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Investigation of properties of fluorogypsum-slag composite binders – Hydration, strength and microstructure

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ABSTRACT

A composite binder of high strength and low water absorption has been developed using industrial by-products fluorogypsum, granulated blast furnace slag and Portland cement. The development of strength in the binder at an early age is attributed to the conversion of anhydrite into gypsum and at later age is due to the formation of ettringite and tobermorite, as a reaction of slag with lime produced during the hydration of cement. These cementitious phases fill in pores and voids of the hydrating gypsum crystals to form a dense and compact structure of low porosity and low pore volume. The reaction products formed during the hydration period were confirmed by scanning electron microscopy and X-ray diffraction. The reduction in porosity and low pore volume of binders, as studied by mercury intrusion porosimetry, are responsible for attainment of high strength and better stability towards water in composite binders than the conventional gypsum plaster.

1. Introduction

Every year, more than 300 million tonnes of industrial solid wastes are being produced by various industries in India and governments are seeking ways to reduce the dual problems of disposal and health hazards from the accumulation of by-products. In recent years, the use of waste materials in the construction industry has become an important option, as it offers cost reductions, energy savings, and reduced CO_2 emissions from the production of Portland cement, as well as reduced environmental impacts of construction materials. The predominant industrial by-products that can be effectively used include chemical gypsum, slag, fly ash and lime sludge.

Out of different waste materials being generated, the use of by-product gypsum is significant not only from the point of view of its disposal, but to avail the dross for conserving high quality natural gypsum and to produce new building materials. By-product gypsum is available to an extent of about 13.0 million tonnes per annum in India from phosphoric acid, hydrofluoric acid and intermediate dye industries and not more than 15.0% is utilized by the cement industries, soil reclamation etc. The limited application of the by-product gypsum is due to the adverse impacts of certain undesirable impurities present in the gypsum on the engineering properties and performance of building materials. Gypsum products are known for their fire resistance, thermal insulation and acoustic properties. However, due to its low water resistance and mechanical strength, gypsum is not a suitable material for external construction works [1–3]. The protection of gypsum requires prevention against penetration of moisture to avoid any damage to the plaster. The gypsum obtained from different sources can be utilized as supplementary raw materials in the production of composite binders as investigated by various researchers. Some attempts were made to produce gypsum-cement-pozzolana binders [4–6] with phosphogypsum. Investigations revealed that a gypsum binder made of a phosphogypsum anhydrite and blast furnace slag mixture 70–24% with appropriate activators achieved 23 MPa strength after 28 d curing at 27 °C under high humidity [7] and a mixture composed of 75% gypsum, 20% OPC and 5% micro silica as cementitious binder developed a strength of up to 17 MPa after 200 d under water [2,8]. Other researchers reported that blends of 41% gypsum, 41% OPC and 18% natural pozzolana gained 22 MPa strength after 95 d; blends of 50–80% fluorogypsum, 15–50% OPC and 0–5% fly ash achieved 32 MPa strength after 180 d and blends of 75% fluorogypsum with blast furnace slag and metakaolin samples showed 14 MPa strength after 360 d of curing in wet conditions [9–11]. The above quoted references reported cementitious systems of various compositions using different sources of gypsum, which developed strength during various curing periods.

In India about 4.0 million tonnes of fluorogypsum, a cardinal waste material of the hydrofluoric acid industry, is produced per annum and poses the problem of disposal and pollution hazards

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as it sets too slowly to form any useful building materials, thus creating an environmental nuisance. The literature survey revealed that not much work has been done on the utilization of fluorogypsum as a main component in composite binders. The objective of this research was to convert fluorogypsum into a composite binder by enhancing its hydration properties and performance through the use of an appropriate processing method. Underlining this, a systematic study is undertaken for the utilization of fluorogypsum, as a main component, by admixing it with blast furnace slag and ordinary Portland cement into a composite binder suitable for construction work. The mechanical properties, hydration mechanism, microstructure and durability under water of the composite binders are discussed in the paper. A comparison between the strength and performance in water of the composite binders and commercially available gypsum plaster (β-$CaSO_4.1/2H_2O$) used as a reference material is also presented.

2. Experimental and materials

2.1. Raw materials

2.1.1. Fluorogypsum

The sample of fluorogypsum was analysed for various constituents such as SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, CaF_2 and pH as per standard test procedures [12,13] and characterised by differential thermal analysis (DTA) (Stanton Red Croft, UK), X-ray diffraction (Rigaku D-Max 2200) and scanning electron microscopy (SEM) (LEO 438 VP, UK). The results of the chemical analysis given in Table 1 show that fluorogypsum possesses a high purity along with free acid (pH 2.8), which is responsible for its hygroscopic nature and may corrode the grinding media. Therefore, the pH of the fluorogypsum was adjusted to 8–8.5 by addition of 0.5–1.0% of lime ($Ca(OH)_2$) and then ground in a ball mill to a fineness of 90% passing through a 90 μm IS sieve.

2.1.2. Granulated blast furnace slag

The chemical analysis of granulated blast furnace slag tested as per IS:4032-2005 [14] is shown in Table 2. The composition of blast furnace slag satisfied the requirements of IS: 12089-2008 [15] as the ratio of CaO + MgO + Al_2O_3 to SiO_2 was found to be 1.6, which was greater than a minimum value of one as specified in the standard. Therefore, it was found suitable for making Portland slag cement and other cementitious materials [16,17].

2.1.3. Portland cement

The physical and chemical analysis of Portland cement tested as per Indian Standards [14,18] are given in Table 3.

2.2. Preparation and testing of composite binders

The mixture composition of composite binders prepared by blending the beneficiated ground fluorogypsum with granulated blast furnace slag (Fineness 400 m^2/kg (Blaine), Portland cement and activators in different proportions followed by inter-grinding in a ball mill to a fineness of 410 m^2/kg (Blaine) are given in Table 4. The composite binders were tested for different properties as per methods specified in Indian Standards and compared with properties of conventional gypsum plaster or Beta-hemihydrate plaster (β-$CaSO_4.1/2H_2O$).

Table 1
Chemical composition of fluorogypsum.

Constituents	(%)
CaF_2	1.32
SiO_2 + insoluble in HCl	1.65
Al_2O_3+Fe_2O_3	0.65
CaO	42.2
MgO	0.05
SO_3	55.1
Loss on ignition	0.31
pH	2.8

Table 2
Chemical composition of granulated blast furnace slag.

Constituents	(%)
Silica	36.8
Al_2O_3	16.5
Fe_2O_3	0.36
CaO	34.9
MgO	7.46
$Na_2O + K_2O$	1.7
S	0.92
MnO	0.85
SO_3	0.37
Insoluble residue	0.039

Table 3
Physico-chemical properties of Portland cement.

Property studied	
Chemical constituents (%)	
Silica	24.2
Al_2O_3	3.39
Fe_2O_3	3.2
CaO	62.62
MgO	3.21
$Na_2O + K_2O$	1.7
SO_3	1.8
Loss on ignition	0.45
Physical properties	
Specific gravity	3.1
Setting time (min)	
Initial	240
Final	300
Compressive strength (MPa)	
3 d	33.0
7 d	45.0
28 d	56.5

2.3. Testing

2.3.1. Setting time

The initial and final setting times of composite binders were determined using a Vicat needle as per Indian Standard [18].

2.3.2. Compressive strength and bulk density

To determine the compressive strength and bulk density of the composite binders, cubes of size 25 mm were cast at normal consistency. The samples were cured for 24 h at room temperature and then placed at 27 °C ± 2 °C in a sealed desiccator containing water for different hydration periods up to 90 d. The specimens cured for respective hydration periods were dried at 42 °C ± 2 °C for 2 d and the compressive strength was determined as per IS: 4031(2002). The average value of six specimens is reported. The compressive strength of composite binders was within a 5% variation level of the arithmetic average. The bulk density of the dried specimen in kg/m^3 was calculated by dividing the mass of the specimen by the overall volume of the cube.

2.3.3. Transverse strength

The specimens of size 200 mm × 25 mm × 25 mm hydrated for 28 d were tested for transverse strength under three point load-

Table 4
Mix proportions of composite binders.

Designation	Mix Composition (% by wt.)				
	Fluorogypsum	Granulated blast furnace slag	OPC	Activator	
				Na_2SO_4	$KAl(SO_4)_2$
F1	70	20	10	1	–
F2	75	15	10	1	–
F3	50	48	2	1	–
F4	70	20	10	–	1

ing on a span of 150 mm in a universal testing machine. A uniform cross head speed of 2.5 mm per minute was applied, until failure.

2.3.4. Soundness and chemically combined water

The soundness of binder samples was tested by the Le-Chatelier Clamp expansion test as per the method described in IS: 6909-1994 [19] and chemically combined (bound) water of the hydrated binder was determined as per the ASTM standard: C 471M-01(2012) [13].

2.3.5. Thermal conductivity

To assess the performance of gypsum based composite binder under elevated temperatures, it is important to determine its thermal conductivity. This property plays a significant role for the use of composite binder as a construction material with thermal comfort. The thermal conductivity of the hydrated composite binder was determined by the guarded hot plate method [20]. In this method two identical specimens of size 300 mm × 300 mm × 12 mm prepared from the composite binder were placed on either side of a horizontal heater assembly and sandwiched between the cooling plates. This arrangement was then placed in a large insulated box packed with insulating material to reduce edge losses and convective heat transfer.

2.3.6. Performance in water

The binder cubes were dried to a constant mass at 42 °C ± 2 °C and then immersed in tap water for different periods. The temperature of the tap water was maintained at 25 °C ± 2 °C and the water was changed successively after each 7-d immersion period. The water absorption was tested as per the method given in the Indian Standard [21]. The porosity was determined by using the following relationship as reported in the literature [22]:

$$\text{Porosity} = \frac{W\,ds}{dw} \qquad (1)$$

where W is the mass gain, ds is the dry density of gypsum cement and dw is the density of water. For the measurement of pore-size distribution, a fragment of each hydrated specimen with a mass between 0.5 g and 1.0 g was taken from the inner one third of the 25 mm cube sample. The sample was dried to constant mass in an oven at 42 °C ± 2 °C and then stored in a vacuum desiccator over silica gel until being tested. The pore size distribution was determined by using a "Pore sizer 9320" mercury intrusion porosimeter with a maximum mercury intrusion pressure of 210 MPa and the value of the contact angle was taken to be 117°.

2.4. XRD and microstructure studies

X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques were used to study the reaction products formed during the hydration of composite binders which are responsible for the progressive enhancement of strength. The inner portion of crushed hydrated binder samples was taken and dried in an oven at 42 °C ± 2 °C and then ground with a mortar and pestle to a fineness sufficiently passing through a 150-μm IS sieve for these studies.

3. Results and discussion

3.1. Characterization of fluorogypsum

The results of the characterization of fluorogypsum as evaluated by DTA, XRD and SEM are given in Figs. 1–3 respectively. DTA (Fig. 1) shows the appearance of endothermic and exothermic peaks at 140 °C, 250 °C, and 930 °C. These peaks are due to the conversion of poorly weathered dihydrate into hemihydrate ($CaSO_4 \cdot \frac{1}{2} H_2O$), soluble anhydrite ($CaSO_4$ (II)) and anhydrite ($CaSO_4$ (I)) plaster. The XRD (Fig. 2) pattern shows that major peaks are of anhydrite having high intensity and minor peaks of gypsum ($CaSO_4 \cdot 2H_2O$) with low intensity. The SEM (Fig. 3) shows that the majority of crystals in fluorogypsum were anhedral to subhedral prismatic interspersed with lathes in the agglomerated form. Twinning of some crystals may also be noted.

3.2. Properties of composite binders

The properties of the composite binders are reported in Table 5. It can be seen that all mixture compositions F1, F2, F3, and F4 have much longer setting time, higher flexural strength and lower consistency as compared to commercial available gypsum plaster. The soundness of all mixture compositions is within the maximum specified value of 5 mm as prescribed in the Indian standard. However, the mix composition F1 has comparatively lower values of consistency, setting time, and soundness and a higher value of flexural strength as compared to other mixture compositions.

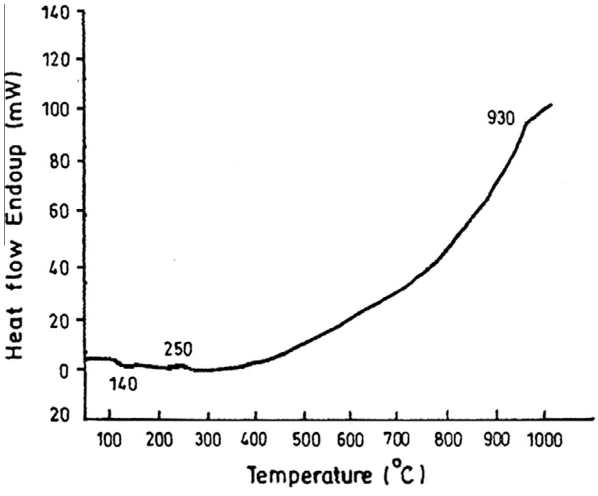

Fig. 1. Differential thermogram of fluorogypsum sample.

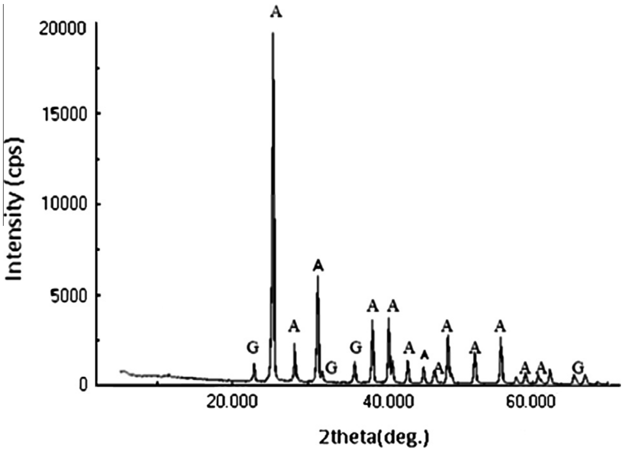

Fig. 2. X-ray diffractogram of fluorogypsum sample (A: Anhydrite, G: Gypsum).

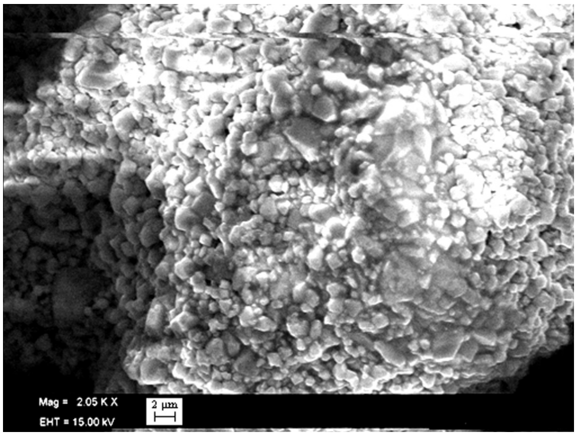

Fig. 3. Scanning electron micrograph of fluorogypsum sample.

3.3. Compressive strength

The development of compressive strength of composite binders and gypsum plaster with hydration periods are shown in Fig. 4. It can be seen that strength of composite binders increased considerably with the increase in hydration period, but the strength of the gypsum plaster remained almost constant after one day of curing. Furthermore, the composite binders have higher strengths than that of the gypsum plaster. The enhancement in the strength of composite binders with curing period clearly indicates that the porous structure formed in the early stage is successively being filled up by hydration products (ettringite and tobermorite). As such, the gypsum matrix becomes denser, contributing high strength to the composite binders. The chemically combined water of different mixture compositions of composite binders hydrated for different periods shown in Fig. 5 revealed that chemically combined water of all compositions increased with hydration period, which confirmed the increased formation of gypsum from anhydrite plaster and of ettringite and tobermorite. The maximum achievement in compressive strength and a higher value of chemically combined water were observed for F1.

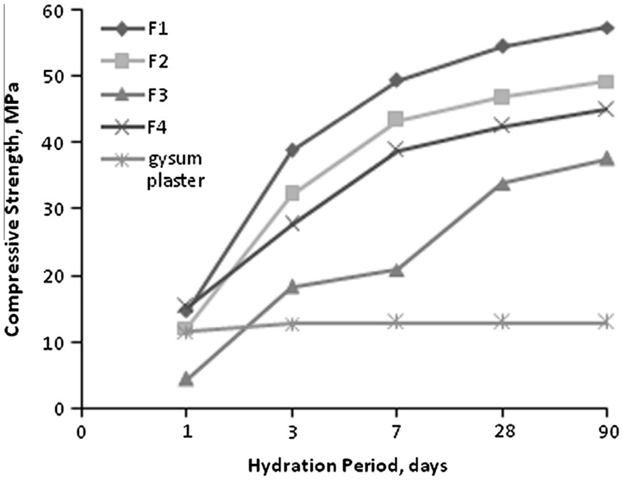

Fig. 4. Effect of hydration period on compressive strength of composite binders.

3.4. Water resistance of composite binders

The water absorption and compressive strength of composite binders of mixture compositions F1, F2, F3, and F4 were measured after different immersion periods in water as depicted in Figs. 6 and 7, respectively. Fig. 6 shows that the water absorption of all compositions increased with an increase in immersion period and is much lower than that of the gypsum plaster. The absence of leaching in composite binders imparts an acceptable water resistance property to them as compared to the conventional gypsum plaster which showed leaching after 3 d immersion in water.

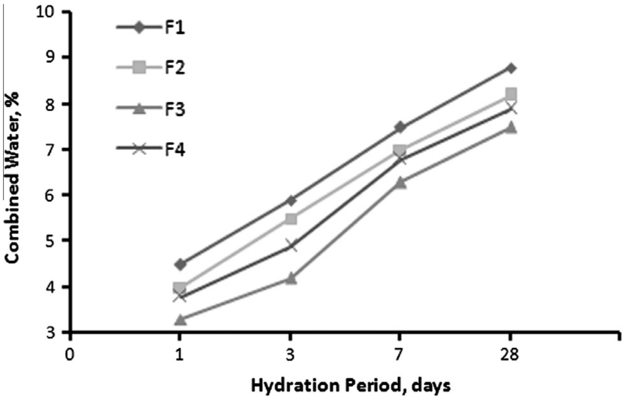

Fig. 5. Effect of hydration period on chemically combined water of composite binders.

Table 5
Physical properties of composite binders.

Designation	Consistency (%)	Setting time (min)		Flexural strength, 28 d (MPa)	Soundness (mm)	Thermal conductivity (W/mk)
		Initial	Final			
F1	28	62	194	7.3	0.90	0.17
F2	29	68	210	6.9	0.95	0.175
F3	29.8	51	185	4.8	1.0	0.168
F4	29.5	75	240	5.5	1.2	0.163
Gypsum plaster	63	25		2.6	–	0.13

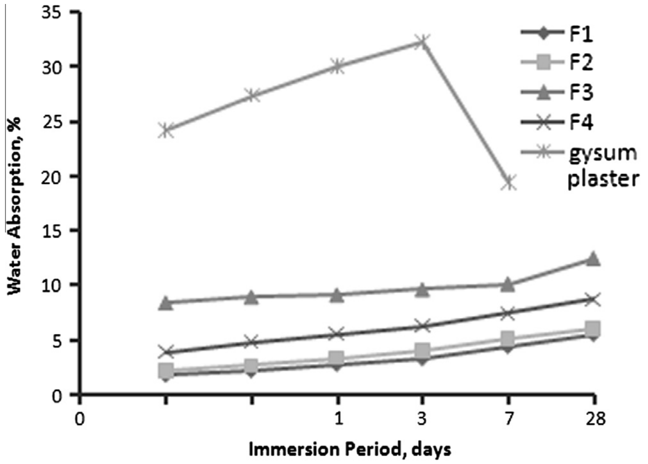

Fig. 6. Effect of water immersion period on the water absorption of composite binders.

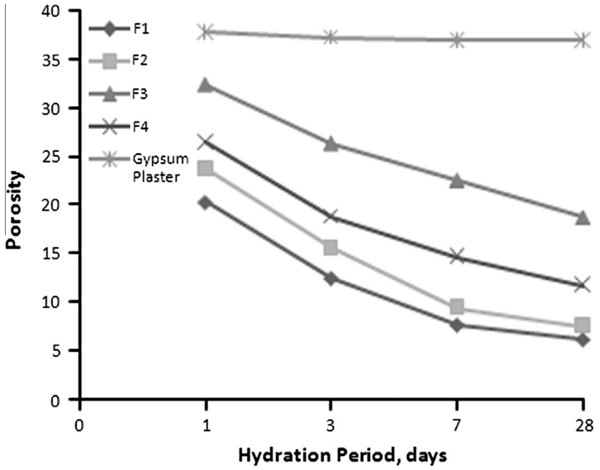

Fig. 8. Effect of hydration period on porosity of composite binders.

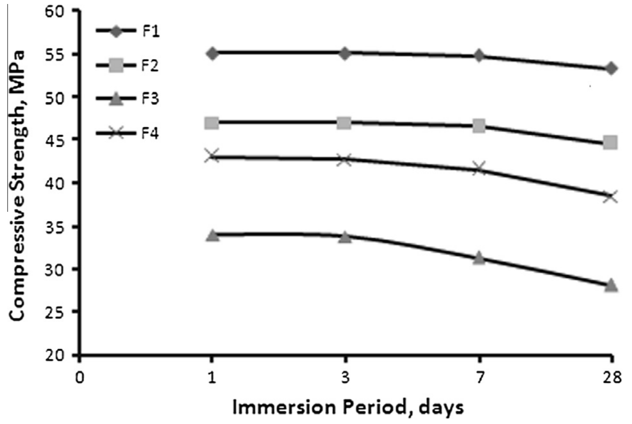

Fig. 7. Effect of water immersion period on the compressive strength of composite binders.

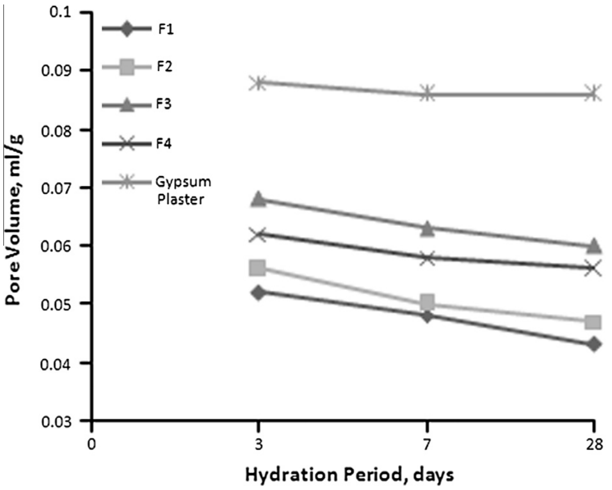

Fig. 9. Effect of hydration period on total pore volume of composite binders.

It can be seen from Fig. 7 that with the increase in immersion period, the strength of the composite binders ranked in the following order: F1 > F2 > F4 > F3. It was noticed that after 28 d immersion of the specimens in water, the mixture compositions retained F1: 96.7%, F2: 94.6%, F3: 82.8% and F4: 89.5% strength of their pristine values.

3.5. Porosity and pore volume

The attainment of high strength and low water absorption with hydration period may be correlated with porosity and refinement of pore volume of these composite binders. The results for porosity and total pore volume intruded by mercury with hydration period are shown in Figs. 8 and 9, respectively. It can be seen from Fig. 8 that the porosity decreased with increase in the hydration period of all mixture compositions. The rate of decrease of porosity was higher at early stages of hydration, but became approximately linear after 7 d of hydration in all compositions. No change in porosity of gypsum plaster was observed with curing period (Fig. 8). Fig. 9 illustrated that the total pore volume of composite binders decreased with hydration period whereas the total pore volume of gypsum plaster was found to be independent of the hydration period. It can be further seen (Figs. 8 and 9) that the porosity and total pore volume of gypsum plaster is much higher than those of the composite binders. These results amply demonstrated the higher pore structure refinement for the composite binders with hydration period than that occurring in gypsum plaster.

3.6. Hydration of composite binders

The acceleration of setting time, development in strength, and water resistance of composite binders with hydration period can be attributed to an increase in solubility and rate of dissolution of gypsum anhydrite in the presence of chemical activators. In fact, the chemical activators added to the composite binder react with the anhydrite $CaSO_4$ obtained from fluorogypsum as per the mechanism given below:

$$\underset{\text{Anhydrite}}{mCaSO_4} + \text{Activator}\cdot nH_2O \text{ -------- } \underset{\text{unstable double salt}}{\text{Activator}\cdot mCaSO_4\cdot nH_2O}$$

$$\text{Activator}\cdot mCaSO_4\cdot nH_2O \text{ ------- } \underset{\text{Gypsum}}{mCaSO_4\cdot 2H_2O} + \text{Activator}\cdot(n\text{-}2m)H_2O$$

$$\text{Activator}\cdot mCaSO_4\cdot nH_2O - \underset{\text{Gypsum}}{mCaSO_4\cdot 2H_2O} + \text{Activator}\cdot(n-2m)H_2O.$$

These unstable double salts formed by chemical activators, concentrate on the surface of anhydrite $CaSO_4$ and are then decomposed into various ions (Ca^{2+} ions, activator cations, SO_4^{2-}). These ions bind water to form gypsum crystals. The above mechanism can be correlated with the findings of Wenheng et al. [23].

The addition of granulated blast furnace slag and Portland cement enhanced the water resistance of composite binders as Al_2O_3 and SiO_2 contributed by the slag in presence of $Ca(OH)_2$ released during the hydration of Portland cement, reacts with gypsum to form ettringite and tobermorite as per the following chemical reactions:

$$\underset{\text{Slag glass}}{16CaO\cdot 13SiO_2\cdot Al_2O_3} + 3CaSO_4\cdot 2H_2O + Ca(OH)_2 \downarrow \underset{\text{Ettringite}}{3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O} + \underset{\text{Tobermorite}}{13CaSiO_3\cdot xH_2O}$$

These cementitious phases fill in the pores and voids of the hydrating gypsum crystals and thus the porosity of the binder is further reduced leading to a hard, dense and compact matrix having better stability and higher resistance towards water than the plain gypsum plaster. These results corroborate the findings of Yang et al.

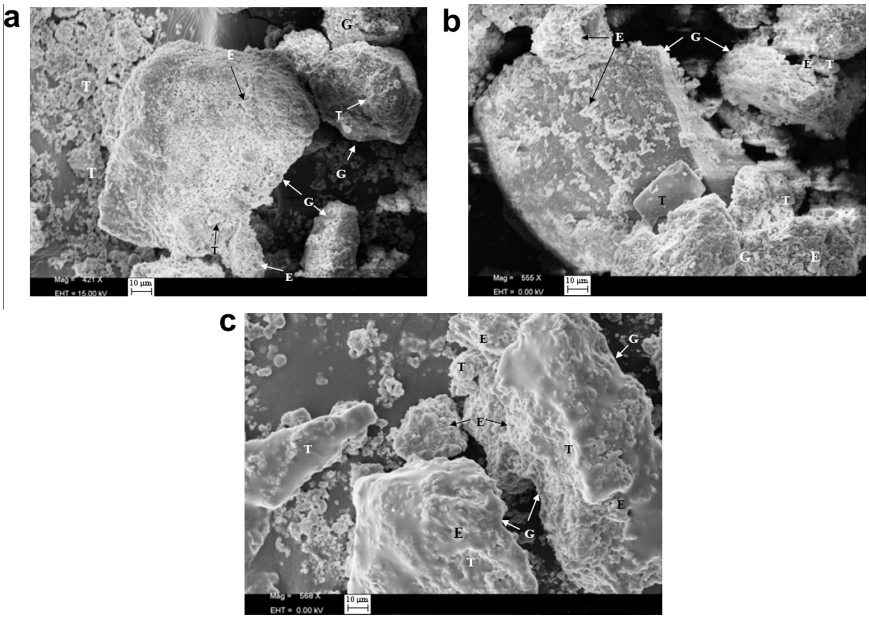

Fig. 10. Scanning electron micrographs of composite binder 'F1' hydrated for (a) 3 d, (b)7 d and (c) 28 d (G: Gypsum, E: Ettringite, T: Tobermorite).

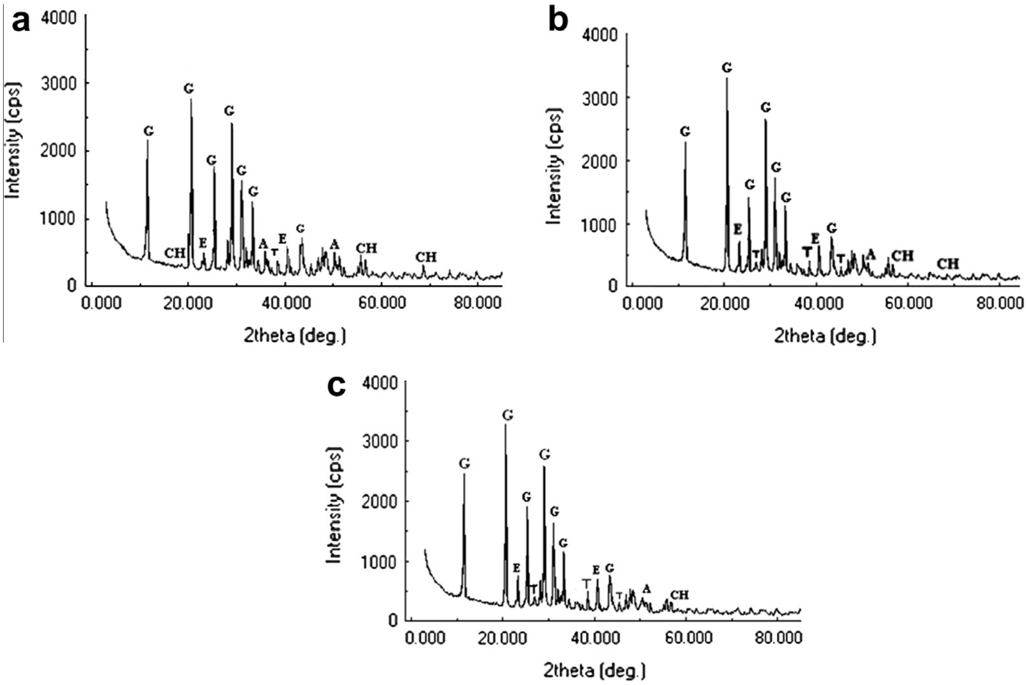

Fig. 11. X-ray diffractograms of composite binder 'F1' hydrated for (a) 3 d, (b) 7 d and (c) 28 d (A: Anhydrite, G: Gypsum, E: Ettringite, T: Tobermorite, CH: Calcium hydroxide).

[24]. The mixture composition F1 has been selected for SEM and XRD studies on the basis of maximum achievement of strength and water resistance with curing period.

3.7. Scanning electron microscopy

The SEM images of composite binder F1 hydrated for 3 d, 7 d, and 28 d are shown in Figs. 10a–c, respectively. Fig. 10a shows that at 3 d hydration, anhydrite gypsum binds with water to form short prismatic, tabular and rhombic-shaped crystals, and formation of plate-like gypsum crystals was also observed. The size of gypsum crystals gradually increased with hydration period to make a diamond–shaped crystal structure which is responsible for high strength (Fig. 10b and c). In Fig. 10c, needle and prismatic crystals of ettringite and formation of euhedral-like CSH crystals along with cross cutting gypsum crystals can be seen. The mixture of ettringite and hydrated CSH fill into the porous structure of gypsum crystals and make them denser. This phenomenon reduced the water absorption and porosity of composite binders and imparts acceptable water resistance to them. These findings have been supported by other workers [25,26].

3.8. X-ray diffraction

XRD data at 3 d, 7 d and 28 d for the composite binder F1 are illustrated in Fig. 11a–c, respectively. Fig. 11a shows the formation of prominent peaks of gypsum due to the conversion of anhydrite plaster into gypsum at 11.5^0, 20.7^0, 29^0, 31.2^0, 33^0 and 43.3^0. The appearance of characteristic peaks of calcium hydroxide at 18.5^0, 56^0, and 68^0 indicate the hydration of cement. Furthermore, the diffraction peaks of other reaction products, that is, ettringite at 21.5^0 and 41^0 and tobermorite (CSH) at 38.5^0 and 43^0 were also observed. The peaks of unreacted anhydrite plaster were obtained at 36.8^0 and 52.5^0. It can be further seen that the intensity of gypsum peaks increased and anhydrite plaster decreased with hydration period. It amply demonstrates the continuous conversion of anhydrite into gypsum plaster. It can also be noticed that the intensity of the peaks of ettringite and tobermorite gradually increased while that of $Ca(OH)_2$ peaks decreased due to its consumption as the hydration reaction proceeds.

4. Conclusions

From the study reported above, the following conclusions can be drawn:

1. A composite binder of high strength and low water absorption has been developed using industrial by-products fluorogypsum, granulated blast furnace slag and Portland cement.
2. The formation of gypsum, ettringite and tobermorite were confirmed by scanning electron microscopy and X-ray diffraction.
3. The increase in chemically combined water with the progress of hydration confirmed the conversion of anhydrite plaster into gypsum.
4. The reaction products i.e., ettringite and tobermorite are filling in pores and voids of the hydrating gypsum crystals to form a dense and compact structure with reduced porosity and pore volume, and thus imparts high strength and better stability towards water to the composite binder than the conventional gypsum plaster.
5. The enhancement in properties of the composite binder suggest that it could be used in making prefabricated building elements like bricks, blocks, boards etc. and in other construction applications i.e., masonry, concrete and external and internal plastering works.

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