INFLUENCE OF FIBRE LOADING, FIBRE LENGTH, FIBRE ORIENTATION AND VOIDS ON THE CHARACTERISTICS OF COMPRESSION AND INJECTION MOULDED CELLULOSE FIBRE-REINFORCED POLYLACTIDE (PLA) COMPOSITES

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Abstract

The influences of fibre loading, fibre orientation, fibre length and voids on the mechanical characteristics of lyocell fibre-reinforced polylactide (PLA) composites with a fibre loading of 20, 30 and 40 mass-% were analysed. Composites were produced via compression moulding (CM) and injection moulding (IM). An increasing tensile strength was observed with an increasing fibre loading from 20 to 40% for CM composites, while the highest values of IM samples were measured for a fibre loading of 30%. A better fibre/matrix adhesion and compaction was observed for IM composites and the fibre orientation differed not significantly from that of the CM composites. Addtionally the tensile strength of extracted fibres was analysed and has shown only slightly lower values of IM fibres as compared to CM fibres. Despite these findings strength values of IM composites are significantly lower than that of CM composites. It was shown that the fibre length of IM composites drastically decreased during processing with an increasing fibre loading. Additionally, a high proportion of voids was found in the central part of IM samples reinforced with 40% fibres resulting in a notch effect and reduced tensile strength in combination with the low fibre aspect ratio. The lower mechanical characteristics of IM samples with a fibre loading of 20 and 30% compared to CM samples are attributed predominantly to the lower fibre aspect ratio.

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1. Introduction

It is well known that properties of cellulose fibre-reinforced composites are difficult to compare. Characteristics of fibres and matrix itself [1,2,3], fibre loading [4,5,6], fibre/matrix adhesion [7,8,9], fibre orientation [10,11], fibre length [1,12,13], compaction (a good compaction means a low volume of voids) [14] and the production process as well as the procedural settings may affect the composite properties clearly.

For a relatively new composite system like cellulose fibre-reinforced bio-based plastics it is very important to investigate the influences of CM and IM on the resulting composite characteristics systematically to understand the relationship between properties and structures of fibres, matrix and the composite. Mechanical properties of composites with different fibre loadings, fibres of variable fineness, compaction, fibre orientation, fibre length and potential damage of the fibres by the different processes (CM vs. IM) were investigated. Regenerated cellulose fibres (lyocell) were used as reinforcement in a polylactide (PLA) matrix.

2. Materials and methods

As reinforcing fibres Tencel® lyocell fibres were supplied by Lenzing AG (Lenzing, AT) with fibre fineness values of 1.3, 3.3, 6.7, 12.0 and 15.0 dtex. The length of the staple fibres was in the range between 38 and 100 mm. The fibres were analysed with tensile tests at a gauge length of 3.2 mm and a testing speed of 2 mm/min. Polylactide (PLA) was used as a matrix and was supplied in fibre form (Ingeo fibres type SLN 2660 D; Eastern Textile Ltd., Taipei, TW) with a fineness of 6.7 dtex and a staple fibre length of 64 mm. Fibres were produced from a NatureWorks™ 6202 D PLA with a density of 1,24 g/cm³, a melting temperature of 160 - 170 °C and a glass transition temperature of 60 - 65 °C. Composites were produced with an compression (CM) and injection moulding (IM) technique and were investigated with respect to their tensile characteristics, their void distribution and fibre orientation via µ-CT [15]. Fibres were extracted from the different composites and were analysed for their tensile strength after processing as well as their average and medium fibre length L. The critical fibre length L_c (fibre length which is necessary for a reinforcement effect in a composite was determined in a previous study [17]) An detailed description of the used methods can be found in [16].

3. Results and discussion

The tensile strength of CM and IM composites reinforced with different fine lyocell fibres of variable fibre loadings is shown in Figure 1. The IM neat PLA matrix shows a significantly higher tensile strength than the CM sample which may be based on the process-related parameters.

Despite of the higher tensile strength of the neat PLA matrix, significant lower strength values of the IM composites were measured (compare Figure 1). The differences in tensile strength between CM and IM samples increase significantly with an increasing fibre loading. The largest difference was found for a fibre loading of 40%.

As shown in Figure 1 (left) a significant improvement of tensile strength was achieved for CM composites with increasing fibre loading up to 40%. IM samples reached the highest tensile strength at a fibre loading of 30% (Figure 1, right). The tensile strength of IM composites reinforced with 40% fibres was statistically at the level of the PLA-matrix or even lower. Since the IM PLA-matrix has a higher tensile strength than CM PLA, a degradation of the matrix which may lead to lower strength of the IM composites can be excluded. To test if fibres are more damaged by IM than by CM, fibres have been extracted from an IM and CM lyocell/PLA composite and were investigated with a tensile test. The damage of IM fibres is slightly higher but in comparison to the values of CM fibres not significant.

Among other things the decreasing tensile strength of the composites reinforced with fibres of increasing fibre diameter may be explained with the lower tensile strength of the coarser fibres (compare [16]). Moreover, the specific fibre surface decreases with an increasing fibre diameter leading to a smaller bonding surface between fibre and matrix. As shown in Figure 1 the influence of the fibre fineness on the tensile strength is larger for IM composites than for CM composites. This phenomenon may be attributed to a better compaction of the IM composites due to a more efficient mixing process and the high pressure in the IM machine compared to CM specimens. Consequently, the higher specific fibre surface has a more important influence.

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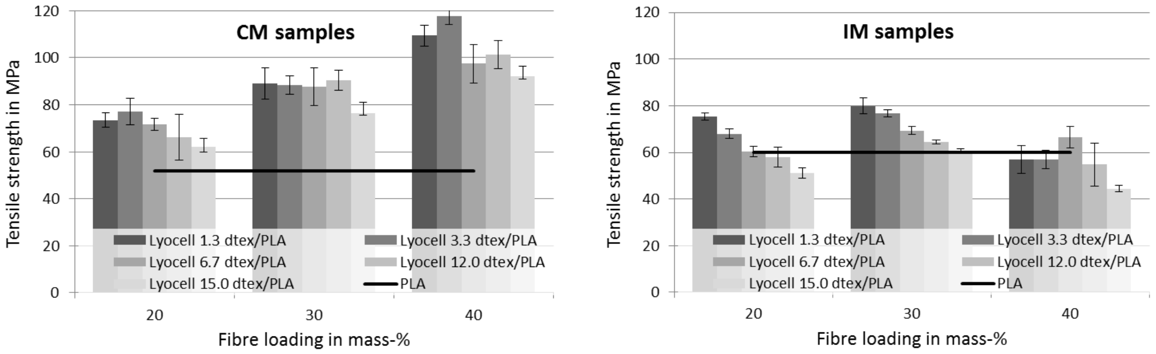

Figure 1: Tensile strength of PLA composites reinforced with 20, 30 and 40 mass-% lyocell fibres of variable fineness produced with the CM process (left) and the IM process (right) (data from [15]).

Voids were visually detected with SR μ-CT micrographs (compare [15]). Figure 2A shows void proportions in CM 20% lyocell 1.3 dtex/PLA. The voids are formed mainly at the top side of the sample. The reason for this is the degassing and evaporation of residual moisture. Since the gases cannot escape upwards, a porous layer is formed which can have a negative influence on the mechanical properties.

No voids were identified for IM composites with fibre loadings of 20 and 30 mass-%. Nevertheless, at a fibre loading of 40% a high amount of voids was found in the central part of the sample (compare Figure 2B). These voids may be the reason for reduced tensile strength.

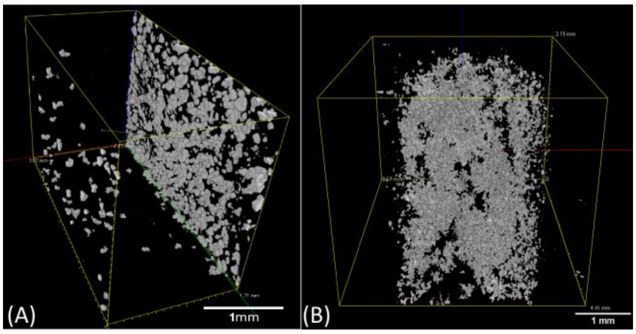

Figure 2: Voids (light grey) detected with SR-μ-CT of CM 20% lyocell 1.3 dtex/PLA and IM 40% lyocell 1.3 dtex/PLA (data from [15]).

Tensile fracture surfaces of CM and IM composites were investigated via scanning electron microscopy (SEM). Longer fibre pull-outs and larger gaps were visible in the fracture surface of the CM samples compared to the IM samples. This phenomenon can be explained with the better compaction and fibre/matrix adhesion of IM samples leading to a better stress transfer from the matrix to the fibre.

Despite of the considerably better fibre/matrix adhesion in IM samples, the higher tensile strength was determined for the CM samples. The investigation of fibre orientation and fibre length may lead to possible explanations for this phenomenon. According to the established hypothesis it is assumed that fibres which are processed with the IM technique are shorter and less oriented in length direction to the specimen axis as compared to CM composites.

To elucidate the different behaviour with respect to the mechanical properties of the CM and IM composites, measurements of fibre orientations were taken on the basis of the SR μ-CT images for selected samples (compare [15]).

No differences of the fibre orientation angles in dependence on the fibre fineness were detected for the IM composites. However, an increasing fibre loading led to a reduction of axially oriented fibres. With a fibre-reinforcement of 20 mass-%, 31 to 34% of fibres are oriented in axial direction. With an

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increasing fibre loading up to 40 mass-% this value decreased clearly to 24 to 27%. Due to the larger number of fibres, there is a higher probability that fibres interfere with each other during processing, leading to a lower preferred orientation.

For the CM specimens another trend was observed. The frequency of axially oriented fibres increases significantly with an enlarging fibre cross-section. While a fibre loading of 20 mass-% lyocell 1.3 dtex/PLA leads to a frequency of 29% axially oriented fibres, for lyocell 6.7 dtex/PLA and for lyocell 15.0 dtex/PLA a value of 30% and 42% was found, respectively. As described for the IM samples significantly lower values were measured for a fibre-reinforcement of 40 mass-%.

Based on the SR µ-CT measurements the samples were gradually analysed in different layers. The results show a different behaviour for CM and IM samples. For the IM samples a higher number of axially oriented fibres was measured in the edge regions of the sample. For the CM specimens a consistent orientation was determined across the layers.

The various fibre orientation of CM and IM samples is based on the different processing techniques. In the present work the CM specimens were prepared from a composite board made from multilayer webs with a fibre orientation mainly in production direction. Apart from the melt front of the PLA matrix no flow processes appear during CM, while the fibres processed with the IM technique are oriented due to various factors like flow processes, fibre/fibre interaction, fluid pressure, back pressure, design of the tool, mould temperature, melt viscosity, shear deformation et cetera.

The lower preferred fibre orientation in the centre of the IM samples indicates a swirling of the fibres. During IM, the fibres are oriented predominantly parallel to the flow direction. Freezing of the polymer at the cavity wall results in a fixation of the fibres. At the boundary layer of the melt, shear flow leads to a preferred fibre orientation (parallel to the flow direction). In the centre of the sample, the shear flow is lower resulting in fibre-swirls. The different fibre orientation of CM and IM samples is confirmed by Fara & Pavan [10] for CM 50% glass fibre-reinforced PA 6.6 and IM 50% glass fibre-reinforced polyarylamide. The authors describe a constant fibre orientation of the CM samples over the sample thickness, and a lower fibre orientation angle in axial direction of the IM specimen in the middle of the sample compared to the shell layers. Thus, for our cellulose fibre-reinforced composites we can show compareable results to glass fibre-reinforced composites.

An orientation factor η_O indicates the reinforcement effect of an individual fibre as a function of the fibre orientation angle φ. The fibre orientation factor can be calculated according to equation 1. The higher the orientation factor (highest value = 1) the higher the fibre orientation in axial direction.

$$\eta_O = \cos^2(\varphi) \qquad (1)$$

Table 1: Mean fibre orientation angles (φ) and orientation factors (η_O) of CM and IM lyocell/PLA composites.

	Compression moulding		Injection moulding	
Composite	φ in °	η_O	φ in °	η_O
20% lyocell 1.3 dtex/PLA	24.5	0.776	23.6	0.786
40% lyocell 1.3 dtex/PLA	27.8	0.731	31.2	0.687
20% lyocell 6.7 dtex/PLA	22.5	0.805	23.9	0.781
40% lyocell 6.7 dtex/PLA	26.8	0.743	27.5	0.736
20% lyocell 15.0 dtex/PLA	19.6	0.832	24.5	0.775
40% lyocell 15.0 dtex/PLA	22.2	0.802	30.7	0.693

The results of η_O for CM and IM samples are given in Table 1. φ and η_O exhibit a slightly higher longitudinal fibre orientation for CM specimens. The use of coarser fibres leads to a better fibre orientation during the carding process of the fibre webs for the CM samples. For IM samples the fibre fineness has no significant influence on the average fibre orientation angle. An increase of fibre mass content from 20 to 40% resulted in a lower frequency of axially oriented fibres for both, CM and IM samples. It should be noted that these differences are not high enough to explain the significant differences and trends of the tensile strength between IM and CM composites. It is hypothesised that the fibre length has a significant influence. This aspect is analysed in detail as follows.

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While the fibres are only slightly shorten by the CM method, the IM process leads to a clear shortening of the fibres [13]. To determine the fibre length distribution of IM samples, the fibres were extracted from the composites. Table 2 shows the measured mean and median fibre length values. As shown for the finest and coarsest lyocell fibres the fibre length increases with a higher fibre loading. However, the aspect ratio decreases due to the larger fibre diameter. An increasing fibre loading results in shorter fibres due to the higher friction and fibre/fibre interactions as well as the higher viscosity of the melt.

Table 2: Fibre length (L), aspect ratio (L/d) and critical fibre length (L_c) of extracted lyocell fibres from IM composites reinforced with fibres of variable fibre fineness and different fibre loading (mass-%).

Fibre (fibre loading)	d in µm	L in µm		L/d		L_c in µm
		Mean	Median	Mean	Median	
Lyocell 1.3 dtex (20%)	10.5	841	489	80	47	220
Lyocell 1.3 dtex (30%)		730	467	69	45	
Lyocell 1.3 dtex (40%)		533	254	51	24	
Lyocell 3.3 dtex (20%)	16.7	1152	602	69	36	351
Lyocell 3.3 dtex (30%)		861	580	52	35	
Lyocell 3.3 dtex (40%)		663	275	40	16	
Lyocell 6.7 dtex (20%)	23.9	1426	962	60	40	500
Lyocell 6.7 dtex (30%)		983	661	41	28	
Lyocell 6.7 dtex (40%)		1034	544	43	23	
Lyocell 12.0 dtex (20%)	31.9	1920	1712	60	54	669
Lyocell 12.0 dtex (30%)		1078	720	34	23	
Lyocell 12.0 dtex (40%)		712	569	22	18	
Lyocell 15.0 dtex (20%)	35.7	1956	1733	55	49	748
Lyocell 15.0 dtex (30%)		1089	724	31	20	
Lyocell 15.0 dtex (40%)		979	589	27	16	

Characteristic values for the fibre/matrix adhesion are determined in previous studies [17]. On this basis, conclusions can be drawn which allow a comparison of the critical fibre length L_c to the actual measured fibre length L in the composite. As presented in Table 2, the measured average fibre length is larger than L_c. However, if the median values are considered for composites reinforced with 40 mass-% fibres it can be seen that these values are very close or even below the values of L_c. The calculated critical aspect ratio is 21 (aspect ratio which is necessary to achieve a reinforcement effect in a composite, [16]). A nearly linear relationship of the measured aspect ratio as a function of the fibre diameter was found with a coefficient of determination (R^2) of 0.80 to 0.94.

Since the granules for IM were not produced with a standard compounding process, but from shredded CM sheets with a uniform cutting length it is assumed that the different fibres have a similar length before IM. Measured shortening effects can therefore be attributed solely to the IM process. An increase of fibre loading results additionally in a stronger shortening of the fibres and a lower aspect ratio (Table 1 and Table 2). The relationship between fibre loading and aspect ratio is almost linear (R^2 = 0.95 to 0.99). The low tensile strength of 40 mass-% reinforced IM composites can thus be attributed to the high porosity in the centre of the sample, the lower orientation and the low aspect ratio of the fibres. Even though a reinforcement effect with a fibre loading of 20 and 30% should be achieved for finer fibres (1.3 dtex - 6.7 dtex); the fibre length L is larger than L_c and the compaction is even better as described for CM composites. Nevertheless, a lower tensile strength was detected for IM samples. As a main reason the smaller fibre length in IM samples compared to CM samples was identified. It is assumed that a fibre length on the level of L_c leads to a reinforcing effect, however, the fibre cannot provide its full reinforcing effect since debonding of fibres under mechanical stress usually starts at the fibre ends [1,2]. The shorter the fibre, the easier it is to pull the fibre out of the matrix. Additionally, more fibre ends exist which may lead to the initiation of debonding. Thus, beside L_c a specific fibre length must exist which is needed for a maximum load transfer from the matrix to the fibre. For our composites it can be concluded that the fibre length in IM samples is not sufficient to use the full

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potential of the reinforcing fibres resulting in a lower tensile strength. Beckermann & Pickering [18] calculated for hemp, that the fibre length needs to be $L = 4 \cdot L_c$ in a PP matrix to achieve the full reinforcement potential. For this calculation, the authors used the modified rule of mixtures according to Kelly & Tyson [19] (equation 2, with the composite strength σ_C, the fibre strength σ_F the fibre volume fraction V_F, the fibre length efficiency factor η_L, the fibre orientation factor η_O, the matrix strength σ_M and the volume fraction of the matrix V_M).

$$\sigma_C = \sigma_F\, V_F \cdot \eta_L \cdot \eta_O + \sigma_M \cdot V_M \qquad (2)$$

$$\text{with} \quad \eta_L = 1 - (L_c/2 \cdot L) \qquad \text{for } L \geq L_c$$
$$\text{or} \quad \eta_L = L/(2 \cdot L_c) \qquad \text{for } L < L_c$$

In equation 2 a perfect fibre/matrix adhesion of the straight aligned fibre is assumed. According to Kelly & Tyson [19] the fibre can be fully loaded at a fibre length of $L = L_c$; but only in a small area in the middle of the fibre. With an increasing fibre length this area is enlarged resulting in a higher stress transfer and tensile strength of the composite.

The model of Kelly and Tyson describes a decrease in tensile strength at a fibre length of $L >> L_c$. The Kelly-Tyson model considers the fibre strength at the corresponding clamping length (fibre length). The fibre tensile strength decreases with an increasing clamping length due to the increasing probability for defects in the fibre volume. Beckermann & Pickering [18] argue that a larger fibre length than L_c has no negative influence on the tensile strength of the composite. Long fibres may break under tension before the composite fails and the two broken parts of the fibre can be loaded again [20]. The Kelly-Tyson model only considers one break per fibre. Nevertheless, using this model makes it possible to determine the fibre length which is necessary for a maximum reinforcing effect.

By determining a regression equation of the fibre tensile strength as a function of the clamping length (results not shown), it is possible to predict the fibre tensile strength at different fibre lengths. If the Kelly-Tyson model is applied to 30% lyocell/PLA composites one obtains the graphs shown exemplary in Figure 3. The fibre length factor was calculated on the one hand with the fibre tensile strength depending on the clamping length and on the other hand with the fibre strength determined at a gauge length of 3.2 mm. The critical fibre length values for the different fine lyocell fibres were taken from Table 2.

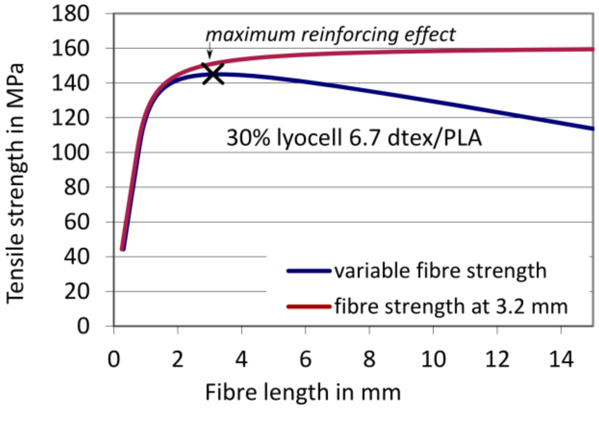

Figure 3: Tensile strength of a 30% lyocell 6.7 dtex/PLA composite according to equation 2 as a function of the fibre length. Values were calculated on the one hand with a tensile strength determined at specific clamping lengths (fibre length) and on the other hand with a fibre tensile strength measured at a gauge length of 3.2 mm. The fibre length necessary for the maximum reinforcement effect is shown as intersection.

An increase in tensile strength with increasing fibre length is visible for all composites (Figure 3). Above a certain fibre length an approximation to a maximum value occurs. When the fibre tensile strength is used for a variable gauge length the values of the composite decrease above a certain fibre length. If a constant fibre strength is used for the calculation, the composites´ tensile strength rises

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asymptotically. The fibre length at maximum tensile strength of the composite calculated with variable fibre tensile strength is the required fibre length for the maximum reinforcing effect. In the case of lyocell 1.3 dtex, lyocell 6.7 dtex and lyocell 15.0 dtex this fibre length corresponds to 2.35 mm, 3.00 and 4.65 mm, respectively. It can be concluded that the minimum fibre length for the maximum reinforcement effect is mainly dependent on the fibre/matrix adhesion, the fibre tensile strength and the fibre fineness. The investigation of different fibre loadings has revealed that the minimum fibre length is not affected.

Since the fibre orientation is only slightly different between CM and IM samples reinforced with lyocell fibres of a fineness between 1.3 and 6.7 dtex, the compaction of IM specimens with fibre loadings of 20 and 30 mass-% is even better and the tensile strength of the extracted fibres from CM and IM composites differed not significantly from each other, the fibre length - and especially the fibre aspect ratio - is mainly responsible for the higher reinforcing effect in CM composites.

4. Summary and conclusion

It can be concluded that the composites´ tensile strength is affected by a number of properties and processing parameters. In addition to fibre and matrix characteristics and the amount of fibre loading, processing parameters, compaction, voids, fibre orientation and fibre length have an influence on the characteristics of cellulose fibre-reinforced PLA composites. IM samples reinforced with fibre loadings of 20 and 30 mass-% have a better compaction than CM samples resulting in a better wetting and a higher influence of the fibre fineness on the tensile strength while an increasing tensile strength was determined with increasing fibre loading for CM composites. In contrast to this, tensile strength of IM samples decreased at a fibre loading of 40%. This behaviour is based among other things on the fibre aspect ratio which was determined to be below the critical aspect ratio and the presence of voids in the centre of 40% IM lyocell/PLA. An increase of the fibre loading leads to a lower preferred fibre orientation in CM and IM composites. The fibre tensile strength was not significantly reduced by IM compared to CM. Fibres processed with the CM technique have a fibre length $L >> L_c$. The IM process leads to a decrease in fibre length with an increasing fibre loading; the aspect ratio decreased more clearly for coarser fibres. To reach the maximum reinforcing effect, the fibre needs to be multiple longer than L_c. Thus, the smaller fibre length is mainly responsible for the lower tensile strength of IM samples compared to CM samples. It is assumed that the use of a sufficient fibre length and an elimination of voids would lead to higher strength values of IM samples compared to CM samples.

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