



A NOVEL PULL-OUT DEVICE USED TO STUDY THE INFLUENCE OF PRESSURE DURING PROCESSING OF CEMENT-BASED MATERIAL REINFORCED WITH COIR

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Abstract

This paper deals with the use of plant fibres, such as coconut, as reinforcement in cementitious materials; such additions modify the mechanical properties of the composite in its hardened state. This enhancement depends on both the fibre's physical and chemical properties. For reinforcement of the mineral matrix, the interfacial properties are paramount in ensuring fibre effectiveness. Fibre reinforced cementitious material can also be enhanced by processing, such as extrusion, that will improve the interfacial bond by the application of pressure.

An original experimental procedure has been carried out to study the influence of cementitious matrix consolidation pressure on interfacial bond strength; samples with a coir bundle were made to be submitted to pull-out tests from the hardened paste. Results show that the fibre-matrix adhesion can be controlled by matrix consolidation in the fresh state. It suggests that high-pressure processing such as extrusion can improve the mechanical behaviour of the interfacial bond between coir and cementitious matrix leading to better composites mechanical properties. Therefore, forming process requires careful analysis in order to optimize the reinforcement ability of the natural fibre.

Keywords:

coir reinforcement, cementitious material, fibre pull-out, interfacial bond, extrusion

1 INTRODUCTION

Over the last decade, plant fibre reinforced cement-based materials have received increasing attention. Compared with synthetic fibres, plant fibres can provide a significant reduction in processing costs and have many environmental advantages: biodegradability, renewability, and a favourable life cycle assessment (LCA) [Le Duigou 11]. Plant fibres are widely available in most developing countries and often derive from agricultural wastes, and therefore could be considered as a potentially useful local material. Among natural fibres, coir is a low cost fibre [Dicker 14] extracted from the tissues surrounding the seed of the coconut palm (*Cocos nucifera*). The coir's natural function is to protect the nut from breaking, by absorbing the shock when the coconut falls, and from rotting by reducing water penetration. Coir is also the only natural fibre resistant to salt-water exposure. These properties are achieved by means of the complex multilayer microstructure and biochemical composition of coir. The main producers of coconut palms in the world are India, Sri Lanka, Brazil and Southeast Asia. In 2009, worldwide, coir production was of the order of 500 000 tons per year [Ali 13].

The biochemical composition of coconut fibre depends on its origin and maturity (brown and green coir). Coir is composed of around 45% cellulose, which is half that of other commonly used bast fibres such as flax or hemp. Cellulose microfibrils act as reinforcement within the fibre; therefore, the low quantity of cellulose combined with high microfibrillar angle (around 30-50°) induces low stiffness and tensile strength; however, ductile properties could be reached (higher strain at break) [Defoirdt 10, Satvanaravana 82]. A high amount of lignin, between 20 and 59 %, is found in coir; this is combined with a moderate quantity of hemicelluloses: 8-28% [Agopyan 05, Justiz-Smith 08, Hejazi 12]. Cement-based materials are currently reinforced with synthetic fibres (steel, glass and synthetic polymeric fibres) to enhance their mechanical properties, notably their flexural strength and toughness. The principal advantage of synthetic fibres is that they are standardized and chemically inert in a cementitious matrix. To reinforce cement-based materials with plant fibres in place of conventional fibres requires a careful analysis, as vegetal fibres absorb water [Hejazi 12, Ali 11] and could release extractive compounds into the fresh mix. These compounds can affect the setting time [Savastano 99, Savastano 00], prevent cement hydration [Bilba 03,

Stancato 05] and therefore modify the interface area between fibre and matrix in the fresh state and during hardening [Nozahic 12]. Problems such as a set-retarding or even set-inhibiting effect when mixing vegetable particles with mineral binders can create a complex Interfacial Transition Zone (ITZ) between lignocellulosic aggregates or fibres and the binder matrix. This ITZ can alter the efficiency of reinforcement ability. Plant fibres are used in ordinary concretes and in soil reinforcement [Agapyan 05, John 05, Pacheco-Torgal 11]. Careful selection of plant fibres, which takes into account its physico-chemical nature, is highly important and has to follow a biomimetic approach. Coir could therefore be a good candidate to enhance cement toughness, but not suitable for producing high performance structural concrete [Ali 12]. As a comparison, the strength of the interfacial bonds in plant fibre reinforced thermoplastics composites depends on many parameters, including the fibre's surface biochemistry, roughness and residual thermal stress due to differential expansion between fibre and matrix [Baley 06, Le Duigou 14]. In the case of fibre reinforced cementitious composites, Bentur and Mindness [Bentur 07] noted three ways in which fibres can effectively improve the mechanical performance of a brittle matrix: 1) physical and chemical adhesion; 2) friction; 3) mechanical anchoring due to the fibre surface roughness. In addition, they considered that the mechanical properties of cementitious material are significantly improved in the case of low matrix porosity. Low porosity can result from the processing parameters, such as a low water/cement mass ratio ($W/C < 0.3$), casting pressure [Ali 12] or extrusion [Shao 01]. Direct characterization of reinforcement/concrete interface bond strength is a tedious work and requires specific devices such as pull-out tests [Bouazaoui 08, De Lorenzis 02] and a wide range of samples, especially when dealing with natural fibres such as cellulose fibres [Morissey 85] or coconut fibres [Ali 12]. Concerning pull-out experiments on fibre-reinforcements, the samples consisted in beams and the fibre direction was not controlled into the mineral matrix. It makes the data very difficult to interpret. In the other hand, for rope-concrete and steel bar reinforcement, the experimental size is at least one scale higher than for fibre reinforcement. The first purpose of the present paper is to directly characterise the coir fibre/cementitious matrix adhesion and further understand the interfacial phenomena during hardening and at hardened state of the matrix. Second purpose is to evaluate the optimized processing conditions i.e consolidation pressure during casting or extrusion on the interfacial properties of coir fibres/cementitious matrix. An original pull-out device is developed with samples whose consolidation pressure has been controlled.

2 MATERIALS AND METHODS

2.1 Materials

The matrix has been chosen to produce an extrudable paste in its fresh state. The paste has consisted of a mix of cement and kaolin, with a cement/kaolin ratio of 1:1 by weight, and a water/cement ratio of 0.4. This mixture was able to fill the narrow cavities in the purpose made moulding device [Khelifi 13a]. This type of mixture was chosen here for two main reasons: First, kaolin is usually added to cement pastes or

mortar for extrusion process as it provides cohesion in the fresh mix and decreases internal and wall friction inside the extruder. The second reason is to explore the development of cement or lime stabilized clay blocks as low-cost and low-environmental-impact building materials.

The kaolin used was a Powdered Polwhite BB from Imerys ® (Kaolins de Bretagne, Ploemeur, France). The specific gravity of the clay was 2.65, the largest clay grain size approximately 40 μm and mean grain size of the order of 9 μm . The specific area of the kaolin powder was 105 cm^2/g . A Portland cement CEM I 52,5N CE CP2 with a specific gravity of 3150 kg/m^3 was used. The specific surface of this cement, measured using a Blaine apparatus, was 3390 cm^2/g . The mean standard compressive strengths (of a standard mortar) of this cement were high: 28 MPa after 2 days and 63MPa after 28 days of cure.

Compressive and flexural strengths (at 28 days) of the matrix were measured on 40 x 40 x 160 mm^3 samples. The mean flexural strength and compressive strength were 5.9 MPa and 18.8 MPa respectively. So its direct tensile strength can be estimated around 3.8MPa, and its shear strength around 7.5 MPa, using the empirical formula of [Boulekbache 12].

The coir fibres used here came from Indonesia. Two types of fibres were studied: raw fibres from Sumatra Island and manufactured fibres from Western Java Island provided by a car-seat padding manufacturer. The two fibre types were treated in different ways. The treatment of raw fibres was as follows: the coir bales were soaked in water for three days to release compounds and then hackled to extract some of the dust and pith. Manufactured fibres have undergone the same treatment as raw fibres but with a supplementary water cleaning process. Some of the manufactured fibres were also manually rewashed in the laboratory to remove almost all other particles. This latter treatment consisted of immersion of the fibres in tap water for 24 hours and hand washing to remove any residual chemical compounds.

Whatever their origin and treatment, the fibres are organized in bundles of single fibres which allow sufficient length for tensile characterization and pull-out testing. In addition, bundles of individual fibres require only a few manufacturing operations limiting therefore the cost of material. The nomenclature used in this paper is RF for 'raw farmed fibres', MF for 'manufactured fibres' and LF for 'manufactured fibres rewashed in the laboratory'.

2.2 Analysis of coir fibres

Fibres geometry

Tensile strength and interfacial bond strength depends on fibre cross-section and perimeter. However, coir fibres have a section rather elliptic than circular as evidenced by [Nam 11]. In the present study, only the apparent mean diameter of each fibre, which is similar to their thickness, was evaluated using an optical microscope. Three diameters were measured and an average value was computed and used as the mean diameter for each of the 125 fibres that were used for the tensile and pull-out tests. The fibres with a diameter variation of more than 20% by centimetre of length were removed. Therefore, the assumption of a circular cross-section may generate an uncertainty up to four percent in pull out tests and 8% for tensile tests [Charlet 11].

Tensile characterization of fibres

Tensile characterization was performed on 30 different fibres following the protocol described by [Baley 12]. The fibres were glued to a paper frame with a gauge length of 10 mm. The frame was clamped in a universal MTS type tensile-testing machine equipped with a 100 N capacity load cell, and loaded at a constant crosshead-displacement rate of 1 mm/min to rupture. A constant cross-section was assumed for tensile properties estimation.

Fibres water absorption

Fibre water absorption was measured following the method of [Nguyen 09] on hemp shives: a given weight of fibres is placed in a drying oven at 60°C until the stabilization of the weight, corresponding to about 72 hours. 1 gram of fibres is then poured in tap water during 5, 10, 15, 30, 45, 60 minutes and 24 hours. Fibres are drained on a sieve screen to extract the superficial water from the fibres skin. Fibres are then weighted. The absorption ratio is given by the weight gain to dry weight ratio.

Study of the coir influence on cement hydration

To study the effect of fibre extractives in water on cement hydration, the leachate obtained from fibre immersion was studied. In the case of plant fibre reinforced cementitious composites, extractives are a major concern as they may modify the cement hydration. Based on a previous work [Bourmaud 10], the test method proposed by [Dubois 56] was used to estimate overall sugar quantity using colorimetric measurements inside the water-washing bath of coir fibres. A more indepth analysis of the nature of extractives was carried out using the method of [Blumenkrantz 73]; this gives information on the amount of uronic acids (UA) within the whole sugar leachate. Uronic acid is typical of pectins polysaccharide.

Then, the Vicat test, was used to measure the setting time. This test showed directly if cement hydration was delayed or prohibited by the presence of fibres. Two standards mix formulations were studied: one with tap water and the other with the water that have been used to wash the fibres whose sugar analysis have been previously done (24 hours fibre immersion).

2.3 Pull-out test method

Sample preparation

Batches were prepared in a 0.03 m³ capacity mixer pan. Firstly, cement and kaolin were blended 2 minutes at low rotation speed (0.37 s⁻¹ - 140 rpm). Water was then poured and mixed with the dry ingredients. The paste mixing consisted in three steps: 120 s at 0.37 s⁻¹ (140 rpm), 60 s at rest for bowl scraping and 480 s at high speed (0.74 s⁻¹ 280 rpm).

A purpose made device was developed to manufacture the samples for pull-out tests (Fig. 1). It consisted of two drilled polymer plates, the lower plate is the bottom mould with a 1 mm diameter hole where fibre can go through. The top plate, with a 5 mm diameter hole, constitutes the mould cell into which the paste can be poured and pressed. An aluminium punch is used to apply a constant pressure after casting. During the setting up of the mould, the top plate holes were lubricated with a mould release oil to facilitate removing of the specimens. After assembling the two plates, the aluminium punch helps handling the fibres through the holes. The paste was weighted until target value corresponding to the volume of paste equivalent

to a 5 mm thick sample is reached. The fresh paste was then poured into the cell and a load was applied for three minutes whatever the specimens prepared. The applied load varied from 500 kPa to 1000 kPa in increments of 250 kPa. Due to consolidation pressure, loading values controlled the specimen thickness variation. Above a load of 750kPa, this thickness variation was accentuated by extrusion of the paste through the 1 mm-diameter hole. After 24 h in the mould, the device was dismantled and the specimen pulled out using the punch. Specimens were then kept in a chamber with controlled humidity (70%) and temperature (20°C). Fibre pull-out tests were carried out after a cure of 28 days.

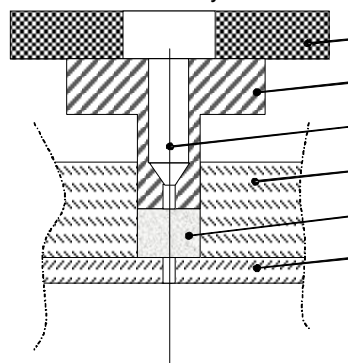


Fig. 1: Specimen manufacturing device

Pull-out tests

Pull-out tests were performed using the same device that was used for the tensile characterization of fibres. The top of the coir fibre was clamped by the jaw in a universal MTS type tensile-testing machine equipped with a 50 N capacity load cell and loaded at a constant crosshead-displacement rate of 1 mm/min. Two blades blocked the matrix and thus interfacial area was sheared and tension was applied to the fibre (Fig. 2).

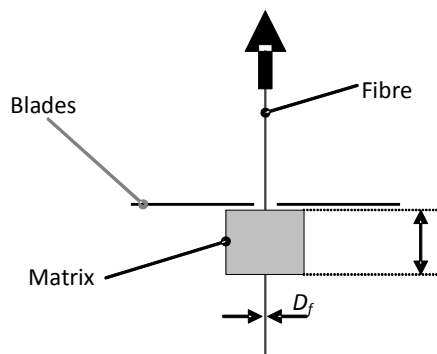


Fig. 2: Pull-out test

At least 16 specimens were made for each type of fibre (RF, MF and LF) and for three applied loads (500 kPa, 750 kPa, 1000 kPa) (1 kg, 1.5 kg, 2 kg). For each valid test (no slipping of the tensile tester jaws), force and displacement were recorded.

The average interfacial bond strength was considered to be the apparent interfacial shear strength (IFSS) using the assumption of a uniform interfacial stress distribution at the fibre/cement interface:

$$IFSS = \frac{F_{bond}}{\pi t D_f} \quad (1)$$

Where F_{bond} is the first maximum force and t is the matrix thickness (Fig. 2). D_f is the average mean fibre

diameter within the cement cylinder as described in 2.2.

3 RESULTS AND INTERPRETATION

3.1 Coir fibre characterisation

Tensile tests

Typical force/ displacement curve of tensile test of coir fibre is shown in figure 3. Before reaching a threshold strain of 0.3 %, the fibre behaviour is linear elastic. Then, loss of linearity with an associated high failure strain is observed. This tensile behaviour could be directly linked to the microstructure composition, organization (microfibrillar angle) and internal reorganization during test, as also observed for bast fibre such as flax and hemp fibres [Baley 12, Placet 14].

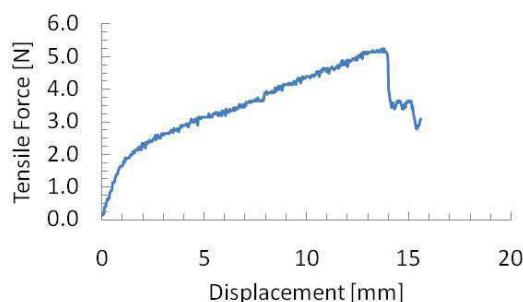


Fig. 3: Typical tensile behaviour of a coir

These high levels of elastic strain can be of great interest when combined with brittle concretes that exhibit poor tensile properties. Indeed, during fracture of the composite, such fibres could bring additional energy dissipation mechanism by both internal reorganization and interfacial friction.

Figure 4 shows the distribution of fibre diameter within the sample population (125 fibres of all types were used in this distribution) plotted in Weibull coordinates. Results show that the diameter is scattered in a range between 131 μm and 430 μm , with a mean value of 298 μm and a standard deviation of 63 μm .

Figure 5 shows the Young's modulus and tensile strength distributions as a function of diameter for 25 fibres of the studied coir fibres. Scatter of mechanical properties with respect to diameter was investigated and it was noted that this trend is typical of natural products [Defoirdt 10, Satvanaravana 82, Agopyan 05, Ali 11, Nam 11, Baley 12]. Assuming a constant cross-section, the tensile strength was of the order of 116 ± 36 MPa and Young's modulus was 4.9 ± 1.3 GPa. These parameters were of the same order regardless of fibre type (RF, MF or LF). These values are in the lower range of those reported in literature, especially the tensile strength: $E_{\text{coir}} = 4\text{-}6$ GPa, $\sigma_{\text{coir}} = 120\text{-}304$ MPa [Defoirdt 10]. Among the 25 samples studied, no trend can be observed concerning the dependency of mechanical behaviour on fibre diameter. The dispersion in geometry and mechanical characteristics due to the plant variability could be a benefit in the case of composite materials, as shown by [Fratzl 07].

Water absorption

Figure 6 shows the water absorption kinetic of the selected coir fibres for this study. One has to keep in mind that the way to evaluate water uptake do not differentiate bonded water onto chemical groups and free water within single and bundles fibres multi-scale

porosity. Thus values need to be taken with caution as a comparison. Coir fibres can absorb twice less water than flax or hemp fibres with similar protocol especially due to their biochemical composition, Indeed, coir fibres have a high quantity of lignin (around 50%) with a low A/B component of surface energy and thus a low affinity with water [Tran 13] is in accordance to their functional role in the nature to prevent rioting. It is an interesting characteristic of this fibre regarding its potential behavior into the cementitious fresh mixture

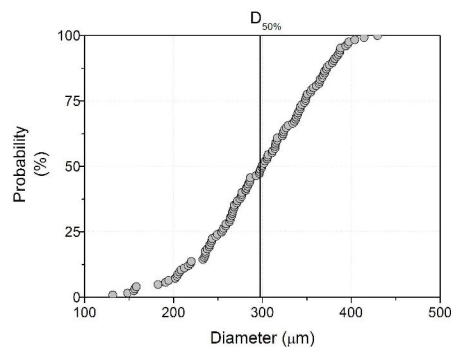


Fig. 4: Diameter distribution for the 125 coir fibres used in this study. The average Diameter D_{50} is plotted in the figure and is equal to 297 μm .

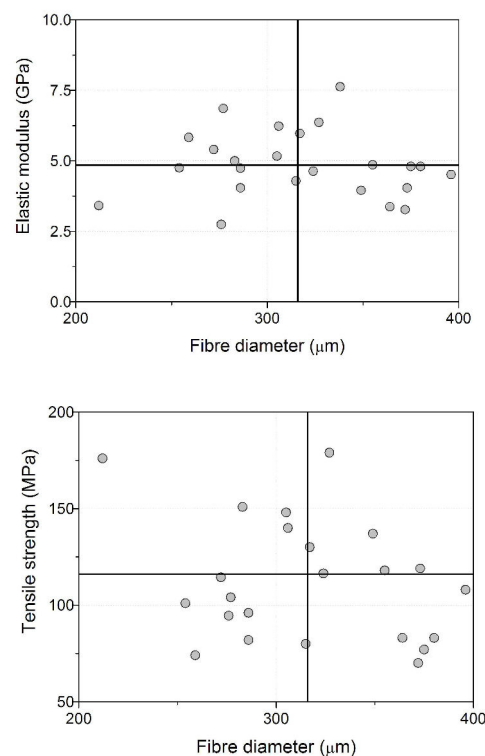


Fig. 5: Longitudinal Elastic modulus and tensile strength versus fibre diameter for 25 coconut fibres; the horizontal and vertical black lines shows the average values of the mechanical properties and the diameter respectively.

Fiber extractives and influence on cement hydration

Total quantity of sugar removed from the coir fibre during washing procedure was measured at $3.2 \cdot 10^{-2} \pm 2.5 \cdot 10^{-3}$ mg/ml. This value is one decade lower than results obtained with a similar weight of flax fibres (24 hours immersion): $2.1 \cdot 10^{-1} \pm 3.7 \cdot 10^{-2}$ mg/ml.

The quantity of Uronic Acid (UA) leachate for coir was $3.62 \cdot 10^{-2} \pm 5.5 \cdot 10^{-3}$ mg/ml which is very close to the total amount of sugar released.

Hence, water washing of coir fibre generated a limited release of sugar compounds. This limited release can be explained again by the water resistant nature of coir; this being the case even for raw fibres that were soaked for three days in water at the end of the farming process. Coir fibres may have a lower inhibiting effect on the cement hydration unlike other plant fibres (flax for example) which contain more extractives.

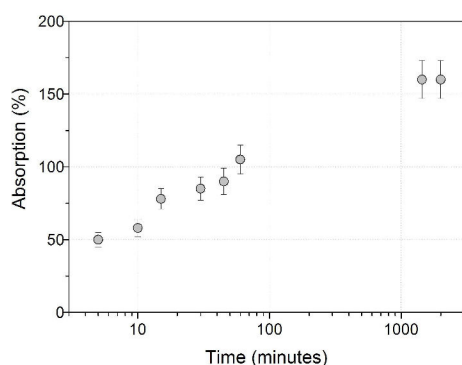


Fig. 6: Water absorption kinetic of the coir.

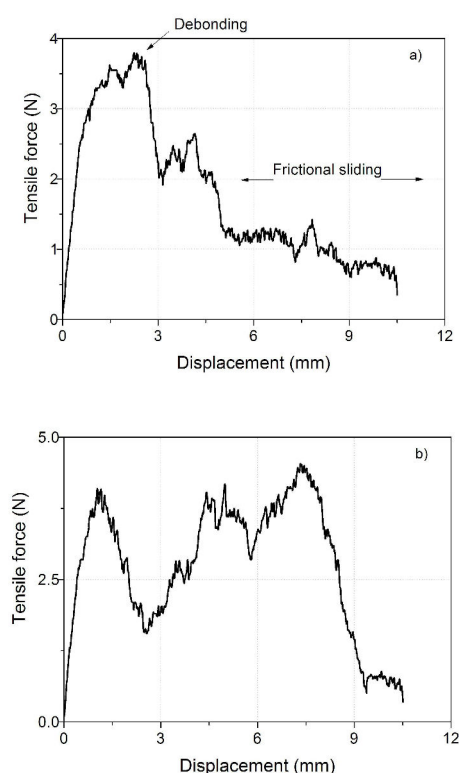


Fig. 7: Displacement-Force curve obtained from pull-out experiments; (a) sliding of the fibre within the matrix after the first peak; (b) tensile force growth after the first peak.

Complementary result with Vicat test have evidenced that no significant difference was found for the setting times between tap water and washing water: an initial setting time of about 220 minutes, and a final setting time of 320 minutes are evaluated for both materials. These observations confirm that coir fibres are, thanks to their low amount a released sugar substances, a

suitable vegetal fibre to improve cement mechanical behaviour (tenacity).

3.2 Pull-out tests

Two kinds of debonding behaviour were observed (Figures 7(a) and 7(b)). Both behaviours have an initial stage where the fibre undergoes linear elastic strain up to a peak that corresponds to the failure of fibre/matrix bonds. The second stage corresponds to the sliding of the fibre within the matrix after the first peak. If the bottom end of the fibre is still within the matrix and if the fibre diameter is constant, tensile force should reach a steady state that corresponds to the solid friction force of the fibre surface on the matrix.

The difference in frictional behaviour observed on figures 7(a) and 7(b) could be explained by variations of fibre perimeter along the fibre length. Figure 7(a) is typical for fibres whose diameter decreases towards its bottom end, while figure 9(b) is typical for fibres with an increasing section, the latter leading to a higher force growth after the first peak. Hence, roughness and section variation of coir fibres could be natural advantages to improve interfacial toughness by providing improved anchorage ability.

Various consolidation pressures, typical for the extrusion process (500 to 1000 kPa), were applied during pull-out sample manufacturing. The results obtained from pull-out tests are shown in Figure 8.

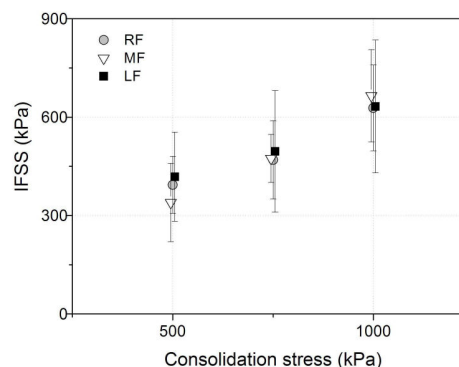


Fig. 8: Evolution of interfacial shear strength as a function of consolidation pressure for the three fibre types: Raw fibres obtained directly from the farm (RF), manufacture fibres (MF) and manufacture fibres rewashed in laboratory (LF)

Interfacial shear strength (IFSS) was computed using Eq.1, as explained section 2.3. The mean IFSS increases almost linearly with the consolidation pressure (coefficient of determination $R^2=0.94$). Mean IFSS varies from 400 to 600 kPa. It is higher than results previously reported in published work on coir pull-out tests [Ali 13], which ranged from 55 to 372 kPa. However, a high scattering is observed, and is due to the variability of fibres shape and tensile behaviour. An adhesion bonding mechanism between coir and cement appears to be driven by the consolidation pressure on the fibre. Manufacturing with an extrusion process instead of casting is therefore advisable in order to optimize the IFSS. During extrusion, fibre may be submitted to a constant and high pressure when the fresh material crosses the die. This pressure is likely to improve the interfacial bond strength and then the mechanical behaviour of the final composites. This result is in agreement with the improvement of ductibility and tensile strength of plastic fibers reinforced cement-composites [Shao 01].

Different physical mechanisms can be involved in the bond strength improvement. One can assume that the consolidation of the cement-based matrix (i.e. liquid and void filtration) due to the load applied during casting is the origin of the increase in the bond strength. However, it has been shown that kaolin/cement pastes are not likely to undergo important liquid filtration during short loading as the one performed during casting [Khelifi 13a]. In the work by Khelifi et al. consolidation phenomenon has significant effect only for a loading time of the order of dozens of minutes. Moreover, no filtrated liquid was flowing outside the sample as observed during extrusion of cement-based materials [Perrot 07, Khelifi 13a]. Therefore, the matrix remains homogeneous during the process and is not considered as responsible for the bonding stress increase.

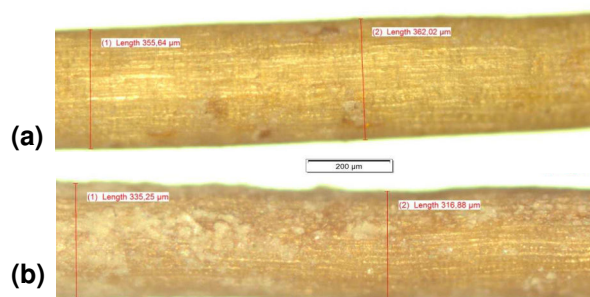


Fig. 9: Surface observation by optical microscopy of two fibres after the pull-out test: (a) fibre with 500kPa of consolidation load; (b) fibre with 1500kPa of consolidation load.

Another phenomenon that can be involved is the improvement of the interfacial zone between fibers and matrix as observed during extrusion of PVA fibre reinforced cement composites [Shao 01]. As observed in Figs. 9(a) and (b), a higher consolidation load induces more residual grains on the extracted fibre and the fibre surface appears to be roughened. Higher adhesion is achieved, which is due to a higher radial pressure on the fibre surface during the consolidation stage and hardening of cement paste. The debonding behaviour appears to be purely interfacial in the case of Fig. 9(a) while it is only partially interfacial in the case of Fig. 9(b). It follows that above a given consolidation state, interfacial cohesion with some mineral particles becomes higher than the cohesion of the matrix. Nevertheless, the de-bonding of some single mineral particles doesn't seem to correspond to the shear collapse of the matrix. As computed in section 2.1. shear strength and direct tensile strength are one order higher than IFSS values observed Figure 8. The results of figure 8 can contribute to the formulation of an analytical model to predict the interfacial behaviour as a function of the stress state in the mineral matrix during consolidation.

The mechanical adhesion can be understood thanks to soil mechanics or powder compression theories. The interfacial bonding at the fibre/matrix interface depends on the radial stress applied during consolidation. Assuming Coulomb's law at the interface the debonding tangential stress can be written as:

$$IFSS = \tau_z = \mu \sigma_{r, residual} + c \quad (2)$$

Where c is the cohesion and μ is the friction coefficient at the coir/matrix interface. $\sigma_{r, residual}$ is the residual radial stress along the fibre due to consolidation during

processing. This consolidation is linked to the pressure applied after casting and it depends on different phenomena: the stress transmission in the matrix during loading, the fibre and cementitious matrix shrinkages during maturation, the creeping of fibre with time.

3.3 Discussion

As shown by eq.2, it is required to evaluate the residual stress acting on the fiber in order to predict the evolution of the IFSS with the consolidation load. This residual stress depends on many parameters such as the consolidation load amplitude, the mechanical behaviour of both the fiber and the matrix and even materials shrinkage and creep.

Stress transmission during consolidation

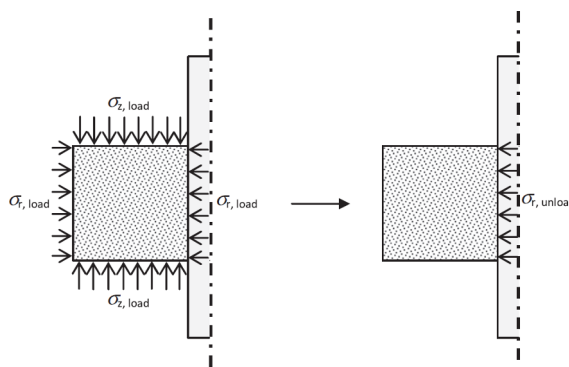


Fig. 10: Stress state at the outer boundaries of the cementitious matrix

Powder and soil mechanics often use a proportionality law between axial stress and radial stress in compression die. According to [Jacky 44] the radial stress applied on the fibre wall in the fresh state during the loading phase $\sigma_{r, load}$ can be expressed as follows:

$$\sigma_{r, load} = (1 - \sin \phi) \sigma_z \quad (3)$$

Where ϕ is the internal angle of the granular packing of the matrix. In non-saturated conditions, Khelifi et al. found 22° with kaolin paste and 35° for mortars [Khelifi 12, Khelifi 13b]. Taking an internal angle of 22° , it gives $\sigma_{r, fresh load} = 0.625 \sigma_z$.

The consolidation stress is applied for only three minutes. The load is then removed and the sample is pulled out of the device after 24 hours (figure 10). The matrix will be submitted to an elastic release. The stress state at the outer boundaries of the cementitious matrix will pass from $\sigma_{r, load}$ to 0 in radial direction, and from $\sigma_{z, load}$ to 0 in axial direction. A residual stress $\sigma_{r, unload}$ will be effective on the inner wall of the matrix (corresponding to the fiber/matrix interface). This residual stress is correlated to the elastic behavior of the matrix and of the fibre.

During compression, one can assume that the fiber is purely elastic, and that the matrix is almost plastic. During relaxation, matrix is supposed to be more rigid than the fiber: [Bourmaud 09] measure radial elasticity modulus E_{Rf} by nano-indentation of several plant fibres. They found longitudinal by radial elastic modulus ratios E_{Lf} / E_{Rf} between 6 and 9. Taking, $E_{Lf} / E_{Rf} = 8$, and measures of E_L shown on Fig. 5, we obtain radial elastic modulus between 250MPa and 1000MPa for the coir bundle. After 24 hours, the Young modulus of the matrix will be around 3000MPa. The high variability of coir elastic parameters can then explain the high scattering in the pull-out results.

Fibre and matrix dimensional variations during hardening

The fibre can absorb water in the fresh state and release it during hardening, with limited shrinkage. Following the experimental protocol, the absorption kinetic of the coir and the limited amount of water in the matrix at fresh state, the size variation of the fiber due to swelling should be very low. Shrinkage of cement-clay pastes during hardening is also a well-known phenomenon. For example, [Khelifi 13a] measured a shrinkage of about 0.75% after 28 days hardening of mortars with high amounts of kaolin. Due to the annular geometry of the matrix (a matrix cylinder crossed by a fiber), its shrinkage will increase the compressive load on the fibre, as in the case of a shrinkage ring test [Zhang 13], while fibre drying will reduce the residual radial stress.

Mechanical creep within the fibre and the matrix

This creep is generally neglected, as in [Tran 13]. Nevertheless, to take this phenomenon into account, a coefficient k could be added to the residual strain, as suggested in [Zhang 13]:

$$\varepsilon_f - \varepsilon_m \rightarrow k(t) (\varepsilon_f - \varepsilon_m) \quad (4)$$

Where k is a time-dependent creep parameter, ε_f and ε_m are respectively the fiber deformation by shrinkage and the matrix deformation by shrinkage.

Further developments are needed to improve the understanding of each of the phenomena cited above and to properly understand the variability of the results, but the development conduces to a dependency of the IFSS versus consolidation stress. The case of this study is very theoretical: one fibre bundle centred in a cylinder-matrix. However, the residual stress due to the differences of elastic parameter, and the adhesion phenomena discussed above will be present in real cases as extrusion or compression of fibre reinforced cementitious materials.

4 CONCLUSION

To optimize cement tenacity, coir fibre are used following inspiration from their function in the nature. Their tensile behaviour exhibits energy dissipation ability due to their specific hierarchical microstructure. Coir/cement interfacial shear strength is studied by an original pull-out method. Experimental results have evidenced higher values compared to those reported in the literature.

It has been shown that, on the mean, a higher consolidation pressure results in a better interfacial bond strength despite the high results variability due to the high dispersion of the fibers geometrical and mechanical properties. The variability in diameter and elasticity of these fibres could also be an asset to the behaviour of the composite. The two modes of debonding described in this paper show the influence of the diameter variation along a fibre. The proper optimized fibre to limit interfacial debonding should be a fibre with a randomly varying diameter that will ensure sufficient anchoring. In nature, coconut fibres are rough and therefore can only provide good anchorage if they are sufficiently long with a possible tortuous arrangement of the fibres within the matrix.

The influence of pressure on bond strength shows that manufacturing with an extrusion process instead of casting is therefore advisable in order to optimize the

interfacial properties. During extrusion, fibres are submitted to a constant and high pressure when the fresh material crosses the die. This pressure improves the interfacial bond strength and then the mechanical behaviour of the final composites.

Further investigations must be carried out to estimate the geometrical variations of the mineral matrix and fibres due to elasticity, stress relaxation, drying and matrix hardening.

5 REFERENCES

- [Agopyan 05] V. Agopyan, H. Savastano Jr, V.M. John, M.A. Cincotto, Developments on vegetable fibre-cement based materials in Sao Paulo, Brazil: an overview, *Cement and Concrete Composites*, 27 (2005) 527-536.
- [Ali 11] M. Ali, Coconut fibre: A versatile material and its application in engineering, *Journal of Civil Engineering and Construction Technology*, 2 (2011) 189-197.
- [Ali 12] M. Ali, A. Liu, H. Sou, N. Chow, Mechanical and dynamic properties of coconut fibre reinforced concrete, *Construction and Building Materials*, 30 (2012) 814-825.
- [Ali 13] M. Ali, N. Chow, Experimental investigations on coconut-fibre rope tensile strength and pullout from coconut fibre reinforced concrete, *Construction and Building Materials*, 41 (2013) 681-690.
- [Baley 06] C. Baley, F.d.r. Busnel, Y. Grohens, O. Sire, Influence of chemical treatments on surface properties and adhesion of flax fibre and polyester resin, *Composites Part A: Applied Science and Manufacturing*, 37 (2006) 1626-1637.
- [Baley 12] C. Baley, A. Le Duigou, A. Bourmaud, P. Davies, Influence of drying on the mechanical behaviour of flax fibres and their unidirectional composites, *Composites Part A: Applied Science and Manufacturing*, 43 (2012) 1226-1233.
- [Bentur 07] A. Bentur, S. Mindness, *Fibre reinforced cementitious composites*, second edition, New York, 2007.
- [Bilba 03] K. Bilba, M.A. Arsene, A. Ouensanga, Sugar cane bagasse fibre reinforced cement composites. Part I. Influence of the botanical components of bagasse on the setting of bagasse/cement composite, *Cement and Concrete Composites*, 25 (2003) 91-96.
- [Blumenkrantz 73] N. Blumenkrantz, G. Absoe-Hansen, New method for quantitative determination of uronic acids, *Analytical Biochemistry*, 54 (1973) 484-9.
- [Bouazaoui 08] L. Bouazaoui, A. Li, Analysis of steel/concrete interfacial shear stress by means of pull out test, *International Journal of Adhesion and Adhesives*, 28 (2008) 101-108.
- [Boulekbache 12] B. Boulekbache, M. Hamrat, M. Chemrouk, S. Amziane, Influence of yield stress and compressive strength on direct shear behaviour of steel fibre-reinforced concrete, *Construction and Building Materials*, 27 (2012) 6-14.
- [Bourmaud 09] A. Bourmaud, C. Baley, Rigidity analysis of polypropylene/vegetal fibre composites after recycling, *Polymer Degradation and Stability*, 94 (2009) 297-305.
- [Bourmaud 10] A. Bourmaud, C. Morvan, C. Baley, Importance of fiber preparation to optimize the surface

- and mechanical properties of unitary flax fiber, *Industrial Crops and Products*, 32 (2010) 662-667.
- [Charlet 11] K. Charlet, A. Beakou, Mechanical properties of interfaces within a flax bundle - Part I: Experimental analysis, *International Journal of Adhesion and Adhesives*, 31 (2011) 875-881.
- [Defoirdt 10] N. Defoirdt, S. Biswas, L.D. Vriese, L.Q.N. Tran, J.V. Acker, Q. Ahsan, L. Gorbatikh, A.V. Vuure, I. Verpoest, Assessment of the tensile properties of coir, bamboo and jute fibre, *Composites Part A: Applied Science and Manufacturing*, 41 (2010) 588-595.
- [De Lorenzis 02] L. De Lorenzis, A. Rizzo, A. La Tegola, A modified pull-out test for bond of near-surface mounted FRP rods in concrete, *Composites Part B: Engineering*, 33 (2002) 589-603.
- [Dicker 14] M.P.M. Dicker, P.F. Duckworth, A.B. Baker, G. Francois, M.K. Hazzard, P.M. Weaver, Green composites: A review of material attributes and complementary applications, *Composites Part A: Applied Science and Manufacturing*, 56 (2014) 280-9.
- [Dubois 56] M. Dubois, K. Gilles, J.K. Hamilton, P.A. Rebers, Colorimetric method for determining of sugars and related substances, *Anal Chem*, 28 (1956) 350-366.
- [Fratzl 07] P. Fratzl, H.S. Gupta, F.D. Fischer, O. Kolednik, Hindered Crack Propagation in Materials with Periodically Varying Young's Modulus—Lessons from Biological Materials, *Advanced Materials*, 19 (2007) 2657-2661.
- [Hejazi 12] S.M. Hejazi, M. Sheikhzadeh, S.M. Abtahi, A. Zadhoush, A simple review of soil reinforcement by using natural and synthetic fibers, *Construction and Building Materials*, 30 (2012) 100-116.
- [Jacky 44] J. Jacky, The coefficient of earth pressure at rest, *Journal for Society of Hungarian Architects and Engineers*, (1944) 355-358.
- [John 05] V.M. John, M.A. Cincotto, C. Sjostrom, V. Agopyan, C.T.A. Oliveira, Durability of slag mortar reinforced with coconut fibre, *Cement and Concrete Composites*, 27 (2005) 565-574.
- [Justiz-Smith 08] N.G. Justiz-Smith, G.J. Virgo, V.E. Buchanan, Potential of Jamaican banana, coconut coir and bagasse fibres as composite materials, *Materials Characterization*, 59 (2008) 1273-1278.
- [Khelifi 12] H. Khelifi, Matériaux argileux stabilisés au ciment et renforcés de fibres végétales : formulation pour l'extrusion, in, UBS, Lorient, 2012.
- [Khelifi 13a] H. Khelifi, A. Perrot, T. Lecompte, G. Ausias, Design of clay/cement mixtures for extruded building products, *Materials and Structures*, 46 (2013) 999-1010.
- [Khelifi 13b] H. Khelifi, A. Perrot, T. Lecompte, D. Rangeard, G. Ausias, Prediction of extrusion load and liquid phase filtration during ram extrusion of high solid volume fraction pastes, *Powder Technology*, 249 (2013) 258-268.
- [Le Duigou 11] A. Le Duigou, P. Davies, C. Baley, Environmental Impact Analysis of the Production of Flax Fibres to be Used as Composite Material Reinforcement, *Journal of Biobased Materials and Bioenergy*, 5 (2011) 153-165.
- [Le Duigou 14] A. Le Duigou, A. Kervoelen, A. Le Grand, M. Nardin, C. Baley, Interfacial properties of flax fibre-epoxy resin systems: Existence of a complex interphase, *Composites Science and Technology*, 100 (2014) 152-157.
- [Morissey 85] F. Morissey, R. Coutts, P. Grossman, Bond between cellulose fibers and cement, *International journal of Cement Composites and Lightweight Concrete*, 7 (1985) 73-80.
- [Nam 11] T.H. Nam, S. Ogihara, N.H. Tung, S. Kobayashi, Effect of alkali treatment on interfacial and mechanical properties of coir fiber reinforced poly(butylene succinate) biodegradable composites, *Composites Part B: Engineering*, 42 (2011) 1648-1656.
- [Nguyen 09] T.-T. Nguyen, V. Picandet, S. Amziane, C. Baley, Influence of compactness and hemp hurd characteristics on the mechanical properties of lime and hemp concrete, *European Journal of Environmental and Civil Engineering*, 13 (2009) 1039-1050.
- [Nozahic 12] V. Nozahic, S. Amziane, Influence of sunflower aggregates surface treatments on physical properties and adhesion with a mineral binder, *Composites Part A: Applied Science and Manufacturing*, 43 (2012) 1837-1849.
- [Pacheco-Torgal 11] F. Pacheco-Torgal, S. Jalali, Cementitious building materials reinforced with vegetable fibres: A review, *Construction and Building Materials*, 25 (2011) 575-581.
- [Placet 14] V. Placet, O. Cissé, M. Lamine Boubakar, Nonlinear tensile behaviour of elementary hemp fibres. Part I: Investigation of the possible origins using repeated progressive loading with in situ microscopic observations, *Composites Part A: Applied Science and Manufacturing*, 56 (2014) 319-327.
- [Perrot 07] A. Perrot, C. Lanos, Y. Melinge, P. Estellé, Mortar physical properties evolution in extrusion flow, *Rheologica Acta*, 46 (2007) 1065-1073.
- [Ramakrishna 05] G. Ramakrishna, T. Sundararajan, Studies on the durability of natural fibres and the effect of corroded fibres on the strength of mortar, *Cement and Concrete Composites*, 27 (2005) 575-582.
- [Satvanaravana 82] K.G. Satyanarayana, C.K.S. Pillai, K. Sukumaran, S.G.K. Pillai, Structure property studies of coir fibres from various parts of coconut tree, *Journal of Materials Sciences*, 17 (1982) 2453-2462.
- [Savastano 99] H. Savastano Jr, V. Agopyan, Transition zone studies of vegetable fibre-cement paste composites, *Cement and Concrete Composites*, 21 (1999) 49-57.
- [Savastano 00] H. Savastano Jr, P.G. Warden, R.S.P. Coutts, Brazilian waste fibres as reinforcement for cement-based composites, *Cement and Concrete Composites*, 22 (2000) 379-384.
- [Shao 01] Y. Shao, J. Qiu, S.P. Shah, Microstructure of extruded cement-bonded fiberboard, *Cement and Concrete Research*, 31 (2001) 1153-1161.
- [Stancato 05] A.C. Stancato, A.K. Burke, A.L. Beraldo, Mechanism of a vegetable waste composite with polymer-modified cement (VWCPMC), *Cement and Concrete Composites*, 27 (2005) 599-603.
- [Tran 13] L.Q.N. Tran, C.A. Fuentes, C. Dupont-Gillain, A.W. Van Vuure, I. Verpoest, Understanding the interfacial compatibility and adhesion of natural coir fibre thermoplastic composites, *Composites Science and Technology*, 80 (2013) 23-30.
- [Zhang 13] J. Zhang, Y. Gao, Z. Wang, Evaluation of shrinkage induced cracking performance of low shrinkage engineered cementitious composite by ring tests, *Composites Part B: Engineering*, 52 (2013) 21-2.