Effect of Maleic Anhydride Modification on the Mechanical Properties of a Highly Filled Glass Fibre Reinforced, Low-Viscosity Polypropylene for Injection Moulding

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Abstract

Short glass fibre reinforcement is one of the most important measures to improve the mechanical properties of injection moulded parts. The need for increasing performance leads to high fibre contents which is a critical fact due to the contrasting behaviour of strength, stiffness and impact properties. In this study a high-fluidity polypropylene (PP) is compounded with short glass fibres up to 70 weight percent and comparatively investigated with the adding of maleic anhydride (MAH) as compatibilizer. Due to the high fluidity of the PP, which ensures a good embedding of the fibres, the tensile modulus increases up to 65 weight percent, tensile strength up to 50 weight percent of glass fibre content. Adding the MAH increases strength and impact properties, while the tensile modulus only shows an improvement at high fibre content. The influence of the compatibilizer effect diminishes at higher fibre contents, especially for the impact strength which could so far not be clarified exactly. Reasons could be the reduction of fibre length at higher fibre contents and the increasing influence of the brittle fibre component. With the adding of MAH the contrasting behaviour of the impact properties in comparison to strength and stiffness can be reduced and the optimum over all mechanical properties can be shifted to higher fibre contents.

Keywords: injection moulding, glass fibre, polypropylene, maleic anhydride, viscosity

1. Introduction

Lightweight design using fibre reinforced plastics (FRP) has taken a leading role in reducing weight in kinematic systems, since fibre-plastic composites are characterised by highly direction-bound specific mechanical properties, as well as a high functional integration potential. Classical textile reinforced composite parts are preferentially made in processes based on thermosetting plastic that are run on long cycle times, as well as with low levels of automation. To be able to produce fibre reinforced lightweight structures in large quantities, thermoplastic technologies like injection moulding are increasingly used (Mitschang & Hildebrandt, 212). Injection moulding is one of the most economical and flexible processes to produce thermoplastic structural parts such as load carriers (Drummer & Gröschel, 2014) or even foamed sandwich structures (Roch, Menrath, Huber, Henning & Elsner, 2013). It is distinguished from other technologies by the wide variety of materials that can be processed, the short cycle times and the many degrees of freedom in the moulding geometry design (Hopmann et al., 2012).

To cope with the mechanical performance and properties demanded, highly fibre filled thermoplastic materials are frequently used for injection moulded parts. Here, in upstream compounding processes, the initial polymer is enhanced with chopped glass fibres. The fibre addition and embedding are performed using classical plasticising and injection processes. The fibre length in the initial granule is limited to a range from 0.2 to approx. 15 mm for reasons of process execution. It is well known, that the high shear stress in the plasticising and injection processes causes the fibres to break, this being essential for compounding (Fisa, 1985) and injection molding process (Franzen, Klason, Kubat, & Kitano, 1989). The resulting average fibre length in the part is substantially less than the initial fibre length; it ranges from 0.1 to 2 mm.

Besides the resulting fiber length the mechanical properties are influenced mainly by fiber volume content and adhesion promoters. This has been an issue of research for many years and is well known for typical injection molding grades. The resulting fibre length in the finished part has a significant effect especially on the impact

properties. The stiffness of glass fibre reinforced thermoplastic injection moulded parts achieves a very high level with fibres only a few tenth millimeters long, which hardly increases as a function of greater fibre length. A significant increase in strength is observed with fibres longer than 1mm, while once fibre lengths are greater than 10 mm, a saturation level is reached (Thomason, 2002a). Another increase in the resulting part fibre lengths mainly results in an increase in impact resistance. Consequently, using textile reinforced prepregs in injection moulding processes is primarily interesting for impact loaded structures. As a result, these parts are predominantly used in the car industry, for front end, door and rear end parts. Fundamental investigations into embedding the textile prepregs (Khondker, Yang, Usui, & Hamada, 2006), the thermoforming mould design, strategies to preheat insert structures (Giessauf, Pillwein, & Steinbichler, 2008) and ways to improve the bonding of inserts and injected melt were carried out (Heinzle, Huber, Henning, & Elsner, 2014). Optimisation of the fibre-filled injection moulded parts' mechanical properties through textile reinforcements reduces suitability for large series production because of the cycle times required and the part costs.

For this reason, an increase in the performance of the mechanical characteristics in the field of short fibre reinforcement is also sought. Using highly filled plastics, particularly stiffness can be enhanced further. However, the other mechanical properties, such as fracture toughness and impact resistance, are reduced as a function of the fibre concentration (Thomason, 2006). Therefore, extensive investigations were made by Thomason (2007 & 2002b) to the influence of maleic anhydride (MAH) on the properties of long glass fibre reinforced polypropylene at difference fibre content up to 70 weight-%. MAH is typically used for enhancing the glass fibres' bonding characteristics to non-polar plastics, such as PP (De Roover et al., 1995), (Mäder, Moos, & Karger-Kocsis, 2001). Thomason observed an optimum of MAH effect on the mechanical properties, in particular tensile strength as well as impact properties, at about 40% glass fibre content. With higher fibre content the mechanical properties enhanced to a lesser extent. He justified this with the decreasing ratio of the concentration of maleated molecules in the matrix to the glass fibre surface area.

When adding high fibre content and using modifiers to improve the fibre-matrix bonding, attention has to be paid to the processing of these materials in the compounding and injection moulding processes. Increasing fibre content results in an increase in the melt viscosity, fibre-fibre interaction and thus to a greater mechanical stress on the fibres during compounding and injection moulding, which, in turn, negatively affects the resulting fibre length (Thomason, 2001). As a consequence, in the case of high fibre content, it seems to be advantageously to use injection molding grades with low melt viscosity, which are primarily not designed for conventional injection moulding. While many investigations (Thomason, 2001-2007) were made for PP types in the typical range of 15-35 g/10 min for the melt flow index (MFI), there is a lack of research for using high fluidity polymers in combination with high fiber contents. The viscosity is mainly adjusted through the molecular weight and molecular weight distribution. When polymers with low viscosity are used, a better wetting of the glass fibres and higher interfacial shear strength may also be observed (Yoshida, Ishiaku, Okumura, Baba, & Hamada, 2006). However, reduced molecular weights and thus shorter polymer chains in the initial polymer negatively affect the toughness and cause the unreinforced polymer to become brittle. With this study, we want to investigate the mechanical behaviour a polypropylene grade with low viscosity at the limits of injection moulding and compare the mechanical properties that result from the addition of short glass fibres up to a high filler content as a function of the added MAH. This way, the obtainable mechanical properties of fibre filled injection moulding parts are represented in summary, thereby considering feasible material and process parameters.

2. Experimental

2.1 Materials

The polypropylene homopolymeric Moplen HP500V, supplied by LyondellBasell (Rotterdam, Netherlands) was used for the experiments. The MFI measured according to ISO 1133 (230 °C/2.16 kg) was 120 g/10 min. Diminishing ease of flow due to the fibre load was verified in viscosity measurements. Injection moulding types above an MFI of 80 g/10 min are suitable only for very thin walled parts and long flow paths due to their high fluidity and obvious tendency towards embrittlement.

Chopped strands of the CS 7952 (28/779) glass fibre type supplied by Lanxess (Cologne, Germany) of 4,5 mm initial length (fibre diameter 14μm) and with silane sizing was used. The coating volume quantified through loss on ignition according to DIN EN ISO 1887 amounted to 0.52 weight-%.

Based on typical research works (Ho, Su, Wu & Chen, 1993), (Bettini & Agnelli, 2002), (Fu, He & Chen, 2010), (Guo et al., 2006) a grafted polypropylene with maleic anhydride acid (PP-g-MAH) was added to the plastic as a compatibiliser at 2 weight-% (Scona TPPP8012 from Byk Kometra, Schkopau, Germany). The content of the

bound MAH in the compatibiliser was approximately 1 weight-%. The MFI of the compatibiliser was specified with 90 g/10 min (190 °C/2.16 kg) and thus lies at the level of the injection moulding type used.

The PP employed was compounded with glass fibre content from 2 to 70 weight-% and the modifier was added to the master batch during the compounding process. In the abbreviation, the fibre mass content is given as a number following polypropylene. The polypropylene additionally enhanced with MAH also includes the small letter g (example: PP with MAH modifier and 30 weight-% glass fibres corresponds to PPg30).

2.2 Processing and Tests

Compounding to add the glass fibres as well the PP-g-MAH was performed at a laboratory compounder (Noris Plastic ZSC 25/40D) with a co-rotating twin screw, screw diameter of 25 mm and 40 length/diameter ratio. The fibres were added via a side feeder. Compounding was followed by a strand take-off, cooling in a water bath and pelletisation. The materials were injection moulded according to DIN EN ISO 294-1, type A, using an Arburg Allrounder All Drive 370 injection moulding machine with 60 tons of clamp force. The multi-purpose test specimens produced were used for mechanical characterisation. All mechanical tests were executed at 23° C room temperature and 50% relative air humidity. The tensile test was carried out according to DIN EN ISO 527-2, tensile modulus of elasticity was measured at 1 mm/min, and tensile strength and elongation at break were measured at 5mm/min. Both notched and unnotched specimens were used to measure the Charpy impact strength according to ISO 179-2 (edgewise). The heat resistance (heat deflection temperature - HDT) was found according to DIN EN ISO 75 for HDT/A.

The fibre length was analysed for PPg10, PPg40 and PPg70 in order to investigate how the fibre content and, as a result, the increase in viscosity, affect the fibre length reduction during compounding and injection moulding process. The fibre length was determined for the tensile test specimens produced. Test specimen sections were ashed, and small sample quantities of it were separated on specimen slides in water. A count was performed for each of the two sample quantities under a digital microscope with image-supported length measurement. All of the fibres within a section of the image were counted. Altogether, there were at least 500 fibres per sample.

Viscosity was measured by means of a high pressure capillary rheometer and a disk rheometer in the injection moulding relevant shear rate range from 10 to more than 10,000 1/s for the unmodified PP, unfilled, as well as up to a fibre content of 60 weight-%. The measurements were executed, analogously to the measured value specifications of the melt flow index, at 230 °C. Zero shear viscosity and viscosity at small shear rates were determined by plate rheometer for the unfilled polypropylene by means of a frequency test. The complex viscosity thereby found was converted by means of the Cox/Merz relationship into a steady state shear viscosity. Furthermore, the storage and loss modules were measured.

3. Results and Discussion

3.1 Rheological Properties

Figure 1 illustrates the non-Newtonian behaviour of the unfilled polypropylene. The dynamic viscosity is characterized by a pronounced shear thinning. The transition range from the quasi-Newtonian range into the non-Newtonian one amounts to $\dot{\gamma} = 10/s$ and zero shear viscosity $\eta_0 = 101$ Pa·s. The loss and storage modules measured by the plate rheometer show a pronounced viscous behaviour up to the shear rate range relevant for injection moulding. It was impossible to determine the point of intersection of loss and storage modules in the frequency range to be measured; it is situated at shear rates > 300 Pa·s.

The increase in viscosity due to the fibre load was determined for the PP using the capillary rheometer. Figure 2 elucidates the viscosity curves found as a function of the shear rate and the fibre content. The typical shift of the master curve along the viscosity axis as a function of the fibre content can be seen very clearly. However, only in the unfilled PP and PP10 the curves measured intersect in the range of shear rates > 100 1/s. This is due to measuring inaccuracies in the capillary rheometer. A slight shift towards lower shear rates and an enlargement of the transition range from the quasi-Newtonian to non-Newtonian flow properties can be ascertained. Viscosity increases at maximal fibre load in comparison with the unfilled polypropylene by a factor of 10. The excellent ease of flow of the PP is maintained even with high fibre content and is still clearly below the viscosity values of injection moulding types with a MFI < 10.

In comparison with the unmodified PP, hardly any change in viscosity is to be expected for the PPg since both have a similar viscosity level, and the modifier has only been added at 2 weight-%. As the literature indicates, for fibre filled polypropylene with MAH modification, slight increases in viscosity in comparison with unmodified polypropylene are also seen in the melted state due to the enhanced fibre-matrix bonding and in the neat PP due to the cross-linking between the polymer chains caused by the MAH. However, the distinctions, as they are typical in

injection moulding processes are almost unobservable at higher shear rates (> 100 1/s) (Romero-Guzmán, Romo-Uribe, González & Cruz-Silva, 2008), (Abacha & Fellahi, 2005).
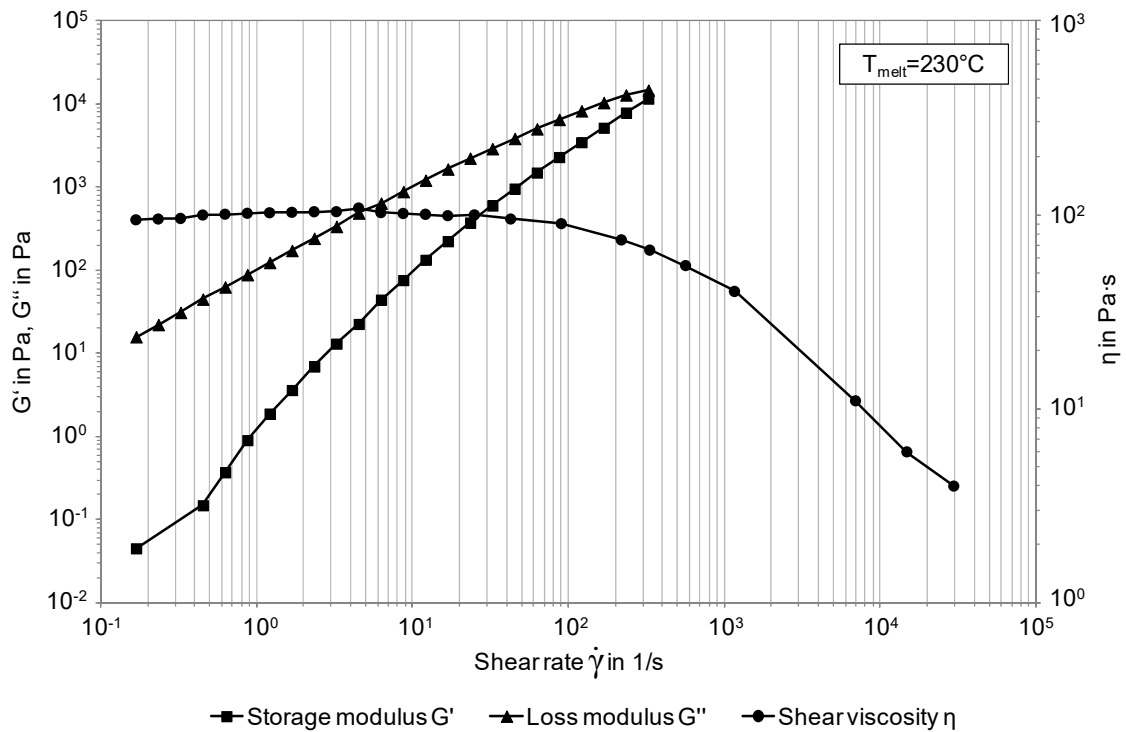

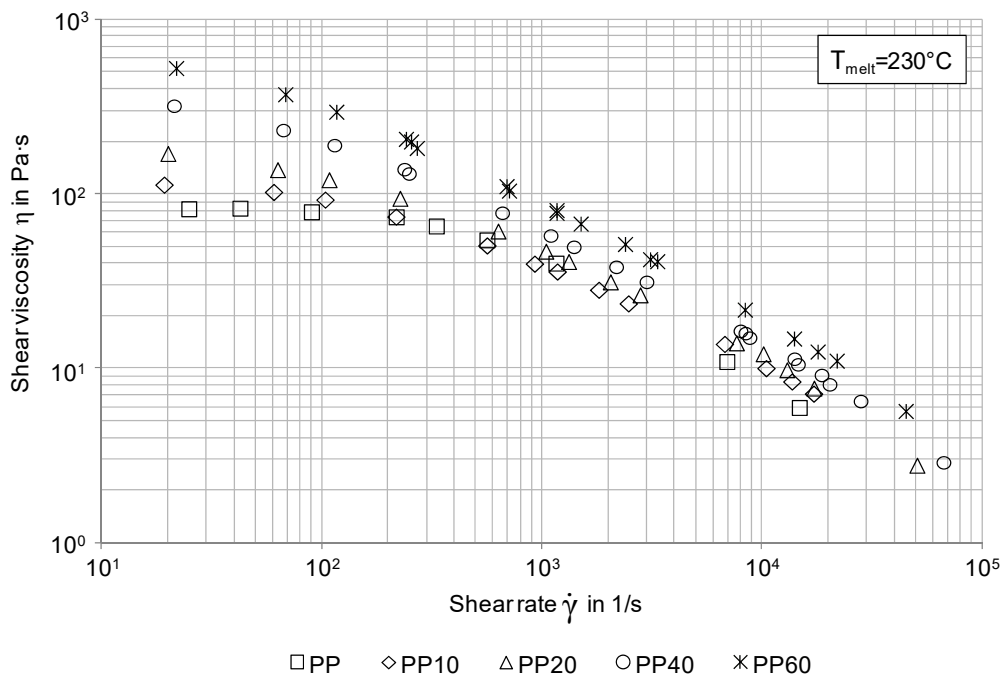Figure 1. Storage modulus, loss modulus and shear viscosity for the unfilled PP

Figure 2. Shear viscosity for PP with different content of short glass fibre in weight-%

3.2 Mechanical Properties

We first explored how the fibre content without additional modification of the polymer by MAH affects the mechanical parameters. Table 1 sums up the mechanical parameters found. The neat PP shows typical values of strength and stiffness for injection moulding grades. However, the Charpy impact properties are much lower than for common PP types due to the slow molecular weight and narrow molecular weight distribution, which enables the high fluidity. A significant change in the mechanical parameters can be detected even at low fibre content values of 2 weight-%. Elongation at break and impact strength (notched and unnotched) strongly decrease, while tensile modulus increases by 50% even at this stage. Tensile modulus becomes almost linear with a further increase in the fibre content, and a decrease can only be observed starting at 65 weight-%. Tensile strength also increases as a function of fibre content; however, its maximum has already been achieved at 40 weight-%. As expected, the impact strength of the notched samples is clearly less than the parameters of the unnotched samples, whereby the fibre content results in a clearly different curve of the parameter values.

Table 1. Mechanical properties of the PP at different fibre content

Fibre content in weight-%	0	2	5	10	20	30	40	50	60	65
Tensile modulus in MPa	1,237	1,900	2,390	2,900	4,840	6,660	8,400	10,100	10,100	9,380
Tensile strength in MPa	27.5	32.7	37.7	43.6	58.0	61.9	62.9	61.2	46.2	39.7
Elongation at break in %	>50	14.50	5.62	3.55	2.01	1.52	1.22	1.01	0.87	0.78
Charpy unnotched in kJ/m²	87.4	28.0	22.2	20.2	16.9	13.3	10.9	9.8	7.4	6.6
Charpy notched in kJ/m²	1.97	1.46	1.78	2.23	3.23	2.97	3.25	3.29	3.12	0.65
HDT in °C	57,4	57.0	70.3	86.5	127.3	--	137.3	--	--	127.5

Figure 3 brings together all mechanical properties, normalised to 100%, which corresponds to the parameter achieved maximally. Tensile strength, notched impact strength and HDT achieve a high level even beginning at 20 weight-%. Tensile modulus alone further increases at values greater than 20 weight-%. Unnotched impact strength significantly decreases even at a fibre content of 2 weight-% and diminishes further down to 10% of the unfilled plastic. Notched impact strength also decreases at first, but increases again at 10 weight-% to an amount greater than the initial level. The maximum of approximately 60% above the value of the unfilled polypropylene is achieved even with a short fibre content of 20 weight-%. Consequently, the optimum of all parameters is to be found in the range from 20 to 50 weight-%.

Adding MAH into the melt as a modifier during compounding causes all of the mechanical properties considered to increase due to enhanced fibre-matrix bonding. However, the alteration as a function of the fibre content differs. The modifier affects the mechanical properties even in the unreinforced plastic. A slight increase in stiffness and strength can be observed, and also a significant embrittlement of material, which can be particularly demonstrated with a strong decrease of unnotched Charpy impact strength (Table 2). The possible causes for this may come from the formation of intermolecular hydrogen bonds of the carboxyl groups due to the MAH functionalization. This to the end results in physical cross-linking of the polymer chains and lead to the embrittlement.

At first, no differences can be observed in terms of stiffness as a function of the fibre content. Only beginning with a fibre content of 40 weight-% does adding MAH bring out a stronger increase in the tensile modulus in comparison with the unmodified PP. Moreover, the maximum is only achieved at 65 weight-%. In comparison with unmodified PP, tensile strength is higher at low fibre contents. The differences are reinforced as a function of the increasing fibre content up to 40 weight-%. Then the tensile strength of the PPg achieves a value around 60% greater than that of the unmodified one. The strength diminishes again as a function of higher fibre content; however, the difference in strength due to the MAH remains. In elongation at break, there is almost none, while in heat distortion temperature only slight increases can be seen when using MAH. The increases in HDT can be identified consistently for all fibre contents (Figure 4).
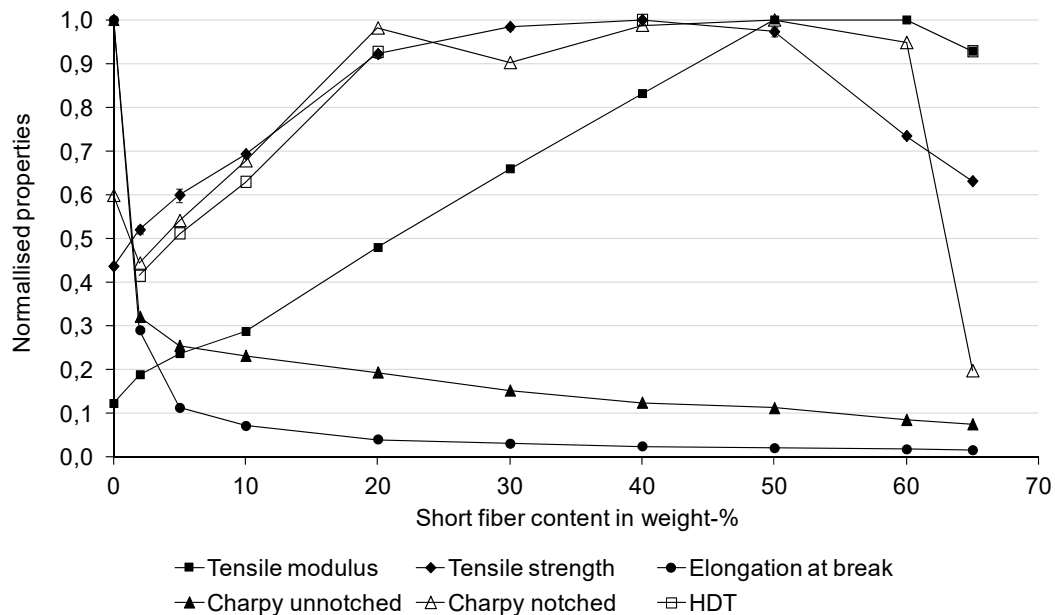
Figure 3. Normalised properties for the PP to the short fibre content

Table 2. Mechanical parameters of PPg with MAH modifier at different fibre content

Fibre content in weight-%	0	2	5	10	20	40	60	65	70
Tensile modulus in MPa	1,500	1,870	2,320	2,870	4,500	8,900	12,700	13,500	13,000
Tensile strength in MPa	31.5	34.1	40.0	48.4	65.7	101.0	91.8	76.7	60.3
Elongation at break in %	>50	16.30	7.87	5.26	3.50	2.66	1.09	0.75	0.53
Charpy unnotched in kJ/m²	22.4	29.4	22.1	23.4	35.4	46.6	25.2	13.3	9.4
Charpy notched in kJ/m²	2.16	1.61	2.00	2.98	7.80	9.75	6.47	2.89	3.01
HDT in °C	62.7	63.0	82.7	98.2	139.9	145.1	--	--	144.1

The comparison of the parameters shows that the improvement due to the bonding agent MAH becomes effective starting from a value of approximately 20 weight-%. The differences in the tensile modulus are only to be seen above very high fibre contents, such as 50 weight-%. When adding MAH and taking into account all mechanical parameters, the optimum of fibre reinforcement is slightly shifted towards higher fibre contents and is to be seen at approximately 40 weight-%.

It is noteworthy that the properties are enhanced to a lesser extent when adding PP-g-MAH at fibre contents of more than 40 weight-%. This can be seen in terms of the tensile strength and the impact strength, but not for the tensile modulus. For this reason, for the polypropylene with a glass fibre content of 30 weight-%, the ratio of the PP-g-MAH added was varied. This variation was to find out whether the decreasing difference between the unmodified and the modified polypropylene can be explained by the decreasing percentage of MSA content which is available for adhesive fibre bonding. Figure 5 elucidates the tensile strength and Charpy impact strength parameters as a function of the PP-g-MAH added in an exemplary manner. In these curves, there are hardly any alterations in the property values' level when varying the content of the PP-g-MAH added over a range from 2 to 10 weight-%. This phenomenon could also be observed for the tensile modulus and the notched Charpy impact strength and coincides with the results obtained in other studies (Fu, He & Chen, 2010). Consequently, the deterioration of the mechanical properties of the PPg above 40 weight-% has to be explained by other reasons. Since, for the tensile modulus, the parameters above 40% are also even higher than those of the unmodified polypropylene, the changes in the fibre-matrix bonding cannot be regarded as the primary reason.
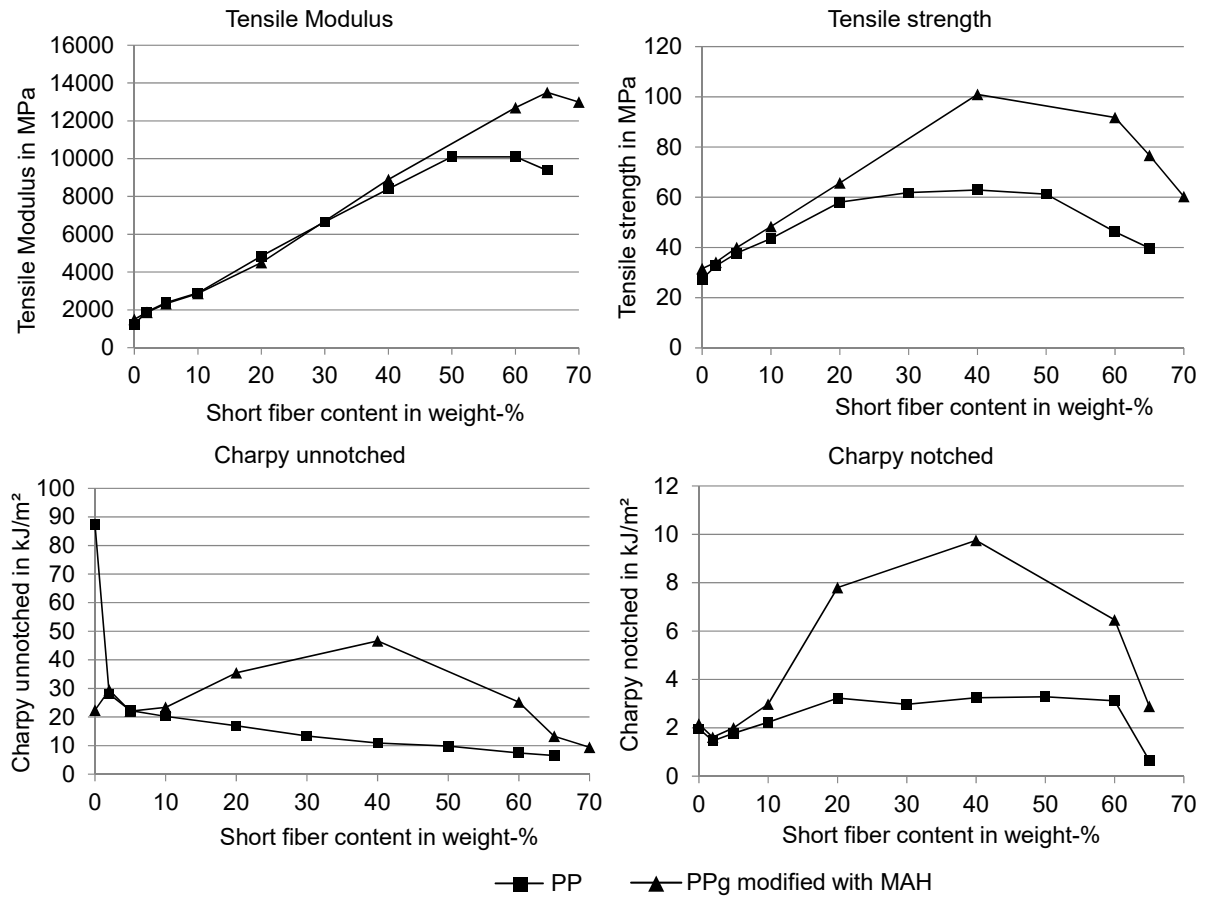
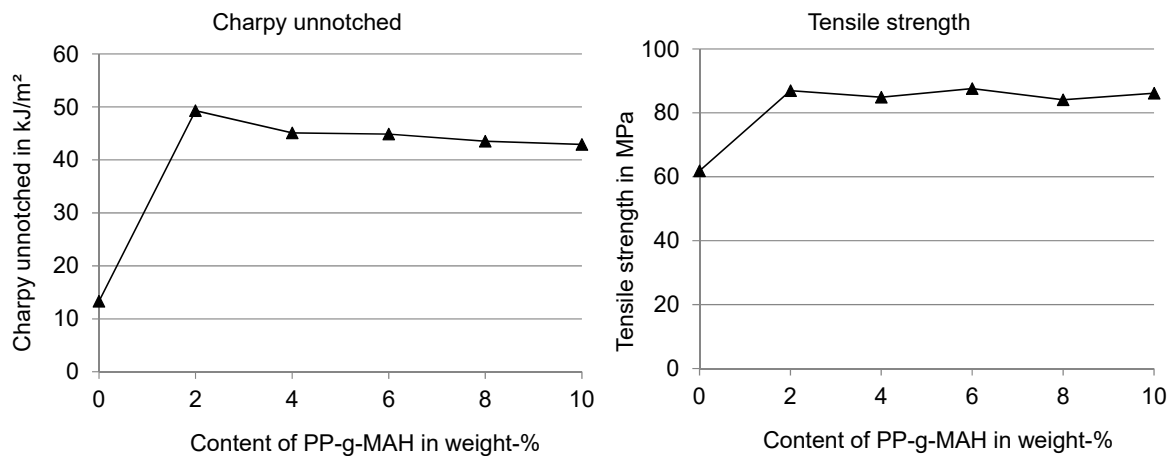Figure 4. Comparision of mechanical parameters for PP and PPg

Figure 5. PP-g-MAH content acting on the mechanical properties of PPg with 30 weight-% fibre content

The higher fibre contents lead to an increase in the melt viscosity and thus to higher shear stress during processing. As a consequence the fibre length reduces due to the fibre breakage. Figure 6 shows the measured frequency of the fibre length in a normal distribution for 10, 40 and 70 fibre weight percent. The fibre length analysis provided average fibre lengths of 0.67 mm for PP10, 0.26 mm for PP40 and 0.20 mm for PP70. As expected, fibre length diminishes as a function of fibre content, while fibre length reduction clearly weakens between PP40 and PP70.

The decreasing content of longer fibres in increasing fibre content can be clearly seen. Fibre length distribution becomes tighter and is shifted towards lower fibre contents. However, even at the fibre content of 70 weight percent the average aspect ratio of the fibres is around 12, which is a result of the use of the high fluidity polymer.

The reduction of average fibre lengths mainly results in a decrease of impact resistance. The energy absorption under impact loading results from the energy absorption potential of the elastic matrix and from the fibre reinforcement due to the introduction of the forces into the fibres, crack deflection and energy absorption by fibre pullout at break. The latter depend on the existing fibre-matrix adhesion. With increasing fibre content, the influence of the fibre-matrix adhesion weakens, since the matrix ratio for introducing forces into the fibres is reduced. Also the fibre length for crack deflection and pullout is reduced. This in combination leads to the diminishing effect of MAH modification at high fibre contents. In general, the observable deterioration of mechanical properties at high fibre contents may primarily be explained by the decreasing fraction of the volume of the matrix that is available for homogeneous force introduction into the fibres.
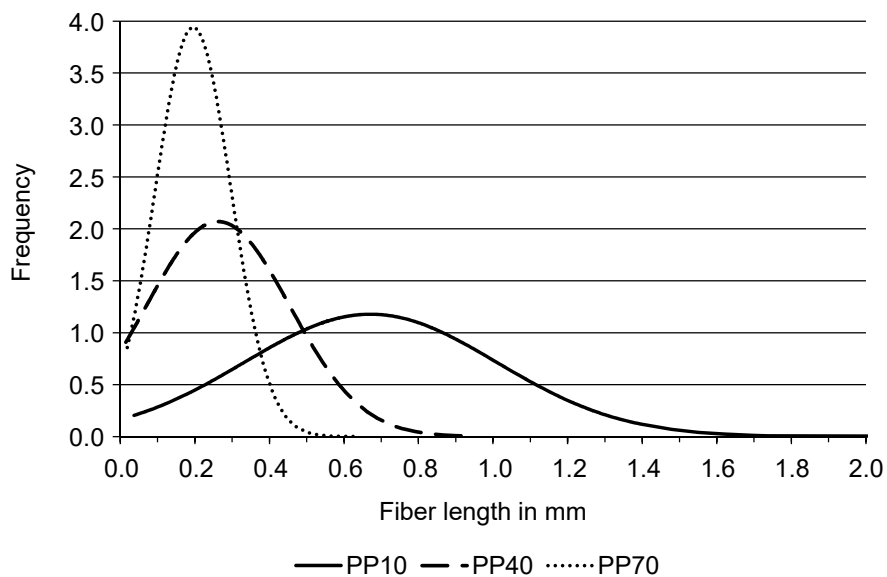

Figure 6. Fibre length distribution of the fibre reinforced PP

4. Conclusion

The reinforcement of polypropylene with short glass fibres during the injection moulding process allows a significant strengthening of the mechanical properties through technologically simple measures. The performance increase is limited to about 70 percent by mass of fiber content due to the process technologically maximum achievable added amount of chopped fibers in the compounding process. By using low-viscosity PP these high fiber contents can be achieved, but their low average molecular weight and narrow molecular mass distribution have a negative effect on the impact strength properties in unreinforced state.

High fiber contents lead in particular to the improvement of the modulus of elasticity. The impact strength and notched impact strength run contrary to the tensile modulus in case of unmodified PP, while tensile strength reaches its maximum at almost 40 percent by weight. However, by adding the MAH the impact characteristics in particular can be improved. After an abrupt decrease, the impact strength increases when adding minute fiber quantities back to 50% of the initial value at about 40 percent by weight of fiber content. The tensile strength maintains excellent characteristics up to about 60 percent by weight when a major drop can be seen.

The modification of the PP with MAH results in an optimum of mechanical properties between 40 and 50 percent by mass of glass fiber content, strength and impact resistance decreasing again thereafter. The added amount of MAH is relatively independent of the selected fiber content. Higher added amounts compared to the usual dosage of 2 percent by mass of PP-g-MAH, at a fraction of the bound MAH of 1 percent by mass, do not improve the usual properties such as strength and impact resistance.

Acknowledgments

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