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Effect of phosphatic and fluoride impurities of phosphogypsum on the properties of selenite plaster

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Abstract

The effect of phosphatic and fluoride impurities present in waste phosphogypsum on the setting time, strength development and morphology of selenite gypsum plaster have been studied. The results showed that soluble phosphates retard the setting and strength development of plaster while soluble fluorides decrease the setting time and reduce the density and strength. The gypsum crystals in the set plaster are modified from to euhedral to subhedral prismatic, rhombic, tabular and lath-shaped crystals of variable sizes interspersed with subhedral to anhedral needles. It is concluded that the formation of prismatic, rhombic and lathelike crystals retards the normal setting and strength development of selenite gypsum plaster.

Keywords: Fineness; Crystal size; Compressive strength; Fluoride; Phosphate

1. Introduction

Over 5.0 million tonnes of phosphogypsum are produced as by-products of the phosphoric acid industry per annum in India. Phosphogypsum is always contaminated with the impurities of P_2O_5 as monocalcium phosphate, dicalcium phosphate and tricalcium phosphate, fluoride as sodium fluoride, sodium silicofluoride or calcium fluoride, organic matter and a small quantity of soluble alkalies [1–3]. These impurities adversely influence the workability, setting time and strength development of the plaster [4,5]. The industrial potential of gypsum is realized largely through its ability to form quality-grade plaster. It is therefore essential to beneficiate phosphogypsum to reduce harmful impurities present in it. Researches have shown that after beneficiation, the phosphogypsum plaster possesses similar characteristics as that obtained with the natural gypsum plaster [6–8]. The effect of phosphogypsum impurities has been reported on the properties of cement and plaster [9–14]. These impurities decrease the strength and workability of the plaster, which is affected by the slight variation in the nature and quantity of the impurities. The studies reported in this paper are confined to the effect of phosphatic and fluoride impurities on the properties of gypsum plaster produced from selenite gypsum.

2. Experimental

2.1. Raw material

The selenite gypsum and phosphogypsum samples procured from Bikaner, Rajasthan and Albright Morarji Pandit Ambernath (Thane), respectively, were used for making calcined gypsum or gypsum plaster. The chemical composition of phosphogypsum and mineral selenite gypsum samples are reported in Table 1. The phosphogypsum sample was procured from the single-step process (dihydrate route) of phosphoric acid manufacture. The P_2O_5 and F impurities are present in three different forms: (i) on the surface of gypsum crystals as water-soluble compounds (H_3PO_4, $Ca(H_2PO_4)_2 \cdot H_2O$, NaF), (ii) substituted in the lattice of gypsum crystals (effectively solid solutions of $CaHPO_4 \cdot 2H_2O$ or Na_2SiF_6) in gypsum and (iii) as insoluble compounds ($Ca_3(PO_4)_2$, CaF_2). These impurities are the reaction products of phosphate rock and sulphuric acid while manufacturing phosphoric acid. The selenite gypsum contains only small amounts of impurities (principally silica, alumina and iron oxide) and its pH is almost neutral. The

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Table 1
Chemical composition of phosphogypsum and mineral selenite gypsum

Constituents	Phosphogypsum (%)	Selenite gypsum (%)
P_2O_5	0.92	–
F	1.5	–
Organic matter	0.13	–
Alkalies (Na_2O+K_2O)	0.46	–
SiO_2 + insoluble in HCl	6.60	2.13
$Al_2O_3+Fe_2O_3$	5.86	1.06
CaO	27.76	31.41
MgO	0.25	1.41
SO_3	39.65	45.48
$CaSO_4 \cdot 2H_2O$	85.25	97.70
pH	2.8	6.9

low pH of phosphogypsum indicates that it contains an excess of acidic impurities.

2.2. Preparation of plaster of paris or calcined gypsum

Selenite gypsum of about 97.0% purity was ground to pass a 150-μm Indian Standard (IS) sieve and calcined at 150 °C for 5.0 h in the gypsum calcinator devised by Central Building Research Institute, Roorkee, India [15]. The calcined material was desiccated over anhydrous calcium chloride in a closed vessel for a period of 24 h to avoid any contamination.

2.3. Reagent used

The following reagent-grade chemicals similar to major impurities present in phosphogypsum were added to the calcined selenite:

Phosphates: H_3PO_4, $Ca(H_2PO_4)_2 \cdot H_2O$, $CaHPO_4 \cdot 2H_2O$ and $Ca_3(PO_4)_2$
Fluorides: NaF, Na_2SiF_6, Na_3AlF_6 and CaF_2

2.4. Preparation of slides for microscopic examination

Soluble reagents (H_3PO_4, $Ca(H_2PO_4)_2 \cdot H_2O$ and NaF) in the range 0.5–1.5% as detected in phosphogypsum were dissolved in distilled water. The sparingly soluble reagents ($CaHPO_4 \cdot 2H_2O$, Na_2SiF_6 and Na_3AlF_6) were uniformally mixed with selenite gypsum plaster. For making slides to be used for microscopic examination, the plaster was placed on the slide and blended with 3.0 ml of a solution of soluble phosphates and fluorides. The plaster containing sparingly soluble reagents was gauged with distilled water on the slide itself. The plaster was allowed to set for 24 h to dihydrate gypsum. The dihydrate gypsum thus formed in the chemical media was studied for its shape and size under the Petrographic Leitz Pan Phot microscope (Germany). The pH value of the gypsum slurry containing various chemicals was measured using a digital pH meter Model CL 46 (Toshniwal, India).

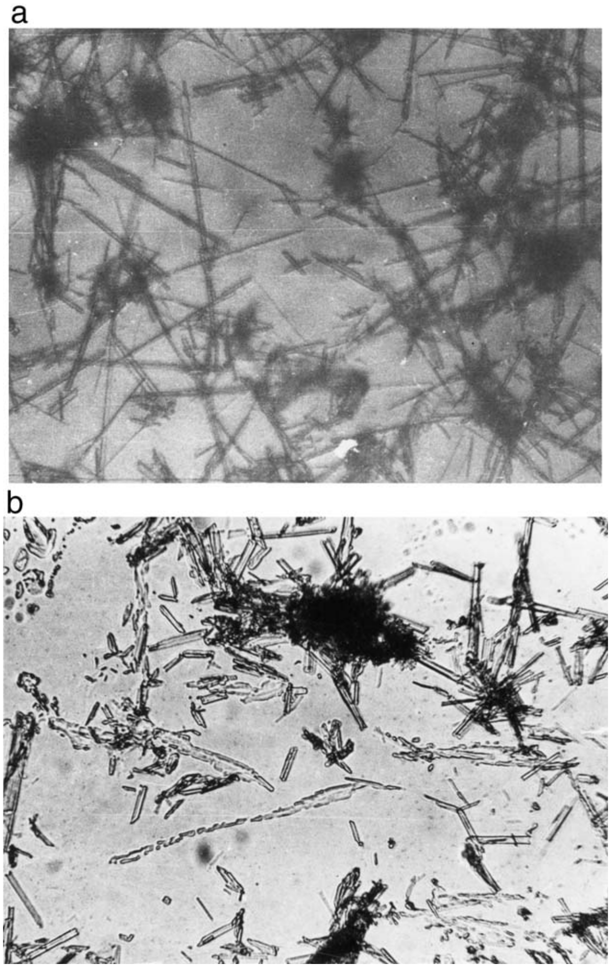

Fig. 1. Photomicrographs of (a) hardened selenite gypsum plaster and (b) phosphogypsum plaster (×45).

2.5. Properties of set plaster in chemical media

Properties such as consistency, setting time, bulk density and compressive strength of the selenite plaster in the presence of phosphatic and fluoride compounds were determined as per IS: 2542 (Part 1)-1978 [16]. To determine compressive strength, 5 × 5 × 5-cm cubes were cast at nor-

Table 2
Physical properties of selenite and phosphogypsum plasters

Sample no.	Water demand for normal consistency (%)	Setting time (min)	Bulk density (kg/m^3)	Compressive strength (MPa)
1. Selenite	60.0	8.0	1300	14.2
2. Phosphogypsum	68.0	4.0	1180	8.4

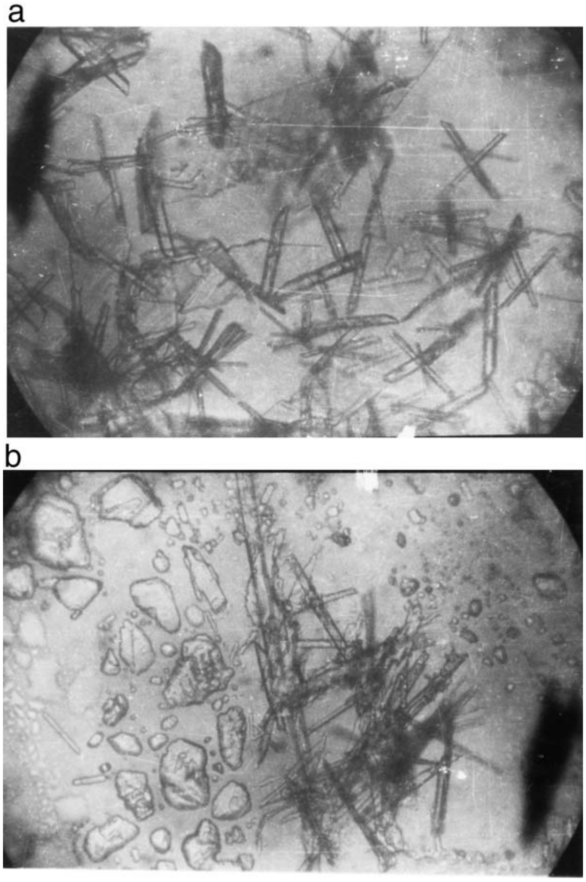

Fig. 2. Photomicrographs of hardened selenite plaster in the presence of (a) 0.5% H_3PO_4 and (b) 1.0% $Ca(H_2PO_4)_2{\cdot}H_2O$ (×45).

mal consistency. The water demand for normal consistency is expressed in grams per 100 g of plaster.

3. Results and discussions

3.1. Morphology and physical characteristics of selenite and phosphogypsum plasters

The typical morphology of set selenite gypsum plaster is shown in (Fig. 1a). There are clusters of euhedral needles that are fairly elongated and well developed. The crystals often appear interlocked. On the contrary, the morphology of set phosphogypsum plaster (Fig. 1b) shows predominantly a mixture of badly developed prismatic, lath- and needle-shaped crystals forming agglomeration. The formation of such type of crystals confirm adverse effect of impurities present in phosphogypsum. The physical properties of selenite and phosphogypsum plasters are listed in Table 2.

It can be seen that selenite plaster has higher values for setting time, bulk density and compressive strength than the phosphoplaster while the consistency is lower in the former than the latter. The fall in setting time and strength values in phosphoplaster can be attributed to the increase in consistency and to the formation of clustered subhedral and anhedral deformed prismatic and lath-shaped crystals of variable sizes. The development of such type of crystals instead of conventional needles found in selenite plaster may be due to the bad effect of impurities of phosphates and fluorides that remain intact in phosphogypsum even during calcination.

3.2. Effect of addition of phosphatic and fluoride compounds on the morphology and physical properties of selenite plaster

3.2.1. Effect of phosphatic compounds

3.2.1.1. Orthophosphoric acid (H_3PO_4). On addition of 0.5% H_3PO_4 acid to the selenite plaster, the hardened plaster showed formation of deformed prismatic and tubular-shaped crystals. Besides these crystals, a few small, agglomerated anhedral needles were also formed (Fig. 2a). On further increasing the concentration of H_3PO_4 from 1.0% to 1.5%, the size and number of crystals were grossly reduced. The formation of these crystals may be ascribed to the selective adsorption of H_3PO_4 on the growing faces of the selenite gypsum plaster, thereby modifying the crystal habit. The physical properties of plaster containing H_3PO_4 are listed in Table 3. Data showed significant reduction in bulk density and compressive strength values (compared with selenite plaster; Table 2) with the increase in H_3PO_4 concentration. The setting time too is greatly retarded due to the formation of temporary coatings on the growing faces of the selenite crystals by the H_3PO_4 solution. Data also show an enhancement of consistency with the increase in H_3PO_4 concentration. This may be attributed to the bulky nature and fineness of the crystals.

3.2.1.2. Monocalcium phosphate monohydrate ($Ca(H_2PO_4)_2{\cdot}H_2O$). The addition of 0.5% $Ca(H_2PO_4)_2{\cdot}H_2O$ to the selenite plaster showed the formation of short

Table 3
Effect of the addition of H_3PO_4 acid and $Ca(H_2PO_4)_2{\cdot}H_2O$ on the physical properties of selenite plaster pastes at normal consistency

Sample no.	Property	H_3PO4 (%)				$Ca(H_2PO_4)_2{\cdot}H_2O$(%)			
		0.5	0.75	1.0	1.5	0.5	0.75	1.0	1.5
1	Water (%)	62.0	64.0	64.0	66.0	62.0	62.5	63.0	64.0
2	Setting time (min)	15.0	20.0	24.0	26.0	12.0	12.5	13.5	15.0
3	Bulk density (kg/m^3)	1200	1170	1165	1160	1270	1260	1240	1210
4	Compressive strength (MPa)	10.03	9.20	8.25	7.36	11.22	10.8	10.2	8.63

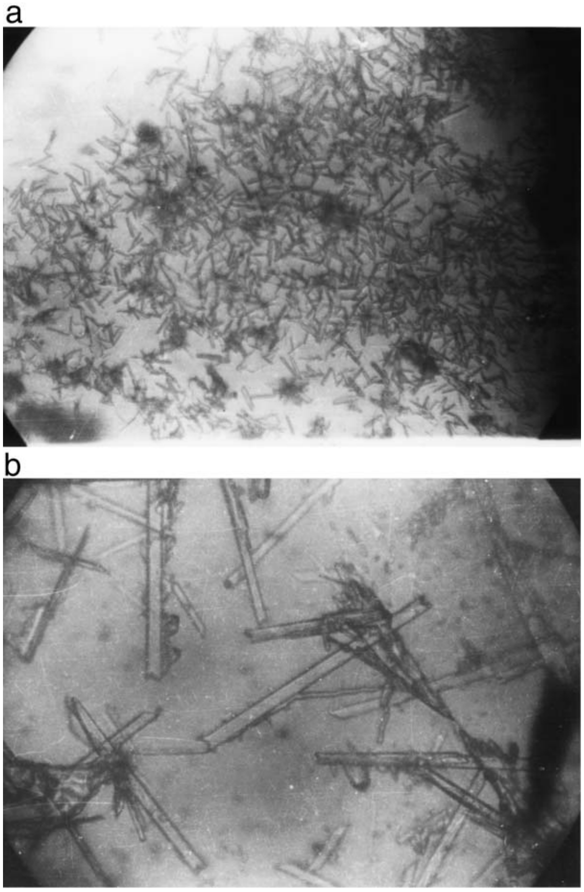

Fig. 3. Photomicrographs of hardened selenite plaster in the presence of (a) 0.5% $CaHPO_4 \cdot 2H_2O$ and (b) 0.5% $Ca_3(PO_4)_2$ (× 45).

euhedral to subhedral needles. On increasing the concentration from 0.75% to 1.0%, the size and quantity of the needles are reduced with respect to the selenite plaster. The presence of prismatic crystals with twinning habit was noted as the predominate phase (Fig. 2b). On increasing the compound concentration to 1.5%, the needle crystals are further reduced, which may be ascribed to the decrease in pH of the gypsum slurry with the increase in compound concentration. The physical properties of the selenite plaster in the presence of $Ca(H_2PO_4)_2 \cdot H_2O$ are shown in Table 3.

It can be seen that strength values were reduced to a lesser extent as compared to strength results of plaster obtained on admixing H_3PO_4. The fall in strength can be attributed to the formation of modified prismatic crystals with twinning and disappearance of needles. The setting time was found to be retarded to a lesser extent than the results recorded with H_3PO_4. The retardation of setting time may be ascribed to the formation of calcium phosphate produced by the interaction of H_3PO_4 (released on hydrolysis of $Ca(H_2PO_4)_2 \cdot H_2O$) with the gypsum.

$$Ca(H_2PO_4)_2 \cdot H_2O + xH_2O = CaHPO_4 + H_3PO_4 + (x+1)H_2O$$

$$H_3PO_4 \rightarrow 3H^+ + PO_4^{3-}$$

$$3Ca^{2+} + 2PO_4^{3-} \rightarrow Ca_3(PO_4)_2$$

3.2.1.3. Dicalcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$). On addition of 0.5% $CaHPO_4 \cdot 2H_2O$ to the selenite plaster, very fine euhedral needles were formed as the cardinal phase interspersed with occasional twinning (Fig. 3a). On further increasing the compound concentration from 0.75% to 1.0%, the size and quantity of needles were reduced. The formation of tuberous (rounded) bodies were also noticed. Further increase in the concentration of $CaHPO_4 \cdot 2H_2O$ to the selenite plaster further decreases the proportion of needle-shaped crystals. The major reason for the formation of such type of crystals can be assigned to the decrease in adsorption of $CaHPO_4 \cdot 2H_2O$ on the growing faces of gypsum crystals. Therefore, the crystal habit is restricted to the formation of needle-shaped crystals because of the low solubility of $CaHPO_4 \cdot 2H_2O$ (0.0286 g/100 ml H_2O) resulting in minimal release of H_3PO_4 acid. The decrease in pH values of the media was observed with the increase in the compound concentration. The physical properties of the hardened selenite plaster in presence of $CaHPO_4 \cdot 2H_2O$ are listed in Table 4.

Data show that compressive strength and bulk density were reduced with the increase in concentration of $CaHPO_4 \cdot 2H_2O$. However, the fall in strength was less when compared with the strength results obtained in Table 3. The improvement in compressive strength was mainly due to the formation of needle-shaped crystals. The setting time is also less retarded than the results achieved with H_3PO_4 as well as $Ca(H_2PO_4)_2 \cdot H_2O$. This is probably due to less availability of H_3PO_4, which could form temporary coatings over the growing dihydrate crystals.

Table 4
Effect of the addition of $CaHPO_4 \cdot 2H_2O$ and $Ca_3(PO_4)$ on the physical properties of selenite plaster pastes at normal consistency

Sample no.	Property	$CaHPO_4 \cdot 2H_2O$ (%)				$Ca_3(PO_4)_2$ (%)			
		0.5	0.75	1.0	1.5	0.5	0.75	1.0	1.5
1	Water (%)	63.0	62.5	62.0	62.0	60.0	61.0	61.5	62.5
2	Setting time (min)	9.0	11.5	12.0	13.0	8.0	9.0	9.5	11.0
3	Bulk density (kg/m^3)	1280	1260	1260	1230	1280	1275	1270	1266
4	Compressive strength (MPa)	12.04	11.64	10.96	9.65	13.3	12.8	12.6	12.1

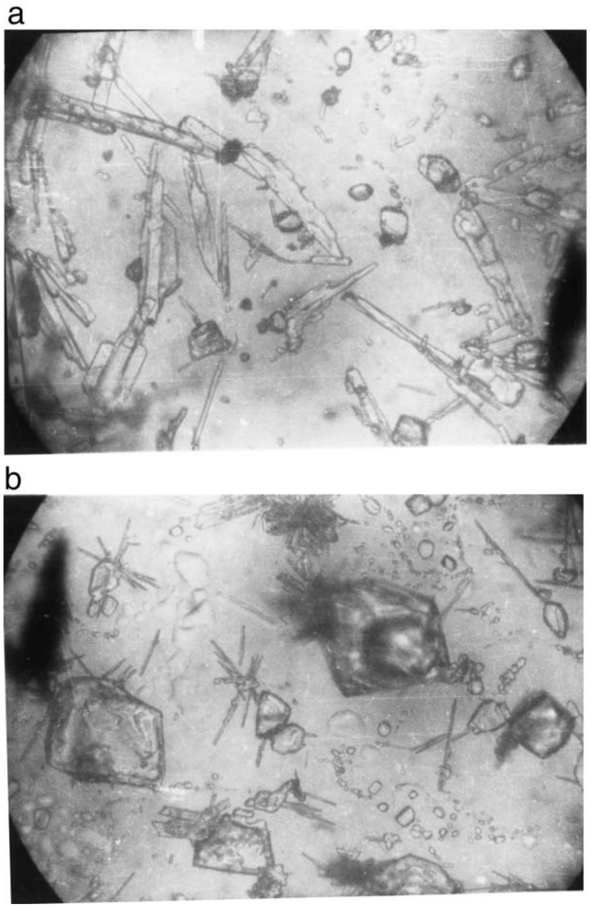

Fig. 4. Photomicrographs of hardened selenite plaster in the presence of (a) 0.1% NaF and (b) 1.5% NaF (× 45).

3.2.1.4. *Tricalcium phosphate ($Ca_3(PO_4)_2$).* The addition of 0.5% $Ca_3(PO_4)_2$ (passing a 90-μm IS sieve) to the selenite plaster indicated the formation of agglomerated euhedral to subhedral needle-shaped crystals (Fig. 3b). The needles were reduced on increasing the compound concentration from 1.0 to 1.5%. The needle-shaped crystals can be attributed to the very low solubility of $Ca_3(PO_4)_2$ (0.0089 g/100 ml H_2O), which releases a very small percentage of free H_3PO_4 acid into the gypsum paste. The low concentration of H_3PO_4 is not sufficient to be adsorbed on the growing nuclei of dihydrate crystals to cause any variation in the usual needle-shaped crystals. Only the size of the needles was affected. The properties of selenite plaster hardened in the presence of $Ca_3(PO_4)_2$ are reported in Table 4.

It can be seen that the compressive strength and bulk density values of the plaster were reduced only slightly when compared with the data reported in Table 2. However, these values were higher than the corresponding values obtained on using H_3PO_4, $Ca(H_2PO_4)_2{\cdot}H_2O$ and $CaHPO_4{\cdot}2H_2O$ (Tables 3 and 4).The increase in compressive strength is due to the formation of needle-shaped crystals. Data further showed that setting time was retarded slightly when 0.75% to 1.5% $Ca_3(PO_4)_2$ was blended with the selenite plaster. The slight retardation of the setting time may be attributed to the low solubility of $Ca_3(PO_4)_2$ in water. Thus, the availability of free H_3PO_4 is not sufficient to form surface coatings over the dihydrate and a weak retardation of the plaster took place. The consistency of plaster was seen to be close to that of plain selenite plaster.

3.2.2. *Effect of fluoride compounds*

3.2.2.1. *Sodium fluoride.* On addition of 1.0% NaF to the selenite plaster, a major phase of anhedral deformed prismatic and lath-shaped crystals were formed (Fig. 4a). On increasing the salt concentration from 0.5% to 1.0%, well-defined, euhedral, rhombus-shaped crystals were formed (Fig. 4b). The variable take-up of fluoride compound by the various faces of dihydrate gypsum results in the formation of deformed crystals of different habits. The appearance of rhombus-shaped crystals is due to selective adsorption of NaF on the crystal surfaces. The corresponding physical properties of the hardened selenite plaster are listed in Table 5.

It can be seen that the strength and bulk density of the plaster were both reduced. The decrease in strength can be ascribed to the formation of anhedral prismatic and rhombus-shaped crystals of deformed boundaries. The rapid setting of the plaster may be assigned to the microcrystallites of CaF_2 and Na_2SO_4 compounds produced during the setting and hardening of the plaster ($CaSO_4{\cdot}2H_2O + 2NaF \rightarrow Na_2SO_4 + CaF_2 + 2H_2O$) that may serve as nuclei.

3.2.2.2. *Sodium silicofluoride (Na_2SiF_6).* The addition of 0.1% Na_2SiF_6 to the selenite plaster, long, well-developed euhedral needles of hardened plaster were formed. A few needles of fibrous nature were also noted. The low adsorption of Na_2SiF_6 on the growing faces of the dihydrate crystals may be responsible for the development of needle-shaped crystals. On increasing the concentration of

Table 5
Effect of the addition of NaF and Na_2SiF_6 on the physical properties of selenite plaster pastes at normal consistency

Sample no.	Property	NaF (%)				Na_2SiF_6 (%)			
		0.5	0.75	1.0	1.5	0.5	0.75	1.0	1.5
1	Water (%)	61.0	62.0	62.0	63.0	60.5	63.0	63.0	63.0
2	Setting time (min)	5.0	3.0	2.5	2.0	6.0	5.0	5.0	5.0
3	Bulk density (kg/m^3)	1230	1200	1150	1100	1230	1200	1190	1180
4	Compressive strength (MPa)	12.01	11.66	9.24	8.88	12.5	11.8	10.2	9.60

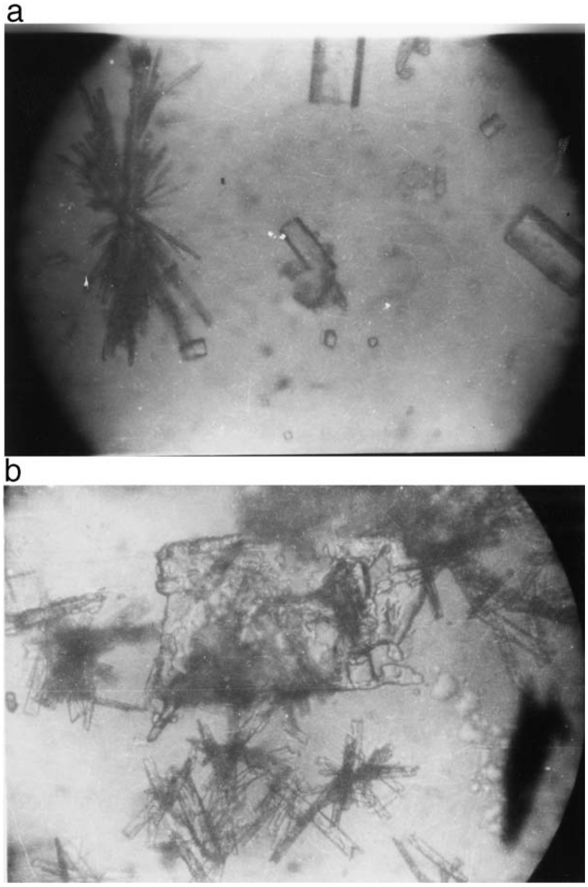

Fig. 5. Photomicrographs of hardened selenite plaster in the presence of (a) 0.5% of Na_2SiF_6 and (b) 1.0% Na_3AlF_6 (× 45).

Na_2SiF_6 from 0.3% to 0.5%, well-developed tubular shaped crystals were formed as majority phase (Fig. 5a). On further increasing the concentration of Na_2SiF_6 to 1.0%, mainly euhedral tabular-shaped crystals interspersed with radiating needles were formed. The change in crystal habit from needle to tabular and radiating habit may be correlated with the adsorption of increasing amounts of Na_2SiF_6 on the gypsum nuclei. The physical properties of the hardened plaster in the presence of Na_2SiF_6 are listed in Table 5.

It can be seen that bulk density and compressive strength were reduced as the concentration of Na_2SiF_6 increased. However, these results are much better than the results reported for NaF. The decrease in strength of the plaster is due to the formation of tabular-shaped crystals having less cohesion among themselves. The reduction in setting time and consistency of the plaster can be attributed to the formation of tabular-shaped crystals of bulky nature.

3.2.2.3. Sodium fluoroaluminate (Na_3AlF_6).

On addition of a small amount of 0.1% Na_3AlF_6 to the plaster, fine needles were formed as the predominant phase. Some prismatic and radiating fibrous strands were also precipitated. With the increase in Na_3AlF_6 concentration to 0.5% euhedral prismatic and radiating needles were formed. With the addition of 1.0% Na_3AlF_6, the hardened plaster showed the appearance of euhedral prismatic-type crystals as a major phase (Fig. 5b). The properties of selenite plaster containing Na_3AlF_6 are listed in Table 6.

Data showed a fall in bulk density and compressive strength of the plaster but the values were higher than the results achieved on addition of NaF and Na_2SiF_6 (Table 5). This can be ascribed to the formation of needle-shaped crystals in abundance in association with euhedral prismatic crystals.

3.2.2.4. Calcium fluoride (CaF_2).

On addition of 0.15% CaF_2 (passing a 90-μm IS sieve) to the plaster, fine subhedral needle-shaped crystals were formed. On increasing the compound concentration from 0.3% to 0.5%, agglomerated needles were obtained along with prismatic crystals (Fig. 6). With the addition of 1.0% CaF_2, prismatic crystals were formed as the major phase. The crystals were characterised by sharp boundaries with small size. The formation of needle-shaped crystals initially was due to low availability of fluoride because of the low solubility of CaF_2 in water (0.0018 g/100 ml). Therefore, adsorption of fluoride on the growing crystal surfaces of dihydrate gypsum was also mild; hence, normal hydration of plaster took place leading to the formation of needle-shaped crystals. The increase in CaF_2 concentration provides an excess of fluoride in the paste and thus modifies the crystal habit to prismatic-shaped crystals. The physical properties of the selenite plaster in CaF_2 media are reported in Table 6. It can be seen that bulk density and compressive strength of plaster were reduced slightly when the concentration of CaF_2 was 0.15%. This was because of the formation of needle-shaped crystals. As CaF_2 concentration was increased, the bulk density and compressive strength were reduced. The extent of reduction in these parameters was far less than the results obtained with NaF, Na_2SiF_6 and Na_3AlF_6 (Tables 5 and 6).The marginal fall in strength and

Table 6
Effect of the addition of Na_3AlF_6 and CaF_2 on the physical properties of selenite plaster pastes at normal consistency

Sample no.	Property	Na_3AlF_6 (%)			CaF_2 (%)			
		0.1	0.5	1.0	0.15	0.3	0.5	1.0
1	Water (%)	64.0	64.5	66.0	57.0	57.0	58.0	59.0
2	Setting time (min)	6.0	5.5	4.0	6.0	6.0	6.0	8.0
3	Bulk density (kg/m^3)	1240	1220	1200	1290	1280	1280	1260
4	Compressive strength (MPa)	12.80	12.08	11.40	13.6	13.0	12.6	12.4

Fig. 6. Photomicrograph of hardened selenite plaster in the presence of 0.5% CaF_2 (× 45).

the density of the plaster is due to change in crystal habit and reduction in the needle-shaped crystals. The efficacy of fluoride compounds may be summarized as follows:

$NaF > Na_2SiF_6 > Na_3AlF_6 > CaF_2$

4. Conclusions

1. On addition of soluble phosphates (H_3PO_4,$Ca(H_2PO_4)_2$) and fluorides(NaF), the crystal habit was found to change from needle to prismatic, rhombic and lath type of crystals.
2. When sparingly soluble or less soluble phosphates ($CaHPO_4{\cdot}2H_2O$, $Ca_3(PO_4)_2$) and fluorides (Na_2SiF_6 and Na_3AlF_6) were used, the persisting needles and growth of euhedral prismatic, lath, radiating fibres and tabular-shaped crystals of variable sizes and quantities were observed.
3. A maximum fall of compressive strength was found with the use of soluble phosphatic and fluoride compounds.
4. No correlation was observed among the morphology, setting time, strength development and the pH of the gypsum plaster.
5. The order of action of different phosphatic and fluoride compounds on the properties such as consistency, setting time, bulk density and compressive of selenite plaster was found to be:

$$H_3PO_4 > Ca(H_2PO_4)_2{\cdot}H_2O > CaHPO_4{\cdot}2H_2O > Ca_3(PO_4)_2$$

and $NaF > Na_2SiF_6 > Na_3AlF_6 > CaF_2$

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