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Effects of processing and materials variations on mechanical properties of lightweight cement composites[1]

Seung Bum Park [a], Eui Sik Yoon [b], Burtrand I. Lee [c,*]

[a]*Department of Civil Engineering, Chungnam National University, Taejon, Korea*
[b]*Department of Structural Systems and Site Evaluation, Korea Institute of Nuclear Safety, Taejon, Korea*
[c]*Department of Ceramic and Materials Engineering, Clemson University, Olin Hall, PO Box 340907, Clemson, South Carolina 29634-0907, USA*

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Abstract

Low-density/low-cost cement composites were fabricated. Carbon and alkali-resistant glass fibers were used to reinforce the matrix of industrial by-products; fly ash with silica fume, Portland cement, and calcium silicates were mixed in different proportions. The additional low density was obtained by adding perlite and foaming agents followed by hot water curing. The composites also were prepared by autoclave curing for comparison. The mechanical properties were improved by increasing the amount of silica fume, fly ash, and fibers. Both carbon fibers and alkali-resistant glass fibers were effective in reinforcing the matrices, but carbon fibers were superior to glass fibers. Fabrication techniques for producing lightweight cement composites that can substitute for autoclaved lightweight concrete was developed.

Keywords: Fracture toughness; Mechanical properties; Cement; Silica fume; Fiber reinforcement

Demand is increasing for affordable, lightweight construction materials with superior mechanical properties. Low density in cement composites often is obtained by sacrificing mechanical properties needed for practical construction applications. This problem may be solved by cement composites reinforced with high-performance fibers and other low-cost additives. It has been demonstrated by many researchers [1–20] that carbon fibers are particularly effective in reinforcing and reducing the density of the composites.

For autoclaved lightweight concrete (ALC), quick lime (CaO) usually is added to cement for its effectiveness in strength development and satisfying the hydration behavior. These effects are caused by the formation of tobermorite phase ($5CaO{\cdot}6SiO_2{\cdot}5H_2O$) of calcium silicate from the reaction between silica and lime under a hydrothermal condition. Gypsum ($CaSO_4$) is added to reduce the flow loss by controlling cohesivity and setting time in ALC [21–26].

Calcium silicates (CS) are chemically stable, refractory hydrates with superior thermal insulating properties by the high-porosity body, which is accompanied by the low-density structure.[27] It can be easily fabricated by reacting lime (CaO) and fine silica (SiO_2) at 170°C to 250°C under saturated steam pressure [28]. Ma and Brown [29] explained the reaction and pozzolanic effect of silica and alumina in fly ash with lime and gypsum.

Among the industrial by-products, fly ash is produced from coal-fired power stations in large amounts. It is desirable to utilize the waste by-product for lightweight composite building materials, not only for economic but also for environmental reasons. Fly ash has been shown to be beneficial in cement concretes [19,29–33]. By incorporating fibers, high specific strength and high fracture toughness composites with low density at reasonable cost can be obtained. To demonstrate this possibility, we report the results of our investigation on fabrication of three compositional groups (fibers, low-cost additives, and lightweight additives) of cement composites followed by curing in hot water and in an autoclave for a comparison.

1. Experimental procedure

1.1. Materials

Early strength Portland cement was obtained from Ssangyong Cement Co., Ltd., Korea. Fly ash (FA) of particle size <42 μm was obtained from Boryung Coal-fired

* Corresponding author. Tel.: (+1) 864-656-5348; Fax: (+1) 864-656-1453; E-mail: burtrand.lee@ces.clemson.edu.

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Table 1
Chemical compositions in wt% and physical properties of early strength Portland cement, fly ash, silica powder, and silica fume

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	Ignition loss	Specific gravity at 20°C	Blaine surface area (m^2/g)
Cement	19.2	5.9	2.8	61.7	3.6	4.2	0.15	1.4	3.14	0.450
Fly ash	65.4	25.5	4.3	1.2	1.0	1.0	0.21	3.6	2.14	0.312
Silica powder	92.9	3.8	0.5	0.3	0.8	—	—	1.0	2.60	0.448
Silica fume	92.0	1.7	2.5	0.6	—	—	0.33	—	2.21	26.32

Power Plant (Korea). Silica powder (SP) of 7.0-μm diameter was obtained from Jirae Silica Mining Co., Korea. Fine silica fume (SF) of 1.36-μm particle size was obtained from Anglo-Alpha Co. (South Africa). The compositions and properties of these materials are given in Table 1.

Lime and gypsum (α-$CaSO_4 \cdot 1/2H_2O$) having specific gravity of 2.75 were obtained from Baikwang Materials Co., Korea. To produce lightweight composites, a foaming agent, high-purity hydroxy silicon ester, obtained from Lucky Development Co., Korea, was used in fly ash/perlite composites. For the same purpose, perlite of 2.2-mm mean particle diameter with density of 0.34 g/cm^3 (Samson Perlite Co.) was used. ALC-grade aluminum metal powder used as a foaming agent in fly ash/calcium silicate/cement composites was obtained from Comalco Smelting Co., Australia. For carbon fibers (CF), polyacrylonitrile (PAN)-based CF of average length of 10 mm were obtained from Korea Steel Chemical Co., Ltd. Alkali-resistant glass fibers (GF) were obtained from Asahi Glass Co., Japan and polypropylene fibers (PF) from Fibermesh Co., U.S.A. The relevant properties of the fibers are listed in Table 2. Superplasticizer (SPL) used was obtained from Master Builder Ltd., Korea.

1.2. Mixing and composite fabrication

The optimum mixing ratios of ingredients preventing materials segregation and yielding proper workability were determined by preliminary experiments. Mixing formulations of the cement matrix are given in Table 3. In FA/SF/CS/cement composites the mass% ratio of water to solid was kept constant at 66, and the fiber levels were varied from 0 to 5.0 vol% of the matrix. The aluminum powder foaming agent was added at 0.07 wt% level of the total solids to yield low-density matrix. An Omni Mixer (Chiyoda Technical & Industrial Co., Japan) of 30-l capacity was used to mix the ingredients. The mixing times were 3 min for dry blend of cement, FA, SF, lime, gypsum, and SP followed by 2 min of a primary blend with distilled water and superplasticizer. In the secondary blend, fibers were added in 2 to 3 min with stirring followed by mixing for 2 min; then the foaming agent and perlite in the case of FA/perlite/cement composites was added, followed by mixing for 1 min. After 2 min of continuous mixing, cement was added. The mixture was cast into molds at room temperature (RT) and left for 5 h until the foaming was complete.

Table 2
Relevant physical properties of reinforcing fibers

Type fibers	Diameter (μm)	Length (mm)	Specific gravity (20°C)	Young's modulus (GPa)	Tensile strength (GPa)
Carbon	6.8	10	1.78	225.6	3.43
Glass	14.1	10	2.60	57.9	2.45
Polypropylene	88.0	13	0.91	3.5	0.69

For initial curing the composite slurry was placed in a constant temperature humidity chamber at 50°C and 80% RH for 5 h. After 1 day in room air, the cured composites were transferred to an autoclave for final curing at 180°C and 981 kPa for 5 h. After 5-h autoclave curing, the composites were dried in a ventilated ambient condition for about 1 week until the water content in the composites fell below 10%. Hot water curing was done in 70°C water for 1 day.

1.3. Testing of composites

Possible degradation of the reinforcing fibers in the hot alkali water environment was pretested by placing 10 g of the various fibers in 600-mL saturated solution of calcium hydroxide (0.138 g/100 mL water at 20°C), which was placed in an autoclave at 180°C and 981 kPa for 5 h. The autoclaved fibers were washed with cold water to remove residual calcium hydroxide. Then the fibers were inspected for mass loss and visually observed for physical degradation.

The tensile strengths were determined using 10-cm diameter × 20-cm long cylindrical specimens according to ASTM C 496. Flexural strengths were determined by using 7 × 7 × 20-cm specimens under three-point loading with 18-cm span according to the Japan Concrete Institute (JCI) method SF-4 at 0.5 mm/min loading rate. The load vs. deflection curves were recorded by an X-Y recorder.

The compressive strengths and specific gravity were determined by following KSF testing methods 2701 and 2459, respectively. The 90% dried 10 × 10 × 10-cm cubic composite specimens were placed on a 25-ton computer-controlled Shimadzu Universal Testing Machine under 98.1 kPa/s loading rate. The compressive modulus of elasticity (MOE) was determined following the method of ASTM C 469 using 10-cm diameter × 20-cm long cylindrical specimens attached with a 6.8-cm wire strain gage. The MOE was located at 40% of the ultimate stress and secant modulus of the strain. The flow test for FA/perlite/cement com-

Table 3
Proportion and composition of cement composites

CF/SF/cement				
SF/cement (wt%)	Water/cement (wt%)	SPL (wt%)		CF (vol %)
30	40	5		0, 1, 2, 3, 4, 5
FA/perlite/cement				
FA:cement by wt	Perlite content (vol% FA + cement)	Foaming agent (wt% FA + cement)	Water/ (FA + cement) (%)	Flow no.
40:60	50	0	39.0	176
		0.1	40.5	178
		0.3	41.4	182
		0.5	42.0	181

FA/SF/CS/cement						
Wt % proportion					Vol/% fibers	Wt/% water
SiO_2 powder	(FA + SF)*	Lime	Gypsum	Cement	(f/m)†	(w/m)†
62	—	17	3	18		
57	5	17	3	18	0, 0.5, 1.0	
52	10	17	3	18	1.5, 2.0	66
42	20	17	3	18		

* FA (fly ash) and SF (silica fume) in equal amounts.
† f is fibers; m consists of SiO_2 powder, FA, SF, lime, gypsum, and cement; w is water.
SPL, superplasticizer.

posites was carried out following Korea Standard Method L5105. Flexural strengths for CF/SF/cement composites were determined by using 4 × 4 × 16-cm rectangular bar specimens with 10-cm span placed on a 25-ton computer-controlled Shimadzu Universal Testing Machine.

2. Results and discussion

2.1. Fiber damage by autoclaving

The physical integrity of the fibers under the autoclaving condition of hot calcium hydroxide are given in Table 4. Carbon fibers were virtually inert under the condition, and glass fibers showed no changes in physical appearance, although they exhibited slight weight gain due to the reaction between SiO_2 and $Ca(OH)_2$ forming calcium silicate hydrates. On the other hand, PF showed clear degradation by the heat and alkali, indicating that it is unsuitable for reinforcing calcium silicate/cement matrices in ALC conditions. Although the weight change of PF is insignificant, the lumped form of PF suggests that the fibers were melted at the autoclaving temperature of 180°C and resolidified upon cooling. Thus, only CF and GF were used in further fabrication of composites.

Table 4
Percent residual weight and appearance of fibers after autoclaving in hot calcium hydroxide

Fibers	% wt	Fiber appearance
Carbon	103	No visible change
Glass	110	No visible change
Polypropylene	104	Fibers lumped together and transformed to granular

2.2. Bulk density of the composites

Density of the FA/SF/CS/cement composites are given in Table 5 for all different proportions of the ingredients. The density values increased by 3.8%, 8.4%, and 23.4% when FA + SF loadings were increased by 5wt%, 10wt%, and 20

Table 5
Density and compressive elastic modulus of fiber-reinforced fly ash/silica fume/calcium silicates/cement composites

FA + SF (wt%)	Fiber (vol%)	Density (g/cm^3)	Compressive elastic modulus (GPa)
5	0	0.51	1.97
	1.0	C 0.52	1.92
		G 0.53	1.98
	2.0	C 0.52	1.92
		G 0.53	1.93
10	0	0.54	2.14
	1.0	C 0.54	2.12
		G 0.55	2.18
	2.0	C 0.55	2.02
		G 0.55	2.08
20	0	0.60	2.35
	1.0	C 0.62	2.20
		G 0.62	2.38
	2.0	C 0.62	2.09
		G 0.65	2.14

C, carbon fibers; G, glass fibers.

wt%, respectively. The density values of 0.51 to 0.55 g/cm^3 meet the criteria for ALCs [21,34] except the ones with 20 wt% FA + SF.

When the autoclave curing temperatures were varied at 140°C, 160°C, 180°C, and 200°C keeping other parameters constant, the density of composites with no fibers were 0.53, 0.52, 0.50, and 0.50 g/cm^3, respectively. This means that the curing temperature has little effects on reducing the density of the matrix above 180°C. Comparing the density with respect to fiber type, GF tends to exhibit little higher values at a given volume loading. This must be attributed to the slightly higher density of GF itself and stronger interfacial bonding between the fibers and matrix. If one wants the density below 0.6 g/cm^3, which is the limit for ALCs, it must be kept at the FA+SF level below 20 wt%.

Fig. 1 shows the bulk density as a function of perlite loading with the hydroxy silicon ester foaming agent. The density decreased essentially linearly with increase in perlite and foaming agent loading regardless of curing conditions. However, 70°C hot water curing yielded lower density than the composites cured in autoclave. The statistical analysis for the effect of perlite and foaming agent on bulk density is shown to be significant even at the 1% significance level. It was revealed that foaming agent addition is more effective than perlite addition for reducing the density of lightweight FA/cement composites. If these composites are to substitute for ALC, the density of FA/cement composites must be below 1.5 g/cm^3 and the compressive strength at least 14.7 MPa [21,34]. In that case, the optimum perlite loading should be 150% to 200%, 80% to 100%, 50%, and 30% by volume with respect to FA+cement per foaming agent of 0 wt%, 0.1 wt%, 0.2 wt%, and 0.3 wt%, respectively.

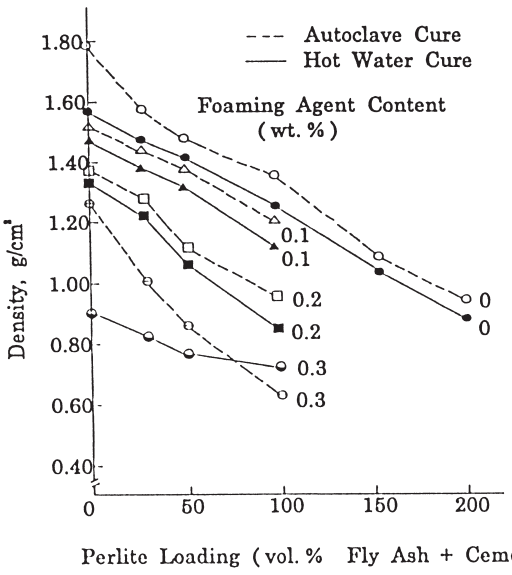

Fig. 1. Bulk density changes for FA+cement composites as a function of perlite and foaming agent loading and curing conditions.

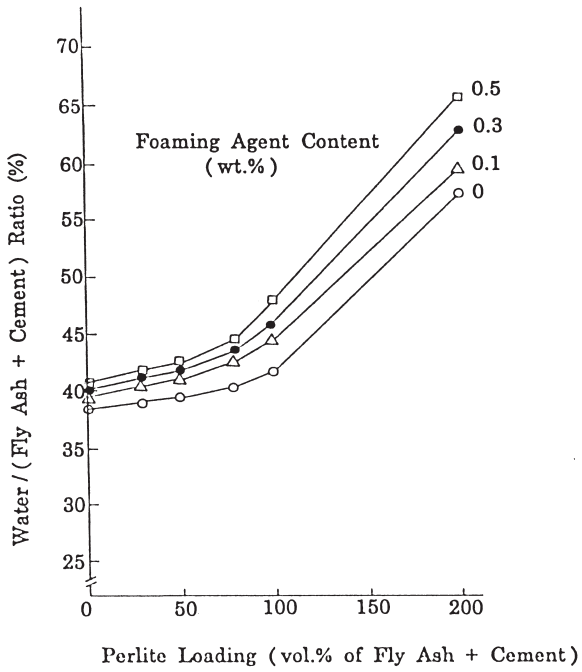

Fig. 2. Relationship between water to FA+cement ratio and perlite and foaming agent loading for a flow value of 180.

In Fig. 2 it is shown that perlite addition for lightweight composites requires more water in FA/cement/perlite composites, especially the vol% of perlite above 100. The organic foaming agent exhibited a tendency to lower the flow number for gaining lower density.

The statistical analysis for the effect of perlite and foaming agent content shows that the perlite and foaming agent addition is significant for increasing the ratio of water to FA+cement at the 1% significance level. The effect of perlite is shown to be larger than that of foaming agent for increasing the water to FA+cement ratio.

2.3. Compressive strength and compressive modulus of elasticity

The compressive strength and compressive elastic modulus of FA/SF/CS/cement composites increased, as shown in Fig. 3 and Table 5, respectively, with the increase in FA+SF content and fiber loading. The MOE, however, decreased somewhat with the increase in fiber loading. Carbon fibers are shown to be superior to glass fibers in increasing the compressive strength, due to the much superior Young's modulus as given in Table 2. The effectiveness of fiber reinforcement on the compressive strength is diminished for fiber loading above 1.5 wt% due to the difficulty in dispersing the fibers homogeneously in the matrix [1,2,6].

The effect of the silicon ester foaming agent on compressive strength of FA/perlite/cement composites is shown in Fig. 4. The strength decreased in linear fashion as the foaming agent and perlite loading increased. Autoclave curing exhibited higher strength throughout the foaming agent and perlite ranges. This coincides with the higher density of the

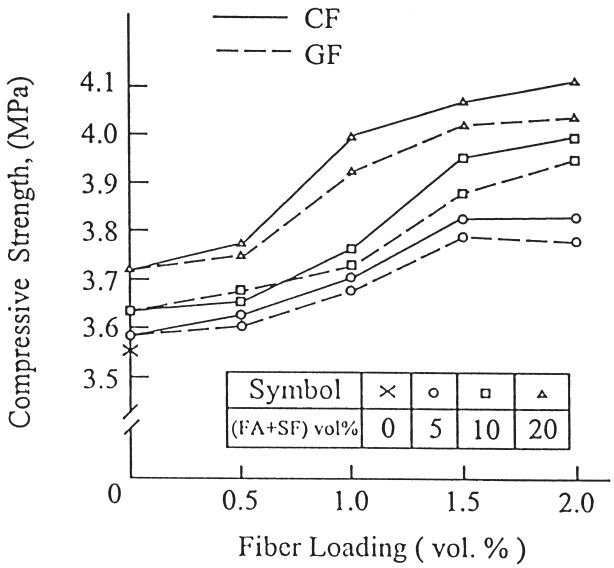

Fig. 3. Compressive strength of FA/SF/CS/cement composites as a function of fibers and FA+SF loading.

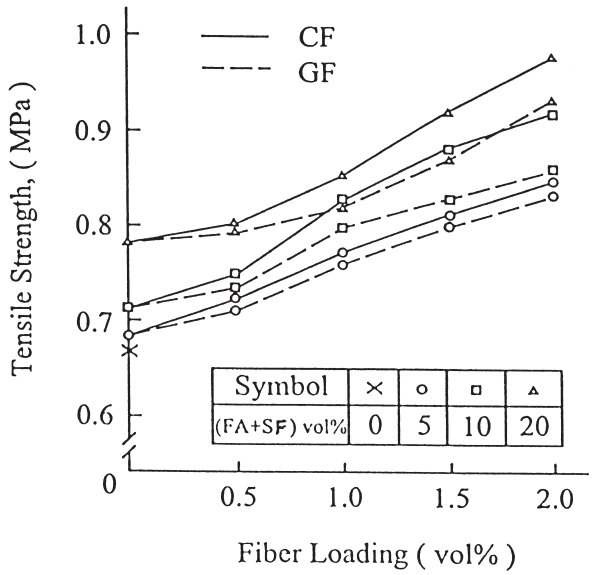

Fig. 5. Tensile strength of FA+SF/CS/cement composites as a function of fibers and FA+SF loading.

composites cured in autoclave than that in hot water. The factors affecting the compressive strength were statistically analyzed. All of the factors—perlite, foaming agent, and curing methods—were significant even at the 1% significance level. However, the effect of perlite addition in reducing compressive strength was more pronounced than the other two factors.

2.4. Tensile strength

The tensile strength values of FA/SF/CS/cement composites are plotted in Fig. 5. The trend is identical to the compressive behavior described in the previous section with Fig. 2. The tensile strength increased with FA+SF loading increase as well as the increase in fiber loading. Here again CF was shown to be more effective in yielding high tensile strength composites than GF for the same reasons applied to the compressive strengths, i.e., CF has higher tensile strength and higher aspect ratio.

Statistical analysis for the effect of the addition of FA+SF and fibers to cement shows that the fiber reinforcement is significant even at the 1% significance level, indi-

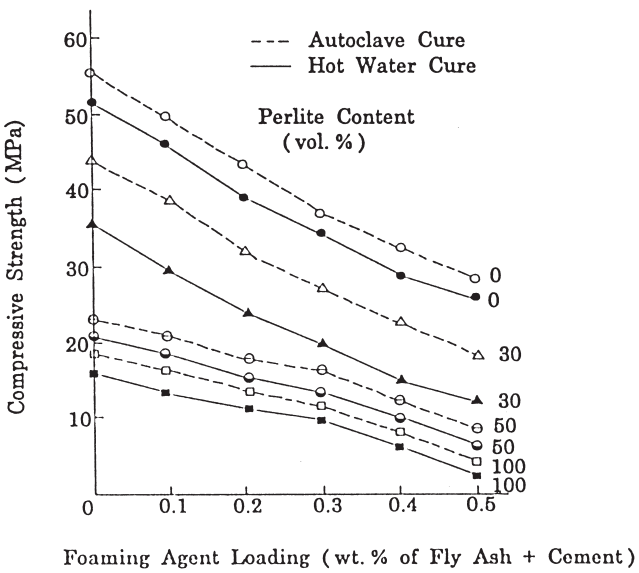

Fig. 4. Compressive strength of FA/cement composites as a function of perlite and foaming agent loading with different curing conditions.

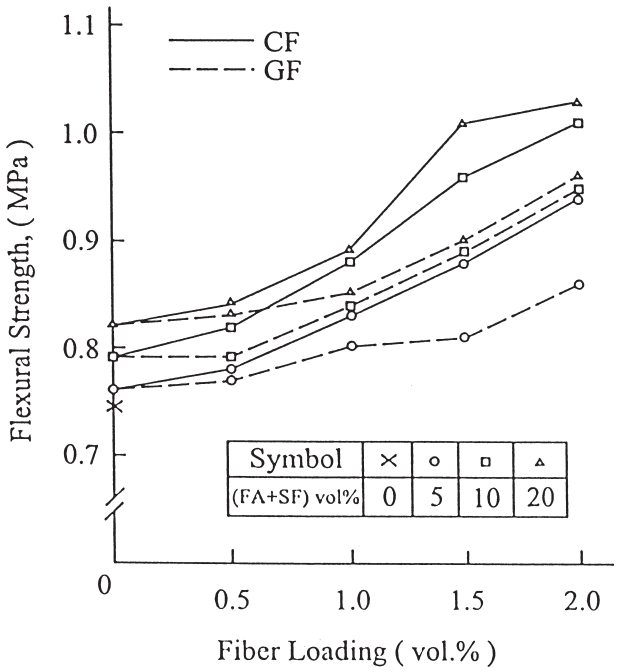

Fig. 6. Flexural strength of FA+SF/CS/cement composites as a function of fibers and FA+SF loading.

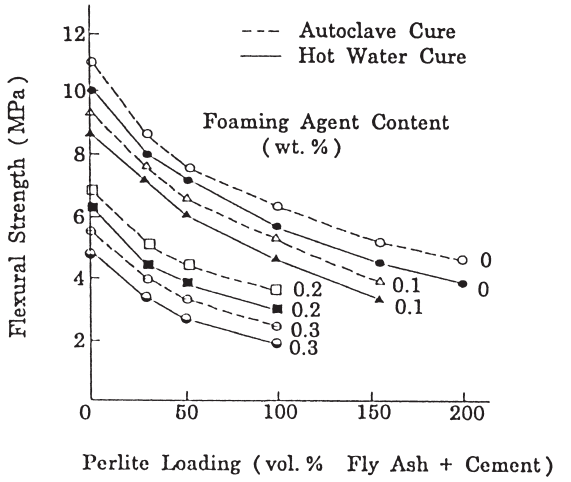

Fig. 7. Flexural strength of FA/cement composites as a function of perlite and foaming agent loading with different curing conditions.

cating that fibers are very effective for increasing tensile strength of the composites. It is also shown that FA+SF addition is effective at the 5% significance level for increasing tensile strength.

2.5. Flexural strength

The effectiveness of fiber reinforcement for flexural strength in cement-based composites is well known [3,35]. The flexural strengths of fiber-reinforced FA+SF/CS/cement composites are plotted in Fig. 6. As FA+SF loading increased from 5vol% to 20 vol%, the mean flexural strength increased from 1.95 to 11.6%. In Fig. 6 it appears that FA+SF beyond 10% loading is less effective for increasing the strength. This was more noticeable for glass fibers than carbon fibers and for increasing fiber loading. This trend is agreeable with the tensile and compressive strengths discussed in the previous sections.

The results of analysis of variance for the effect of the addition of fibers and FA+SF show that they are significant at the 1% and 5% significance levels, respectively. However, FA+SF addition, at the 5% significance level is shown to be less effective than fiber addition in increasing the flexural strength of these composites.

Fig. 7 shows the flexural strength as a function of perlite loading for different curing conditions and the amount of foaming agent added. The reduction in strength is similar to the reduction in bulk density shown in Fig. 1. However, autoclave curing maintained 10% to 30% higher strength than composites cured in hot water, which agrees with the density values. Statistical analysis of the effect revealed that all of the experimental conditions—perlite, foaming agent, and curing conditions—are significant at the 1% significance level. Hence, the effect is large in the descending order of foaming agent, perlite, and curing condition.

2.6. Load-deflection curves and deformation capacity

Flexural load vs. deflection curves for 10 wt% FA+SF in FA+SF/CS/cement composites are shown in Fig. 8. The integrated areas under the curves were taken as flexural toughness (FT) of the composites following JCI SF-4 method [36]. They generally showed sharp linear increases up to the maximum flexural load regardless of the kind of fibers. Beyond the maximum load, the curves varied asymmetrically depending on the fiber loading.

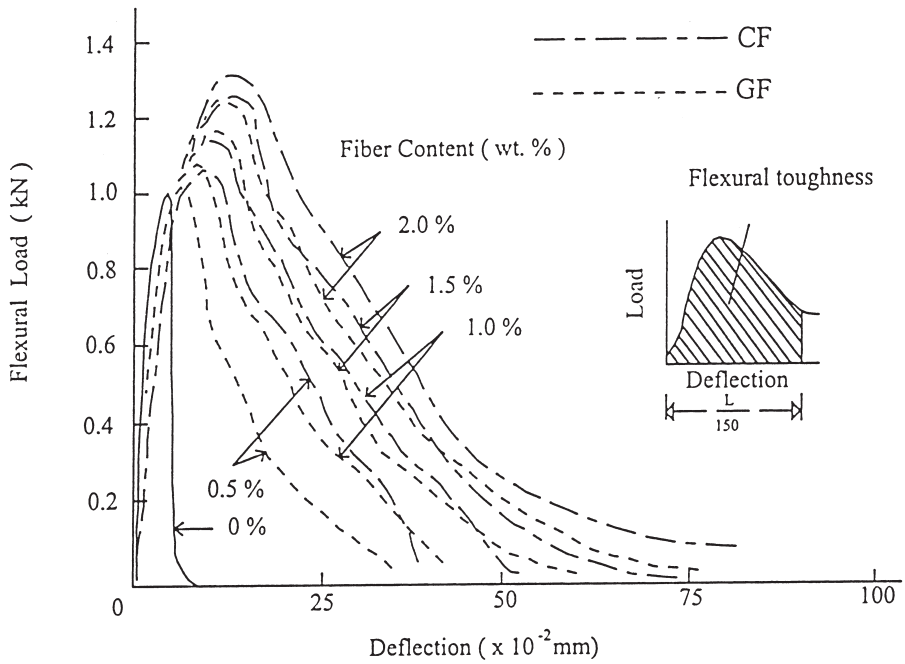

Fig. 8. Flexural load-deflection curves for composites of 10% (FA+SF)/52% SP/17% lime/3% gypsum/18% cement with different fiber loadings.

The FT increased as the fiber loading increased in all other loadings of FA+SF similar to one shown for 10% FA+SF in Fig. 8. Although the contribution of FA+SF to the FT is clear, the effect of fiber addition on toughening of the composites is unequivocal. At lower fiber loading, the effectiveness of CF is even more pronounced as compared with GF. That is, as the fiber loading changed from 0.5vol% to 2.0 vol%, the FT increased from 3.5× to 9.5× for GF and from 5.4× to 10.8× for CF. The toughening effect must be from the combination of strengthening the matrix and increasing the interfacial bonding between the fibers and matrix under the autoclaving conditions [1].

3. Conclusions

Normal procedures for preparing lightweight cementious composites comparable to ALCs incorporating industrial by-products and calcium silicates reinforced by carbon or glass fibers were developed. The composites exhibited very low density ~1.6 to 0.5 g/cm^3 with good mechanical properties to be used as lightweight construction materials.

It is clear that the compressive, tensile, and flexural strength and toughness increase are due to the addition of carbon and alkali-resistant glass fibers. Carbon fibers were superior to glass fibers, for a given weight loading, due to the higher volume loading of carbon fibers and the superior chemical and mechanical properties of the fibers themselves. At 2 vol% fiber loading, approximately 11× increase in the flexural toughness was exhibited by carbon fiber reinforcement in FA/CS/cement composites.

In the case of FA/perlite/cement composites, an increasing amount of water is needed to maintain the workability of the composite paste as the perlite and foaming agent loading is increased. Perlite and the organic foaming agent are effective in reducing the bulk density at the expense of reducing compressive and flexural strengths. Autoclave curing yields higher bulk density of the composites and thus higher strengths than does hot water curing. The effect of perlite addition is more pronounced than are the foaming agent and curing methods in lowering strengths, that is, foaming agent is more effective in lowering the density than perlite.

To substitute for ALC, the optimum amounts of perlite are 150% to 200%, 80% to 100%, 50%, and 30% by volume of FA+cement with corresponding foaming agent of 0, 0.1, 0.2, and 0.3 wt% of FA+cement, respectively.

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