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lass Fibre Reinforced Water-Resistant Gypsum-ased Composites

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tract

effect of length and content of E-type glass fibre the strength properties (flexural, tensile and act) of water-resistant gypsum binder was lied. The data show that maximum strength was ined on using 4·0% of glass fibres 50 mm long. performance of gypsum binder and that of n plaster composites in water was investigated mmersion. The results show absence of leach- of the matrix in gypsum binder as compared plain plaster composites. The mechanical erties of gypsum binder composites obtained curing in high humidity (> 90·0% relative idity at 27 ± 2°C) and in water, in air and by ral weathering were compared. In general, an ease in composite strength was noticed but best lts were found when the composites cured in humidity.

he durability of glass reinforced composites has assessed after exposure to alternate wetting drying cycles at 27–50°C. The strength reduced the weight loss increased with the increase in erature. The maximum fall in strength occurs °C.

words: Gypsum binder, composites, glass s, density, flexural strength, wetting and dry- cycles, tensile strength, ettringite, impact gth.

NTRODUCTION

sum plaster, like other inorganic cements, is g in compression but weak in tension and has impact strength. These brittle characteristics ent the effective utilization of the high com- sive strength in structural applications. Some improvement can be achieved by incorporating organic fibres (e.g. sisal, coconut).[1] A much greater improvement could be expected to result by incorporating glass fibre reinforcement in the gypsum plaster matrix and a composite of improved tensile and impact strength can be produced.[2] The fire resistance of such composites is far superior to that of plaster reinforced with organic fibres.[3] The strength characteristics of the glass reinforced gypsum composites can approach that of sheet timber but they have higher densities and fixing problems.[4]

It is well known that gypsum plaster and plaster products are not suitable for external use on account of the solubility of gypsum in water (2 g/litre). Attempts have occassionally been made to make gypsum water-repellent.[5–7] Owing to the high cost of these treatments, the processes developed so far could not be adopted commercially. Therefore, a water-resistant gypsum binder suitable for masonry work and plaster has been developed utilizing by-product phosphogypsum plaster, slag/fly ash, cement and a small quantity of organic retarder.[8] In India, about 4 million tonnes of phosphogypsum is produced annually from over a dozen phosphoric acid fertilizer plants. It contains impurities such as phosphate, fluoride, quartz and organic matter[9] which adversely affect the setting and hardening of plaster products. Extensive work regarding its processing and utilization has been accomplished at the Central Building Research Institute, Roorkee.[10,11]

There are two main methods of reinforcing gypsum with fibre materials. One method is to concentrate the fibres in the tensile zone of the resulting structural element so as to match the external tensile force and use the matrix to match the external compressive force. The other method

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to disperse the glass fibre uniformly in the trix so as to form a homogeneous mixture. This ures a high degree of stress distribution by the e, which then acts as a crack arrester. The nogeneous dispersion of the glass fibre in the trix ensures uniform distribution of fibres and ss when the resulting composite material is med into sheets. It is this method which has n selected for the development of new compo- material in the present work.

Glass fibre reinforced gypsum binder com- ites were produced by using E-type glass fibre newly developed water-resistant gypsum der. The effect of glass content on the proper- of composites and its durability, studied by ernate wetting and drying cycles at 27–50°C in ural weathering, in air (at ambient tempera- e) and in water, is reported in this paper. The ults of these investigations are discussed.

MATERIALS

Gypsum binder

the present study, a water-resistant gypsum der was produced by blending ground granu- d slag, ordinary portland cement and an anic retarder with calcined phosphogypsum hemihydrate) in a ball mill to obtain a uniform duct. Phosphogypsum processed by washing neutralization was used for making the cal- ed material. The main hydration products in gypsum binder were indentified as gypsum, ingite and tobermorite. The binder possesses d water resistance as it does not show leaching water up to 28 days of immersion, while plain ster shows leaching after three days of immer- n in water.

The physical properties and chemical composi- n of the gypsum binder are given in Tables 1 2.

Glass fibre

the reinforcing material in this programme, pped uncoated E-type glass fibres (FGP India , New Delhi, India) were used. The physical perties of the glass fibre are given in Table 3.

EXPERIMENTAL PROGRAMME

Preparation and testing of composites

spray suction technique was used to cast the ss fibre reinforced composites. An air-tight funnel was fabricated with a top made of a perforated metallic plate. The funnel was attached to a vacuum pump. Before casting composites, a wet linen cloth was placed on the perforated plate followed by brass moulds of dimensions 150 mm × 50 mm × 12 mm. A gypsum binder–water slurry was prepared at 67% consistency (quantity of water required for 100 g of binder to produce a workable mix) and poured into the brass moulds up to a thickness of 5 mm. The chopped glass fibres were then placed randomly over the surface of the gypsum binder followed by another layer of gypsum binder and glass fibres. After the extraction of water for 15 min, the sheets were demoulded and stored under 90% RH at 27 ± 2°C for a period of 1–28 days.

For impact and thermal conductivity tests, the glass fibre reinforced boards (250 mm × 250 mm × 12 mm) were cast and cured in high humidity in a sealed container for a period of 28 days and then dried at 42°C for two days.

Table 1. Physical properties of water-resistant gypsum binder

Fineness, cm^2/g	3 100
Setting time, min	
Initial	70
Final	145
Bulk density, g/cm^3	1·2
Compressive strength, MPa (28 days)	35·0
Soundness, mm	1·60
Water absorption, %	6·0
pH	11·5

Table 2. Chemical composition of gypsum binder

Consituent	*Percentage by wt*
SiO_2 + insoluble in HCl	8·20
$Al_2O_3 + Fe_2O_3$	9·00
CaO	37·30
MgO	1·80
SO_3	39·65
P_2O_5	0·15
F	0·058
Organic matter	0·090
$Na_2O + K_2O$	0·089
Loss on ignition	4·10

Table 3. Physical properties of E-type glass fibre

Diameter of the fibre filament, μm	8–10
Number of filaments in a strand	204
Tensile strength of glass fibre, MPa	1750
Young's modulus of glass fibre, MPa	6890–7600

TESTING

1 Flexural strength
exural strength was determined on 50 mm 150 mm specimens tested under three-point ading on a span on 135 mm in a universal test-g machine. A constant cross-head speed of 2·5 m/min was used for all flexural tests.

2 Tensile strength
) determine tensile strength, 25 mm × 150 mm ecimens were tested. The specimens were ımped in the flat grips of the Instron testing achine, which had a self-aligning joint at the top .d loaded at an overall elongation rate of 2·0 m/min; the ultimate load was recorded. The uge length of the specimen was maintained at ; mm during the testing for all samples.

3 Impact strength
falling-weight method as specified in IS2380-)63 was adopted for testing impact strength. ccording to this method, the test specimens (250 m × 250 mm × 12 mm) were evenly supported a rebated square frame without fastening. A ɔck, whose weight was previously recorded, ving a mild steel hemispherical end with a dius of 25 mm was allowed to fall first from a ight of 25 mm measured from the upper surface the test specimen and then from successive ights rising in increments of 25 mm until failure the test specimens occurred.

4 Thermal conductivity
e thermal conductivity of gypsum binder mposites was determined by the guarded hot-ıte method as described in the IS3346-1966 ecification of a method for the determination of thermal conductivity of thermal insulation ıterials. In this method two identical gypsum ıder composites of size 300 mm × 300 mm × 12 n placed on either side of a horizontal heater sembly were sandwiched between the cooling ıtes. This arrangement was then placed in a ge insulated box packed with insulating ıterial to reduce edge losses and convective heat nsfer.

DURABILITY STUDIES OF GYPSUM)MPOSITES

e durability of the glass reinforced gypsum nposite was examined by determining its behaviour in (1) water; (2) air, natural weathering and high humidity at 27 ± 2°C; and (3) wetting and drying cycles.

5.1 Wetting and drying cylces

The gypsum composite strips (150 mm × 50 mm), hardened in over 90·0% relative humidity at 27 ± 2°C for a period of 28 days, were subjected to alternate wetting and drying cycles at different temperatures from 27 to 50°C. One cycle of wetting and drying comprised heating the strips for a period of 16 h at different temperatures followed by cooling for 1 h and then immersing them in water for a period of 7 h.[12] After a certain number of cycles, the flexural strength and weight loss of dry strips were determined.

The microstructure of the composites was studied with a scanning electron microscope (SEM; Phillips, The Netherlands).

6 TEST RESULTS AND DISCUSSION

6.1 Optimization of glass fibre

The effect of glass fibre, i.e. its content and length, on the physical properties of the gypsum binder was studied. The relationship between flexural strength and glass content at different fibre lengths is given in Fig. 1. It can be seen that the strength of the composite reached a maximum value at 4·0% glass fibre content and then fell off. The fall in strength may be attributed to the decrease in the quantity of binder and improper compaction, which induces voids in the composites.

The relationship between tensile strengths and glass content is given in Fig. 2. It indicates a similar type of behaviour to that observed in the case of flexural strength. Figure 3 gives a linear relationship between impact strength and glass content. The increase in impact strength is due to a crack-arresting mechanism induced in the composites by incorporating the glass fibres. A crack originating in the highly stressed tensile zones of the matrix propagates and, on reaching the fibre, grows along the weak interface of the matrix and fibre. Thus, the energy of the impact or fracture is dissipated along the fibre–matrix interface and the fibres are pulled out.

Figure 4 shows the relationship between the density and the glass content. With an increase in the glass content, the density remains fairly constant.

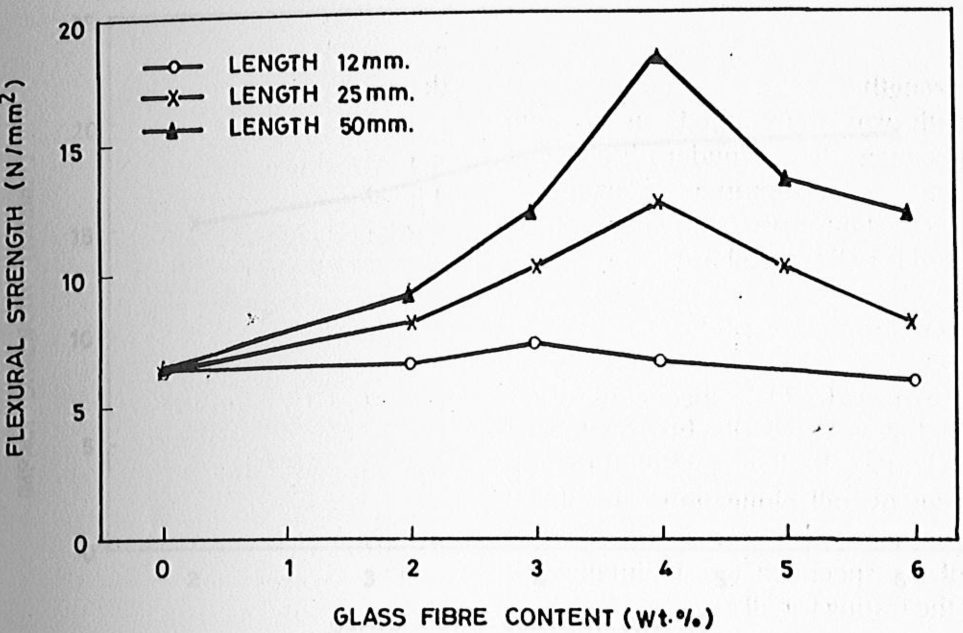


Fig. 1. Effect of glass fibre content on flexural strength.

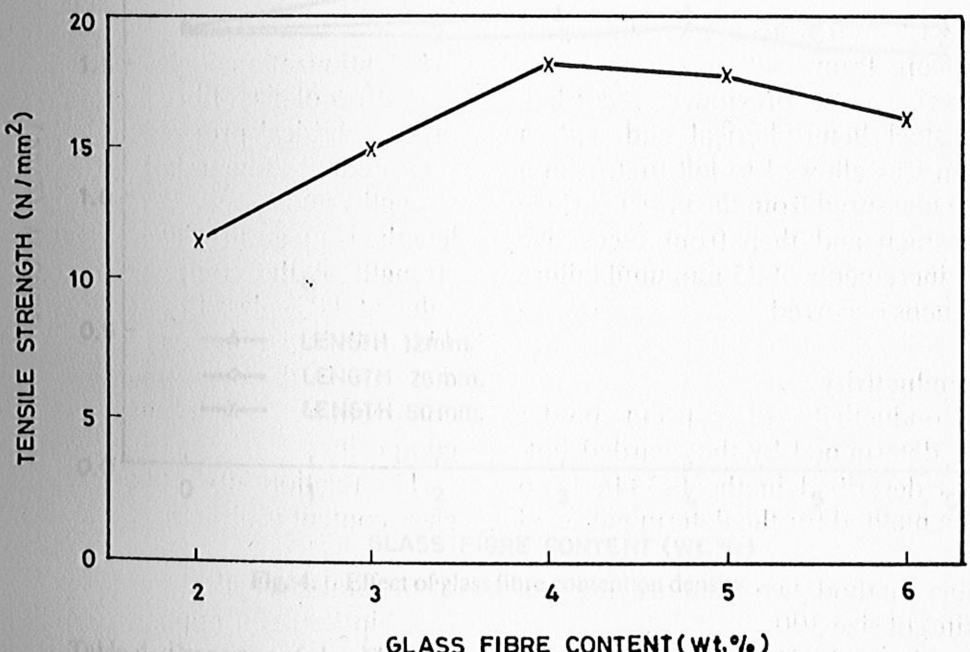


Fig. 2. Tensile strength and glass content of glass reinforced gypsum binder.

here is no variation in the quality of experi- ital gypsum binder composites as measured in is of flexural strength, impact strength and ile strength. The flexural strength of gypsum ler composites (six) was within 7% variation l of arithmetic average while the tensile and act strengths of gypsum binder composites in each case) lay within 5% variation levels of arithmetic average.

On the basis of optimization of glass fibre, composites were cast with 4% of their glass fibres 50 mm long. The properties of these composites were compared with those of plain gypsum plaster composites cast with a similar type of glass fibre. The results are reported in Table 4.

It can be seen that gypsum binder composites have higher strength than the ones made with plain phosphogypsum plaster. The data show that

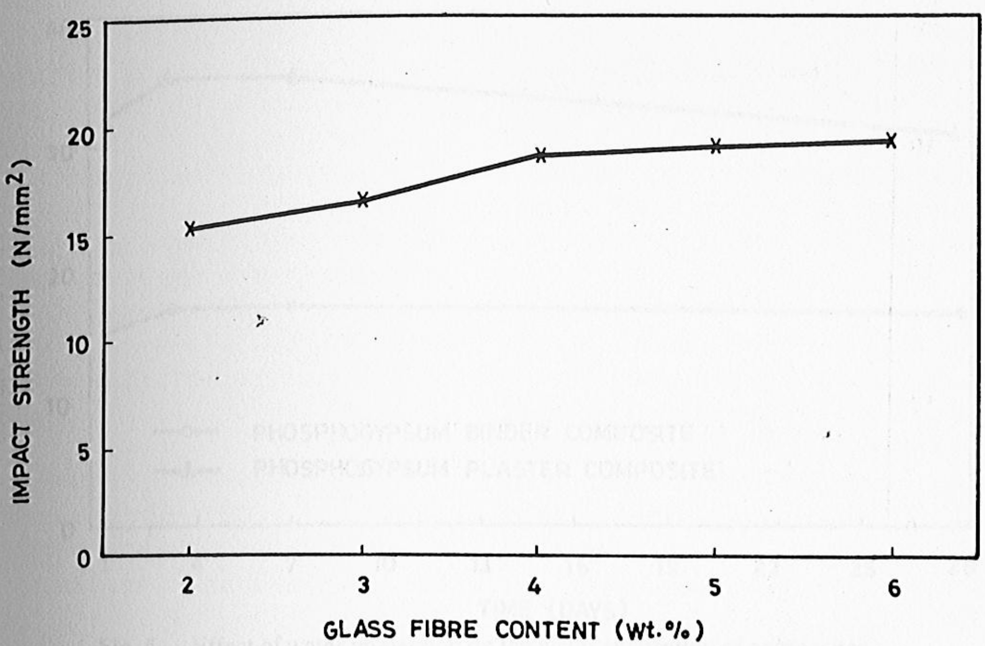


Fig. 3. Impact strength and glass content of glass reinforced gypsum binder.

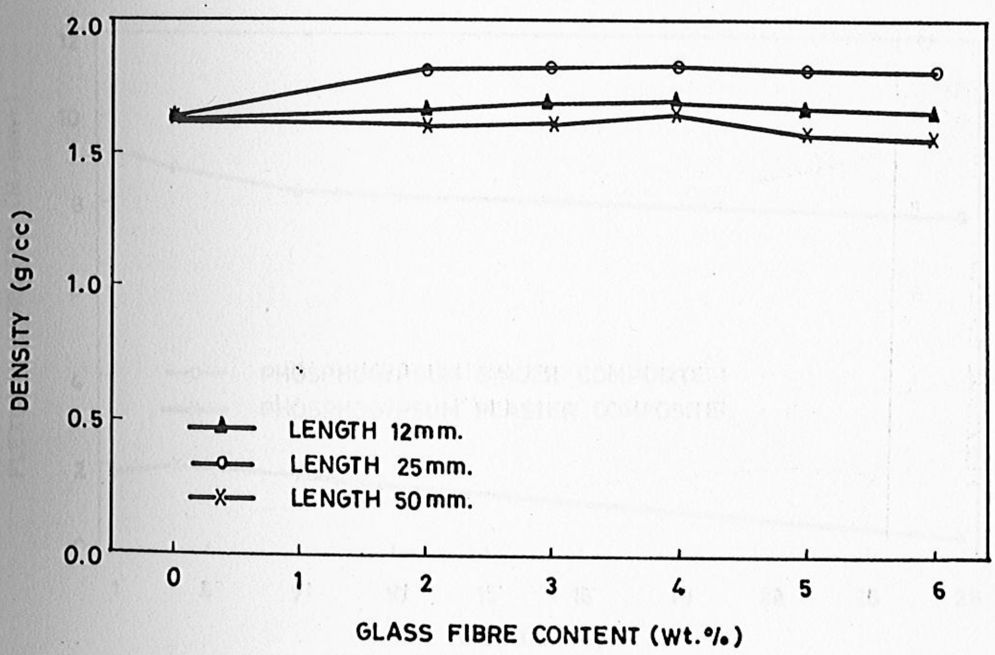


Fig. 4. Effect of glass fibre content on density.

Table 4. Properties of glass fibre reinforced gypsum binder composites

Property	*Gypsum binder composites*	*Plain gypsum plaster composites*
Bulk density, g/cm^3	1·628	1·20
Consistency, %	65·00	81·00
Flexural strength, MPa		
1 day	10·70	4·96
3 days	12·17	4·97
7 days	13·21	4·98
28 days	22·00	4·96
Tensile strength, MPa (28 days)	18·00	2·75
Impact strength, N/mm^2 (28 days)	18·60	10·20
Thermal conductivity, kcal/m h°C	0·09	0·12

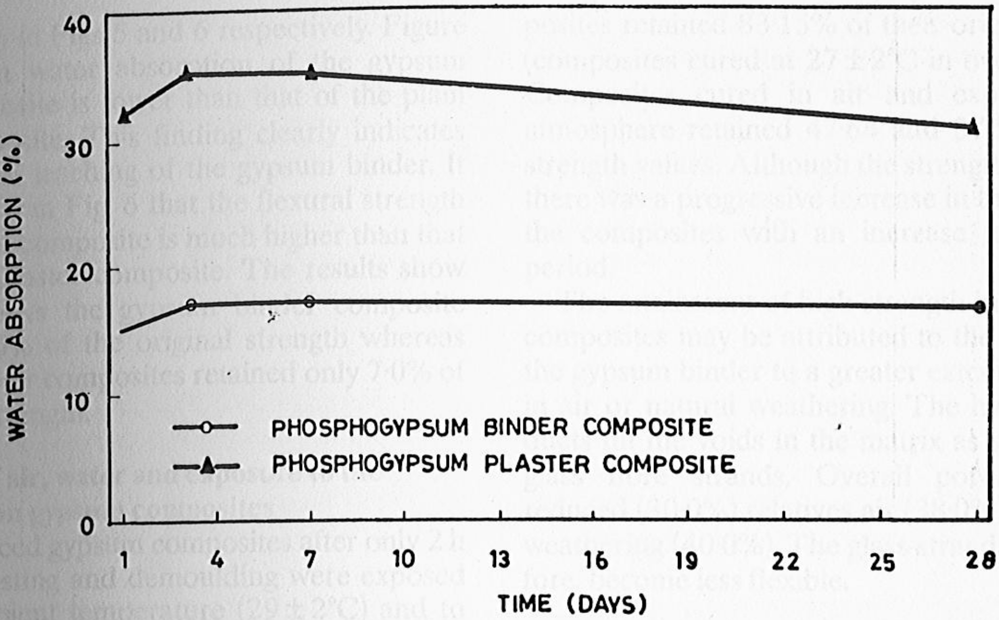


Fig. 5. Effect of water immersion on the water absorption of composites.

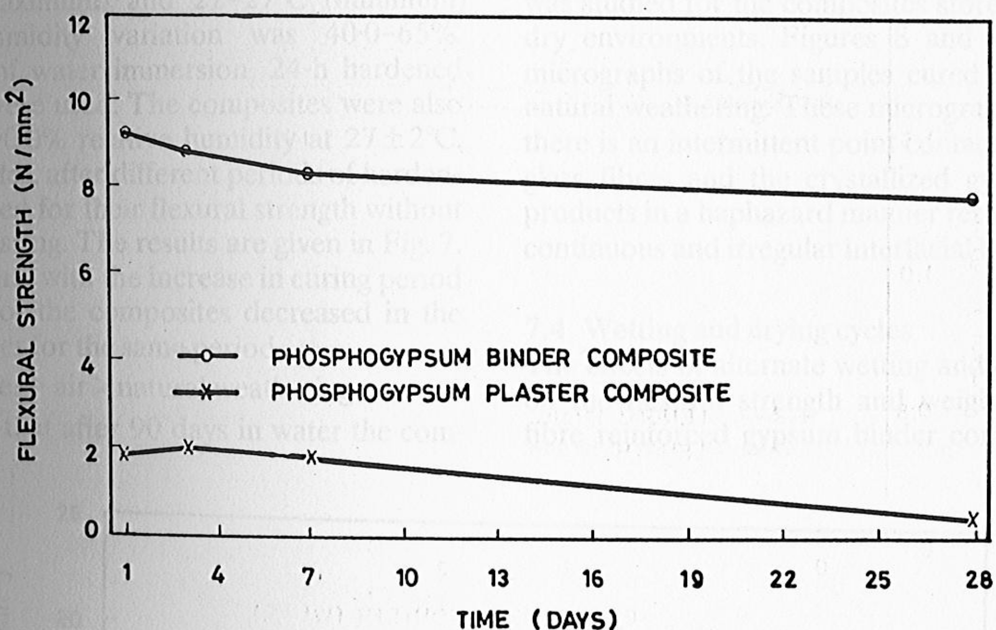


Fig. 6. Effect of water immersion on the flexural strength of composites.

strength of the binder increased from one day 28 days but the strength of the plain gypsum ster composites remained almost constant r one day of curing. The high strength development in gypsum binder over the plain plaster der composites can be ascribed to the nentitious phases formed ($C_3S_2H_x$, C_3AH_6, .$CaSO_4.32H_2O$, $Ca(OH)_2$)[8] and to the low sistency of the binder.

7 DURABILITY OF GYPSUM COMPOSITES

7.1 Performance of composites in water

The fibre reinforced gypsum binder composites hardened for 28 days were dried and then immersed in water to measure their water absorption and flexural strength after different periods.

The effect of immersion in water on the water absorption and flexural strength of binder compo-

:s is shown in Figs 5 and 6 respectively. Figure ;hows that water absorption of the gypsum der composite is lower than that of the plain ster composite. This finding clearly indicates absence of leaching of the gypsum binder. It ı be seen from Fig. 6 that the flexural strength :he gypsum composite is much higher than that the plain plaster composite. The results show t at 28 days the gypsum binder composite ained 42·0% of the original strength whereas plain plaster composites retained only 7·0% of original strength.

Effect of air, water and exposure to the ıosphere on gypsum composites

ɔre reinforced gypsum composites after only 2 h ɛr their casting and demoulding were exposed air at ambient temperature (29 ± 2°C) and to outside atmosphere for a period of 1–90 days. e atmospheric temperature variation was -45°C (maximum) and 22–27°C (minimum) l the humidity variation was 40·0–65%. the case of water immersion, 24-h hardened nposites were used. The composites were also ɔsed to 90·0% relative humidity at 27 ± 2°C. e composites, after different periods of harden- were tested for their flexural strength without her processing. The results are given in Fig. 7. evident that with the increase in curing period strength of the composites decreased in the owing order for the same period.

Water > air > natural weathering

vas found that after 90 days in water the composites retained 83·15% of their original strength (composites cured at 27 ± 2°C in over 90% RH). Composites cured in air and exposed to the atmosphere retained 47·64 and 37·50% of their strength values. Although the strengths decreased, there was a progressive increase in the strength of the composites with an increase in the curing period.

The attainment of high strength in water-cured composites may be attributed to the hydration of the gypsum binder to a greater extent than occurs in air or natural weathering. The hydration products fill the voids in the matrix as well as in the glass fibre strands. Overall porosity is thus reduced (30·0%) relatives air (38·0%) and natural weathering (40·0%). The glass strands may, therefore, become less flexible.

7.3 Microstructure of the interface

The microstructure of the fibre–matrix interfaces was studied for the composites stored in wet and dry environments. Figures 8 and 9 are typical micrographs of the samples cured in water and natural weathering. These micrographs show that there is an intermittent point contact between the glass fibres and the crystallized gypsum binder products in a haphazard manner resulting in a discontinuous and irregular interfacial bond.

7.4 Wetting and drying cycles

The effects of alternate wetting and drying cycles on the flexural strength and weight loss of the fibre reinforced gypsum binder composites kept

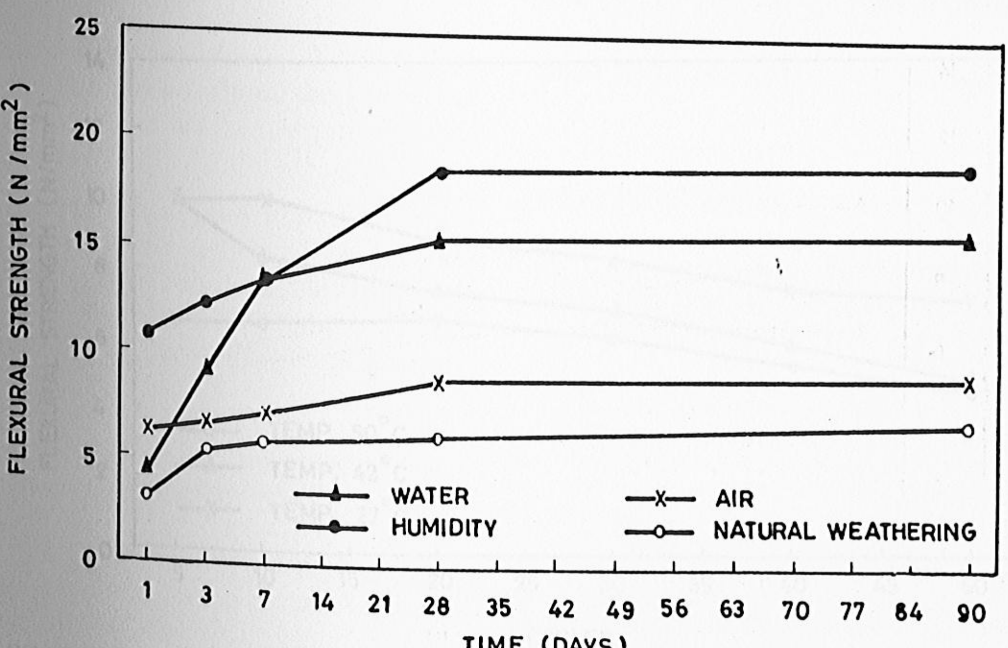


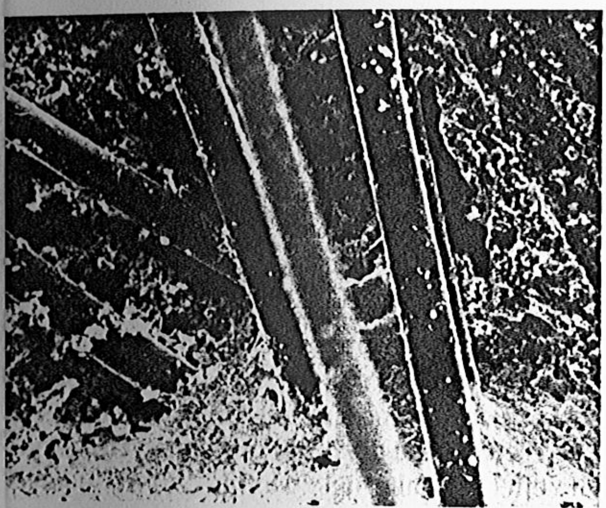
Fig. 7. Durability of composite in water, humidity, air and natural weathering.

temperatures from 27 to 50°C are shown in gs 10 and 11 respectively.

It is clear from Fig. 10 that on increasing the mperature from 27 to 50°C the flexural strength reduced. The maximum fall in strength occurs 50°C. However, there were no cracks in the mposites even after 50 cycles. It can be seen om Fig. 11 that the weight loss increased from 7 to 50°C with the increase in wetting and drying cles. The maximum increase in the weight loss n be noticed at 50°C.

The fall in the strength and increase in the eight loss of the composites can be correlated ith the decomposition of the ettringite ($C_3A.3CaSO_4.32H_2O$) phase formed during the ydration of the gypsum binder and with an increase in the temperature beyond 27°C. This finding is in agreement with the studies carried out by Ghorab & Kishar[13] on the stability of calcium sulphoaluminate hydrates (ettringite) by investigating the effect of increasing temperature on the solubility of sulphoaluminate. It has been found that when the solubility values for sulphate and alumina and the pH are 0·2025 g SO_4^{2-}/litre (168 mg SO_3/litre), 0·0298 g Al/litre (56·2 mg Al_2O_3/litre) and 11·0, respectively, then the ettringite remains stable. Normally the phenomena takes place at 30°C. Similar results were also reported by Lea.[14]

When the temperature is enhanced to 60°C, the solubility of sulphate is increased to 0·45 g SO_4^{2-}/litre after 14 days of stirring the ettringite in water



g. 8. Bonding in glass fibre reinforced gypsum binder red in water (Magnification × 288).

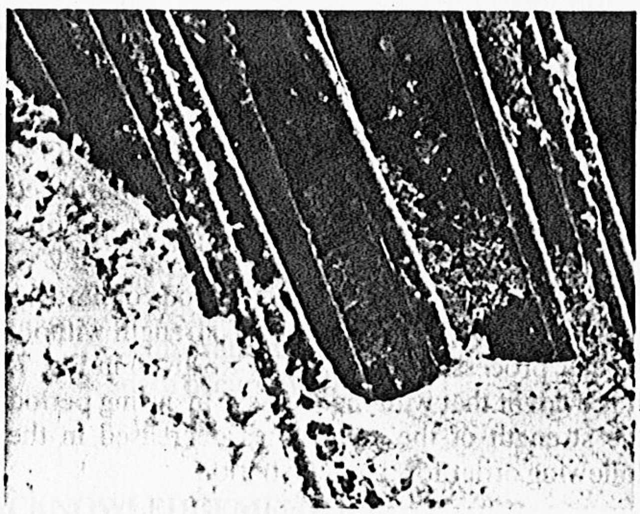


Fig. 9. Bonding in glass fibre reinforced gypsum binder cured by natural weathering (Magnification × 288).

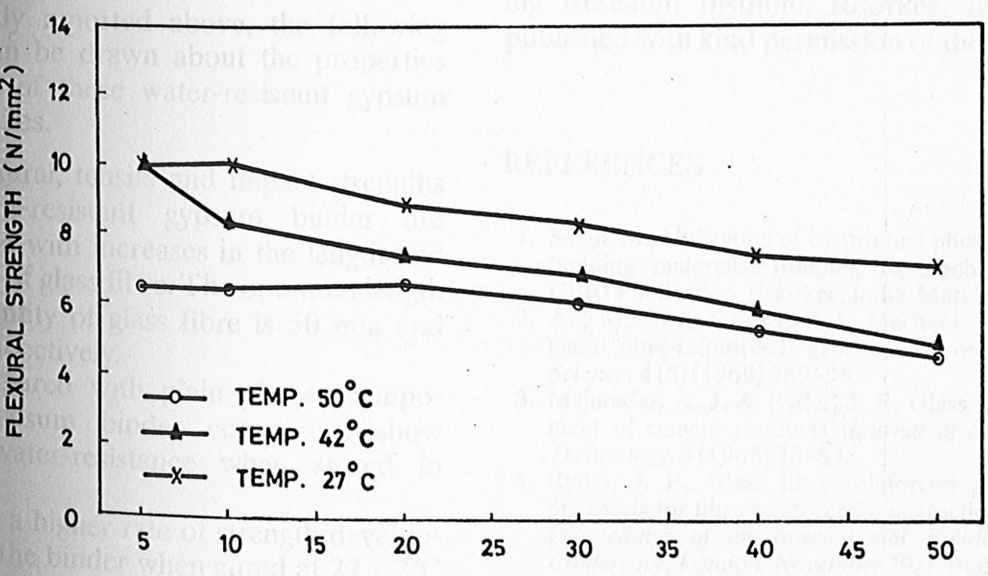


Fig. 10. Effect of alternate wetting and drying cycles on the flexural strength of the composite at different temperatures.

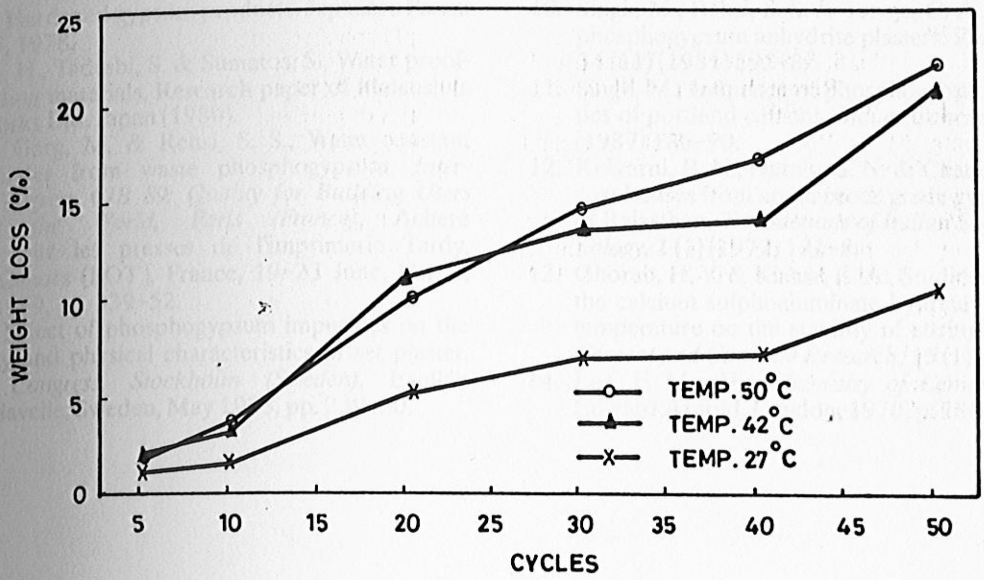


Fig. 11. Effect of alternate wetting and drying cycles on the weight loss of the composite at different temperatures.

ile the aluminium concentration does not differ rkedly from that at 30°C. X-ray diffraction ılysis of the ettringite at 60°C showed broaden- of lines and formation of a few lines belonging monosulphate. The similar behaviour was ;erved in the case of gypsum binder composites) after 50 wetting and drying cycles at 60°C.

CONCLUSIONS

m the study reported above, the following clusions can be drawn about the properties durability of these water-resistant gypsum der composites.

1) The flexural, tensile and impact strengths of water-resistant gypsum binder are increased with increases in the length and quantity of glass fibre. The optimum length and quantity of glass fibre is 50 mm and 4·0% respectively.
2) As compared with plain plaster composites, gypsum binder composites show better water-resistance when stored in water.
3) There is a higher rate of strength development in the binder when cured at 27 ± 2°C in over 90·0% RH than when cured in water, in air or by natural weathering.
(4) When subjected to alternate wetting and drying cycles, the strength is reduced and the weight loss is increased with an increase in temperature. The maximum fall in strength occurs at 50°C.

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REFERENCES

1. Singh, M., Utilization of by-product phosphogypsum for building materials. Building Research Note No. 9, CBRI Publication, Roorkee, India, March 1988.
2. Ali, M. A. & Grimer, F. J., Mechanical properties of glass fibre-reinforced gypsum. *Journal of Material Science*, 4 (5) (1969) 389–95.
3. Majumdar, A. J. & Ryder, J. F., Glass fibre reinforcement of cement products. *Journal of Society of Glass Technology*, 9 (1968) 78–84.
4. Ryder, J. F., Glass fibre reinforced gypsum plaster prospects for fibre reinforced construction materials. In *Proceedings of an International Building Exhibition Conference, London, November 1971*. Building Research Establishment, Garston, 1972, pp. 69–89.
5. Schmidt, H., Fietsch, G., Grohmann, R. & Gruem, J. H., British Patent 22314106, 1980.

Shihara, I., Hardened gypsum products. Japanese Patent 160 52325, 1978.

Mutsuhasu, H., Tadashi, S. & Sumatos, S., Water proofing of building materials. Research paper of Matsushita Electric Works Ltd, Japan (1980).

Singh, M., Garg, M. & Rehsi, S. S., Water resistant gypsum binder from waste phosphogypsum. *International Congress CIB 89: Quality for Building Users throughout the World, Paris (France)*, Achere d'imprimev sur les presses de l'imprimerie Tardy, Quercy a Cahors (LOT), France, 19–23 June, Vol. 2, Theme 2, 1989, pp. 339–52.

Singh, M., Effect of phosphogypsum impurities on the morphology and physical characteristics of set plaster. *9th CIB Congress, Stockholm (Sweden)*, Exellan Grafiska, Gavelle, Sweden, May 1983, pp. 239–50.

10. Singh, M., Rehsi, S. S. & Taneja, C. A., Development of phosphogypsum anhydrite plasters. *Zement-Kalk-Gips*, **34** (11) (1981) 595–8.
11. Singh, M., Influence of phosphogypsum on two properties of portland cement. *Indian Concrete Journal*, **61** (7) (1987) 186–90.
12. Kulkarni, B. N., Narain, S. N. & Chandawat, B. S., Low cost houses from argillaceous grade gypsum in arid zone of Rajasthan. *Transactions of Indian Society Desert Technology*, **2** (1) (1977) 121–8.
13. Ghorab, H. Y. & Kishar, E. A., Studies on the stability of the calcium sulphoaluminate hydrates Part 1: Effect of temperature on the stability of ettringite in pure water. *Cement and Concrete Research*, **15** (1) (1985) 93–9.
14. Lea, F. M., *The Chemistry of Cement and Concrete*, Edward Arnold, London, 1970, p. 186.