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Linen Fibres Based Reinforcements for Laminated Composites

Abstract
Bast fibres have been used as fillers in plastics for many years, most often in the form of very short fibres or mats. If it is desired to produce composites with high mechanical properties the short fibres need to be replaced by continuous forms such as roving, yarn and fabrics to improve the mechanical parameters, e.g. tensile strength or flexural strength of composites reinforced with natural fibres. This study, conducted within FP7 project No 214467 NATEX: "Aligned Natural Fibres and Textiles for Use in Structural Composite Applications", covers the development of composites reinforced with fabrics prepared from flax fibres. The current article describes a study on blended yarns containing flax and polymers prepared by using different spinning methods and their application in the production of fabrics suitable for composite reinforcement. The results of the study showed that the way the flax and polymer fibres are distributed in the yarn cross section influences the mechanical parameters of composites.

Key words: *flax, natural fibres, blended yarn, composite reinforcement.*

Introduction

Natural fibres have become valuable raw materials for the reinforcement of composites [1 - 3, 5]. Lignocellulosic natural fibres, mainly flax and hemp, can be used for composites in the form of loose mass, roving, yarn or textiles in 2D or 3D structures: woven fabrics, knitted fabrics, and non-wovens [4]. The reinforcement of composites can be prepared from pure natural fibres or from blends of natural fibres and polymer filament or staple fibres [4]. The necessity of the development of light composites with desired mechanical properties has been forced by different industry sectors, e.g. automobiles, ships, trains, machine constructions and others, which has led to the undertaking of work on composites reinforced with natural fibres by many researchers [6 - 13].

The composites developed, dedicated to structural application, need to meet requirements in terms of good mechanical properties. The aim of the study undertaken was the investigation of composite reinforcements based on hybrid textiles made of flax fibres in blends with thermoplastic polymer fibres like polylactic acid (PLA), polypropylene (PP) and polyethylene terephthalate (PET). These bio-based and oil-based polymers were selected because they cover 85% of the structural applications of composites, including transport systems (automotive), energy systems (solar power), agricultural machinery (tractor casings) and shipbuilding. The main advantage of composites from natural fibres and thermoplastic biopolymer PLA is biodegradability. Even though composites made of natural fibres and oil-based polymers are not biodegradable, they are attractive because they are characterised with good mechanical properties.

The study was a part of research conducted within FP7 project No 214467 NATEX: "Aligned Natural Fibres and Textiles for Use in Structural Composite Applications", where natural fibre reinforcements were developed for composites potentially applicable in the transport industry and renewable energy sector. The main objective of the project was to promote the utilisation of natural fibres in structural applications where traditionally glass fibres have been usually used. Based on flax fibres and manmade polymer, blended yarns were developed for the preparation of hybrid fabric suitable for laminate reinforcement. The laminated composites used in the study were obtained by employing the vacuum consolidation processing technique.

Investigation of the textile reinforcement was conducted in the following sequence: fibres → yarn → fabric → composite, which is illustrated in ***Figure 1***.

To achieve the high mechanical performance of composites reinforced with natural fibres, it is necessary to pay attention to fibre distribution in the matrix because this aspect strongly influences the quality of the processing and final composite [13].

The mixing of natural fibres and manmade polymer to obtain hybrid yarns dedicated to woven reinforcements was conducted with the application of the following methods:
- Mixing of both types of short fibres in a mass at the carding stage,
- Twisting of two yarns – 100% flax and polymer filament,
- Core yarn formation with the use of flax fibres as the core and polymer filament as the sheath.

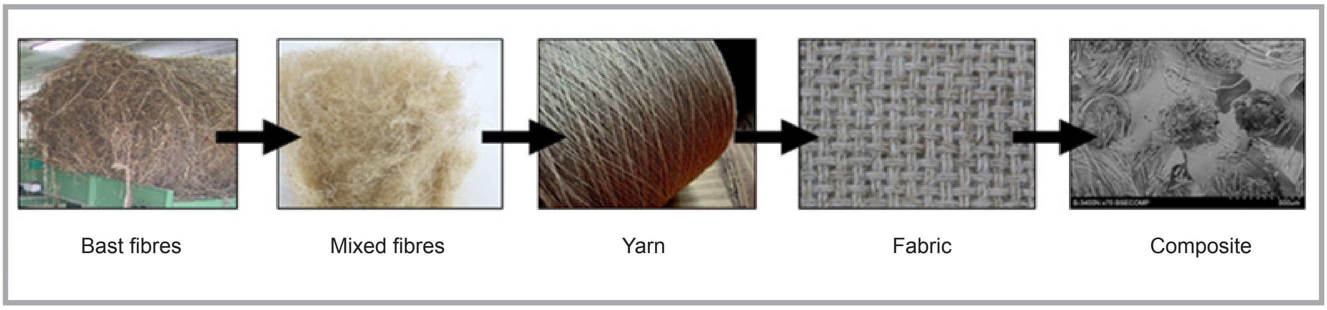

Figure 1. *Sequences of natural fibres reinforcement investigation.*

Application of different spinning methods allowed to obtain diversity in the distribution of flax and polymer fibres in cross sections of the yarns. Fibre distribution in the yarn plays a significant role in the creation of proper conditions for fibre wettability and adhesion between fibres and matrices, which in consequence has an effect on the composite formation and its properties [14 - 16].

Blended yarns were spun using the following spinning systems:
- cotton ring spinning system,
- woollen spinning system,
- twisting two yarns: flax and man-made yarn,
- hollow spindle spinning system.

The mechanical properties of all the types of fibres, yarns and fabrics were evaluated in terms of their physical and mechanical properties to confirm which parameters have a significant effect on laminates reinforced by these textiles.

Materials

Fibres

Fabrics dedicated to the reinforcement of laminated composites were made from two groups of raw material: flax noils and thermoplastic polymer fibres: polypropylene, polyester and polylactic acid (PLA).

The flax noils were prepared according to the requirements of the spinning system selected; the mechanical modification applied allowed to obtain fibres with a length suitable for the spinning system chosen, low linear density and high purity.

The flax noils used in the study were characterised by the following parameters:
- Length of noils – 88.6 mm
- Linear density – 1.05 tex

Application of the cotton ring spinning system requires adaptation of the flax fibre length to a proper size, similar to cotton; hence the cottonisation of the flax noils was conducted. The average length of the flax fibres was 46.2 mm and the linear density 0.83 tex after mechanical modification (cottonisation).

The following thermoplastic polymers in the form of staple fibres and filaments were applied to the blends with flax fibres to obtain hybrid yarns:
- staple polymer fibres for the cotton and woollen spinning systems:
- PLA – S 111 XNS 134A: 1.65 dtex × 38 mm,
- polypropylene – 3.3 X/50 HY3000 Natural White Hyg
- polyester – Grilon KE 170 (FIBRECORE, UK)
- filament polymer:
  - for the hollow spindle spinning system – PLA, 84 dtex
  - for twisted yarn of natural fibre single yarn with filaments:
    - 225 dtex PLA filament 98 t.p.m S
    - 2×150 dtex PLA filament 98 t.p.m S
    - 220 dtex PP ex Draker 98 t.p.m S
    - LPET POY dtex 560 f96 98 t.p.m S

Blended yarn - natural/man-made fibres

The application of different spinning systems for the manufacture of blended yarns from flax and man-made fibres allowed a diverse way of mixing and laying the fibres in the yarn cross section. In the case of the cotton or woollen spinning systems, flax and polymer fibres were mixed with staple man-made fibres on a carding machine, which resulted in random but uniform distribution of fibres throughout the whole yarn.

Longitudinal SEM images of all types of blended yarns developed in the study are presented in ***Figures 2 – 10***, whereas the SEM views cross section of yarn: FLAX/PLA (Core-Flax) 100 tex, Hollow spindle spinning technique are presented in ***Figure 11***.

A twisted yarn was obtained by joining two different yarns: flax single yarn and polymer filament, resulting in fibres grouped in two bundles in the cross section of the yarn; half of the yarn cross section was covered by flax fibres and the other half by man-made fibres, which is visible in ***Figures 8*** and ***9***. Fibre distribution in the cross section of hollow spindle yarn was very different – flax fibres were concentrated as a core in the central part of the yarn cross section and were surrounded by man-made polymer filaments, as can be seen in ***Figure 10***.

The preparation of blended yarn with the use of the spinning systems described above creates a variety of conditions influencing the interaction between the yarn and matrices used for the formation of laminates investigated in the study.

Table 1. *Properties of blended yarns made from flax and man-made fibres spun with the use of different spinning methods.*

Yarn composition and nominal linear density	Real linear density,	Breaking force,	CV of breaking force,	Elongation,	CV of elongation,	Tenacity,	Nominal twist,
	PN-EN ISO 2060:1997	PN-EN ISO 2062:2010					PN-EN ISO 2061:2010
	tex	N	%	%	%	cN/tex	twist/m
Flax/PP 50/50 Cotton ring spinning 105 tex	105	10.5	15.5	12.4	36.9	10.0	337
Flax/PLA 50/50 Cotton ring spinning 116 tex	116	10.2	13.1	7.4	36.8	8.8	447
Flax/PET 50/50 Cotton ring spinning 138 tex	138	6.4	16.7	8.6	3.4	4.6	375
Twisted yarn: 1×flax 110 tex + 2× LPET POY 560 dtex f 96 98 t/m S	223	24.2	7.5	5.4	16.6	10.8	89
Twisted yarn: 1×flax 200 tex + 8× PLA filament 225 dtex 98 t/m S	407	68.1	10.0	30.9	7.3	16.7	86
Flax/PLA 50/50 Wool spinning 110 tex	110	6.0	10.2	14.5	46.3	5.5	358
Flax/PP 50/50 Wool spinning 110 tex	110	6.0	21.0	15.1	16.0	5.4	288
Flax/PET 50/50 Wool spinning 110 tex	118	4.2	14.9	10.1	50.6	3.5	334
Flax/PLA Hollow Spindle (Core-Flax) 100 tex	86	10.2	24.1	1.6	15.8	11.9	15 000 turns of spindle /min

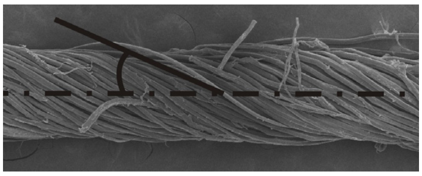

Figure 2. *Longitudinal yarn view FLAX/PP, 105 tex, cotton ring spinning.*

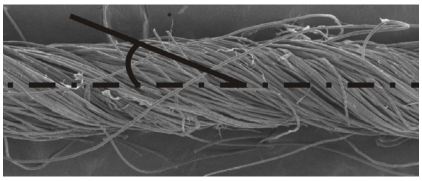

Figure 3. *Longitudinal yarn view FLAX/PLA, 116 tex, cotton ring spinning.*

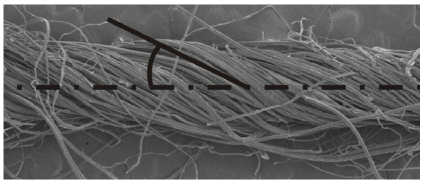

Figure 4. *Longitudinal yarn view FLAX/PET, 138 tex, cotton ring spinning.*

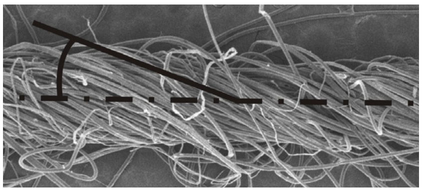

Figure 5. *Longitudinal yarn view FLAX/PET, 110 tex, wool spinning.*

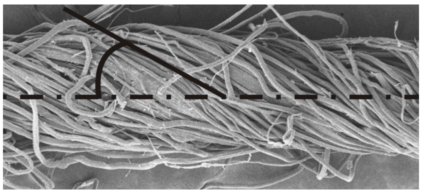

Figure 6. *Longitudinal yarn view FLAX/PP, 110 tex, wool spinning.*

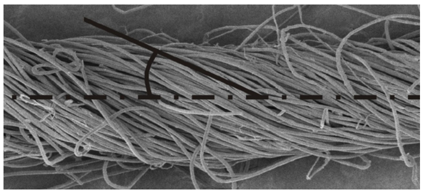

***Figure** 7. Longitudinal yarn view FLAX/PLA, 110 tex, wool spinning.*

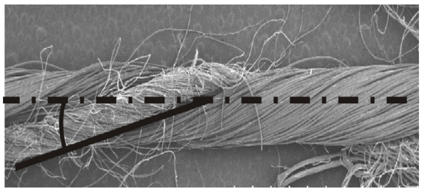

Figure 8. *Longitudinal view of twisted yarn FLAX 110 tex +2× LPET POY 560 dtex f 96, 227 tex.*

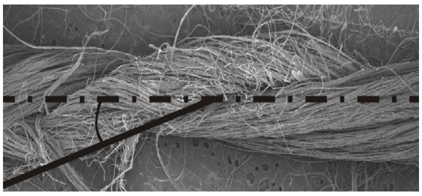

Figure 9. *Longitudinal view of twisted yarn FLAX 200 tex + 8× PLA filament 225 dtex f 98, 407 tex.*

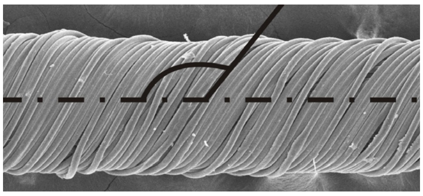

Figure 10. *Longitudinal view of yarn: FLAX/PLA (Core-Flax) 100 tex, Hollow spindle spinning technique.*

All the blended yarns used in the study were tested according to international standards. Results of the yarn evaluation are presented in ***Table 1***.

Mechanical properties of the yarn, such as the breaking force and tenacity, are key elements in terms of the possibility of conducting further processes of textile manufacture. In the study, blended yarns prepared by twisting two separate yarns e.g. 100% flax and polymer filament showed the best mechanical properties. This was related to:

- The system of yarn production: the tenacity of the final yarn resulted from the sum of the tenacity of components with increased strength from the additional twist applied to combine the two yarns
- The fact that continuous filaments are usually stronger than staple fibre yarns
- Significant differences in the linear density of the twisted yarn compared to other blended yarns. Yarns spun by the wool spinning system were characterised with poorer mechanical properties due to the loose irregular structure with many free ends of fibres sticking out from the yarn, which is visible in ***Figures 5*** and ***6***. Blended yarns spun by the cotton ring system were characterised with a higher tenacity in comparison to the wool spinning system – the fibre positions were more regular in relation to the yarn axis. The tenacity of blended flax/PET yarns spun by the cotton as well as wool spinning system were very low, which was a consequence of the low quality of PET fibres used.

Due to the very low tenacity of blended flax/PET yarn spun by the wool spinning system, this yarn was excluded from the further processes of fabric preparation. Other blended yarns with a tenacity below 7 cN/tex were used for fabric manufacture by applying special process conditions, such as reduction of the weaving velocity.

Blended yarn spun using the hollow spindle spinning system had very good mechanical properties. The polymer filament was applied as a sheath covering the flax fibre core, resulting in an increase in yarn tenacity.

Fabric prepared from blended yarn developed

Blended yarns spun from flax and thermoplastic polymer fibres were used in the weaving process to manufacture fabric samples dedicated to laminate reinforcement. All fabric samples were made in plain weave, but with a different thread

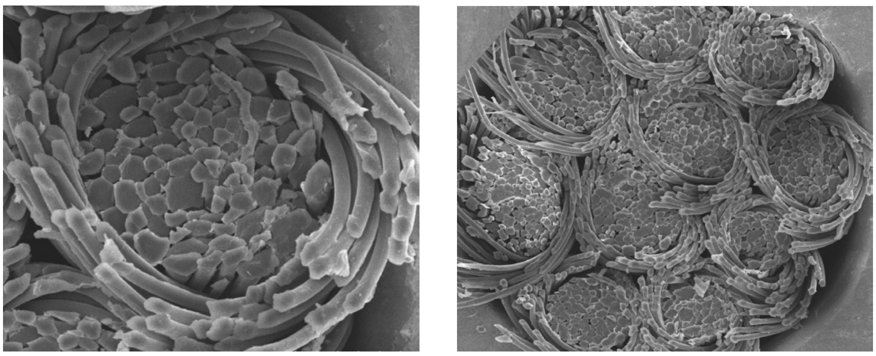

Figure 11. *Cross section of yarn: FLAX/PLA (Core-Flax) 100 tex, Hollow spindle spinning technique.*

__Table 2.__ Properties of fabrics made from blended yarns.

Sample	Material		Weave	Thread density,	Mass per square metre,	Average breaking force,	CV of breaking force,	Average elongation,	CV of elongation,	Air permeability,
				PN-EN 1049-2:2000	PN-ISO 3801:1993	PN-EN ISO 13934-1:2002				PN-EN ISO 9237:1998
				threads/dm	g/m^2	N	%	%	%	l/m^2/s
498	Flax/ PP 50/50 105 tex, 337 t/m Cotton ring spinning	Warp	plain 2/2	108	237	513.1	4.4	20.0	8.7	2282
		Weft		104		514.4	7.6	21.0	7.0	
499	Flax/ PLA 50/50 116 tex, 447 t/m Cotton ring spinning	Warp	plain 2/2	108	255	458.2	6.7	10.6	5.8	2834
		Weft		108		495.9	5.7	12.8	9.5	
500	Flax/ PET 50/50 138 tex, 375 t/m Cotton ring spinning	Warp	plain 2/2	95	266	259.6	4.1	15.6	4.3	1842
		Weft		95		264.8	3.4	17.8	16.6	
501	1x flax 110tex +2x LPET POY 560 dtex f 96 98 t/m S	Warp	plain 2/2	60	272	456.0	0.6	67.7	79.4	3946
		Weft		50		417.0	4.1	6.3	6.4	
506		Warp	plain 2/2	82	391	675.3	9.3	102.7	7.6	1526
		Weft		80		642.6	6.0	39.9	137.7	
504	1x flax 200tex +8x PLA filament 225 dtex 98 t/m S	Warp	plain 1/1	31	270	789.0	5.3	30.9	4.1	5327
		Weft		32		727.5	4.8	31.0	0.6	
505		Warp	plain 1/1	41	356	1022	6.8	34.1	0.9	2894
		Weft		43		1022	2.3	35.0	1.5	
515	Flax/PLA 50/50 110 tex 358 t/m Wool spinning	Warp	plain 2/2	100	233	269.4	5.7	11.9	7.3	2349
		Weft		105		257.8	4.8	15.7	5.2	
516	Flax/PP 50/50 110 tex 288 t/m Wool spinning	Warp	plain 2/2	100	233	410.9	9.1	20.8	13.1	2069
		Weft		105		346.8	5.1	23.0	4.1	
599	Flax/PLA 100tex Hollow Spindle	Warp	plain 2/2	100	216	644.9	9.3	5.7	2.7	8200
		Weft		100		577.0	6.3	6.3	6.5	

density in the warp and weft directions due to different yarn linear densities, which caused that the fabrics made from twisted yarns had the least number of threads per decimetre. Properties of the fabrics prepared from blended yarns are presented in ***Table 2***.

The mechanical properties of fabrics are influenced by the mechanical properties of the yarn used and the number of threads per unit length in the woven structure. The highest values of breaking force were shown by fabric Nos. 505, 504 and 506, made of twisted yarn, as well as No 599, made of hollow spindle yarn. The fabric made of blended yarns spun by the wool spinning system were characterised with the lowest value of breaking force due to the low tenacity of the yarns.

Experimental

There are different organic matrices such as the thermoplastic, thermosetting and elastomeric that combined with appropriate fibres can be used in the synthesis of composite materials. For the formation of laminates the following thermoplastic matrices were applied:

- Bio-based - polylactic acid (PLA) (Trader-Lorenzo Ciani, Chaney International S.a.S., Prato, Italy) - renewable, biodegradable, aliphatic polyester derived from lactic acid.
- Oil-based:
  - polypropylene (PP) (Drake Extrusion Ltd., UK) - a semicristalline apolar thermoplastic material.
  - polyethylene terephthalate (PET) (BIELTEX, Spain) - characterised by toughness, high heat resistance and stiffness; PET is engineered thermoplastic with excellent strength and rigidity for a broad range of applications.

The laminates were formatted with the use of the vacuum consolidation technique for thermoplastics. Each fabric was cut to produce four rectangular pieces, with the size of each being similar to a 25 cm square. The fabric samples prepared were laid flat on an aluminium plate and sealed in a vacuum bag.

The cloth was dried under a vacuum at 80 °C for three hours to eliminate the risk of sample shift during the process. It was known that some fabrics, particularly those containing thermoplastic fibres, were liable to change shape if dried in an oven at a high temperature, which is caused by the thermoplastic fibres in the fabric shrinking at elevated temperatures. It was found that the application of moderate pressure could constrain the fabric so that it did not change shape or dimension.

None of the fabrics distorted during drying. Mass loss was typically around 2.0-2.5%.

The fabrics were placed one over the other in a way that warp directions were aligned. The stack was placed between aluminium sheets using PTFE films for release. A thermocouple was attached to one of the central layers of fabric at about 25 mm from one corner. The sheet was then loaded into a press, which had been preheated. Each sheet was pressed for 5 minutes at 195 °C under an applied pressure of 3 MPa based on the nominal sheet area (due to the different sizes of sheet the load was varied from one pressing to the next). Timing was started once the temperature recorded by the thermocouple in the sheet exceeded 190 °C. All the sheets reached the final temperature of 195 ± 2 °C, and then were cooled under pressure to below 50 °C before being removed from the press.

It was noted that in all cases there was resin loss from the sheet – about 10 - 25 mm width around the edge, so the fibre/resin ratio would change during pressing from that present in the yarn.

Pressed sheets were left for 2 - 3 days at ambient conditions before being tested. The sheets were cut to make samples for uniaxial tensile tests and density measurements. The tensile test samples were cut with their axis parallel to the warp direction of the fabrics. The testing was conducted using an Instron mechanical testing machine. The sample was extended at 3 mm min^{-1} and the load recorded. To obtain an accurate figure for Young's modulus, an extensometer was used. The density was measured using Archimedes' principle.

Results

Table 3 shows the tensile strength and modulus of the sheets which were pressed. The results have been grouped together according to the composition of the materials. It can be concluded that, in general, the strength of the composites' reinforcement with the hybrid fabrics developed in this study (excluding fabric from hollow spindle yarn) is the lowest for laminates with PLA matrices. The tensile modulus value is the lowest for composites with PP matrices, samples No 498 & 516 and PET commingled sample No 500.

To calculate the theoretical densities, the following values were assumed for the individual components in the composites:

Flax	1.50 g/cm^3
Polypropylene	0.92 g.cm^3
Poly lactic acid	1.24 g/cm^3
PET	1.40 g/cm^3

The lower than expected density values of the laminates developed indicate that some porosity appeared during the composite formation, despite a slight loss in the lower density component during pressing.

The PLA containing fabrics produced composites with a high elastic modulus but very low strength, apart from the material prepared from hollow spindle yarn, gave the best properties of all the samples tested in the study. The fabrics based on flax and LPET gave composites with a high modulus and reasonably high strength. The mechanical properties of the laminate prepared with sample 500 were very poor.

Table 3. *Properties and construction of laminates made from blended fabrics & thermoplastic matrices.*

Fabric code	Tensile modulus, GPa	Elongation at break, %	Tensile strength, MPa	Actual density, g/cm^3	Theoretical density, g/cm^3	Matrix material
498	7.1	2.3	53.6	1.11	1.14	PP commingled
516	6.5	2.0	46.3	1.08		
499	11.6	0.4	37.2	1.36	1.36	PLA commingled
515	11.1	0.8	39.3	1.34		
504	9.1	1.0	43.8	1.36		PLA filament 225 dtex
505	10.0	1.0	43.9	1.34		
599	12.5	1.4	83.4		1.36	PLA filament sheath (hollow spindle yarn)
500	5.2	2.5	31.4	1.36	1.45	PET commingled
501	10.8	1.3	78.3	1.41		LPET POY 560 dtex
506	10.1	1.2	63.9	1.42		

Discussion

It would appear from the results of this study, that the introduction of thermoplastic yarns or fibres as the source of the matrix material for a natural fibre based textile reinforcement gives composites characterised with different mechanical properties of composites depending on the type of thermoplastic material.

Commingled PLA gives a lower strength but higher stiffness of laminates than commingled PP. The melting points of PLA & PP are both around 160 °C, although this does not mean they show the same melt flow at 195 °C. LPET is amorphous and does not have a well-defined melting point. Crystalline PET melts at 260 °C, so one would not expect a normal PET fibre to produce a properly consolidated material, which may explain why laminate 500 was weak and of low density: it was not properly consolidated. The two laminates made with LPET show a high strength and high modulus, although the modulus is not generally as high as the laminates made with PLA.

A comparative analysis of the laminates formed in terms of the reinforcement properties shows some differences between laminates reinforced by pairs of fabric samples No 501 and 506 as well as 498 and 516. Both samples in each pair of the fabrics were made from the same raw materials but differing in their structure.

Considering the pair of laminates reinforced with fabrics No 501 and 506, it was found that fabric with lower density and breaking strength gives a composite characterised with better mechanical properties, e.g. tensile strength and modulus. Both laminates reinforced with fabrics 501 and 506 were prepared in the same way and with the same matrices used, but differences in the mechanical properties of the composite's properties are visible, which can be explained by the looser fabric structure (which is confirmed by higher air permeability) of the reinforcement, which allows better polymer penetration and fibre bonding. It is visible that good conditions for resin penetration have a stronger effect on the improvement in composite tensile strength than the good mechanical properties of the reinforcement applied.

The pair of fabrics 498 and 516, prepared from the same raw materials: 50% flax/50% PP were characterised with very similar structure parameters, but the yarns used for the fabric manufacture were spun by different spinning systems. Comparison of laminates with hybrid fabric samples 498 and 516 indicate that the tensile strength, tensile modulus and elongation at break are higher for laminate reinforced with fabric made from yarn spun with the cotton ring system, which can be explained that in the yarn spun by the wool spinning method, the fibres were tangled and irregularly laid in reference to the yarn axis and a lot of free ends of fibres were sticking out (***Figure 6***), which influenced the reduction in tenacity of the yarn and hence the breaking force of the fabric. This irregular fibre laying with numerous fibre ends sticking out from the yarn and fabric structure caused a reduction in the composite's mechanical properties. According to the results of studies described by Coroller *et al* [13] and Krucińska *et al* [14], the homogeneity of fibre distribution in the matrix is an important aspect for the composite's mechanical properties.

In the case of yarn made with the cotton ring spinning method, the fibres were laid more regularly and tightly, as is visible in ***Figure 2***; such a structure had a positive effect on the laminate reinforced by the fabric prepared from this yarn. Additionally the higher air permeability of fabric 498 in comparison to 516 gives an indication of possibly better polymer penetration between flax fibres, which improved laminate formation processing and composite properties.

The values of tensile strength of the laminate pairs reinforced with fabrics 504 and 505 as well as 499 and 515 are the lowest and did not show significant differences. This indicates that the application of bio-based thermoplastic polymer PLA in this structure of commingled sheet with flax fibres is not a proper solution for a composite dedicated to structural application, even though the values of their modulus are at a high level.

The composite with the highest strength and stiffness is the one reinforced with fabric made of hollow spindle yarn. This yarn is built from sheath - highly twisted PLA filament and a core which is low twisted flax yarn. The flax fibres are almost aligned parallel and close to the axis of the yarn, which resulted in the high homogeneity of flax fibre distribution and their unidirectional position in the laminates. This fact has a positive effect on the mechanical properties of the composite [13, 14]. During compression moulding the sheath melts to form a matrix and this flows into the core of the yarn. The flax fibres remain aligned with the yarn direction and hence are in optimum orientation for maximum strength and stiffness. In addition, the core of the hollow spindle yarn is relatively loose, which is confirmed by the high air permeability of the fabric; hence it is easy for the molten resin to impregnate the yarn. Since PLA and flax fibres are both polar, the matrix readily wets the fibres to give a good bond.

Conclusion

The results of the study showed that the structure of hybrid reinforcement of composites made of commingled natural fibres and thermoplastic polymer has a strong effect on their mechanical properties. The factors influencing composite quality are the homogeneity of fibre distribution in the matrices, the yarn and fabric structure, mainly their density and uniformity, and applied matrices. The mechanical properties of yarns and fabrics used as reinforcements do not influence the tensile strength of the composites.

It can be concluded that the structure of natural fibre reinforcement of the composite is responsible for ensuring proper conditions of adhesion between the fibres and matrices, e.g. resin penetration and fibre bonding, and finally for the composite's mechanical properties.

The fabric woven from a blended yarn made using the hollow spindle spinning system showed the best structure suitable for composite formation of all samples tested, e.g. uniform flax fibre distribution and their parallel position in the yarn, effective PLA filament contact with flax fibres and low textile density. Mechanical properties of the laminate based on the hybrid fabric made of hollow spindle yarn showed the highest value.

As a result of the study, the new biomaterials have been used to develop a complete panel system structure used in photovoltaic solar and thermal solar systems, several tractor casing parts, and in shipbuilding applications in order to manufacture access doors to the different compartments located in the navigating bridge.

Acknowledgement

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