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Novel cellulose fibre reinforced thermoplastic materials

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Abstract

Spun cellulose fibres from the viscose, lyocell and carbamate processes have been used to reinforce thermoplastic commodity polymers, such as polypropylene (PP), polyethylene (PE) and (high impact) polystyrene (HIPS) as well as poly(lactic acid) (PLA) and a thermoplastic elastomer (TPE) for injection moulding applications. A specially developed double pultrusion technique has been employed for compounding. Fibres were analysed in single fibre tensile tests. Strength, stiffness, impact strength, and heat distortion temperature (HDT) were determined for injection-moulded standard test specimen and structural features were revealed by scanning electron microscopy. A strong reinforcing effect was observed in all cases. In particular, high tenacity tyre cord rayon gives excellent composite strength and impact strength, often doubling or tripling the pristine matrix values. In the case of PP, Lyocell type fibres provide enhanced stiffness and HDT, and thus the combination of both fibre types leads to a balanced composite property profile. The PE case is very similar to PP. For HIPS mainly strength and stiffness is increased, while for TPE the property profile is changed completely. With PLA, a biogenic and biodegradable composite with excellent mechanical properties is presented.

Introduction

Natural cellulosic fibres originating from wood or annual plants were being used for reinforcing both thermoplastic materials and thermosets for a number of years (see, e.g., Bledzki and Gassan 1999). In the field of injection moulding, which is the topic of the present paper, bast fibres (flax, hemp, jute etc.) are used to replace glass fibres as the predominating reinforcing component for low to medium price technical applications (mainly with polypropylene (PP) as matrix). This is motivated by advantages over glass fibres such as lower cost, lower density (1.5 g/cm^3 vs. 2.5 g/cm^3), lack of abrasion to the processing equipment and ease of incineration. Although progress has been made in properties and processing in the past few years (compare, e.g., Snijder and Oever 2005 for jute–PP) larger scale industrial applications are not yet implemented. This might be caused not only by problems in a steady supply of fibre material with guaranteed quality, but also by deficits in impact behaviour typical for natural fibre composites. The low impact strengths are likely to originate from the internal composite structure of the lignocellulosic fibres themselves causing brittle fracture events within the fibres.

Spun Cellulose fibres, on the other hand, possess all the advantages of a man-made industrial fibres in terms of supply, quality control, and uniformity, maintaining the advantages mentioned above for natural fibres. At moderate price they offer remarkable mechanical properties (strengths can be above 50 cN/tex (750 MPa), moduli up to 2000 cN/tex (30 GPa)) which make them a promising candidate for reinforcing thermoplastic matrices. Compression-moulded short-cut rayon-reinforced PP was studied for the first time by Amash and Zugenmaier, 1998 (see also Amash and Zugenmaier 2000), showing up the great potential of this fibre–matrix combination. The potential of Lyocell fibres for reinforcing PP was investigated by Lützgendorf et al. 2000. In the group of W. Glasser, Tencel fibres were used for unidirectionally reinforced cellulose ester composites including both the processing route (Seavey et al. 2001) and fibre surface modifications (Seavey and Glasser 2001). A further attempt to use a spun cellulosic fibre as polypropylene reinforcement for injection moulding applications has been found in the literature (Paunikallio et al. 2003, 2004). First results from our laboratory for injection moulded composites with rayon tyre cord yarn and a polypropylene block copolymer are published in Weigel et al. 2002 (see also Fink et al. 2004 and http://www.new-composites.com).

In the present paper the capabilities of spun cellulose fibres of various origin (viscose, lyocell, carbamate) to reinforce thermoplastic matrices, i.e. polypropylene, polyethylene, high impact polystyrene, poly lactic acid, and a PP-based thermoplastic elastomer are explored. A special two-stage pultrusion process developed in this laboratory (Gassan et al. 2003) is used to incorporate and cut cellulosic spun yarns in the polymer matrix and produce pellets ready for injection moulding. Structural features of injection moulded test bars are revealed by scanning electron microscopy. Tensile and impact properties are studied and the thermal stability is examined by heat distortion temperature (HDT) measuremets. A more detailed account concerning variations in fibre load, type of coupling agent, and fibre length distributions is given elsewhere (Ganster et al. 2005).

Experimental

Materials

Cellulose fibres

Important mechanical properties of spun cellulose fibres, including those used in the composites which are marked with an asterisk, are presented in Table 1, all measured in this laboratory (cf. sec. Experimental – Methods). Cordenka 700 is a rayon tyre cord yarn provided by Cordenka GmbH Obernburg, Germany and is produced by a special variant of the viscose process. Enka viscose is a typical viscose fibre for textile applications produced by Enka GmbH and Co. KG, Oberbruch, Germany. Viscose sliver was kindly provided by Kelheim Faser GmbH, Germany, and taken from the staple fibre production line (non-wovens applications) by separating a sliver of approximately 2 ktex.

In contrast to the former three, the filament yarn NewCell is spun using a non-derivatising Lyocell process with *N*-methyl morpholine-N-oxide/water as the solvent. The fibre had been manufacured by Akzo Nobel NV, The Netherlands, but is out of production at present. Another Lyocell fibre is Tencel, kindly provided by Tencel Ltd., Grimsby, UK (today part of Lenzing AG, Austria), again separated from a staple fibre sliver (textile and non-wovens applications) to give an appropriate overall titre suited for incorporation into the composite matrix.

Table 1. Titre and mechanical properties of spun cellulose fibres (single fibre measurements).

Material	Titre	Strength			Elongation		Modulus		
	dtex	cN/tex	MPa Average	cN/tex s.d.[b]	% Average	% s.d.	cN/tex Average	GPa Average	cN/tex s.d.
Cordenka 700[a]	1.8	56	833	4	13	2	1300	20	100
Enka viscose	2.8	21	308	1	24	1	704	11	25
Viscose sliver[a]	1.3	23	338	3	12	4	730	11	70
NewCell	1.3	40	603	2	9	1	2060	31	285
Tencel sliver[a]	1.4	37	552	4	11	2	1500	23	170
Carbamate[a]	1.5	24	365	1	8	1	1450	22	60

[a]Used in composite.[b]Standard deviation.

Finally, Carbamate is a cellulose fibre spun in this institute via the CARBACELL process (Voges et al. 2002), an environmental-friendly alternative to the viscose process using urea as the derivatising agent.

Additionally, a 100% Tossa jute yarn with 238 tex, purchased from J. SCHILGEN GmbH & Co., Germany with a yarn tensile strength of 32 cN/tex and a yarn modulus of 1000 cN/tex (own measurements) was used.

Matrix materials

The matrix polymers used in this study as well as some important characteristics as given by the producers are listed in Table 2. The PP is a light flowing block copolymer suited for injection moulding applications. The PE is a high density polyethylene for injection moulding of larger parts, like crates and boxes. HIPS is an impact-modified polystyrene used for the manufacture of TV cabinets and audio equipments. PLA is a biogenic and biodegradable poly(lactic acid) polymer for extrusion and thermoforming applications. An injection moulding grade in this price segment is not available at present. Finally, TPE is a thermoplastic elastomer on the basis of PP with processing conditions similar to PP and rubber-like behaviour at room temperature.

Coupling agents and additives

For coupling the cellulose fibres to the PP and the TPE matrix, maleic anhydride-grafted polypropylene (MAPP) has been used with a high MFI (450 g/10 min at 190 °C and 21.6 N) and a graft level >1 wt% the trade name being Fusabond MD353D (Du Pont). PE has been coupled with Fusabond E MB-100D (Du Pont) with and MFI of 2 g/10 min at 190 °C and 21.6 N and a graft level >1 wt%. For HIPS a poly(styrene-co-maleic anhydride) with 25 wt% maleic anhydride (SMA 3000F, Cray Valley) and one with 7 wt% maleic anhydride and M_w 224000 (Aldrich) was used. PLA has not been coupled at all, due to the absence of a suitable product. An amount of 3 wt% coupling agent with respect to the matrix polymer has been proven to be sufficient in all cases. The talcum used as an additive in one case was Luzenac A7C (Luzenac Naintsch, Austria), a compacted product with diameters below 6.5 μm for 90% of the material.

Methods

Compounding

A pultrusion technique was applied with a conventional co-rotating twin screw extruder (Haake Rheocord 9000 PTW 25) equipped with a coating die assembly to cover a number of (continuous) filament tows or sliver with the molten matrix-coupling agent mixture which was pre-mixed before fed into the extruder. PLA was thoroughly pre-dried in a vacuum oven at 60 °C in the presence of P_2O_5 to avoid water initiated degradation during extrusion. The maximum temperature of the extruder and the die were 200 °C and 195 °C, respectively. The coated yarns were cooled with water and cut into pellets of usually about 4 mm length. Then the pellets were dried overnight at 110 °C for PP and PE, 80 °C for HIPS and at 60 °C *in vacuo* in the presence of P_2O_5 for PLA. In a second step, the pellets were extruded with the same extruder under the same conditions to homogenise the fibre–matrix mixture. The screw configuration was chosen such that appropriate mixing elements were included to guarantee the dispersion of the fibres in the matrix. After cooling the thread was cut into final pellets of defined lengths between 3 and 5 mm with diameters between 2 and 4 mm. Apparent pellet densities (bulk densities) are above 300 g/l in all cases.

Table 2. Matrix polymers used in this study and selected manufacturer's data.

Trade name	Abbreviation	Producer	MFI [g/10 min]	Strength [MPa]	Modulus [GPa]
Stamylan P 412MN40	PP	Sabic	37 at 230 °C, 22 N	26 (yield)	1.55 (bend)
Hostalen GC 7260	PE	Basell	23 at 190 °C, 5 N	30 (yield)	1.35 (tesile)
Lacqrene 4240	HIPS	Atofina	4 at 200 °C, 50 N	26 (yield)	2 (bend)
PLA 2002D Nature Works	PLA	Cargill Dow	4–8 at 190 °C, 22 N	53	3.5
Sconablend TPE 60x111	TPE	Ravago	not specified	4[a]	0.02[a]

[a] Own measurements.

As a standard, a fibre fraction of 25 wt% has been used for all composites considered in the present paper. Polypropylene composite properties as a function of Cordenka fibre load (wt%) have been studied elsewhere (Ganster et al. 2005), giving slopes of 1.6 MPa/wt%, 72 MPa/wt% and 0.25 kJ/m^2/wt% for strength, modulus and notched Charpy impact, respectively.

Injection moulding

Standard test specimens were prepared according to DIN EN ISO 527–2 (for tensile test) and DIN EN ISO 179 (for bending and Charpy impact test) using an injection moulding machine (ES 200/50, Engel, Austria), operating with a nozzle temperature of 210 °C and specific injection pressures between 30 and 70 bar. In contrast to earlier work (Ganster et al. 2005) also a different tool with slightly bigger gates has been used for the preparation of the test bars.

Mechanical testing

Single fibre measurements were performed using a Zwick Z 020 universal testing machine (Zwick Co., Germany) with a 10 N load cell, a clamp separation of 20 mm and a testing speed of 10 mm/min. The fineness has been determined gravimetrically.

Tensile strength and modulus of the composites were measured according to DIN EN ISO 527 and 178, respectively with a universal testing machine (Zwick 020) using the injection-moulded standard test specimen. However, the tensile modulus was determined as the maximum derivative at the beginning of the stress–strain curve measured at 50 mm/min testing speed. Charpy impact strengths of the composites were determined with an impact tester (PSW 4J) according to DIN EN ISO 179 standard in the flatwise, unnoched, or the edgewise notched (notch type A) modes. The test samples were conditioned at 23 °C and 50% relative humidity for several days before testing and all the tests were performed under the same conditions.

Heat distortion temperature (HDT)

Heat distortion temperature was measured along the lines of DIN EN ISO 75, practice A. However, a DMA (TA Instruments 2980) was used as the temperature chamber and the force applying device and therefore air instead of oil was used as the surrounding medium. Comparison to proper ISO 75 measurements performed elsewhere showed that the DMA generated values are somewhat lower (in the order of 5 °C) than the proper ones.

Scanning electron microscopy

Cryo-fractured surfaces were generated by breaking the test bars under liquid nitrogen conditions and subsequent sputtering with Pt with a thickness of 4 nm. The fracture surfaces were studied with an SEM Jeol JSM 6330 (Jeol Corp., Japan) at 5 kV.

Results and discussion

Fibre characterisation

Results of single fibre measurements for various spun cellulose fibres are shown in Table 1. Cordenka tyre cord yarn gives the best values for strength and the best values for modulus among the viscose type fibres, as to be expected for a technical filament fibre. On the other hand, the NNMO type fibres NewCel and Tencel sliver are stiffer with lower elongation and reduced tenacity. The carbamate fibre has strengths in the range of textile viscose, while its modulus is typical for a technical fibre.

Composites with polypropylene matrix

Fibre type variation

In Figure 1, tensile strength, tensile modulus and elongation at break are shown for injection moulded composite standard test bars with 25 wt% fibre load as a function of fibre type. For comparison, the values for the pristine matrix polymer are included. A clear reinforcing effect for all fibre types is obvious: strength and modulus are increased considerably, while the elongation is reduced, as expected. Cordenka and Tencel give the best effects in terms of strength with values of 74 and 66 MPa, respectively. Modulus is best for Tencel and jute with 3.6 and 3.3 GPa, respectively. This is accompanied with the lowest values for elongation at break, both 4%, in sharp contrast to Cordenka, which has the highest elongation (10%) for all reinforced materials. The high scatter in elongation for the pristine PP is due to the statistic nature of the yielding process. No yielding is observed for the reinforced samples.

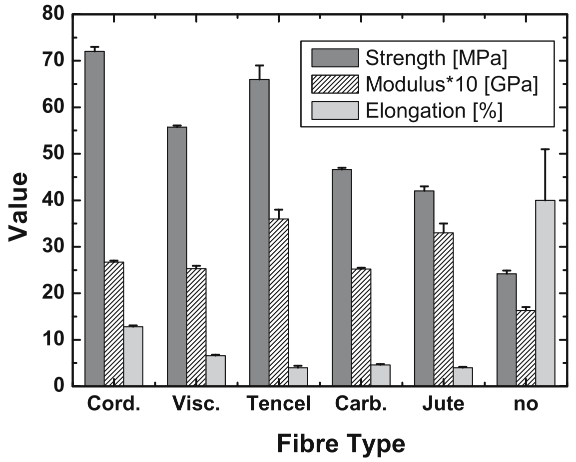

Figure 1. Strength, modulus, and elongation for injection-moulded composite test bars with 25% fibre load as a function of fibre type and for the pristine polypropylene.

In Figure 2, Charpy unnotched impact strength, Charpy notched impact strength and heat distortion temperature are displayed in the same manner as in Figure 1. By far the best Charpy values among the reinforced samples are found for Cordenka. Notched Charpy impact strength is 14 kJ/m^2 and the unnotched experiment it does not even fail (as the pure PP). The high stiffness level for Tencel and jute found in Figure 1 is confirmed in Figure 2 by the high HDT values. That means even under elevated temperatures a good bending stiffness is maintained for these composites. On the other hand, the Cordenka reinforced composites, i.e. the best performing material in terms of strength and impact strength, give values in the range of 80 °C, which is somewhat low for a series of applications, e.g. in the automotive industry (see below).

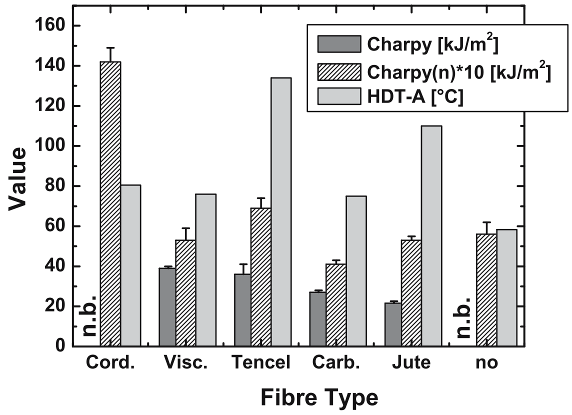

Figure 2. Charpy and notched Charpy impact strength, and heat distortion temperature for injection moulded composite test bars with 25% fibre load as a function of fibre type and for the pristine polypropylene (n.b. – not broken).

Differences in the macroscopic behaviour of cellulose fibre reinforced-PP can also be supposed from the microscopic cryo fracture surfaces of the composites, studied in the range from 500 to 1 μm. In Figure 3, left, an overview for a composite not coupled at all is given. No matrix material adheres to the fibres which are pulled out to a large extent. The coupling with MAPP works for all the three fibre types shown in Figure 3, right (Cordenka) and Figure 4, left (Tencel) and right (jute). However, differences are obvious. Cordenka is heavily covered with matrix polymer and the stumps of pulled out and broken fibres are short. Tencel is also covered with matrix, but not as thick as in the Cordenka sample, and the fibres are pulled out more. Jute is well anchored in the matrix, but breaks in a way that shows its internal composite structure. Differences in the broken fibre morphology between Cordenka and Tencel are also observed.

In order to provide a link between fibre and composite properties, a modified rule of mixtures can be applied for strength and modulus (Bader and Hill 1993) by using so-called efficiency factors accounting for finite fibre length, orientation and, in case of strength, coupling effects

$$\sigma_{\mathrm{exp}} = \eta_\sigma v_\mathrm{f} \sigma_\mathrm{f} + (1 - v_\mathrm{f})\sigma_\mathrm{m} \quad \text{and}$$
$$E_{\mathrm{exp}} = \eta_\mathrm{E} v_\mathrm{f} E_\mathrm{f} + (1 - v_\mathrm{f}) E_\mathrm{m},$$

where σ_{exp} and E_{exp} are the measured composite strength and modulus values, $\sigma_\mathrm{f}, \sigma_\mathrm{m}$ and E_f, E_m those for the fibre and matrix, respectively, and v_f the fibre volume fraction (0.17 in the present case). The two equations can be considered as the definition of the efficiency factors η_σ for strength and η_E for modulus. In the limit $\eta \to 1$ the rule of mixtures for a parallel two phase (matrix and fibre) model are retrieved. Results are given in Table 3. Obviously, the best performing cellulose spun fibre in terms of efficiency is the viscose sliver. Jute, on the other hand gives low strength efficiency and high modulus efficiency at the same time. But here it should be kept in mind that the results are calculated with yarn properties, such that the fibre modulus is measured too low and thus η_E is too high. The same holds true for η_σ and

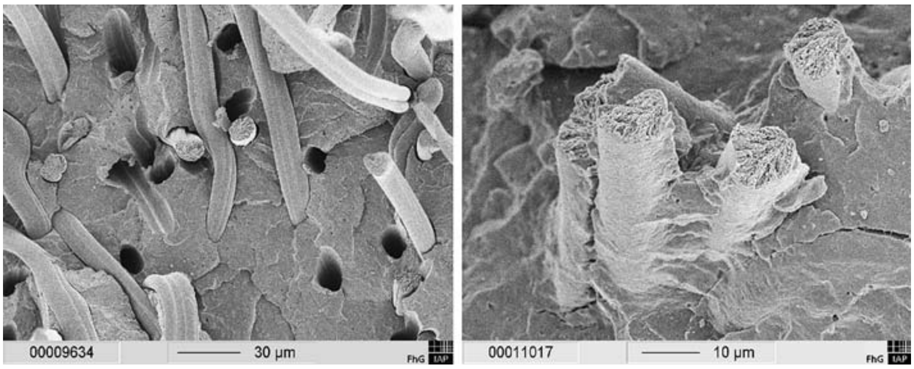

Figure 3. SEM cryo-fracture surface images of uncoupled Cordenka–PP composite (left) and coupled composite with 3 wt% MAPP (right).

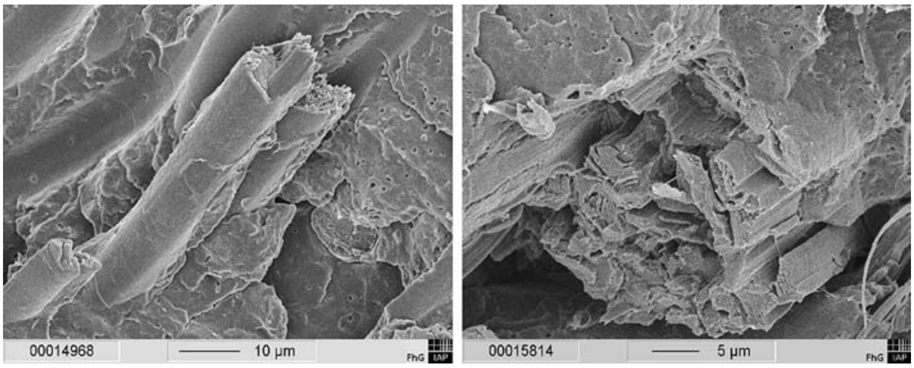

Figure 4. SEM cryo-fracture surface images of 3% MAPP coupled Tencel–PP composite (left) and jute–PP composite (right).

Table 3. Strength (η_σ) and modulus (η_E) efficiency factors for polypropylene reinforcement with 25 wt% cellulose fibres according to Table 1 and jute.

Fibre type	η_σ	η_E
Cordenka 700	0.37	0.39
Viscose sliver	0.63	0.64
Tencel sliver	0.50	0.58
Carbamate	0.43	0.32
Jute	0.27	0.78

thus the strengthening efficiency is even lower than 0.27, hinting again at the internal composite structure as the cause for breakage. For Cordenka, which gives the best composites, the efficiency factors are moderate, demonstrating that other effects not covered in these simple rules of mixture are significant.

Mixed reinforcement

In order to obtain a balanced property profile in terms of stiffness/HDT on the one hand and strength/impact strength on the other, the results of the preceding section suggest a combination of reinforcement fibre types. Additionally, the classic stiffener talcum has been used to reach this goal. In Figures 5 and 6, results for mixed reinforcement are presented in the same manner as in Figures 1 and 2. The composition of the composites were as follows: Cord./talc. (25 wt% Cordenka + 10 wt% talcum), Cord./Jute (22 wt% Cordenka + 8 wt% jute), Cord./Tencel (18 wt% Cordenka + 7 wt% Tencel). In each case 3 wt% of MAPP coupling agent has been added to the PP matrix. Obviously, for all compositions, a good stiffness (around 3 GPa) and HDT (>100 °C) is combined with high strength (>65 MPa) and high impact strength (>70 kJ/m^2 for unnotched and >12 kJ/m^2 for notched Charpy). Finding even higher notched Charpy values for the Cord./Jute composite than for the pure Cordenka one is ascribed to the slightly higher overall fibre load and the scatter of the values.

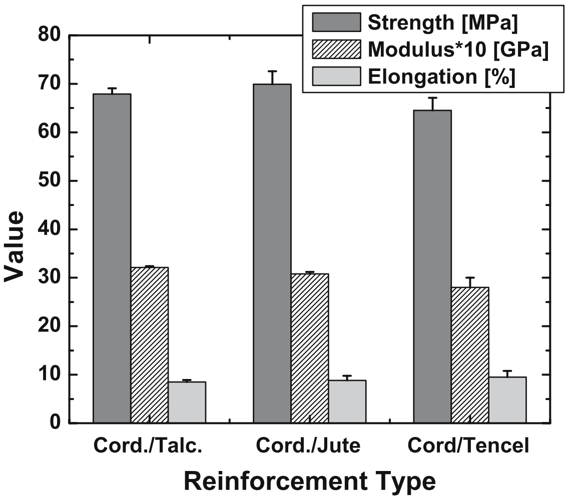

Figure 5. Strength, modulus, and elongation for injection moulded composite test bars with mixed reinforcement (for compositions see text).

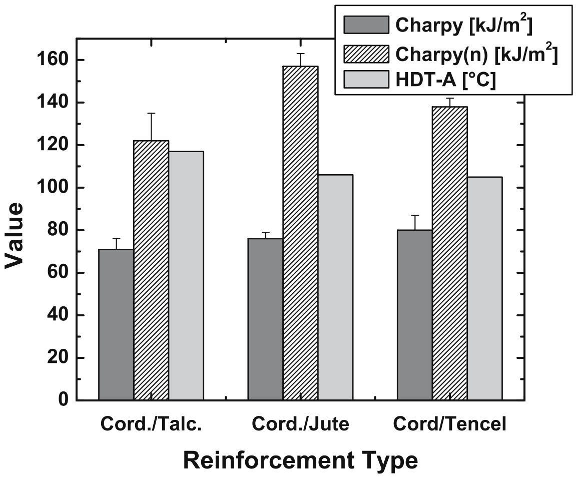

Figure 6. Charpy and notched Charpy impact strength, and heat distortion temperature for injection moulded composite test bars with mixed reinforcement (for compositions see text).

Composites with alternative matrices

Polyethylene

Polyethylene, like PP, belongs to the class of inexpensive commodity thermoplastics and is worth testing as a matrix material. Results for the mechanical testing of Cordenka reinforced polyethylene (PE) composites together with the values for the pure PE are shown in Figure 7. The effective reinforcement of the matrix by the cellulose spun fibre is obvious. Tensile modulus is almost tripled, strength and notched impact strength are more than tripled and increased 5-fold, respectively. With 79 kJ/m^2 unnotched Charpy is on a very high level. The pure PE does not fail in this experiment. This is, of course, the best result that can be obtained. However, it must be kept in mind that the modulus of the pristine PE is quite low. Therefore, the forces acting on the sample surface during impact, which are responsible for crack initiation, are comparatively low. In that way, low modulus materials are favoured in such kind of test.

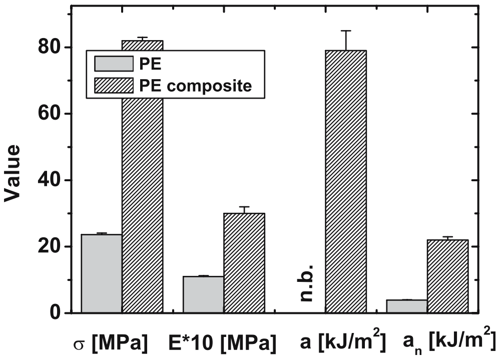

Figure 7. Tensile Strength(σ), modulus (E), Charpy unnotched (a) and notched (a_n) impact strength for injection moulded PE test bars with 25 wt% Cordenka and for the pristine PE (n.b. – not broken).

High impact polystyrene

In line with PP and PE, polystyrene is another inexpensive commodity polymer, giving clear but rather brittle products. Therefore, the polymer is often modified with rubber components giving so called high impact polystyrene (HIPS). Due to its brittleness, the unmodified polystyrene could not be reinforced with the processing method used in this study and thus HIPS was tested as a still inexpensive matrix polymer. Results for mechanical properties of the pristine material and of composites with 25 wt% Cordenka reinforcement are presented in Figure 8. The pure polymer has good impact properties, but a rather low modulus and, in particular, tensile strength. The latter are increased by reinforcing with Cordenka already without using a coupling agent. The right two sets of columns in Figure 8 represent the values for composites with 3 wt% coupling agent of different architecture. 25% MA means the styrene–maleic

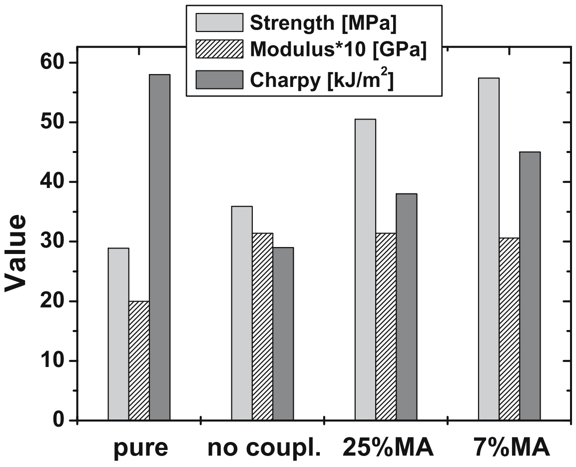

Figure 8. Strength, modulus, and unnotched Charpy impact strength for pure HIPS and composites with 25% Cordenka without coupling and with two different coupling agents (see text).

acid anhydride copolymer with 25% maleic acid anhydride, as specified in the experimental section, while 7% MA stands for the 7% maleic acid anhydride product mentioned there. Obviously, the lower MA concentration favours both strength and impact strength. This can be explained by the characteristics of the coupling mechanism. On the fibre side, the anhydride group reacts with the cellulose hydroxyl groups to form ester linkages (cf., e.g., Felix and Gatenholm 1991) and on the matrix side, the styrene loops of the coupling agent are entangled in the matrix polymer. The loops are very short for 25% MA and become longer for the 7% MA coupling agent improving in that way the anchorage in the matrix polymer. Further improvement is therefore to be expected by (a) decreasing the MA concentration and (b) using a grafted product instead of a copolymer to improve the accessibility.

Poly(lactic acid)

A quite different matrix type investigated in this study is poly(lactic acid) (PLA). This is a biogenic and biodegradable polymer with remarkable mechanical properties available in various types (mostly thermoforming and extrusion) at moderate prices. Reinforcing this material with a cellulosic (spun) fibre opens up new opportunities to design biogenic and biodegradable composites with excellent mechanical performance. However, care must be taken to avoid the presence of water during processing to prevent degradation of the polyester backbone by hydrolysis.

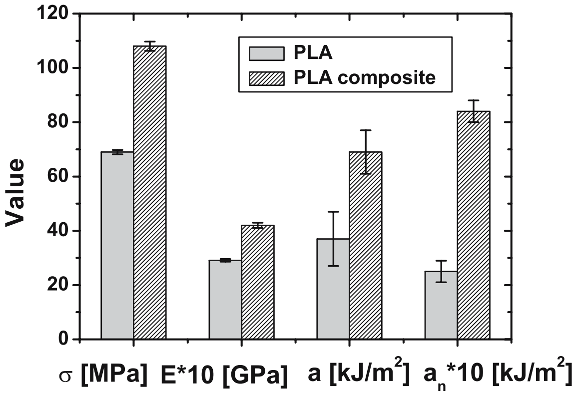

Figure 9. Tensile Strength(σ), modulus (E), Charpy unnotched (a) and notched (a_n) impact strength for injection moulded PLA test bars with 25 wt% Cordenka and for the pristine PLA.

Results for composites with 25% Cordenka and for the pristine PLA are presented in Figure 9. The increase in strength and modulus is about 50%. Again, the Cordenka fibre proves to be an excellent impact modifier: unnotched impact strength is doubled and notched Charpy values tripled. All these values are obtained without using a coupling agent, which, to the authors knowledge, is not commercially available at present.

Polypropylene-based thermoplastic elastomer

Finally, a thermoplastic elastomer has been tested for its possibilities to be reinforced with the cellulosic spun fibre Cordenka, as shown in Figure 10

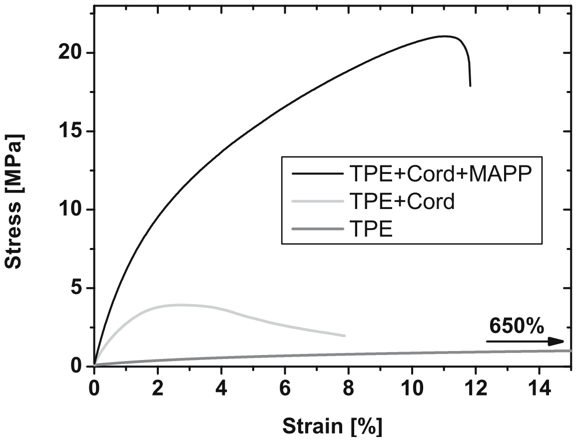

Figure 10. Stress–strain curves for injection moulded TPE composite test bars with 25% Cordenka with and without MAPP coupling agent and for the pristine TPE.

by the stress–strain curves of injection moulded TPE composite test bars with 25% Cordenka with and without MAPP coupling agent and for the pristine TPE. Again coupling with MAPP is very effective. The property profile of the material is changed drastically. Strength is increased 5-fold to 20 MPa and the modulus is 38 times higher than for the pristine matrix. The elongation of the pure TPE of 650% is reduced to 12%, a value found for the reinforcing fibre. In that way, a different type of material is formed for the present fibre content of 25 wt%. By variation of the fibre load it should be possible to tailor the mechanical properties in a way to obtain intermediate values between the extreme cases pure TPE and 25% fibre composite (maybe except for the elongation).

Conclusions

Cellulose spun fibres are suited for reinforcing thermoplastic polymers such as polypropylene, polyethylene, high impact polystyrene, poly(lactic acid) and polypropylene-based thermoplastic elastomers. Important mechanical properties like strength, stiffness and impact strength are increased considerably, often they are doubled or tripled. In particular, high tenacity technical viscose fibres like tyre cord yarn prove to be excellent strength promoters and impact modifiers, while Lyocell type fibres act as stiffeners enhancing the heat stability (HDT) for polypropylene composites. An appropriate combination of fibres (or addition of inorganic fillers) leads to a well balanced property profile in terms of stiffness vs. impact strength in the polypropylene case. For all the matrix materials, except for poly(lactic acid) (PLA), the cellulose fibres are coupled to the respective matrix by adding small amounts of maleic acid anhydride grafted or copolymerised matrix material. In the case of biogenic and biodegradable PLA composites, excellent mechanical properties are obtained without coupling.

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