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The hornification of vegetable fibers to improve the durability of cement mortar composites

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ABSTRACT

The use of vegetable fibers to reinforce a cement-based matrix has its weakest point in terms of durability. The alkalinity of the matrix and the volumetric instability of the fibers are the main causes of the loss of resistance of vegetable fiber-reinforced cement mortar composites.

The aim of this study was to determine the effects of the previous hornification of the vegetable fibers on the mechanical performance and durability of softwood kraft pulp and cotton linters cement mortar composites. For this purpose, composites containing 4 wt.% of the hornificated fibers and the untreated ones were prepared with both fiber types. The mechanical performance of these composites was tested after 28 days of cure treatment and after aging with four wet–dry cycles. Results indicated that the previous treatment of fibers had beneficial effects on the mechanical performance and durability of the resulting cementitious composites.

1. Introduction

The use of vegetable fibers to reinforce brittle matrices such as cement mortar or concrete constitutes an interesting possibility that offers many advantages with respect to the utilization of other fibers or reinforcements. Due to their mechanical properties, vegetable fibers improve the ductility, flexibility and crack resistance of the resulting material, among other benefits [1]. As a consequence of their hydrophilic nature, vegetable fibers are ideal candidates for hydraulic matrices. Due to their low cost, the use of vegetable fibers in fiber–cement materials constitutes a very interesting option for the building industry, mainly in less developed countries or countries that need low cost constructions [2–5]. Finally, vegetable fibers are obtained from renewable sources and are biodegradable, and are therefore "eco-friendly".

Despite all the aforementioned advantages, the industrial production of cement base composites reinforced with vegetable fibers is currently limited by the lack of durability of these materials [6,7]. In this sense, the scientific community has been making substantial efforts in the last decade to improve the durability of vegetable fiber-reinforced cement matrix composites (VFRCMC) [3,8].

Many studies have related the presence of calcium hydroxide in the cementitious matrix with the degradation of the vegetable fibers, and thus with the loss of durability of the VFRCMC [3,6,7,9,10,11–13]. Mohr et al. [6] established the following sequence of damage that occurs in the vegetable fibers when the composite is subjected to various wet–dry cycles: (a) loss of adherence between the fiber and the matrix after the second wet–dry cycle; (b) reprecipitation of the hydrated compounds within the void space at the former fiber–cement interface during the first ten wet–dry cycles; (c) full mineralization, and thus the embrittlement of the vegetable fibers after ten wet–dry cycles. Likewise, Tolêdo Filho et al. [7] demonstrated alkaline attack of the fibers after various wet–dry cycles. Using an X-ray diffraction technique and Thermogravimetric Analysis Claramunt et al. [11,12] corroborated the migration of the hydration compounds of the cement to the vegetable fibers according to the following process: (a) in the first dry cycle the transversal section of the vegetable fibers is reduced due to the loss of water. This reduction causes loss of adherence with the matrix and the appearance of void spaces at the fiber–matrix interface; (b) in the subsequent wet cycle, the water dissolves the hydration compounds of the cement (calcium hydroxide). The vegetable fibers absorb this dissolution of calcium hydroxide and thus swell; (c) in the second dry cycle, water is lost

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by evaporation and the calcium hydroxide precipitates on the surface and in the lumen of the fibers. During the subsequent wet–dry cycles there is a "pump-like" effect with the consequent densification of the surface and lumen of the fibers with products with high alkalinity.

There are basically two options for improving the durability of the VFRCMC. One possibility is to modify the composition of the matrix in order to reduce or remove the alkaline compounds. Tolêdo Filho et al. used pozzolanic additions to precipitate the calcium hydroxide of the matrix as calcium silicate hydrate, or treatments with a higher concentration of CO_2 to precipitate the calcium hydroxide as calcium carbonate [14,15]. This modification is an effective way to guarantee the durability of the VFRCMC but the dosage of the cementitious materials must be analyzed carefully in order to achieve similar or higher performance compared to the composites prepared only with ordinary Portland cement.

The second way to increase the durability of the VFRCMC is to modify the surface of the fibers with chemical or physical treatments to increase their stability in the cementitious matrix [16–18]. Some of these treatments imply the use of chemical reagents and could be complex; making the industrial processes more expensive.

Hornification is an irreversible effect that appears on cellulosic fibers when they are subjected to drying and rewetting cycles. This effect, quantified as the percentage reduction in water retention values (WRV), principally causes shrinkage of the fibers due to the formation of hydrogen bonds in cellulose, and does not modify the resistance of the fibers [19,20]. Claramunt et al. [20] analyzed this effect in softwood pulps and fibers from cotton linters and proved that the hornification process causes a substantial decrease in the WRV of these fibers, mainly the softwood pulp fibers. With respect to the morphology of the fibers, hornification does not modify the length of the fibers but significantly decreases their wall thickness while fiber width remains constant.

The reduction in the WRV of the hornificated fibers could have beneficial effects on VFRCMC. On the one hand, these fibers have a higher dimensional stability, and thus higher fiber–matrix adherence is expected. On the other hand, as a consequence of the lower WRV, a reduction in the formation of incrustations of calcium hydroxide on the surface and lumen of the fibers and thus a reduction in the degradation of the cellulose in the cementitious matrix may occur.

The main objective of this study was to analyze the influence of prior hornification of the vegetable fibers on the mechanical performance and durability of the cement mortar composites. To achieve the aforementioned objective, two types of cellulosic fibers (chemical pulp from softwood and cotton linters) with or without prior hornification treatment, were used to prepare different specimens of cement mortar composites. The mechanical performance of these composites was tested after 28 days of curing and after four wet–dry aging cycles.

2. Materials and experimental procedures

2.1. Materials

UNE-EN 197-1:2000 [21] Type I cement supplied by *Ciments Molins* (Spain) was used in this research. Table 1 shows the chemical composition of this cement (provided by the supplier). The sand used was supplied by the company Sibelco and was subjected to grinding with a Jar mill (1000 cm^3 of capacity and spheres of 12 mm) at 400 rpm during 5 min to achieve a maximum particle size of 0.1 mm. The particle size was measured using sieve series following UNE-EN 933-2:1996 standard [22]. Sika Viscocrete-3425 fluidizer, obtained from Sika S.A.U., was used at a maximum dosage rate of 40 g/1000 g of cement to aid workability.

Table 1
Chemical composition of the cementing material used in this study.

Chemical	Oxide equivalent (wt.%)
SiO_2	20.17
Al_2O_3	5.34
Fe_2O_3	3.85
CaO	63.93
MgO	0.91
Na_2O	0.05
K_2O	0.35
SO_3	4.00
Loss on ignition	0.80
Insoluble residue	0.05
C_3S	54.16
C_2S	7.64
C_3A	16.97
C_4AF	11.70

Unbleached softwood kraft pulp (*Pinus insignis*) with a 53 Kappa number (7.8 wt.% of lignin), supplied by Smurfit Kappa Nervión, S.A. (Spain), and cotton linters provided by Celsur (Cotton South, S.L., Spain), were used as reinforcement.

2.2. Fiber treatment

The hornification of the fibers was achieved by four drying and rewetting cycles. The sequence of the drying and rewetting cycles was as follows: (1) drying in an oven with air recirculation at 60 °C for 7 h; (2) rewetting by soaking overnight; (3) disintegration of the wet pulp for 30,000 revolutions (ISO 5263-1:2004 [23]). This step consisted in a mechanical treatment in water where the dried interlaced fibers were separated from one another without appreciably changing in their structural properties. A normalized pulp disintegrator was used; and (4) filtration of the pulp suspension through a Buchner funnel equipped with a wire screen (150 mesh). The first filtrate was recirculated during the pad formation with the objective of retaining pulp fines. At the end of the process the fibers were dried at 60 °C for 12 h and stored in sealed bags until the composite samples were prepared.

2.3. Fiber characterization

2.3.1. Water retention value (WRV)

The WRV was determined by centrifugation according to ISO 23714:2007 [24].

2.3.2. Viscosity measurements

Viscosity was measured according to ISO 5351-1:2004 (TAPPI T 230, 1994) [25] using cupriethylenediamine (CED) as a solvent and a Schott capillary viscometer.

2.3.3. Fiber dimensions

The length, width, lineal mass (coarseness), and curl index were measured on a Kajaani FS300 Analyzer according to ISO 16065-1 [26]. Measurements were taken from more than 10,000 fibers.

2.3.4. X-ray diffractometry

X-ray diffraction (XRD) was performed using a Siemens D-500 diffractometer with Cu Kα radiation ($\lambda = 0.154$ nm) operating at 40 kV and 30 mA. The crystalline-to-amorphous ratio of the cellulose pulps was determined using the empirical procedure first proposed by Segal et al. [27]. This method consists of estimating the crystallinity index for Cellulose I (Cr.I.) according to the following equation:

$$Cr.I(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

where I_{002} is the maximum intensity (in arbitrary units) of the diffraction from the (0 0 2) plane at $2\theta = 2.6°$ and I_{am} is the intensity of the background scatter measured at $2\theta = 16°$

2.4. Composite preparation

2.4.1. Cement mortar composition

To analyze the effect of the hornification treatment on the mechanical properties and durability of the composites, two series of 36 specimens were prepared with the treated and as received wood fibers and cotton linters. One of the series was subjected to a process of accelerated aging. The theoretical and experimental composition of the specimens and the treatments applied to the fibers and the composites are presented in Table 2. The ratio cement:sand:water used was 1:1:0.4 [13]. The fiber amount was fixed at 4 wt.% of the mixture for both, the composites prepared with the kraft pulp and the ones prepared with the cotton linters. This wt.% is the maximum quantity of fibers which allows to prepare homogeneous composites. To verify the repeatability of the results, each group of samples was made up twice 30 days apart.

Table 2
Reference and composition of the prepared cement mortar composites.

Sample reference*	Kind of fiber	Hornification	Aging	Cement (g)	Sand (g)	Fiber (g)	Water mortar (g)		Water/ cement initial		Water loss (g)		Solid loss (g)		Final cement (g)		Final water (g)		Final water/ cement		Water/ cement average
Mixture**							1	2	1	2	1	2	1	2	1	2	1	2	1	2	
KP	Softwood pulp	No	No	365	365	30	451	445	1.24	1.22	220	224	10	14	355	351	231	221	0.65	0.63	0.64
KP-A	Softwood pulp	No	Yes	365	365	30	440	455	1.21	1.25	221	231	10	12	355	353	219	224	0.62	0.63	0.63
HKP	Softwood pulp	Yes	No	365	365	30	451	453	1.24	1.24	235	229	18.6	16	346	349	216	224	0.62	0.64	0.63
HKP-A	Softwood pulp	Yes	Yes	365	365	30	452	453	1.24	1.24	226	235	16.7	15	348	350	226	218	0.65	0.62	0.64
CL	Cotton linter	No	No	365	365	30	417	409	1.14	1.12	197	185	13.2	11	352	354	220	224	0.63	0.63	0.63
CL-A	Cotton linter	No	Yes	365	365	30	415	421	1.14	1.15	188	198	12	13	353	352	227	223	0.64	0.63	0.64
HCl	Cotton linter	Yes	No	365	365	30	430	416	1.18	1.14	204	205	9.51	11.5	355	354	226	211	0.64	0.60	0.62
HCl-A	Cotton linter	Yes	Yes	365	365	30	417	411	1.14	1.13	189	196	11.2	12	354	353	228	215	0.64	0.61	0.63

* KP: kraft pulp; CL: cotton linter; H: hornificated; A: aged.
** To verify the repeatability of the results, each group of samples was mixed twice with 30 days of difference. Each group of samples correspond to mixture 1 or mixture 2.

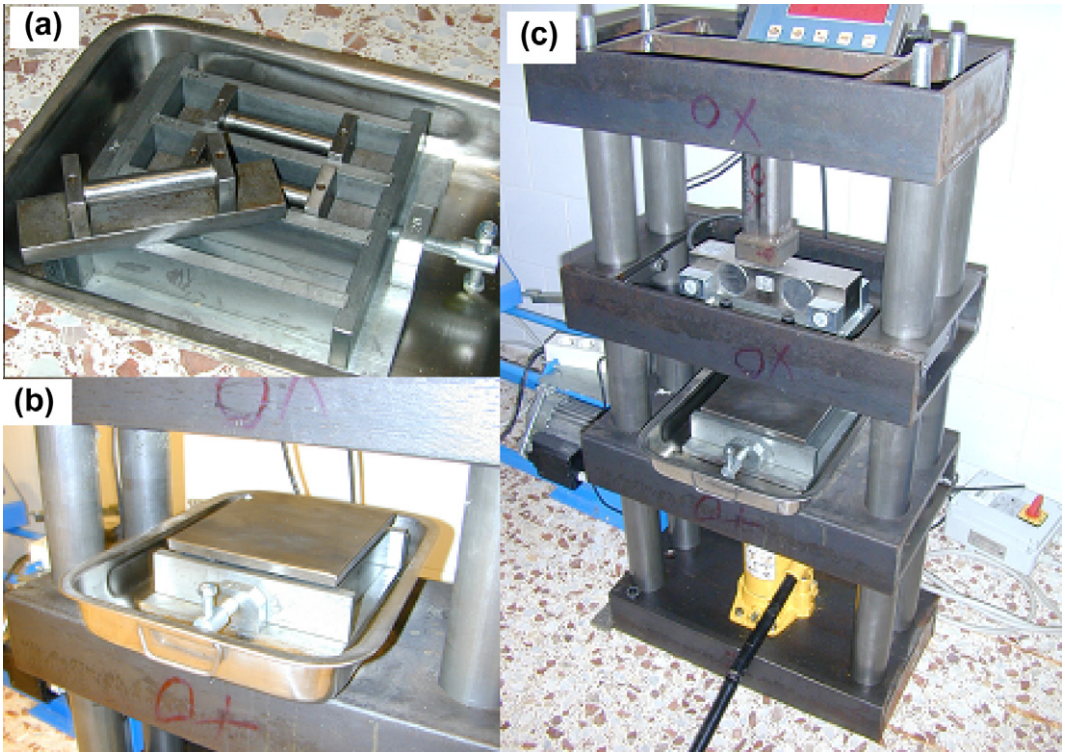

Fig. 1. Mould and press system used to prepare the specimens. (a) The moulds, (b) the lid used to cover the mould, and (c) mould placed on the hand-press.

2.4.2. Mixing process

The mixture of vegetable fibers with the cement mortar matrix was prepared as follows:

1- Dispersion of 30 g of dried fibers (kraft pulp or cotton linters) in 1.5 L of water, followed by mixing for 5 min at 60 rpm.
2- Filtration and compression of the fibers to achieve a mass of 230 g.
3- Dispersion of 365 g of cement and 5 g of the fluidizer in 0.25 L of water followed by mixing for 1 min at slow speed (140 rpm).
4- Dispersion of 365 g of sand in the mixture of cement and fluidizer followed by mixing for 1 min at slow speed and then 1 min at fast speed (285 rpm).
5- Dispersion of the fibers (kraft pulp or cotton linters) in the mixture and mixing for 3 min at slow speed. After standing the paste for 5 min 5 g of fluidizer were added and the paste was mixed for 3 min at slow speed. If it was necessary to incorporate more water the mixing process was longer.

2.4.3. Specimen preparation

For each composition prismatic specimens were prepared for compression and flexural tests. The mould used was UNE-EN 196-1:2005 [28] type with internal dimensions of 40 × 40 × 160 mm^3 modified to allow the compression of the specimens to 20 mm in thickness. This modification was also useful for removing the excess water and increasing the compaction of the material [29,30]. Fig. 1 illustrates the modification to the mould, which involved adding three lids provided with easels (Fig. 1a) and a lid that covers the mould and allows the homogeneous distribution of the load (Fig. 1b). All rims of the mould were microscored to allow the evacuation of water with the minimum loss of cement and sand. The mass placed in each of the three parts of the mould was optimized to almost fill the mould (875 g ± 0.1 g). The mould was placed on a plate and then compacted on a vibrating table for 2 min at 60 Hz, after which it was hand-pressed to reach a final pressure of 0.4 MPa for 24 h (Fig. 1c). The pressing process was carried out in several stages to allow the evacuation of water through the microslots of the mould. Five steps of 5 min each and an increase of 0.08 and 0.1 MPa were performed to reach the maximum pressure. The system used allowed the lixiviate of the sample to be measured. This lixiviate was dried and weighed to accurately determine the water/cement ratio in the specimen. As shown in Table 2, regardless of the initial water used, the final ratio was always between 0.62 and 0.64.

After demoulding, the specimens were cured for 28 days in a curing box at 20 ± 1 °C and 95% relative humidity. Three specimens were prepared for flexural tests and six specimens for compression tests for each composition.

2.4.4. Accelerated aging of the composites (wet/dry cycling)

To analyze the durability of the composites, one of the series prepared was artificially aged using four wet/dry cycles. This method has been widely used to study the degradation of vegetable fibers in cementitious matrices [31–33].

To establish the optimum wet/dry cycle length to ensure the full saturation and drying of the composites, three extra specimens were firstly dried in an oven at 60 °C and then immersed in water. Readings of gain and loss of mass with respect to the just cured specimens were taken every 3 h until constant mass. The mass change over time for the wetting and drying cycles is shown in Fig. 2. The wet/dry cycle used for this study was 72 h drying in an oven provided with open air circulation at 60 °C and 96 h soaking in water at room temperature.

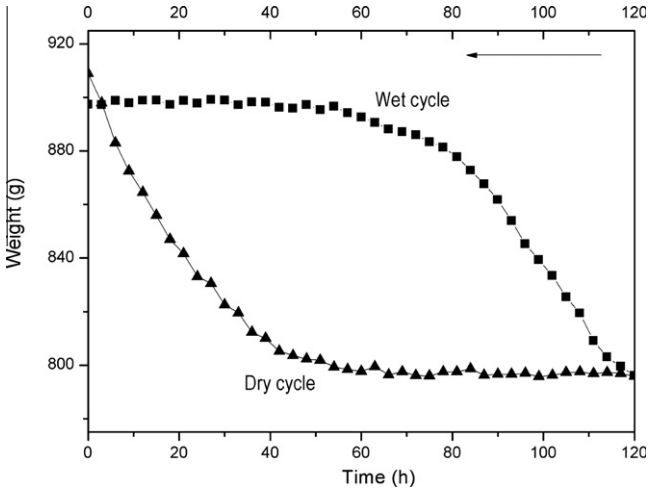

Fig. 2. Variation in mass with time of the VFRCMC specimens subjected to wet/dry cycles.

To avoid "heat shock" in the specimens, the drying process was always initiated with the oven at room temperature, and the wetting process with the water at 60 °C but without heating.

2.5. Composite characterization

2.5.1. Mechanical characterization of the composites

The mechanical characterization of the composites was performed via both flexural and compressive tests using an Incotecnic press equipped with a maximum load cell of 30 kN. The flexural tests were performed with a three-point bending configuration at a load speed of 50 ± 10 N/s. The compressive tests were performed on specimens of 40 × 20 mm, with a 2:1 width:thickness ratio. The load speed for the compression tests was 2400 ± 200 N/s. Both flexural and compressive tests were performed following the UNE-EN 196-1:2005 methodology [28]. Toughness was defined as the area under the load–deflection curve until 40% of the maximum force, following the recommendations of the 49-TFR "Energy absorption on FRC sections" of RILEM [34].

2.5.2. Microstructural characterization

The fibers morphology in the composites was characterized using a Jeol JSM 6400 scanning electron microscope (SEM). The surface of a dried suspension of the fibers was made conductive by sputtering deposition of a thin layer of gold. 1000× magnifications were used.

3. Results and discussion

3.1. Physical and morphological characterization of the hornificated fibers

Table 3 shows the results of the physical and morphological characterization of the kraft pulp and cotton linter fibers as received and after treatment with the wet/dry cycles.

The phenomenon of hornification was quantified as the percentage reduction in the WRV measured in the pulp after they were subjected to the drying and rewetting cycles. As shown, WRV decreased with the number of cycles, with this decrease being more significant in the fibers from kraft pulp than the cotton linters, due to the presence of hemicelluloses on the softwood pulps.

Table 3
Physical and morphological characterization of the kraft pulp and cotton linters.

	Number of wet–dry cycles	Water retention (%)	Length (mm)	Width (μm)	Aspect ratio (length/width)	Lineal mass (mg/m)	Curl index (%)	Viscosity (cm^3/g)	Crystallinity (%)
Kraft pulp	Initial	126	1.37	28.28	48.4	0.256	15.96	968	56.8
	I	112	1.49	28.32	52.6	0.252	15.79	945	57.1
	II	102	1.37	26.65	51.4	0.231	13.51	916	57.8
	III	97	1.32	26.72	49.4	0.261	12.15	902	60.4
	IV	90	1.36	25.91	52.5	0.252	12.94	888	58.0
Cotton linters	I	58	0.79	20.14	39.2	0.244	25.66	755	78.7
	II	54	0.81	19.95	40.6	0.24	26.98	741	82.0
	III	52	0.86	19.61	43.9	0.261	26.78	770	81.0
	IV	50	0.84	19.63	42.8	0.237	27.16	779	80.9

As expected any significant changes were observed in the length and width of the fibers after the drying and rewetting cycles [35,36].

The fiber curl index, defined as the gradual and continuous curvature of the fibers, was found to decrease with the drying and rewetting cycles of the kraft pulp. This decrease could be related to an increase in the stiffness of the fibers as a consequence of the hornification process. Nonetheless, the curl index of the cotton linters showed a slight increase with the drying and rewetting cycles. The effects were less noticeable in these fibers, probably as a consequence of the lower percentage of hornification (quantified as the percentage reduction in water retention values) than in the fibers from the kraft pulp.

The viscosity of chemical pulps dissolved in CED solution is considered to be a measure of the degradation of cellulosic material and an indication of its general strength potential [37]. As shown in Table 3, the changes in the CED viscosity with the drying and rewetting cycles were insignificant. Only a slight decrease was observed in the kraft pulp.

The crystallinity index (Cr.I.) obtained from X-ray diffractograms for the kraft pulp and the cotton linters and its variation with the drying and rewetting cycles are shown in Table 3. The

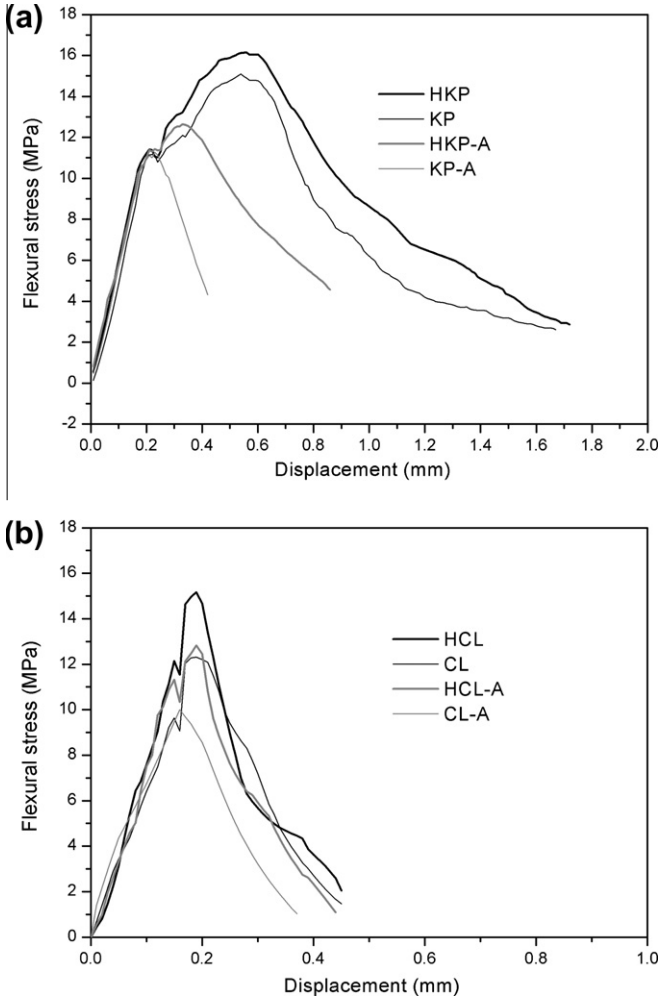

Fig. 3. Typical flexural curves of the composites reinforced with the hornificated fibers and with the untreated fibers. The thin lines correspond to the aged composites.

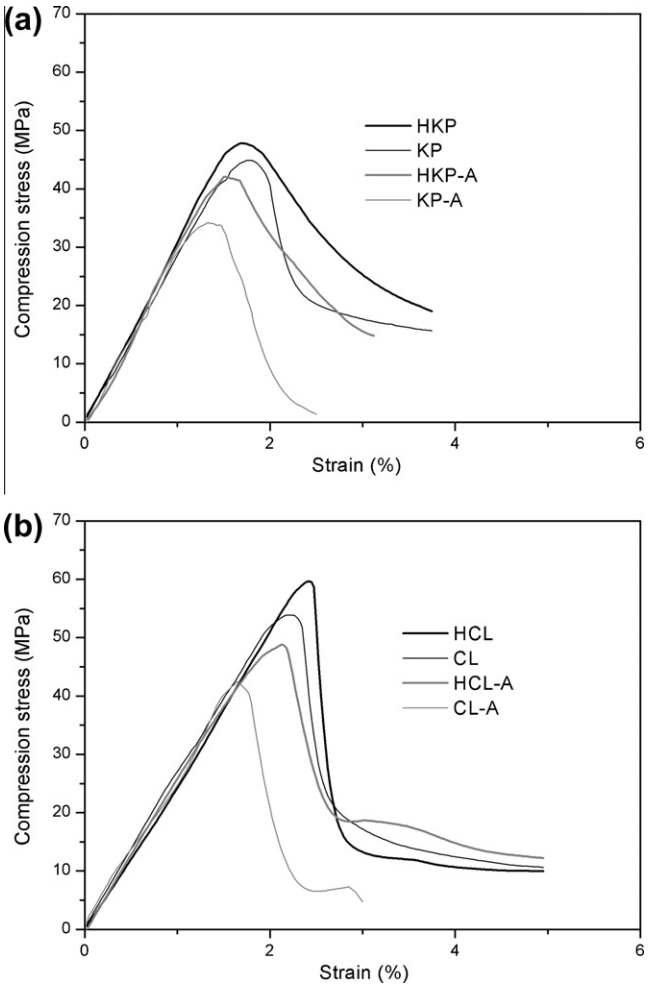

Fig. 4. Typical compression curves of the composites reinforced with the hornificated fibers and with the untreated fibers. The thin lines correspond to the aged composites.

cotton linters had significantly higher values of Cr.I. than the kraft pulp (around 20% higher). The higher crystallinity of the cotton linters with respect to the kraft pulp is related with the higher α-cellulose content in the cotton linters. Nevertheless, no significant differences in crystallinity were found with the drying and rewetting cycles.

3.2. Effect of fiber hornification on the mechanical properties of the composites

Figs. 3 and 4 show the typical load–deflection curves for the specimens analyzed under flexural and compressive tests. Overall, the composites prepared with the hornificated fibers had superior mechanical properties. The typical stress–strain behavior for a cement mortar reinforced with fibers shows first a line with high slope and a very linear stress–strain relationship. This behavior basically corresponds to the work of the matrix. After reaching the maximum stress of the matrix, the specimen fractures, this fracture being more or less abrupt depending on the ductility of the material. Nevertheless, if the mortar is reinforced with fibers there is a stress transfer from the matrix to the fibers beyond the maximum stress point that depends on the reinforcement capacity of the fibers and their adhesion to the matrix. As shown in Fig. 3, although both the composites with the kraft pulp and the cotton fibers had a similar maximum stress, the former showed less steeper curves than the latter, probably due to the lower stiffness of the kraft pulp fibers (due to the lower crystallinity of these fibers with respect to the cotton linters).

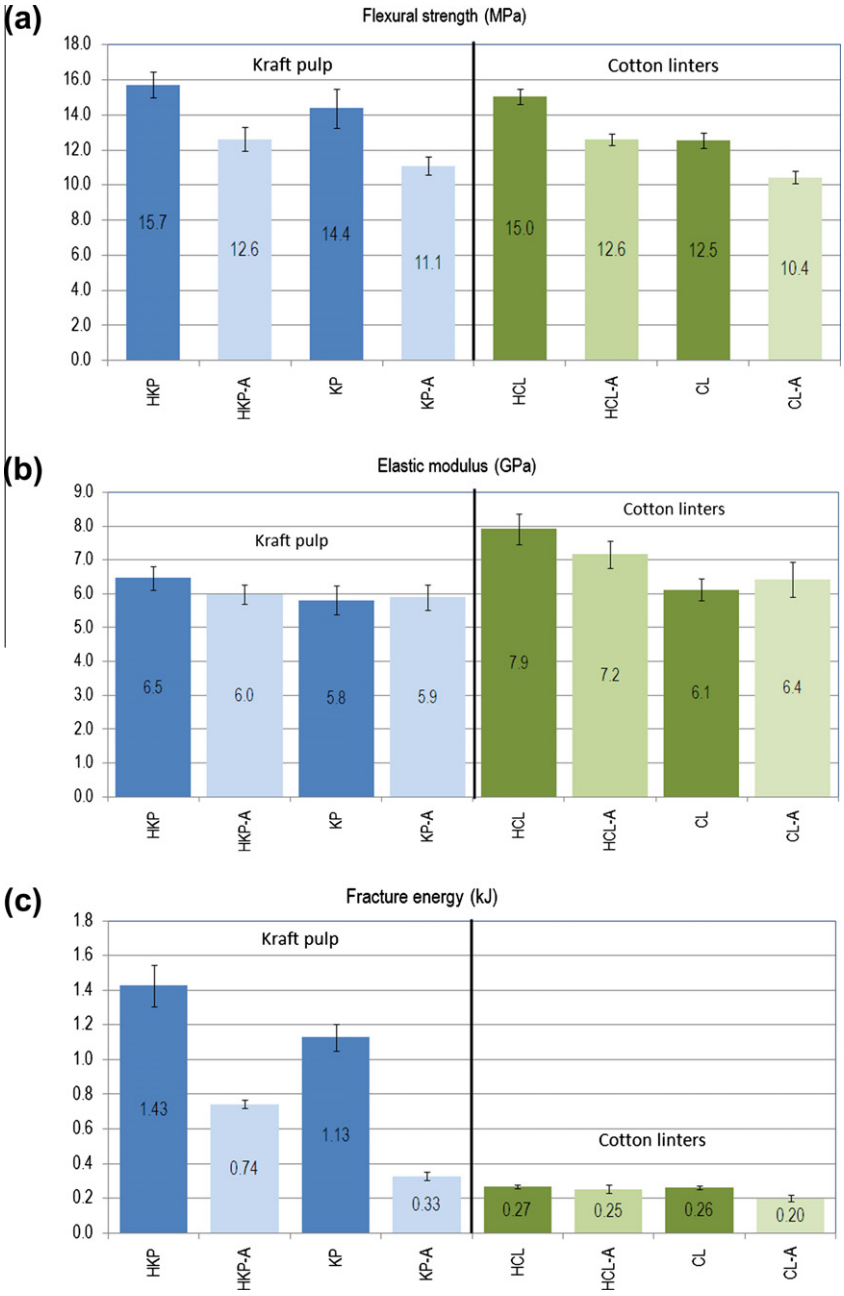

Fig. 5. Flexural test results for the composites prepared with the kraft pulp (left) and cotton linters (right). The light colors correspond to the aged specimens: (a) average values of the maximum strength, (b) average values of the elastic modulus, and (c) average values of the fracture energy.

During compression (Fig. 4), the matrix developed the resistance of the composite. Overall, the fibers increased the porosity of the composite and the maximum stress decreased with respect to the neat matrix. Nevertheless, near to the fracture point, the specimens tended to form a fracture with a bi-pyramidal shape, and the fibers had a "bond" effect that increased the ductility of the material. In this case, the lower stiffness of the fibers from the kraft pulp led to higher deformation values compared to the composites with cotton linters. On the other hand, it must be noted that the cotton linter fibers used were shorter and had a lower WRV than the kraft pulp fibers, leading to the formation of composites with lower matrix porosity (as could be concluded from the lower water/cement ratios of these composites).

3.2.1. Flexural texts

Bending tests are the most useful for showing the reinforcing effects of the fibers with respect to the matrix in cementitious composites. Thus such mechanical tests could be very useful for analyzing the effect of the hornification of the fibers on the mechanical performance and durability of the composites.

As shown in Fig. 5a, the composites reinforced with the hornificated fibers had a flexural strength of between 8% (kraft pulp) and 16% (cotton linters) higher than the composites prepared with the untreated fibers. The elastic modulus followed the same tendency (Fig. 5b). In a similar way, the fracture energy (Fig. 5c) was between 20% (kraft pulp) and 5% (cotton linters) higher in the composites prepared with the hornificated fibers. As expected, there was a loss of resistance in the composites after aging. Nevertheless, while those containing hornificated fibers maintained their post cracking strength and resistance, the composites prepared with the untreated fibers showed a similar behavior to that of mortar without any fiber reinforcement, it is to say cracks after reaching the maximum stress.

Although there were no significant differences between the flexural strength of the composites prepared with kraft pulp or cotton linters, the fracture energy was about five times higher for the composites reinforced with kraft pulp fibers. These results could be explained by the higher ductility of the kraft pulp fibers with respect to the cotton linters. Thus, as shown in Table 2, kraft pulp fibers had higher WRV values and a lower crystallinity index and, consequently, were more flexible. Moreover, the higher aspect ratio of these fibers led to an increase in adherence and consequently to a higher fracture energy in the composites.

On the other hand, although as mentioned previously the absolute values of flexural strength and the fracture energy were higher for the composites reinforced with kraft pulp, after aging the specimens, the loss of resistance was also considerably higher for these composites. This phenomenon could be related to the higher loss of durability of the kraft pulps with respect to cotton linter fibers in the cementitious matrix due to the differences in the morphology

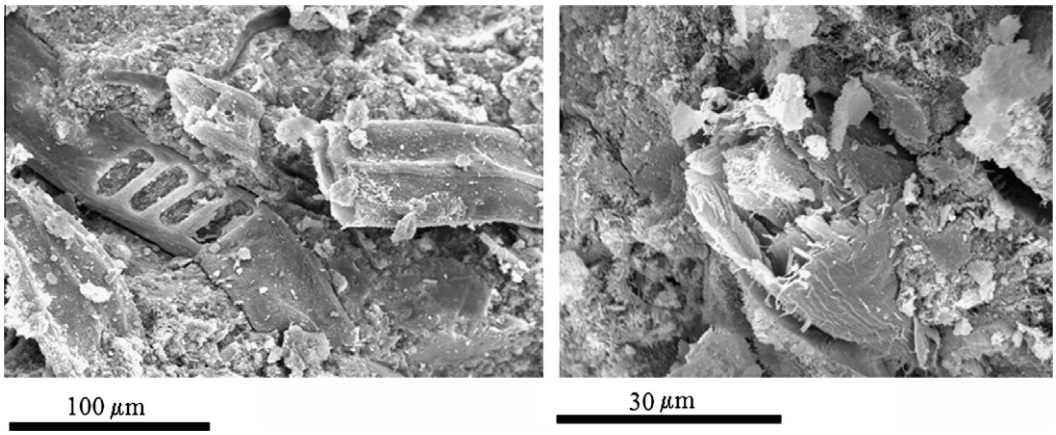

Fig. 6. SEM micrographs of the fracture surface of the aged composites reinforced with kraft pulp: (a) micrograph showing the presence of the cement hydration compounds on the surface and lumen of the softwood pulp fibers, (b) micrograph of a broken fiber showing the presence of the cement hydration compounds in the lumen.

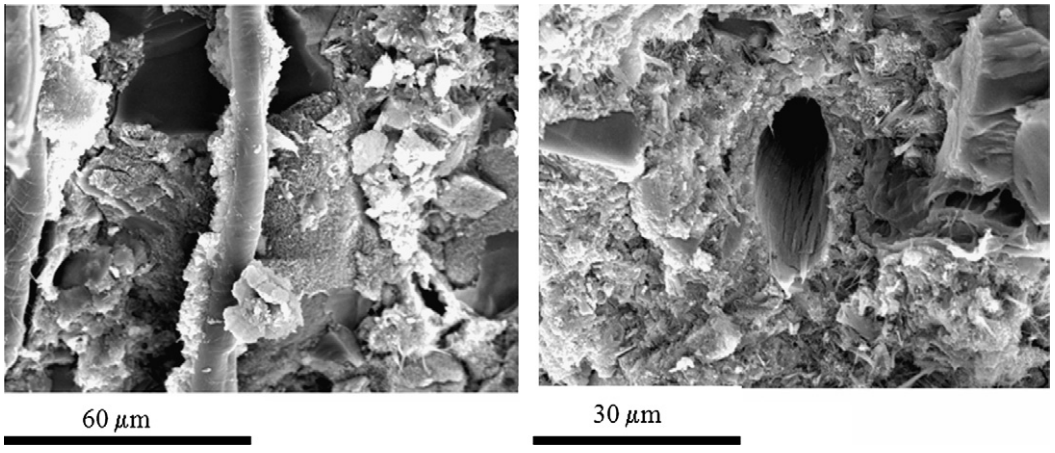

Fig. 7. SEM micrographs of the fracture surface of the aged composites reinforced with cotton linters: (a) micrograph showing the presence of the cement hydration compounds on the surface of the cotton linter fibers, and (b) micrograph showing the absence of the cement hydration compounds in the lumen of the broken cotton linter fibers.

and WRV between these fibers. Thus, as a consequence of the higher WRV of the kraft pulp fibers these fibers would be expected to have a lower dimensional stability and higher loss of adherence with the matrix. On the other hand the morphology of the kraft pulp, with pits on its surface, could favor the precipitation of hydration products of the cement in the lumen of the fibers and, consequently, facilitate the degradation of the fibers in the cementitious matrix. These hypotheses were confirmed by SEM observations of the fracture surface of the composites. As shown in Fig. 6 the fibers of kraft pulp in the aged composites were saturated with the precipitation products of the cement, mainly in the zones near the pits. These products on the surface and in the lumen of the fibers cause degradation of the fibers, which lose their reinforcement capacity. A similar effect occurred in the cotton linters (Fig. 7). However, these fibers did not contain precipitation products in the lumen due to the lower permeability of their surface. Moreover, as shown in Fig. 7 (right), the fibers were less damaged than those in the kraft pulp.

3.3. Compressive tests

In the compression tests, the matrix has greater importance than the fibers and thus the reinforcement effects are less evident. In agreement with the flexible tests, as shown in Fig. 8a, the hornificated fibers showed higher resistance than the untreated ones with increases of between 7% and 10% for the kraft pulp and cotton

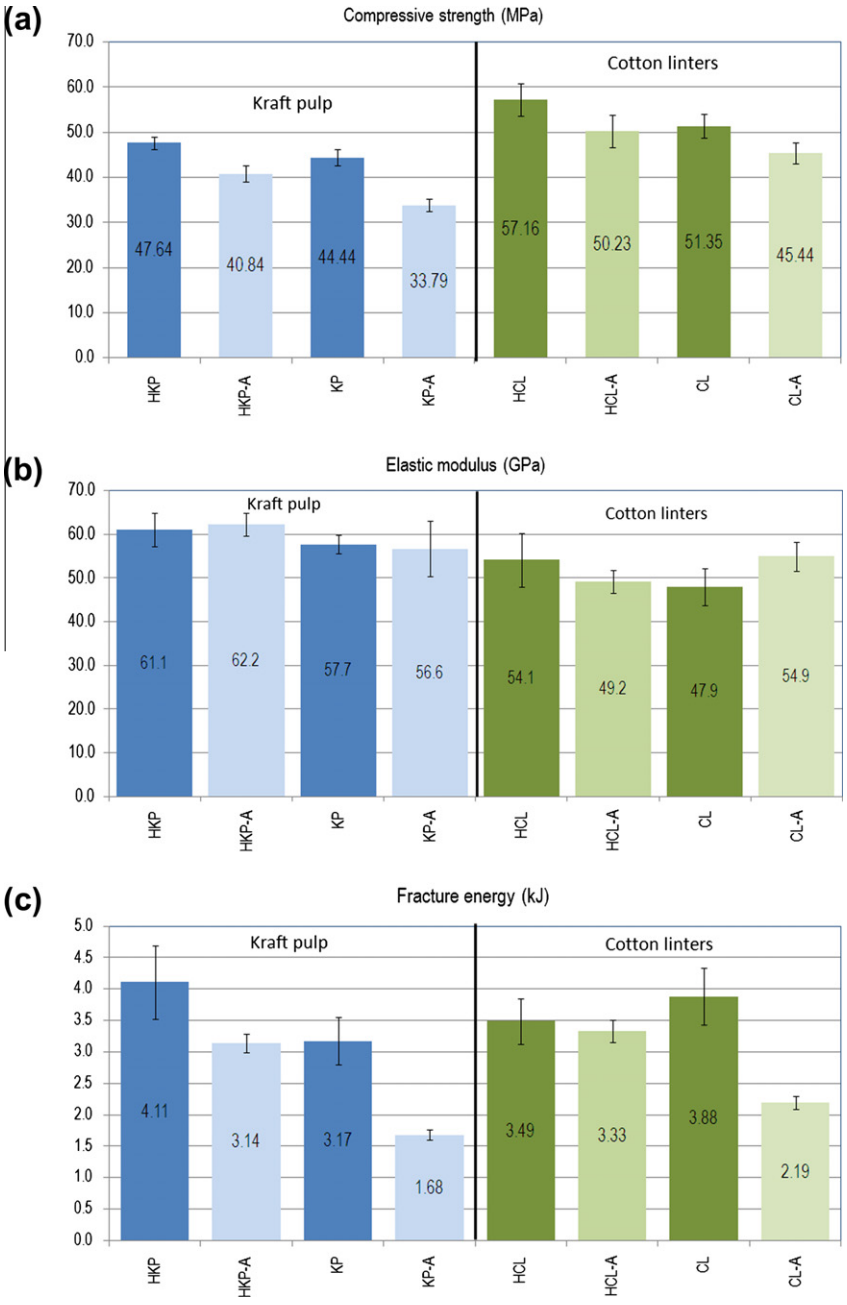

Fig. 8. Compressive test results for the composites prepared with the kraft pulp (left) and cotton linters (right). The light colors correspond to the aged specimens: (a) average values of the maximum strength, (b) average values of the elastic modulus, and (c) average values of the fracture energy.

linters respectively. After aging, the loss of resistance of the composites showed a similar tendency to that observed in flexural tests. The elastic modulus followed the same tendency (Fig. 8b).

With respect to the fracture energy (Fig. 8c), the values were always higher for the composites reinforced with the hornificated fibers than those with untreated fibers. In this sense, after aging, there was 24% decrease in the fracture energy values in the composite with the hornificated kraft pulp versus a 47% decrease in that with untreated fibers and a 5% decrease in the composite reinforced with the hornificated cotton linters versus a 44% decrease in that with untreated fibers. These values confirm the beneficial effect of the treatment of the fibers on the reinforcement effect and durability of the composites.

4. Conclusions

The main conclusion of this study is that a simple and environmentally friendly method (the hornification process) can be used to improve the durability of mortars reinforced with cellulosic fibers.

The particular conclusions are as follows:

1. The prior hornification of the fibers of kraft pulp and cotton linters improved the durability of cement mortar composites although it did not prevent the partial loss of their mechanical reinforcement.
2. The prior hornification of the fibers of kraft pulp and cotton linters improved the mechanical performance of their composites, with around 8% (kraft) and 16% (cotton linters) higher values in flexural tests and around 7% (kraft) and 10% (linter) higher values in compressive tests with respect to the untreated fibers.
3. The prior hornification of the fibers of kraft pulp and cotton linters improved the mechanical performance of their aged composites, with around 13% (kraft) and 21% (cotton linters) higher values of flexural strength and around 20% (kraft) and 10% (linter) higher values of compressive strength with respect to the untreated fibers.
4. Reinforcement with kraft pulp led to cement mortar composites with more than a fivefold higher flexural fracture energy than those reinforced with cotton linter pulp. Kraft pulps are very effective fibers for cementitious composites that work under bending forces.
5. The lower permeability of the fibers of cotton linters resulted in lower degradation of the fibers and, as a consequence, less loss of resistance in the aged composites. The permeability of the kraft pulp fibers, with pits on their surface, facilitated the degradation in the interior of the fibers and, as a consequence, the loss of resistance was higher than that in the cotton linter fibers.

This study demonstrates the beneficial effects of the prior hornification of the fibers as a simple and environmentally friendly method of improving the durability of mortars reinforced with fibers from kraft pulp and cotton linters.

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