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Development of novel non-woven *Triumfetta cordifolia* **bast fibers and polylactide fibers biocomposites**

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Abstract

This study focuses on the development of needle-punched nonwoven composites reinforced with a *Triumfetta cordifolia* (TC) bast fiber based on a polylactide (PLA) matrix. Flax and hemp fibers, already known for their use in automotive applications, are used as a reference for comparison purposes. TC bast fibers from the equatorial region of Cameroon were extracted by a water retting process. Nonwoven felts were obtained by mixing TC fibers with PLA (50:50 weight ratio) through carding-napping process. The composite materials were obtained by thermocompression of the nonwoven felts with two thickness ranges (2 mm and 3 mm). The results obtained in tensile and flexural tests showed that composite materials with an optimal thickness of 2 mm displayed better mechanical performances. The anisotropy of the mechanical properties in traction and flexion of these composites was highlighted. It was found that the tensile and flexural mechanical properties in the transverse direction (CD) are always superior to those in the machine direction (MD). Compared to flax or hemp nonwoven-reinforced biocomposites, TC fibers-based nonwoven biocomposites showed slightly lower maximum tensile and flexural stiffness values of 2882.7 MPa and 3908.6 MPa respectively. Thermogravimetric analysis revealed that the addition of the plant fibers increased the thermal stability of PLA and the ash content to 8%. TC nonwoven fiber reinforcement composites can be used in the automotive interior.

Keywords: Bast fiber, *Triumfetta cordifolia* fiber, polylactic acid, carded-napped non-woven, biocomposites.

1. Introduction

The awareness of ecological, social and economic crises has prompted researchers to develop new low-cost and environmentally friendly materials by incorporating natural fibers (jute, flax, hemp, Kenaf) as reinforcements for compositess, especially for car interior components in the automotive industry (Lee et al., 2009; Alves et al., 2010; Merotte et al., 2016; Carus and Partanen 2017; Ishikawa et al., 2018 ; Hadiji et al., 2020; Elseify et al., 2023). The attractiveness of natural fibers as reinforcement materials in such applications is motivated by their various advantages, including low cost, low specific mass, flexibility in processing, competitive mechanical properties, recyclability, workability, safer working conditions, and above all their ecological aspect (Segovia et al., 2016; Komal et al., 2019; Manaii et al. 2019; Jawaid et al., 2020; Ndengue et al., 2021). However, ~ 80% of industrial plant fibers are obtained from only three crops namely jute, flax, and coir (Elseify et al., 2021).

Triumfetta cordifolia (TC) is a fast-growing emerging tropical crop traditionally used to produce ropes. It could be produced in large quantities to increase the global availability in fibers, while being beneficial to the local economies. Previous studies, including our own preliminary work, have demonstrated the potential of this fiber for composite applications (Snewitz et al., 2016; Mewoli et al., 2020). Grosser et al. (2018) showed that a polylactide (PLA) matrix reinforced with unidirectionally arranged TC fibers exhibited mechanical properties comparable to those of PLA/hemp composite. Njom et al. (2022) developed green hybrid composites by combining bleached TC and bagasse fibers with a natural rubber (NR) matrix. The authors showed that the combination of two fibers improves the physical and mechanical properties of the NR matrix and these hybrid composites are good candidates for engineering applications.

As an alternative to glass fibers, composites with non-woven reinforcements of natural fibers combined with polymer fibers (polyester, polypropylene or PLA) are widely used especially in automotive interior components since they combine lightness, flexibility, interesting specific mechanical properties, acoustic efficiency and a good cost/performance ratio (Behlouli et al., 2016; Renouard et al., 2017; Baley et al., 2019; Zhang et al., 2019; Thilagavathi et al. 2019). Their good specific mechanical properties have been highlighted in many applications (Hadiji et al., 2020). For example, Lee et al. (2009) showed that composites reinforced with carded-needle PLA/kenaf (50/50) nonwovens can be successfully used to make automotive headliners. Chocinski-Arnault et al. (2013) investigated the mechanical and damage behavior of nonwoven composites with flax yarn and thermoplastic matrices polypropylene (PP), PLA and polyamide 11 (PA11). The authors showed that in addition to the anisotropic behavior, the type of matrix used influences the stiffness of the composites exhibit anisotropic behavior to the reinforcement, the type of matrix used has an influence on their stiffness. Alimuzzaman et al. (2013) fabricated flax fiber reinforced PLA nonwoven biocomposites through the air-laid route, the authors showed an increase in mechanical properties and thermal stability of the biocomposites with the addition of flax. Kandola et al. (2018) studied the mechanical performance and fire resistance of biocomposites and compared them to similarly produced glass fiber reinforced PP and PLA composites for automotive applications. They showed that the tensile and flexural moduli of PLA composites were higher than those of PP composites and that composites with PLA have a lower propensity to burn than PP composites. Tse et al. (2018) highlighted the flexural behavior of hybrid composites with recycled carbon fiber staple (FCr) nonwovens mixed with flax and PLA fibers as a matrix (FCr/flax/PLA) fabricated by wet process. Their results showed that the mechanical properties of the composite in bending increase in contrast to composites reinforced only with carbon fibers. The above-mentioned works on natural fiber reinforced nonwoven polymer composites were mainly interested in the study of their mechanical properties. Due to these good properties cited above, nonwoven biocomposites are a major asset for the automotive industry and for a variety of applications (Baley et al., 2019; Gager et al., 2019a).

Based on this literature review (Fig.1). this paper examined the potential of TC fibers as reinforcements for nonwoven composites. The TC fibers and PLA fibers were mixed, carded, napped, and consolidated by needling into nonwoven mats before curing by thermocompression. Flax and hemp fibers were also processed in the same conditions for comparison. All biocomposite panels (PLA/TC, PLA/linen and PLA/hemp) were manufactured with a reinforcement/matrix weight ratio of 50:50. Tensile and three-point bending measurements were performed on these biocomposites and their behavior was compared. The thermal stability and degradation of the biocomposites were evaluated by thermogravimetric analysis (TGA).

2. Materials and methods

2.1. Raw materials

Three types of plant fibers are used in this work: Triumfetta cordifolia bast fibers collected in Cameroon and extracted by water retting according to the method used in the literature (Mewoli et al., 2020) and cut by a cutter set to a length of 120mm; flax fibers (Linum usitatissimum L.) are provided by Argylin (Normandy, France) and hemp fibers provided by the CETELOR (Epinal, France). These different fibers are used as reinforcement in the nonwoven with polylactide fibers (PLA INGEOTM Biopolymer type SLN 2660D) as matrix. They are supplied by Max Model SAS (France), they have an average length of 64 mm, an average diameter of 6 Dtex and a density of 1.24 g/cm3. Table 1 shows the properties of the plant fibers, non-woven felts.

Table 1

Physical and mechanical properties of plant fibers and nonwoven reinforcements used

2.2. Manufacture of non-woven biocomposites

The manufacturing process of the biocomposites is illustrated in Fig. 1. First, using the cardingnapping-needling technology of CETELOR (Epinal, France), the needle-punched nonwoven structures were elaborated from the mixture of PLA/TC, PLA/flax and PLA/hemp fibers. The vegetable fibers and the thermoplastic polymer fibers of PLA are introduced in a ratio weight of reinforcement/matrix of 50:50 in the Laroche opening machine at a speed of 1800 rpm, where they are individualized and mixed in order to be conveyed in a loader and then in the carding machine of Bonino brand which makes it possible to obtain homogeneous webs. These undergo at the exit of the carding machine, a topping where they are piled up to form a web of webs of a certain mass density followed by a consolidation by needling of 150 blows/cm2, which leads to the non-woven structure. It should be noted that the orientation of the needle-punched web is close to a 90° angle with respect to the machine direction (MD) of the reinforcing roll.

In a second step, the biocomposites are shaped by thermocompression, on a branded hydraulic press (LabTech Scientific 50T) at EcoTechnilin SAS (Normandy, France) from superimposed nonwoven felts to reach a mass per unit area of 1500 ± 50 g/m², then hot-pressed at 200° C and 40 bars for 180 seconds before being cooled down at 50 bars for an additional 40 seconds to obtain rigid sheets of 250 x 200 mm² dimensions. Two thicknesses (2 and 3 mm) were fixed by the use of wedges.

Fig. 1. Process for the manufacture of biocomposites.

2.3. Measurement of density, fiber density fraction and porosity rate

To calculate the constituents of the nonwoven, the dichloromethane (DCM) resin digestion method (PLA) in accordance with the literatures of Alimuzzaman, (2014) and Akonda et al. (2018) was used. The anhydrous mass of the sample was determined before and after matrix digestion. Non-woven felt (PLA/plant fiber) specimens were soaked in a bath containing 100ml DCM at room temperature to digest the matrix. Sintered glass crucibles, oven, desiccants, DCM, conical flasks, and a balance were used in this experiment. For these experiments, the average values were calculated from four measurements and considered as the final value. The fiber content, void content, and volume fraction of plant fibers were calculated using the following equations (1) - (6):

$$
W_f(\%) = \frac{m_3 - m_1}{m_2 - m_1} \times 100\tag{1}
$$

Where, W_f is the fiber content as a percentage of the initial mass; m_I is the sintered glass crucible (g); m_2 is the initial mass of the sample and glass crucible (g); m_3 is the final total mass (g) of crucible and residue after digestion.

$$
W_r(\%) = 100 - P_f \tag{2}
$$

To measure the density of the biocomposite, the calculation was based on the assumption that the mass of the final plate is the sum of the mass of the fibers (reinforcements) and the resin. The total volume of the biocomposite was realized with the measurements of the plate dimensions ($V_c = L \times l \times l$ t). V_c : total volume of biocomposite in (m³); L: length of biocomposite plate in (mm); l: width of biocomposite plate in mm; t : thickness of biocomposite plate in (mm). The density of the biocomposite is given by equation (3):

$$
\rho_c = \frac{m_c}{v_c} \tag{3}
$$

Where, ρ_c is the density of the biocomposite m_c ; is the mass of the biocomposite weighed in (g) and V_c total calculated volume in m³.

$$
V_f = W_f \times \frac{\rho_c}{\rho_f} \tag{4}
$$

Where V_f is the fiber content as a percentage of the initial volume, W_f is the fiber content as a percentage of the initial mass, ρ_c is the density of the test sample (g/m³), ρ_f is the density of the flax, hemp and *TC* fibers $(g/m³)$.

$$
V_r = (100 - W_f) \times \frac{\rho_c}{\rho_f} \tag{5}
$$

Where, V_r is the PLA content as a percentage of the initial volume; W_f is the fiber content as a percentage of the initial mass; ρ_c is the density of the test sample (g/m³); ρ_r is the density of PLA (g/m^3) .

The porosity rates are determined on the composite materials after thermocompression using equations (6).

$$
\Phi = 100 \left[W_f \times \frac{\rho_c}{\rho_f} + (100 - W_f) \times \frac{\rho_c}{\rho_r} \right]
$$
\n(6)

Where, ϕ is the void content as a percentage of the initial volume, W_f is the fiber content as a percentage of the initial mass, ρ_c is the density of the specimen (g/m³), ρ_f is the density of the flax, hemp and TC fibers (g/m³), ρ_r is the density of PLA (g/m³).

2.4. Mechanical characterization of the biocomposite

The tensile and bending behavior of PLA biocomposite materials was studied in both directions of the reinforcement, MD (Machine Direction) and CD (Cross Direction) as well as the influence of the thickness of PLA biocomposites reinforced with TC, linen and hemp fibers.

2.4.1. Traction

Static tensile tests were performed on the materials using an INSTRON model 4206 testing machine equipped with a 100 kN load cell with a crosshead speed of 2mm/min and a nominal length of 150mm between the jaws. The tests were performed on specimens in a controlled atmosphere (23°C and 50% RH), according to the ISO 527-4 standard. The tensile modulus was calculated in the linear part of the stress-strain curve, at the beginning of the load between 0.05% and 0.1%.

2.4.2 Three-point bending

Three-point bending tests were performed in accordance with BS EN ISO 14125 to determine the bending behavior. Five specimens of each sample were tested on an INSTRON model 4301 with a 30 kN load cell at a crosshead speed of 2 mm/min. The deformation in the middle was given by the displacement of the center load element, and the specimen dimensions were 100×25 mm2 with a span ratio of 1:20. All tests were performed at a temperature of $23 \pm 2^{\circ}C$ and a relative humidity of $50 \pm 5\%$. Modulus of elasticity in bending, E_f was calculated in the linear part of the stress-strain curve, at the onset of loading between 0.05% and 0.25%.

2.5. Examination of the microstructure by scanning electron microscopy (SEM)

Scanning electron microscope observations were performed on cross sections of nonwoven composites and a fracture face examination was performed on tensile fractured specimens to examine the probable adhesion of the matrix-fiber interface. The specimens were coated with a thin layer of gold by sputtering with an Edwards metallizer before being analyzed with a HITACHI TM 3000 Scanning Electron Microscope (SEM) with an accelerating voltage of 15 kV.

2.6. Thermogravimetric analysis (TGA)

A NETZSCH STA 449 F3 Jupiter analyzer was used to measure the stability and thermal decomposition of plant fiber/PLA biocomposites. Approximately 30±1 mg of milled fiber sample powder was placed on the platinum stage and heated with a temperature ranging from 20°C to 600°C at a heating rate of 20 K.min-1 in a nitrogen atmosphere.

3. Results and discussions

3.1 Microstructure of materials

Table 2 shows the main characteristics of the PLA/TC, PLA/linen and PLA/hemp composite plates produced in this study. In the SEM images of the cross sections of the materials (Fig. 2), it is possible to see the presence of pores that are mainly located in the matrix and at the fiber/matrix interface. These pores are formed during the consolidation of the nonwoven structure by needling (Gager et al., 2019a). Indeed, during this process, the needles in their penetration movements form bonding points (knots and perforations) in the nonwoven structure which leaves voids in the latter. Moreover, the random orientation of the fibers in the nonwoven composite can also impede the flow of the matrix or trap air (Tse et al.,2018).

At low thickness T2 (porosity level $\phi = 40\%$), the pores did not appear connected and the fibers are well impregnated into the PLA with good fiber/matrix adhesion as shown in Fig. 2 (d, e, and f). At a thickness T3 (at more than $\phi = 60\%$ porosity) Fig. 2 (a, b, and c), the pores appeared larger and connected to each other which consequently generates a poor impregnation of some fibers. This leads to a decrease in the quality of the fiber/matrix adhesion and to poor mechanical properties of the composites. (Martin et al., 2016; Gager et al., 2019b; Hadiji et al., 2020).

Fig. 2. SEM images of cross sections of biocomposites made with different thicknesses 3mm:(a) PLA/TC (b) PLA/linen, (c) PLA/hemp _ Thicknesses 2mm:(d) PLA/TC (e) PLA/linen, (f) PLA/hemp.

On the other hand, such high porosity is ideal for ensuring good acoustic properties due to a porosity network that tends to scatter and attenuate acoustic waves (Merotte et al., 2016; Renouard et al., 2017). Finally, due to the lack of flexibility of TC fibers, it appeared that PLA/TC/T3 biocomposites have more pores, with larger dimensions than PLA/linen and PLA/hemp (in Fig. 2).

Table 2

Characteristics of biocomposite plates made with measured thickness, porosity $(\%)$, fiber mass ratio (W_f) and fiber volume fraction (V_f) of composites.

3.2. Mechanical properties of biocomposites

3.2.1. Tensile behaviour

The results of these tensile tests showed that the average moduli varied from 1940.9 MPa to 3265.6 MPa in the CD direction and from 1442.2 MPa to 2959.5 MPa in the MD direction. As for the maximum stresses at break, values of 11.9 MPa to 27.1 MPa in the CD direction and 11 MPa to 26 MPa

in the MD direction were found. As for the maximum strains, they vary from 1.3% to 2% in the CD direction and from 1 to 1.7% in the MD direction. It can be seen that, depending on the direction of loading, the mechanical performances of all biocomposites (stiffness, strength) in the CD direction are higher than those in the MD direction (Table 3). This anisotropy is generated by the carding and topping steps during the nonwoven web manufacturing process (Miao and Shan, 2011; Martin et al., 2016). As a result, the CD direction is more resistant to tensile loading.

the results observed in Fig. 3 showed that the difference in thickness (T2 and T3) of the composites has a great influence on the mechanical properties of the biocomposites, regardless of the MD or CD orientation. It can be seen that biocomposites with low thicknesses of 2 mm (low porosity) have higher mechanical properties compared to those with thicknesses of T3. Indeed, in agreement with literature data, when the thickness of the composite is lower, the volume fraction of the fibers increases, which has the effect of decreasing the voids in the material and increasing the stiffness and the maximum stress. (Chocinski-Arnault et al., 2013). Regarding the influence of the fiber, Table 1 indicated that nonwoven felts based on TC fibers showed a lower tensile behavior compared to the reference fibers.

Fig. 3. Evolution of the tensile mechanical properties of PLA composites along the MD and CD directions.

it appeared from this study that the incorporation of TC fibers significantly strengthens PLA matrix but did not achieve the performance of flax and hemp. This could be due to the short elementary fiber length (average length of 0.7 cm) and to the brittle character (high lignin content 18.9 %) compared to flax and hemp fiber.

Table 3

Tensile mechanical characteristics and properties of biocomposites for the MD and CD directions.

3.2.2. Tensile failure mechanism

The Fig. 4 shows the SEM image of the composite specimen fracture. It is also possible to see that the composites of small thickness T2, (Fig. 4 a, b and c), have short fiber extraction lengths after composite fracture. This can be justified by a good adhesion to the interface. In Fig. 4 (a and d) we can also observe the brittle character of the TC fiber compared to composites of the same thickness (b and c). This translates into a brittle failure of the PLA/TC composite.

Fig. 4. SEM observation of tensile fracture surfaces in the CD direction of PLA matrix biocomposites. Biocomposites T2: (a) PLA/TC, (b) PLA/linen, PLA/hemp; Biocomposites T3: (d) PLA/TC, (e) PLA/linen and (f) PLA/hemp.

3.2.3. Flexural behaviour of biocomposites

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The mechanical flexural property results showed an improvement in the flexural performance of the matrix with the addition of TC, flax and hemp fibers (Fig. 5). It was observed that the mechanical properties in bending in the MD direction are weaker than in the CD direction due to the strong anisotropy generated by the coating mentioned previously in the tensile test. Indeed, the obtained moduli varied from 1940.9 MPa to 4397.4 MPa in the CD direction and from 1539.8 MPa to 4257.5 MPa in the MD direction. Compared to the tensile test results, the flexural moduli are slightly higher in both loading directions. The stresses are also higher compared to the tensile stresses at break ranging from 20.2 MPa to 61.2 MPa in the CD direction and from 17.8 MPa to 55.4 MPa in the MD direction. The flexural strains varied from 1.4 % to 2.2 % and are higher than the tensile strains. Thus the materials showed better performance in bending in the CD direction.

Fig. 5. Evolution of flexural mechanical properties of PLA matrix materials in the CD and MD directions.

Table 4

Characteristics and bending mechanical properties of biocomposites for the MD and CD directions.

From Fig. 5 it can be observed that the thickness has a great influence on the mechanical properties in flexion. The values of modulus and maximum bending stress of T2 thickness composites are about twice those of T3 thickness specimens. In the light of these results, it appears that the mechanical performance in flexion of biocomposites reinforced with TC fiber nonwovens is close to those reinforced with flax or hemp. Again, the low porosity provides better mechanical performances. It should also be noted that these biocomposites have a high flexural deformation. Such a capacity of the materials to deform is an important criterion for the targeted automotive applications.

3.3. Thermogravimetric analysis (TGA)

In all TGA curves (Fig. 6a), two main regions of degradation can be observed. One is due to the thermal degradation of cellulose, hemicelluloses, and lignin of the fibers (Lee et al., 2009; Ornaghi et al., 2014; Teixeira et al., 2019; Legrand et al., 2020) and the other, at higher temperature, is attributed to the depolymerization of PLA (Lee et al., 2009; Ketata et al., 2021). Good thermal stability of biocomposites is observed for $T < 239^{\circ}$ C corresponding to the stability limit for TC plant fibers (Mewoli et al., 2020).

Fig. 6. (a) TG curves and (b) DTG curves of pure PLA and biocomposites.

The onset of thermal decomposition of biocomposites is slightly higher than that of pure PLA, indicating that the addition of the fibers increases its thermal stability. The DTG curves (Fig. 6b) gave degradation temperatures of the samples from 239 to 419 °C. In view of these results observed on the curves (TGA and DTG), it appeared that, the manufacturing temperature of the biocomposites must remain well below 239 °C, in order to avoid the thermal degradation of the fibers, which could strongly affect the mechanical properties of the biocomposite. Finally, Figure 6 showed that the incorporation of fibers in the PLA matrix improved the residual ash content to more than 8%.

Conclusion

At the end of this study on the development of new nonwoven composites manufactured by cardingnapping-needling based on TC fibers, the results obtained during the experimental campaign have

It has been shown that the addition of TC fibers in the PLA matrix significantly improved its mechanical strength. The mechanical properties in traction and flexion (stiffness and resistance) in the CD direction were high compared to the MD direction. This was due to the strong anisotropy linked to the preferential orientation of fibers. The tensile mechanical performance of TC fiber reinforced composites is slightly lower than that of non-woven linen and hemp composites due to the morphology of the TC fiber which is of short length and brittle character. On the other hand, the bending stiffness is close to that of the reference composites. The stiffness of nonwoven composites was strongly influenced by the thicknesses. Thus, an increase in the thickness of the composite degraded its mechanical properties. The addition of TC, flax, and hemp plant fibers to the PLA matrix improved its thermal stability and also increased the residual ash content of the composites. The composites of TC fiber nonwoven reinforcements developed during this study have shown properties that make them serious candidates in a perspective of diversification of composites commonly used in the automotive field.

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