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Application of lignin as natural adhesion promoter in cotton fibre-reinforced poly(lactic acid) (PLA) composites

Nina Graupner

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Abstract This study investigated how lignin—used as a natural adhesion promoter in biodegradable, thermoplastic cotton fibre-reinforced composites—influences the composites' mechanical properties. Composites with fibre mass proportions of 40% were produced by compression moulding. Poly(lactic acid) (PLA), a biopolymer, served as matrix. Cotton/PLA composites with and without lignin content were manufactured. As reference samples of bast fibre-reinforced composites, kenaf/PLA composites were produced under the same conditions. The composites were tested for stiffness, tensile strength, elongation at break and impact strength. Fractured surfaces were analysed using scanning electron microscopy (SEM). The results of the composite investigations showed that the addition of lignin has an influence on the cotton/PLA composite characteristics. SEM investigations showed that the adhesion between fibre and matrix could be improved by the addition of lignin. Tensile characteristics like tensile strength and Young's modulus could be improved clearly, while the impact properties were decreased.

Introduction

Today, in the time of climatic change, natural fibre-reinforced plastics are an interesting alternative to composites based on glass and petrochemical fibres. Due to their good lightweight construction potential and good crash characteristics, they are used more and more as reinforcement for construction materials. Nevertheless, application is often limited to non-visible components in the automobile industry [1] or to products for which there is little demand like salvers or briefcases. In order to conquer further areas of application and to satisfy the increasing demand for natural composites, new high-quality materials need to be designed, and further material combinations need to be tested.

The strength of the bond of fibre and matrix is substantial for the mechanical properties of a composite. Fibre/matrix adhesion is a complex process with many factors interacting with each other. It can be affected by the choice of materials, manufacturing methods, processing parameters, surface treatments of the fibres or by additives like adhesive agents. Thus, several studies dealing with the optimisation of fibre/matrix adhesion in natural fibre-reinforced composites have already been conducted.

Bhat et al. examined the adhesion between fibres and different thermoplastic matrices. For this purpose, they produced cotton composites with matrices in the form of foils or fibres. Then they examined the composites' mechanical properties; the best fibre/matrix adhesion and the highest composite strength could be achieved by using a matrix in the form of fibres. In addition, the processing parameters during production have a decisive influence on the composite properties [2]. For example, Müller et al. examined the influence of the processing temperature on the strength of natural fibre-reinforced thermoplastics. The result is a temperature curve which indicates that a too low press temperature (too high viscosity of the matrix) as well as a too high press temperature (start of fibre degradation) leads to a decreased composite strength [3]. Likewise, pre-treating the fibres with different chemicals has a strong influence on

N. Graupner
Faserinstitut Bremen e.V. (FIBRE),
Am Biologischen Garten 2, 28359 Bremen, Germany

Present Address:
N. Graupner (✉)
University of Applied Sciences Bremen,
Faculty 5 – Biomimetics/Biological Materials,
Neustadtswall 30, 28199 Bremen, Germany
e-mail: nina.graupner@hs-bremen.de

the composite properties. Mehta et al. analysed the consequences of different surface treatments like alkalis, silanes and acrylonitriles. The test was carried out with hemp needle felt/unsaturated polyester resin (UP) composites. It is quite evident that mechanical and thermal composite properties could be increased tremendously by these surface treatments [4]. Joffe et al. examined fibre/matrix adhesion with the fragmentation test. Their results also show that fibre/matrix bonding can be increased by fibre treatment [5]. Endres et al. investigated whether maleic anhydride-grafted (MAH-g) coupling agents improve the mechanical properties of injection-moulded natural fibre-reinforced thermoplastics. They found out that the mechanical properties of these composites could be improved by up to 40% with an optimised coupling agent content as well as with an optimal MAH-content proportional to the fibre surface [6].

Beside the fragmentation test, microscopic examination can be used for evaluating fibre/matrix adhesion. Michaeli et al. tested different microscopic examination methods—i.e. scanning electron microscopy (SEM), transmission electron microscopy, energy-dispersive X-ray analysis and atomic force microscopy—to evaluate the bonding surface in flax fibre-reinforced thermosets [7].

In own investigations, compression-moulded natural fibre-reinforced thermoplastic PLA composites were manufactured. As reinforcement, we used bast fibres such as hemp, kenaf and seed hair like cotton. In the bast fibre-reinforced composites, we observed a good allocation of fibre and matrix. The matrix melted evenly and the fractured surface showed no delaminations. Nevertheless, in the cotton fibre-reinforced composites the matrix melted unevenly, and the bonding of fibre and matrix, i.e. of the single webs in the multilayer web, was weak.

While bast fibres like hemp or kenaf contain a portion of lignin, cotton fibres lack this component. We assumed that lignin strengthens the bond of fibre and matrix. In the context of this work, we examined to what extent adding powdery lignin changes the mechanical properties of cotton fibre-reinforced PLA. As reference, we also examined kenaf/PLA composites. All results were compared to a pure PLA matrix sample.

Experimental procedure

Fibres and polymers

For the composite production, cotton fibres (*Gossypium hirsutum L.*) from the provenience CIS-ELS/Uzbekistan with a density of 1.56 g/cm^3 were used. The fibres were supplied by Alpearse (Bremen, Germany).

For the bast fibre-reinforced reference sample, chopped yield-retted kenaf fibres (*Hibiscus cannbinus L.*) were supplied by Holstein Flachs (Mielsdorf, Germany) in September 2004.

As matrix, the biopolymer poly(lactic acid) (PLA) was chosen. It contains a NatureWorks^{TM} PLA polymer 6202D supplied by Cargill Dow LLC (Minnetonka, Minnesota, USA). This biopolymer has a density of 1.24 g/cm^3, a melting temperature of 160–170 °C and a glass transition temperature of 60–65 °C. It was supplied in fibre form.

For the additional lignin treatment of some of the cotton/PLA webs, lignin powder was used. Lignin of the type Hardwood kraft eucalyptus with a melting point of approximately 122 °C is derived from the plant *Eucalyptus globulus*. It was supplied by the Federal Research Centre for Forestry and Forest Products (BFH) in Hamburg (Germany).

Fibre characteristics

Prior to the composite production, fibres were tested for their single element strength, collective strength, elongation at break, Young's modulus and fibre width. These investigations took place in standard climate at 20 °C and 65% relative humidity according to DIN EN ISO 139.

Tensile test of fibres

Collective strength of fibre collectives by using a Stelometer The tensile strength was measured on fibre collectives by a Stelometer (Spinlab, Knoxville, Tennessee, USA) according to ISO 3060. The oriented fibre collective was clamped over 15 mm width in Pressley clamps using a spacer of 3.2 mm. The clamps were covered with leather for testing the cotton fibres and with PVC foil (150 μm for the top part, 300 μm for the bottom part) for testing the kenaf fibres. Ten collectives of cotton fibres and 20 collectives of kenaf fibres were tested to obtain a representative set of results. The strength of fibre collectives in cN/tex can be calculated from the mass of the tested collective in kilogram divided by the mass-related fineness of the tested collective in tex. The mass of the collective was measured with an accuracy of 0.01 mg.

Single element test by using Dia-Stron A testing instrument from Dia-Stron Ltd. (Andover, UK) was used to measure the force-elongation characteristics of the fibres. The individual elements are glued to reduce the influence of clamping and tested at a gauge length of 3.2 mm. The cross-sectional area of each element is measured by means of a laser beam. The sample is then automatically transferred to the tensile testing system. After the tensile test, the sample containers are removed and the next samples are tested automatically. Ninety individual elements are prepared in advance and are inserted into the system automatically by the auto sampler. The software determines the strength of

surfaces in N/mm^2. In consideration of the density, fineness-related values in cN/tex can be calculated. The analysis programme allows the determination of the true zero point, elongation at break, Young's modulus and breaking energy.

Determination of fibre fineness by using Fibreshape

For the investigation of the fibre width with the Fibreshape measuring system, the fibres were distributed on a slide frame of 40 × 40 mm and a glass width of 2 mm (Gepe, Zug, Switzerland). Four slide frames were prepared per sample and scanned with a Dimage Scan Multi Pro (Konica Minolta Photo Imaging Europe GmbH, Unterfoehring, Germany) at a resolution of 4,800 dpi. The images were analysed with the image analysis software Fibreshape 4.35 (IST AG, Vilters, Switzerland). The evaluation was done with a calibrated measuring mask (ALFM 48 Z K 5 D00).

Fibre processing and composite production

Composites with a fibre mass content of 40% were manufactured. The mass portions refer to the dry weight. Fibre and matrix were mixed during carding.

Cotton/PLA and kenaf/PLA composite mixtures were produced. As reference, webs of pure PLA were made. The fibres were oriented into a multilayer web by using a lab roller card (Anton Guillot, Aachen, Germany) with a working width of 30 cm. The fibre orientation was predominantly in length direction, parallel to the machine direction. To avoid damage of fibres due to carding, the fibres were conditioned for at least 24 h at 20 °C and 65% relative humidity.

After carding, the webs were cut into parts of 323 × 223 mm. The initial weighing of the pre-cut parts took place in consideration of the density of the tested fibres in relation to the volume of the fabricated boards. Cotton/PLA boards with and without lignin were produced. For the boards with lignin content, 5 g powdery lignin was filled equal into the webs by a sieve.

The dimensions of the boards are 323 × 223 × 2 mm. As a spacer an aluminium frame with external dimensions of 343 mm × 243 mm was used. For easier removal of the composite panel from the mould, the frame was treated with a parting compound from the company Marbocote Ltd. (Cheshire, UK). Prior compression moulding, the multilayer webs were dried for 3 h at 105 °C in a forced-air-oven (Memmert, Germany). After drying, the multilayer web was centred on the bottom pressure plate in the aluminium frame. Compression moulding was done with a hydraulic press type KV 214.01 (Rucks Maschinenbau GmbH, Glauchau, Germany) capable of applying pressure and controlled heating to both upper and lower plates. The press has a nominal pressure force of 1,000 kN, a maximum temperature of 330 °C and a pressure table of 600 × 600 mm^2. The pressure was set to 4.2 MPa and was maintained for 20 min at 180 °C. Before demoulding, the boards were cooled down to approximately 60 °C.

After that, the composites were cut into specimens with a moulding cutter for the different mechanical testing experiments.

Composite characteristics

The composites were examined for their tensile and impact properties. The pure PLA, without fibre-reinforcement, acted as the reference for the experiments.

Before testing, the samples were conditioned for at least 24 h at 23 °C and 50% relative humidity according to DIN EN ISO 291.

Tensile test

Tensile strength, elongation at break and Young's modulus were determined according to DIN EN 61. For this experiment, the composites were cut into waisted specimens (type I, DIN EN 61).

The tensile specimens were tested in a Zwick Z250/SNA universal testing machine (Zwick/Roell, Germany) with a displacement speed of 2 mm/min. The gauge length was 100 mm. Respectively, at least five specimens were tested at 0° machine direction of the roller card. The mechanical properties determined were maximum strength, strength at break, deformation at maximum load, deformation at break and Young's modulus. The computed results correspond to average values of samples with the measured specimens.

Charpy impact test

For the Charpy impact strength rectangular, unnotched specimens of 80 × 10 × 2 mm were manufactured and tested according to DIN EN ISO 179. Impact strength was measured on a Thwing-Albert Frank (Uffenheim, Germany) testing machine type 53302 operating with a pendulum length of 225 mm and a pendulum size of 1 J. For each testing procedure, at least five specimens at 0° machine direction were tested. All results were taken as the average reading of the measured samples.

Scanning electron microscope analysis (SEM)

Morphological examinations were carried out using a CamScan CS 24, a SEM manufactured by Obducat CamScan Ltd. (Cambridgeshire, UK). All specimens were sputtered with a layer of gold–palladium prior to SEM observations and mounted on aluminium holders using double-sided electrically conducting carbon adhesive tabs.

The SEM investigation was used to study the fracture surface of the tensile specimens of the composite samples.

Results

Fibre characteristics

Different methods to examine the fibre characteristics were applied. An overview is given in Fig. 1. The precise values can be taken from Table 1.

The fibre width was measured with the Fibreshape system. With widths of 15.6 μm, cotton fibres were almost three times finer than kenaf fibres (43.3 μm).

Tensile strength was determined on the one hand as collective strength measured by Stelometer and on the other hand as single element strength measured by using Dia-Stron. The cotton fibres' collective strength (15.1 cN/tex) was less than half of their single element strength (36.9 cN/tex). Kenaf fibres had a collective strength of 27.8 cN/tex and a single element strength of 41.6 cN/tex. The collective strength is always lower than the single element strength.

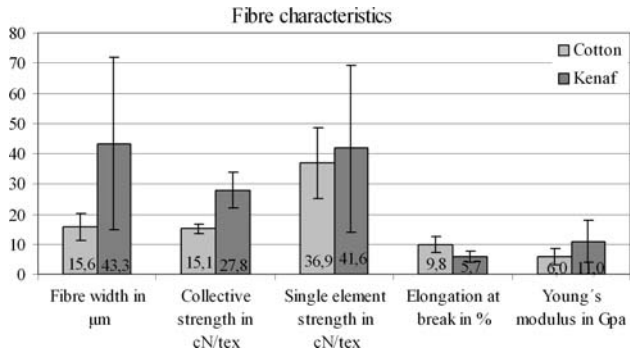

Fig. 1 Comparison of cotton and kenaf fibre characteristics measured by different methods

Young's modulus and elongation at break were also determined by using Dia-Stron. Cotton fibres with a Young's modulus of 5,979 MPa are less stiff than kenaf fibres with a value of 10,995 MPa. The elongation at break of cotton fibres (9.8%) is clearly higher than that of kenaf fibres (5.7%).

The measured fibre characteristics lead to the assumption that tensile strength and stiffness of cotton/PLA composites are much lower than that of kenaf/PLA composites. Because of their high elongation at break, they must have considerably higher impact strength than kenaf/PLA composites.

Composite characteristics

The mechanical characteristics of cotton/PLA composites with and without lignin, kenaf/PLA composites and the pure PLA sample were measured with the tensile test according to DIN EN 61 and the Charpy impact test according to DIN EN ISO 179. An overview of the measured composite characteristics is given in Table 2.

Optical examinations were carried out using a SEM.

Tensile properties

An overview of the tensile strength of the composites compared to the values of the pure PLA matrix is given in Fig. 2. As supposed, the highest value (53 MPa) could be measured in kenaf/PLA composites due the high fibre strength of kenaf. The lowest value (30 MPa) was measured in the pure PLA sample.

Cotton/PLA composites showed a tensile strength of 41 MPa. Compared to the pure PLA matrix, the tensile strength could be increased considerably. By treating the multilayer webs with lignin, the tensile strength could be increased to 45 MPa. This is a 9% increase.

Table 1 Overview about the measured fibre characteristics

Fibre characteristics	Indicated value	
	Cotton	Kenaf
Single element strength by Dia-Stron, cN/tex	36.92 (±11.70)	41.57 (±27.71)
Young's modulus by Dia-Stron, MPa	5978.86 (±2622.16)	10994.73 (±6781.36)
Elongation at break by Dia-Stron, %	9.80 (±2.66)	5.68 (±1.77)
Collective strength by Stelometer, cN/tex	15.14 (±1.44)	27.76 (±5.88)
Fibre width by Fibreshape, μm	15.60 (±4.50)	43.30 (±28.6)

Table 2 Mechanical cotton/PLA and kenaf/PLA composite characteristics, referred to the pure PLA

Sample	φ_F, %	Tensile strength, MPa	Young's modulus, MPa	Elongation at break, %	Charpy impact strength, kJ/m^2
Cotton/PLA	40	41.20 (±2.04)	4242.3 (±634.98)	2.95 (±0.47)	28.71 (±4.39)
Cotton/PLA + lignin	40	45.44 (±0.58)	5234.27 (±168.51)	2.36 (±0.24)	23.90 (±2.42)
Kenaf/PLA	40	52.88 (±4.45)	7138.57 (±1329.89)	1.05 (±0.10)	8.97 (±2.67)
Pure PLA	0	30.08 (±8.00)	3820.18 (±166.33)	0.83 (±0.26)	22.42 (±4.11)

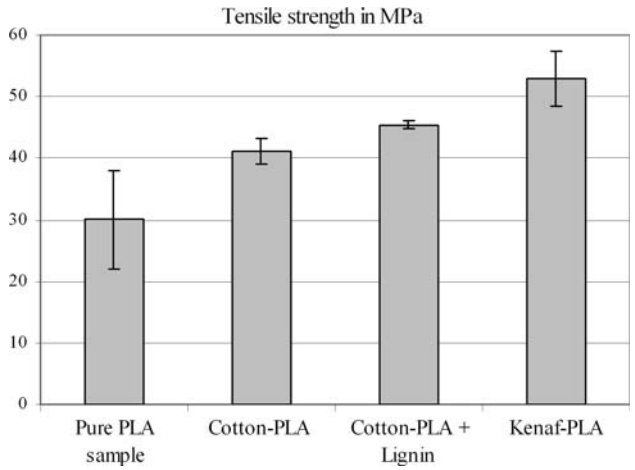

Fig. 2 Tensile strength of the composites compared to the pure PLA sample

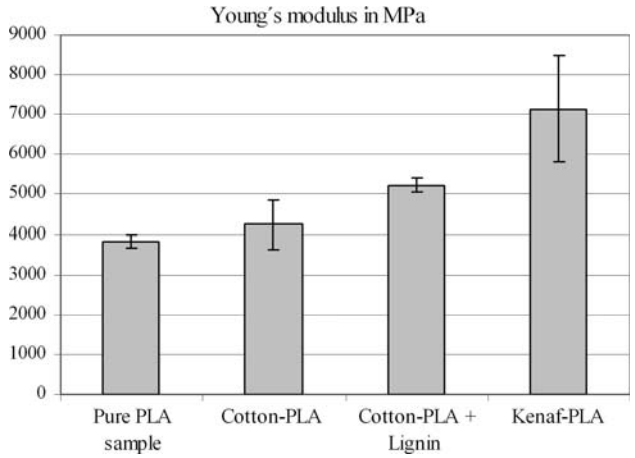

Fig. 3 Young's modulus of the composites compared to the pure PLA sample

The Young's moduli of the different composites are shown in Fig. 3 next to the values of the pure PLA matrix. Again, the highest values (7,139 MPa) were measured in bast fibre-reinforced kenaf/PLA composites. The pure PLA matrix possessed the lowest stiffness (3,820 MPa). A reinforcement effect concerning stiffness could be achieved by using untreated cotton. The measured Young's modulus was 4,242 MPa. By treating the cotton multilayer webs with lignin, an increase of the Young's modulus to 5,234 MPa could be achieved. This is a 19% increase.

Impact properties

Another tendency could be determined examining the Charpy impact strength. Figure 4 compares the impact strength values of the different composites to the values of the pure PLA matrix.

The pure PLA matrix had an impact strength of 22 kJ/m^2. With values of 9 kJ/m^2, the bast fibre-reinforced kenaf/PLA composites had by far the worst values. Compared to the pure matrix, no reinforcement effect concerning impact strength could be achieved by using kenaf fibres.

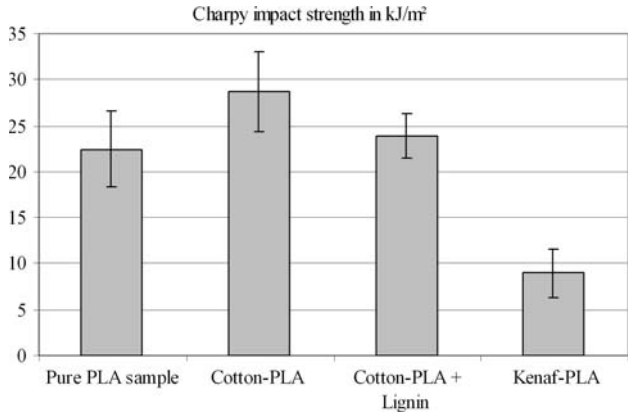

Fig. 4 Charpy impact strength of the composites compared to the pure PLA sample

The best value (29 kJ/m^2) was measured in cotton/PLA composites, followed by cotton/PLA treated with lignin (24 kJ/m^2). Thus, the impact strength was decreased by the treatment with lignin by approximately 17%. This result was also reflected by the elongation at break (see Table 2). The following values were measured: cotton/PLA = 2.95%, cotton/PLA + lignin = 2.36% and kenaf/PLA = 1.05%.

SEM analysis

The microscopic examination of the fractured surfaces of the tensile specimens of the composites was carried out by using SEM analysis.

As for the fractured surface of cotton/PLA, it was already macroscopically recognisable that the individual layers of the multilayer webs did not interconnect optimally (Fig. 5). Delaminations were obvious, even more during the SEM analysis (Fig. 6). Delaminations are marked by darts. The fractured surface of the cotton/PLA composites treated with lignin showed fewer delaminations (Fig. 7). This indicates that lignin improved the connection of the individual webs in the multilayer web. In kenaf/PLA

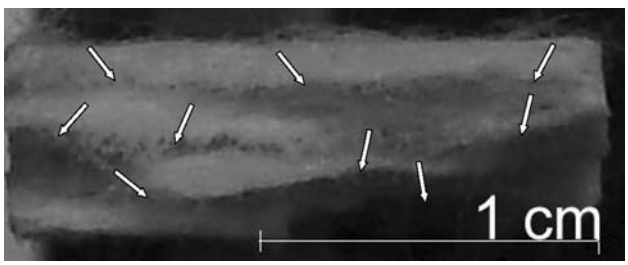

Fig. 5 Delaminations of a fractured surface of a cotton–PLA composite (delaminations are marked by darts)

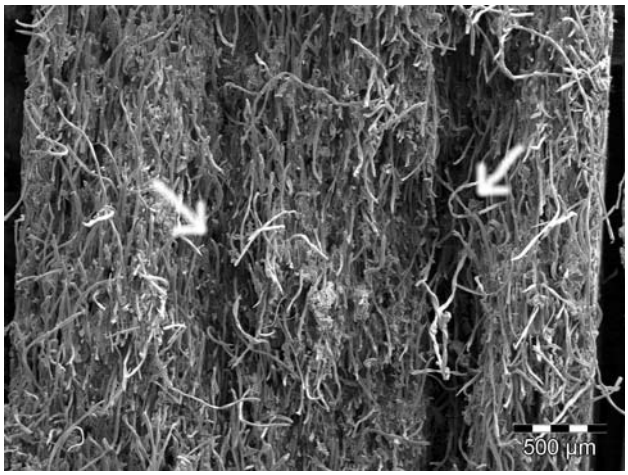

Fig. 6 SEM photograph of a fractured surface of a cotton/PLA composite (overview)

Fig. 7 SEM photograph of a fractured surface of a cotton/PLA + lignin composite (overview)

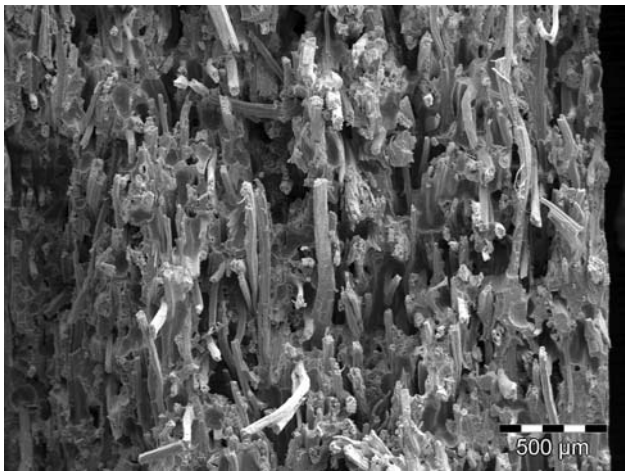

Fig. 8 SEM photograph of a fractured surface of a kenaf/PLA composite (overview)

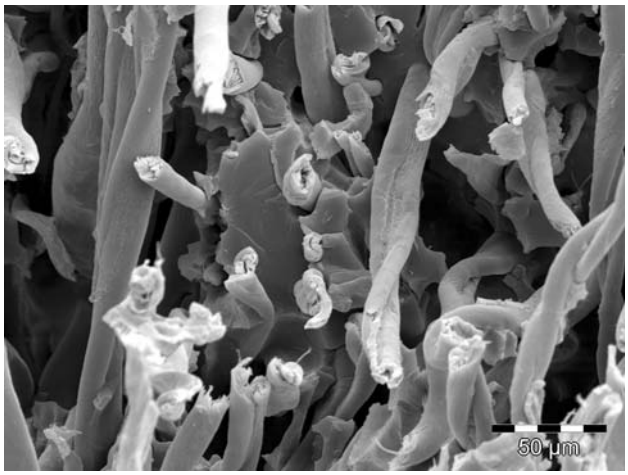

Fig. 9 SEM photograph of a fractured surface of a cotton/PLA composite

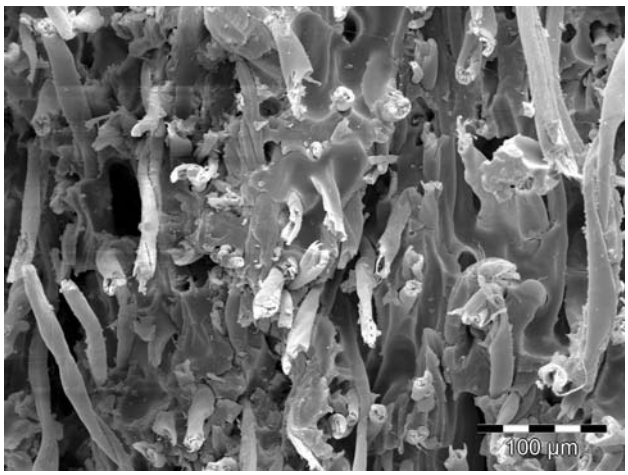

Fig. 10 SEM photograph of a fractured surface of a cotton/PLA + lignin composite

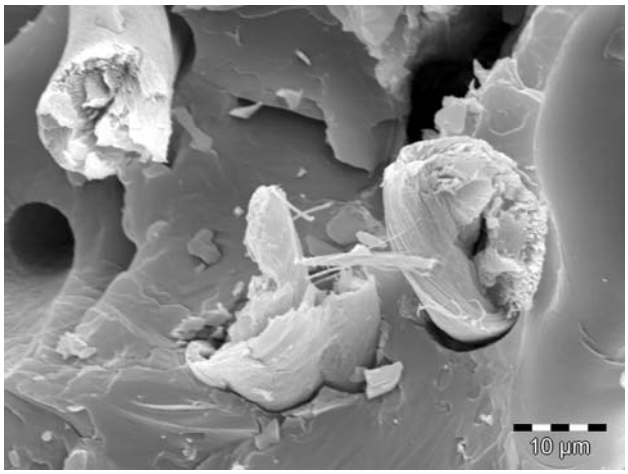

Fig. 11 Detailed SEM photograph of a fractured surface of a cotton/PLA + lignin composite

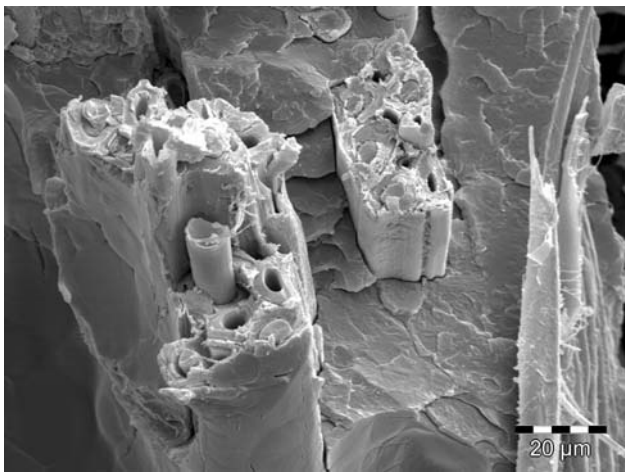

Fig. 12 Detailed SEM photograph of a fractured surface of a kenaf/PLA composite

composites, no delaminations were recognisable (Fig. 8). Closer examinations showed that fibre pull-outs in the untreated cotton/PLA composites (Fig. 9) were more severe than in the lignin-treated PLA composites (Figs. 10 and 11). The fractured surface of the kenaf/PLA composite is shown in Fig. 12.

Discussion

The assumption that lignin improves the fibre/matrix adhesion is supported by the described test results.

Cotton does not naturally contain lignin, whereas kenaf contains 12–14% [8].

The SEM pictures show no delaminations of the single fibre webs in the fractured surface of kenaf/PLA composites (Fig. 8), while cotton/PLA composites clearly delaminate (Fig. 6). By adding lignin to cotton/PLA composites, a better connection between fibre and matrix and between the single fibre layers of the multilayer web could be achieved, resulting in reduced delaminations (Fig. 7). This is reflected by the mechanical characteristics: compared to pure cotton/PLA composites, adding lignin increased the tensile strength by approximately 9%, the Young's modulus by approximately 19% (Figs. 2 and 3).

Impact strength and elongation at break are directly correlated. The impact strength is usually increased by a higher elongation at break. Fibres containing much cellulose mostly have high elongation at break values. Cotton has a cellulose content of 88–96%; kenaf only contains 58–63% [8]. Elongation at break and impact strength are directly correlated. This can be seen in Tables 1 and 2. The high elongation at break of raw cotton fibres of 9.8% increased the elongation at break in the composites to 2.95%. Raw kenaf fibres have an elongation at break of 5.7%. Kenaf/PLA composites showed a value of 1.05%. The bad impact strength of kenaf/PLA composites (9 kJ/m^2) could be traced back partly to the low elongation at break. Despite a low elongation at break (0.83%), the pure PLA matrix with an impact strength of 22 kJ/m^2 was more than twice as strong as kenaf/PLA composites (Fig. 4). Theoretically, the impact strength values of kenaf/PLA should have been higher due to its higher elongation at break compared to the pure matrix. Presumably, the presence of lignin causes embrittlement of the composites. Müssig and Bax [9] and Oksman et al. [10] reported similar results in their works. They examined PLA composites reinforced with bast and synthetic cellulose fibres (100% cellulose): Compared to the pure PLA matrix, the impact strength of bast fibre-reinforced composites was clearly reduced, whereas the impact strength of man-made cellulose fibre-reinforced composites was very high.

While tensile properties like tensile strength and Young's modulus were clearly improved by adding lignin to cotton/PLA composites, the impact strength was reduced by approximately 17% compared to pure cotton/PLA composites. Elongation at break also decreased from 2.95% to 2.36%. This supports the assumption that the presence of lignin improves adhesion between fibre and matrix as well as the bonding between single layers in the multilayer web. However, at the same time the impact strength decreases because of the embrittlement of the composites.

Conclusions

The objective of the work was to investigate the effects of lignin in cotton/PLA composites, its influence on mechanical properties and its potential as a natural adhesion promoter in natural fibre-reinforced composites. The assumption was that lignin improves the fibre matrix adhesion in cotton fibre-reinforced PLA composites. For this purpose, cotton was well suitable because the fibres do not contain any lignin. Thus, cotton/PLA fibre webs with a cotton mass proportion of 40% were manufactured by carding. Some of the fibre multilayer webs were treated with powdery lignin. As reference samples, bast fibre-reinforced kenaf/PLA composites were produced. Kenaf contains natural lignin. The fibre mass content was also 40%. The boards were manufactured by compression moulding. Pure PLA boards without reinforcement acted as further reference samples.

The composites and the pure PLA sample were examined in terms of their tensile characteristics (tensile strength, Young's modulus and elongation at break) as well as their impact characteristics by the Charpy impact test. Optical investigations of the fibre/matrix interface were carried out by SEM.

The results of the SEM analysis showed that adhesion between fibre and matrix as well as between individual layers of the multilayer webs could be improved by the presence of lignin. While the untreated cotton/PLA composites showed clear delaminations of the individual layers of the multilayer web, less delaminations were found in the lignin-treated cotton/PLA. No delaminations were found in the kenaf/PLA composites.

Compared to the untreated cotton/PLA composites, lignin treatment increased tensile strength and Young's modulus by 9% and 19%. On the other hand, the impact strength was decreased by 17%. However, it was still higher than that of the pure matrix. In contrast, kenaf/PLA composites had barely half the impact strength of the pure matrix.

The measured results prove the assumption that the presence of lignin improves fibre/matrix adhesion. However, the impact strength is reduced by the embrittlement of the composites.

Thus, after further optimisations, lignin could be used in natural adhesion promoters for lignin-free fibres in a PLA matrix if an increase of tensile strength and stiffness is required. The moisture and odour development during processing needs to be kept under control.

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