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Mechanical Properties of Natural Fiber Composites

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Abstract
Please provide a 200 word synopsis of the article, which will be used to summarise the work when presented online.
This topical review covers the mechanical properties of natural fibre composites (NFCs) that allow applications in many application fields ranging from infrastructure to transportation. Climate change alerts on the importance of sustainability and NFCs offer opportunities when compared to conventional glass-fibre composites. Advantages and drawbacks of NFC versus synthetic composites are considered and discussed. Mechanical properties are assessed both statically and dynamically. The hydrophilicity and its consequences on damage and deformation mechanisms are also addressed. The key to making well-reinforced composites is by ensuring that there is a strong interface between the reinforcing phase and the matrix phase. The interfacial bonding quality is thus discussed through various testing methods and the effects of surface treatments are presented.

Keywords
Please provide 10-15 keywords, which will be used for indexing purposes only. These should be listed alphabetically.
Plant fibre composites, Engineering applications, Mechanical properties, Quasistatic and dynamic behaviours, Hydrophilicity, Damage, Interface, Interfacial strength

Glossary

Bast fibre, high performance fibre extracted from plant stem
Glass fibre, synthetic fibre made of E or S glass
Hydrophilicity/hydrophobicity, attraction (or repulsion) to water
Interface, interface between the polymer matrix and the fibres
Interphase, transition region between fibre and the matrix.
Natural fibre, fibre of natural origin (animal, plant and mineral)
Tensile strength, resistance to tensile fracture (Pa)
Yarn, a thread formed by elementary fibres twisted together

Nomenclature

GFRP glass fibre reinforced plastics
MAPE maleated polyethylene
MAPP maleated polypropylene
NFC Natural fibre composite
PA11 Polyamide
PE polyethylene
PLA polylactide
PP Polypropylene
τ interfacial shear strength
F Force (N)
d fibre diameter
l embedded fibre length (m)
l_f fragment length (m)
l_c critical fragment length (m)
σ_f fibre strength (Pa)

Introduction and context

Natural fibre composites (NFCs) allow alternatives to synthetic carbon and glass fibre composites in the context of sustainable materials for a broad range of applications spanning from building structures to aeronautics (Shahzad, 2011; Yashas Gowda et al., 2018; Girijappa et al., 2019; Ramesh, 2019). Natural fibres mentioned in this chapter are plant-based but it should be noted that animal fibres like cocoon silkworm or spider silk can also been used (Ku et al., 2011; Oushabi, 2019). In fact, natural fibres comprise many different categories, as they may have animal, vegetable and mineral origins. For the sole vegetable origin, subcategories are defined according to their being wood (generally waste materials), extracted from stems, leaves or seeds. The review focuses on high performance natural bast fibres being extracted from plant stem, an example being hemp fibre that is used to illustrate further the topic. So far, the review can be extended to other categories and literature is cited accordingly. The environmental marks of NFCs can be much reduced in terms of energy and emission as compared to conventional ones. In the case of Hemp versus Glass, the marks are lower of an order of magnitude and

more (Table 1). As a matter of fact, the natural fibre composites are gaining market share even though the evolution is incremental and glass remains largely dominant (Fig. 1).

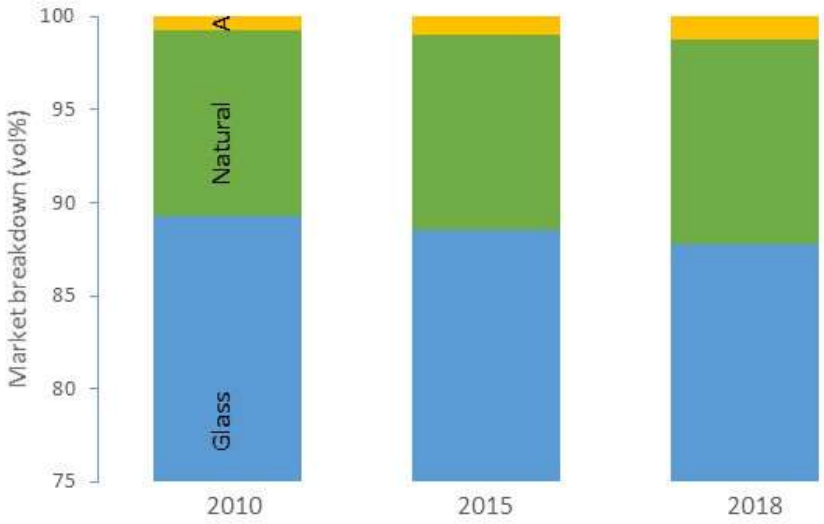


Figure 1. Market breakdown of fibres over the last decade (A for Aramid). A slight favorable evolution in the direction of natural fibre use is observed (according to Lucintel, 2019).

	Hemp fibre	**Glass fibre**
Power consumption (MJ)	3.4	48.3
CO2 emission (kg)	0.64	20.4
SOx & NOx emission (g)	2.15	11.7

Table 1. Environmental parameters in the production of 1kg of fibres (according to Shahzad, 2011).

NFCs can be classified according to the size of the fibres, namely short (randomly dispersed), medium (using mat or non-woven) and long fibre (unidirectional or woven), together with the associated matrix namely thermoplastic or thermoset. The process used fixes the combination of both bearing in mind that matrices are to be confined to temperatures compatible with NFCs. Therefore, polyethylene (PE), polypropylene (PP) thermoplastic matrix allows for extrusion, compression and injection moulding of the composites, while epoxy and polyurethane thermoset composites can be manufactured with resin transfer moulding, sheet and bulk moulding compound. The overall performance depends on the component properties (matrix and fibre) and on many other interdependent parameters as illustrated in Fig. 2, notably the interfaces as discussed extensively below.

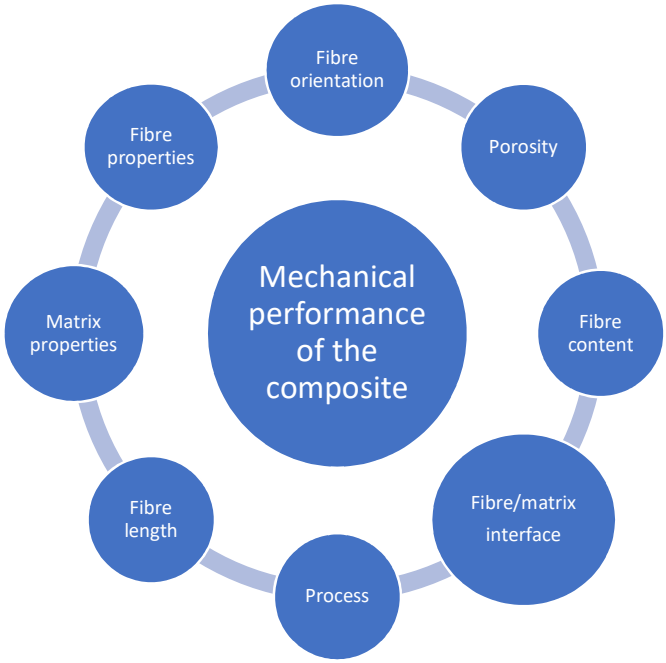


Figure 2. Influent parameters governing the performance of the composite.

Massive production of polymers in the last century has opened the use of composite materials. Noticeably, natural fibres could have been used originally as shown by the attempts by Ford company already around 1940, who incorporated natural materials to produce organic car body (Evans et al., 2005). Composites have entered all sectors including packaging, building, transportation (automotive, naval, and aeronautics), communication (Evans et al., 2005).

Progressively, NFCs have become used in most of the cited sectors although the mutation remains slow and the market is still dominated by glass fibre composites (Fig. 1). Nonetheless, the massive use of composites causes serious issues in the production (Table 1) and in the disposal of wastes. New regulations are in favour of NFCs either in terms of energy, raw material or disposal. Recycling routes are also to be explored and considered from the design step and here again natural fibres hold great advantage. The natural fibres represent an interesting low-cost and CO_2 neutral resource, as they can be extracted from many different plants like Banana, Flax, Hemp, Jute, some of them being waste products from the agroindustry (Kiruthika, 2017; Evans et al., 2005). Noticeably, the complete sustainability of the composites requires considering bio-based polymer matrices (La Mantia et al., 2011; La Rosa et al., 2014). Thermoplastics exhibit a reduced environmental impact as compared to thermosets, which cannot be recycled. On the other hand, thermosets offer higher mechanical performances.
It is important to emphasise that natural fibres also benefit from a reduction of weight when compared to their glass counterpart (Le Bourhis, 2014) that is of utmost interest for the transportation sector and in particular for the aeronautics (Table 2). Taking again the example of hemp, a factor about two is obtained in terms of density. Still, elementary hemp fibre competes in terms of elastic modulus when tensile strength reduction is the main drawback.

	Hemp fibre	Glass fibre
Density	1.4	2.5
Stiffness (elastic modulus, GPa)	70	70
Strength (tensile, GPa)	0.7	3
Hydrophilic	yes	no

Table 2. Properties of elementary fibres (order of magnitude).

High performance bast fibres extracted from plant stem offer weight saving around 10 to 30%. Flax, jute, hemp bast fibres represent the largest sources used by the manufacturers. These plants are cultivated and their production can be renewed on an annual basis to the demand of the manufacturers. Noticeably, the hemp culture is almost pesticide free while this is not the case for the other plants. The performances achieved by the fibres and their NFCs allow producing interior automotive parts as well as exterior components like bumpers, spoilers (Table 3) (Evans et al., 2005, Koronis et al., 2013). As regards the aeronautics, the applications concentrate on the interior (Scarponi et al., 2017). So far, the composites have to meet crucial capability in terms of flammability and impact loading. Structural and infrastructure are also addressed in sectors demanding moderate strength. Load bearing beam, roof panels for tanks, pedestrian bridges have been introduced. Naval applications have been also considered (La Rosa et al. 2014; Castegnaro et al., 2017). In particular, La Rosa et al. emphasised that the life cycle assessments (LCA) of bio-based polymer composites compare favorably with petroleum-based products (La Rosa et al. 2014).

	Examples of component
Transportation	Aeronautics: Interior panels; Automotive: Insulation, furniture, package shelves, Interior/exterior, door panels, seat backs panelling, bumpers, spoilers; Railway, Interior, door leaves; Naval, hull, panels
Infrastructure, building	Insulation, filling agent, moderate load bearing beam, roof panel, wind mill blade
Packaging	Textile, bag, filter

Table 3. Some applications of NFCs.

The two following sections concern mechanical properties both under static and dynamic modes. Mechanical properties addressed here are of utmost importance for the structural or semi-structural applications. A major drawback of natural fibre composites regards the moisture sensitivity. Therefore, this key point is also considered. Last section focuses on fibre/matrix interface that plays a major role in the resistance to fracture and fatigue.

1. Mechanical properties

Despite a steadily expanding body of work demonstrating the potential of natural fibres, industry adoption in load-bearing engineering applications is still reluctant due to a general lack of confidence in their mechanical performance, their complex loading behaviour and the relative immaturity of research compared to that of synthetic fibres (Mahboob et al., 2018). In this section, quasistatic and dynamic mechanical properties of NFCs will be discussed.

1.1 Quasistatic behaviour of natural fibre composites

In order to evaluate the potential of use of NFCs in engineering applications, many authors have determined the quasistatic behaviour of natural fibre composites in tensile, flexural and compressive loadings. For example, Ku et al. proposed a review on tensile properties of short plant fibres composites (Ku et al., 2011), Krishnan et al. have studied the tensile and flexural behaviour of hemp/polyester composites with different kinds of fibre orientations (Krishnan et al., 2020) and Van Vuure et al. have compared compressive properties of composites with three different natural fibres: flax, bamboo and coir (Van Vuure et al., 2015). In order to rank the large databank of mechanical properties of NFCs available in literature, Shah proposed different types of selection charts (Shah, 2014). Figure 3 presents for example a comparison of the absolute and specific tensile properties of various plant fibre composites with glass fibre reinforced plastics (GFRP). It can be seen in Figure 3 that NFCs, particularly (i) unidirectional thermosets and thermoplastics, and (ii) nonwoven thermoplastics, perform exceptionally well against similar GFRPs in terms of both absolute and specific stiffness. However, both the absolute and specific tensile strength of NFCs tend to be lower than that of GFRPs. Consequently, in terms of tensile properties, it can be said that NFCs may be potential alternatives to GFRPs in stiffness-critical applications, but not in strength-critical applications (Shah, 2014).

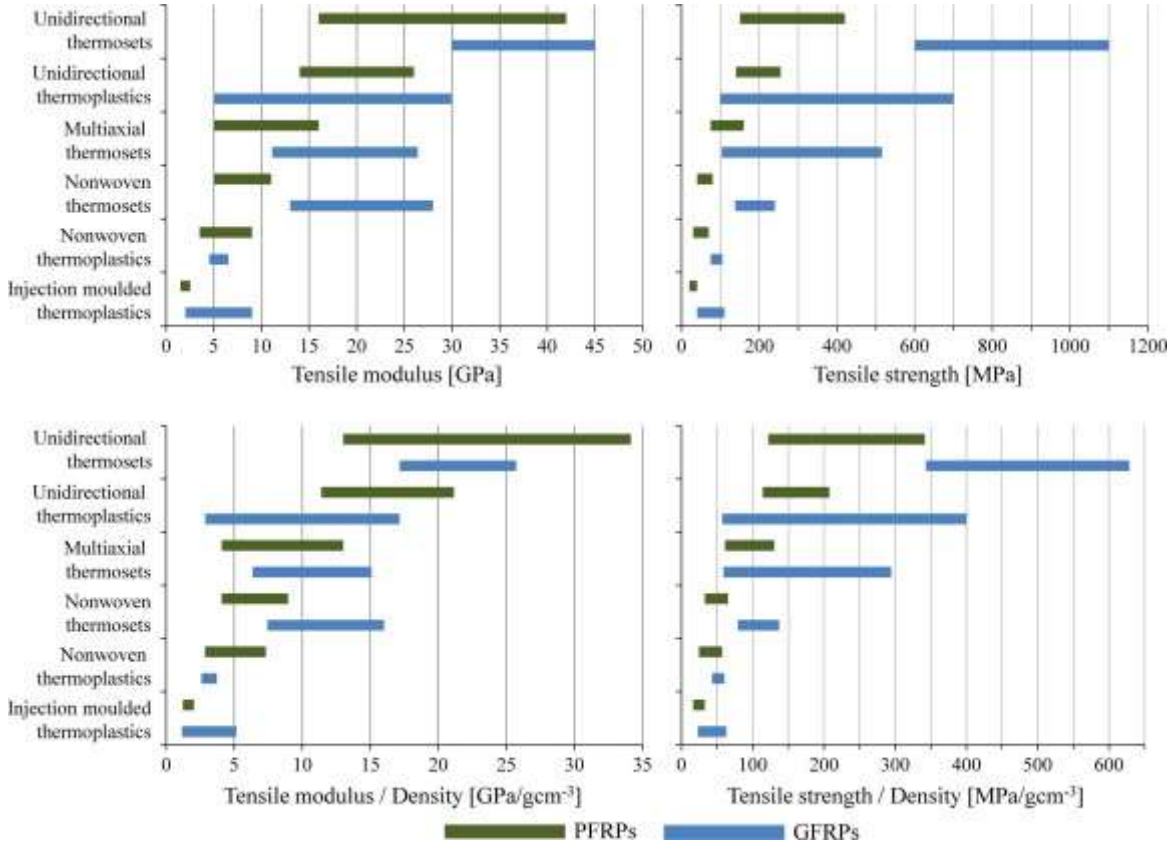
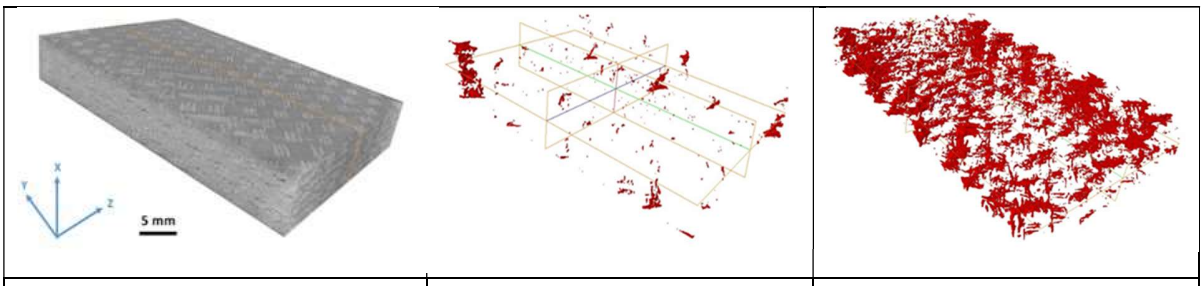


Figure 3. Comparison of the absolute and specific tensile properties of plant fibre reinforced plastics (PFRPs) with E-glass reinforced plastics (GFRPs). Reprinted from (Shah, 2014), with permission from Elsevier.

Because of the high moisture sensitivity of natural fibre composites, a crucial issue is to assess the influence of water on their mechanical properties. Several studies have shown the loss of rigidity and strength under quasistatic loading after moisture absorption in NFCs (Shahzad, 2011; Azwa et al., 2013; Moudood et al., 2019; Dayo et al., 2020). Some models have been developed to take into account the moisture influence on mechanical behaviour of NFCs. For instance, Pan et al. have proposed a nonlinear constitutive model for unidirectional natural fibre reinforced composite considering the moisture absorption induced swelling and irreversible energy dissipations (Pan et al., 2014). In order to better understand the degradation phenomena occurring in NFCs submitted to moisture environment, it is necessary to characterise the corresponding damage and deformation mechanisms. Different types of techniques can be used to characterise damage and strain in NFCs: optical and electronic microscopy (Pickering et al., 2016; Malinowski et al., 2018), acoustic emission (De Rosa et al., 2009; Assarar et al., 2011), digital image correlation (Perrier et al., 2015; Unlusoy et al., 2019), ultrasonic scanning (Lebaupin et al., 2019; Fischer et al., 2019), infra-red thermography (Boccardi et al., 2018; Zhang et al., 2018) and X-ray micro-tomography (Rasket et al., 2012; Perrier et al., 2017). Figure 4 illustrates the ability of X-ray micro-tomography to analyse damage development in NFCs. The damage quantity after tensile failure has been measured in a ±45 woven hemp/epoxy composite for non-aged and water-aged specimens.

Results demonstrated that the relative volume of damage in the water-aged sample is more than twenty times higher than that in the non-aged specimen. This explains the degradation of the mechanical properties for NFCs after moisture exposure.



(a)	(b)	(c)

Figure 4. Damage analysis by X-ray micro-tomography in ±45 woven hemp/epoxy composite. (a) 3D reconstruction at initial state, (b) and (c) damage after tensile failure in non-aged and water-aged samples respectively.

1.2 Dynamic behaviour of natural fibre composites

As regards NFCs dynamic behaviour, fatigue and impact loadings are responsible for many, if not most, failures in engineering structures. Indeed, dynamic repetitive loadings (vibration, rotation, wind and wave action, turbulence, pressurization, etc.) at levels much lower than ultimate strengths result in sudden and catastrophic failure due to internal damage accumulation over a period of time. Impact loadings may be induced by falling foreign objects that can cause a drastic reduction in the composite mechanical properties. Understanding fatigue and impact behaviours of natural fibre composites is thus essential for the efficient and predictable design of load-bearing components.
In their review, Mahboob et al. have shown that fatigue testing parameters (e.g. frequency, loading ratio) and material variables (stacking sequence, fibre content) influence longevity (Mahboob et al., 2018). In the field of short fibre composites, Liber-Knec et al. have shown that addition of flax fibres resulted in improvement of fatigue strength of PLA-based composite (Liber-Knec et al., 2015) and Kanny et al. have studied fatigue life of sisal/PP composites (Kanny et al., 2013). Concerning mat reinforced polymers, it has been shown by Shahzad et al. (Shahzad et al., 2014) that hemp/polyester composites exhibit less fatigue sensitivity as compared to glass fibre composites, despite having poorer absolute fatigue strength. The same conclusion can be made for long fibre composites from the work of de Vasconcellos (de Vasconcellos, 2013). By considering absolute values, the woven hemp/epoxy composite is not at the level of glass/epoxy composites. But, as it can be seen in Fig. 5, there is a decrease in the rate of fatigue degradation during cyclic loading revealed by the differences in slopes of the S-N curves. The hemp/epoxy composite is less sensitive to fatigue loading than the glass/epoxy composites, which gives an additional advantage to the use of NFCs for industrial applications.

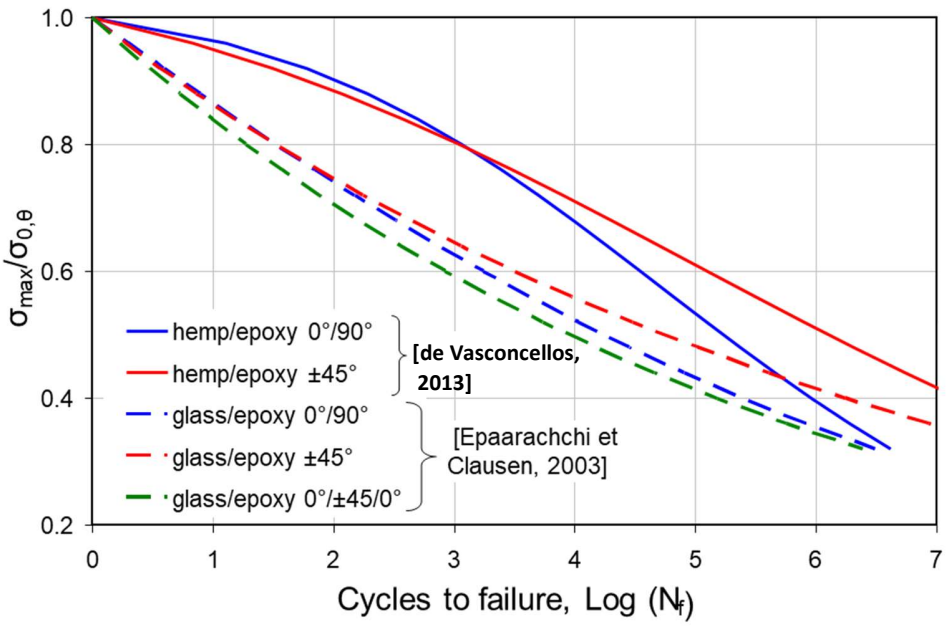


Figure 5. Comparison of fatigue S-N curves for a hemp/epoxy composite and a glass/epoxy composite.

The other type of loading that is crucial for engineering applications is the impact loading. There are many factors controlling impact strength properties of NFCs (Thomason et al., 2018; Al-Maharma et al., 2019). The impact velocity has a great influence on induced damage, as it has been shown by comparing damages created by a laser shock or by a falling dart impact in a hemp/epoxy composite (Touchard et al., 2017). In laser impacted samples, the induced damage is located near the back face while in mechanically impacted samples, damage appears close to the front face and propagates towards the back face. The natural fibre length plays also a significant role on the impact behaviour of NFCs. The comparison of impact damage in short, medium-length and long plant fibre composites has revealed that, despite identical damage mechanisms – i.e. interfacial debonding, matrix cracks and fibre failure -, damage quantity and distribution vary greatly in NFCs made of fibres of different lengths (Malinowski et al., 2018). Another influencing factor on impact properties is the stacking sequence in laminates. Fig. 6 clearly illustrates the differences in impact-induced damage for flax/PA11 laminates. This study has been performed in the frame of Lebaupin PhD work (Lebaupin, 2016). Micro-tomography images of flax/PA11 composites impacted at 3.6 J are presented in Fig. 6 for four different lay-ups: unidirectional $[0]_8$, cross-ply $[0/90]_{2s}$, sandwich-like $[0_2/90_2]_s$ and quasi-isotropic $[45/0/45/90]_s$. It is observed that laminates with fibres stacked in the same orientation such as in unidirectional or sandwich-like lay-up are seriously affected by mechanical impact. The induced damage is more significant in these lay-ups than in the two other configurations. This high quantity of damage allows dissipating a large amount of energy during impact loading for these two stacking sequences, but these lay-ups also exhibit poor impact resistance. On the opposite, the micro-tomography analysis shows that damage can be limited by introducing alternated plies or 45° plies in the stacking sequence. Indeed, the quasi-isotropic composite has the

smallest induced damage and the highest peak load. It is thus necessary to choose the most suitable lay-up, in terms of impact behaviour, for each considered industrial application.

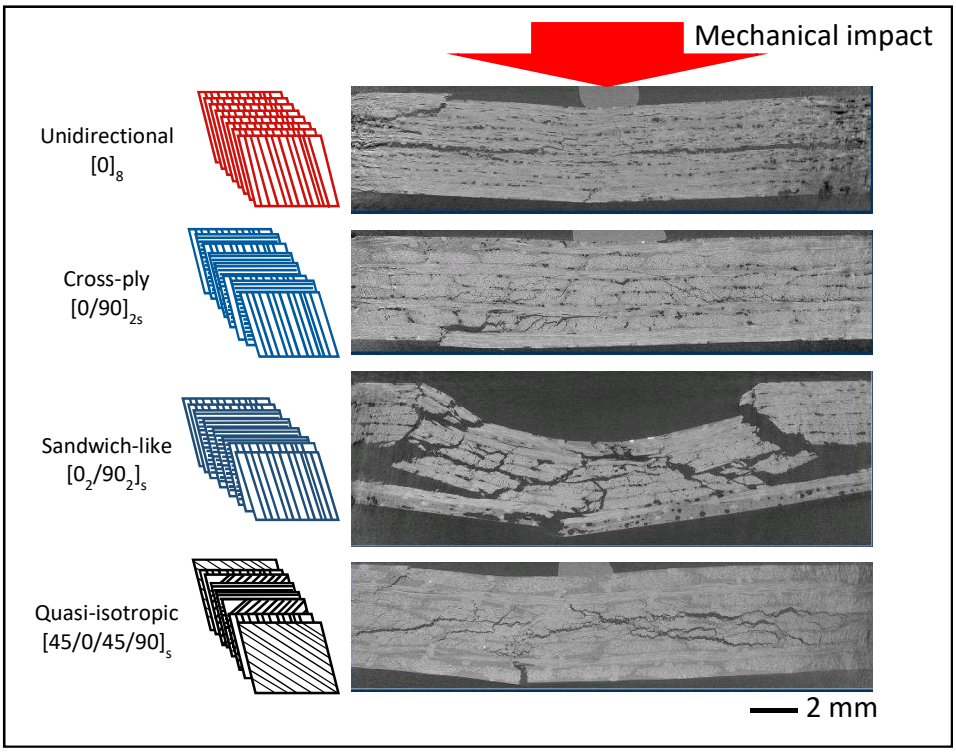


Figure 6. Micro-tomography images of flax/PA11 composites impacted at 3.6J for four different lay-ups: unidirectional $[0]_8$, cross-ply $[0/90]_{2s}$, sandwich-like $[0_2/90_2]_s$ and quasi-isotropic $[45/0/45/90]_s$.

It is also interesting to compare the impact behaviour of NFCs with other types of composites. For example, comparisons of hemp/epoxy with glass/epoxy composites (Touchard et al., 2017), and of flax/epoxy with basalt/epoxy ones (Seghini et al., 2019) have been performed. In both cases, plant fibre composites exhibit a lower peak reaction force value and a better energy absorption capability than glass or basalt fibre composites. The superior capability of NFCs to absorb energy during the impact loading can be related to the high damping properties of plant fibres and to the development of significant damage inside the composites.

All these results show that the mechanical properties of NFCs present interesting aspects compared with classical composites. However, in order to enhance the mechanical strength of NFCs, whether for quasistatic or dynamic loadings, a crucial issue is the need to improve the adhesion quality at the fibre-matrix interface.

2. Interface

As highlighted above, the properties and performance of a composite rely on its components namely the polymer matrix, the reinforcing fibre and the interface. Latter term has to be defined more precisely since the transition from the fibre to the matrix is not abrupt but encompasses an interphase. The interfacial region controls the stress transfer between the matrix and the reinforcement, which depends on the adhesion level as detailed in the next sections. The improvement of the mechanical properties of the composite may require the pre-treatment of the natural fibre as discussed in section 2.4.

2.1 What is an interface?

The interface appears between the two components of the NFCs namely the fibre and the matrix. The stiff and strong fibres reinforce the polymer matrix on a level depending on the adhesion between both. Adhesion or bonding results from molecular interdiffusion, electrostatic forces, chemical reaction and mechanical interlocking, one of them being dominant (Jacob, 2005 et al.; Zhou et al., 2016; Latif et al., 2018). As emphasised above, the transition from the fibre to the matrix is not abrupt but consists of a heterogeneous intermediate material. This is particularly evident in Fig. 7a where entanglement of fibre and polymer molecules is observed. The transition region or interphase possesses none of the properties of the fibre and the matrix. The nature of the interphase varies with the specific composite system as illustrated in Fig. 7. It is important to note that the interphase is not a distinct phase as it has no clear boundaries. Its extent is difficult to assess although nanoindentation tool has allowed significant progress as discussed in section 2.3.1. It is believed to be of the order of a few µm. A strong interface allows an efficient stress transfer from the matrix to the fibre and hence higher stiffness and strength values for the composite. However, upon fracture a weaker interface would allow deviation and dissipation. The interfacial strength can be measured by a group of techniques discussed in the next section.

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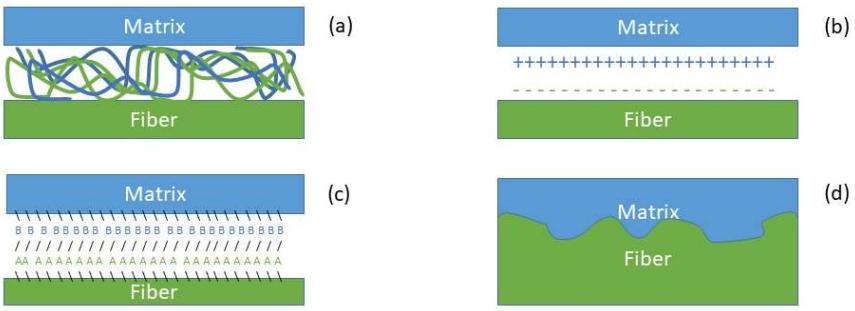


Figure 7. Fibre-matrix interface with (a) molecular inter diffusion, (b) electrostatic bonding, (c) chemical bonding , and (d) mechanical interlocking.

2.2 Testing an interface

The main mechanical characterization techniques used for interface fall in three main categories. In a single fragmentation test, a fibre is embedded in a dogbone matrix specimen and pulled until fragmentation reaches a saturation (Fig. 8a) (Jacob et al., 2005; Zhandarov et al., 2005; Zhou et al., 2016). The critical fragment length that can break, l_c, allows determining the interfacial shear strength τ as

$$\tau = \sigma_f d/2l_c \qquad \text{Eq. 1}$$

where d is the fibre diameter and σ_f the fibre strength. The second category of test also considers either a single fibre being embedded in a matrix block or in a droplet (Oushabi, 2019). In the first case, the fibre is pulled out, the force F required allowing the determination the fibre-matrix shear strength τ (Fig. 8b):

$$\tau = F/\pi dl \qquad \text{Eq. 2}$$

where l is the embedded fibre length. In the case of the microbond test with an ellipsoidal droplet (Fig. 8c), the force required to debond the fibre against the scissors allows a similar value to be determined with l being the embedded fibre length in the droplet.

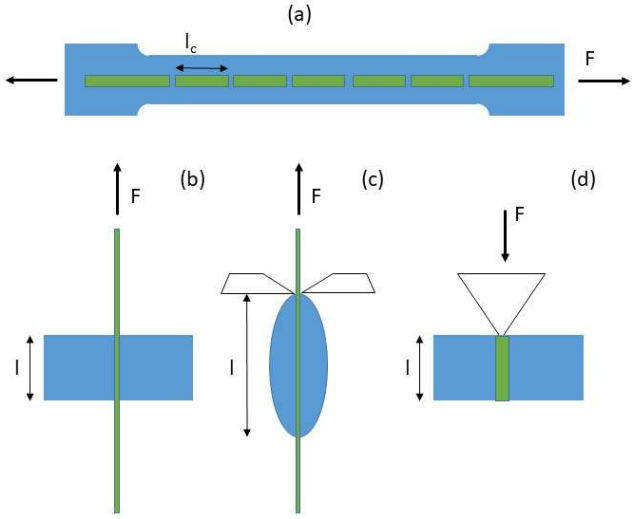


Figure 8. Micro-mechanical techniques to measure the strength of fibre-matrix interface (a) single fibre fragmentation, (b) single pull-out test, (c) microbond test, and (d) micro-compression.

It should be noted that one can pass from one equation to the other (Eq. 1 to Eq. 2 and vice versa) when assuming a uniform stress distribution at the interface, a uniform fibre diameter as well as a uniform wetting of the fibre by the matrix (dogbone, block or droplet). So far, tests show large difference in the results of shear strength τ when considering the two main approaches. This is attributed to different stress distribution, due to variable specimen geometry, different behaviour at failure and the friction occurring between the fibre and the matrix during a fragmentation and a pull test. In practice, higher values of interfacial strength are obtained with the pullout test than with the fragmentation test (Graupner et al., 2014). In the last category (Fig. 8d), we find the indentation approach where a diamond

tip is used to push out the fibre. In contrast to the other approach above, actual composite specimens can be used as long as they can be polished carefully without damaging the interfaces. However, indentation push-out has limitation as it is dependent on the assumption made to describe the stress field around the fibre when also the surrounding fibres cannot be neglected. While this method works well with well-defined glass fibres, it becomes tedious with natural fibre bundles. So far, we shall see that using nanoindentation the interphase can be investigated (section 2.3.1).

2.3 Example with hemp-epoxy interface

The development of woven hemp/epoxy composites for semi-structural applications requires a better understanding of the adhesion mechanisms at the interface between the hemp yarns and the epoxy matrix. In woven composites, yarns are close and interfere with each other. To avoid such influence, single yarn composites can be considered. Different types of analyses have been performed on such composites and are presented below: measurement at the interface of the evolution of mechanical properties, using nanoindentation and Digital Image Correlation techniques, and determination of the interface adhesion quality, by means of laser shock technique and fragmentation tests.

2.3.1 Evolution of the nanoindentation response at the interface

Interfacial region rules the mechanical transfer between the polymer matrix and the fibre reinforcements. This region or interphase is difficult to scrutinise, its extension depending upon the system. Nanoindentation offers an ideal probe of this interfacial region (Le Bourhis, 2014; Gibson, 2014). Loading-unloading procedure can be adapted to the length scale, the polymer and fibre response while the automatic displacement table offers the possibility of scanning and mapping the region. A single hemp yarn epoxy composite has been elaborated to scan mechanically the interfacial region (Perrier et al., 2016). Fig. 9 shows a series of Berkovich indents made with a displacement step of 5 µm. The hemp yarn is constituted by several elementary fibres, which can be individual or grouped in bundles (Fig. 9).

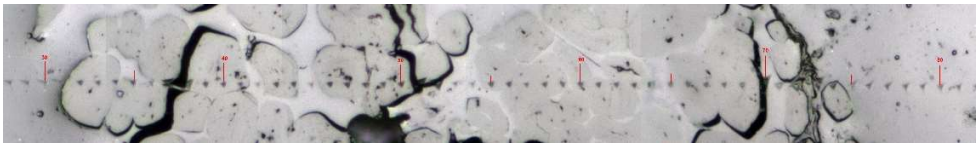


Figure 9. Nanoindentation scan made in a single hemp yarn epoxy composite as observed by optical microscopy. The distance between indents is 5 µm.

Each loading-unloading curve allows to extract locally the elastic response in terms of reduced modulus (Le Bourhis, 2014). A strong contrast of reduced modulus is obtained between the fibres and the matrix with a factor of more than three between both. The data are rationalised once plotted as a function of the distance from fibre edges (Fig. 10). Such a representation allows detecting the intermediate region between the fibre and the epoxy matrix. In the present case, a progressive change of the elastic response is detected with an interphase region being about 2 µm in size. The interphase might be difficult to detect as illustrated in the case of cellulose fibre reinforced polypropylene (Lee et al., 2007).

These authors emphasise that the convolution of the probe with the interface is to be considered carefully. It is interesting to note that Nair et al. also worked on cellulose fibre reinforced PP composites (Nair et al., 2013). The interphase region in the specimen was observed to be dependent on the matrix used. A 0 % maleated polypropylene (MAPP) showed a narrow interphase with a steep modulus gradient in, while the use of MAPP significantly increased the interphase thickness, resulting in a more gradual change in modulus from fiber to matrix. As discussed in more detail in section 2.4, maleated matrices offer indeed improvement of mechanical properties of related composites.

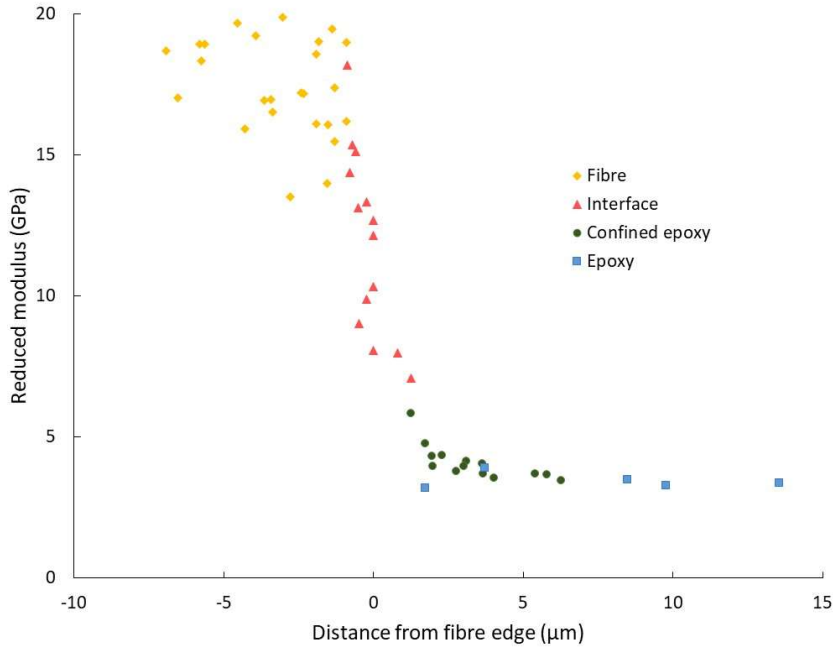
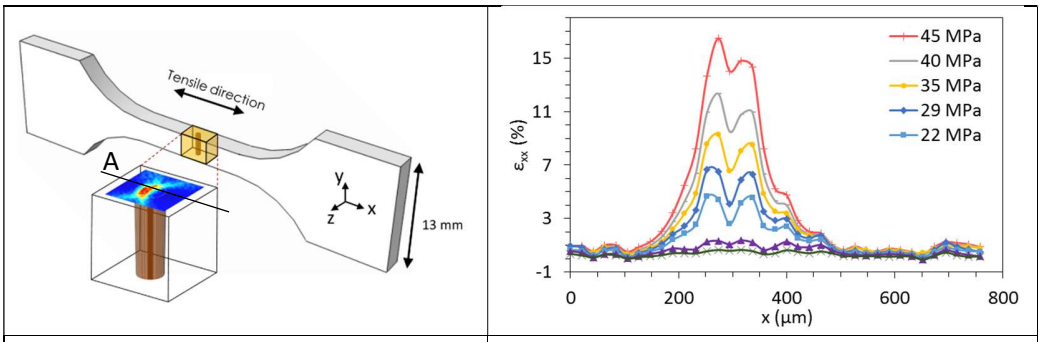


Figure 10. Reduced modulus for hemp/Epoxy composite versus distance from fibre edge (set to 0) as extracted from the nanoindentation loading-unloading curves. The transition is not abrupt but reveals an interphase.

2.3.2 Strain fields measured by Digital Image Correlation (DIC)

The principle of DIC technique is based on a unique random pattern, recorded twice, once before loading the sample and once after the sample is deformed. The first picture taken at the initial state is the reference picture, which is compared to a second one taken at a deformed state. The first picture is divided into small sub-windows, each one being a measurement point characterised by its grey level distribution. The analysis consists in seeking, for each sub-window of the reference image, the most similar one in the deformed image (in terms of spatial distribution of grey levels), using a correlation function. In this study, single hemp yarn specimens with hemp yarn oriented at 90° in regard to the tensile direction were prepared by laying a mixture of paint and particles of 200 nm diameter (Fig. 11a). This made it possible to obtain a fine random pattern on the material surface. The thickness of the paint layer was

about 33 µm. In order to perform measurements with high spatial resolution, tests were carried out with a micro tensile tester placed under an optical microscope (Perrier et al., 2016).
Fig. 12 shows longitudinal strain maps measured on the edge of a single yarn composite, with the yarn tilted of 90°. For the different applied stress levels, the same colour code has been used; in this way, the evolution of strain is easily showed. The measurements show that strain distributions are highly heterogeneous, with their values increasing with the applied stress level. Strains are localised inside the section of the yarn (represented by the dotted circle). For an applied stress of 45 MPa, which corresponds with 2.31 % of macroscopic strain, the maximum strain value measured by DIC is nearly 17 %.



a) b)

Figure 11. a) Single yarn composite samples with hemp yarn oriented at 90° in regard to the tensile direction. b) Longitudinal strain values along line A measured by DIC at different applied stress levels on the edge of 90° oriented yarn specimens in a single hemp yarn/Epolam sample.

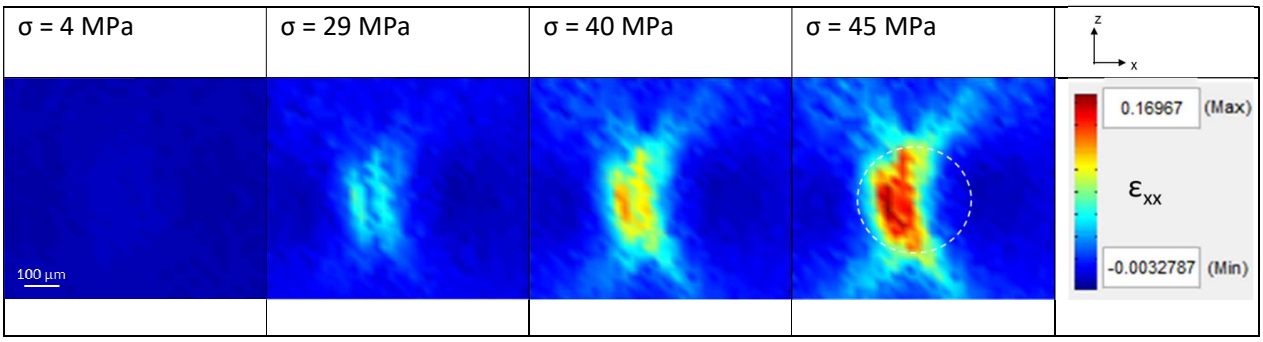


Figure 12. Evolution of strain fields ε_{xx} obtained by DIC versus the applied stress on the edge of a 90° oriented single hemp yarn/Epolam sample.

The evolution of the measured strain can be more accurately described by plotting in a same graph the strain values for different stress levels of the specimen during a tensile test. Fig. 11b shows the longitudinal strain values along the line A for different applied stress levels. It can be

seen that the strain increases progressively and is localised at the yarn section. These results allow to investigate the local strain mechanisms at the yarn/matrix interface, showing the strong heterogeneities in strain fields which develop at the interface.

2.3.3 Interface bonding quality tested by laser shock

The laser shock wave method can create a short but intense internal loading into the shocked sample. Thanks to the wave transmission/reflection through the irradiated material, localised tension in the loading direction can be generated. The laser shock adhesion test (LASAT) technique can be used for example to test the adhesion quality of bonded composite materials (Ecault et al., 2016).
This method has also been used in order to study the adhesion between a single hemp yarn and an epoxy matrix (Perrier et al., 2016). Samples were made of a single hemp yarn embedded in epoxy resin. Each specimen was cut into square samples, with dimensions of about 10 x 10mm^2 and thickness about 3 mm. In these samples, the single yarn was centered in the x direction and positioned along the z direction closer to the back face than to the front face (Figure 13). Front faces were covered with aluminum paint.

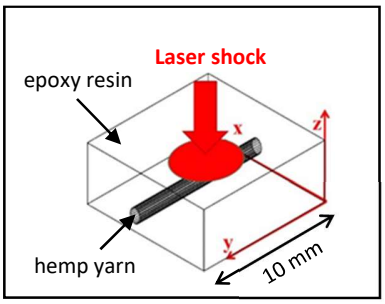


Figure 13. Composite samples for laser shock tests: single hemp yarn embedded in epoxy resin.

After the shocks, all samples were recovered and damage micromechanisms have been analysed by microscopic observations. With transmitted light, it was possible to observe single-yarn samples through the matrix (Fig. 14). Two different types of damage induced by laser shock have been observed: resin cracks appear only for high laser intensity levels, and specific cone-shaped interfacial damage appears for lower intensity values. At the yarn-matrix interface, the observed micro-damage is quite unusual: it initiates at one interfacial point, probably a small defect at the interface, and propagates from the yarn surface to the resin. The central axis of the resulting three-dimensional cone shaped cracks is perpendicular to the yarn. It has been observed that this interface damage appears above a given laser intensity value, which corresponds to the damage threshold of the interface. Its quantity and size increase with the laser intensity levels.
The second type of damage consists of large cracks in the resin, localised between the yarn and the specimen back face, as shown in figure 14. These cracks are due to the boundary conditions induced by the sample holder, leading to a bending phenomenon in the specimen for high laser intensity level. As for interface damage, these resin cracks appear above a given level of the laser intensity, corresponding to resin damage threshold. These damage thresholds, given in terms of laser intensity levels, are directly linked to the corresponding stress values induced in the material by the shock wave propagation.

Therefore, it allows characterising the mechanical adhesion quality at yarn/matrix interface and the resin damage threshold. It has been determined that hemp/epoxy interfacial damage starts at 0.31 GW/cm² and resin cracks start at 0.57 GW/cm² (Perrier et al., 2016). A numerical simulation by finite elements has also been performed to get further insight into laser shock wave propagation in such samples.

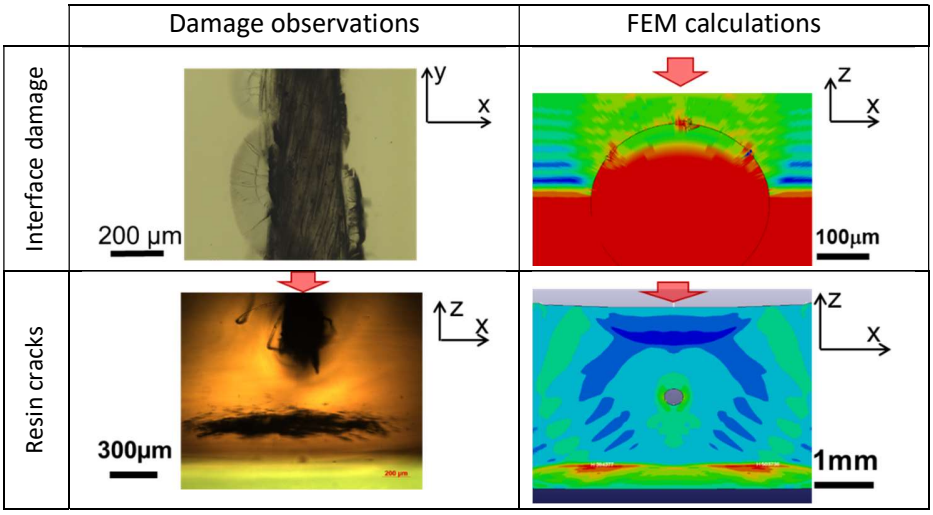


Figure 14. Damage observations in single hemp yarn/epoxy composite samples after laser shock tests and corresponding σ_{zz} fields obtained by FEM calculations.

The qualitative finite element model (FEM) represents a slice of a sample in the (x-z) plane, with a thickness of one element of 1 µm. The diameter of the hemp yarn section is 300 µm. The laser shock has been simulated by a loading with a Gaussian-like shape in time. As observed in Fig. 14, FEM calculations lead to three damaged zones at the yarn/matrix interface, which can be related to the micro-damage experimentally observed. Moreover, calculations also show an area of overstresses σ_{zz} near the sample back face, corresponding to the observed resin cracks. These preliminary results demonstrate the ability of the laser shock test to study and quantify the mechanical quality of yarn/matrix interface, which is needed to help design of such composites.

2.3.4 Fragmentation tests

Fragmentation tests are usually performed on elementary glass or carbon fibres embedded in polymer matrix (Zheng et al., 2020; Yao et al., 2017; Swaminathan et al., 2018). Nonetheless, this test method applied to natural fibre composites suffers from several limitations. The length of elementary hemp fibres, about 15 mm, is very small for elaborating a dogbone composite specimen. Moreover, in structures made of woven hemp composites, the load-bearing interface is the yarn-matrix interface. Therefore, the interfacial adhesion should be assessed at the yarn scale. A special mould was developed

to elaborate composite samples with a single hemp yarn aligned in the loading direction. Single yarn composite specimens were manufactured with resin injection moulding technique using EPOLAM 2020 epoxy resin from Axson. The specimens were dogbone shaped, with a total length of 50 mm. When applying a tensile loading to these samples, the load is transferred through the matrix into the yarn by means of shear stress at the interface. Yarn failure occurs when this transferred stress reached the tensile strength of the yarn. The yarn will continue to fracture into shorter length fragments as the load increases, until the yarn fragments are so small that the tensile stresses induced in the yarn can no longer reach the yarn tensile strength. At this point, a state of saturation is reached, and the fragmentation process ceases. It is thus possible to measure the fragment lengths, l_f, by optical microscopy and to determine the corresponding critical fragment lengths (Eq. 3) (Ohsawa et al., 1978):

$$l_c = \frac{4}{3}\, l_f \qquad \text{Eq. 3}$$

An example of hemp-epoxy sample after fragmentation test is presented in Fig. 15. At the end of the test, after the sample failure, several fragments are visible in the gauge length.

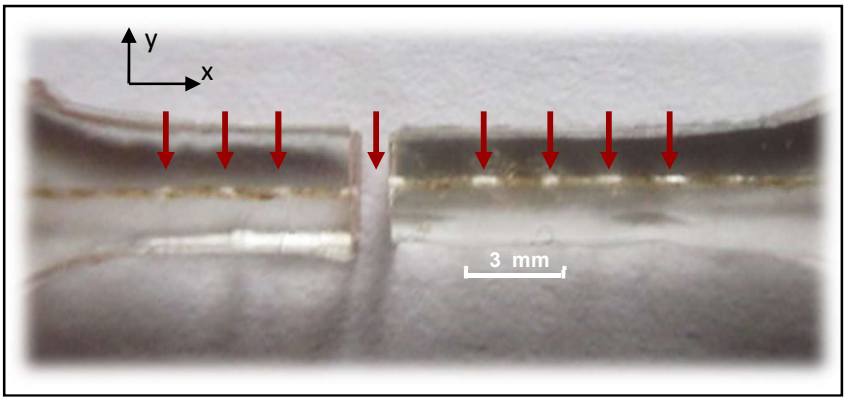


Figure 15: Single hemp yarn-epoxy composite sample after fragmentation test.

The mean value of l_c determined for hemp/epoxy interface is 3.0 ± 0.15 mm, which gives a mean interfacial shear strength (τ) value (Eq.1) of 14.4 ± 4 MPa (Barbiere et al., 2018). Moreover, X-ray micro-CT observations with a very fine resolution (1.5 µm per voxel) allowed the observation of interfacial debonding in the vicinity of the yarn fragment extremity (Fig. 16).

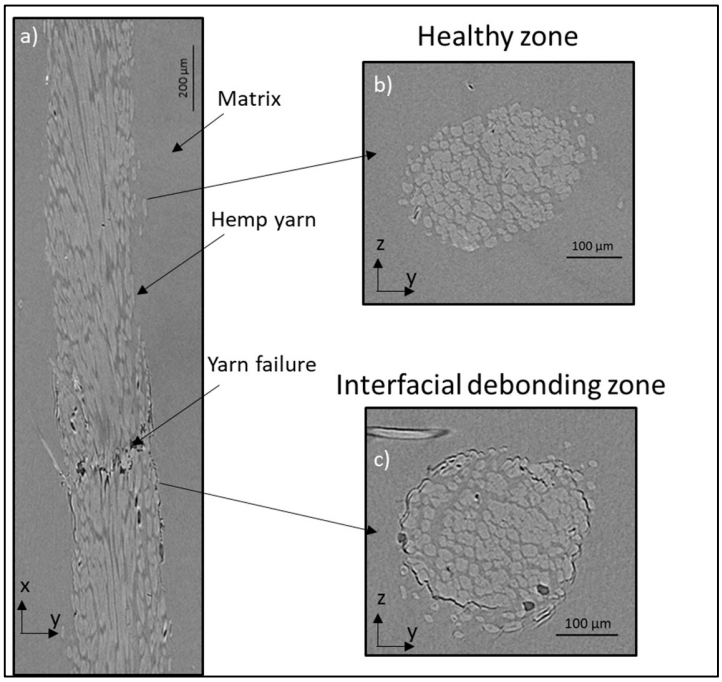


Figure 16: Micro-CT observations of a single hemp yarn-epoxy composite sample after fragmentation test in the vicinity of a yarn fragment extremity: a) longitudinal view of a yarn failure zone, b) cross section in the healthy zone, c) cross section in the debonding zone.

Two different zones are revealed in Fig. 16. The healthy zone, far from the yarn fragment extremity, where the cross-section shows that the interface is undamaged (Fig. 16b), and the debonding zone, near the yarn fragment extremity, where damage is present all around the yarn (Fig. 16c). These observations confirm that the load-bearing interface is the yarn/matrix interface.

2.4 Fibre surface treatments

Natural fibres are mostly formed by cellulose, hemicellulose and lignin (Kabir et al., 2012; Liu et al., 2017). Cellulose fibres are highly polar materials and their compatibility with very apolar matrices is highly problematic. Furthermore, due to their polar nature they are very hydrophilic materials, which also poses restrictions on their use in some applications (Zafeiropoulos et al., 2007). A possible solution is to improve fibre-matrix interface by using compatibilizers and adhesion promoters (Shahzad, 2012). Their hydrophilic character is a major drawback as it represents the risk of lesser interfacial contact with the polymer matrix, water uptake on long duration use, microorganism growth (Liu et al., 2017). Those deleterious processes affect the long-term applications of the NFCs and have to be prevented. The hydrophilic character has its origin in the presence of hydroxyl (–OH) groups on the cellulose component of the fibre and carboxylic acid (–COOH) on the hemicellulose one. Treatments using mercerization, silanization, acetylation, allow reacting with or replacing the (–OH) groups producing fibres more resistant and more compatible to the polymer matrix (Kabir et al., 2012; Mohit et al., 2018; Oushabi,

2019). Moreover, the hydrophilic character being modified, natural fibres are better wet by the hydrophobic polymer matrix. Oushabi reviewed the literature and showed that the pull-out interfacial shear strength was largely improved upon such treatment regardless of the fibre-matrix system (Oushabi, 2019). Interestingly, this improvement is shown to be correlated with SEM revealed matrix residues observed on the fibre after the test (Oushabi, 2019). On the other hand, maleated matrices like polyethylene or polypropylene (MAPE & MAPP) offer a better coupling to the natural fibres (Latif et al., 2018; Oushabi, 2019) correlated with the presence of a thicker interphase region (Nair et al. 2013). In the work of Seghini et al., it has also been demonstrated that oxygen and tetravinylsilane plasma treatments allow enhancing the adhesion of flax yarns with epoxy matrix (Seghini et al., 2020). By using fragmentation tests on single flax yarn composite samples, it has been shown that flax yarns with pp-TVS deposition after a 100 W plasma oxygen pretreatment lead to a τvalue twice as large as the one obtained with untreated flax yarns. Overall, these approaches promote interfacial adhesion, fibre dispersion, load transfer. High quality composites can then be obtained (La Mantia, 2011).

3. Conclusion and prospects

Composite materials have been considerably developed in search of weight gain. Conventional composites become challenged by always improving natural composites. Also, immense applications of NFCs are being opened in the framework of sustainability. NFCs represent strong alternatives of conventional composites used in different important industries. Applications cover transportation, infrastructures and packaging. Some of them are very demanding and it is important to assess the mechanical performance notably the toughness and fracture behaviour of these composites. A particular attention has been paid to quasistatic and dynamic (notably fatigue) behaviours where NFCs present interesting performances compared with conventional composites. The main drawback of the use of natural fibre remains its hydrophilicity, which is being circumvent thanks to preliminary treatments. Such treatments allow improving fibre durability against moisture as well as wettability of the polymer matrix. The obtained NFCs show stronger interface and mechanical performance. Albeit these promising aspects, green composites based on natural fibre still need to undergo fundamental investigations to assess their capabilities and potential as conventional composites replacement. The research field is very active as illustrated in the review and will further open new opportunities for NFCs applications.

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