

Study of Hygral Behavior of NON-asbestos Fiber Cement Made by Similar Hatschek Process

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Fiber cement, similarly to all cementitious materials, undergoes dimensional and volumetric changes when it is exposed to dry and wet environments. When exposed to natural weathering, cement-based materials absorb and may also release water to their surroundings via a very complex inherent pore structure. In this work initialized a few years ago, some properties of asbestos-free fiber cement composites were studied, such as density, porosity and loss or absorption of water. For the understanding of the behavior of this material, modifiers were employed in the cement matrix such as styrene-acrylic, styrene-butadiene and vinyl acetate-versatic vinylester copolymers to verify their influence on the variables described above. All composites were analyzed by mercury intrusion porosimetry (MIP). For monitoring the expansion/shrinkage movements, the specimens were exposed inside a controlled environmental chamber with constant temperature and relative humidity (RH) during the test time. The results showed no interference on hygral behavior because particular casting process of these composites but, as showed by literature, increase of toughness. Also, it was possible to verify that moisture movement within the asbestos-free fiber cement can be very complex and the variables density and porosity (pore structure) play a very important role in the expansion/shrinkage properties of these composites.

Keywords: *copolymers, fiber cement, moisture movement, expansion/shrinkage*

1. Introduction

Cement-based composites exposed to natural weathering absorb water and may also release water to the surroundings via a very complex inherent pore structure. The extent of pore water exchange and the speed at which it occurs depend on numerous factors¹, for example, the type of application (roofing or steel frame system), the shape of the product, the temperature of the surrounding environment, the relative humidity, the weather conditions, the porosity and pore structure of the material, the orientation of structure (anisotropy), the type of fiber (cellulose or synthetic), the type of coating or impregnation applied and the age of material.

After fiber cement undergoes expansion or shrinkage it may produce cracking and also to be aesthetically displeasing and eventually can lead to structural unsoundness and poor durability in a component. In Brazil, frequently failures have been observed in joints of asbestos-free fiber cement sheets because the excessive moisture movements of these composites.

Various forms of shrinkage are associated with cementitious materials and since drying shrinkage is among the most troublesome, it is a useful tool for the performance evaluation in the development of improved materials.

It is evident from the factors mentioned above that it would be an extremely difficult task to describe the exchange of water in the pores of cement-based fiber composite during exposure to natural weathering. In fact not much has been reported in the literature regarding the moisture movement of asbestos-free fiber cement.

In general, it is observed that in concrete the presence of large volume fractions of rigid aggregates restrains the amount of moisture-induced movements. In fiber cement composites the volume concentration of fibers is very low compared to the aggregate volume and the matrix tends to be slightly richer in binder in comparison with normal concrete.

Although the transportation of pore water in fiber cement composites is complex, it is important to describe in general terms the behavior of the material under certain climatic conditions as the exchange of water in the pores results in dimensional changes (expansion or shrinkage) within the product. It is the reason for the study of the effect of water movement within the composites.

This paper shows the influence of density, porosity (pore structure) and sorption of water in the moisture movements of asbestos-free fiber cement where exchanges in these variables were made by the use of different copolymers.

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2. Literature Review

2.1. Asbestos-free fiber cement composites

Over the last decades many research studies related to the substitution of asbestos by other raw materials have been published. In this way, natural fibers from different softwood and hardwood species and synthetic fibers such as polypropylene (PP) and polyvinyl alcohol (PVA) have been an important group of studied fibers.

Natural fibers are frequently characterized by the low cost and poor durability because of vulnerability to chemical decomposition in the alkaline environment of cement. Regardless, these fibers are very important in countries with agricultural based economies².

Synthetic (polymeric) fibers have become more attractive in recent years for reinforcement of cementitious materials. In this way, PP and PVA have been studied extensively, with fibers formulated and produced specifically for reinforcement of cement-based composites. However, polymeric fibers present low modulus of elasticity compared to asbestos. To increase the strength of the composites, the fibers must have a modulus of elasticity greater than that of matrix. For cementitious materials, where the modulus of elasticity ranges from approximately 15 to 30 GPa, this condition is difficult to meet with synthetic fibers. Moreover, fiber-matrix interface may constitute a problem to the engineering of these composites³.

Asbestos is a natural occurring fibrous silicate very compatible with cement. Its chemical structure and its fiber size make composites of higher strength and lower moisture movements than asbestos-free cement-based composites.

This situation confirms the importance of understanding the hygral behavior of these composites, one of the most important variables for asbestos replacement, principally in applications such as corrugated sheets for roofing and flat sheets for dry-wall buildings.

2.2. Polymer addition

The concept of polymer modification is well known and the first patent was obtained in 1923. This patent refers to the use of cement as filler in polymer matrix. In 1924, the first patent was published using the concept of polymer modification for cement mortar and concrete⁴⁻⁷.

As a principle of polymer modification, it is very important that both Portland cement hydration and polymer film formation proceed well in order to produce a monolithic matrix phase with a network structure in which the cement hydrate phase and polymer phase interpenetrate. The modification with redispersible polymer powders is similar to that of latex formation except that it also involves the redispersion of the polymer powders⁸⁻¹⁵. In these cases, the modification is governed by both Portland cement hydration and polymer film formation processes in their binder phase¹⁶⁻²². The cement hydration process generally precedes the polymer film formation by coalescence of polymer particle.

According to the theory, during the hydration of Portland cement the hydrophilic part of the polymer is oriented towards the water phase whereas the hydrophobic part heads towards the air phase (pores and capillaries not filled

with water). Upon drying, the water is taken away and the hydrophobic particles coalesce together and form the film.

Drastic changes in the matrix properties such as density and porosity can be achieved by combining the matrix with polymers and it can also reduce the shrinkage of the composites³. It is the reason by which in this work the polymer addition is showed as one alternative to the studies of moisture movements of non-asbestos fiber cement. Due to their hygroscopic nature, fiber cement composites are sensitive to moisture changes in the material and in the ambient environment. Several studies have been conducted to assess the performance of pulp fiber-cement composites under different moisture states, particularly in the wet and oven-dry states.

It was possible to verify in the literature some works about polymer-modified cement matrix reinforced by E-glass and neither about other kind of fiber. As related²³, the GRFC (Glass Fiber Reinforced Cement) was developed by Bijen and Jacobs using a special acrylic polymer (Forton) which was developed for that purpose. In the production of this composite, the tiny polymer latex particles infiltrate and fill the spaces between the filaments in the strand and eventually coalesce into a film. Thus they can provide a protective effect, both to reduce the chemical attack, and to reduce the extent of the microstructural mechanism, by eliminating the growth of dense and rigid hydration around the filaments. Also, some authors describe the formation of the film in the matrix provide an additional sealing effect to slow down moisture movement, and consequently the expansion/shrinkage of fiber cementitious composites²⁴⁻²⁷.

3. Experimental

3.1. Material

The materials employed for composite preparations were Portland Slag Cement according to EN 197-1 – European Standards²⁸ - type (CEM II /B-S), cellulose pulp (solombola, Russian specie), Chinese PVA fibers (length: 6 mm; diameter: 20 μ m), carbonate powder as inert filler, silica fume and styrene-acrylic, styrene-butadiene and Va/Veova copolymers. Some characteristics of these materials are shown in Tables 1-6.

Table 1. Chemical analysis of Portland cement.

Chemical analysis of ordinary Portland cement (% by mass)		
Element	Average of 4 analyses	Standard deviation
SiO ₂	19.52	0.10
Al ₂ O ₃	5.03	0.05
Fe ₂ O ₃	3.02	0.04
CaO	64.76	0.10
MgO	0.62	0.03
SO ₃	2.85	0.03
CO ₂	2.25	0.28
K ₂ O	0.70	0.01
Loss on ignition	2.86	0.16
Residue	0.27	0.03

Table 2. Physical characteristics of styrene-acrylic copolymer dispersion.

Physical analyses	
Solids content (% by mass)	50 ± 1*
Viscosity (MPa.s)	2,000-5,000
Glass Transition (°C)	~ -8
Apparent density (g/cm ³)	1.0 ± 0.1*
Particle size (µm)	80 ± 10*

*Standard deviation.

Table 3. Physical characteristics of Va/Veova copolymer powder.

Physical analyses	
Solids content (% by mass)	99 ± 1
Glass Transition (°C)	~ +9
Apparent density (g/cm ³)	0.45 to 0.60
Particle size (µm)	80 ± 10

Table 4. Physical characteristics of styrene-butadiene copolymer dispersion.

Physical analyses	
Solids content (% by mass)	50 ± 1
Viscosity (MPa.s)	2,000-5,000
Glass Transition (°C)	~ +3
Apparent density (g/cm ³)	1.0 ± 0.1
Particle size (µm)	80 ± 10

Table 5. Chemical analysis of carbonate filler.

Element	(%)
SiO ₂	6.48
Al ₂ O ₃	1.67
Fe ₂ O ₃	1.10
MgO	18.8
CaO	31.6
Na ₂ O	< 0.10
K ₂ O	0.40
SO ₃	0.14

Table 6. Chemical analysis of silica fume.

Element	(%)
SiO ₂	93.5
Al ₂ O ₃	0.43
Fe ₂ O ₃	0.45
MgO	0.47
CaO	0.23
Na ₂ O	0.16
K ₂ O	0.83
SO ₃	-

3.2. General procedures

The established compositions are shown in Table 7. For the analyzes of the influence of density, porosity and pore distribution on moisture movement of the composites, the range was formed by four compositions with different kinds of copolymer (A - without copolymer; B - styrene-acrylic addition; C - Va/Veova addition and D - styrene-butadiene addition).

The following procedure was employed for the mixing of fiber cements: (i) submission of the unrefined cellulose pulp to a stirring process in water and post-refining by several passes through 300 mm disc refiner until the achievement of CSF 220 mL refinement degree; (ii) dispersion of PVA fiber in water; (iii) dispersion of silica fume by ultrasonic equipment; (iv) mixing of all these materials in water; (v) addition of carbonate filler; (vi) addition of ordinary Portland cement; (vii) high-speed mechanical mixing during 300 s; (viii) sheet molding by vacuum dewatering process; (ix) cutting of the specimens (160 mm × 40 mm × 5 mm) 24 h after production; (x) cure in water until the 28th day of age; (xi) preparation of the specimens for physical and mechanical tests and mercury intrusion porosimetry (MIP).

3.3. Physical and mechanical characterization tests

Water absorption and bulk density results were obtained from the average of ten specimens for each formulation, following the procedures specified by ASTM C 948-81²⁹. Mechanical (flexural) tests were performed in a universal testing machine Emic DL-30000 equipped with 5 kN load cell. A span of 135 mm and a deflection rate of 5 mm/min were adopted in the flexural test. The deflection during the bending test was collected by the deflectometer positioned in the down side of the specimen. Modulus of rupture (MOR) and toughness were obtained. The absorbed energy was calculated by the integration of the load-deflection curve to the point corresponding to a reduction in load carrying capacity to 50% of the maximum observed. Toughness was defined as the energy absorbed during the flexural test and divided by the cross sectional area of the specimen.

3.4. Soak & dry accelerated ageing cycles

To study the influence of product age on the studied variables a soak & dry accelerated ageing cycles test was employed. It involved comparative analysis of the physical properties (water absorption and bulk density - except moisture movements) and mechanical performance of the composites before and after this test. The specimens were repeatedly immersed in water at 20 °C ± 5 °C during 170 min, followed by interval of 10 min, and then exposed to the temperature of 70 °C ± 5 °C for 170 min in a ventilated oven and final interval of 10 min. This procedure is similar to ISO 8336³⁰, however it was based on recommendations of the EN 494³¹ Standards and carried out in automated equipment for the application of the successive cycles. Each soak & dry set represents one cycle and was performed by 100 times (i.e., 100 cycles). The moisture movements were measured thru the expansion and shrinkage of the fiber cements composites, before and after their exposition to the

natural ageing of these materials (i.e., six months exposed to weathering).

3.5. Water Sorption

The expansion/shrinkage properties of the composites were monitored in a controlled environmental chamber. The relative humidity and temperature were pre-conditioned (70% RH and 40 °C respectively) and kept constant by specific equipment (Thermotron). The specimens with 28 days of age were also pre-conditioned. A group of 20 specimens was established for each composition (A, B, C and D as in Table 7) being the first ten specimens pre-conditioned by submerging in water for 48 h at 25 °C and the other ten specimens dried in the temperature of 105 °C by 48 h. After these procedures, all specimens were positioned inside the chamber and their expansion/shrinkage measured each week by a digital caliper. The behavior of the specimens was then plotted in expansion/shrinkage versus time curves.

3.6. Moisture movement

For the determination of the moisture movement of the fiber cements, the specimens were pre-conditioned in the temperature of 105 °C by 48 h. After this, they were cooled and measured by a digital caliper. Subsequently, the specimens were submerged in water at room temperature by 48 h. The moisture movement was obtained as function of the linear changes based on the length at dry and saturated conditions.

3.7. Mercury intrusion porosimetry (MIP)

MIP analyses were carried out in a PoreSizer 9320 – Micromeritics equipment. At 28 days the specimens were cut with a diamond saw and submitted to ultrasonic

cleaning equipment during 10 min. The samples were initially removed and vacuum-oven-dried at 40 °C for 24 h. The porosimeter used for pore size distribution was able to apply a pressure of approximately 410 MPa. For the analysis of the results, the intruded mercury values (intruded mercury volume by sample mass, in mL/g) were arranged within four distinct pressure intervals corresponding to different pore size ranges. The selected pore size ranges permit a good approach to cement matrix properties based on intruded mercury volumes. According to literature⁹, these pore size ranges can be correlated to cement matrix properties as shown in Table 8.

All analyses mentioned above were carried out in the Faculty of Animal Science and Food Engineering of Pirassununga (FZEA), Institute of Physics of São Carlos and in the Institute of Chemistry of São Carlos, University of São Paulo.

4. Results and Discussion

Table 7 shows the mixture proportions analyzed. Tables 9-13 show the results of analyses. Results of moisture movement are shown in Table 14.

As seen in Table 10, the copolymer additions not change water absorption of the fiber cements in the contents employed. It is unlike the literature for cementitious composites such as mortars and concretes reinforced by glass fiber²⁴⁻²⁷. The authors attribute this particular behavior to water excess for mixing and consequently vacuum form to simulate Hatschek process adopted by the industry. In this way, it turns not possible the formation of polymer film into matrices. Despite to be common the use of flocculent polymers in Hatschek process, there is not works in the literature about the use of these copolymers for fiber cement

Table 7. Mixture proportions (% by mass).

Fiber cement	Ordinary Portland cement	Silica fume	Carbonate filler	PVA fiber	Cellulose fiber	Copolymer (*)	Approximated water/cement ratio
A without copolymer	80	8	7	2	3	-	0.22
B styrene-acrylic	80	8	7	2	3	2.7	0.22
C Va/Veova	80	8	7	2	3	2.7	0.22
D styrene-butadiene	80	8	7	2	3	2.7	0.22

*Percentage of copolymer addition applied to the total dry mass.

Table 8. Classification of pore size in hydrated cement paste/fiber cement.

Designation	Pore diameter	Properties affected/Relations
Hydrated phases (gel) porosity	< 10 nm	Shrinkage and creep
Medium capillaries	10-50 nm	Strength, permeability, creep and shrinkage
Large capillaries	50 nm-1µm	Strength and permeability
Fiber/matrix voids	300-800 nm	Cellulose fibers
Large capillaries/ entrained air	> 1µm	Strength
Fiber/matrix voids	1.5 µm	Unrefined cellulose fibers
Large fiber/matrix voids	2 µm	Fiber/matrix failure

Table 9. Mechanical properties at 28 days of age.

Fiber cement	Modulus of rupture MOR (MPa)	SD*	Limit of proportionality (MPa)	SD	Toughness (kJ/m ²)	SD
A	11.35	0.90	9.05	1.05	2.65	0.85
B	10.50	1.25	7.00	0.90	3.40	1.15
C	11.30	1.45	8.90	2.10	2.95	1.30
D	11.20	1.35	9.50	1.15	2.60	0.90

*Standard deviation.

Table 10. Physical characteristics at 28 days of age.

Fiber cement	Water absorption (%)	SD*	Bulk density (g/cm ³)	SD	Apparent porosity (%)	SD
A	20.55	1.25	1.65	0.05	33.40	1.15
B	20.00	0.60	1.65	0.01	33.05	0.75
C	20.85	1.40	1.60	0.05	33.75	1.35
D	20.40	0.55	1.60	0.02	32.65	0.55

*Standard deviation.

Table 11. Mechanical properties after soak & dry cycles.

Fiber cement	Modulus of rupture MOR (MPa)	SD*	Limit of proportionality (MPa)	SD	Toughness (kJ/m ²)	SD
A	11.50	1.00	9.85	1.45	1.25	0.40
B	11.25	0.35	9.05	1.05	1.80	0.45
C	10.55	0.80	9.80	1.35	1.65	0.50
D	12.90	1.20	11.60	2.00	1.80	0.70

*Standard deviation.

Table 12. Physical characteristics after soak & dry cycles.

Fiber cement	Water absorption (%)	SD*	Bulk density (g/cm ³)	SD	Apparent porosity (%)	SD
A	18.50	0.80	1.65	0.03	30.70	0.90
B	20.65	1.20	1.55	0.03	32.20	1.20
C	18.00	1.45	1.65	0.04	29.40	1.85
D	17.00	2.20	1.62	0.06	27.70	2.60

*Standard deviation.

Table 13. Results from porosimetry analyses.

Fiber cement	Opened porosity at 28 days (%)	Opened porosity 6 months old (%)	Real density at 28 days (g/cm ³)	Real density 6 months old (g/cm ³)	Mean diameter at 28 days (μm)	Mean diameter 6 months old (μm)
A	6.53	6.17	1.64	1.64	0.0243	0.0501
B	7.64	6.70	1.53	1.62	0.0312	0.0423
C	7.62	5.48	1.63	1.76	0.0269	0.0406
D	7.52	6.13	1.59	1.70	0.0361	0.0620

formulations. In this way, these copolymers appear not to be appropriated to this process and probably the polymer matrix phase it was not made into cement hydrate phase. Considering the results of porosimetry, it turns evident because as known the use of polymers decrease the pore diameters. Table 13 not evidences this behavior.

However, the principal aim of copolymer addition was accomplished, that is, changes in the density and porosity variables as showed in the Table 13. The mercury intrusion porosimetry (MIP) showed to be an important technique to measure the influence of the copolymers in these variables.

In relation to flexural tests, all copolymer additions increased the toughness of the fiber cements as described by the literature³. It was also observed that the soak and dry procedure decreased the energy absorption of the composites. These results have probably correlation to copolymer degradation in the alkaline environment of Portland cement matrices and the increasing of Young's modulus of these composites from densification of the matrixes as showed in Table 13.

Figures 1-8 show the moisture sorption of the fiber cements under evaluation. It is pointed out that movement of water within composites is very complex. Regardless

Table 14. Moisture movement.

Fiber cement	Moisture movement at 28 days (mm/m)	SD*	Moisture movement at 6 months (mm/m)	SD
A	2.0	0.1	4.0	0.4
B	1.4	0.1	4.3	0.3
C	2.1	0.3	3.8	0.2
D	1.9	0.2	2.4	0.4

*Standard deviation.

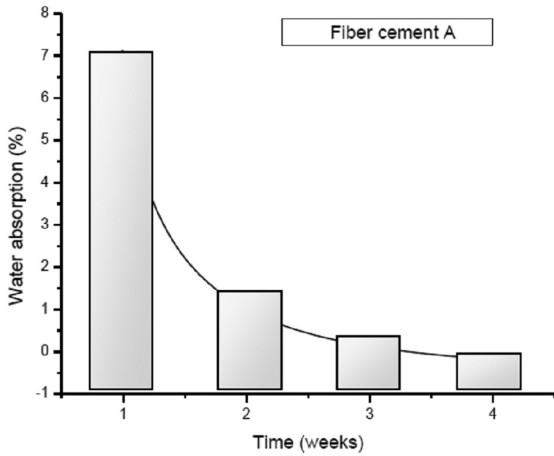


Figure 1. Water absorption evolution up to 28 days – fiber cement A.

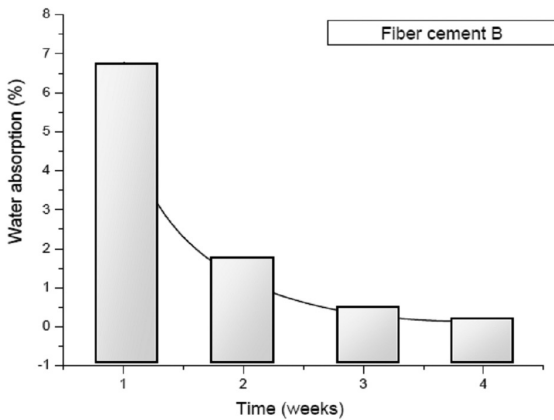


Figure 2. Water absorption evolution up to 28 days – fiber cement B.

of this, the composites performed hygral behavior with intensive loss of water and water adsorption during the first weeks and tendency to be close to zero after certain period of time. For the conditions established in the present study, this stabilization was observed after four weeks.

The moisture movements perform the principal target of this study and are showed in Table 14. The changes in the variables density and porosity made composites with different properties. Both the bulk density and the water absorption strongly influence the moisture expansion/shrinkage of the asbestos-free fiber cement. On the other hand, the total porosity did not show to be very important.

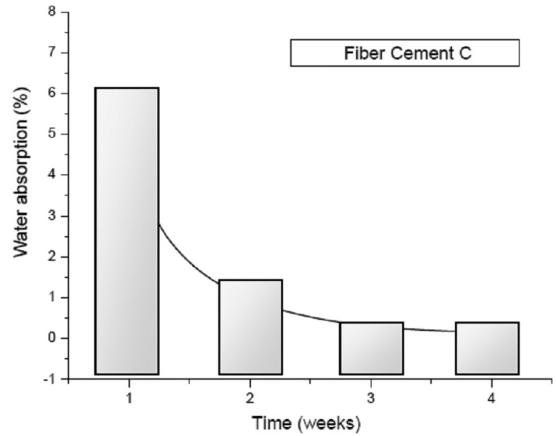


Figure 3. Water absorption evolution up to 28 days – fiber cement C.

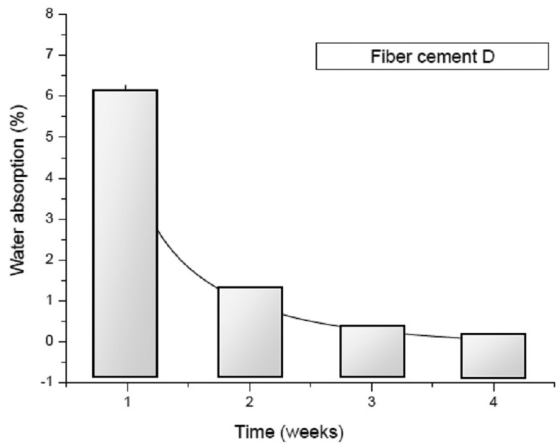


Figure 4. Water absorption evolution up to 28 days – fiber cement D.

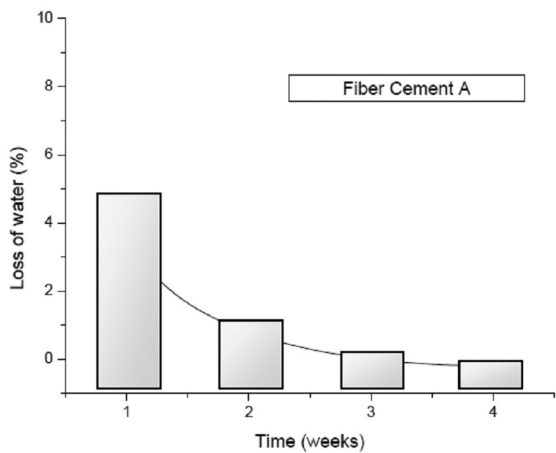


Figure 5. Loss of water evolution up to 28 days – fiber cement A.

Despite of that, the pore structure or pore size distributions showed to be the most important characteristic in the control of moisture movement of these composites.

Figures 9-16 show the curves of intruded mercury volume in function of pore diameter (incremental volume) for the fiber cements under analyses. Pore size distribution

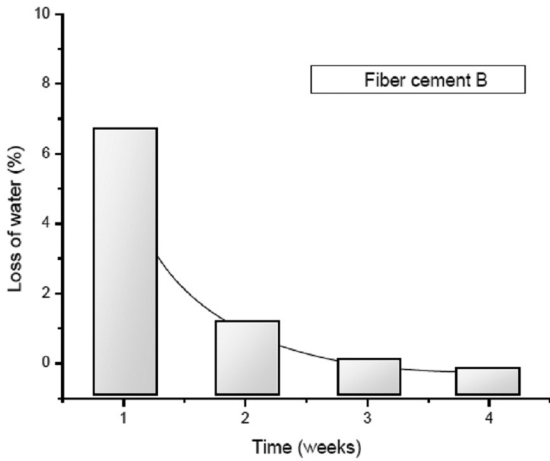


Figure 6. Loss of water evolution up to 28 days – fiber cement B.

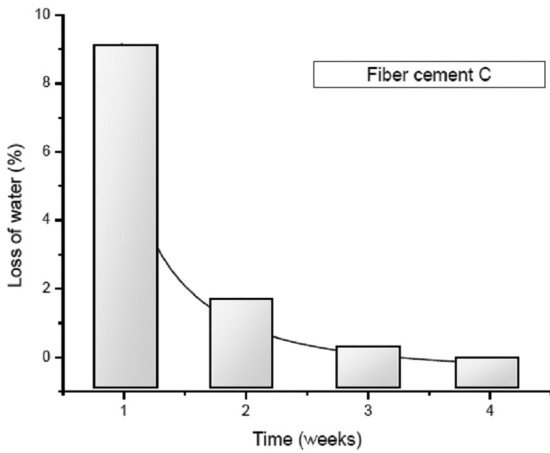


Figure 7. Loss of water evolution up to 28 days – fiber cement C.

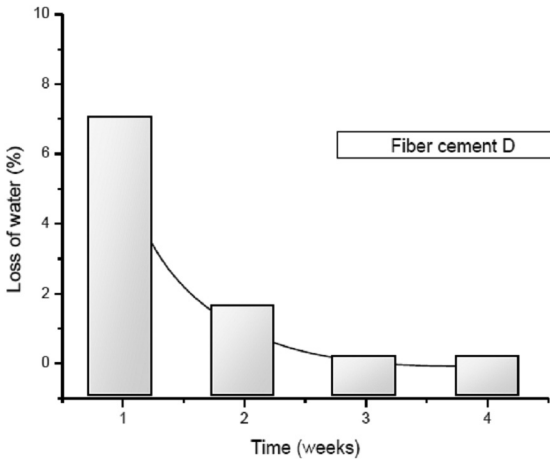


Figure 8. Loss of water evolution up to 28 days – fiber cement D.

curves exhibit several characteristic peaks showed in Table 8. The peaks around 3-10 nm and 40-100 nm are related to pores of gel (hydrated phases) and medium capillaries respectively. These peaks showed to be higher at 28 days than after 6 months of age. Probably, these results are from matrix carbonation and continued hydration of

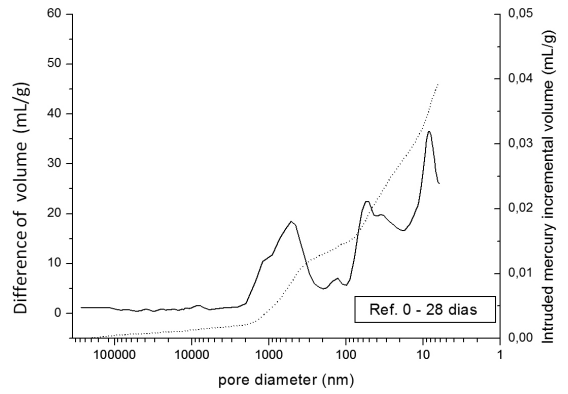


Figure 9. Porosimetry of fiber cement A at 28 days.

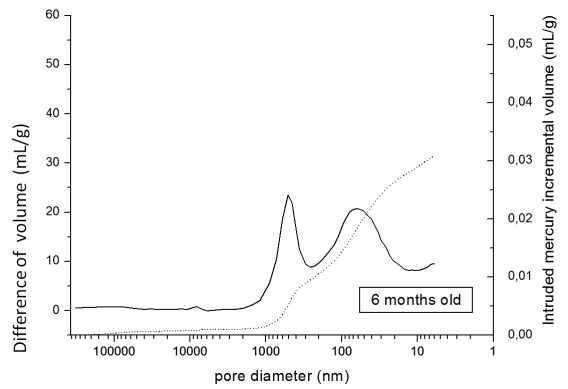


Figure 10. Porosimetry of fiber cement A at six months of age.

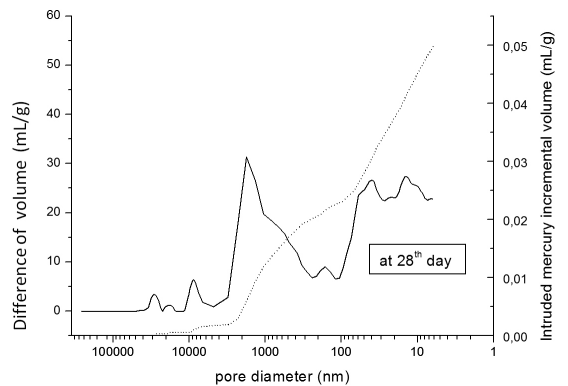


Figure 11. Porosimetry of fiber cement B at 28 days.

ordinary Portland cement phases. The decrease of these peaks showed correlation to the increment of moisture expansion/shrinkage of the analyzed composites. The peaks around 1-5 μm are probably from voids in the fiber/matrix interface. These peaks showed to be higher in the composites of lower moisture movements.

The density of the non-asbestos fiber cement plays a very important role in the moisture expansion/shrinkage of these composites like their pore structures or pore distribution.

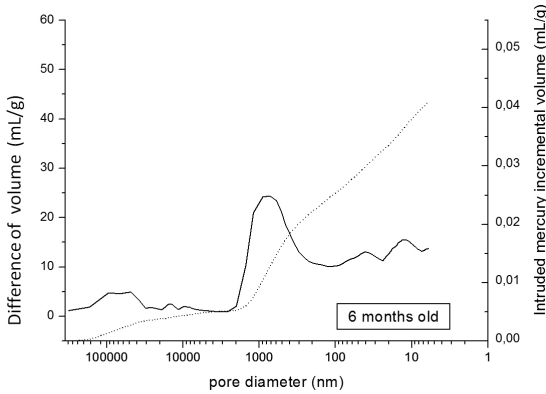


Figure 12. Porosimetry of fiber cement B at six months of age.

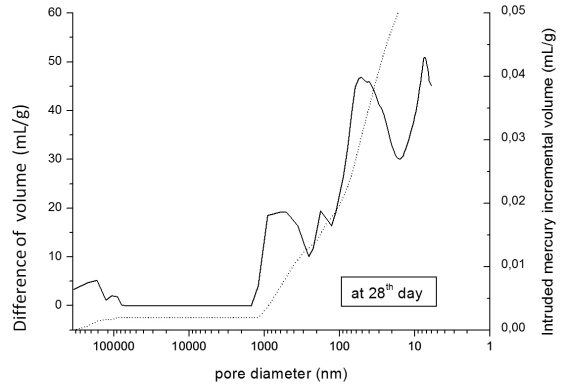


Figure 15. Porosimetry of fiber cement D at 28 days.

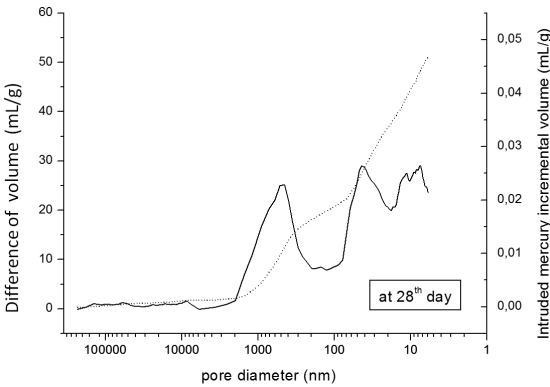


Figure 13. Porosimetry of fiber cement C at 28 days.

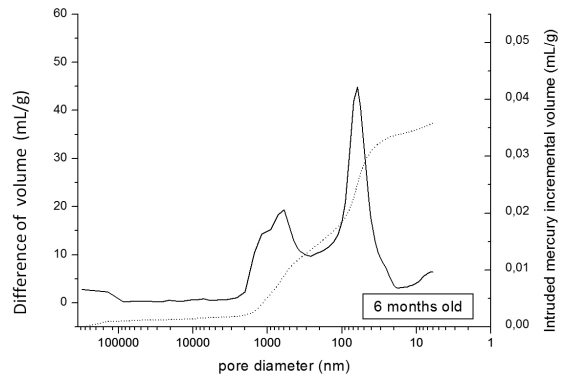


Figure 16. Porosimetry of fiber cement D at six months of age.

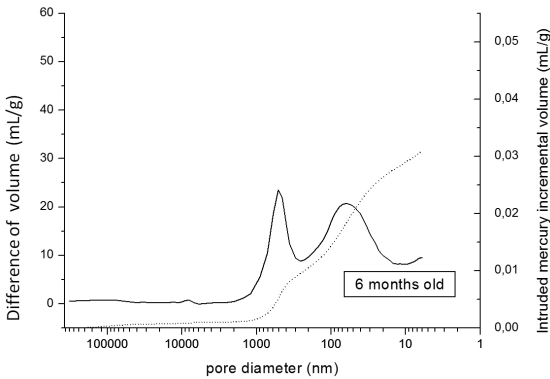


Figure 14. Porosimetry of fiber cement C at six months of age.

Although the hygral behavior of the fiber cements is very complex, concluding remarks are showed below:

- Migration of the pore water of fiber cements can be related to the density;
- Moisture movements (expansion/shrinkage) are related to water absorption and density of these materials and also not strongly related to total porosity;
- Density and porosity are related to each other; however, for products of equivalent density having different pore distribution, the pore structure appeared

to be the most important characteristics to the moisture movements;

- The capillary pores around 40-100 nm have strong influence in the hygral behavior of these composites when exposed to weather;
- The hydration during the time of the non-hydrated phases of cement paste and carbonation may increment the expansion/shrinkage of these composites;
- The pores around 1-5 μm probably relate to voids in the fiber/matrix interface and may collaborate to decrease of expansion/shrinkage of asbestos-free fiber cements;
- The use of copolymers in the Hastschek process is not sufficient to control the moisture movement and expansion/shrinkage of this cementitious composite.

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