NANOFIBRILLATED CELLULOSE (NFC) AS A POTENTIAL REINFORCEMENT FOR HIGH PERFORMANCE CEMENT MORTAR COMPOSITES

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In this work, nanofibrillated cellulose (NFC) has been evaluated as a potential reinforcement for cement mortar composites. Two types of vegetable fibres with different composition and properties (cellulose content and microfibrillar angle), sisal, and cotton linters pulps, were initially characterised in order to assess their reinforcing capability. Sisal pulp was found to be most suitable as reinforcement for the brittle cementitious matrix. Nanofibrillated cellulose was produced by the application of a high intensity refining process of the sisal pulp. It was found that 6 hours of refining time was required to obtain the desired nanofibrillation of the fibers. Cement mortar composites reinforced with both the sisal fibres and the nanofibrillated cellulose were prepared, and the mechanical properties were determined under flexural tests. The cement mortar composites reinforced with the nanofibrillated cellulose exhibited enhanced flexural properties, but lower values of fracture energy, than the ones reinforced with the conventional sisal fibres.

Keywords: Sisal and cotton fibres; Nanofibrillated cellulose; Cement mortar composites; Mechanical performance

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INTRODUCTION

Over the last few years, a number of researchers have been involved in investigating the exploitation of natural fibres as load-bearing constituents in composite materials (Ardanuy *et al.* 2012; Beckermann and Pickering 2008; Garkhail *et al.* 2000; Heijenrath and Peijs 1996; John and Thomas 2008; Singleton *et al.* 2003; Stamboulis *et al.* 2000). The use of natural fibres as reinforcements is increasing due to their cheapness, their ability to be recycled, and their competitive mechanical properties. The combination of interesting mechanical and physical properties of natural fibres and their environmental benefits have been the main drivers for their use as alternatives for conventional reinforcements, such as steel and harmful asbestos in cement composites (Asasutjarit *et al.* 2007; Pandey *et al.* 2010). Vegetable fibre reinforced cement mortar composites (VFRCMC) constitute a very interesting option for the building industry, mainly in less developed countries or countries that need low-cost constructions (Claramunt *et al.* 2010; d'Almeida *et al.* 2010; Savastano *et al.* 2003a).

So far, most vegetable fibres used in composite applications are fibre bundles, despite their relative low strength of around 600 MPa when compared with the actual fibre cell (up to 1500 MPa of strength). Therefore, even higher intrinsic strengths would be foreseen when nanofibrils are used as reinforcement (Berglund and Peijs 2010; Stamboulis *et al.* 2001).

The production of nano-scale cellulose reinforcing materials and their application in composites materials have gained increasing attention due to their unique characteristics, such as very large surface to volume ratio and good mechanical properties, including a high Young's modulus, high tensile strength, and a very low coefficient of thermal expansion (Kaushik and Singh 2011).

Most applications of these reinforcements have been in reinforcing polymer matrices (Eichhorn *et al.* 2009; Siro and Plackett 2010) but, to our knowledge, they have never been applied in cement mortar matrices. Published works on the field of VFRCMC mainly describe the use of vegetable fibres with several millimetres in length (2 to 10 mm) and diameters ranging between 10 and 30 μm, such as wood pulps, sisal and abaca fibres, or cotton linters (Ardanuy *et al.* 2011; Asasutjarit *et al.* 2007; Claramunt *et al.* 2011; d'Almeida *et al.* 2010; Pacheco-Torgal and Jalali 2011; Ramakrishna and Sundararajan 2005; Silva *et al.* 2009). The use of pulp fibres in comparison to fibres in strand form could result in better fibre–matrix bond, greater reinforcing efficiency and significant increase in flexural strength and toughness (Coutts 2005; Karade 2010; Savastano *et al.* 2003b).

The aim of this work is to evaluate the capacity of nanofibrillated cellulose as a potential reinforcement to produce high-performance ductile cement mortar composites. For this purpose, composites reinforced with nanofibrillated cellulose will be assessed and compared with those reinforced with conventional cellulose fibres. The relationship between the size and extent of fibrillation of the fibres and the mechanical performance of the resulting composites will be established based on the crack-bridging law in composite materials.

MATERIALS AND EXPERIMENTAL DETAILS

Materials

UNE-EN 197-1:2000 Type I cement was used in this research. The sand used was provided 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 for 5 min to achieve a maximum particle size of 0.1 mm. Sika Viscocrete-3425 fluidizer, obtained from Sika S.A.U., was used at a maximum dosage rate of 40g/1000g of cement to aid workability.

Two kinds of vegetable fibres with different composition and properties were used for the production of these cellulose based cement mortar composites. Cotton linters were kindly supplied by Celsur (Cotton South, S.L., Spain) and sisal (*Agave sisalana*) pulp, from a soda-anthraquinone cooking process was kindly supplied by CELESA (Spain).

Fibre Treatment

Nanofibrillated cellulose was prepared by the application of a high intensity refining process in a Valley Beater. Following the ISO 5264-1:1979, 360 g of oven-dried sisal pulp were added to deionised water in such a way as to give a final volume of 23 L, corresponding to a consistency of 1.57% (m/m). The mixture was placed at the Valley Beater device, where the cutting and fibrillation of the sisal fibres took place due to the mechanical action. The extent of fibrillation of sisal fibres was studied for different refining times of 1, 2, 3, 4, 5, and 6 h.

Fibre Characterisation

An initial characterisation of sisal and cotton linters pulp was performed. The fibre dimensions such as length, width, linear mass (coarseness), and curl index were measured according to TAPPI T271 om-02 by using a Kajaani FS300 Analyzer. Measurements were taken from ~5000 fibres. In order to study the intrinsic mechanical resistance of the sisal and cotton fibres, the zero-span tensile index was also determined by using a Pulmac tester. According to ISO 15361:2000, the zero-span tensile index (Z_I) was calculated from Equation 1,

$$Z_I = \frac{Z_T}{G} \quad (1)$$

where Z_I is the zero-span tensile index (kN.m/g), Z_T is the zero-span tensile strength (kN/m), and G is the oven-dry grammage (g/m^2).

In order to perform the zero-span test, homogenous isotropic sheets of sisal and cotton pulps with a grammage of about 60g/m^2 were produced. These sheets were cut into strips and tested according to ISO 15361:2000 (E).

The microstructure and morphology of the initial pulps, and those obtained after different refining times, were analysed by scanning electron microscopy (SEM), using a JEOL JSM-S610 microscope at an accelerating voltage of 10 kV. Prior to examination, a little amount of pulp was diluted in deionised water, and an aliquot of this suspension was dropped on a metallic support surface and dried in an oven for 12 h at 80°C. Finally, the dry pulp surface was sputtered with a thin layer of gold to make them conductive.

X-ray diffraction patterns of the initial pulps were obtained by using a powder X-ray diffractometer at 45kV, 40 mA, and CuKα radiation. The measurements were performed in the 2θ angle range between 5 and 50° with a step size of 0.04° and a time step of 1 s. The sample crystallinity, X_{cr} (%), defined as the ratio of the amount of crystalline cellulose (cellulose I_β) to the total amount of sample material (Thygesen *et al.* 2005), was determined by the Segal method (Equation 2) (Segal 1959), using the intensity of the (200) reflection (I_{200}) and the minimum between the (200) and the (110) reflections (I_{AM}). I_{200} represents both crystalline and non-crystalline material while I_{AM} represents only the fraction of non-crystalline material.

$$X_{cr} = \frac{(I_{200} - I_{AM})}{I_{200}} \quad (2)$$

Composite Preparation

In order to study and compare the reinforcing effect provided by the incorporation of the cellulose fibres and nanofibrillated cellulose in the cementitious matrix, three composites of each type of reinforcement were prepared. The ratio cement:sand:water used was 1:1:0.46 for the cellulose fibres and 1:1:0.67 for the nanofibrillated cellulose, and the reinforcement amount was fixed at 3.3wt.%. This wt.% was the maximum quantity, which allowed for the preparing of the homogeneous mixtures. The composites were prepared following the same procedure described previously (Claramunt *et al.* 2011).

Prismatic specimens of the composites were prepared for measurement of the flexural test. The mould used was UNE-EN 196-1:2005 type with internal dimensions of 40x40x160 mm^3 modified to allow the compression of the specimens to 20 mm of thickness. This modification was also useful for removing the excess water and increasing the compaction of the material (Toledo Filho *et al.* 2009). The modification performed to the mould involved adding three lids provided with easels and a lid that covers the mould and allows the homogeneous distribution of the load. 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 4 MPa for 24 hours. 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 0.8 and 1 MPa were performed to reach the maximum pressure.

After demoulding, the specimens were cured for 28 days in a curing box at 20 ± 1°C and 95% relative humidity. Six specimens of each composite were prepared for flexural test.

According to UNE-EN 196-1:2005, three point bending tests were performed using an Incotecnic press equipped with a maximum load cell of 3kN. The load speed was 50 ± 10 N/s. At least six specimens of each sample were tested, and the values were averaged.

RESULTS AND DISCUSSION

Physical and Morphological Characterisation of the Cellulose Fibres

The reinforcing capability of fibres is governed by parameters such as fibre type, length, aspect ratio, and strength. In order to select the most suitable type of fibre as reinforcement in brittle cement matrices, an initial determination of these parameters was carried out. Table 1 shows the results of the physical and morphological characterisation of the sisal and cotton pulps as received.

As shown in Table 1, sisal fibres are longer but narrower than cotton linters fibres, resulting in a much higher aspect ratio and, therefore, expectable better reinforcing capability. Figure 1 shows the SEM pictures of these fibres at different magnification, where it can be clearly observed that the sisal fibres have a smaller diameter and curl

index than the cotton fibres, which confirms the measurements obtained by Kajaani. The ratio of fines (fibres shorter than 20 µm) present in sisal pulp is around four times lower than the one of cotton linters, which suggests that sisal pulp shows a higher homogeneity in length. The higher the fines ratio in the pulp, the lower the amount of fibres that remained with enough length to provide reinforcement, since fines do not play a significant role as reinforcement due to their short length. In fact, fines can be considered as fillers based on their reinforcing effect. With regard to the fibre intrinsic strength measured by the zero-span tensile index, no differences were found for both types of fibres. The XRD patterns of both conventional sisal and cotton pulps are shown in Fig. 2.

Table 1. Physical and Morphological Characterisation of the Initial Sisal and Cotton Pulps

	Length mm	Width µm	Aspect Ratio (length/ width)	Curl index (%)	Fines (%)	Zero-span tensile index (KNm/g)	Crystallinity (%)
Sisal fibres	1.14	15.96	71.42	26.2	7.89	0.13±0.01	87.2
Cotton linters	0.85	20.15	42.18	31	28.71	0.13±0.02	91.8

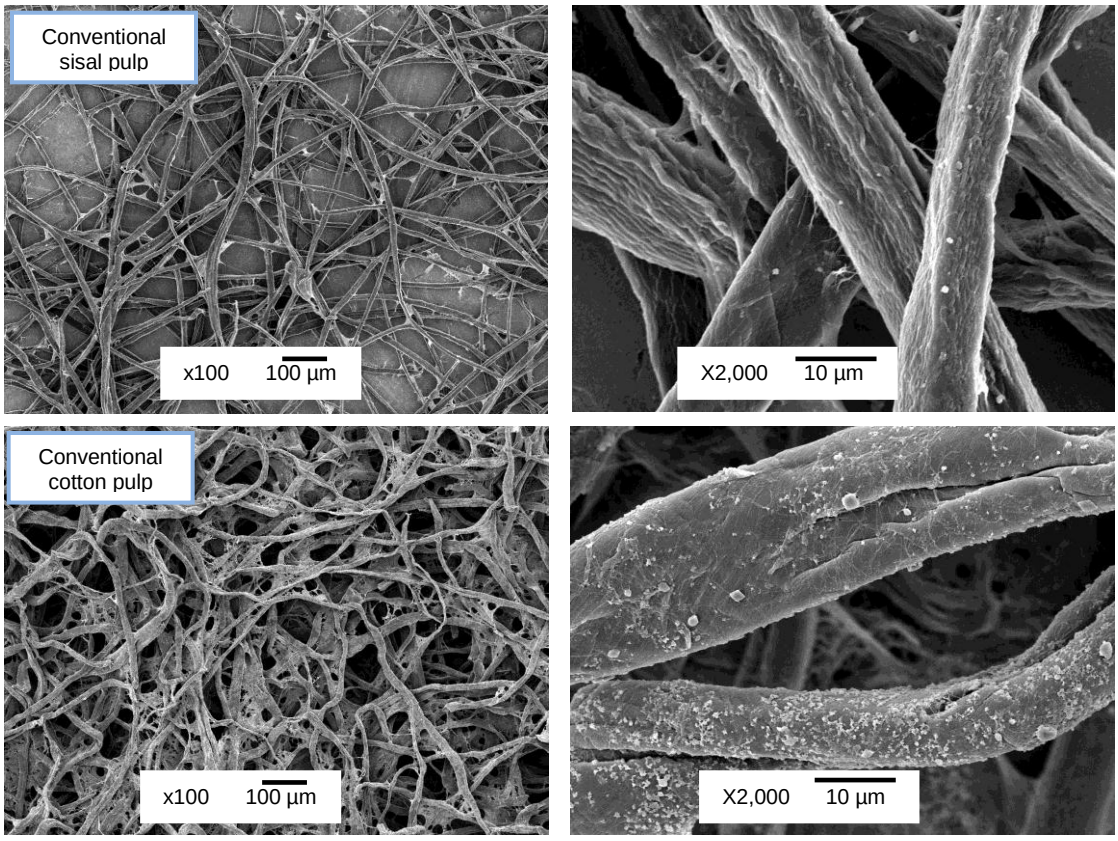

Fig. 1. SEM images at different magnification of conventional sisal pulp (1st row) and cotton linters (2nd row)

The intensity of the reflections corresponding to the crystalline phase of cellulose I ((1-10), (111), (200), and (004)) was slightly higher for cotton, whilst the intensity of the reflection at 21° related to the amorphous material was lower than that of sisal pulp, resulting in a slightly higher crystallinity in the cotton linters (Table 1).

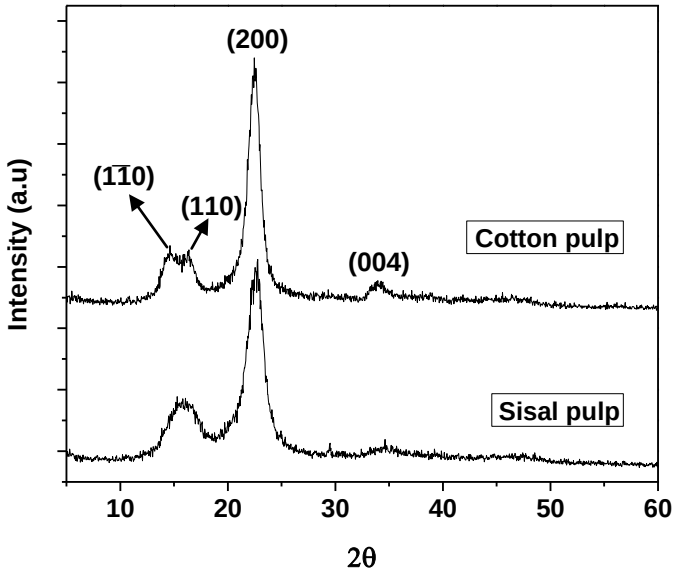

Fig. 2. X-ray diffraction patterns of initial sisal and cotton linters pulps

Taking into account that the crystallinity and the fibre intrinsic strength are rather similar for both types of fibres, the different reinforcing capability is determined by the remaining parameters such as length, aspect ratio, and fines ratio. Thereby, it is expected that sisal fibres will exhibit greater load bearing and crack bridging capabilities due to their low fines content and the high aspect ratio and length of these fibres. Hence, sisal fibres were selected for the preparation of the VFRCMC. Hereafter, allusions to cotton pulp will not be made in the next sections, since this pulp was abandoned for the composite production due to its low reinforcing capability.

The Effect of the Refining Process on the Fibre Microstructure

With the aim of determining the refining time at which cellulose nanofibrils are obtained, the effect of the mechanical treatment on the fibre microstructure was studied by SEM. Figure 3 shows the microstructure of the initial sisal fibres (first row), and of the pulps obtained after 1 (second row), 3 (third row), 5 (fourth row), and 6 (sixth row) hours of refining at low (left) and high (right) magnifications. As previously mentioned, the conventional sisal pulp consists of fibres with a diameter ranging from 10 to 20 μm. After 1 hour of refinement, the fibres remain almost intact, although an initial external fibrillation starts to appear, after which a progressive fibrillation of the fibres can be observed. Therefore, the cutting process was predominant over the fibrillation process for the first refining hour. After 3 hours of refinement, the external fibrillation of the fibres can be clearly observed. An increase of the refining time to 4 hours involves an enhancement of the extent of fibrillation of the fibres.

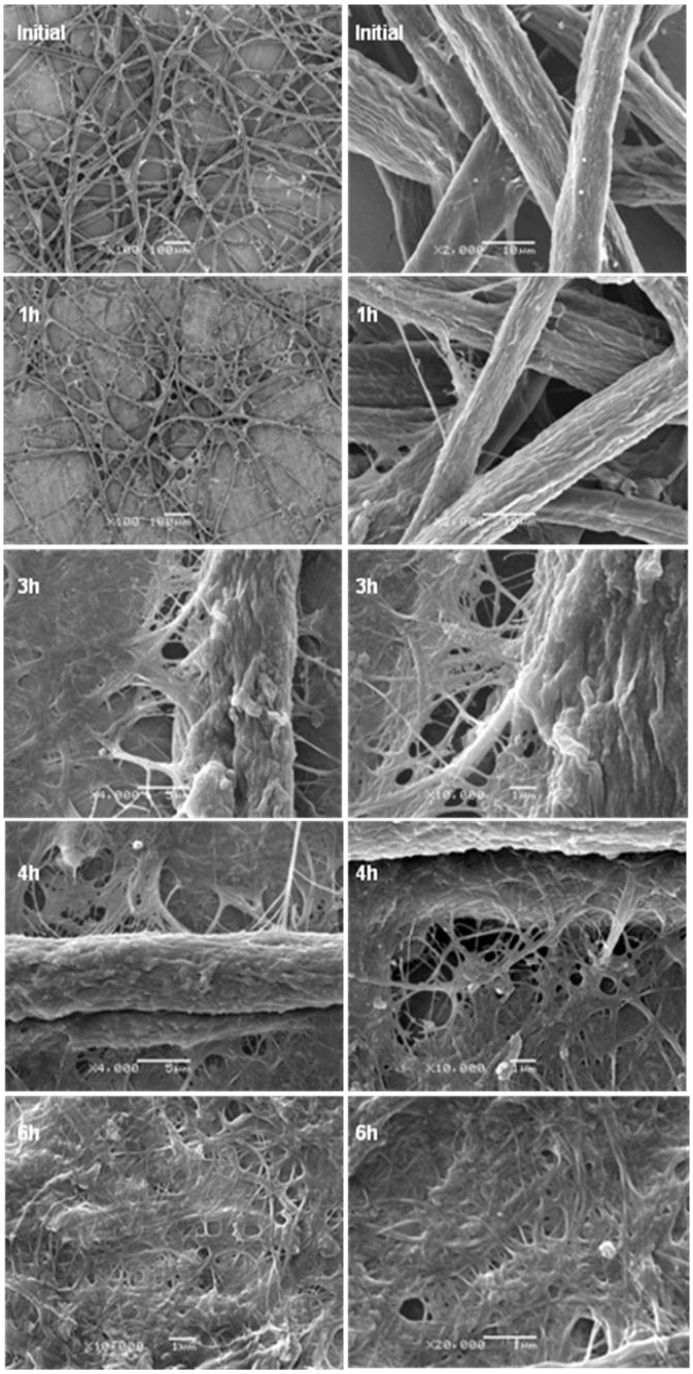

Fig. 3. SEM micrographs of the initial sisal pulp and after different refinement times at low (left) and high (right) magnifications

The initial fibre diameter was reduced to *ca.* 5 μm as a consequence of the fibrillation of the outer layers that creates branches in the fibre, leading to the formation of nanofibrils, increasing in this way the fibre specific surface area. Further refinement (6 hours, bottom) yields highly branched fibres in the nanometer scale, between 25 and 250 nm. These nanofibrils could present a higher intrinsic strength than the initial fibres. This fact, together with the increase of the aspect-ratio, notably enhances the reinforcing

capabilities of these pulps, making them potentially suitable for the production of composites. Moreover, their high specific surface area would potentially favour the interaction with the matrix and therefore more effective fibre-matrix bonding, giving place to composites with enhanced matrix to fibre stress transfer. Taking into account all these issues, the optimum refining time was found at 6 hours, and this nanofibrillated pulp was selected for the preparation of the nanoVFRCMC.

The Effect of Using Cellulose Fibres and Nanofibrillated Cellulose as Reinforcement on the Mechanical Performance of Cement Composites

The mechanical properties obtained from the flexural curves (Fig. 4) of the cement mortar composites reinforced with both, the sisal microfibres and the nanofibrillated cellulose, are shown in Table 2.

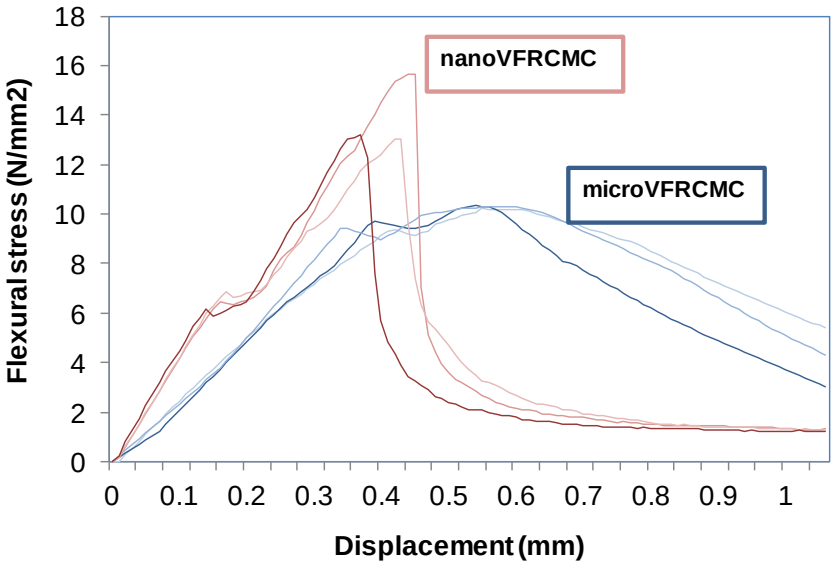

Fig. 4. Flexural stress-displacement curves of the cement composites reinforced with nanofibrillated cellulose compared with cellulose fibres

Table 2. Mechanical Properties of Cement Mortar Composites

Composites	Flexural Modulus GPa	Flexural Strength MPa	Fracture Energy J
Cellulose sisal fibres reinforced cement mortar composites	2.4 ± 0.1	10.3 ± 0.06	759 ± 78
Nanofibrillated cellulose reinforced cement mortar composites	4.1 ± 0.1	14.0 ± 1.5	357 ± 48

A clear improvement of both the flexural modulus and strength can be observed for the composites reinforced with nanofibrillated cellulose compared to those reinforced with the cellulose fibres. As previously mentioned, the reinforcing effect of nanofibrils

was higher than the fibres, since the nanofibrils have higher intrinsic strength and stiffness. However, nanoVFRCMC exhibited a brittle behaviour, as can be easily deduced by the little plastic area in the stress-displacement curve (Fig. 4) and confirmed by the low value of fracture energy. This brittle failure can be attributed to the low crack bridging capacity of the nanofibrils, since their short length is not enough to prevent the growth of the matrix microcracks. Besides the fibre length, the composite toughness is mainly governed by the fibre-matrix bonding. The interfacial bond between the fibre and the matrix needs to be optimized rather than maximized, particularly in vegetable fibre, reinforced composites, which often suffer from a lack of toughness as a consequence of excessively strong fibre-matrix bonding (Berglund and Peijs 2010). The high energy-absorption mechanisms involved in the composite fracture, such as debonding and fibre pullout, are hindered in favour of fibre rupture when interfacial properties are maximized, bringing about a brittle fracture and therefore, low fracture energy. This general relationship between toughness and fibre-matrix bonding could be the reason for the brittle behaviour of the nanoVFRCMC. As previously mentioned, the high specific surface area of the nanofibrils provides an enhanced fibre-matrix interaction as well as an increase of cellulose hydroxyl groups available to hydrogen bond with the cementitious matrix (Coutts 2005), hence an excessively strong bonding is expected to form between the nanofibrils and the matrix, leading to an embrittlement of the composite. Unlike the composites reinforced with nanofibrillated cellulose, the microVFRCMC showed a more plastic behaviour, since the long fibres were more effective in bridging the crack faces. In addition, the interfacial properties of these composites were weaker as a consequence of the low fibre specific surface area, favouring the toughening by debonding and fibre pullout.

Taking into account the mechanical performance of both cement mortar composites, it is proposed that the ideal reinforcement of brittle matrices such as cement mortars would be a combination of both types of cellulose fibres, at the nano- and micrometric level, to match the good flexural properties provided by the nanofibrils (modulus and strength) with the high toughness rendered by the cellulose fibres.

CONCLUSIONS

1. The obtaining of nanofibrillated cellulose was successfully achieved through a simple and chemical-free mechanical treatment, which consists of the application of a high energy refining process.
2. NanoVFRCMC exhibited enhanced flexural properties compared to the conventional VFRCMC, but they showed a brittle behaviour, since the nanofibrils contributed low capability to bridge incipient cracks bridging as a result of their small size. In addition, the highly nanofibrillated cellulose specific surface area results in an enhanced of fibre-matrix interaction, leading to an excessively strong fibre-matrix bonding, which involves a better stress transfer from the matrix to the nanofibres, but, in turn, an embrittlement of the composite.

3. An optimisation of the refining time is required to obtain long nanofibrillated cellulose fibres to improve the ductility while keeping the flexural properties of the nanoVFRCMC.

ACKNOWLEDGMENTS

The authors would like to acknowledge MICINN (Government of Spain) for the financial support of the projects BIA2011-26288 and CTQ2010-20238-CO3-01. The authors wish also to thank to Dr. Fernando Conde from Universidad Complutense de Madrid for the XRD measurements.

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Article submitted: December 10, 2011; Peer review completed: June 7, 2012; Revised version received and accepted: July 5, 2012; Published: July 9, 2012.