

The effect of different mineral additions and synthetic fiber contents on properties of cement based composites

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Received 23 February 2005; accepted 3 February 2006

Available online 23 March 2006

Abstract

The influence of the matrix formulation and different amounts of synthetic fiber on physical and mechanical performance of asbestos free fiber cement was evaluated. Polyvinyl alcohol (PVA) fiber was tested as reinforcement in combination with mechanical and kraft cellulose pulps. Silica-fume, metakaolin and fly ash were used as pozzolanic additions in proportions up to ~14% by mass in combination with ordinary Portland cement and carbonatic filler. Bulk densities of composites varied from 1.5 to 1.7 g/cm³. Synthetic fiber contents higher than 2% by mass (from 4% to 5% by volume of the composite) were unable to promote any further improvement in the mechanical performance of the composites at the age of 28 days. Formulations with silica fume showed better strength performance for the composites after accelerated aging test. The toughness measurements of composites after exposition to soak and dry cycles also showed that silica fume seems to prevent cellulose fiber degradation.

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Keywords: Metakaolin; Flexural strength; Silica fume; Fly ash; Synthetic fiber; Cellulose pulp; Fiber cement

1. Introduction

In recent times, the term pozzolan has been extended to cover all siliceous/aluminous materials, which in finely divided form and in the presence of water, will react chemically with calcium hydroxide (CH) to form compounds with cementitious properties. This generalized definition covers waste products such as fly ash (FA), rice husk ash (RHA), silica fume (SF), and more recently, metakaolin, a man-made material formed by the controlled calcinations of kaolin clay [1]. Sabir et al. [2] observed that portlandite (CH) reacts with the added pozzolan resulting in additional calcium silicate hydrates (CSH). The CH liberated by the hydration of ordinary Portland cement (OPC) does not

make a significant contribution to the mechanical strength and can be harmful to concrete durability. Its elimination or reduction by reaction with the pozzolans can result in greatly enhanced durability and strength to the cement matrix.

In the calcination process (450–600 °C), kaolinite loses the OH lattice water, and it is transformed into metakaolinite, where the Si–O network remains largely intact and the Al–O network reorganizes itself [3]. While kaolinite is crystalline, metakaolinite presents a highly disordered structure and offers good properties as a mineral addition. Metakaolinite reacts particularly well with calcium hydroxide and forms hydrate compounds of Ca and Al silicates in the presence of water. The development of pozzolanic properties in fired clays depends mainly on the nature and abundance of clay minerals in the raw-material, calcination conditions and the fineness of the final product [4–9]. According to Moulin et al. [10], metakaolin is a

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pozzolanic material that is essentially an anhydrous poorly crystallized aluminosilicate produced by the calcination of kaolin, a naturally occurring clay basically containing kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) and, depending on the deposit, other minerals. The blend of metakaolin with ordinary Portland cement (OPC) can contribute to enhanced performance of mortars and concretes.

When mineral additions, such as fly ash, are added with cement, they react with CH to form additional calcium silicate hydrates in the hydrated cement matrix [1,11]. The additional CSH may increase the specific gravity of the matrix and refine the pore structure depending on the hydrated material that is actually formed (Tobermorite, e.g.). The addition of superfine mineral filler such as silica fume has also resulted in a denser material (i.e. with greater bulk density) that presents higher strength and is more durable.

Some mineral additions are finer than cement particles. They improve the particle packing of cement paste, in particular in the transition zone between aggregate and cement paste and fill the pores, thus reducing the permeability. The use of silica fume is based on two characteristics: (1) the effective pozzolanicity and (2) the (submicron) fineness of the material [3,4,12]. One of the basic principles of the materials technology is the necessity of dense packing to create a stronger matrix. The small size of the silica fume particles allows filling up the voids between the much larger cement and aggregate grains and contributes to a less porous composite [13]. There is also an effect of reduction of the size of the average pore which is significant for the mechanical strength of the cement matrix [1].

The use of cellulose fibers in cement composites is important because of the retention of cement particles in industrial processes and some reinforcement effect in the early ages [14,15]. However, natural fibers containing higher amounts of other constituents (as hemicellulose and lignin) are very sensitive to the alkaline environment and are rapidly degraded, causing reduction in the composite durability [16]. Air cured fiber–cements reinforced exclusively with chemical wood pulps also show some reduction in the mechanical performance under continued exposition to natural environment [17] although the alkali degradable materials have been largely removed during the pulping process. It can be partially understood by the embrittlement that cellulose fiber suffers due to its mineralization inside the cement matrix [18]. The problem of reinforcement can be solved by the use of synthetic fibers, which are less sensitive to alkali attack and possess good mechanical properties, resulting in composites with better performance in the long-term [19]. The degradation process of the composite can, therefore, be directly related to the matrix and fiber type, the composite porosity and the aging mechanism.

The objective of this work was to evaluate the effect of different types of pozzolans and carbonate addition on the physical and mechanical properties of cementitious composites containing cellulose and synthetic fibers.

2. Experimental work

2.1. Composite production

Hardened specimens of cement-based composites were produced with dimensions of 40×160 mm, ~ 5 mm of thickness and reinforced with polyvinyl alcohol (PVA) fibers, virgin and waste cellulose pulps. The matrix was composed by ordinary Portland cement (OPC) CPII F and CPI S types (specified by Brazilian Standards NBR 11578 and 5732, respectively) [20,21]. The chemical composition of these cements is given in Table 1. The specific surface area (BET technique) of the CPII F cement is $598 \text{ m}^2/\text{kg}$ and the value for the CPI S cement is $591 \text{ m}^2/\text{kg}$ according to information given by the producers. It reflects a tendency of the Brazilian industry to produce OPC with increased specific surface area [22] that helps to compensate the amount (up to 10% by mass) of carbonate filler added in the formulation of the used cements. Carbonate filler with specific surface area of $451 \text{ m}^2/\text{kg}$ was used as an aggregate. Three different types of pozzolanic materials were used in this study as shown in Table 2. PVA fibers were chosen as reinforcement. The synthetic fibers were approximately 6 mm long, $14 \mu\text{m}$ thick and with modulus of elasticity (MOE) equal to 32.5 GPa as informed by the supplier. A blend of cellulose pulps of long and short fibers was employed. Average length of the short fiber (newsprint mechanical pulp) was 0.47 mm, with fines content (fibers with length under 0.07 mm) of 52%. Long fiber unbleached kraft pulp, was refined up to 70° Shopper Riegler (°SR) and presented the average length of 1.24 mm (fines content of 25%). The Shopper Riegler index is an empirical estimation of the drainability of the cellulose pulp. The high level of the cellulose pulp refinement helps in the efficiency of the fine particles retention by the fiber network during the drainage process. This capability is of particular interest for retention of the matrix constituents during the production of composite slates in industrial scale by the Hatschek (or wet) process [23]. Table 3 shows all formulations used

Table 1
Oxide composition of the ordinary Portland CPIS/CPIIF cements

Compound	Content (% by mass)	
	CPIS ^a	CPIIF ^b
Ignition loss	2.98	3.69
SiO ₂	18.36	19.53
CaO	58.20	60.49
Al ₂ O ₃	4.42	4.71
Fe ₂ O ₃	2.82	3.07
MgO	5.99	4.68
Na ₂ O	0.15	0.10
K ₂ O	1.03	0.96
SO ₃	3.41	2.60
CO ₂	1.93	–
Insoluble residue	0.83	–

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Table 2
Physical and chemical characteristics of the pozzolanic materials

Material	Pozzolanic activity ^a (mg/g)	BET (m ² /kg)	Chemical composition (%)			
			SiO ₂	Al ₂ O ₃	CaO	Ignition loss
Metakaolin	767	20,960	51.2	35.3	2.62	4.57
Silica fume	814	22,500	91.2	0.22	0.22	1.30
Fly ash	197	320	59.6	26.5	1.66	–

Source: Laboratory of Microstructure/PCC—Escola Politécnica—USP, Brazil.

^a Based on Chapelle Method.

Table 3
Formulations of the composites (% by mass of solid raw-materials)

Raw-materials	Formulations								
	MFA1	MFA2	MFA3	MCF1	MCF2	MCF3	MM1	MM2	MM3
Cement CPIS	62.14	61.48	60.83	–	–	–	–	–	–
Cement CPIIF	–	–	–	62.14	61.48	60.83	76.00	75.20	74.40
Metakaolin	–	–	–	–	–	–	5.14	5.08	5.03
Fly ash	13.86	13.72	13.57	–	–	–	–	–	–
Silica fume	5.14	5.08	5.03	5.14	5.08	5.03	–	–	–
Carbonate filler	13.86	13.72	13.57	27.72	27.44	27.14	13.86	13.72	13.57
PVA fiber	1.00	2.00	3.00	1.00	2.00	3.00	1.00	2.00	3.00
Brazilian softwood pulp	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20
Recycled newspaper	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80

and their respective percentage by mass of solid raw-materials. The specimens were produced in the laboratory by slurring the raw-material in water solution (20% of solids) followed by a vacuum drainage of the excess of water and pressing at 3.2 MPa during 5 min. This procedure of preparation is described in detail elsewhere [24]. The final proportion between water/solids in the end of the slurry dewatering and pressing processes can only be estimated and it is expected to be around 0.5 for the amount of fibers in use (Table 3) as discussed by Caldas e Silva [25]. Consequently although the water/cement ratio could not be precisely determined it is expected to be lower for MM mixtures due to the higher proportion of OPC in comparison to the others. Twenty specimens of each formulation were submitted to wet curing for seven days followed by air curing until the age of 28 days when the mechanical and physical performances were assessed. Twenty specimens for each formulation were submitted to the accelerated aging test (soak and dry cycles). This test consists of submerging the specimens into cold tap water for 18 h. After this, they were put in an oven at 60 °C during 6 h, to complete the cycle of 24 h. The aging test is composed by 50 cycles and it is based on the methodology of the European Standards EN-494 section 7.3.5 [26].

2.2. Composite characterization

Bulk density (BD) and permeable void volume (PVV) were determined following the procedures specified in the Brazilian Standard NBR-9778 [27]. The mechanical behavior was based on the Rilem recommendations 49 TFR [28].

It was assessed by a four-point bending configuration. The loading apparatus consisted of two supports a distance $L = 135$ mm apart and two loading points spaced to provide third point loading ($L/3$). A deflection rate of 1.5 mm/min was used for all tests in an Emic DL30000 universal testing machine equipped with load cell of 1 kN [24]. The mechanical properties evaluated at 28 days and after 50 cycles were modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE) and toughness. The degradation of the composites was measured using the Eq. (1) to calculate the R -factor

$$R = \frac{L_2}{L_1} \quad (1)$$

where L_1 = average strength of non-aged (28 days) specimens (+) 0.58 (×) the standard deviation of results; L_2 = average strength of aged specimens (–) 0.58 (×) the standard deviation of results; R = parameter of degradation measurement of specimens after 50 cycles.

3. Results and discussion

Figs. 1–4 show the results obtained from four-point bending tests. Table 4 summarizes physical properties with average values and corresponding standard deviation of all series under analysis.

Stress–strain curves concerning the composites with fly ash addition are shown in Fig. 1. After 28 days (Fig. 1(a)), the flexural strength presented only small alterations related to the different fiber content. Results obtained after 50 cycles of accelerated aging (Fig. 1(b))

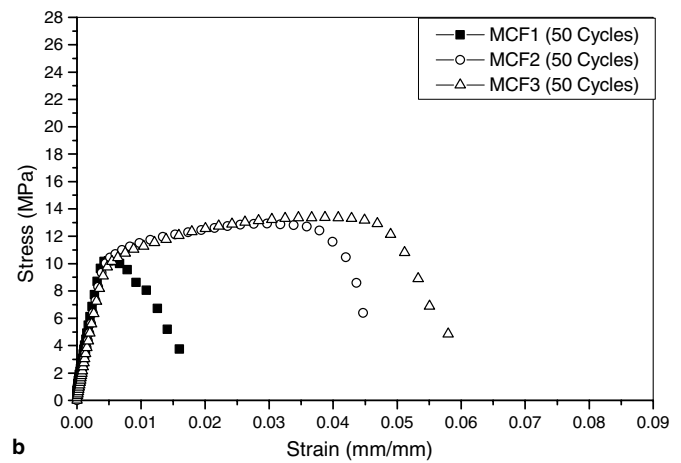
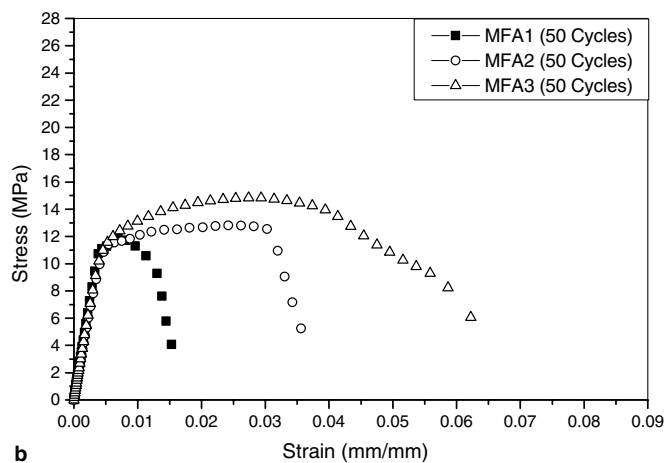
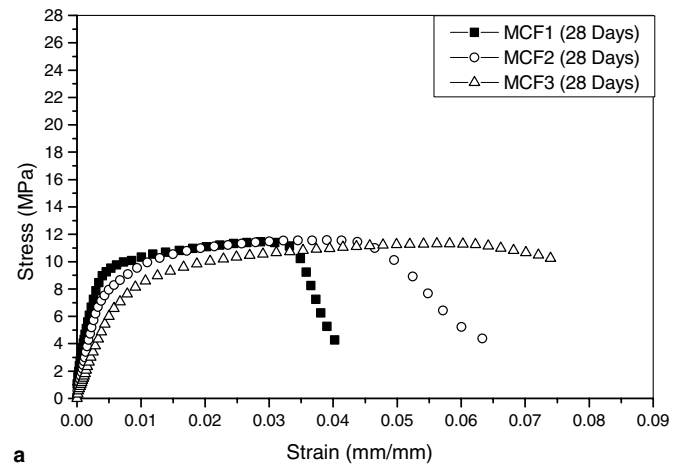
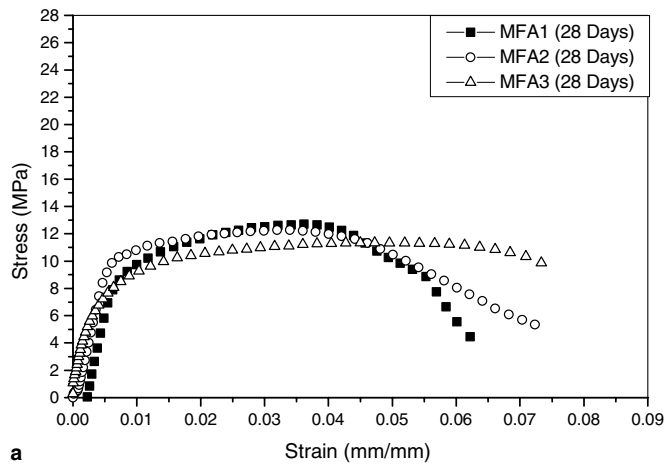


Fig. 1. Matrix with fly ash: stress–strain curves of the composite reinforced with synthetic and cellulose fibers after (a) 28 days of cure and (b) 50 cycles of accelerated aging test.

Fig. 2. Matrix with carbonate filler: stress–strain curves of the composite reinforced with synthetic and cellulose fibers after (a) 28 days of cure and (b) 50 cycles of accelerated aging test.

showed that the formulation MFA3 presented the best mechanical behavior, due to the higher concentration by mass of the PVA fibers. The cellulose fibers are particularly sensitive to alkali attack [29], and this can be better identified in the series with lower content of synthetic fiber. The peeling and the hydrolytic reactions [30] are responsible for the loss of polysaccharides and reduction of the cellulose chain length in alkaline environment (pH \sim 12–13).

On the other hand the accelerated aging test seems to contribute to the improvement of the bond of the fibers as a whole. Since the only relevant change in any of the mixes is the PVA fiber content and there is no significant decrease between the ultimate strength of aged and un-aged composites (Fig. 4(a)) then it is unlikely to suppose that has been much change in the cellulose fibers. However, the strain to failure shown in Fig. 1(b) is greatly reduced after aging particularly for the mix with 1% PVA and this is also reflected in the toughness values. This supports the assumption of brittle failure of the fibers after aging due

to an increase in bond strength of the fibers to the matrix from additional curing.

Additionally the fly ash particles presented small surface area and their pozzolanic activity was considerably low. As a result, the MOR values of the composites were positively affected by the soak and dry cycling specially for the mixture MFA3. The physical parameters showed small changes after accelerated aging test, according to Table 4. The higher amount of fibers led to the reduction of the bulk density of the composite. This reduction can be associated with two factors: (1) change in the phase composition due to modifications in the concentration by mass of the synthetic fibers and (2) reduction of the packing factor of the particles. The encountered results for permeable void volume and bulk density seem to be not consistent with each other. The PVA fibers with high aspect ratio (\sim 430) can be difficult to disperse, and responsible for the inclusion of non-permeable pore spaces in the composite in a variable way, what can interfere in the test results.

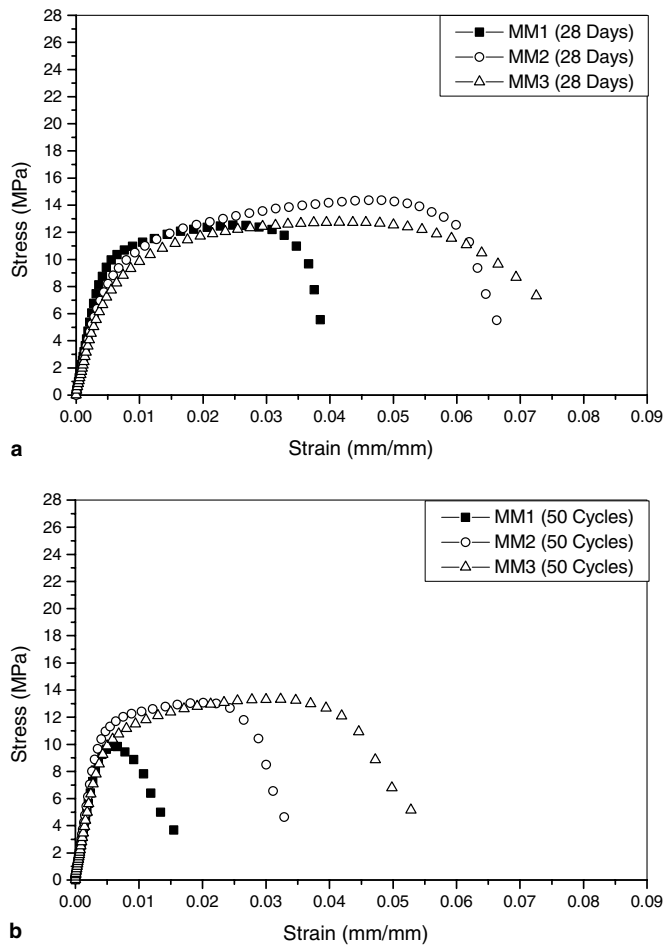


Fig. 3. Matrix with metakaolin: stress–strain curves of the composite reinforced with synthetic and cellulose fibers after (a) 28 days of cure and (b) 50 cycles of accelerated aging test.

The formulation with higher content of carbonate filler and 2% by mass of PVA fiber (MCF2) presented better performance of MOR and LOP after 28 days of curing, compared to the formulations with 1% and 3% fiber, respectively (Figs. 2(a) and 4(a) and (b)). This mechanical behavior can be associated with good array of the fibers and solid particles into the composite. The explanation is complemented by the fact that up to 2% of synthetic fiber can be wetted by the matrix and good bonds are made to the fiber. But beyond this limit there is a decrease in fiber bond probably because amount of matrix is insufficient to fully wet and bond the fiber. This improvement in MOR and LOP occurs despite the expected reduction in the strength of the matrix due to increased porosity because of the presence of the fiber [14].

After the accelerated aging test, the formulation MCF1 had a statistically significant reduction of the toughness, which can be associated with the degradation of the cellulose fibers after 50 soak and dry cycles as depicted in Fig. 2(b). According to Table 4, the formulation MCF1 had a higher bulk density after 28 days of curing and accel-

erated aging test, due to the lower concentration of fibers and higher percentage of granulated particles.

Concerning the formulations with metakaolin, the mixes with 2% and 3% of PVA showed similar toughness values after 28 days (Fig. 3(a)) with clear improvement in relation to the mix with 1% PVA. The explanation for that could be the same as the one for the MCF series. It is also shown that the formulation with 3% of PVA fiber (MM3) presented better mechanical performance than MM2 and MM1 after the accelerated aging test (Fig. 3(b)), due to the presence of higher concentration of PVA fibers, which are not susceptible to the alkali attack. Metakaolin particles are supposed to react with the calcium hydroxide liberated during the hydration process reducing the alkalinity of the cement paste. Such an assumption would also be expected to silica fume (and to a lesser extent the fly ash) and is based in the pozzolanic activity of these materials as presented in Table 2. However, no significant contribution to the mechanical performance of the composites was observed. As expected, the increase of the PVA concentration induced the reduction of bulk density of the composites (Table 4).

Comparisons of mechanical properties can only be made between series of composites with the same mineral addition due to the differences in composition as shown in Table 3. The results of MOR, LOP and toughness obtained for all formulations are summarized in Fig. 4. The values of MOR for all formulations (MFA, MCF and MM) containing 1% of synthetic fibers at 28 days were higher than those after 50 cycles of aging. For the composites with 2% and 3% by mass of PVA, MOR is higher after 50 cycles, with exception of MM2 (Fig. 4(a)). The LOP showed a consistent improvement for all mixtures after the accelerated aging test and such a behavior indicates the improvement of the composite prior to generalized cracking due to the better adhesion of fibers in the matrix. The presence of greater quantities of PVA fibers is more relevant for toughness results (Fig. 4(c)). While for MOR the differences between 28 days and 50 cycles of accelerated aging are up to 25%, for the toughness these differences are very significant mainly for composites with 1% and 2% of PVA, with reductions exceeding 75%. This behavior could be explained by the improvement of fiber adhesion due to the densification of the interfacial transition zone after the accelerated aging tests [31]. This general behavior can be visualized in the micrographs of fracture surface of composites shown in Fig. 5. The incidence of pulled out fibers seems to reduce after the exposition to the 50 soak and dry cycles.

The results of R -value calculated according to standard EN-494 are presented in Fig. 6. The standard establishes that the R -factor should be equal or above 0.7. The results for all matrices are in agreement with the EN-494 and several of them reached R -values for MOR in excess of the unity, which is an indication of the material densification instead of the decomposition of reinforcing fiber. These results can also be a signal that the curing process

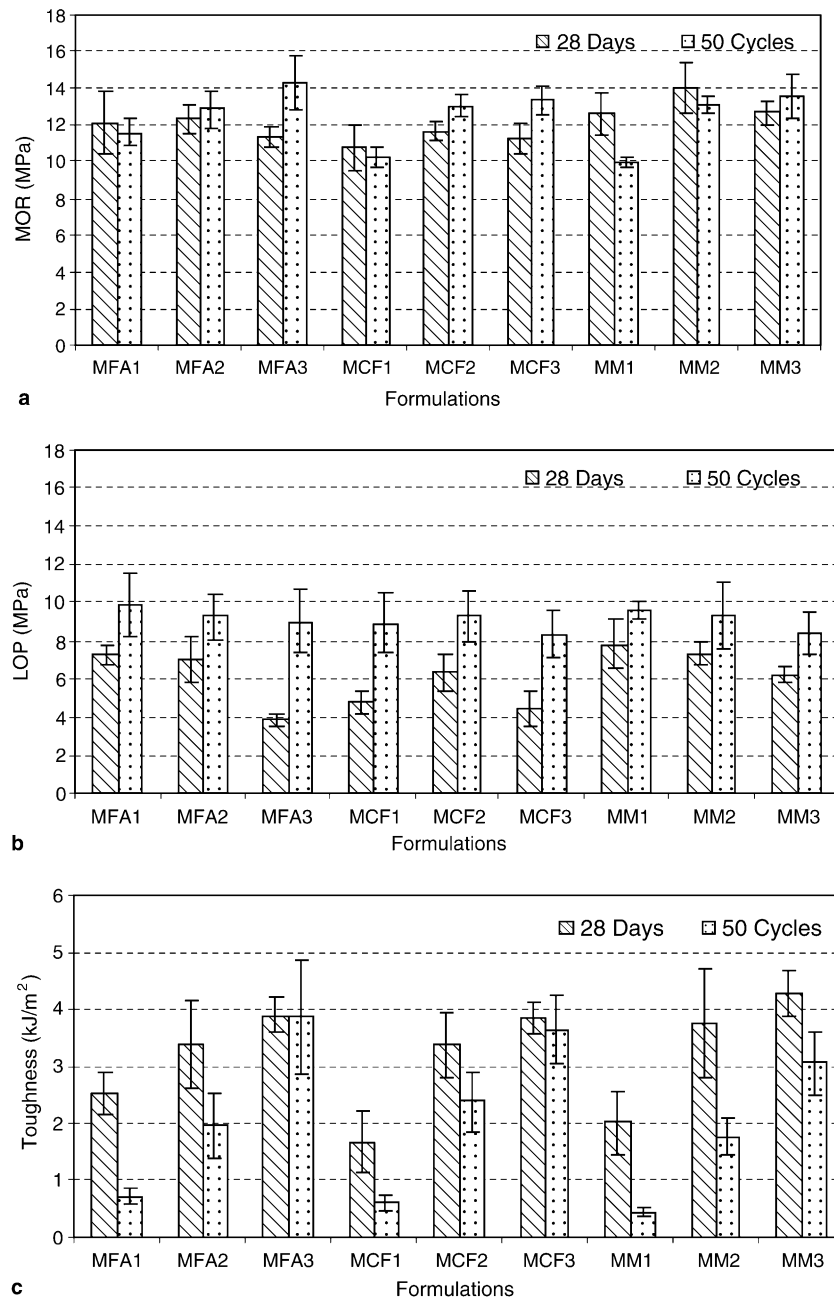


Fig. 4. Comparison of average values of (a) MOR, (b) LOP and (c) toughness. Vertical bars indicate single standard deviation.

should be improved since the wet phase of the aging tests is helping with the continuation of the hydration process of the cementitious matrix. Such a performance is in agreement with similar studies on durability of cementitious pastes reinforced by a combination of cellulose and synthetic fibers [32].

The present study confirms the better performance of mechanical strength of aged composites with hybrid reinforcement in comparison to fiber–cement with vegetable fiber as the sole reinforcement [33]. The same observation is valid for MOE with average values in excess of 13 GPa, what represents a considerable increase in relation

to MOE values under 5 GPa encountered by Savastano et al. [33] for composites reinforced with natural fiber after 13 months of age.

4. Conclusions

Physical properties

- As general tendencies, the lowest permeable void volume was related to the formulations with metakaolin additions (MM) both at 28 days of age and after the accelerated aging test. The MM formulations contained

Table 4
Physical properties^a of composites with mineral additions

Formulation	Bulk density (g/cm ³)		Permeable void volume (% by volume)	
	28 days	50 cycles	28 days	50 cycles
MFA1	1.53 ± 0.03	1.55 ± 0.02	32.3 ± 1.6	31.9 ± 0.5
MFA2	1.52 ± 0.04	1.53 ± 0.02	32.1 ± 1.2	30.4 ± 1.1
MFA3	1.49 ± 0.03	1.48 ± 0.04	30.7 ± 0.7	30.7 ± 0.7
MCF1	1.67 ± 0.02	1.61 ± 0.03	30.7 ± 0.8	32.5 ± 1.8
MCF2	1.57 ± 0.04	1.54 ± 0.07	32.8 ± 0.9	32.5 ± 2.5
MCF3	1.50 ± 0.03	1.56 ± 0.03	31.7 ± 0.9	31.6 ± 0.9
MM1	1.66 ± 0.03	1.70 ± 0.03	30.9 ± 0.6	28.2 ± 1.4
MM2	1.64 ± 0.02	1.69 ± 0.01	29.2 ± 0.3	29.0 ± 0.8
MM3	1.55 ± 0.05	1.58 ± 0.03	29.5 ± 0.8	30.9 ± 1.0

^a Single standard deviation of sample means indicated.

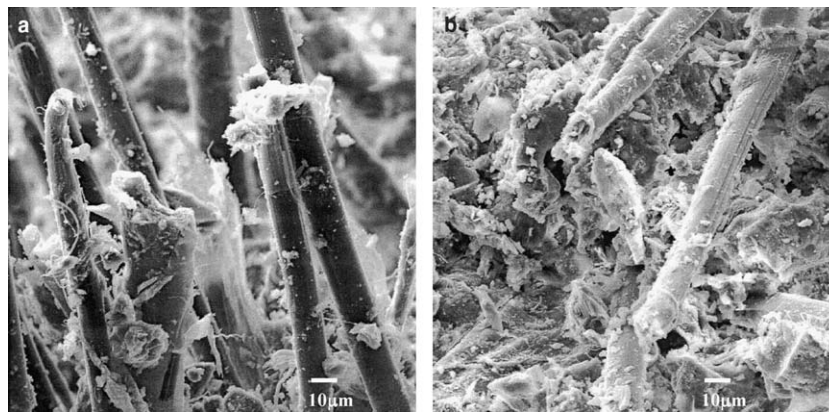


Fig. 5. Pull-out of PVA and cellulose fibers: (a) 28 days and (b) after 50 cycles.

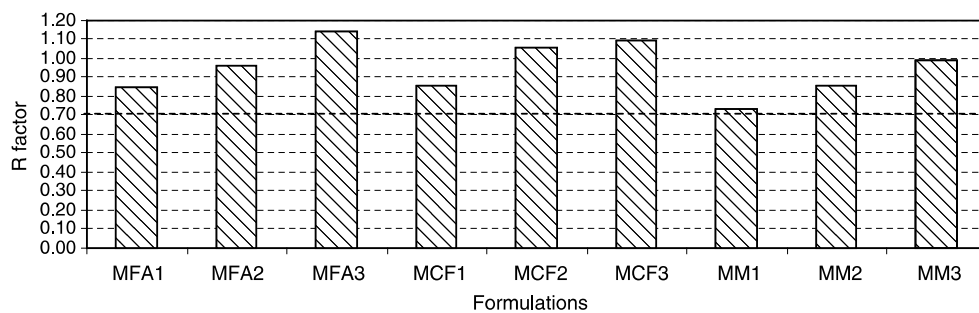


Fig. 6. *R*-factor for MOR related to soak and dry test.

the highest percentages by mass of OPC. On the other hand, the highest values of permeable void volume were presented by the MCF series, which contain the highest amounts of carbonate filler.

Mechanical performance

- The reduction in the strain to failure and toughness after 50 soak and dry cycles was more abrupt to the formulations containing lower concentration by mass of the PVA fibers. This behavior can be understood by the degradation of cellulose fiber and its more significant effect

in the mechanical performance of the composite with lower amount of synthetic fiber. The observed reduction of strain to failure and toughness values can also be attributed to the densification of fiber-matrix transition zone and consequently to the improved bond of the fibers.

- Several series showed increased MOR after the aging test and with $R > 1$ in some cases. The values of LOP also showed a consistent increase after the soak and dry cycles. It could be a consequence of densification and continued hydration of the cement phase of the composite caused by the soak and dry cycles. These

results attest the necessity of improved conditions of curing for formulations containing pozzolanic additions.

- The categories of formulations (MFA, MCF and MM) with 2–3% by mass of synthetic fiber presented better mechanical strength comparing results after and before aging tests, all of them with *R*-values higher than 0.85. The formulation MCF with 2% of PVA fiber can be considered one of the most promising due to the favorable relation between the mechanical performance and the advantageous proportion of raw-materials.

New studies should be carried out to evaluate the performance of cement-based matrices with different types of cellulose and synthetic fibers as alternative formulations to asbestos fiber–cement. It is of particular interest to attend construction markets of developing countries mainly focused on the use of corrugated sheeting production by Hatschek process.

Acknowledgements

The research is part of a collaborative work of the University of São Paulo and the fiber–cement industries Permatex Ltda. and Imbralit Ltda., Brazil. The authors express their gratitude to the State of São Paulo Research Foundation (Fapesp), the Research and Projects Financing (Finep), the National Council for Scientific and Technological Development (CNPq), and the Co-ordination for the Improvement of Higher Education Personnel (Capes) for their support, and to Mr. Gustavo H.D. Tonoli, Mr. Leandro Cunha and Mr. Zaqueu Dias de Freitas for their skilful assistance at the Laboratory of Rural Construction of University of São Paulo, Brazil. The authors would also like to thank Dr. Marie-Ange Arsène from Université des Antilles et de la Guyane for helping with MEV studies. They are also in debt with the external referees of the peer-review system of Cement and Concrete Composites that anonymously contributed to the improvement of the discussion of the proposed manuscript.

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