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Influence of Natural Fibre Treatment on Interfacial Adhesion in Biocomposites

Abstract
Adhesion interaction in the fibre-matrix interface is important in all composites used in load-bearing applications. To improve interfacial adhesion, various treatment methods of the fibre surface are applied. In this work the influence of chemical and/or mechanical treatment on the adhesion interaction of polyester (UP) resin or poly(lactic acid) (PLA) polymer composites reinforced by flax or cotton yarns was investigated by means of contact angle measurements and uniaxial tensile testing. It was obtained that the tensile strength of UP or PLA biocomposites increases when cotton and flax fibre undergo mercerisation, twisting or pre-tension, while the effect of bleaching was negligible. The combination of treatment methods is especially effective when mercerised or twisted fibres are pre-tensioned. The investigations proved that fibre treatment creates possibilities to obtain natural fibre reinforced biocomposites with mechanical properties comparable to those of glass fibre reinforced composite.

Key words: *polymer-matrix biocomposites, flax and cotton fibre, adhesion interaction, mechanical testing.*

Introduction

Today mankind is greatly concerned with the harmful influence of plastics on human health, as well as environmental pollution. Renewable, recyclable and bio-degradable plastics can make a difference to the environment today and tomorrow. However, most bioplastics cannot compete economically in their present state of technological development, with the domination of petroleum based plastics. A more viable solution for composite manufacturing would be to combine petroleum and bio-based recourses to develop cost-effective products. Today biocomposites are considered as bio-pol-

ymers or synthetic polymers reinforced with natural fibres [1, 2]. Natural fibres are classified according to their origins, coming from plants, animals or minerals. Considering the relationship of price and mechanical properties, flax, jute and hemp are used as optimal reinforcement fibres. Biodegradable matrix polymers comprise both thermoset (furan, crops and oil based) and thermoplastic (lignin, poly(lactic acid), starch based (polyhydroxybutyrate) resins [1]. Among matrix polymers poly(lactic acid) (PLA) has generated great interest as one of the most innovative materials [3]. Previous investigations have proved that PLA can be processed similarly to polyolefin and works well as matrix material for natural fibre composites [4].

To replace non-degradable high strength synthetic and man-made fibres with natural fibres, the most important problems to be solved are their insufficient fibre-matrix adhesion and mechanical properties [2]. Interfacial adhesion is extremely important in all composites used in load-bearing applications. Improper adhesion leads to the debonding of components, which can cause the premature failure of the composite [5]. Therefore a number of chemical and mechanical surface modification studies on a variety of natural fibres have been devoted to enhancing interfacial adhesion between fibres and the polymer matrix. Conventional chemical modification methods are alkalisation, acetylation and bleaching. These methods are more frequently used due to their relative simplicity, low cost and efficiency. However, they are generally

performed under static treatment conditions [6].

Bleaching is the oldest textile yarn treatment method used until nowadays. During bleaching lignin, waxy residue and soils are removed from the natural fibre surface [7 - 9]. For this reason, the fibre cellulose content increases and the surface roughness enhances. Irregularities of the fibre surface improve mechanical interlocking at the interface [8].

One of the most popular chemical treatments of natural fibres is with alkali, also called mercerisation [10]. Sodium hydroxide is used to break hydrogen bonding in the network structure of the fibre's cellulose, thereby increasing the fibre's surface roughness. This treatment also removes lignin, wax and oils covering the external surface of the fibre's cell wall, depolymerises the native cellulose structure and exposes short length crystallites [10].

A combination of different fibre treatment methods is also used [8]. In manufacturing processes, to improve finishing, wetting and coloration properties, natural fibres can be mercerised and half-bleached, or fully mercerised and bleached.

Chemical fibre treatment methods are water based, resulting in high environmental pollution [11]. In comparison with chemical methods, mechanical treatment is more eco-friendly. Mechanical methods, such as stretching and twisting, do not change the chemical composition of the fibre; they modify its structural and

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Table 1. *Main characteristics of matrix polymers.*

Matrix polymer	Physical properties	Value
Unsaturated polyester resin	Viscosity Brookfield LVF, mPa	1100 - 1300
	Content of styrene, wt. %	43 ± 2
	Specific gravity, g/cm^3	1.1
Poly(lactic acid)	Relative viscosity (Viscotek method)	3.1
	Specific gravity, g/cm^3	1.24
	Melt index, g/10 min (210 °C)	15 - 30
	Melt temperature, °C	160 - 170

surface properties, thereby influencing the mechanical interlocking process of the polymer [2].

During twisting, an increase in the helix angle of the fibre increases the radial pressure, causing the overall strengthening of natural fibre yarns [12]. At the same time, the direction of principal stress changes – the higher the helix angle of the fibre, the lower the yarn strength. Because of these two competing effects, a twist level, called optimal twist, exists at which the maximum fibre strength is achieved. Also it is considered that for impregnated fibres with a higher twist level, a higher tension generates greater internal forces preventing the separation of the fibre and resin. Frictional forces between fibres and resin improve the load transfer among yarns [12]. To evaluate the interaction of these factors, optimisation of the fibre twist level for natural fibre reinforced composites is a key parameter [13].

Reinforcement pre-tension has several effects on the polymer-matrix composite's properties. The influence of pre-tension on both the interaction at the interface and matrix toughness was thoroughly investigated in [14, 15]. Pre-tension improves the flexural strength of matrix polymer by inducing compressive forces, which increases the toughness of the matrix. Compression is conditioned by stretching fibres during composite moulding. When the matrix solidifies due to the release of tension, a reinforcement compressive

force is applied to the matrix as the reinforcement seeks to contract to its original unstretched length. The matrix polymer becomes tougher and will have higher flexural strength [14]. At the same time, compressive forces generate frictional forces, which improves interaction at the interface [15]. Although the pre-tension technique is mainly used to improve the flexural strength of matrix polymer, in the case of natural fibres, pre-tension improves the tensile strength of the composite as well [16]. Vegetable fibres have a lack of orientation compared to that of high-orientated synthetic and man-made fibres. However, it is well known that during the tension process, natural fibre alignment and orientation are improved and its tensile strength increases [17]. Regretfully the effect of pre-tension on the properties of polymer biocomposites has not been studied.

The aim of this work was to evaluate the influence of various treatment methods of natural fibres on the adhesion interaction at the interface in order to obtain a polymer-matrix biocomposite with comparable properties to those of synthetic fibre reinforced composites. Particular attention was paid to the influence of natural fibre pre-tension on the biocomposite's mechanical properties.

■ Experimental

Materials
Flax (105 tex and 68 tex) and cotton (70 tex) fibre yarns, provided by JSC "Siūlas", Lithuania, were used as reinforcements. These natural fibre yarns were made from two single yarns twisted in the Z direction. Flax was chosen as a common high strength vegetable fibre for composite manufacturing. Cotton is attractive because of its higher elasticity due to lower fibre orientation (spiral angle up to 45°), which can ensure a higher effect of pre-tension. Also for comparison, 70 tex E-glass fibre roving, R & G Faserverbund-Werkstoffe GmbH,

Germany, was used. This fibre was silane treated in order to enhance adhesion interaction with unsaturated polyester resin.

For fibre impregnation, biodegradable poly(lactic acid) pellets 6201D, obtained from Nature Works LLC, USA, and orthophthalic polyester resin Polylite 440-M850, from Reichhold AS, Norway, were applied. The methyl ethyl ketone peroxide catalyst Norpol MEK 1 (2 wt%) with a colour indicator for polyester resin curing process initiation was added before resin application. The resin was mixed with the catalyst until its blue colour became grey. The main properties of the materials used are listed in **Table 1.**

Treatment methods
Mercerisation was carried out by immersing flax and cotton fibres in 5% sodium hydroxide (NaOH) solution for 24 h and afterwards in 30% NaOH solution for 1 min. After rinsing with demineralised water, the fibre was low loaded (*ca.* 6 MPa) and dried for 2 h at a temperature of 100 °C.

Bleaching was applied only on flax yarn with a linear density of 105 tex. It was immersed into 20% hydrogen peroxide solution containing NaOH (3.5 g/l) (pH = 11), heated at a temperature of 80 - 85 °C for 2 h, and afterwards rinsed with demineralised water. After rinsing, the low loaded (*ca.* 6 MPa) fibre was dried for 2 h at 120 °C.

Natural fibre bleaching with subsequent mercerisation were carried out using the methods described above.

Fibres with different twisting levels were produced on a twisting machine, type PL 31. The flax yarns (105 tex) were twisted to twist levels of 140 – 260 turns/m, while cotton yarns – to 360 – 500 turns/m.

Fibre pre-tension was applied during the joining with the polymer matrix. The upper end of the fibre was fixed, and the lower end was pre-tensioned under the load equivalent to 40% of the flax fibre's (105 tex – 10.0 N and 68 tex – 5.0 N) and 70% of the cotton fibre's (70 tex) (5.0 N) tensile strength. After that the fibres were impregnated with resin.

Preparation of composites
UP composites were obtained using the silicone sheets system with dumb-bell
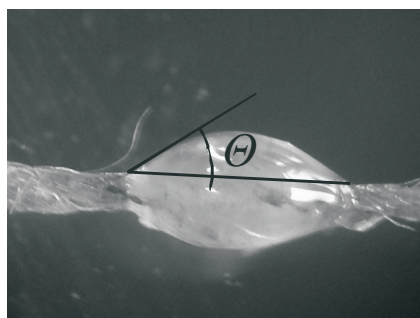
Figure 1. *Scheme of the contact angle measurement*

shaped openings with $55 \times 3 \times (2 \pm 0.1)$ mm operating dimensions of the sample. Continuous single yarn reinforcement was placed lengthwise in the sample mould and filled with polyester resin. The samples were hardened for 24 h at ambient temperature. After this the samples were taken out of the moulds and additionally air hardened for 24 h at ambient temperature.

PLA polymer pellets were melted in a laminating machine - NOVA 45 (Reliant Machinery, United Kingdom) at 190 - 200 °C temperature and sheets were formed at 48.3 kPa pressure for 25 s. Single continuous fibre was placed between the two PLA sheets obtained and laminated by the same method and conditions. Samples were cut into specimens of $100 \times 10 \times (0.8 \pm 0.1)$ mm dimensions.

Characterisations

The drop-on-yarn system was used to determinate the contact angle. Drops of liquid UP resin and viscous PLA polymer melted at 210 °C were placed on the fibre and hardened at room temperature for 24 h (*Figure 1*). For contact angle measurements only symmetrical droplets were chosen, seeking to eliminate the influence of droplet geometry on the contact angle value [18]. Droplets were observed by optical microscope - MBS-9 (magnification 6×) and captured with a CCD camera. Using the software program *Image J,* the contact angle values between the polymer and fibre surfaces were measured from the images obtained. Four specimens for each sample were prepared and averages of test results were calculated.

Flax and cotton fibre surface morphology was investigated using a scanning electron microscope, (SEM) Quanta 200 FEG, FEI Netherlands, at 20 keV (low vacuum). All microscopic images were made under the same technical and technological conditions: electron beam heating voltage – 20.00 kV, beam spot – 5.0, magnifications – 200×, 2000× and 5000×, work distance – 6.0 mm, low vacuum – 80 Pa, detector – LFD.

Tensile tests of yarns were carried out at room temperature using a universal testing machine - Zwick Z005 (Zwick/Roell Group, Germany) with a load cell of 50 N according to the requirements of Standard LST EN ISO 2062. The machine was equipped with special jaws for yarn testing. A cross-head speed of 250 mm/min and gauge length of 500 mm were used. Six specimens were tested for each set of samples and mean values calculated.

Tensile tests of the resins and composite samples were carried out at room temperature using a bench top materials testing machine - Tinius Olsen H25K-T UTM with a load cell of 1 kN (Tinius Olsen Group, Great Britain). A cross-head speed of 5 mm/min and gauge length of 55 mm were used. Six speci-
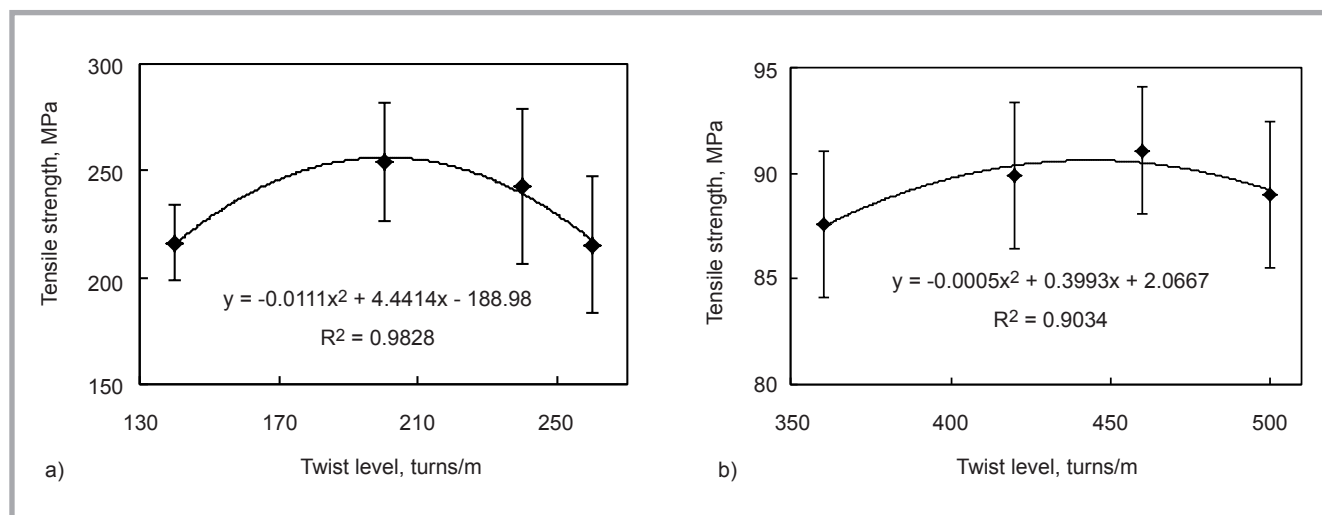
mens were tested for each set of samples and mean values were calculated.

■ Results and discussions

Properties of composite components

The properties at tension of the composite's components are summarised in *Table 2.* As can be seen, strength properties are dependent on the fibre nature – flax fibre has an almost twice higher breaking force and tensile strength compared to those of cotton fibre with similar linear density. Meanwhile the elongation at break of cotton fibre is more than five-times higher compared to that of flax. On the other hand, synthetic E-glass fibre with a linear density of 70 tex is stronger than the natural fibres investigated, even at a higher linear density. However, E-glass fibre shows a significantly lower deformation ability – the elongation at break reaches only 0.9 %.

The change in linear density also influences fibre tensile properties (*Table 2*). An increase in flax fibre linear density from 68 tex to 105 tex increases its strength and deformation by about 20 %. An important control parameter for natural fibre is its twist level. The influence of twisting on flax and cotton fibres' ten-

Table 2. Properties at tension of fibre reinforcements and polymer matrix to be used for composite preparation.

Property	Yarn:				Resin	
	Flax		Cotton 70 tex	Glass 70 tex	UP	PLA
	68 tex	105 tex				
Breaking force, cN/tex	18.5 ± 1.9	22.7 ± 2.3	10.3 ± 0.4	21.0 ± 1.7	–	–
Tensile strength, MPa	175.0 ± 10.5	216.0 ± 11.6	87.6 ± 3.5	245.0 ± 9.6	18.8 ± 2.6	24.1 ± 3.4
Elongation at break, %	2.0 ± 0.3	2.4 ± 0.3	10.6 ± 1.5	0.9 ± 0.1	6.8 ± 1.0	3.1 ± 0.4

$$y = -0.0111x^2 + 4.4414x - 188.98$$
$$R^2 = 0.9828$$

$$y = -0.0005x^2 + 0.3993x + 2.0667$$
$$R^2 = 0.9034$$

Figure 2. Influence of twist level on natural fibre's tensile strength: a – 105 tex flax; b – 70 tex cotton.

Table 3. Wetting of different fibres with UP resin and PLA.

Resin	Yarn	Contact angle, °	
		Untreated fibre	Mercerized fibre
UP	Flax	30 ± 1	20 ± 1
	Cotton	23 ± 1	16 ± 2
	Glass	26 ± 2	–
PLA	Flax	22 ± 2	18 ± 2
	Cotton	16 ± 2	14 ± 2
	Glass	20 ± 1	–

sile strength is shown in *Figure 2.* Initially the twist level increase causes the yarn strength of natural fibre to increase, but further twisting tends to weaken the yarn. Flax fibre yarn with a linear density of 105 tex reaches its maximal tensile strength (255 MPa) at 200 turns/m, while cotton fibre yarn of 70 tex density reaches its maximal strength of 91 MPa at a markedly higher twist level – 460 turns/m. Besides the fibre strength, the amount of twist also affects the impregnation level of fibres with resin in the manufacturing process of composites. At the increased twist level, fibres become more compact and clogged for resin to penetrate into the yarn. Thus the twist level should be kept as low as possible for optimal composite mechanical properties to allow for proper fibre wetting.

Flax and cotton fibres were impregnated with poly(lactic acid) (PLA) and unsaturated polyester (UP) resin. The tensile strength of thermoplastic PLA polymer is about 30% higher than that of thermoset UP (*Table 2*). However, the deformation ability of PLA resin is more than twice lower.

Effect of interaction at the fibre-resin interface on composite properties

The wetting behaviour of fibre in the liquid matrix plays an important role in the fabrication of fibre reinforced polymer composites. The ability of UP and PLA resins to wet twisted natural and synthetic fibres is presented in *Table 3*. Irrespective of the fibre nature, thermoplastic PLA shows better wetting properties – the contact angle is 30 - 45% lower compared to that of thermoset UP resin (16 - 22° for PLA and 23 - 30° for UP resin). In both resin cases the lowest contact angle values are characteristic of cotton fibre, which may depend on the chemical composition of the fibre surface layer investigated. In comparison to cotton, flax fibre walls have a higher amount of hemicelulose, waxes and oils, which, in turn, can affect the polarity of the fibre surface [2].

The adherence mechanism between the fibre and matrix is complex. It is affirmed [18, 19] that interfaces with thermoset resins are governed by chemical bonds, while adherence between fibres and thermoplastic resins proceeds due to weak bonds such as Van der Waals interactions and mechanical anchoring induced by compressive residual stress. Besides this the porous cell wall structure of natural fibres may induce liquid penetration and a higher viscoelastic response to wetting compared to synthetic glass fibre [20].

It may be supposed that the higher ability to wetting of fibres with resin has an influence on higher composite mechanical properties. Therefore fibre modification can be used to enhance the wetting ability. As is clear from *Table 3*, mercerisation decreases the contact angle of natural fibre wetting with PLA polymer by 10 - 20%, and in the case of UP resin – by 30 - 35%. Such an increase in wetting ability can be related not only to changes in flax and cotton surface chemical structure, but also to variations in the fibre's surface morphology. As can be seen from *Figures 3.a* and *3.c*, before treatment the flax and cotton fibre surfaces are covered by non-cellulose plies. Besides this flax fibres contain so-called internodes along their length, running transversally across the fibres. After mercerisation, the surfaces are clean and fibres are aligned (*Figures 3.b* and *3.d*).

Tensile properties of natural fibre reinforced UP and PLA composites are shown in *Figure 4*. It is evident that even single yarn reinforces the polymer matrix. Higher tensile strength is obtained in the case of PLA matrix composites.

Generally the maximal possible strength of the composite is about 50% of the theoretical strength of the reinforcement [21]. Irrespective of the linear density, the tensile strength of flax fibre reinforced UP composites reaches 12% of the fibre strength, whereas for the cotton fibre reinforced UP system it is 30% (see *Table 3*). In the case of flax and cotton fibre reinforced PLA composites, the changes are more noticeable (18% and 31%, respectively). It may be supposed that the reinforcement effect of cotton fibre with lower strength is more efficient due to the higher matrix ability to wet the cotton fibre.

Generally the reinforcing effect of the resin matrix with natural fibre can be
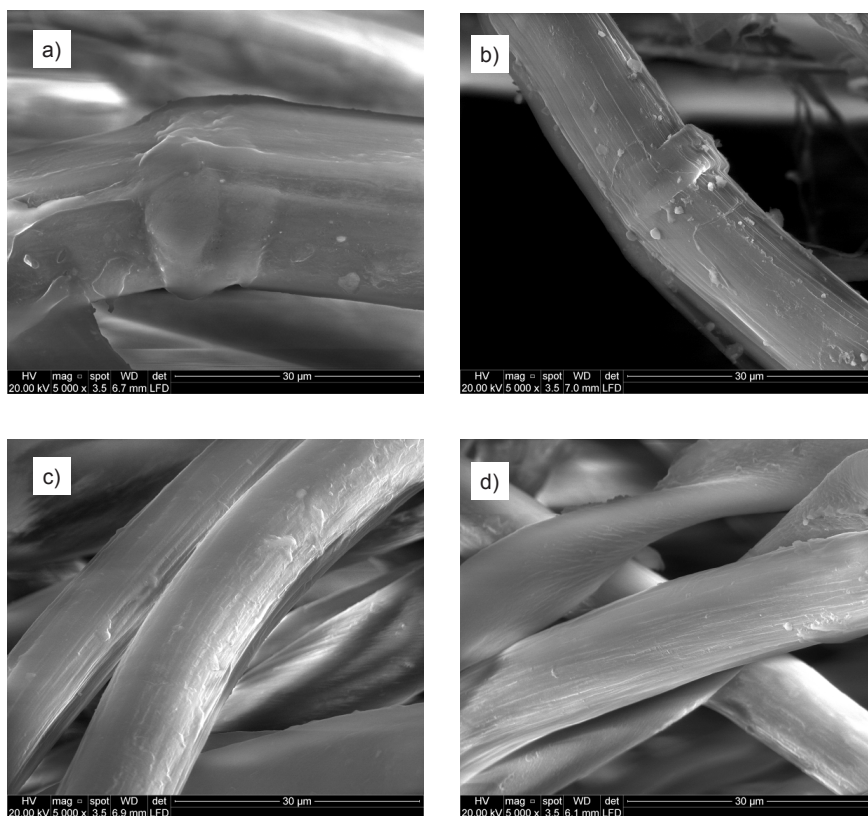
Figure 3. SEM micrographs of flax (a, b) and cotton (c, d) fibres: a, c – untreated; b, d – mercerised.

considered to be low. The tensile strength of UP and PLA polymers reinforced with cotton fibre increases by only 10 - 13%, while when reinforced with flax fiber of similar linear density it is 21 - 31%. A higher degree of tensile strength increase of the polymer matrixes (45 - 66%) is observed in the case of high linear density (105 tex) flax fiber. Meanwhile, the tensile strength of glass fibre reinforced UP and PLA composites reach 29.2 and 47.0 MPa, respectively i.e. the glass fibre increases polymer matrix strength *ca*. 1.6 - 2.0 times (55 - 95%).

On the other hand, an increase in the strength of natural fibre reinforced polymer matrix composites can be achieved by improving adhesion interaction at the fibre-resin interface through chemical or/and mechanical treatment of the fibre surface. Comparison of the effect of various fibre treatment methods on the tensile properties of single natural fibre yarn reinforced UP and PLA composites is presented in *Figure 4*. As can be seen, the bleaching of flax has only a negligible influence on the UP and PLA composite's properties: the tensile strength of the bleached flax/UP composite is only 7% higher than that in the case of untreated flax fibre, while the bleached flax/PLA composite's strength even decreases (from 39.9 MPa down to 34.7 MPa). Meanwhile flax and cotton fibre mercerisation leads to a conspicuous increase in the composites' tensile properties, especially in the case of the PLA matrix (ca. 35 - 50%) (*Figure 4.b*). However, the complex chemical treatment – mercerisation of fully bleached flax fibre (105 tex) – again decreases the composite strength to the level of the bleached flax/PLA composite. Such bleaching influence on the composite strength can be related to the strong effect of the chemical treatment, which removes too much hemicellulose and the fibres could be shifted to a more vulnerable state [7]. On the other hand, it can be related to the high laminating temperature of PLA composites (190 - 200 °C), because cellulose fibres are not sufficiently thermostable. Meantime the tensile strength of mercerised-bleached flax/UP composites obtained at ambient temperature is at the same level as that of mercerised flax/UP (*Figure 4.a*).

Mechanical treatment – twisting, pre-tension or a combination of these methods – reinforces all the composites investigated (*Figure 4*). The twisting of yarns shows a higher influence on the strength of cotton
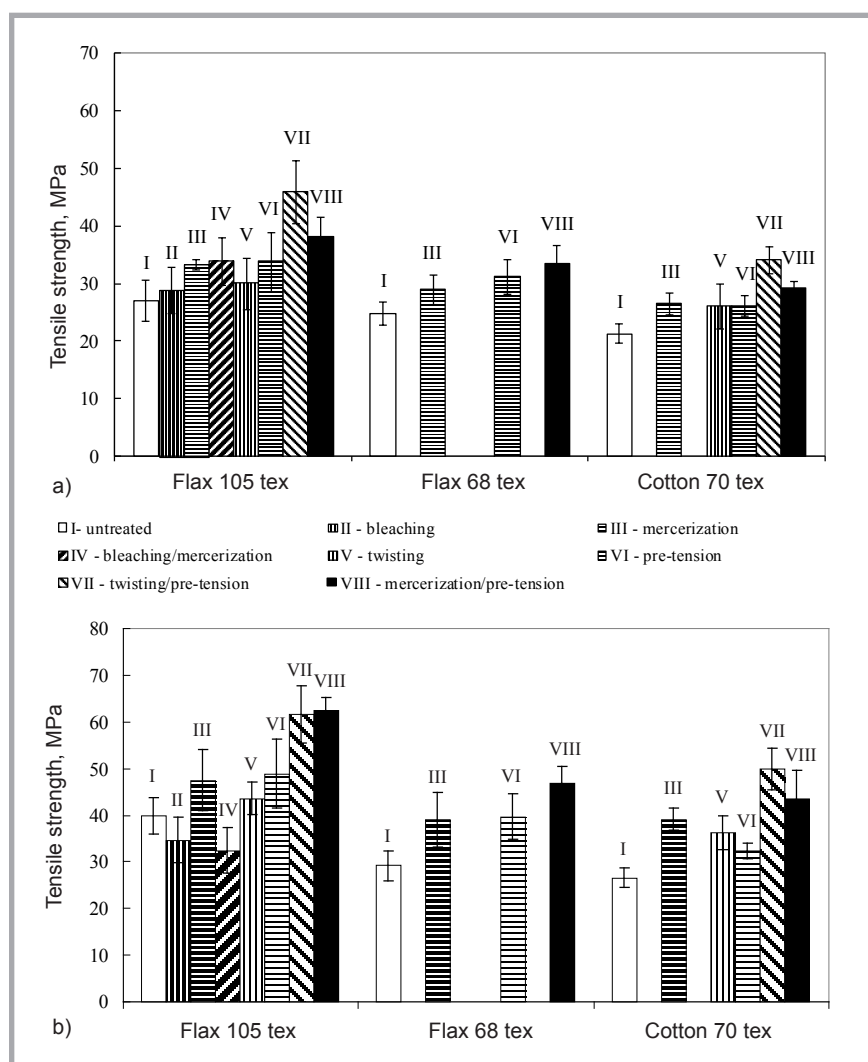
Figure 4. *Influence of fibre treatment methods on the tensile strength of UP (a) and PLA (b) composites.*

fibre reinforced composites. In the case of cotton reinforced UP and PLA composites, their tensile strength increases by 23% and 37%, respectively, whereas for the flax fibre reinforced composites, only a 10% strength increase is observed.

It should be mentioned that the fibre twist level at the maximal tensile strength was chosen for the adhesion interaction investigations (200 turns/m – for flax, and 460 turns/m – for cotton) (see *Figure 2)*. The higher effect of cotton on the composite strength can be explained through the different cross-section shapes of flax and cotton fibres. There are more individual cotton fibres in the yarn than in the case of flax. The number of fibres in the cross-section of yarn has a determinant effect on its quality and on the load transfer among fibres [22].

The pre-tension of fibre also has an influence on the composite strength increase. Such mechanical treatment in-

creases flax/resin composite strength by a higher degree than twisting; especially flax fibres of low linear density (68 tex) are sensitive to pre-tension. A greater effect on the composite strengthening is obtained when twisted flax and cotton fibres are pre-tensioned. Such a combination of mechanical treatment methods allows to obtain 1.6 - 1.9 times stronger UP and PLA composites compared to those obtained with untreated fibres. Thus the pre-tension of twisted natural reinforcements allows to obtain UP and PLA biocomposites with a strength close to that of glass fibre reinforced composites (29.2 and 47.0 MPa, respectively).

The effect of mechanical pre-tension on the macro- and microstructure of flax and cotton fibres can be seen in *Figure 5* (see page 128). Pre-tensioned flax and cotton fibres are orientated more parallel to the yarn axis compared to unstreched ones (*Figure 5.a, 5.b, 5.d & 5.e*). The fibres
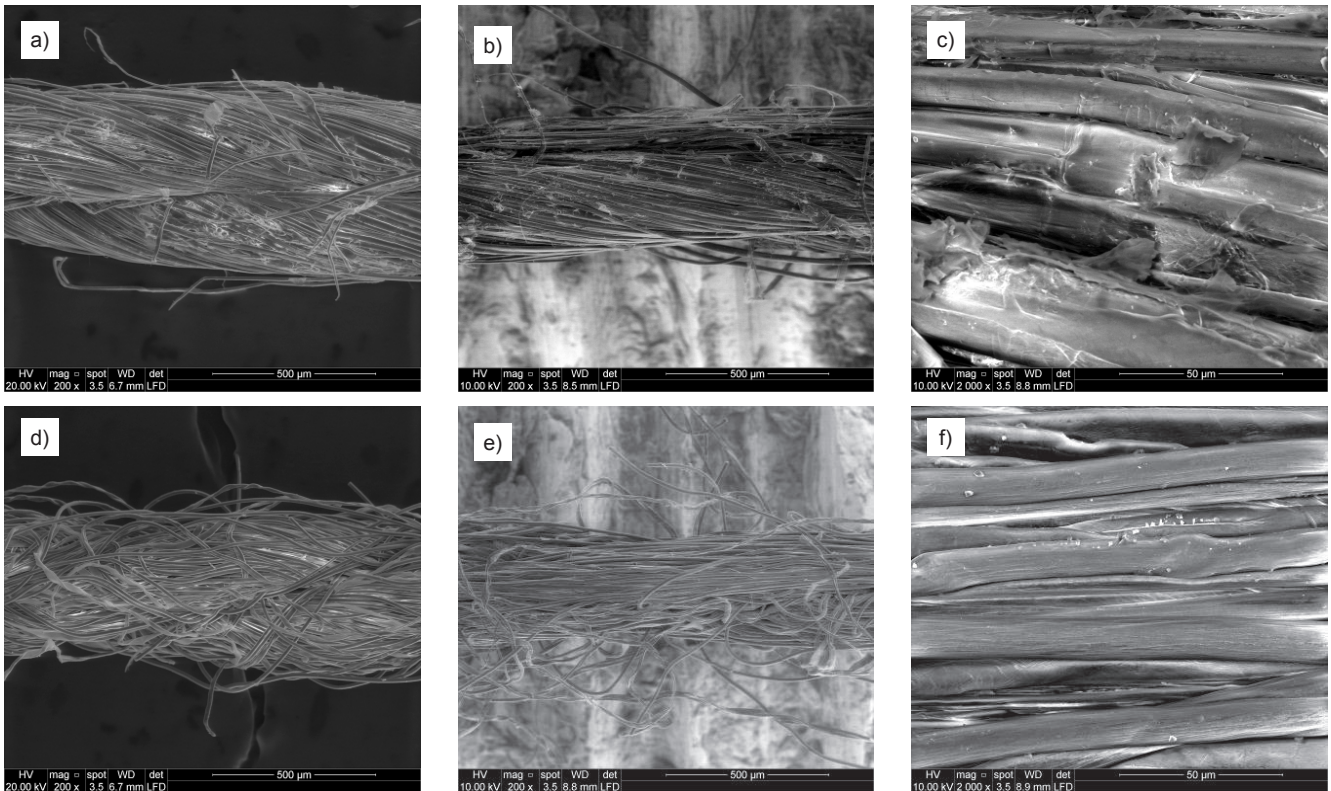
Figure 5. *SEM micrographs of flax (a – c) and cotton (d – f) fibres: a, d – unstretched; b, e – pre-tensioned (magnification 200×); c, f – pre-tensioned (magnification 2 000×).*

in pre-tensioned yarns are placed closely (***Figure 5.c, 5.f***), therefore such yarns are more rigid, inflexible and have higher tensile strength.

A similar influence on the tensile properties of the composites is shown by the pre-tension of mercerised flax or cotton fibres. The enhanced load transfer efficiency of pre-tensioned mercerised 105 tex flax fibres can be seen in ***Figure 6***. As is common, the highest tensile strength is shown by mercerised flax

fibre yarn (252.6 MPa). However, the mechanical strength of the single yarn reinforced composite is mainly determined by the polymeric matrix, i.e. PLA (24.1 MPa). As can be seen, flax fibre increases the strength of the PLA matrix, but its value depends on the fibre treatment method. Whereas untreated flax brings about a 66% matrix strength increase, fibre mercerisation increases the matrix strength nearly twice, while the additional use of pre-tension leads

to a three-times higher increase in strength.

Thus the combination of vegetable fibres mechanical and chemical treatment methods creates possibilities to obtain fibre reinforced polymer biocomposites with similar mechanical properties to those of synthetic composites.

■ Conclusions

The chemical and/or mechanical pre-treatment of vegetable fibres has an influence on adhesion interaction in unsaturated polyester or poly(lactic acid) matrix biocomposites. Single flax or cotton fibre yarn reinforced PLA matrix is characterised by a higher strength than that of the UP. Regardless of lower strength, the reinforcement effect of the cotton fibre is more efficient than that of flax due to the higher wetting ability.

The mercerisation of flax and cotton fibres increases the composite strength, while bleaching shows a negligible or even negative effect on the composite properties. Twisting and pre-tension of fibres increase their rigidity and the packing level in the yarn, resulting in improved tensile properties of composites. The pre-tensioning of twisted fibres leads to markedly higher polymer matrix
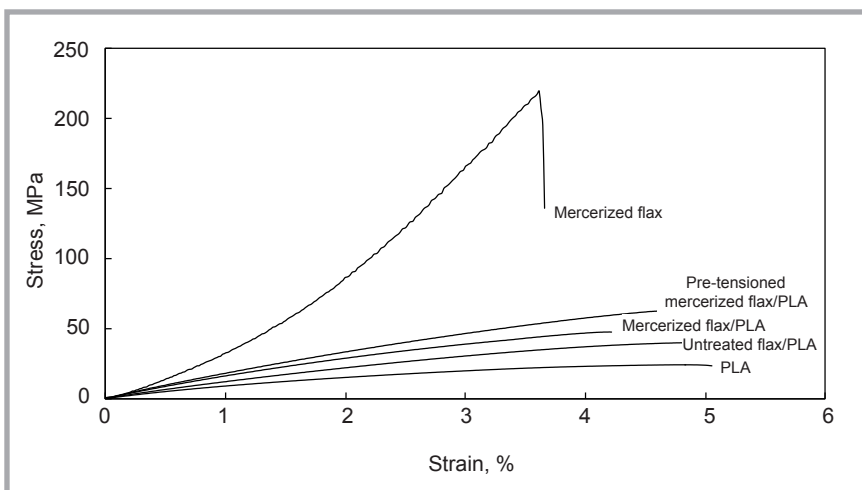
Figure 6. *Stress-stain curves of flax fibre, PLA matrix and flax fibre reinforced PLA composites.*

strengthening, permitting to obtain bio-composites with mechanical properties comparable to those of glass fibre reinforced polymer matrix composites.

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- Polymers and the environment
  - Biodegradable polymers
  - Polymeric biomaterials
  - Polymers and resins in water systems
  - Recycling of polymer materials

Invited Lectures (selected)
- *Andrzej K. Błędzki* 'Cellulose fibres substitude of glassfibres in biocomposites'
- *Danuta Ciechańska* 'Biomass as a source of functional polymeric materials'
- *Zbigniew Floriańczyk* ' Polimeric materials on the basis of unorganic-organic polymers'
- *Andrzej Gałęski* 'Composites and nanocomposites on the basis of polilactide'
- *Marek Kowalczuk* 'Synthesis and properties of biodegradable poli(ester-urethanes) and their application'

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