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***Triumfetta cordifolia*: A Valuable (African) Source for Biocomposites**

Peter Grosser,^a Carolin Siegel,^b Christoph Neinhuis,^a and Thea Lautenschläger^a

The tradition of using naturally occurring plant fibers is still alive in Africa. In the Uíge province of northern Angola, bast fibers from *Triumfetta cordifolia* serve as the basis for everyday objects, such as baskets, mats, fishing nets, and traditional clothing. The fibers exhibit a Young's modulus of 53.4 GPa and average tensile strength of 916.3 MPa, which are comparable to those of commercial kenaf fibers. These values indicate a high potential for use as a reinforcement in biocomposites. Based on this promising mechanical and physical profile of individual fibers, different biocomposites were produced with polylactide (PLA) as a matrix. The obtained composites were analyzed mechanically, physically, and visually. Unidirectionally arranged PLA/33% *T. cordifolia* composites with continuous fibers showed the highest Young's modulus (10.79 GPa \pm 1.52 GPa) and tensile strength (79.37 MPa \pm 14.01 MPa). These composites were comparable to those of PLA/30% hemp composites (10.9 GPa and 82.9 MPa, respectively) and therefore have economic potential.

Keywords: Bast fibers; Biocomposites; Lightweight; Tensile tests; Young's modulus

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INTRODUCTION

In recent years, problems caused by the excessive consumption of fossil fuels have become increasingly visible. This development has triggered the formation of numerous new markets based on renewable resources. Established industries have also seen the potential of these new markets and have started to use bio-based materials. For example, in the automotive sector, natural fibers are now used as reinforcement material in various interior components, such as parcel shelves and interior door panels (Carus and Partanen 2017). Additionally, an entire new sector for natural fiber-reinforced plastics has been established and is steadily growing. These developments have led to an increasing demand for commercial fiber plants and renewed the interest in fiber plants that were once used commercially, but have fallen into disuse. The tropical plant *Triumfetta cordifolia* A. Rich can be regarded as an example of such a plant.

Triumfetta cordifolia is a fast-growing shrub and is widespread in moist areas of tropical Africa. Most local communities in these regions know the plant for its versatile medicinal properties or use it as food (Brink and Achigan-Dako 2012). Only in a few areas is *T. cordifolia* known to produce strong bast fibers, which also have an industrial history. In the 1950s, Belgium imported large amounts of these fibers from the DR Congo and used them to produce coffee bags (Brink and Achigan-Dako 2012). However, the fibers are rarely used today. In the Uíge province in northern Angola, people still use *Triumfetta*

traditionally to produce ropes, textiles, mats, baskets, and other products (Senwitz *et al.* 2016).

In tensile tests, *T. cordifolia* fibers show an average Young's modulus of 53.4 GPa, which is approximately twice that of flax (27.6 GPa) and jute fibers (26.5 GPa) and comparable to that of kenaf fiber (53 GPa). The tensile strength of *T. cordifolia* fibers (916.3 MPa) is similar to that of kenaf fibers (930 MPa) (Faruk *et al.* 2012; Senwitz *et al.* 2016). Moreover, *T. cordifolia* fibers show a density of 1.26 g/cm³, comparable to those of flax (1.5 g/cm³), hemp (1.48 g/cm³), kenaf (1.45 g/cm³), and jute fibers (1.3 g/cm³), which indicates the high potential of these bast fibers for use as reinforcement in biocomposites (Faruk *et al.* 2012; Mansor *et al.* 2013; Senwitz *et al.* 2016).

The first attempts to compound entire fiber bundles in a thermoplastic polylactide (PLA) matrix (NatureWorks 2004) led to poor results and had a Young's modulus below 3 GPa (Petit 2016). However, because the mechanical and physical properties of the fiber are promising, a new approach was chosen, which produced long fiber-reinforced thermoplastics (LFRTs), as well as continuous fiber-reinforced thermoplastics (CFRTs). The results were compared to further evaluate the potential of *T. cordifolia* bast fiber as a reinforcing component.

EXPERIMENTAL

Materials

Fibers

The fibers originated from *T. cordifolia* plants growing in the province of Uíge (municipalities Bembe and Uíge) in northern Angola. The bast fibers were isolated from harvested *T. cordifolia* stems by peeling the whole bark, including the fibers, off of the wood. The fibers were subsequently separated from the bark using a knife (Senwitz *et al.* 2016). To ease the extraction of the fibers from adjacent cells, the samples were immersed for three weeks in plastic buckets containing tap water, which is a process called retting. This process microbially degrades non-cellulosic material (*e.g.*, pectins) and helps to separate single bast fibers from adjacent cell material (Tahir *et al.* 2011). It has been previously reported that retting has positive effects on the mechanical characteristics of *T. cordifolia* fibers (Senwitz 2015).

Fibers from two different harvesting dates were used in this study. The first harvest took place in November 2016, at the beginning of the rainy season in this region. The second harvest took place in February 2017, during the middle of the rainy season. The fibers harvested in November (Figs. 1A and 1I) are referred to as 'N-fibers' and those in February (Figs. 1E and 1L) as 'F-fibers'.

Matrix

The biodegradable polymer PLA (Indigeo Biopolymer 2003D, NatureWorks, Minnetonka, MN, USA) was used as the matrix material to produce the biodegradable composites.

Methods

Preprocessing

Because the use of whole fiber bundles as reinforcements in thermoplastics have been found to have negative side effects (pull-outs, inhomogeneous distribution, fractures,

etc.), the bundles were separated (Petit 2016). Therefore, a close-meshed comb was used to isolate the individual fibers.

To compare several types of composites, two different fiber lengths were used. One half of the material was cut to a length of 0.5 cm to 1 cm (long fibers, Figs. 1B and 1F), the second half was left uncut after the combing process and included fibers of 10 cm to 15 cm length (continuous fibers, Figs. 1J and 1M).

The long fibers had to be brought into an appropriate form to be used in the film-stacking process with the PLA foil. Therefore, the material was transformed into nonwoven fabrics *via* the wet-laid method (DIN EN ISO 5269/2 2005), using a sheet former (System Rapid-Köthen, Frank-PTI, Birkenau, Germany). This process includes the swirling up of the fiber material together with water, before the whole mixture gets withdrawn through a screen. The resulting nonwoven fabrics, approximately 20 cm in diameter and 1 mm in thickness (Figs. 1C and 1G), were then vacuum dried.

The continuous fibers were parallelized and straightened using a flat iron. The unidirectionally (UD)-arranged continuous fibers (Figs. 1J and 1M) were fixed on the PLA foil for easier handling in the following processes.

All of the fiber materials were dried overnight in a drying oven at 105 °C. This step evaporated residual moisture and thus avoided the formation of evaporation airlocks during the following plasticizing process in the heating press.

Film-stacking

To embed the fiber material, the film-stacking method was used. This method can be used for short and long fibers, as well as continuous fibers, unlike injection molding and the extrusion method. The fiber material and PLA film were alternately layered.

Each LFRT consisted of three nonwoven fabrics (approximately 1-mm thickness) embedded into ten PLA foils (100- μ m thickness). The fiber content in the LFRTs was 40% (dry matter content). For the CFRTs, two layers of UD-arranged continuous fibers (approximately 1-mm thickness) were embedded into eight PLA foil layers. The fiber content in the CFRTs was 33%. The compounds were then plasticized in a hot press (WPM Werkstoffprüfmaschine Leipzig DP 1500, WPM Leipzig GmbH, Markkleeberg, Germany) at a temperature of 190 °C and pressures of 5 kN and 15 kN for the LFRTs and CFRTs, respectively. Subsequently the composites were cooled down between iron plates. The results were circular nonwoven LFRTs (diameter = 20 cm) and square UD-CFRTs (10 cm \times 10 cm), as shown in Fig. 1 (LFRTs: Figs. 1D and 1H; CFRTs: Figs. 1K and 1N).

From the available materials, five LFRTs were manufactured, three of which contained N-fibers and two contained F-fibers. Eight out of the 14 CFRTs contained N-fibers and six contained F-fibers.

Tensile tests

All of the composites were mechanically characterized and evaluated by determining the Young's modulus, tensile strength, and breaking strain *via* static tension tests in accordance with DIN EN ISO 527-1 (2012). Complying with this standard, specimens were cut from the composites using a laser cutter (Legend EXT, EPILOG, Houten, The Netherlands).

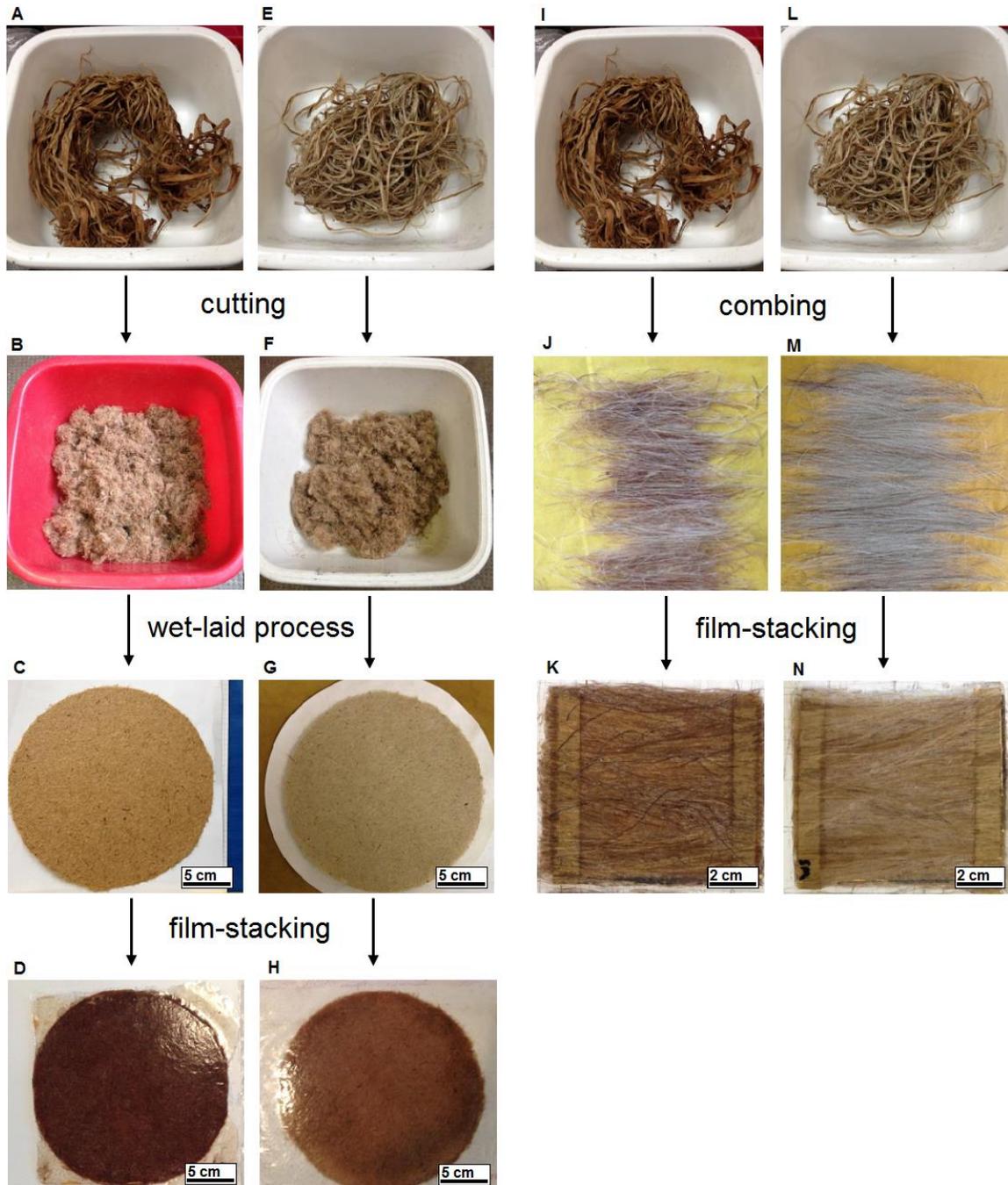


Fig. 1. Pictures of the compounding processes with *T. cordifolia* fibers in the PLA: LFRTs with N-fibers (A - D) and F-fibers (E - H); CFRTs with N-fibers (I - K); and F-fibers (L - N)

The LFRTs were cut into 15 specimens each, resulting in a total of 75 LFRT specimens (45 N-fibers and 30 F-fibers). Nine specimens were cut out of each CFRT, resulting in 126 CFRT specimens (72 N-fibers and 54 F-fibers). Because the CFRTs are anisotropic, one plate from each harvest was cut perpendicular to the fiber orientation (specimens: nine N-fibers and nine F-fibers) and acted as controls for the remaining specimens, which were cut parallel to the fiber orientation (63 N-fibers and 45 F-fibers).

The tensile tests were conducted on a universal testing machine (Inspekt 10 Desk, Hegewald & Peschke, Nossen, Germany) in compliance with DIN EN ISO 527-1 (2012).

The pre-load amounted to 5 N, and the distance between the clamps was 55 mm. The strain was measured *via* a video extensometer (Videoextensometer ME46 NG, Messphysik Materials Testing GmbH, Fürstenfeld, Österreich) using the program WinextNG (Messphysik Materials Testing GmbH, Fürstenfeld, Österreich).

Density measurements

The composite density was determined using a digital caliper (Absolute Digimatic, Mitutoyo, Kawasaki, Japan) to calculate the specimen volume. The weight was measured with a precision scale, with an accuracy of 0.01 g. The density was calculated by dividing the weight by the volume.

The density was also calculated by using the fiber content and densities of the PLA and fibers. The latter was determined using a glass pycnometer (50 cm³, Karl Hecht GmbH & Co KG, Sondheim v. d. Rhön, Germany) and precision scale with accuracies of 0.05 mL and 0.01 g, respectively. The calculated densities served as comparative values to identify failures or unwanted inclusions (*e.g.*, trapped air) in the composites.

Microscopic analysis

To evaluate the composite structure and quality, grinding probes were fabricated by embedding pieces of the composites in epoxy resin (EPOThin, Buehler AG, Uzwil, Schweiz). Air bubbles within the resin were removed in a vacuum chamber (Cast 'n Vac, Buehler AG, Uzwil, Schweiz) prior to hardening. Thereafter, the specimens were ground and polished (EcoMet/AutoMet 250, Buehler AG, Uzwil, Schweiz) and then investigated with a microscope (magnification: 40x, Olympus BX51, Olympus, Tokyo, Japan). Pictures of the microsections were taken with the software VidMess (TSO DATA GmbH, Osnabrück, Germany).

For the anisotropic UD-CFRTs, microsections were made both parallel and perpendicular to the fiber orientation.

RESULTS AND DISCUSSION

Tensile Tests

Both types of composites made using *T. cordifolia* F-fibers showed better mechanical properties compared with those made using N-fibers (Tables 1 and 2).

Long fiber-reinforced thermoplastics

The PLA/40% LFRTs made with N-fibers (Fig. 1D) showed an average Young's modulus of 3.77 GPa ± 0.52 GPa, which was slightly above that of the PLA matrix (3.5 GPa). The F-fiber LFRTs (Fig. 1H) had a Young's modulus of 4.02 GPa ± 0.54 GPa. Other PLA/natural long fiber composites, like PLA/30% nonwoven flax LFRTs, showed a remarkably higher stiffness (8 GPa) than the *T. cordifolia* nonwoven LFRTs, as can be seen in Table 1 (Bodros *et al.* 2007). The same result applies to PLA/40% hemp long fiber (alkali-treated) composites that had a Young's modulus of 8.5 GPa (Hu and Lim 2007). However, the values of the *T. cordifolia* LFRTs were still up to 25% higher than those measured for the PLA/35% *T. cordifolia* fiber bundle UD-composites (2.95 GPa ± 0.66 GPa) reported in an earlier study (Petit 2016).

The tensile strengths of both the N-fiber (36.5 MPa ± 2.94 MPa) and F-fiber LFRTs (40.91 MPa ± 4.23 MPa) were lower than that of the PLA matrix (53 MPa). Composites

with PLA/30% nonwoven flax LFRTs had a strength of 100 MPa, which was twice that of the *T. cordifolia* LFRTs (Bodros *et al.* 2007). The LFRTs breaking strain was $1.18\% \pm 0.17\%$ (N-fibers) and $1.3\% \pm 0.23\%$ (F-fibers), which was well below that of the PLA (6%). The PLA was naturally more flexible, which was because of its polymeric nature.

Table 1. Tensile Properties of the Different Natural LFRTs

Material (LFRT)	Treatment	Pre-form	Plasticizing Method	Young's Modulus (GPa)	Tensile Strength (MPa)
PLA ¹	-	-	-	3.5	53
PLA/40% <i>T. cordifolia</i> (N-fibers)	Retting	Nonwoven	Film-stacking	3.77 (0.52)	36.5 (2.94)
PLA/40% <i>T. cordifolia</i> (F-fibers)	Retting	Nonwoven	Film-stacking	4.02 (0.54)	40.91 (4.23)
PLA/30% flax ²	Retting	Nonwoven	Film-stacking	8	100
PLA/40% hemp ³	Alkali	-	Hot press	8.5	54.6

References: ¹NatureWorks (2004), ²Bodros *et al.* (2007), ³Hu and Lim (2007); values in parentheses indicate standard deviations

Using *T. cordifolia* long fibers as a reinforcement material did not obtain the desired results regarding the stiffness and strength in comparison with other PLA/natural fiber composites. Composites with similar or better mechanical properties can be made using residual biomass (*e.g.*, papaya bast fibers) as a reinforcement material, which is more ecologically friendly (Lautenschläger *et al.* 2016).

Continuous fiber-reinforced thermoplastics

Because UD-composites with continuous fibers generally have a higher stiffness and strength (parallel to the fiber orientation) than long fiber composites, it was not surprising that the *T. cordifolia* UD-CFRTs had better properties than the LFRTs from the same plant (Lengsfeld *et al.* 2015). They showed an average Young's modulus of $7.26 \text{ GPa} \pm 0.74 \text{ GPa}$ when manufactured with N-fibers (Fig. 1K) and $10.79 \text{ GPa} \pm 1.52 \text{ GPa}$ with F-fibers (Fig. 1N). This equated to an increase of 192% (N-fibers) and 268% (F-fibers) compared with the LFRTs.

Comparing the Young's modulus of the CFRTs with that of PLA/35% *T. cordifolia* fiber bundle UD-composites ($2.95 \text{ GPa} \pm 0.66 \text{ GPa}$) from an earlier study, the direct influence of fiber isolation on the composite stiffness, which increased by at least 246%, was observed (Petit 2016).

The Young's modulus of the F-fiber CFRTs (10.79 GPa) were comparable to those of other natural fiber UD-composites with 30% hemp and PLA, which had a Young's modulus of 10.9 GPa (Islam *et al.* 2010). It should be noted that an additional alkali pretreatment, as well as textile semi-finished products in the form of unidirectional warps, were used as the reference hemp composite, which implicated additional positive impacts on the composite properties (Islam *et al.* 2010). The tensile strength of the N-fiber ($60.41 \text{ MPa} \pm 9.36 \text{ MPa}$) and F-fiber CFRTs ($79.37 \text{ MPa} \pm 14.01 \text{ MPa}$) were 65% and 94% higher than those of the *T. cordifolia* LFRTs, respectively. The tensile strength of the N-fiber CFRTs was comparable to that of the PLA/30% hemp CFRT mentioned above (82.9 MPa) (Islam *et al.* 2010).

Overall, the mechanical properties of the *T. cordifolia* UD-composites were comparable to similar composites with commercial bast fibers. Still, there is considerable potential for better CFRTs because other studies have shown better results when using different methods. One example is PLA/50% flax composites produced by vacuum molding that resulted in a Young's modulus of 19 GPa (Madsen *et al.* 2008).

Table 2. Tensile Properties of the Different Natural Fiber CFRTs

Material (CFRT)	Treatment	Pre-form	Plasticizing Method	Young's Modulus (GPa)	Tensile Strength (MPa)
PLA/33% <i>T. cordifolia</i> (N-fibers)	Retting	UD fibers	Film-stacking	7.26 (0.74)	60.41 (9.36)
PLA/33% <i>T. cordifolia</i> (F-fibers)	Retting	UD fibers	Film-stacking	10.79 (1.52)	79.37 (14.01)
PLA/35% <i>T. cordifolia</i> ¹	Retting	UD bundle	Film-stacking	2.95 (0.66)	-
PLA/30% hemp ²	Alkali	UD warp	Film-stacking	10.9	82.9
PLA/50% flax ³	-	UD fibers	Vacuum molding	19	-

References: ¹Petit (2016), ²Islam *et al.* (2010), ³Madsen *et al.* (2008); values in parentheses indicate standard deviations

The CFRT specimen cut in the cross direction to the fibers showed the lowest stiffness (N-fiber: 1.3 GPa \pm 0.07 GPa; F-fiber: 2.13 GPa \pm 0.34 GPa) of all of the composites and a low tensile strength (N-fiber: 6.08 MPa \pm 0.43 MPa; F-fiber: 9.89 MPa \pm 1.15 MPa).

Density Measurements

The fiber densities of the *T. cordifolia*, determined with the pycnometer, were 1.264 g/cm³ \pm 0.07 g/cm³ (N-fiber) and 1.282 g/cm³ \pm 0.08 g/cm³ (F-fiber), which were similar to that of jute fibers (1.3 g/cm³) and slightly lower than that of flax (1.5 g/cm³), hemp (1.48 g/cm³), and kenaf fibers (1.45 g/cm³) (Faruk *et al.* 2012; Mansor *et al.* 2013). Consequently, the *T. cordifolia* fiber composites were among the lightest natural bast fiber composites, provided the same polymer is used. The composite density was determined by measuring and weighing individual specimens, which revealed low values (0.92 g/cm³ for the LFRTs and 1.05 g/cm³ for the CFRTs). Because these values were lower than those of the individual components, the composite densities were calculated for comparison based on the individual densities of the fibers and matrix, as well as their mass percentage in the composite. The PLA matrix had a density of 1.25 g/cm³. The N-fibers (1.26 g/cm³) were slightly less dense than the F-fibers (1.28 g/cm³). Therefore, the N-fiber composites had a calculated density of 1.256 g/cm³ for the LFRTs and 1.255 g/cm³ for the CFRTs, which were slightly lower than those of the F-fiber LFRTs (1.263 g/cm³) and CFRTs (1.261 g/cm³).

The large differences between the experimentally determined and theoretically calculated densities implied that air was trapped in the composites.

Composite Morphology

The microsection images confirmed the assumption that both the LFRTs and CFRTs contained a considerable amount of air trapped between the fibers (Fig. 2). Also, other failures were visible in the microsections. The LFRTs showed fiber displacements, which resulted in an inhomogeneous composite structure and lower quality (Fig. 2). Additionally, the probability of structural failure was increased because parts of the composite had a lower fiber content. In comparison, the UD-CFRTs (Fig. 3) showed less fiber displacement, but had similar amounts of air bubbles between the fibers and matrix. The transverse microsection (Fig. 3A) clearly showed where and how the trapped air bubbles cause ruptures in the composites. Ruptures occurred along the interface between the fiber and matrix, which indicated a lack of proper fiber-matrix bonding and is a key factor in the composite quality (Drzal and Madhukar 1993). At the edge of the specimen, defective areas were seen, some of which were likely caused by the laser cutter.

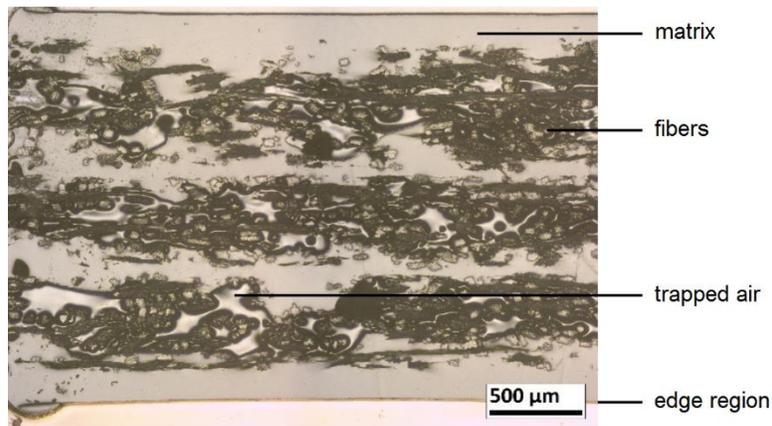


Fig. 2. LFRT microsection

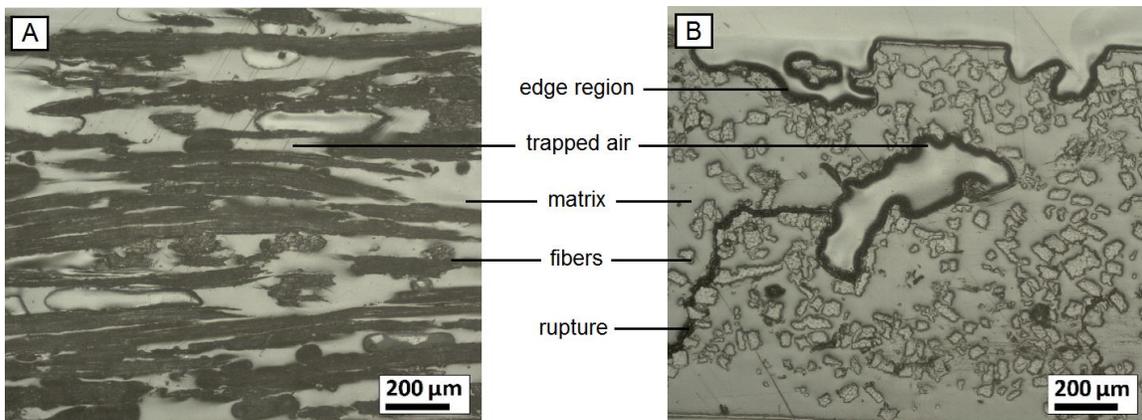


Fig. 3. CFRT microsections along the fiber direction (A) and transverse to the fiber direction (B)

Discussion

Tensile tests and a visual examination of the *T. cordifolia* composites revealed new insights into the suitability of different fiber compounding processes. Various defects, such as trapped air bubbles that were not expelled from the composites during the plasticizing process, showed that there is still room for improvement. The air bubbles, fiber displacements, and damaged edges reduced the performance of the composites. The

CFRTs, especially those containing F-fibers, showed mechanical properties comparable to similar PLA/30% hemp composites, without applying the chemical treatments or semi-finished textile products used by Islam *et al.* (2010).

The better performance of the composites containing F-fibers was explained by the general growth phases of *Triumfetta*. New shoots emerge at the beginning of the rainy season, around October through November, in this region. Because young shoots are not yet fully developed during this time, *i.e.*, the fibers are not fully lignified, the November harvest included both weak young fibers and fibers from shoots of the previous year. In contrast, the February harvest included new stem material with fully developed fibers, which could have been the reason for the higher average stiffness and tensile strength of the composites with F-fibers.

The experimentally determined densities of the composites were comparatively low, which was most likely caused by the air inclusions. Because the density directly influences the specific stiffness (Φ), and therefore the potential for light-weight applications of the material, even the higher calculated densities of the PLA/*T. cordifolia* composites showed considerable potential. As for the CFRTs, the specific stiffness calculated from the measured densities reached up to 7.97 GPa/g/cm³ \pm 0.96 GPa/g/cm³ (N-fiber CFRT) and 10.35 GPa/g/cm³ \pm 1.53 GPa/g/cm³ (F-fiber CFRT). Because enclosed air reduces the density, resulting in a higher specific Young's modulus, and because of reproducibility, the theoretically calculated density values were applied as well (ignoring trapped air), which resulted in lower specific stiffnesses (5.78 GPa/g/cm³ \pm 0.71 GPa/g/cm³ for the N-fiber CFRT and 8.56 GPa/g/cm³ \pm 1.42 GPa/g/cm³ for the F-fiber CFRT). However, in this case, the measured Young's moduli were lower than in composites without defects, which should have resulted in a higher specific stiffness for the N-fiber CFRTs if there were no defects. Nevertheless, the obtained values were comparable to those of similar PLA/30% hemp composites (8.26 GPa/g/cm³) (Islam *et al.* 2010; Faruk *et al.* 2012). Because *T. cordifolia* bast fibers have one of the lowest densities compared with other plants (Senwitz *et al.* 2016), it is a promising source for biocomposites.

The UD-CFRTs performed better than the LFRTs in terms of the stiffness and strength. Also, the effect of using individual fibers isolated from fiber bundles became obvious. The CFRTs containing separated fibers showed an increased stiffness that ranged from 246% to 366% compared with the CFRTs utilizing entire fiber bundles (Petit 2016).

There was still room for improvement because the manufactured composites suffered from insufficient fiber-matrix bonding. Ruptures that were observed in the CFRT microsections transverse to the fiber direction clearly indicated this problem (Fig. 3B). To improve the fiber-matrix contact, and thus the composite quality, different approaches should be applied to prevent the formation of air pockets. This could include vacuum treatment or additional drying steps prior to the plasticizing process. When the fiber material is drier, the probability is lower for water evaporation and air bubble formation during the compounding step at high temperatures. Different chemical treatments of natural fibers have shown positive effects on the fiber-matrix adhesion in composites. For example, alkali-treatment with sodium hydroxide or benzylation with benzoyl chloride improved the adhesion (Li *et al.* 2007; Khan *et al.* 2016). Another possibility to increase the contact between the fiber and matrix is the usage of coupling agents. Zein (a corn protein) and silane have shown good effects as coupling agents in natural fiber composites (John and Anandjiwala 2009; Wang *et al.* 2010). Other plasticizing methods (like vacuum molding) and higher pressures during compounding steps could additionally prevent composites from having weak fiber matrix adhesion and increase the mechanical properties.

Composites with PLA/50% flax manufactured *via* vacuum molding have resulted in a high stiffness of 19 GPa, which indicated the suitability of this method for PLA/natural fiber composites (Madsen *et al.* 2008). For an additional increase in the composite quality, textile semi-finished products (prepregs) could be manufactured from the fibers before plasticization (Lengsfeld *et al.* 2015).

CONCLUSIONS

1. Composites with isolated *T. cordifolia* fibers showed considerably increased mechanical properties when compared with composites containing whole fiber bundles from the same plant.
2. The *T. cordifolia* F-fibers were better suited as a reinforcement material in biocomposites than the N-fibers.
3. Composites with 33% UD-CFRTs showed higher stiffness and strength values than the composites with 40% LFRTs.
4. The mechanical characteristics of the CFRTs were comparable to similar PLA/hemp biocomposites.
5. Both the LFRTs and CFRTs contained air inclusions.
6. Further improvement is possible by using suitable coupling agents, textile semi-finished products, chemical treatment, or adapted plasticizing methods.
7. Further studies with *T. cordifolia* fibers are recommended because the fibers showed a high mechanical potential.

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