

EXPERIMENTAL INVESTIGATION OF TENSILE PROPERTIES OF FLAX FIBRE-REINFORCED COMPOSITES

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Renewable materials for aeronautical structures have the potential to contribute to an eco-efficient sustainable aviation. High-performance flax fibres, a promising substitution for glass fibres, are available on the market and currently comprehensively studied. With bio-resins, it is possible to increase the bio-content of composite structures over 50%. However, the mechanical properties of flax fibres are highly influenced by the chosen matrix and process parameters. This paper investigates the tensile properties of composites that are produced from two epoxy, seven bio-epoxy resins and one polyfurfuryl alcohol (PFA) resin in combination with five different flax fibres. It was found that the resin type strongly influences the flax fibre, with PFA inducing a brittle fracture behaviour and lowering the tensile strength for 40.5%, however increasing the stiffness for 17% compared to epoxy matrix. The bundle type of UD sheets and BD weaves are a key factor affecting strength and stiffness with hackled flax, and rovings having the best properties for UD and BD, respectively. Furthermore, the fibre volume content is a crucial parameter influencing mechanical properties, which strongly depends on resin type and production process. Good mechanical properties can be achieved, however with the finding that there is still a great potential for further improvements on the quality and hence the mechanical properties of flax fibre-reinforced composites.

Nomenclature

BD	bidirectional
FFRC	flax fibre-reinforced composite
FVC	fibre volume content
SD	standard deviation
UD	unidirectional
(.) ₀	parallel to fibre direction
(.) ₉₀	perpendicular to fibre direction
(.) ₄₅	45° to fibre direction
E	young's modulus
E^I, E^{II}	primary, secondary young's modulus
G	shear modulus
ρ_A	grammage
R_m	tensile strength
σ	tensile stress
τ	shear stress
τ_{max}	ultimate shear stress
ϵ	strain
ϵ_f	fracture strain

1. INTRODUCTION

Renewable materials for eco-efficient transportation structures are increasingly gaining more importance. However, bio-composites were already used in the 1940s for automotive applications [1] motivated by the high mechanical performance regarding its weight. In recent years, the research on bio-composites is driven by their low environmental footprint due to the reduced energy input during production [2–6]. Research developments have shown that flax fibre reinforcements offer mechanical and lightweight potential for modern composite structures [7, 8], which has already been demonstrated in the automotive industry for door panels and boot liners, valued for their low density, good mechanical and acoustic properties, tool-friendly processability, and splinter- and fracture-free damage characteristics [9, 11]. However, aeronautical structures must typically comply with stricter standards. Therefore, flax fibre composites for aviation are still in the research stage due to the high requirements in terms of strength, stiffness and operational longevity. Nevertheless, the low density, hence comparable specific mechanical properties to glass fibres and the high inherent damping properties, offer a great potential in helicopter structures [10].

Within the scope of the federal research project *InteReSt II*, a representative helicopter cabin door of the lightweight helicopter EDM Aerotec CoAX 2D/2R is used to demonstrate and evaluate the technological concept of

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hybridising carbon fibre composites and natural fibre composites [13]. The goals are to significantly increase the fraction of renewable materials up to more than 50%, to improve the life cycle assessment for semi-structural aerospace components and to investigate the impact of an aviation environment on FFRC. The preceding research project *InteReSt* has already shown that an application of suitable hybrid carbon/flax composites for the aerospace sector is possible and that ecological and economic advantages are emerging [12, 14]. However, there is still a potential to increase the fraction of renewable materials. Additionally, the difficulty of a continuous investigation of specific flax fibre/resin material combinations is their rapidly changing market. Availability drives the material selection and sets limitations to targeted properties like bio-content. The FVC of FFRCs is equal to the composite's bio-content when epoxy resin is used as the matrix. This means that for the targeted bio-content available, flax fibre prepregs with epoxy resin are sufficient for a FVC higher than 50%, but that the hybridisation with CFC is then limited. Either using flax prepregs with bio-resins or infusing flax fibres with bio-resins are possible solutions. A market study has been conducted to provide FFRCs that are currently available on the European market and that are relevant for aeronautical applications. Due to the goal of developing a lightweight structure, it is necessary to find the strongest and stiffest flax fibre-reinforced composite. Hence, the flax fibres as well as the influence of the resin on the flax fibre must be investigated regarding their mechanical properties. This paper provides a broad overview on currently available high-performance flax fibre-reinforced composites suitable for semi-structural aeronautical components and studies their experimentally derived tensile properties.

2. MATERIALS AND METHODS

The market research of prepregs, bio-resins and flax fibres was driven by the factors of availability, mechanical properties and bio-content. The selected materials in this study are experimentally evaluated on their tensile and shear strength, young's and shear modulus, bilinear stiffness and fracture behaviour in 0° and 90° for UD fibres as well as $0^\circ/90^\circ$ and $\pm 45^\circ$ for BD twill weave fibres.

The investigated FFRCs use flax fibres of the two producers *Bcomp Ltd.* and *Ecotechnilin*. For this study, the lightest UD of $110 - 150\text{g}/\text{m}^2$ and BD of $150 - 200\text{g}/\text{m}^2$ materials are selected, which is due to the requirement for the design of the lightweight helicopter cabin door. The compared fibres are listed in Table 1 and shown in Fig. 1 and 2. The image of B1 is made from the prepreg while all the others are from dry materials. The UD fibres of Bcomp, B1 and B2, are flax rovings connected with threads perpendicular to fibre direction. The advantage of rovings is a homogenous fibre distribution across the material. The fibre bundle type of Ecotechnilin's UD material, L1, is hackled flax, which promises good tensile properties due to the fibre's low twist angle. Looking at the BD flax weaves, B3

of Bcomp is a 2/2 twill made from rovings compared to Ecotechnilin's L2, which is made from yarns. However, low twist rovings are expected to have a 20% higher bundle stiffness compared to yarns [29].

The investigated matrix materials are petrochemical-based and bio-based epoxy resins as well as one polyfurfuryl alcohol (PFA) resin, referred to as furan resin. All investigated resins are listed in Table 2 with their total bio-content including hardener. Bio-based epoxy resins make use of low-cost and environmentally-friendly renewable natural sources such as plant oils and starches as building blocks for polymers [15, 16]. The selected seven bio-based epoxy resins have bio-contents ranging from 20.8% to 39.2% including hardener, six of which are used for vacuum assisted resin infusion (VARI) and one comes with a prepreg. The two petrochemical-based epoxy resins are provided in prepregs from Fiberpreg and Ecotechnilin. The selected furan resin CE, also delivered as prepreg, is a thermosetting bio-based resin derived from crop waste like bagasse from sugar production or corn cob residues, which has great fire retardancy and lower toxicity and VOC (volatile organic compounds) emissions compared to phenolic resins [26, 27]. Furan resins promise a highly ecological alternative to conventional epoxy and phenol resins with similar mechanical properties [17–19]. According to the manufacturer, the bio-content including hardener is around 98%, which is the main reason for selection. However, it is known that furan resins can have high porosity depending on the curing cycle. Also, water is used as solvent that is released during processing, which can pose problems considering the hydrophilic behaviour of the flax fibre [17].

Three manufacturing techniques are applied in this study: 1) VARI is chosen due to the high variability of fibre and resin types, which allows to independently design the material properties such as the bio-content. 2) Prepreg is chosen due to the great applicability in complex three-dimensional moulds. 3) Wet lay-up is used for the Fire-Green resin in order to allow its fire-proof particles to distribute equally in the composite structure. All three procedures are followed by vacuum/autoclave curing under pressure in order to increase the FVC.

Table 1: Investigated Flax Fibres

	fibre	type	ρ_A [g/m^2]
B1	Bcomp ampliTex 5032 UD	UD	120
B2	Bcomp ampliTex 5057 UD	UD	150
B3	Bcomp ampliTex 5043 twill 2/2	BD	200
L1	Ecotechn. FLAXTAPE UD 110	UD	110
L2	Ecotechn. FLAXDRY BL 150	BD	150

The composites are produced on polished steel plates as panels of 300×300 mm, with the number of plies required to reach the thickness according to the norm. The panels are subsequently cut into test specimens. All materials under investigation are cut using a water-cooled buzz saw,

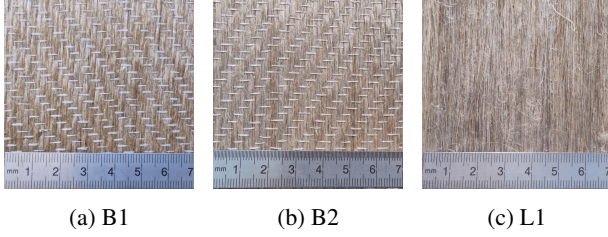


Fig. 1: UD flax fibre material

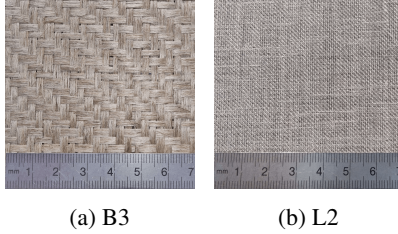


Fig. 2: BD flax fibre material

with the exception of B1-FPB, which is cut using guillotine shears due to a availability of cutting machinery. For that reason, the influence of the cutting method on tensile strength is also investigated due to the hydrophilic behaviour of the flax fibre. The findings are presented in the next chapter. The width and thickness of the test specimens are measured at three positions each with a micrometer gauge and a sliding caliper, respectively.

The tensile tests are carried out on an Instron 4505 in accordance with DIN EN 2561 (UD0) [20], DIN EN 2597 (UD90) [21], DIN EN 6031 (BD45) [22] and DIN EN ISO 527-4 (BD0) [23] with a sample size of 5-8 specimens for each test configuration. As seen in Fig. 3, the strain is optically measured with digital image correlation (DIC) utilising the system GOM Aramis, which receives the force input as analogue signal from the Instron. The test article's preparation is crucial for DIC measurements in order to capture the strain field. The test article must be coated non-glare white before a black stochastic pattern can be applied due to the requirement of a strong contrast of the applied speckle pattern on the test article for good measuring quality [28]. The advantage of using DIC to measure strain over traverse path data from the tensile testing machine is the increased accuracy. At the start of each tensile

Table 2: Investigated Resins

	resin (+ hardener)	bio-content [%]
GP33	GreenPoxy 33 + SD 4771	27.0
GP56	GreenPoxy 56 + SD 4771	39.2
IG	InfuGreen 810 + SD 8822	29.0
RT	Resoltech 1800 ECO + 1804 ECO	34.0
PS	Pro-SET M1049 + M2048	26.9
FG	FireGreen 37 + SD 8202	20.8
FP	Fiberpreg EP1.5	0.0
FPB	Fiberpreg EP1.5 Bio	22.0
ET	Ecotechnilin Epoxy	0.0
CE	Composites Evolution PFC502	98.0

Table 3: Investigated Prepregs

prepreg	fibre	resin
Fiberpreg-NAT-120-UD-EP1.5Bio-50	B1	FPB
Fiberpreg-NAT-150-UD-EP1.5-44	B2	FP
Fiberpreg-NAT-200-T-EP1.5-61	B3	FP
Ecotechnilin Flaxpreg-T-UD-110	L1	ET
Composites Evo. PFC502-F150U-5057-50	B2	CE
Composites Evo. PFC502-F200T-5043-50	B3	CE

test, a reference image of the unloaded specimen is taken, also storing force data of the unloaded load cell of the Instron. This serves as reference for the strain calculation. The number of images taken during a tensile test is the resolution of the data, which was between 70 to 140 images per test, depending on the material.

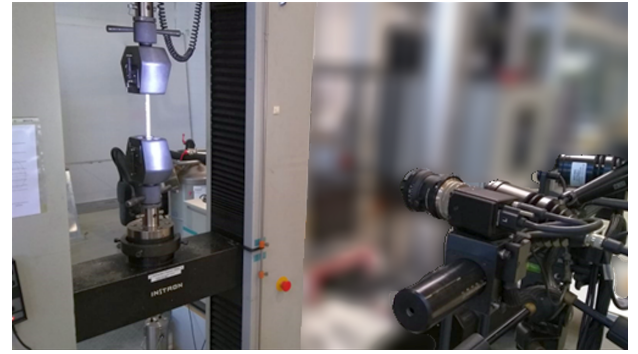


Fig. 3: Tensile test set-up with digital image correlation

Generally, a FVC of 60% is considered as optimum for achieving high mechanical strength with fibres bearing the loads. A higher FVC than 60% causes a drop in strength again due to the fibres no longer being completely wetted by the matrix [24] [25]. The FVC of the produced composite panels are calculated using the density of the flax fibre $\rho_f = 1.47 [kg/m^3]$, the number of plies n_f , the grammage $\rho_A [g/m^2]$, the area $A_f [m^2]$ of the plies and the volume of the composite panel $V_c [m^3]$.

$$FVC = \frac{\rho_A \cdot A_f \cdot n_f}{V_c \cdot \rho_f} \quad (1)$$

3. RESULTS

The results of the tensile tests are provided in form of stress-strain curves for UD material parallel and perpendicular to fibre direction, as well as for BD material parallel and in $\pm 45^\circ$ to fibre direction. The standard deviations of the data are provided as semi-transparent areas in the graphs. For all results, the FVCs must be considered, which are presented in Table 5 and compared and discussed in Fig. 10. The stress-strain curves of the four UD0 prepregs B2-CE, B2-FP, B1-FPB and L1-ET are shown with the transition of E^I to E^{II} that are marked as red dots. Fig. 5 compares the UD0 material produced with the VARI method,

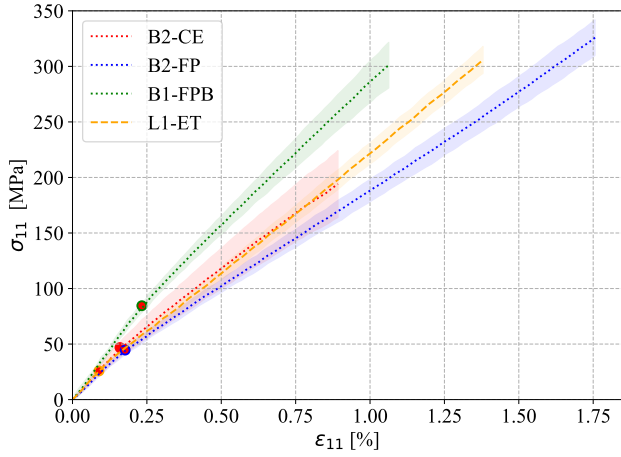


Fig. 4: Prepregs: Stress-strain diagram for UD fibres in 0°

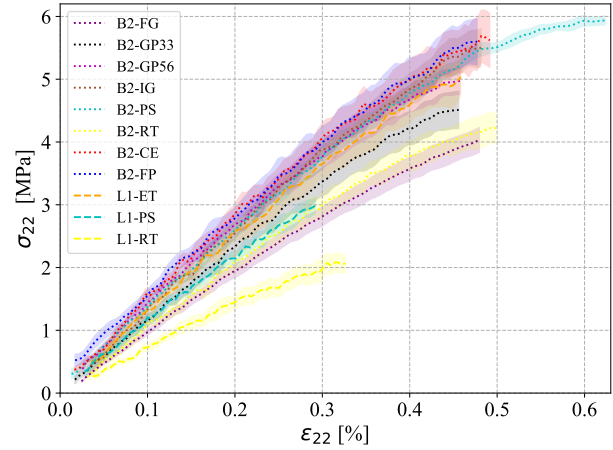


Fig. 6: Stress-strain curve for UD fibres in 90° normalised by FVC

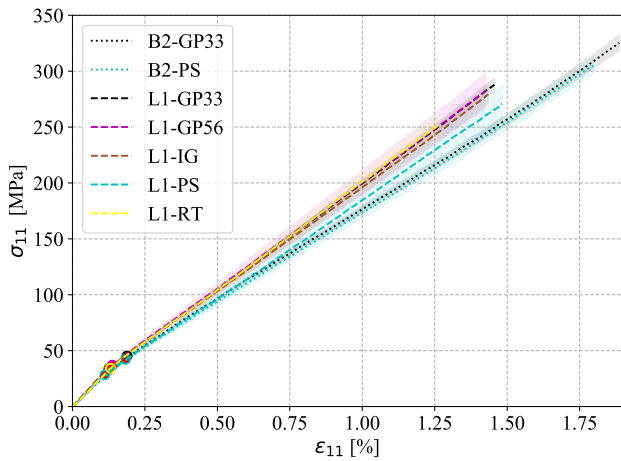


Fig. 5: VARI: Stress-strain diagram for UD fibres in 0°

showing a trend of L1 fibres being $\approx 15\%$ stiffer with $\approx 10\%$ less strength and a lower ϵ_f of around 25%. The ultimate tensile stresses of composites with epoxy resins are 50 – 70% higher compared to the B2-CE with furan resin, which also has the lowest fracture strain. Although the fibre is the load bearing component in UD0, the matrix has a strong influence on the fibres and hence their tensile properties.

The tensile tests for UD90 compare the resins that are carrying the load as well as the fibre-matrix adhesion. The results in Fig. 6 show σ_{22} normalised by the FVC for better comparison. Looking at the fibres, B2 have a higher stiffness, strength and fracture strain compared to L1. A possible contribution could be the polyester (PES) weft yarns of Bcomp UD fibres that hold the UD flax rovings together perpendicular to fibre direction, as seen in Fig. 1.

The data in Fig. 7 shows the stress-strain relations for bidirectional weaves loaded in 0°/90° to the fibre. Similar to the results in UD0, the prepreg with furan resin has considerably lower R_m as well as ϵ_f in contrast to epoxy resin. Comparing the fibres, B3 shows higher strength and

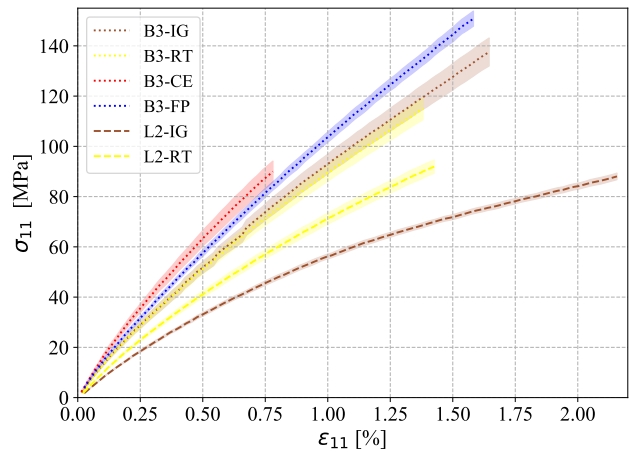


Fig. 7: Stress-strain curve of BD weave in 0°/90°

stiffness compared to the weave L2 of Ecotechnilin. This originates from the different fibre bundle types of the two weaves as described in the previous chapter.

The two BD weaves, B3 and L2, are also tested in 45° to fibre direction, which leads to the relation of shear stress versus shear strain in Fig. 8. Composites with L2 weave show a high fracture strain of up to 17.8% compared to the maximum of 8.5% for B2. The highest shear strength is reached by the prepreg B3-FP of 41.2 MPa.

The bilinear behaviour of the young's modulus parallel to the flax fibre direction can be observed in Fig. 4 and 5 with a decrease of E at yield points between 0.10 – 0.25% of strain. The decrease in stiffness is also shown in Fig. 9 as the incremental young's moduli of B2-CE, B2-FP and L1-ET over strain. Two main stiffness zones can be detected, which are visualised with continuous lines.

The FVC, bio-content and density of the test samples are given in Table 5. In case of multiple test panels per material combination, the average value is used. In Fig. 10 the strength R_m is shown against the FVC for UD0 and BD0. It can be observed that R_m is increasing with a higher

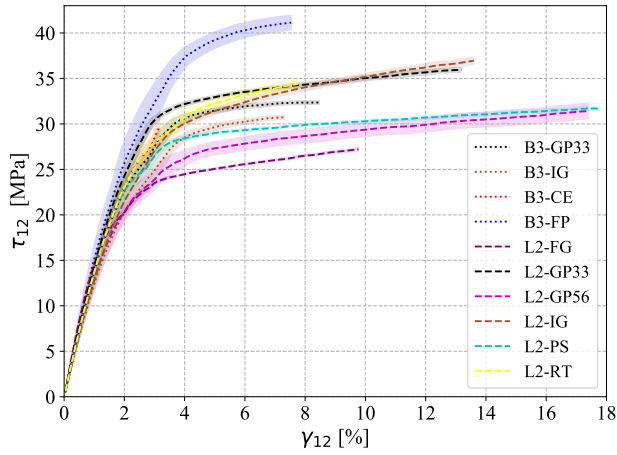


Fig. 8: Stress-strain curve of BD weave in $\pm 45^\circ$

Table 4: Young's moduli and tensile strength for UD0

material	R_m [MPa]	E^I [GPa]	E^{II} [GPa]	ε_f [%]
B1-FPB	301.1	36.88	26.51	1.06
B2-FP	325.9	25.08	17.61	1.75
B2-CE	194.1	29.56	20.40	0.89
B2-GP33	325.3	23.67	16.27	1.85
B2-PS	305.0	23.17	16.49	1.76
L1-ET	311.4	29.20	21.58	1.38
L1-GP33	287.7	26.48	19.06	1.43
L1-GP56	282.1	27.01	18.98	1.39
L1-IG	279.3	26.65	18.60	1.40
L1-PS	270.6	25.61	17.60	1.45
L1-RT	252.2	26.39	19.20	1.24

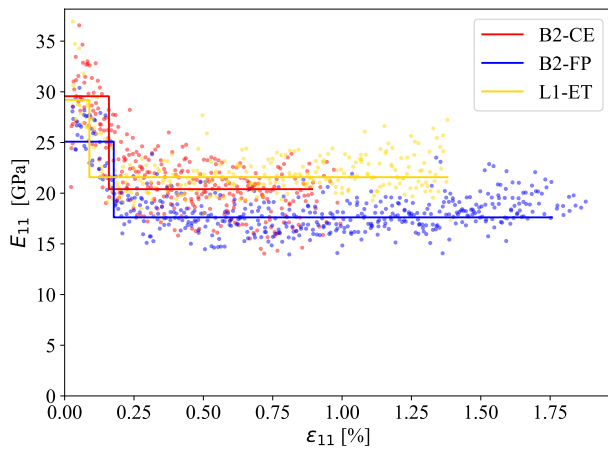


Fig. 9: Bilinear young's modulus of prepregs

Table 5: Properties of investigated materials

material	FVC [%]	bio-content [%]	density [g/cm^3]
L1-PS	41.6	57.3	1.14
L1-RT	40.7	60.9	1.15
L1-GP33	42.8	58.2	1.18
L1-IG	41.2	58.3	1.16
L1-GP56	39.1	63.0	1.15
L1-ET	49.6	49.6	1.11
L2-PS	33.0	51.0	1.20
L2-RT	35.1	57.2	1.14
L2-GP33	33.8	51.7	1.19
L2-IG	34.5	53.5	1.17
L2-GP56	35.2	60.6	1.15
L2-FG	26.6	41.9	1.30
B1-FPB	45.7	57.6	1.14
B2-PS	43.1	58.4	1.22
B2-RT	43.1	62.5	1.11
B2-GP33	33.9	51.7	1.14
B2-IG	43.2	59.6	1.09
B2-GP56	40.0	63.5	1.23
B2-FG	23.2	39.2	1.28
B2-FP	49.8	49.8	1.19
B2-CE	49.8	99.0	1.19
B3-RT	40.4	60.7	1.06
B3-GP33	42.9	58.3	1.17
B3-IG	41.7	58.6	1.12
B3-FP	46.1	46.1	1.19
B3-CE	52.4	99.0	1.16

FVC. The materials B2-CE and B3-CE with furan resins are outliers due to the lower level of strength. An exception is also B2-GP33 with a high strength at a comparatively low FVC. The dot size refers qualitatively to the young's modulus. For UD0 there is no clear trend recognisable, in comparison to BD0 with an increasing E for higher FVC.

A great difference between composites with furan and epoxy resins can be observed in their fracture behaviour. Composites with furan resin show a brittle fracture throughout the test specimens and test methods. Their fracture strains are 0.89% for UD0, 0.49% for UD90, 0.78% for BD0 and 3.19% for BD45. In contrast, the epoxy and bio-epoxy resin display a mixture between brittle and ductile behaviour in fracture. For tests of UD0, the fibres start tearing at several positions across the specimen with fibres sliding off of each other having fracture strains between 1.76% – 1.89% for Bcomp and 1.27% – 1.48% for Ecotechnilin fibres. Tests with BD45 show a more ductile fracture behaviour with fracture strains ranging of 7.33% – 8.48% for Bcomp and 7.5 – 17.8% for Ecotechnilin weaves.

The primary and secondary young's modulus parallel to fibre direction are shown in Fig. 12 of the UD fibres L1 and B2 with SD given in red colour. L1 and B2 are directly compared with the resins PS as well as GP33. L1 of Ecotechnilin has a 10.5% higher E^I with PS and 11.9% higher E^I with GP33 compared to B2. E^{II} with PS and GP33 is 6.7% and 17.1% higher respectively compared to B2. Both fibres show an increase of stiffness with GP33

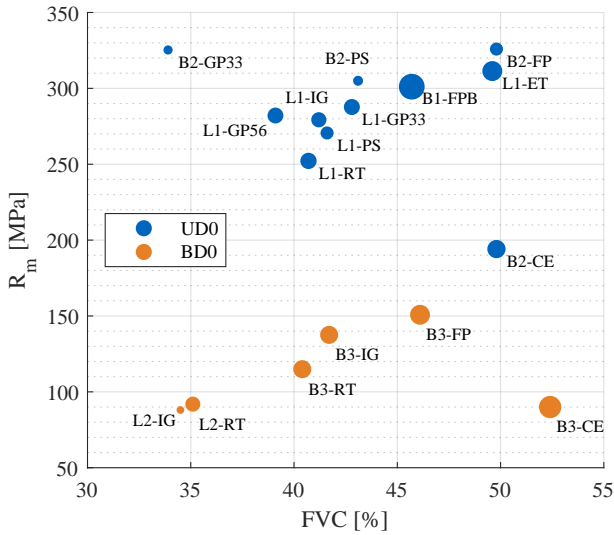


Fig. 10: Strength dependency on FVC

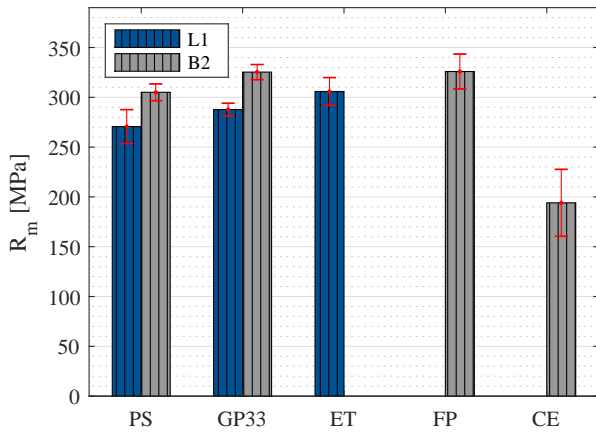


Fig. 11: Tensile strength of UD fibres in 0°

compared to PS. In this comparison, CE has the highest primary and ET the highest secondary young's modulus with 17.9% and 22.5% respectively higher compared to FP. In contrast, the values for R_m show that the Bcomp fibres are stronger compared to Ecotechnilin. Looking at the results for R_m and E of BD0 in Fig. 13 and 14, the weave B3 is 25%-57% stronger and 26% to 64% stiffer compared to L2.

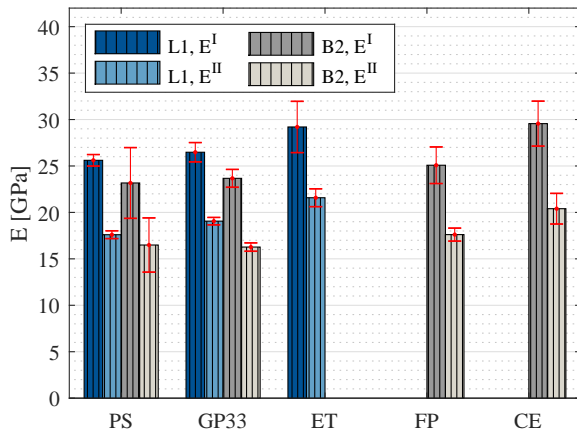


Fig. 12: Young's modulus of UD fibres in 0°

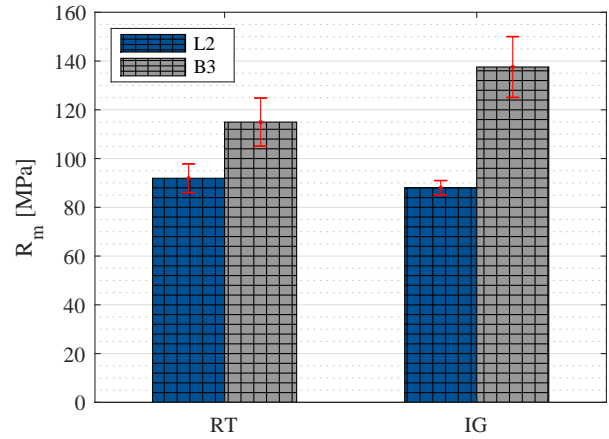


Fig. 13: Tensile strength of BD fibres in 0/90°

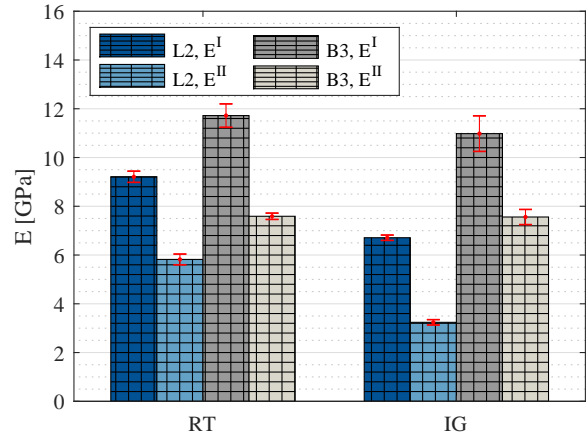


Fig. 14: Young's modulus of BD fibres in 0/90°

The shear strength and modulus of BD45 are compared with the resins GP33 and IG in Fig. 15 and 16. The weave L2 has with both resins a higher τ_{max} and G compared to B3.

Limitations

Errors in the results accumulate due to 1) production process (fibre orientation during layup), 2) sample preparation (cutting processes of test specimens), 3) measurement setup of tensile tests (load cell) and 4) data evaluation, which are not accounted for in the presented data. Due to the hydrophilic behaviour of flax fibres, the influence of cutting processes on test specimens are investigated. Three methods are compared using guillotine shears, laser cutter and water cooled buzz saw. For the shown tensile test results in this study, the water cooled buzz saw was used except for B1-FPB being cut with guillotine shears. The results in Fig. 17 compare the R_m of the preregs B1-FPB, B2-FP and L1-ET. There are no large discrepancies in tensile strength between the methods for epoxy composites with only a slightly smaller SD using the laser cutter. However, for furan the SD using the buzz saw has more than doubled with decrease in R_m . It must be noted that the specimens cut with the buzz saw had glass fibre caps

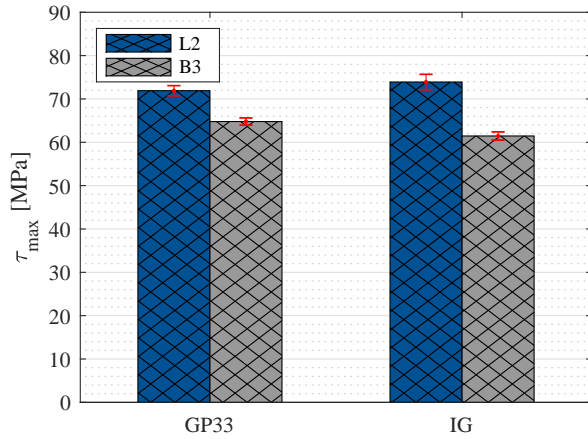


Fig. 15: Shear strength of BD fibres in +-45°

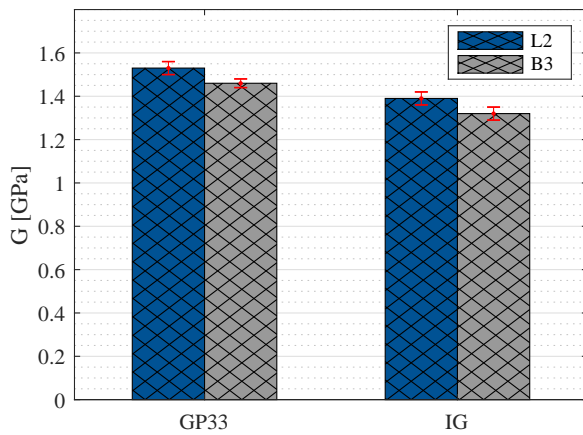


Fig. 16: Shear modulus of BD fibres in +-45°

at clamping. The norm DIN EN 2561 describes specimen types A and B with glass fibre composite caps at the clamping and type C without caps. All data in this study is derived using specimens with caps except for B1-FPB. The comparison for laser cutter shows that without caps, the UD0 test specimen has higher R_m of 6.3%.

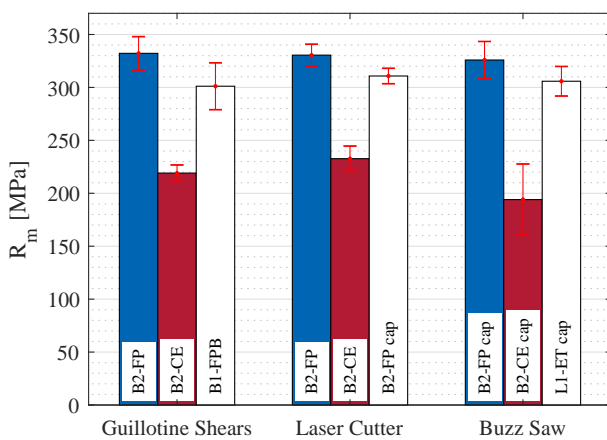


Fig. 17: Comparison of cutting methods for tensile test specimens

4. CONCLUSION

This paper provides an overview of currently available high-performance flax fibre-reinforced composites. Although they are a promising renewable material, its rapidly changing availability is still a limiting factor for a continuous application and research. With the aim of a bio-content of over 50%, the composites in this study were produced in combinations of five different flax fibres and ten resins, which were then experimentally investigated on their tensile properties. The following conclusions are drawn from this study:

1. The data of this study provides a starting point for simulations with FFRCs. In order to complete the set of data, it is however still necessary to provide Poisson's ratios and conduct further tests for compression and shear with UD laminates.
2. The resin type has a strong influence on the mechanical properties of the flax fibre laminates. The greatest difference can be observed between epoxy and furan resin. The latter induces a brittle fracture behaviour and lowers tensile strength to 59.5% compared to epoxy composites. The difference between the epoxy resins is shown in a range of 23.4% with L1 and 6.8% with B2 for strength and a range of 19% with L1 and 7.9% with B2 for stiffness.
3. The fibre volume content is crucial for the mechanical properties of the material. This study shows that this is strongly dependent on the resin system and the manufacturing process. Best results for tensile strength were achieved with prepreg systems having the highest FVC.
4. High standard deviations for some results show that firstly, natural fibres are imperfect compared to synthetic fibres, and secondly that errors can accumulate during production, preparation and testing.
5. The influence of cutting methods and preparation of composite structures must be considered during design, as heat introduction or water contact might influence the mechanical properties.
6. With high-performance flax fibres being still in development, the widely varying differences between the investigated combinations show that there is still a need for further research into process optimisation.
7. The bilinearity of the young's modulus was observed for UD0 fibres B1, B2 and L1. The yield point varies a lot throughout the tested materials with the bio-epoxy prepreg showing the highest strength, stiffness and strain of the primary section.

8. The handling of the raw materials during the production process defines their application. The great advantage of prepregs is the good applicability for complex three-dimensional moulds.
9. Comparing the results of this study to findings of the preceding research project *InteReSt* [10], a higher tensile strength of 20.7% for UD0 and 63.8% for BD0 as well as a 15.3% higher E^I for UD0 has been found.

5. OUTLOOK

There is a large potential to optimise the process and hence the quality for all applied material combinations. Each composite can be improved by adapting moulds, auxiliary materials and the curing cycle. An optimum FVC of 60% should be targeted, which was not reached for the test specimens in this study. Fibre treatment of flax fibres for better fibre-matrix adhesion can further improve strength, as much as up to 23.5% for furan resins and 30% for epoxy resins [25, 30]. When processed correctly, FFRCs can be a suitable substitution for glass fibre-reinforced composites considering their specific mechanical properties [10]. The fracture behaviour must be further investigated as it varies strongly with the resin types and could provide relevant data regarding airworthiness. Finally, cutting methods and mechanical post-treatment of composite structures must be further studied in order to maintain the mechanical properties for complex structures.

Acknowledgment

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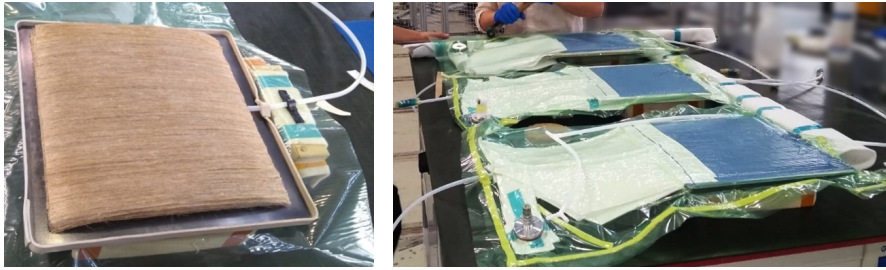
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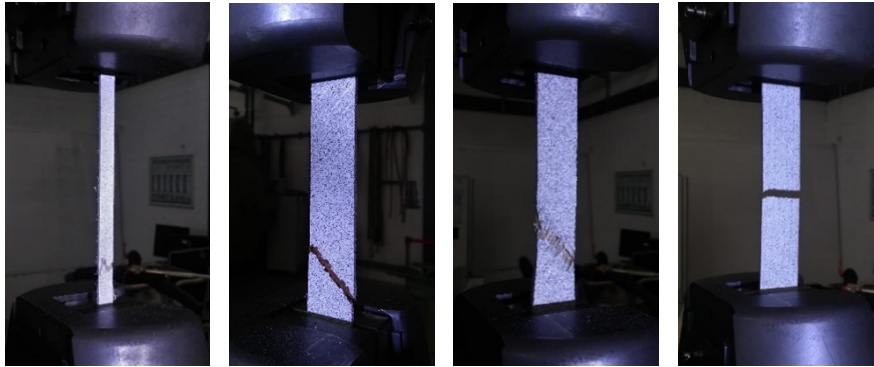
APPENDIX:



(a) L1 fibres

(b) resin infusion

Fig. 18: VARI production process of flax fibres with bio-epoxy



(a) UD0

(b) BD45

(c) BD45

(d) BD0

Fig. 19: Tensile test specimens