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RESEARCH ARTICLE

EFFECT OF CHEMICAL ADMIXTURES ON THE PROPERTIES OF GYPSUM HEMIHYDRATE PLASTER USED AS BUILDING MATERIAL

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ABSTRACT

Investigations carried out deal with the effect of inorganic and organic admixtures in different concentrations on the properties of ß-hemihydrate plasters of mineral gypsum (selenite) and chemical gypsum (phosphogypsum). It has been observed that the addition of these admixtures plays a vital role in the increase of the compressive strength and also improves water resistance of the plaster. Addition of 1 % K_2SO_4 increases about 54 % and 66 % of compressive strength of selenite and phophogypsum plasters respectively, while water absorption decreases upto 30 %. The order of increase in compressive strength and water resistance observed is $Al_2(SO_4)3 < NaCl < Na_2SO_4 < CaCl_2 < K_2SO_4$. Maximum increase in compressive strength and water resistance has been observed at 1.0 % of sugar as organic admixture in gypsum plasters. This shows an increase of about 65 % and 72 % in the compressive strength as well as about 22 and 17 % decrease in water absorption for selenite and phophogypsum plasters respectively. It can be seen that the order of increase in compressive strength and water resistance is starch < sodium tartarate < sugar. The results achieved are encouraging and show that organic admixtures give better performance as compared to inorganic admixtures.

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INTRODUCTION

Man and his perpetual activities have been producing lot of wastes. Over 300 million tones of agro industrial wastes or by-product materials namely gypsum, fly ash, slag, red mud, mine tailings, lime sludge, etc. are produced annually from various industrial processes in India. Different measures have been adopted in different countries to dispose and use of these wastes in effective manner. The constantly rising demands of the housing and construction sector in the country have led to acute shortage of building materials and encourage the scientist, engineers and technologist to play the role for development of building materials utilizing the vast varieties of waste available. By-product gypsum or chemical gypsum is an important waste available obtained from the phosphoric acid, hydrofluoric acid, titanium, intermediate dyes, tartaric acid, citric acid industries and from the separation of salt from sea water. These waste gypsums are popularly called as phosphogypsum, fluorogypsum, titangypsum, H-acid gypsum, tartrarogypsum and citrogypsum and marine gypsum respectively.

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Mineral gypsum or natural gypsum is found in the form of rock is an important building materials not only used in cement manufacture, agriculture or as filler in various commodities, but is a starting material for making plaster. Plaster is produced from the calcination of gypsum ($CaSO_4.2H_2O$) which partially dehydrates to produce a hemi hydrate ($CaSO_4.1/2H_2O$) ($CaSO_4.2H_2O \rightarrow CaSO_4.1/2H_2O + 3/2\ H_2O$). In India about 5.0 million tonnes per annum of phosphogypsum, a waste product of phosphoric acid fertilizer is produced and about 15.0 % is use at present (Singh and Garg, 2000). Efforts have been made in several countries to use phosphogypsum in making cement (Olmez and Enden, 1989), gypsum plaster (Olmez and Enden, 1989), building products (Kumar Sunil, 2003) and binders (Singh and Garg, 1992). Phosphogypsum is always contaminated with the impurities of P_2O_5 as monocalcium phosphate, dicalcium phosphate and tri calcium phospohate, fluoride as sodium fluoride, sodium silicofluoride or calcium fluoride, organic matter and a small quantity of soluble alkalies (Singh, 2005). These impurities adversely influence the workability, setting time and strength characteristics of the plaster (Olmez and Enden, 1989; Singh, 2005). Therefore the beneficiation of phophogypsum is essential before use to reduce its harmful impurities present in it.

After beneficiation, the gypsum is calcined between 120-180^{o}C, either in directly fired rotary kilns or in indirectly heated kettles to produce the ß-hemihydrate or plaster of paris or gypsum plaster. Gypsum plaster offer fast setting and fine finishing and are used mostly for aesthetic works, rapid construction or fire retarding purposes for indoor applications. Its use in outdoors and construction is limited due to the inconveniences of low mechanical strength and poor water resistance; nonetheless, gypsum is a material intensely used worldwide. The production of plaster from gypsum is an energy intensive process and there is a need to develop high strength water resistant plaster for all purpose by incorporation of mineral or chemical admixtures.

In order to achieve the goal, it is therefore proposed to study the effect of chemical admixtures for improvement in the strength characteristics of plaster for use in formulation of water resistant colourless cementitious binder, boards, blocks and plasters. In the present investigations, effects of inorganic and organic admixtures have been studied on mineral gypsum (salenite) and chemical gypsum (phosphogypsum) as strength enhancers for exterior applications. The properties like compressive strength, bulk density, water absorption and porosity were studied to observe the effect of admixtures.

MATERIALS AND METHODS

Raw materials

The phosphogypsum was procured from M/s Rashtriya Chemicals & Fertilizers, Mumbai and selenite gypsum was procured from Bikaner, Rajasthan.

Beneficiation of Phosphogypsum

Phosphogypsum contains impurities of P_2O_5, F organic matter and alkalies which adversely affect the hydraulic properties of the plaster. Hence beneficiation of phophogypsum is important to reduce the impurities to an acceptable level. Phosphogypsum is beneficiated by solubilization of impurities in water, rejection of coarse fraction (5-10 %) over 300 µm sieve (Singh et al., 1996), centrifugation and drying at 110-120^{o}C of washed gypsum. The salenite gypsum, unprocessed and beneficiated gypsum were tested as per methods given in IS: 1288: 1982 and the chemical composition has been shown in Table 1 along with the limits prescribed in Indian Standard: 12679: 1989. The Table 1 shows that after beneficiation the reduction of impurities in phosphogypsum take place which comply the requirements of Indian Standard.

Table 1. Chemical Composition of Beneficiated Phosphogypsum

Constituents (%)	Salenite gypsum	Phosphogypsum Unprocessed	Beneficiated	IS: 12679: 1989
P_2O_5	---	0.477	0.40	Max. 0.40
F	---	0.52	0.38	Max. 0.40
Organic matter	---	0.31	0.13	Max. 0.15
SiO_2 + insoluble Mater	2.13	2.41	0.22	
$Al_2O_3 + Fe_2O_3$	0.71	0.51	0.22	
CaO	31.72	32.41	32.40	
MgO	0.41	0.09	0.06	
SO3	44.2	41.17	44.68	
Na_2O_3+K_2O	0.21	0.22	0.16	Max. 0.30
Loss on ignition	20.05	19.20	19.40	
pH	6.8	3.0	6.30	Max. 5.0

The SEM of the unprocessed and beneficiated phosphogypsum as studied on SEM Model LEO 438VP (UK) are shown in Fig. 1 and Fig. 2 respectively. It can be seen that phosphogypsum (Fig. 1) mostly show appearance of anhedral to euhedral prismatic, lath and tabular type of crystals often agglomerated with occasional twinning. The reduction of impurities has been confirmed by SEM of beneficiated gypsum in Fig. 2 which shows mitigation of agglomeration among the gypsum crystals as well as formation of well developed euhedral prismatic crystals with rounded bodies.

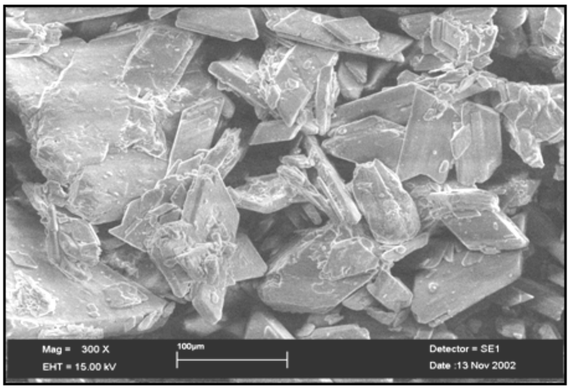

Fig. 1. SEM of unprocessed phosphogypsum

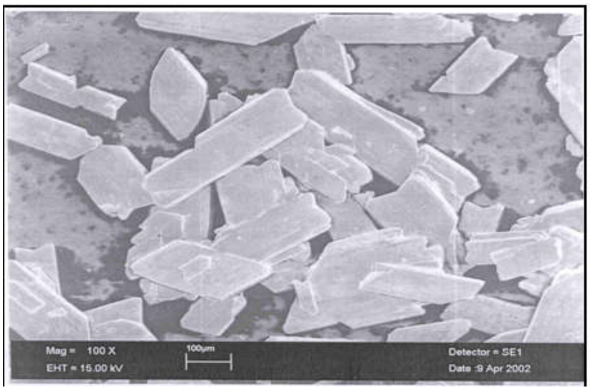

Fig. 2. SEM of beneficiated phosphogypsum

Production of ß-Hemihydrate Plaster

The β-hemihydrate was produced from the beneficiated phosphogypsum and selenite gypsum by calcination at 150-160^{o}C for a period of 4 hrs in trays with intermittent stirring in the oven. The plasters were ground and sieve though 150 µm sieve and tested for physical properties as per IS: 8272-1984. The physical properties are shown in Table 2. A perusal of Table 2 shows that compressive strengths of plasters are complying with the requirements of IS: 8272: 1984. It has been observed that on heating phosphogypsum at 150^{0}C, about 15% of water of crystallization is released from the gypsum lattice in the form of steam. The calcined gypsum containing 4.5-5.3% of combined water is obtained. During transformation of phosphogypsum into β-hemihydrate plaster, the gypsum lattice is broken where by rhombohedral crystal of β-hemihydrate plaster is formed from the monoclinic gypsum. If β-anhydrite is produced directly from the unprocessed phosphogypsum, the impurities of P_2O_5 and F present in the gypsum lattice are released and as a result the pH of plaster is dropped from 2.0 – 3.0 to 2.0 –2.5.

The drop in pH of the plaster affects the setting and strength of the plaster adversely. Hence beneficiation of the plaster by washing and neutralization with optimum quantity of lime or hydrated lime (3-4%) is essential.

Table 2. Physical properties of salenite and phosphogypsum plaster

Plaster strength	Consistency (%)	Setting time (min)	Bulk density (kg/m^3)	Compressive (MPa)
Selenite	60.0	9.0	1300	11.2
Phosphogypsum	62.0	7.0	1220	10.0

Effect of Chemical Admixtures

Various percentage of inorganic (K_2SO_4, Al_2 $(SO_4)3$, $CaCl_2$, NaCl and Na_2SO_4) and organic (sugar, starch and sodium tartarate) chemical admixtures were used as strength enhancers of selenite and phosphogypsum plasters and are shown in Table 3 and 4 respectively. To study the effect of these chemical admixtures on the properties of ß-hemihydrate plasters cubes of size 2.5 cm x 2.5 cm x 2.5 cm of gypsum plasters containing different percentage of inorganic and organic admixtures were cast at respective consistency.

The cubes in their moulds were retained over water in a closed vessel at room temperature for 24 hours. The cubes were removed from the moulds and dried at a temperature of 35±2°C. After complete drying the cubes were weighed and tested for various properties like compressive strength, bulk density, water absorption and porosity.

Compressive strength of cubes was determined as per IS: 8272: 1984. After drying the cubes were cooled at room temperature ~for 15 minutes and tested. The average compressive strength of the five cube specimens has been reported. Bulk density was calculated by dividing the mass of the dried cube with volume. To study the performance in water, cubes of gypsum plaster were dried at 42°C and then immersed. Water absorption and porosity were calculated at different periods of interval at 2 hrs, 8 hrs and 24 hrs.

RESULTS AND DISCUSSION

Effect of Inorganic Admixtures

Effect of inorganic admixtures viz. K_2SO_4 (0.5, 1.0 and 1.5 %), Al_2 $(SO_4)3$ (0.5, 1.0 and 1.5 %), $CaCl_2$ (0.5, 1.0 and 1.5 %), NaCl (0.5 and 1.0 %) and Na_2SO_4 (0.5, 1.0 and 1.5 %) has been studied on the properties of salenite and phosphogypsum plasters and results are shown in Table 3 for change in compressive strength and bulk density. A perusal of Table shows that with the addition of 1.0 % K_2SO_4, maximum compressive strength of 17.3 and 16.6 MPa is achieved by selenite and phosphogypsum plasters respectively as compared to their respective controls. This shows an increase of about 54 % and 66 % in the compressive strength for selenite and phophogypsum plasters respectively. Although all the admixtures added show enhancement in strength at all the percentage studied and the order of increase in compressive

Table 3. Effect of inorganic admixtures on selenite and phosphogypsum properties

Admixture (%)	Selenite plaster Compressive strength (MPa)	Selenite plaster Bulk density (g/cm^3)	Phosphogypsum plaster Compressive strength (MPa)	Phosphogypsum plaster Bulk density (g/cm^3)
Control	11.2	1.30	10.0	1.22
K_2SO_4				
0.5	14.4	1.21	13.0	1.23
1.0	17.3	1.24	16.6	1.25
1.5	16.2	1.30	15.8	1.24
$Al_2(SO_4)_3$				
0.5	13.5	1.14	13.2	1.20
1.0	14.0	1.17	14.7	1.35
1.5	12.6	1.12	12.3	1.32
$CaCl_2$				
0.5	16.2	1.32	15.1	1.35
1.0	14.8	1.23	13.3	1.32
1.5	11.9	1.21	11.1	1.30
NaCl				
0.5	15.5	1.35	16.0	1.35
1.0	14.1	1.27	15.5	1.31
Na_2SO_4				
0.5	14.0	1.21	12.5	1.22
1.0	15.3	1.25	15.8	1.28
1.5	13.8	1.19	12.9	1.25

strength observed is $Al_2(SO_4)3 < NaCl < Na_2SO_4 < CaCl_2 < K_2SO_4$. Bulk density also varies from 1.19 to 1.35 g/cm^3 for both the plasters with addition of different percentage of the select admixtures.

Performance of selenite and phosphogypsum plasters in water was investigated for admixtures K_2SO_4 (1.0 %), Al_2 (SO_4) (1.0 %), $CaCl_2$ (0.5 %) and NaCl (0.5 %) and results were compared with control samples. The effect of chemical admixtures on water absorption and porosity has been studied and the results for selenite and phosphogypsum plasters are shown plotted in Fig. 3 and 4 respectively. It is clearly indicated by these figures that water absorption and porosity increases with time and maximum values have been observed at 24 hrs. It is observed that water absorption and porosity varies from 21.5 to 25.8 % and 27.2 to 32.5 respectively for selenite plaster (Fig. 3) for different admixtures added at 24 hrs. Minimum water absorption and porosity is achieved at 1.0 % dose of K_2SO_4 in the selenite plaster which is about 26 and 13 % lower as compared to control. Similar results have been observed for phosphogypsum plaster in which water absorption and porosity varies from 20.7 to 24.6 % and 25.7 to 32.5 respectively. Minimum water absorption and porosity is shown by 1.0 % dose of K_2SO_4 in the phosphogypsum plaster which is about 30 % lower as compared to control.

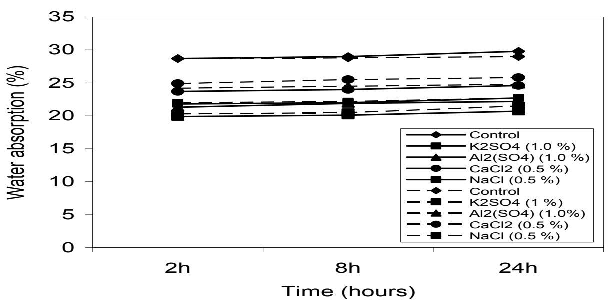

Fig. 3. Effect of inorganic admixtures on water absorption of salenite (---) and phosphogypsum (___) plasters

Effect of Organic Admixtures

Effect of organic admixtures like sugar (0.5, 1.0 and 1.5 %), starch (0.25 and 0.50), and sodium tartarate (0.25, 0.50 and 1.0 %) has been as strength enhancers for gypsum plasters and results are shown in Table 4 for change in compressive strength and bulk density.

A perusal of Table shows that addition of 1.0 % sugar gives maximum increase in compressive strength and is 18.5 and 17.2 MPa for selenite and phosphogypsum plasters respectively as compared to their respective control. This shows an increase of about 65 % and 72 % in the compressive strength for salenite and phophogypsum plasters respectively. It can be seen that all the admixtures added show enhancement in strength and the order of increase in compressive strength observed is starch<sodium tartarate<sugar. The table also shows that increase in bulk density after addition of admixture and varies from 1.29 to 1.39 g/cm^3 for both the plasters with addition of different percentage of the select admixtures.

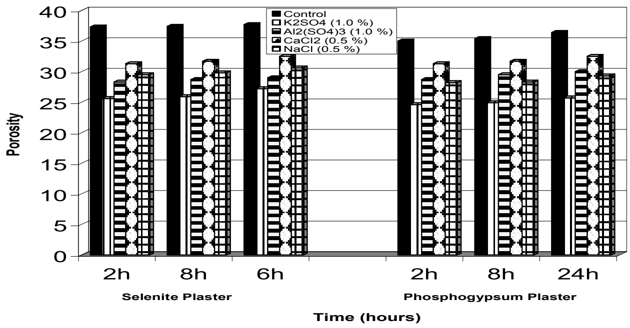

Fig. 4. Effect of inorganic admixtures on porosity of salenite and phosphogypsum plasters

Table 4. Effect of organic admixtures on selenite and phosphopgypsum properties

Admixture (%)	Selenite plaster Compressive strength (MPa)	Selenite plaster Bulk density (g/cm^3)	Phosphogypsum plaster Compressive strength (MPa)	Phosphogypsum plaster Bulk density (g/cm^3)
Control	11.2	1.30	10.0	1.22
Sugar				
0.5	18.0	1.32	15.6	1.31
1.0	18.5	1.35	17.2	1.35
1.5	16.7	1.31	16.5	1.32
Starch				
0.25	16.0	1.37	14.5	1.32
0.50	12.4	1.31	13.8	1.29
Sodium Tartarate				
0.25	18.3	1.39	14.5	1.32
0.50	17.8	1.37	16.2	1.37
1.0	17.0	1.30	16.7	1.39

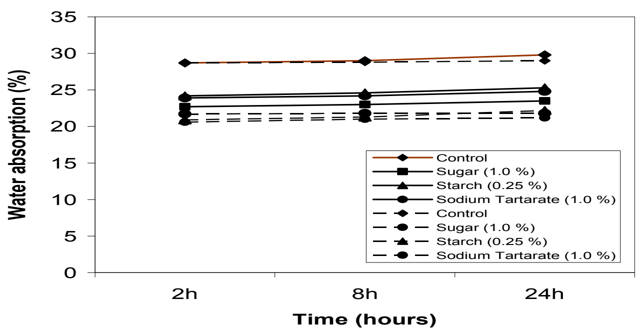

Fig. 5: Effect of organic admixtures on water absorption of salenite (---) and phosphogypsum (___) plasters

Water absorption studies of selenite and phosphogypsum plasters containing organic admixtures sugar (1.0 %), starch (0.25 %) and sodium tartarate (1.0 %) were carried out and results are shown in Fig. 5 and 6 respectively along with controls.

A perusal of these figures shows that water absorption and porosity increases with time and maximum values have been observed at 24 hrs. It is observed that water absorption and porosity varies from 21.8 to 22.5 % and 28.8 to 31.3 respectively for selenite plaster (Fig. 5) containing select admixtures at 24 hrs. Minimum water absorption and porosity is observed at addition of 1.0 % dose of sugar in the selenite plaster which is about 22 and 17 % lower as compared to control. Similar results have been observed for phosphogypsum plaster in showing water absorption and porosity from 23.5 to 25.3 % and 30.9 to 32.8 respectively. Minimum water absorption and porosity shown by 1.0 % dose of sugar in the phosphogypsum plaster is about 21 and 15 % lower as compared to control.

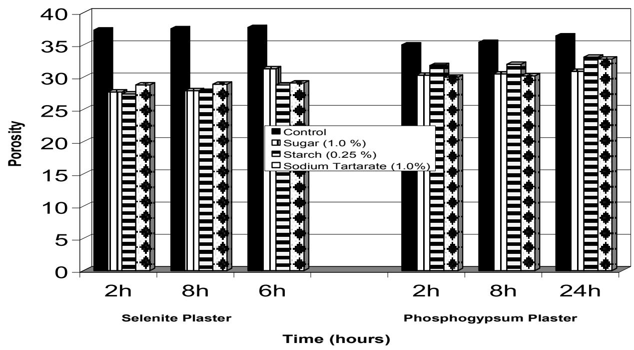

Fig. 6. Effect of organic admixtures on porosity of salenite and phosphogypsum plasters

Further, the results of inorganic and organic admixtures show that the no appreciable difference in value of water absorption and porosity has been observed at all the percentage studied. The results show that with the addition of chemical admixtures significant enhancement in compressive strength and improvement in water resistance as well as porosity of both hemihydrate plasters have been achieved. It is also observed that organic admixtures give comparatively higher strength and lower water absorption and porosity and can be used as strength enhancers after detailed studies including effect on setting time etc.

Conclusion

Present studies involve the effect of different concentrations of inorganic and organic admixture on the properties of ß-hemihydrate plasters of mineral gypsum (selenite) and chemical gypsum (phosphogypsum). It is observed that the addition of inorganic and organic admixtures in selenite and phosphogypsum plaster at different concentration increases the compressive strength and improves performance in water. Maximum compressive strength and water resistance has been observed by the addition of 1 % of K_2SO_4 and sugar in the plasters. Addition of 1 % K_2SO_4 increases about 54 % and 66 % of compressive strength of selenite and phophogypsum plasters respectively as compared to controls, while water absorption has also decreased upto 30 % by the addition of 1 % K_2SO_4. Although all the admixtures added show enhancement in strength at different percentage studied. The order of increase in compressive strength and water resistance observed is $Al_2 (SO_4)3$<NaCl<Na_2SO_4<$CaCl_2$<K_2SO_4. Addition of 1.0 % of sugar as organic admixture in gypsum plasters enhances maximum compressive strength and water resistance.

This shows an increase of about 65 % and 72 % in the compressive strength as well as about 22 and 17 % decrease in water absorption as compared to control for selenite and phophogypsum plasters respectively. It can be seen that the order of increase in compressive strength and water resistance is starch<sodium tartarate<sugar. The results also show that organic admixtures give comparatively higher strength and water resistance and can be used as strength enhancers after detailed studies including effect on setting time etc.

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