POTENTIAL OF OLEAGINOUS FLAX FIBRE AS MORTAR REINFORCEMENT

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Abstract

Natural fibres have been widely used in building materials for centuries and environmental issues lead today to new research in this field. Plant fibre reinforced mortar is one of the rising innovative solutions to develop eco-friendly materials. In this work, the benefits of reinforcing a cementitious or pozzolanic based mortar with oleaginous flax fibre was evaluated and compared with classic polypropylene fibre reinforcement. Fresh state consistency was measured to show the impact of fibre addition and adapt the amount of superplasticizer to maintain the control mortars properties. Standard laboratory bending and compressive mechanical tests were performed on prismatic specimens at 7, 14 and 28 days. Free shrinkage over 28 days was also measured for each composition and the passive ring standard test method was used to determine the age of mortar cracking under restrained shrinkage. Both cementitious and pozzolanic based mortars showed a high flow sensibility when adding polypropylene or oleaginous flax fibres so that superplasticizer was necessary to maintain the fresh state properties. Compressive strength was slightly reduced with both fibres but a higher impact has been noticed at early age with flax ones. In cementitious based mortar, oleaginous flax and polypropylene fibre provided a similar post cracking behavior but the effect of flax reinforcement on pozzolanic based mortar was much more significant. Cracking due to the effect of restrained shrinkage was delayed in each case when adding fibres. Considering these results, oleaginous flax fibre could be a promising eco-friendly solution to replace polypropylene fibre. Further research have to enlighten keys for a better flax fibre distribution in the matrix, to optimize compositions and to evaluate the impact of natural weathering on potential applications.

Keywords:

Biobased building materials; Mineral binder; Fibre reinforcement; Flax; Polypropylene; Mechanical properties; Shrinkage

1 INTRODUCTION

The building construction industry has a major impact on sustainable development, mainly in terms of the use of raw material, greenhouse gas emissions and waste production. Studies on alternative materials are clearly a priority nowadays, to reduce energy consumption and optimize waste management. Bio-based construction materials could take up this environmental challenge.

To reduce the effect of drying shrinkage on coating, enhance ductility and enhance flexural and impact behavior, polypropylene fibres are commonly used nowadays. The fibres mixed in the whole material are used as internal reinforcement. The incorporation of fibres into mineral matrixes is an age old technique used to avoid the brittle behavior of building materials. Straw, horse hair or thin wood parts are some examples of fibres that have been used for thousands of years.

During the last 30 years, several studies have been carried out on the reinforcement of building materials by natural fibres [Ardanuy 2015] [Dittenber 2012] [Santos 2015] [Yan 2014]. Natural fibers, largely available at low cost in developing countries, possess numerous environmental and health qualities and could thus constitute an alternative to conventional fibres [Brandt 2008]. They are made with local, renewable raw materials, they are carbon neutral and their embodied energy is usually low as they can be obtained as industrial by-products (from the textile or paper industries, for example). Their morphology (diameter, aspect ratio, length, roughness) can be adapted to different purposes. They could be a suitable solution but the performance of a composite notably depends on the amount and length of the fibres [Li 2006] and composition has to be optimized to maintain a good global behavior [Li 2004]. The main disadvantages of natural fibres are the variability of their properties (natural growing, extracting method used) and their chemical interaction with the matrix (setting time, long term properties). Recent researches have also studied the potential use of natural or recycled fibres: coconut [Hwang 2016], flax [Page 2017], fishing nets [Bertelsen 2017], human hair [Fantilli 2017a], wool [Fantilli 2017b] or pig hair [Araya-Letelier 2017]. They always revealed a high potential in reinforcing mineral matrixes.

In the context of ecological footprint reduction, the use of local resources and less treatment is a major concern. Oleaginous flax fibres were selected in the present work because they present all the characteristics of ecofriendly byproduct. Oleaginous flax is largely produced an available. Grown for the seed use, the stem could be revalorized to decrease the quantity of waste produced. Oleaginous flax fibres might be convenient as they present high tensile strength and stiffness with a high aspect ratio [Ouagne 2017] [Pillin 2011]. The absolute density of oleaginous flax fibres is close to 1.5 g/cm^3. It is similar to textile flax and hemp fibres density found in the literature [Baley 2004] [Barbulée 2015] [Chafei 2014] [Page 2017].

The question is: what should be the key parameters for a low environmental impact method leading to fibres suitable as mortar reinforcement ?

Considering this goal, raw fibres were used in this study with only rough mechanical transformation. No chemical treatment was applied. Two parameters were used to obtain reinforced mortars with flow time close to reference mortars: water and/or superplasticizer addition. The objective is a similar real use for all mortars tested. The content in oleaginous flax fibre tested was 1% (in binder weight) and the results were compared to plain reference mortars and 0.4% polypropylene reinforced mortars. Comparison was based on short time tests as the first objective is to have convenient mortars regarding fresh state properties and restrained shrinkage behavior.

2 MATERIALS

2.1 Mineral binders

The effect of fibre reinforcement was studied on two different mineral binders. They were selected because of their distinct mechanisms of hardening (hydraulic setting, pozzolanic reaction and carbonation). Moreover, one strategy for improving the durability is to modify the composition of the matrix in order to remove or reduce the alkaline compounds [Ardanuy 2011] [Santos [2015]. The innovative pozzolanic matrix used in this study suits this strategy.

Cement binder (C)

Portland cement is the most widely used hydraulic binder in the world. The cement used for this study is a CEM I 32.5 R. Letter C will refer to this binder in the results section.

Pozzolanic binder (P)

The second mineral binder used in this study was designed in previous works [Magniont 2010][Dinh 2014] to be associated with plant aggregates in composites for the design of precast blocks or with fibres for coatings. It associates 30% of aerial lime and 70% of flash calcined metakaolin. The mix was completed by the addition of two admixtures: 3% of potassium sulfate (K_2SO_4) to improve strength at early age and 1.5% of superplasticizer to reduce the mixing water content. Letter P will refer to this binder in the results section.

2.2 Sand

The sand used in this study is a siliceous rolled 0/2 mm sand complying NF P 18-545 - EN12620/EN13139 standards.

2.3 Polypropylene fibres

Classic polypropylene fibres were used in the present work for casting the reference reinforced mortar. SEM observations show regular well separated fibres with a smooth surface (Fig.1).

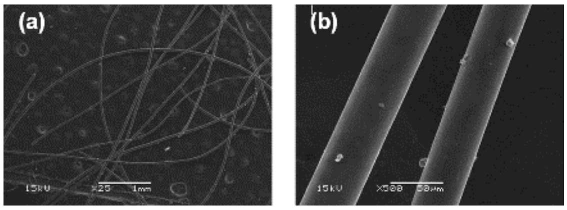

Fig. 1: SEM observation of the polypropylen fibres x25 (a) and x500 (b).

The length chosen was 12 mm. It is the median of the three most widely used (6, 12 and 20 mm) for reducing the effects of restrained shrinkage. The diameter is 30 µm and the density is 0.9 g/cm^3.

2.4 Oleaginous flax fibres

The oleaginous flax fibres (F) are bast fibres, extracted from the stem of the plant. In order to limit the environmental impact of the elaboration process, raw fibres were used in this study with only mechanical transformation and no chemical treatment was applied. Oleaginous flax fibres were retted and stripped. The retting process is applied to fibres in the aim of eliminating the pectin that surrounds the fibres and binds them to each other and to the stem. Natural retting in the field was applied between harvesting the flower and the stem.

Oleaginous flax fibres were obtained through mechanical defibration with a Laroche Cadette 1000 “All Fibre” extraction equipment. This process leads to the production of fibres associated with residues of the woody part of the stem, named shives. Fibres and shives were then separated manually. They were first shacked to make the heaviest parts fall (shives) and then sieved with a 5 mm and 1 mm sieves. After this process, raw fibres were obtained without shive or dust. SEM observations (Jeol JSM-6380LV) were carried out to determine the range of the fibres diameter and to compare the surface condition with polypropylene fibres (Fig.2).

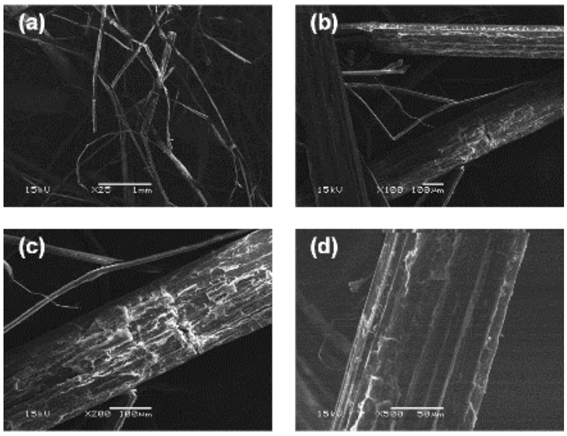

Fig. 2: SEM observation of the oleaginous flax fibres x25 (a), x100 (b), x200 (c) and x500 (d).

They were spread and cut two times in perpendicular directions leaving a 20 mm space between cuts. Oleaginous flax fibres obtained present a large range of length with 80 % of 10 to 40 mm fibres in equal proportion (Fig.3). Around 500 fibres collected in 5 different areas were measured.

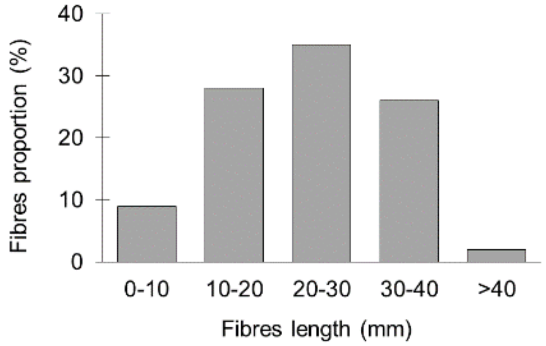

Fig. 3: Oleaginous flax fibres length distribution.

Combined with the length distribution aforementioned, they clearly show heterogeneous type of fibres in length, diameter or roughness to be used in the final composite. Oleaginous flax fibre diameter usually lies between 12 and 20 µm [Pillin 2011] [Ouagne 2017]. In this work, single fibres (10-20 µm) as well as bundles (100-200 µm) will constitute the natural reinforcement for mortars.

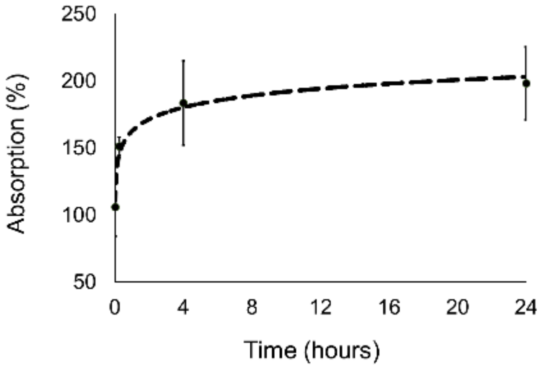

Fig. 4: Oleaginous flax fibres water absorption rate.

Water absorption (Fig.4) is a key parameter that makes a great difference between natural and synthetic fibres when adding in mortars. In order to keep the same amount of water for binder mixing and setting, composition needs to be completed with the quantity of water absorbed by the fibre. Absorption test was realized following the spin-dryer method [Magniont 2010] and the absorbent paper method.

Oleaginous flax fibres used in this study present a water absorption rate between 150% and 200 % during mixing, casting and setting.

3 METHODS

3.1 Compositions and elaboration

The starting point of this study is to maintain the fresh state properties in order to have mortars that can be used for a same large range of applications. The test method chosen is the LCPC flow time apparatus complying NF P18-452 standard. After empirical tests, a mean flow time of 10 s ± 3 s was set as the reference. This range leads to a mortar that can be horizontally cast (manually, vibrating or jolting) as well as vertically applied (manually). To keep the flow time in this range for all mortars, water proportion and superplasticizer addition were modified in fibres reinforced ones.

Compositions (Tab.1)

All the mortars present the same sand / binder weight ratio (s/b = 2). The amount of water was first determined to have the reference mortars (CNF and PNF) in the flow time range previously presented (Fig.5). Water / binder weight ratio (w/b) for reference mortars were 0.42 (CNF) and 0.57 (PNF).

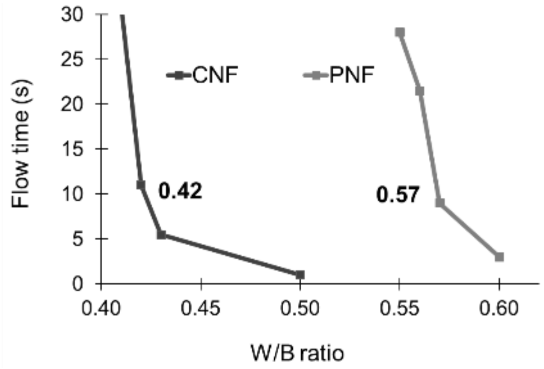

Fig. 5: Reference mortars flow time for different w/b ratio and values complying the flow time range.

For the oleaginous flax fibres composites, the fibres / binder weight ratio (f/b) was 1 %. This ratio constitutes a good compromise for the incorporation of fibres in the fresh paste when having length between 10 and 40 mm. To keep the same amount of water for binder mixing, 150 % by fibre weight of water was added due to flax fibres absorption rate. So that, w/b ratio takes +0.015 with oleaginous flax fibres.

Tab. 1: Mortars composition expressed by binder weight proportion.

	CNF	CFF 1%	CFP 0.4%	PNF	PFF 1%	PFP 0.4%
Cement	1	1	1	-	-	-
Aerial Lime	-	-	-	0.3	0.3	0.3
Metakaolin	-	-	-	0.7	0.7	0.7
Sand	2	2	2	2	2	2
K_2SO_4	-	-	-	0.03	0.03	0.03
Flax Fibres	-	0.01	-	-	0.01	-
Polypropylene Fibres	-	-	0.004	-	-	0.004
Water	0.42	0.435	0.42	0.57	0.585	0.57
Superplasticizer	-	0.004	0.002	0.015	0.019	0.017

For the polypropylene fibres composites, the fibres / binder weight ratio (f/b) was 0.4 %. This ratio has been chosen as a reference reinforcement following industrial recommendations (0.25 to 0.55 %). As polypropylene fibres don't absorb water, w/b ratio wasn't modified when adding fibres.

Adding superplasticizer is necessary to keep the flow time in the range given for both flax or polypropylene fibres reinforced mortars. Good accordance was found when adding 0.4 % and 0.2 % by binder weight for flax and polypropylene fibres reinforced mortars respectively.

Elaboration

The mixing was completed using a mixer complying with NF EN 196-1. Fibres were introduced manually during the 90 s pause included in the mixing method. Free shrinkage and flexural/compressive specimens casting comply with NF-EN 196-3 standard. After mixing, the binder paste was cast in 40x40x160 mm^3 moulds and kept in endogenous conditions in a room at 20°C. They were demolded 24 h after casting.

3.2 Free shrinkage

Total free shrinkage evolution was completed following NF P 15-433 standard. After demolding, samples were stored in controlled conditions room (20°C – 50%RH). Length reference was set 1 hour after demolding and free shrinkage was measured at 2, 3, 4, 24, 48 hours, 7, 15 and 28 days. Three samples were tested for each composition.

3.3 Restrained shrinkage

Passive ring test was used to measure cracking sensibility of compositions regarding restrained shrinkage (Fig.6).

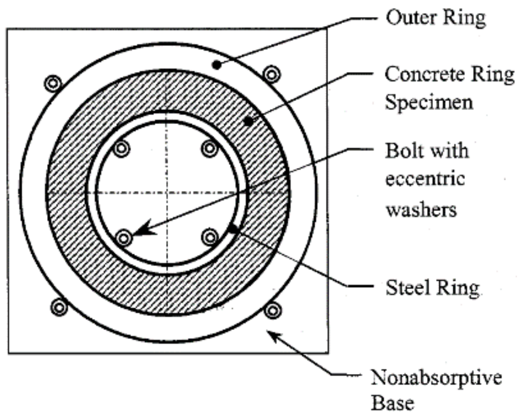

Fig. 6: Plan view of passive ring test used for restrained shrinkage test.

The age at cracking for each mortar was assessed in accordance with ASTM C1581-04 standard (mortar ring dimensions : Ø in. 330 mm, Ø out. 406 mm, h. 150 mm. Two specimens were casted for each composition as preliminary tests.

3.4 Compressive and flexural tests

After demolding, 40x40x160 mm^3 samples were kept in endogenous conditions in sealed plastic bags avoiding any water exchange with the laboratory test room air (20°C and uncontrolled RH).

Flexural strength tests were performed on specimens at 3, 7 and 28 days. They were conducted with a constant displacement rate of 1 mm/min on a Zwick/Roell Proline Z100 5kN universal testing machine. Three samples were tested for each composition.

Compressive strength tests were performed on specimens at 3, 7 and 28 days. They were conducted with a constant loading rate of 2.4 kN/s on a Controlab compression testing machine according to NF-EN 196-3 standard. Six samples were tested for each composition.

4 RESULTS

4.1 Fresh state properties

The goal of this part was to adapt the formulation with water and/or superplasticizer addition to maintain all the mortars in the same range of use (Fig.7). Without adding water to balance flax fibres absorption or superplasticizer when adding fibres, flow time got out the range.

When adding 1% (wgt binder) flax fibres, 1.5% water and 0.4% superplasticizer was needed to maintain reference mortar flow time. Mortars reinforced with 0.4% polypropylene fibres only needed 0.2% superplasticizer addition. These additions were convenient for both type of binder: cement or pozzolanic based.

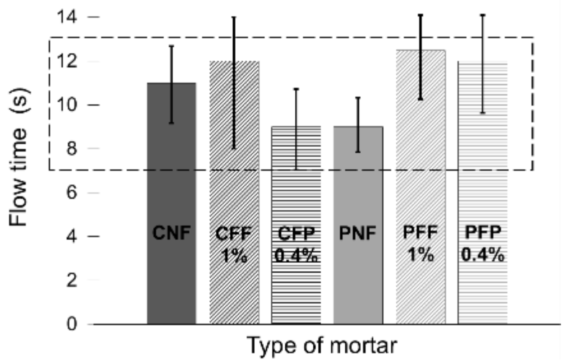

Fig. 7: Flow time for cement and pozzolanic based mortars.

During mixing, polypropylene fibres homogeneously spread in the matrix whereas part of flax fibres remained in little bundles. The high length combined with the high roughness and diameter variability of flax fibres might be the major parameter that causes bundles. Regular spreading might be obtained using industrial cutting solutions to have calibrated fibres.

4.2 Shrinkage behavior

Free shrinkage (Fig.8)

Cement based mortar shrinkage was not significantly affected by flax or polypropylene fibres addition with the formulations considered. The pozzolanic mortar shrinkage slightly decreased for both fibres addition.

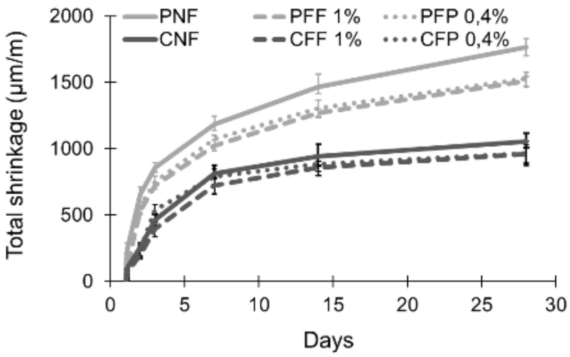

Fig. 8: Total shrinkage between 1 and 28 days after casting for cement and pozzolanic based mortars.

Restrained shrinkage (ring test)

Passive ring tests first showed a very early cracking of pozzolanic based mortars in comparison with cement based. It is due to the higher total shrinkage value and rate combined with lower strength. Composites formulations including fibres addition presented a delayed cracking. The most important delay was obtained with oleaginous flax fibres formulations (Tab.2). As fibres acts after cracking, this delay might be linked to the modification of mechanical properties and to the slightly reduced shrinkage of modified mortars. Mechanical tests are necessary to enlighten this point.

Tab. 2: Cracking appearance during restrained shrinkage ring test

Mortar	Cracking (days)	
CNF	5.8	±0.1
CFF 1%	7.9	±0.2
CFP 0.4%	7.0	±0.6
PNF	1.3	±0.1
PFF 1%	2.1	±0.6
PFP 0.4%	1.7	±0.1

4.3 Mechanical behavior

Compressive strength

Adding 1% by weight binder of oleaginous flax fibres classically reduced the compressive strength of reference mortars (Fig.9).

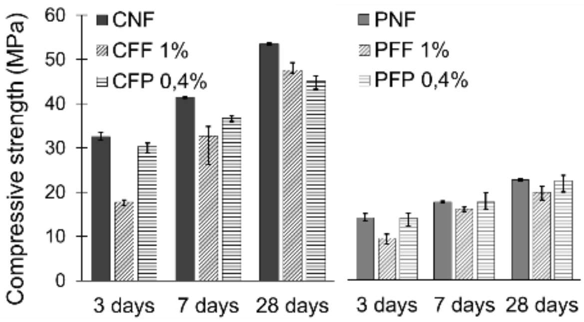

Fig. 9: Compressive strength at 3, 7 and 28 days for cement and pozzolanic based mortars.

Both type of binder were affected by around 10-15% loss at 28 days. The most significant difference was observed with flax fibres at 3 days (cement -45%, pozzolanic -30%). It was attributed to the chemical interaction between binders and water-soluble fibres compounds that causes delay on hardening process [Diquelou 2015] [Sabathier 2017]. Adding polypropylene fibres also reduced compressive strength but the impact is lower and no delay was observed at early age.

Flexural strength and modulus

Flexural strength was less affected by fibres addition and no significant difference was observed at 7 and 28 days (Fig.10). The main difference observed at 3 days with flax addition was due to the hardening delay aforementioned.

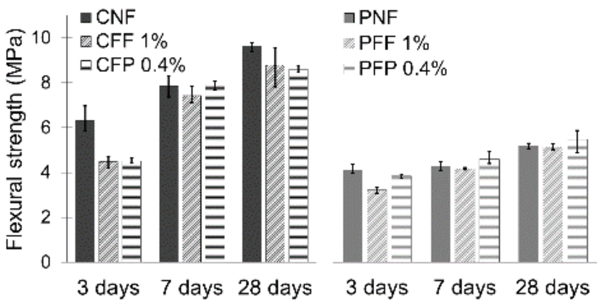

Fig. 10: Maximum flexural strength at 3, 7 and 28 days for cement and pozzolanic based mortars.

Flexural modulus was only significantly lower at 3 days except for the pozzolanic mortar reinforced with polypropylene fibres (Fig.11). This means that during the higher shrinkage rate when cracking appears, modified formulations including flax fibres present a lower modulus. This property leads to lower strength for equal strain in the mortar when subjected to restrained shrinkage. Combined with a slightly lower shrinkage value, cracking is delayed. Moreover, in real coating application, fibres would also act as soon as cracks appear to limit their opening and propagation.

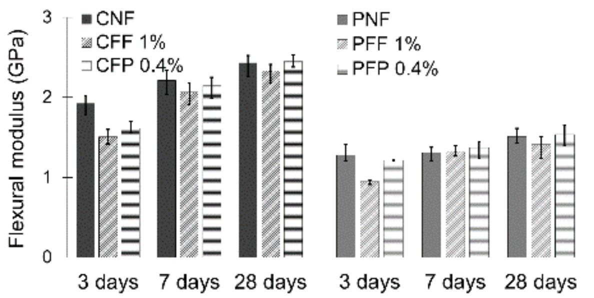

Fig. 11: Flexural modulus at 3, 7 and 28 days for cement and pozzolanic based mortars.

Flexural elastic and post cracking behavior

Lowered modulus can be clearly observed on strength-strain curves at 3 days (Fig.11). As hardening progresses, all mortars modulus reach the same value as reference mortars. Recording flexural strength evolution after cracking to 1% strain, enlighten the residual strength provided by fibres reinforcement.

Adding oleaginous flax raw cut fibres, within the formulations considered in this work, provided equal or superior toughness in comparison with reference polypropylene reinforcement. These results show a high potential of oleaginous flax fibres to limit the effect of restrained drying shrinkage by maintaining residual strength after cracking. Brittle behavior is avoided without important negative effects on rigidity and strength. The variability of length and diameter with the high surface roughness of flax fibres had a negative impact on fibres repartition in the matrix but it might have been an advantage here for residual toughness. Pozzolanic based mortars reinforced with oleaginous flax fibres presented a very constant residual strength of 2 MPa. Considering the area under the curve linked to the fracture energy, incorporating oleaginous flax fibres leads to a very interesting ratio between toughness before and after cracking when flexural strength is around 4 to 5 MPa.

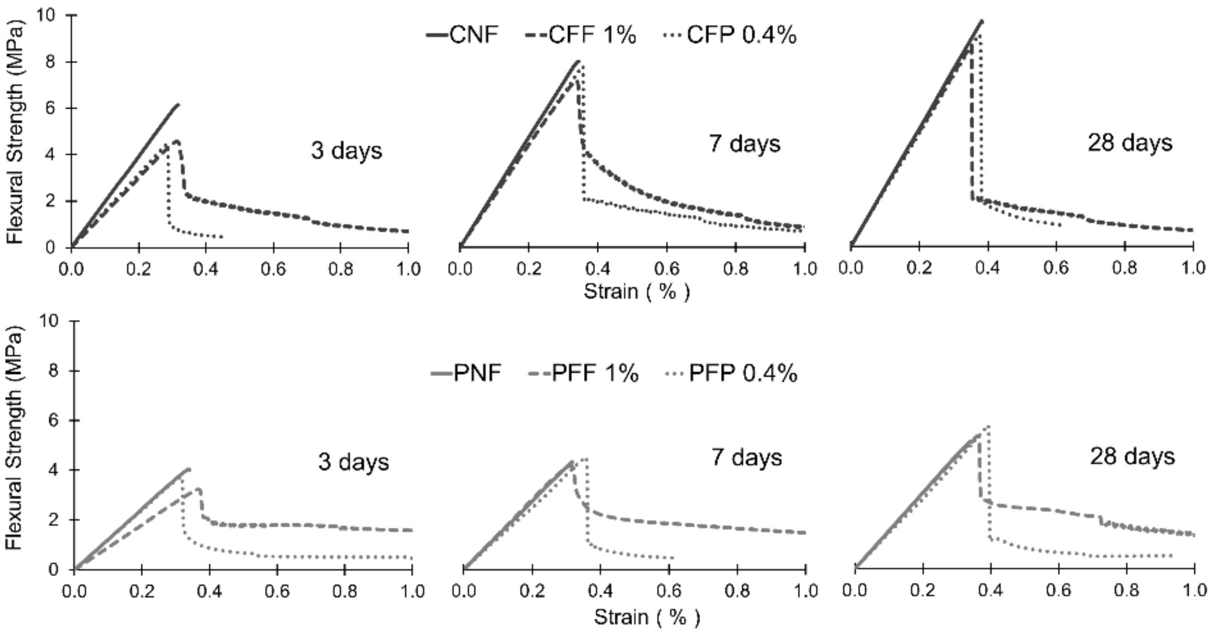

Fig. 12: Flexural strength–strain typical curves at 3, 7 and 28 days for cement and pozzolanic based mortars.

5 CONCLUSION

This study explored the key parameters for a low environmental impact method leading to fibres suitable as mortar reinforcement. Oleaginous flax fibres were used in two different mineral binders: cement and pozzolanic based. Results were compared to plain reference mortars and polypropylene fibres reinforcement. They showed a high potential of fibres with basic elaboration.

Main conclusions are:

- Oleaginous flax or polypropylene fibres addition in mortars (cement based or pozzolanic based) required superplasticizer addition to maintain fresh state properties in the reference mortar range. Average recommended addition is 0.5 x fibres weight.
- Oleaginous flax fibres addition in mortars (cement based or pozzolanic based) also required water addition to balance fibres absorption to maintain fresh state properties in the reference mortar range. Average recommended addition is the amount of water absorbed by fibres at 1 hour.
- Basic mechanical treatment applied in this study does not lead to sufficient homogenous fibres characteristics (length and diameter). This variability causes bundles during mixing and hazardous fibres repartition in the composite. For efficient polypropylene fibres replacement, precise cutting is necessary to have calibrated fibres with a length close to polypropylene one.
- Modifying reference mortars formulations to incorporate fibres does not significantly affect free shrinkage. Pozzolanic based mortars present a slightly lower shrinkage value when adding fibres.
- Oleaginous flax fibres reinforced mortars reduce the effect of restrained shrinkage by delaying cracking. This delay might be linked to the lower elastic modulus at early age for flax fibres reinforced composites.
- Incorporating flax fibres causes significant lower compressive and flexural strength and modulus at 3 days due to fibres-binder chemical interactions leading to hardening delay. At 7 and 28 days, compressive strength remains lower but flexural properties are less affected.
- Post cracking behavior shows a higher residual strength for oleaginous flax fibres reinforcement. This post cracking strength is efficient for high strain and should be able to maintain low cracks opening.

Further studies should precise the followings:

- Are water and superplasticizer recommended additions to maintain flow time convenient for calibrated fibres with different length, f/b ratio and fibres origin ?
- Is efficient fibres cutting enough to provide homogenous fibres spreading into the matrix ?
- Is the global environmental impact of oleaginous flax fibres reinforcement (raw material collect, mechanical transformation, transport, water and superplasticizer addition) lower than polypropylene fibres reinforcement for a dosage leading to the same mechanical benefits ?
- What are the benefits of these composites in a real coating use regarding cracking, long term properties and impact behavior ?

6 ACKNOWLEDGMENTS

Authors acknowledge the support of Agromat-LCA for providing the fibres and Argeco society for providing the metakaolin used in this work.

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