



DEVELOPMENT OF POLYMER-REINFORCED FLAX FIBRE FOR THE STRENGTHENING OF REINFORCED CONCRETE.

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Abstract

This paper presents the development of new environmentally friendly reinforcement composite materials using technical flax fibres. Flax fibres have low energetic production costs, are biodegradable, cost-effective and have specific mechanical properties comparable to those of glass fibres, making them interesting in the field of composite reinforcement. In this study two different applying methods for concrete strengthening are examined, (1) pre-hardened flax composite strips produced by compression moulding using PA11 or epoxy resins, and (2) hand-laid up flexible flax fabrics impregnated on site with ambient temperature curing epoxy resins. Several combinations of matrices and flax fabrics were tested. The composites' mechanical properties and cohesion were determined through tensile loading and delamination tests. The impregnation quality was assessed through SEM microscopy. The reinforcement ability and the adhesion on concrete substrate of the qualified composites were evaluated through pull-out tests. Different numbers of layers and application methods were tested to determine an optimal use of the composite. The tests show very good concrete/FFRP bonding and high mechanical properties, leading to failures in concrete rather than in the composite or at the interface.

Keywords:

Flax Fibre Reinforced Polymer; Concrete Strengthening

1 INTRODUCTION

The reinforcement of concrete structures through the external bonding of glass or carbon fibre reinforced polymers is now an approved and widely used method [1-2]. These materials allow getting an additional tensile force to the concrete surface opposing the deformation and providing an extra layer of protection with the external environment. Yet growing ecological concerns as well as economic considerations with the increasing cost and scarcity of fossil resources justify the substitution of these synthetic fibres to more environmentally-friendly vegetal fibres.

Composites currently used in outdoor building civil concrete are available in two forms: (1) fine pre-hardened polymer strips reinforced by unidirectional fibres glued on site to the concrete, or (2) uni- or bidirectional fabrics impregnated on site by hand in a wet layup process directly on the substrate. Both techniques require a sizing on the concrete by a thermosetting resin curable at ambient temperature. The resins used for outdoor concrete reinforcement must have good adhesion with the concrete as well as with the reinforcing fibres, good weather resistance, especially to moisture or rain, resist to extreme temperatures and chemicals from the concrete or the

environment. [1] For economic reasons, the glass fibre is most commonly used despite its inferior mechanical properties than carbon fibres [11].

Among the potential natural sources, flax fibres (*Linum usitatissimum*) offers the best combination of light weight, low cost, and high specific mechanical properties, comparable to glass fibres [3-4]. Flax cultivated for fibres and linseed are not the same species. 68% of the world's production of long flax fibres, a total of roughly 137 tons in 2010, is originated from France [5]. Numerous attempts to use flax as FRP reinforcement as glass substitution can be found in the literature [12-13], however none as concrete external reinforcement. Many differences between flax and glass fibres prevent the direct transposition from the one technology to the other. The poor compatibility between the hydrophilic natural fibres and hydrophobic matrices of flax FRP (FFRP) has been discussed in many researches for its improvement [11-13], but it still difficult to improve without heavy chemical intervention. Moreover flax fibres are not exactly long-fibres: individual flax fibres are 10 mm to 80 mm long and this observation could be an explanation for the non-linear tensile behaviour described in the literature [6-8,16-18].

In this paper we aim to introduce and characterize a Flax Fibre Reinforced Polymer using the wet layup

process specific to Reinforced Concrete (RC) structure external reinforcement. The influence of the number of folds on the properties is then examined as a critical variable for this specific application.

2 MATERIALS AND METHODS

2.1 Used materials

The flax fabrics used in this study were cultivated and produced in France following the quality and traceability of ISO and NF XP T 25-501 norms specific to technical flax fibres. The flax fibres went through the four usual stages of preparation. The stems of the plant are ripped out and left for natural retting on the ground through the action of micro-organisms separating the fibres from the outer woods. The inner fibres are later extracted from the stem through different mechanical actions called scutching. Heckling

combs the fibres bundles through progressively finer heckles, to be able to finally fold the flax fibres into long ribbons. The ribbons have not been twisted in order to preserve the quality of the technical fibres. The quality of the final ribbon is standardized by mixing fibres from different productions years and flax varieties. Four types of weaving have been used see Table 2. The unidirectional fabrics are hold together by fine weft yarns that have been neglected in all further considerations due to the poor fabric cohesion on their direction. The bidirectional fabrics are woven in 3/3 twill offering balanced properties in both 0/90° directions. The different used epoxy resins are ambient temperature curing bi-components resins. The epoxy and hardeners have been mixed at the specified ratio. Their properties are summarized in Table 1.

Table 1: Characteristics of the flax fabrics





Fabric designation	UD400	UD220	S600	S350
Weight of fabric (g/m ²)	400	220	600	350
Weaving	UD	UD	Twill 3/3	Twill 3/3
Aspect				

Table 2: Characteristics of the used resins

Resin	ρ (kg.dm ⁻³)	E (GPa)	σ_u (MPa)	ϵ_u (%)
Epoxy B	1.21	3.4	97.2	5.2
Epoxy A2	1.25	2.3	27	1.7
Epoxy C	1.10			
Epoxy D	1.10		37	
Epoxy A1	1.06		35	1.35
Epoxy E	1.30	4.5	30	0.9
Epoxy F	1.25	3.0	80	3.0
PA11	1.04	1.5	40	>50.0

2.2 Preparation of the hand-laid composite

In the first part of the study one different type of epoxy resin was used for each specimen, see Table 4. In a second part, flax fibres were impregnated with a low viscosity epoxy (A1) and layered on a more viscous compatible epoxy resin (A2), reproducing the layer enabling the composite to stick to a concrete surface. Flax fabric pieces of a length of 26 cm in the main directions are impregnated with epoxy matrix by hand and layered in the same orientation on waxed wooden plates. The composites are scraped with a trowel to eliminate excessive resin and the air bubbles as well as possible and are left to cure in atmospheric conditions at 20°C for 7 days.

2.3 Preparation of the pre-hardened composite

Two types of pre-hardened composite were realized by an external company FIBROLINE using epoxy F and PA11 resins. Flax fabrics are impregnated layer by layer with resin powder following their D-Preg ® technology and fixed in the fabric through calendar. The impregnated layers are piled with 3 layers in the same orientation and consolidated in a hot-press under 10 bars at 160°C for the epoxy resin and 200°C for the PA11.

Table 3: Characteristics of the tested pre-hardened specimens

Material	Nb Spec.	V ⁱ (%)	V ^p (%)	ρ_{comp} (kg.dm ⁻³)
Epoxy/UD400 x3	5	55	15	1.21
PA11/UD400 x3	4	60	10	1.20

2.4 Density and fibre content measurements

The composite density was measured with a Kern 573 balance. The density of hot pressed hard composite was measured using the dimensions of the plates. The density of wet layup samples was measured by hydrostatic weighing in water, and the density was calculated following Eq.(1)

$$\rho_{comp} = \rho_{air} + \frac{M_{air}}{M_{air} - M_{eau}} \cdot (\rho_{eau} - \rho_{air}) \quad (1)$$

The porosity volume fractions were determined by using the following Eq (2) adapted from Scida et al.[19]:

$$V_p = 1 - \frac{\rho_{comp}}{\rho_m} + \frac{n^p \rho_f^s}{\rho^p} \cdot \left(\frac{1}{\rho_m} - \frac{1}{\rho_f} \right) \quad (2)$$

where ρ_{comp} , ρ_m and ρ_f are respectively the mass density of the composite, the matrix and the dry fibres; ρ_f^s is the areal density of the dry fabric; and n^p is the number of layers. The density of the flax fibres was fixed a $\rho_f = 1540 \text{ kg.m}^{-3}$ following the literature [20].

2.5 Tensile tests on composite

The specimens, 25 mm x 250 mm, are cut out of the composite plates using a circular saw cooled with a water cooling system and left to dry.

Table 4: Characteristics of the tested wet layup specimens

Fabric	Resin	Number of		Thickness (mm)	V ^f (%)	V ^p (%)	ρ_{comp} (kg.dm ⁻³)
		Spec.	Folds				
UD400	B	5	1	2.6	10	5	1,18
	A1	6	1	2.4	11	5	1,22
	C	5	1	2.2	12	3	1,12
	D	5	1	2.2	15	3	1,15
	E	4	1	1.8	14	8	1,30
UD220	A1/A2	4	1	1.5	10	3	1,18
		4	2	2.1	14	6	1,14
		4	3	3.1	14	4	1,15
		6	4	3.7	15	10	1,08
S350	A1/A2	4	1	2.4	10	6	1,14
		6	2	3.2	14	9	1,11
		4	3	4.4	16	11	1,07
S600	A1/A2	7	1	2.6	16	4	1,18
		5	2	4.2	19	10	1,10
		4	3	5.5	21	9	1,11
		5	4	5.8	27	10	1,11

Tensile tests on longitudinal direction were carried out following standardized static tension tests norm ISO 527-1 on a universal traction testing machine ZWICK /1475. The tensile specimens were straightened at a cross-head speed of 1 mm/min until rupture of the specimen. Tensile force was determined through force sensor and longitudinal strain with 10 mm strain gauges glued to the composites specimens surface.

The ultimate strain and tensile modulus were calculated as specifies in the norm using the gross-laminated area for the pre-hardened composites and the net fibre area for the hand-laid composites as introduced in the ACI 440.2R-11. The net-laminated area is calculated using the total cross-sectional area of the cured composite. The net-fibre area is calculated using the known area of the fibres, neglecting the resin, since the wet layup process leads to variable resin content. As a mean to completely disregard the composites thickness, the tensile stress per composites width was also calculated in kN.cm⁻¹. This value gives a clearer indication of the stress the composite could take once at the surface of concrete.

2.6 Observation of microstructure

The microstructure analysis helped to determine the impregnation of the flax fibres and analyse the weakness zones. After rupture through tensile traction, samples of the composite are cut out and metalized with gold/palladium before being observed with a Hitachi S800-1 scanning electron microscope (SEM).

2.7 Characterization of the adherence of the composite on concrete

Concrete plates of 50 x 50 x 4cm were mold and left to harden for at least 28 days. The surface is stripped with a diamond saw. The hand-laid composite were then prepared as described in 2.2 but directly on the concrete plates. The pre-hardened composite were glued on the concrete with a viscous resin (A2). Both types were let to cure 7 days at 20°C.

The bonding test was conducted according to the French standards NF P 18-852. Circle of 5 cm

diameter were drilled in the composite to a depth extending to 3-4 mm into the concrete. Steel rings with a threaded insert are then bond with a fast-setting epoxy on the circles. The test is performed using the pull-out instrument to apply a tensile force until failure occurs. The failure line is noted and the bond strength calculated.

3 RESULTS AND DISCUSSIONS

3.1 Aspect and properties of pre-hardened composite

The PA11 flax composites have a deep brown colour and the Epoxy flax composite are grey-brown. The surface is smooth and the composites have a regular thickness of 1.4 mm.

Fig. 1(a) represents the results of the tensile tests obtained for the pre-hardened Epoxy and PA11 FFRP. The curves are slightly dispersed. This could be a sign for some heterogeneity in the material. The stress-strain curves present two regions of different modulus. Baley et al [6] propose a stress-strain model of their FFRP in tension with a nonlinear initial region that then meet a linear asymptote. Scida et al. [19] have associate this phenomenon by the reorientation of flax microfibrils. Poilâne et al. [8] describe the stress-strain curves of FFRP as "bi-linear" with a bending area called "yield point" and introduce the "apparent modulus" measured from the first region. The modulus E has been measured in a similar way in the first regions of the curves. The yield point is much less pronounced for the PA11 FFRP. This could be due to the specific nature of PA11 that presented a long deformation capacity before breaking. The volumetric percentage of fibre was calculated around $V^f = 55\%$ and the porosity around $V^p = 15\%$ for the Epoxy FFRP; and $V^f = 60\%$ and the porosity around $V^p = 10\%$ for the PA11 FFRP.

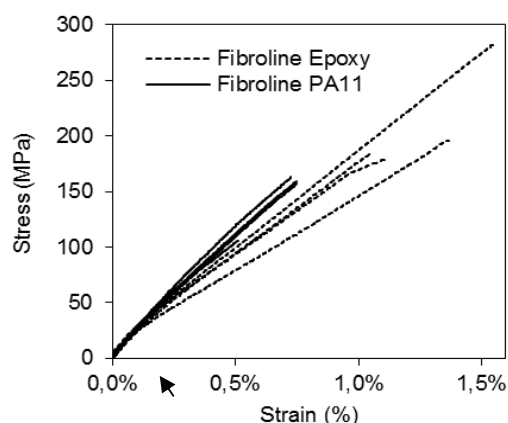
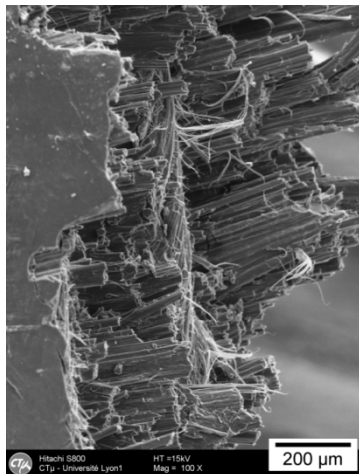


Fig. 1: Stress-strain curves obtained in tensile test of pre-hardened specimens. The arrow points the bending region of the yield point.

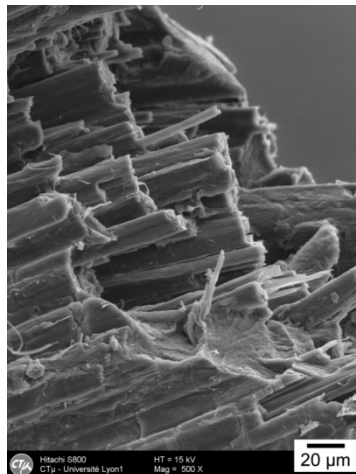
Table 5 shows the tensile properties of the pre-hardened composites with the properties of pre-hardened FFRP Epoxy found in the literature. The epoxy FFRP have similar properties ($E = 15$ GPa, $\sigma_u = 240$ MPa) than those found by Poilâne et al. [9] and Van de Weyenberg [10] with Epoxy FFRP with similar fibres contents. Coroller et al. [7] found higher mechanical properties for similar fibres content for each of the three tested flax species through Resin Transfer Moulding (RTM). Composites produced through pultrusion generally show higher properties which can be explained through the better individualization of the fibres through the high pressure injection of the resin in the bundles. To the opposite of the expected results of a direct mixture law, the PA11 composite shows a higher modulus of $E = 23$ GPa and a lower strain at break $\sigma_u = 146$ MPa. This result proves the importance the compatibility between fibres and matrix.

Table 5: Results of the tensile tests on pre-hardened composite. Comparison with literature.

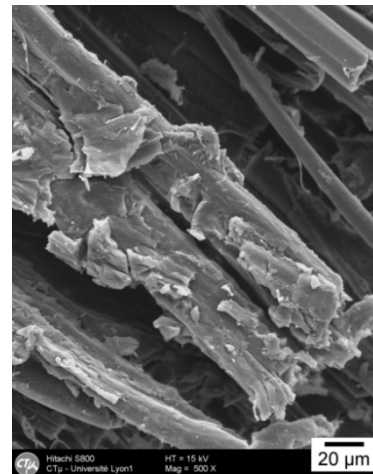
Material	Process	V_f (%)	E (GPa)	σ_u (MPa)	ξ_u (kN/cm)	ϵ_u (%)	Ref.
Epoxy / UD400	Hot Press	55	24.0 ± 0.8	234 ± 15	3.27 ± 0.20	1.17 ± 0.15	-
PA11 / UD400	Hot Press	60	23.7 ± 0.9	165 ± 15	2.30 ± 0.23	0.75 ± 0.06	-
Epoxy/ Flax UD	RTM	32	15.0	132			[16]
Epoxy / Flax pulp	Hot curing	33	16.0 ± 0.7	154 ± 0.9			[17]
Epoxy / Flax UD	Hot Press	44	26.5	325		1.56 ± 0.13	[19]
Epoxy / Flax UD	Hot curing	60	27.5 ± 1.5	298 ± 10		1.69 ± 0.05	[8]
Epoxy / Flax UD	Hot Press	40	22.5 ± 1.5	328 ± 18		1.6 ± 0.20	[6]
Epoxy / Flax UD	Hot Press	50	28 ± 3.6	364 ± 14		1.3 ± 0.01	[7]



(a) Fracture surface of the PA11 FFRP.



(b) Fracture surface within the pre-hardened PA11 FFRP.



(c) Fracture surface within the pre-hardened Epoxy FFRP.

Fig. 2: SEM views of the fracture surface of pre-hardened Epoxy and PA11 FFRP (UD400 x3).

Fig. 2 shows the SEM observations of the fracture surface of the composite after the tensile test. Both composite have broken following steps, making it possible to recognize each of the three fibre layers (Fig. 2(a)). This highlights the heterogeneity of the material. However for the PA11 composite only few fibres are loosened and each layer seem to have broken as a cohesive material (Fig. 2(b)). The PA11 resin shows therefore a good adherence with the natural fibres. In the Epoxy composite more fibres have been laid bare. Epoxy resin residues are visible until the end of the loosen fibres, which is a sign for

good compatibility between the resin and the fibres (Fig. 2(c)). The fracture therefore happened in the matrix and not at the interface between matrix and fibres.

3.2 Aspect and properties of wet layup composite

The manufacturing process influences directly the properties of the composite. Fibres alignment and homogeneous distribution, a good impregnation and absence of porosity are critical to optimize the mechanical properties of the finished product. A good interface allows efficient transfer of mechanical stresses to the fibres during stress. [14]

In the wet layup process the fibres are impregnated with an excess of resin in order to prevent porosities by lack of resin. The excessive resin is evacuated roughly with a trowel. Consequently this process does not fix exactly the percentage of fibres nor the thickness of the composite, and those can vary depending on the worker. In addition the composite is not in a mould during its curing so its surface has the natural roughness of the fibres, which does not allow to accurately determining the thickness for the whole plate. These considerations conduct to use the net-area to calculate the strain and the modulus of the composite, and to calculate the stress per width.

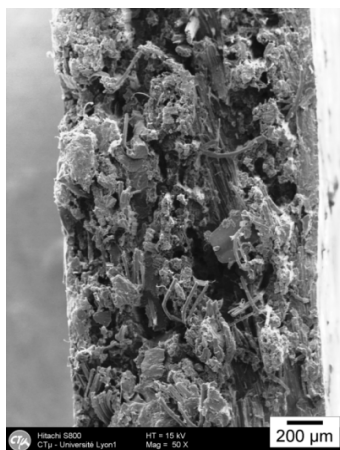
A first series of tests with a UD400 fabric was performed with different resins. Epoxy resins A1, B and E 330 are epoxy resins used for wet layup application of FRP for structural concrete reinforcement on site with glass or carbon fibres. The epoxy resin C is a bio-based epoxy resin and resin D is a highly liquid resin used in civil engineering for sealing cracks in concrete. The results of tests with these resins are summarized in the upper part of Table 6. Following the tensile tests, it appears that the flax fibres are still dry and the bundles are not properly impregnated what can lead to

durability problems through easier water uptake and fast debonding between matrix and fibres. The resin adapted for glass or carbon fibres seem too viscous to impregnate the flax fibres. The FFRP with bio-sourced resin does not exhibit sufficiently high properties to be able to be considered in this context. The specimens with the liquid resin D showed well impregnated bundles and the highest stress per cm. However it does not allow vertical application on concrete substrate, as may be necessary in practice.

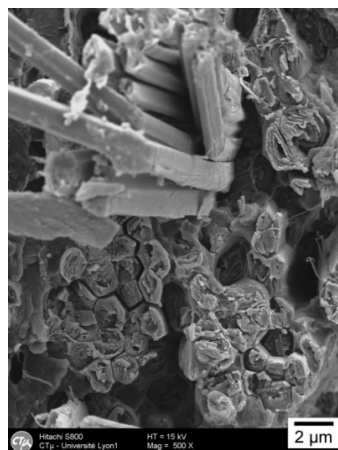
Following these observations a combination of resin was set: a liquid resin to impregnate the bundle of fibres in heart (A2) and a more viscous resin compatible to bond the composite concrete substrate (A1). The SEM observation of the fracture surfaces of this composite (Fig. 3) shows firstly the heterogeneity of the fibres distribution. The fabric layers are clearly visible on Fig. 3(a). As shown in Fig. 3(b) the inside of the fibres bundles are easily laid bare. This is the consequence of a poor impregnation inside the bundles. Nonetheless the impregnated fibres break cohesively with the matrix. The interface between the two resins (Fig. 3(c)) displays no sign of fracture, which confirms their good compatibility.

Table 6 : Results of the tensile tests on wet layup composites.

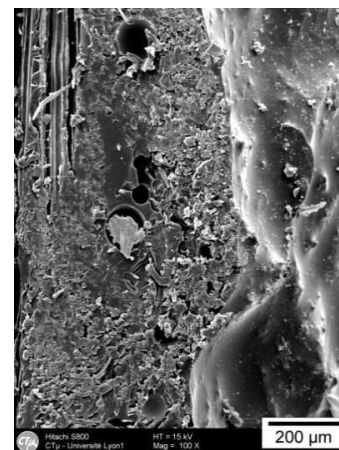
Fabric	Resin	Folds	V ^f (%)	E (GPa)	σ_u (MPa)	ξ_u (kN/cm)	ϵ_u (%)
UD400	B	1	10	31.5 ± 1.8	207 ± 9	1.64 ± 0.09	0.98 ± 0.06
	A1	1	11	26.6 ± 1.5	150 ± 13	1.40 ± 0.14	0.80 ± 0.08
	C	1	12	18.8 ± 3.0	117 ± 6	1.11 ± 0.05	0.71 ± 0.11
	D	1	15	22.3 ± 1.9	193 ± 8	1.84 ± 0.07	1.47 ± 0.08
	E	1	14	28.3 ± 2.1	136 ± 12	1.29 ± 0.11	
UD220	A1/A2	1	10	19.3 ± 1.0	195 ± 12	0.95 ± 0.06	1.29 ± 0.15
		2	14	21.3 ± 1.2	171 ± 16	1.62 ± 0.16	1.29 ± 0.07
		3	14	19.0 ± 1.1	172 ± 17	2.52 ± 0.25	1.37 ± 0.10
		4	15	18.4 ± 1.2	151 ± 10	2.86 ± 0.11	1.25 ± 0.08
S350	A1/A2	1	10	18.8 ± 1.6	172 ± 9	0.84 ± 0.05	
		2	14	21.8 ± 1.5	150 ± 6	1.46 ± 0.06	1.17 ± 0.08
		3	16	23.2 ± 1.5	168 ± 12	2.47 ± 0.17	1.27 ± 0.03
S600	A1/A2	1	16	34.0 ± 1.5	130 ± 8	0.82 ± 0.05	0.97 ± 0.15
		2	19	32.7 ± 3.0	157 ± 12	1.98 ± 0.15	1.06 ± 0.19
		3	21	20.5 ± 0.25	181 ± 6	3.42 ± 0.11	1.17 ± 0.17
		4	27	20.3 ± 0.6	155 ± 4	3.90 ± 0.10	1.16 ± 0.06



(a) Fracture surface of a wet layup Epoxy FFRP (S350 x3).



(b) Fracture surface within the wet layup composite (S350 x3).



(c) Fracture surface between the two Epoxy resins (Epx 342/TFC).

Fig. 3: SEM views of the fracture surface of wet layup Epoxy FFRP (S350 x3 with resins A1/A2)

3.3 Adhesion of the FFRP on concrete substrate

The pull-out test performed on pre-hardened plates bonded with resin E and composite wet layup with resins A1/A2 have both led to rupture of the plate in concrete; at roughly 2.5 MPa. NF P18-840 stipulates that a minimum pull-out strength of 1.5 and 2.0–3.0 MPa must be developed for non-structural and structural repairs respectively. The adhesion of the composite on the concrete substrate is therefore sufficient to consider this technology further.

3.4 Effect of folds number on wet layup composite properties with a resin-duo

Fig. 6 represents a typical result obtained in tensile tests. The curves can be considerably dispersed. This has to be taken into account for further interpretations and a special care given to the standard deviation. The yield point is visible as for the pre-hardened composite. The characteristics of the products can be seen in Table 6.

The properties of wet layup with different fabrics are analysed following the influence of the number of folds and the influence of the weaving nature. The volumetric percentage of fibres increases with the number of folds as well as the percentage of porosity. Indeed with the increasing number of folds, the more the proportion of the bonding layer A1 decreases relative to the whole composite. This parallel evolution supports the hypothesis that the porosity of the composite is mainly concentrated in the bundles and fibres themselves. The number of folds does not appear to impact on the strain as can be seen in Fig.2. The thickness of the composite increases with the number of folds compensating the stress. The apparent initial module does not seem to follow a precise pattern Fig. 5. We do not observe proportional relationship between the volume of fibre and the strain at break or the apparent modulus unlike various publications on the subject [7-8]. This difference can be due to the calculation method of the net area. Nevertheless, it is interesting to note that the final module appears relatively constant (Fig.6).

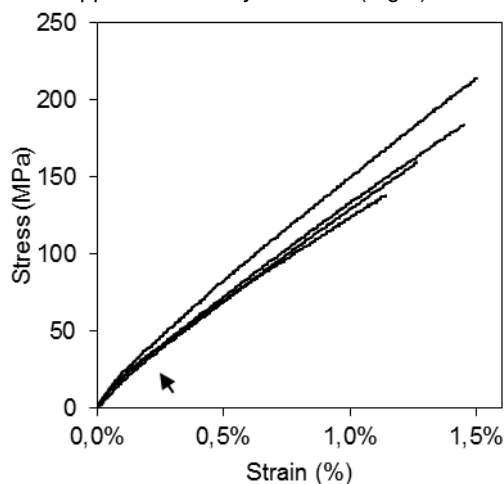


Fig. 4: Typical stress-strain curve obtained in tensile test. (UD220 x3 with resins A1/A2)

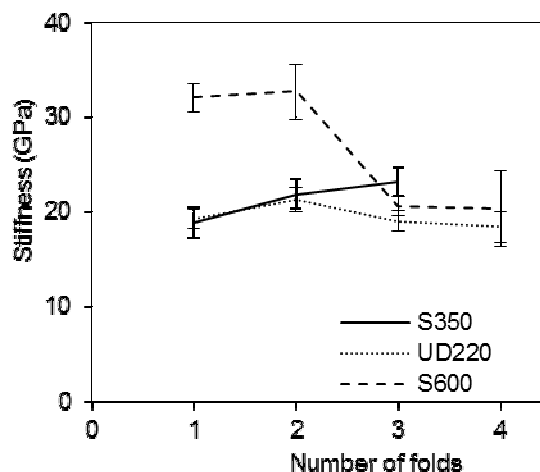


Fig. 5: Evolution of the apparent initial tensile modulus with the number of folds

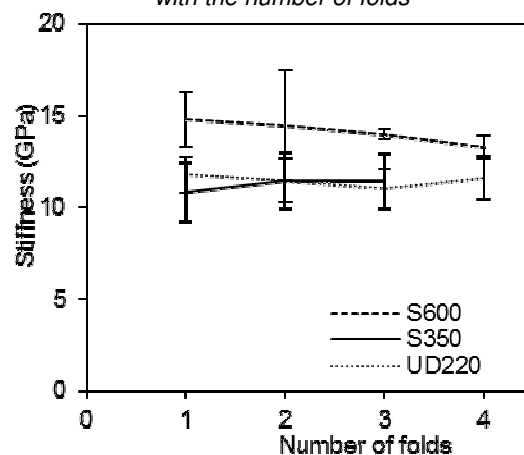


Fig. 6: Evolution of the final modulus with the number of folds

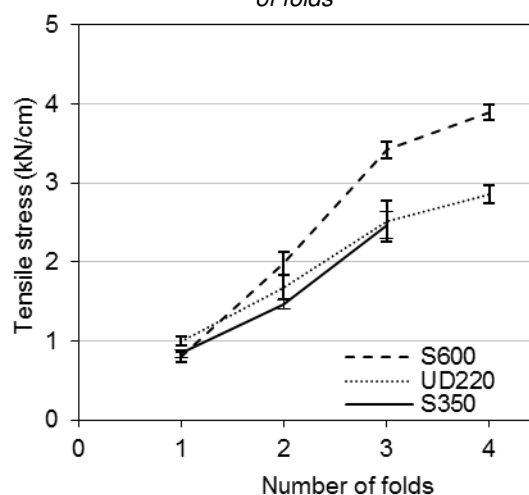


Fig. 7: Evolution of the tensile stress per composite width with the number of folds

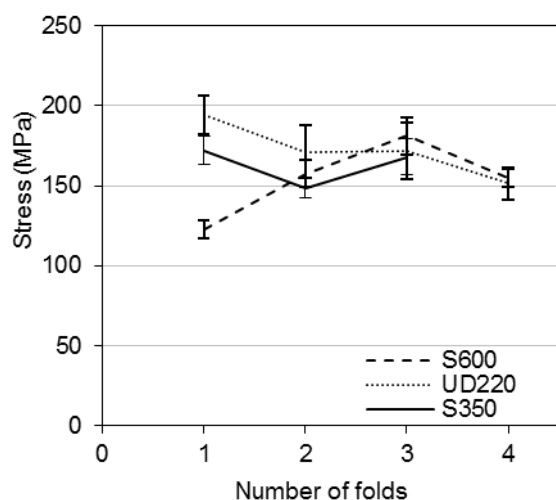


Fig. 8: Evolution of the stress at rupture with the number of folds

The influence of the number of layers on the tensile stress is displayed in Fig. 7. The tensile stress increases with the number of plies of composite in a quasi-proportional manner. Nevertheless, there is a flattening of the curve from the 4th folds. In practice no more than two or three folds are used. The transmission of the stress to the fibres of the outer layers cannot be guaranteed due to the number of intermediate resin. It could be that this trend is visible from the 4th folds. The resin rich areas between the fibres folds are fragility areas of the composites which ages prematurely. Therefore the number of plies should be minimized by using larger yarns. On the other hand the finer the yarn the better the impregnation of the fibres bundles [8]. A balanced weight fabric must thus be determined to ensure both good impregnation and limit the number of folds. The effort taken by the twill 350 g/m² is similar to that taken by the unidirectional 400 g/m². This confirms that only the fibres in the direction of traction work. It is more interesting to work with a unidirectional material in the case of RC reinforcement for which the direction to strengthen is usually known.

4 CONCLUSION

Flax fibres are biodegradable, cost-effective and have specific mechanical properties comparable to those of glass fibres, making them interesting as glass substitution as RC external reinforcement composite. Two types of composites have been developed; pre-hardened Epoxy and PA11 Flax Fibres Reinforced Polymers manufactured by hot pressing in a mould, and wet laid up Epoxy Flax Fibres Reinforced Polymer realized on site. The values obtained for the mechanical properties in tension for the pre-hardened composite were comparable to those obtained in the literature. Both composites present mechanical properties equal to those of commercial wet layup glass FRP ($E \geq 20$ GPa; $\sigma_u \geq 150$ MPa), and have a good adhesion with the concrete substrate.

The investigation of the influence of the number of fabric layers on the properties of wet laid up composite have shown that the usual mixing rule law don't seem to apply, since the mechanical properties don't seem to increase with the percentage of fibres. The evolution of the yield point and the modulus will have to be investigated further. However a quasi-constant strain and a quasi-proportional increase of the tensile stress

per width with the number of layers is a good and important fact for enabling reinforcement design calculations.

This work is a first step to the development of a new material for a Reinforced Concrete (RC) reinforcement. The first results are promising in view of the difficulty to get repeatable results coming from the biological nature of the fibres and the wet layup process. A set of shear adhesion tests and accelerated ageing tests are expected to confirm the potential of this material.

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