity: above 75%		0%	5%	10%	15%	20%
	Trial blocks kept in boiling water for (hrs.)					
1.	00-05 h	NE	NE	NE	NE	NE
2.	05-10 h	NE	NE	NE	NE	NE
3.	10-15 h	NE	NE	NE	NE	NE
4.	15-20 h	NE	NE	NE	NE	NE
5.	20-25 h	C	NE	NE	NE	NE
6.	25-30 h	-	NE	NE	NE	NE

*One part by weight of magnesia and two parts by weight of dolomite (NE, no effect; C, cracked).

iv). Compressive Strength Test

The influence of incorporation of potassium oxalate on compressive strength of the oxychloride cement is studied with the help of standard 50 cm^3cubes prepared from the Indian Standard consistency pastes having potassium oxalate in different amounts. For this purpose standard size trail blocks [70.6 mm X 70.6 mm X 70.6 mm] were prepaid with 1:2 dry mix [by weight of magnesia and dolomite] having potassium oxalate in varying amounts [14,20]. These cubes (moulds) were tested after curing for 30 days as per the standard procedure (Indian Standard Institution 1958; Yadav 1989; Gupta *et al* 1994). The results obtained are shown in table 4.

Table 4: Effect of potassium oxalate on compressive strength of oxychloride cement.

Sl. No.	Observation	Composition of dry mix (% additive)				
Gauging solution: 22□ Be; temperature: 31□□Dry-mix composition: 1 : 2*; and humidity: above 75%						
1.	% Additive (C2H2O4.2H2O)	0%	5%	10%	15%	20%
2.	Compressive strength of the blocks (kg/cm^2)	500	354	232	180	122

*One part by weight of magnesia and two parts by weight of dolomite.

v). Linear Changes Investigations

To find the durability of product, Le-Chateliers test was conducted as per the standard procedure (Gupta 1976; Chandrawat *et al* 1994) standard sized moulds (200 mm X 200 mm X 200 mm) were filled by wet mixes having varying quantities of additive to study the effect of potassium oxalate on linear changes of oxychloride cement. Initial length of beams were measured after 24 hours and final length of the beams were measured after 28 days and the results are recorded in table 5.

Table 5: Effect of potassium oxalate on Linear change characteristics of oxychloride cement.

S. No	Dry Mix Composition (% additive)	Length of Beams (mm)		Change in Length (mm)
		Initial	Final	
1	0%	200.00	200.29	0.29
2	5%	200.00	201.19	1.19
3	10%	200.00	200.80	0.80
4	15%	200.00	200.57	0.57
5	20%	200.00	200.54	0.54

*One part by weight of magnesia and two parts by weight of dolomite.

4. Discussion

Trends in setting of the cement when potassium oxalate is mixed in the dry-mix are shown in table 1.

Volume of gauging solution required for Indian Standard consistency remains almost the same. This suggests about the indifferent nature (slow reactivity) of potassium oxalate with respect to oxychloride cement. Although percentage of the additive (admixture) increases gradually, gross amount of inert fillers (weight of dolomite + weight of potassium oxalate) remains the same. Thus almost constant volume in the amount of the gauging solution for Indian Standard consistency is expected. It is noted that initial and final setting periods increase with increasing amount of an additive. This is due to interlocking chain forming tendencies of additive.

Effects of potassium oxalate Parallely decreasing weights of the trial blocks with time are apparent from the data table 2. Moisture ingress characteristics of oxychloride cement is revealed from the table 3. It is found that water tightness of the trial blocks are increased remarkably due to the reactivity of oxalate ions towards harmful impurities like active lime etc. Excellent moisture sealing characteristics of potassium oxalate are expected as once this harmful di and trivalent metal captions have been converted in to insoluble and inactive crystalline phases, water vapour transmission is almost sealed.

Compressive strength of oxychloride is revealed by the table 4. On increasing proportions of the additive, gradual decrease in the compressive strength is noticed. Plausible reason for this is that with increasing excess of oxalate ions formation of magnesium oxalate take place which results into decrease of active amounts of magnesia and magnesium chloride in the matrix. Consequently chances of formation of strength giving compositions of oxychloride also decrease parallel.

The table 5 summarizes effect of additive on linear change characteristics of the trial beams. It appears that oxalate formed by incorporation of the potassium oxalate increase the crystal lattice gaps within the strength giving compositions. Thus it is observed that initial incorporation of the additive contribute much more to the volume changes than the latter incorporations. The discussions mentioned above can be explained on the basis of the following probable chemical changes during the setting.

$$CaO/MgO + HOH \rightarrow Ca(OH)_2/Mg(OH)_2$$

$$Ca/(OH)_2 + K_2C_2O_4 \rightarrow CaC_2O_4 + 2KOH$$

$$Mg(OH)_2 + K_2C_2O_4 \rightarrow MgC_2O_4 + 2KOH$$

$$2KOH + MgCl_2 \rightarrow Mg(OH)_2 + 2KCl$$

$$MgCl_2 + K_2C_2O_4 \rightarrow MgC_2O_4 + 2KCl$$

$$CaCl_2 + K_2C_2O_4 \rightarrow CaC_2O_4 + 2KCl$$

$$3Mg(OH)_2 + MgCl_2 + 8H_2O \rightarrow 3Mg(OH)_2.MgCl_2.8H_2O$$

$$5Mg(OH)_2 + MgCl_2 + 8H_2O \rightarrow 5Mg(OH)_2.MgCl_2.8H_2O$$

5. Conclusion

- Incorporation of potassium oxalate increases setting periods of oxychloride cement.
- Potassium oxalate improves water tightness of the products in all proportions within the experimental limits.
- It decreases compressive strength of the product within the experimental limits.

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