TPE-MODIFICATION OF WOOD PLASTIC COMPOUNDS FOR ADVANCED RHEOLOGICAL AND IMPACT PROPERTIES

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ABSTRACT

In this study a styrenic thermoplastic elastomer (TPE) lubricant mix for enhancing impact strength and rheological processing behavior of wood fiber-reinforced polypropylene (PP/WF) will be introduced. All PP/WF composites within this investigation consist of a constant matter of 40 wt% WF in combination with 3 wt% of maleated polypropylene (PP-g-MA) and were compounded by a co-rotating twin screw extruder. Four types of dry-blended (DB) TPE-oil-mixes with different TPE/oil ratios were added in amounts of 10 and 20 wt%. The DBs affect a dramatical increase of Charpy impact strength. In addition the elastic modulus was reduced by acceptable degrees. These strong mechanical characteristics are accompanied by an improvement of rheological behavior in processing. All gathered data is analyzed in dependence of used DB composition with respect to mechanical properties and rheological flow behavior by processing parameters and practice oriented flow spiral tests.

Index Terms: polypropylene wood flour composites, impact modification, thermoplastic elastomer, SEBS, rheological behavior, processing oil, WPC

1. INTRODUCTION

The market for Wood Plastic Composites (WPC) shows a steady growth within the last decades, due to their advantages for certain applications in comparison to classical polymer and wood materials. On the one hand, WPCs surpass synthetic plastics, in case of their natural image, lower price and technical properties (higher stiffness, lower thermal expansion coefficient). On the other hand, they outperform classical wood materials regarding their unrestricted formability, moisture resistance and higher durability and longevity.[1,2] Because of these specific features, WPCs are used in Germany especially for decking boards in outdoor areas and for car interiors in the automotive industry.[3] WPC products are processed by extrusion as well as by compression and injection molding. Depending on the processing technology, the wood fiber content could vary up to 80 wt% for

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extrusion of profiles. For injection molding, a natural fiber content between 30 and 50 wt% is typical and increases up to over 90 wt% for compression molding.[4,5] Currently in the automotive industry, injection molding applications of WPCs are far behind those of compression molding. This fact is surprising taking several benefits of injection molding process into account such as high volume production, approachability of complex structures and structural lightweight (in comparison to frequently used talcum filling). WPCs possess a decisive disadvantage with respect to their impact properties which prohibits a wide range use of injection molded WPCs in automobile applications.[6]

According to K. Oksman and C. Clemons[7] there are several ways to improve impact properties of fiber-reinforced composites: 1) increase the matrix toughness; 2) optimize interface filler/matrix with compatibilizers; 3) optimize filler-related properties (content, particle-size and dispersion); 4) length-to-diameter ratio and orientation. The investigation of PP/WF from K. Oksman and C. Clemens is mainly focused on the optimization of fiber-matrix-compatibilization while increasing the polymer matrix PP as a targeted side effect. Several combinations of PP-g-MA, ethylene/propylene/diene terpolymers (EPDM) as well as maleated EPDM (EPDM-MA) and maleated SEBS (SEBS-MA) were tested (all 40 wt% WF). The top performance is reported for a composite composition containing both PP-g-MA and SEBS-MA. The trend of these results regarding enhanced impact strength are confirmed by Kim and Pal[8] for use of PP-g-MA, SEBS-MA as well as neat SEBS which was also tested. Moreover in this context the rheological properties were investigated. The reported plots show, that shear viscosity is largest for SEBS-MA, followed by SEBS and PP-g-MA. In other words the considered impact modifications worsened the flow behavior of WPC melts. Beside the referred disadvantage above with respect to impact properties, the poor processability of WPCs is an frequently mentioned topic. According to L. Haijun and L. Shiang[9] the viscosity of the melt is one of the most important characteristics to be considered when designing any industrial process for WPCs besides mechanical properties of finished component.

The approach of processability enhancement in this investigation is deduced from the general methodology of TPE processing. In case of SEBS, due to its poor processing properties, the copolymers are never used in their pure form and must be compounded with oils, fillers and other polymers. Through the addition of small amounts of PP and high amounts of processing oil is common practice. The resulting SEBS/oil/PP blends have an improved processability and stiffness and are commercialized since the early 1990s.[10-12] Transferring this methodology on WPC processing, the reported compatibility of SEBS, oil and PP offers new possibilities by changing the ratios of components to PP with modifier SEBS/oil. Against this background an investigation of PP/WF with SEBS/oil modification appears promising with respect to advanced impact and rheological properties. The addition of SEBS/oil modificator was realized as dry-blend (DB) and as basis for modification a system of PP/WF (40 wt% WF) with PP-g-MA as compatibilizing agent with 3 wt% was selected. The amount of compatibilizer was determined by earlier investigations, with a maximum for tensile strength and simultaneously no deterioration of impact properties which was caused by amounts above 3 wt%.[13]

2. MATERIALS AND METHODS

2.1 Materials

Matrix polymer was Slovnaft's PP homopolymer Tatrene HG1007 (bulk density 0.55 kg/dm³. melt flow index MFI 10 g/10min at 230 °C and 2.16 kg, ISO 1133). WF (Jeluxyl WEHO 500S) was provided by JELU-WERK Josef Ehrler GmbH & Co. KG. Rosenberg (bulk density 0.27 kg/dm³, L/D ratio 3.36). According to the manufacturer, WF was not pre-treated or modified with chemicals. Used PP-g-MA compatibilizer was SCONA TPPP 8112GA from BYK Additives&Instruments. Wesel; it had a MA-content of 1.4 wt% (bulk density 0.45-0.55 kg/dm³). Tested modificator mixes consisted of two components. Component 1 was a linear high molecular weight styrene-ethylene/butylene-styrene block copolymer (SEBS with butadien/styrenic-ratio BD/SM of 67/33) and a low molecular weight oily component 2. Total Compositions of various PP/WF composites are shown in Table 1.

Table 1 Composition of the various PP/WF composites

Sample Code	Composite composition [wt%]						
	PP	**PP-g-MA**	**WF**	**DB-1**	**DB-2**	**DB-3**	**DB-4**
0	57	3	40	untreated			
1a	47	3	40	10			
1b	37	3	40	20			
2a	47	3	40		10		
2b	37	3	40		20		
3a	47	3	40			10	
3b	37	3	40			20	
4a	47	3	40				10
4b	37	3	40				20

2.2 Processing

At first Component 1 (SEBS) and 2 (low molecular weight oil) were dry blended before the compounding process in compositions of component-ratios 1 to 2 of 90/10, 75/25, 65/35 and 45/55. Used DB compositions are shown in Table 2. Then all WPC samples were compounded by a co-rotating twin screw extruder (Noris Plastics ZSC 25/40D. screw diameter 32 mm, barrel temperature 180°C, screw speed 200 rpm, material output of 10 kg/h) equipped with atmospheric and vacuum degassing. A self -developed screw configuration for fiber-reinforced composites was used (screw-development during project FENAFA[1]). The degassing extruder section removes effectively the residual moisture, therefore no pre-drying step was implemented before compounding. Such approach meets best industrial conditions where no pre-drying step is performed for wood flour, due to time and energy efficiency. The extruded strands were cooled in a water slide system and pelletized. Pelletized WPC samples were then dried 4 hours at 80°C in a dry-air-dryer (Werner Koch Maschinentechnik GmbH).

[1] „Ganzheitliche Bereitstellungs-, Verarbeitungs- und Fertigungsstrategien von Naturfaserrohstoffen“ funded by Fachagentur für nachwachsende Rohstoff e.V. (FNR)

Injection mold processing was performed on an ARBURG Allrounder 370A/600-170 (processing temperature profile 185-205°C and mold temperature 40°C) into standard test specimens DIN EN ISO 3167 type A.

2.3 Mechanical testing

Tensile testing of specimens was performed according to DIN EN ISO 527 on a Zwick/Roell Z010 TN ProLine material test machine. Crosshead speed was 2 mm/min for E-modulus and 10 mm/min for tensile strength and elongation at break. 10 test specimens of each composition were tested.

Impact testing was performed using a CEAST Resil Impactor according to DIN EN ISO 179 Charpy impact method. The unnotched impact energies were determined for 10 test specimens of each composition at room temperatures and at -20°C.

2.4 Rheological testing

The melt flow rate MFR was determined on testing machine MeltFlow@on plus from KARG. Conditions for the compound were according to DIN EN ISO 1133-1 at a temperature of 190°C and a mass of 21.6kg by multiple determinations (at least three repeat measurements).

The flow spiral tests were performed on an ARBURG Allrounder 370A/600-170 at a processing temperature of 190°C with an flow spiral AIM Test Mould System from Axxicon, Specimen dimensions: 1150 x 5 x 3 mm) at a mold temperature of 40°C. The spiral length was determined for at least 5 test specimens on molding pressures of 300, 900, 1200 and 1800 bar.

3. RESULTS AND DISCUSSION

3.1 Characterization DB

The dry-blends were characterized by size exclusion chromatography (SEC). Results of SEC measurements are shown in Table 2. The results are put in context to theoretically approach of composition. For all further calculations and figures the actual composition determined by SEC were used.

Table 2 Compositions of dry-blends*

	SEBS			Oily component		
	theoretical	practical*		theoretical	practical*	
		Area	M_w		Area	M_w
Sample Code	**[%]**	**[%]**	**[10^3 g/mol]**	**[%]**	**[%]**	**[10^3 g/mol]**
DB-1	90	89	260	10	11	0.4
DB-2	75	71		25	29	
DB-3	65	66		35	34	
DB-4	45	47		55	53	

*) SEC determination (eluent: THF, flow rate: 1.00 ml/min, column Set: 3x PL gel mixed B 900mm x 7.5mm, detector: DRI (differential refractometer), temp.: 40°C)

3.2 Mechanical Properties

The mean values and standard deviation of the mechanical properties of PP/WF samples with different content of DB are summarized in Table 3. These results are presented in separate figures to ease an interpretation and point out effects of single component variations.

Table 3 Mechanical properties of PP/WF samples (± values are standard deviations)

	Tensile Properties			Charpy Impact Properties	
	Strength	**E-Modulus**	**Elongation at break**	**Unnotched - at RT***	**Unnotched - at -20°C**
Sample Code	**[MPa]**	**[GPa]**	**[%]**	**[kJ/m²]**	**[kJ/m²]**
0	39.2 ± 0.3	4.3 ± 0.06	2.5 ± 0.2	13.0 ± 1.2	10.1 ± 1.1
1a	32.0 ± 0.3	3.3 ± 0.04	4.8 ± 0.3	18.3 ± 1.0	14.4 ± 1.0
1b	24.6 ± 0.1	2.3 ± 0.02	11.3 ± 0.6	28.9 ± 1.7	23.4 ± 2.1
2a	32.4 ± 0.2	2.9 ± 0.01	5.6 ± 0.3	23.0 ± 1.7	17.8 ± 1.4
2b	26.2 ± 0.1	2.4 ± 0.02	7.6 ± 0.4	26.4 ± 2.2	21.2 ± 2.1
3a	32.4 ± 0.1	2.9 ± 0.03	5.3 ± 0.3	22.6 ± 2.3	17.7 ± 1.3
3b	21.3 ± 0.1	1.8 ± 0.02	9.5 ± 0.9	29.8 ± 3.0	26.1 ± 2.3
4a	30.7 ± 0.1	2.6 ± 0.03	5.5 ± 0.2	22.9 ± 2.1	18.5 ± 1.8
4b	20.4 ± 0.2	1.6 ± 0.02	9.1 ± 1.4	26.2 ± 1.4	23.4 ± 2.4

*) room temperature

Figure 1 provides an overview of percentage changes of mechanical properties relating to the unmodified test sample 0 for test series a) (with 10 wt% DB) as Figure 2 for test series b) (with 20 wt% DB). Whereas the numbers of test samples represent the different DBs used (cf. Table 2). The values of standard deviation were included and interpreted in that way, that bars show the absolute minimum of percentage changes in case of positive and the absolute maximum in case of negative percentage changes for all values according to Table 3. All samples in Figure 1and Figure 2 show in general a decline of tensile strength and E-Modulus and an increase of stiffness and impact strength. The addition of 10 wt% DB affects the percentage changes of mechanical properties less than the addition of 20 wt% DB.

A maximum of overall mechanical properties with 10 wt% DB (cf. Figure 1) was determined for sample 2a with an increase of Charpy impact strength at room temperature by 50% and of elongation at break by 98% along with a decrease of tensile strength by 18% and E-Modulus of 34%. A minimum of properties was determined for sample 1a (with the highest content of SEBS within this series), while Charpy impact strength was improved by 22% and tensile strength was decreased by 20%. Taking all results within test series a) in evaluation there are some remarkable developments. The reduction of tensile strength remains almost constant at very low level until sample 3a, regardless of variation of SEBS/oil ratio (reduction from 8.1 to 1.9 in total composition) only when reducing the SEBS/oil ratio to 0.89 tensile strength went down from 18 to 23% . But in the same range from 1a to 3a there is a distinguishable change in elongation at break (from 65 to 98%) and Charpy impact (from 22 to 50%) behavior. For further reduction of SEBS/oil content these properties remain at this high level while tensile strength decreases.

In Figure 2 the maximum of overall properties was sample 1b with an increase of 296% for elongation at break and 92% for Charpy impact strength while E-modulus is reduced by 49% and tensile strength by 38%. In

contrast to test series a), the test sample 1b has the highest content of SEBS (17.8 wt% in total composition) is the best performing sample related to overall properties (cf. test series a), test sample with highest SEBS content shows poorest mechanical properties). Regarding the effect of SEBS/oil ratio on mechanical properties in test series b) the reduction of tensile strength can be observed between samples 2b and 3b (SEBS/oil ratios of 2.4 to 1.9) within a quite close range. In test series b) remarked trends for E-Modulus, impact strength and elongation at break are less distinctive but still identifiable.

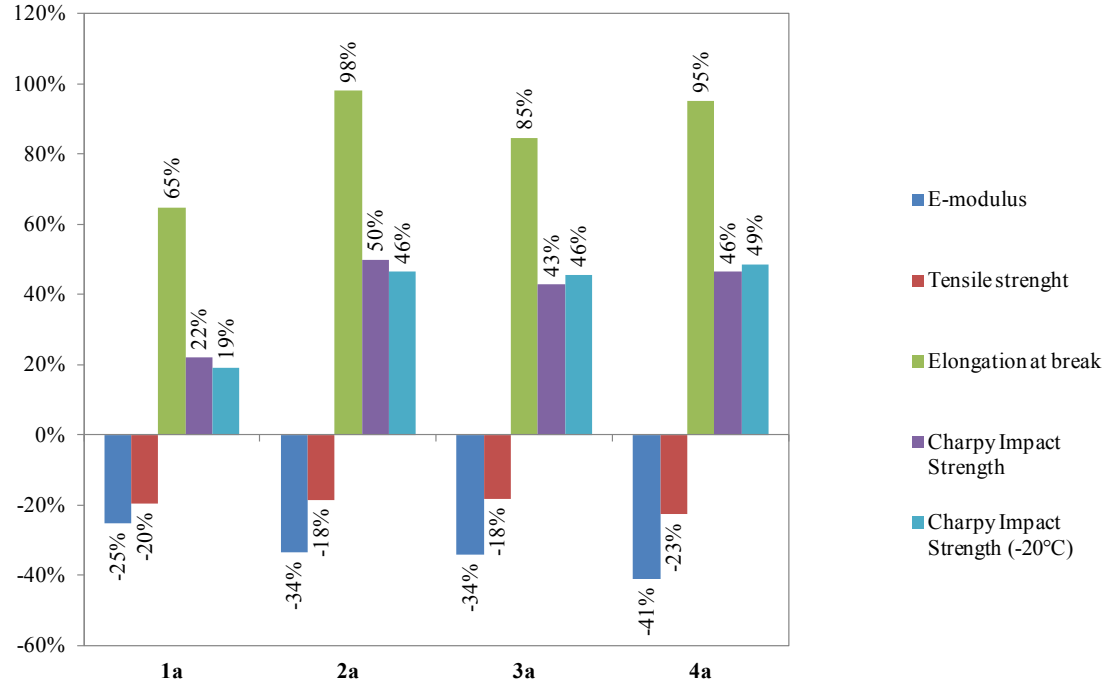

Figure 1: Percentage changes of mechanical properties for addition of 10 wt% DB related to unmodified sample

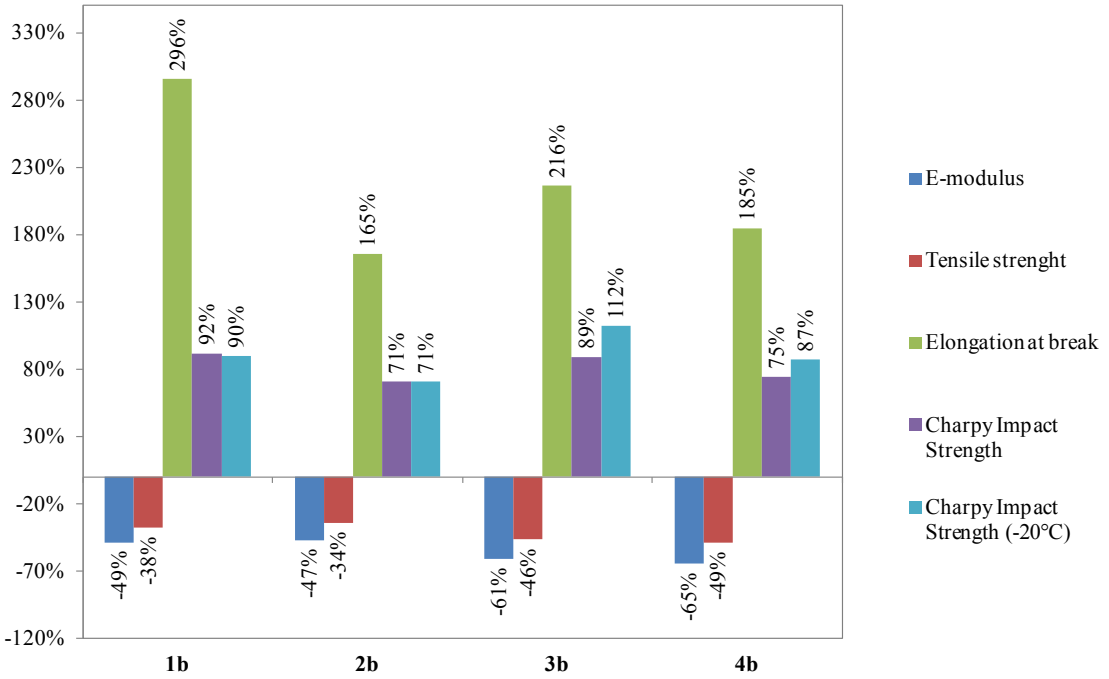

Figure 2: Percentage changes of mechanical properties for addition of 20 wt% DB related to unmodified sample

A competitive evaluation of maxima from test series a) (sample 2a; 7.1 wt% SEBS; 2.9 wt% oil) and test series b) (sample 1b; .17.8 wt% SEBS; 2.2 wt% oil) is delivering following noticeable facts. A factor of 2.4 regarding the wt% of SEBS in total composition increases elongation at break by a factor of 3.0 (from 98% to 296%), Charpy impact strength by a factor of 1.8 (from 50% to 92%), while decreasing tensile strength by a factor of 2.1 (from -18% to -38%) and E-modulus by a factor of 1.4 (from -34% to -49%).

To check the apparent effects of variation of SEBS and oil content in total composition and to discuss the results basing on a different perspective than the SEBS/oil ratio. The mechanical properties are interpreted regarding changing SEBS content (by constant oil-content) and changing oil-content (by constant SEBS content) as follows. Figure 3 shows the total composite composition of test samples 2b and 4a. The oil contents remain almost constant for test sample 2b with 5.8 wt% and 4a with 5.3 wt%, whereas the SEBS content in total composition varies from 4.7 wt% for test sample 4a to 14.2 wt% for test sample 2b. The trends were determined for mechanical properties E-modulus, elongation at break, tensile and Charpy impact strength related to the mean values, but standard deviations are included as well. Tensile strength shows a falling tendency for higher amounts of SEBS. E-modulus is almost unaffected and the elongation at break behavior shows a rising tendency. The trends of Charpy impact strength are difficult to determine because the standard deviation areas are overlapping. For the mean values impact behavior shows a rising tendency. At the background of literature this fact is partially unexpected. According to Kim and Pal[8] higher SEBS contents are related with negative effects on tensile strength and E-modulus and positive effects on impact strength. The determined results within this study confirm the decrease of tensile strength related to higher SEBS contents but the expected increase in Charpy impact strength just in terms.

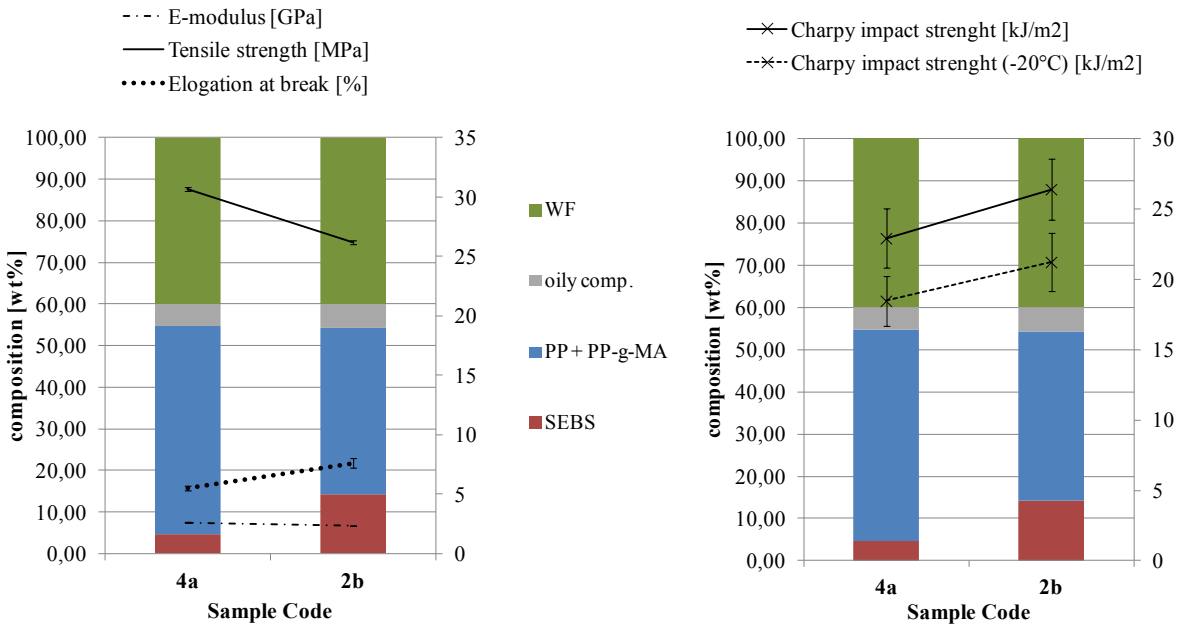

Figure 3: Change of tensile and impact properties related to SEBS content exemplified by comparison of sample 2b (SEBS 14.2 wt%; oil 5.8 wt%) and sample 4a (SEBS 4,7 wt%; oil 5.3 wt%)

Figure 4 shows the total composite composition of test samples 1a and 4b. The SEBS content remains almost constant for test sample 1a with 8.9 wt% and 4b with 9.4 wt%, whereas the oil contents in total composition

varies from 1.1 wt% for test sample 1a to 10.6 wt% for test sample 4b. The trends were determined for mechanical properties E-modulus, elongation at break, tensile and Charpy impact strength related to the mean values, but are standard deviations are included as well. Tensile strength and E-modulus show a falling tendency for higher amounts of oil. The elongation at break and Charpy impact behavior shows a rising tendency (also including the standard deviation range). Taking the increase of impact strength by constant matter of SEBS while rising oil content into account one possible interpretation is provided by G. Holden.[12] The high compatibility of the system PP/SEBS/oil could lead to a better distribution of the SEBS phase within the PP matrix which rises the elastomeric impact on overall mechanical properties according to the enlargement of surface area.[10] Clarification of the observed effect could be provided in further morphological characterizations.

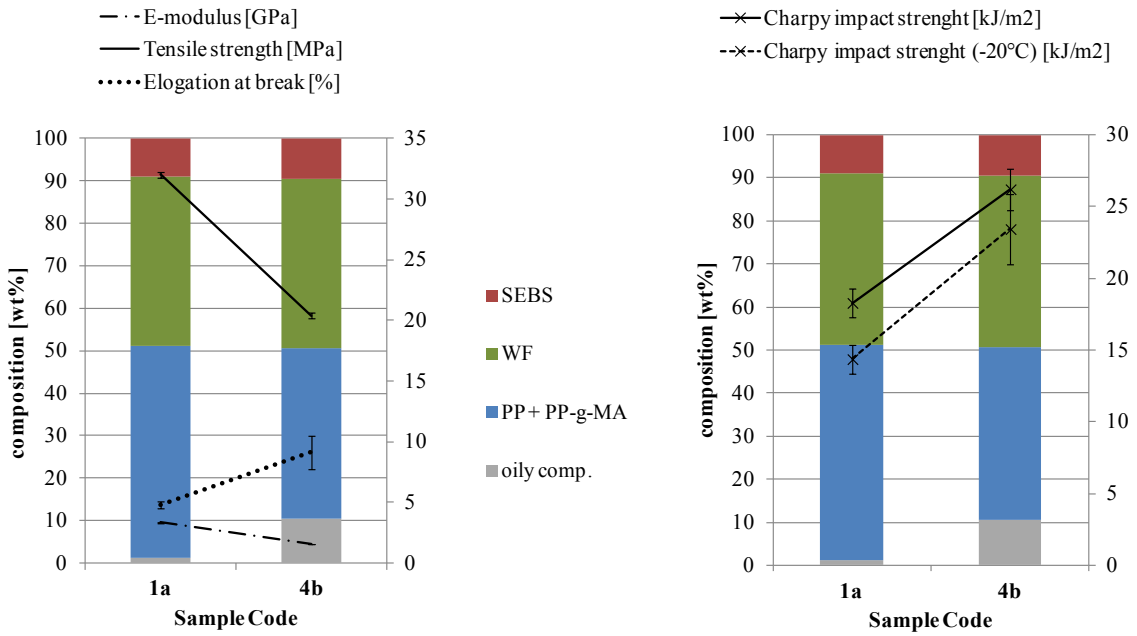

Figure 4: Change of tensile and impact properties related to oily content exemplified by comparison of sample 1a (oil 1.1 wt%; SEBS 8.9 wt%) and sample 4b (oil 10.6 wt%; SEBS 9.4 wt%;)

3.3 Rheological Properties

Conclusions on the rheological behavior during the compounding process of the PP/WF were provided by temperature and pressure sensors within the compounder. Processing parameters of rotary current, the processing melt temperature and pressure (both directly at the nozzle) were monitored.

For discussing values of MFR measurements of WPCs there exist different stances. Experts like Geißle[14] reported about the deficits of MFR measurement with respect to test equipment, measurement process and underlying flow function, which finally lead to higher calculated shear viscosities from MFR then the apparent viscosity. Hansmann and Laufer[15] confirmed this result, but mentioned that the deviation is very small and reported that MFR measurements especially for WPC melts show a very good correlation with measurements by an extrusion slit capillary rheometer.

Without a doubt MFR is the most important parameter for describing the flow behavior of a plastic melt for the plastics processing industry and quality assurance. Therefore within this investigation the MFR value is

discussed as an one-point measurement of shear viscosity by an indirect proportional correlation. All samples were tested at the same testing machine under equal conditions by one single person and the results will be discussed only relatively to each other and only in combination with additional data like melt pressure of compounding process and the results of flow spiral tests.

Table 4 shows the processing parameters recorded under stable processing conditions at constant screw speed (200 rpm). The observed values show an overall down trend regarding rotary current, melt pressure and temperature for both test series (with 10 wt% DB in part A and 20 wt% DB in part B) according to higher oil-contents from samples 0 to 4. The observed decrease of melt temperature indicates an reduction of frictional heat within the compounding process.

Being more specific, related to the value of test sample 0, the samples 1a and 2b show global maxima for the melt pressure. This can be considered as an effect of high SEBS and low oil-contents of these samples. That effect was expected and is reported in literature[8] as well. In addition the determined results of MFR measurements of the test series $b_{)}$ (with 20% DB) are shown in Table 4 part B. The observed trends for test series $b_{)}$ are accompanied by the results of MFR measurements which show an indirect proportional trend and confirm conclusions of the discussion of processing parameters which is emphasized in Figure 5.

Table 4: Processing parameters during compounding and MFR results of test series $b_{)}$

A)

Sample Code	Compounding parameter		
	Rotary current [A]	Melt Temp [°C]	Melt Pressure [bar]
0	30.1 - 31.6	181	22.2 - 23.6
1a	27.1 - 30.4	182	24.2 - 26.9
2a	25.0 - 26.1	180	21.7 - 23.2
3a	24.0 - 25.6	178	18.4 - 20.4
4a	20.9 - 22.4	177	17.3 - 19.2

B)

Sample Code	Compounding parameter			MFR
	Rotary current [A]	Melt Temp [°C]	Melt Pressure [bar]	[g/10min]
0	30.1 - 31.6	181	22.2 - 23.6	120.3
1b	26.8 - 29.0	182	24.2 - 26.9	55.3
2b	21.7 - 23.8	179	25.7 - 28.2	117.4
3b	21.2 - 22.3	177	18.8 - 19.9	158.5
4b	17.5 - 19.0	175	14.7 - 15.5	459.2

In Figure 5 the results of MFR measurements are shown in order of size (smallest to largest) in context of composite composition. *S*ample 1b with the smallest value of MFR has the highest content of SEBS and just a small oily content and lies even beyond sample 0 (without modification). So the high SEBS-content worsens the melt flow behavior regarding to untreated sample 0. In case of sample 2b and 3b, there is shown the effect of the oily component, which reduces the weaken effect of SEBS on the flow behavior. In sample 4b the effect of the oily component exceeds completely the TPE effect.

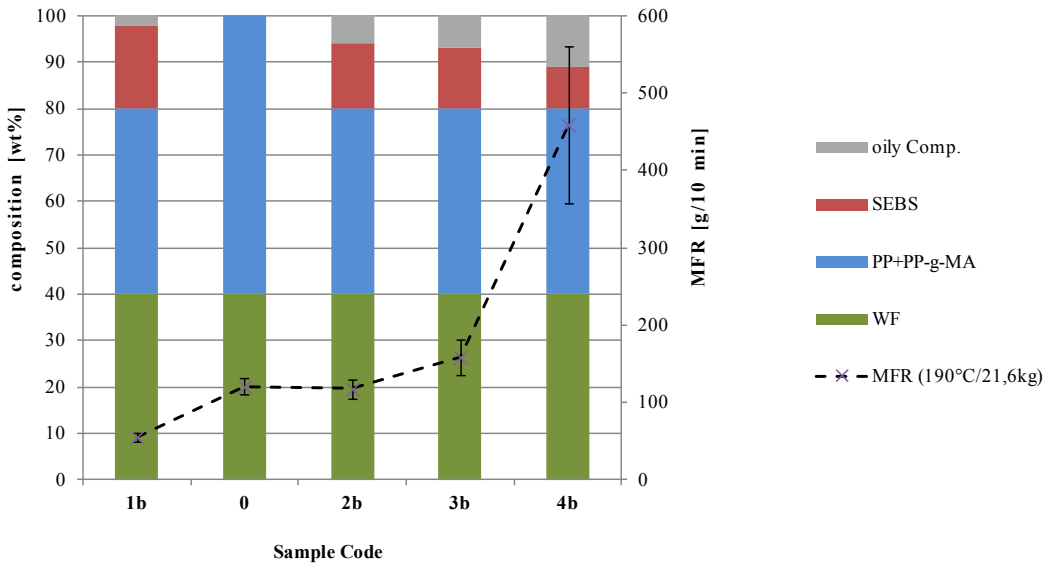

Figure 5: MFR related to different composite compositions

In addition to the above discussed flow behavior during compounding, practice-oriented flow spiral tests were performed for conclusions on the flow behavior regarding injection molding process. These measurements of spiral flow offers a comparative analysis of a material's ability to fill a part with a constant injection pressure. The spiral flow test is performed by injecting a material into a spiral mold. The distance the material travels in the spiral mold is measured in cm and is an indication of the flow behavior of the melt. The results of the test-series $b_{)}$ (20% DB) are shown in Table *5*. The spiral length was determined on injection pressures of 300, 900, 1200 and 1800 bar and put in context to the obtained MFR results. The trends of flow spiral length regarding decreasing SEBS and increasing oily contents confirm conclusions of the discussion of the rheological properties.

Table 5 Flow spiral tests with injection molding conditions (temperature 190°C) and MFR results

	Length of the flow spiral [cm]				MFR
Sample Code	**300 bar**	**900 bar**	**1200 bar**	**1800 bar**	**[g/10min]**
0	14.6	32.3	37.3	55.4	120.3
1b	16.8	38.7	40.7	53.3	55.3
2b	17.0	40.7	49.7	68.5	117.4
3b	12.4	43.7	58.1	79.8	158.5
4b	25.5	62.4	78.2	103.5	459.2

4. CONCLUSION

The objective of this study was to investigate the effects of different SEBS/oil DB on the mechanical properties of PP-g-MA compatibilized PP/WF. The DBs were added in amounts of 10 and 20 wt%. The top performing test sample within test series a) was observed with 10 wt% DB-2. An increase of Charpy impact strength at room temperature of 50% and elongation at break by 98% was observed along with a decrease of tensile strength by 18% and E-modulus by 34%. Top performing test sample of test series b) with 20 wt% DB-1 shows an 92% improvement regarding Charpy impact strength and 296% for elongation at break and a decrease of tensile strength of 38% as E-modulus of 49% but also the worst result related to flow behavior.

Analysis regarding SEBS/oil ratio for test series a) shows a range from 8.1 to 1.9 where tensile strength and E-modulus reduction remains at low levels in combination with oscillating values for impact strength and elongation at break behavior. For test sample series b) these range was quite closer. The point of inversion of this range for sample series b) could be defined more precisely between 2.4 and 1.9, whereas this point for test sample series a) lies between 1.9 and 0.89. Results regarding influences of rising SEBS or oil contents in total composition delivers also interesting results. The investigation shows that the increase of SEBS by a factor of 3 with constant oil content could not affect the impact properties. In contrast to that, varying the oil content by a factor of 9 with constant SEBS contents show an increase of Charpy impact strength. Especially the last observation raises the question for further investigations with respect to distributional effects within the system PP/SEBS/oil by morphological characterization.

The rheological characterization showed that there is a negative effect of SEBS on the relative flow behavior of PP/WF compound in case of high SEBS/oil ratios. This relative effect could be reversed dealing with slightly higher oil contents. The relative flow behavior within compounding and injection molding could be improved related to the measured data. However, these relative trends and results have to be confirmed by additional characterization methods as capillary rheometry, plastographic monitoring or extrusion slit capillary rheometer measurements.

5. FIGURES AND TABLES

5.1 List of Figures

5.2 List of Tables

6. REFERNCES

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