

## Processing of Thermal Sensitive Materials – a Case Study for Wood Plastic Composites

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**Summary.** The influence of processing parameters, *e.g.* throughput and temperature, of the compounding step of wood plastic composites was investigated in this work. The effects on the mechanical properties as well as the influence on the colour of the produced composites were correlated with the different processing parameters. The composites showed a wide range of good processability, only at high temperature and low throughput strong darkening occurred, as well as a loss of tensile strength was found for these samples.

**Keywords.** Melt processing; Compounding; Thermal degradation; Wood plastic composites; Mechanical properties.

### Introduction

Wood plastic composites (WPC) are composite materials from wood and thermoplastically processable matrices, which drew increasing attention over the last years. While the main research efforts were undertaken in the field of extrusion applications [1, 2], where wood contents of over 50 wt% were realized, also injection moulding should be considered for these materials, due to the possibility of getting tailor-made materials.

The main advantages of using wood instead of inorganic materials like talcum or glass fibres are the density of the composite, which is considerably lower and therefore of interest for transportation application, as well as the renewability and enhanced recyclability of the WPC [3].

A main issue for these materials is the processing itself. The wood particles are compounded into the molten polymer matrix by means of an extruder or kneader, at elevated temperatures. Because wood particles are sensitive in terms of temperature, and show degradation at about 200°C, the choice of the polymer itself is limited to types with processing temperatures around this point. Due to this circumstance WPCs are mainly based on polypropylene, polyethylene, and polyvinyl chloride [4], although the share of these polymers depends strongly on the geographical region where the composites are produced and applied.

Because the influences of the processing parameters on the mechanical, optical, and rheological properties have not been investigated and published for injection moulding grade composites so far, this work deals with these topics to point out the critical issues for getting good quality and satisfactory surface of the wood plastic composites.

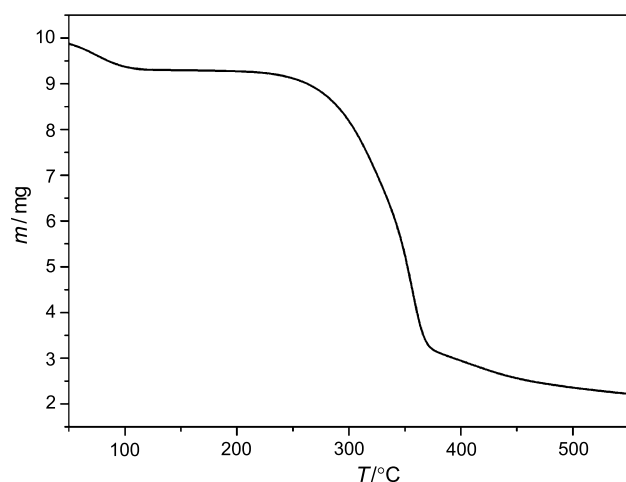
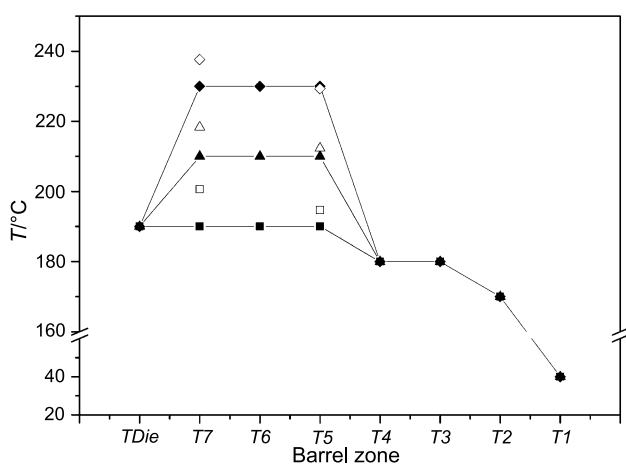
### Results and Discussion

The composites produced for this work as well as the results from mechanical testing are listed in Table 1. While throughput and barrel temperature were altered, each at three different levels, screw speed was held constant. Due to the different throughputs this results into different specific throughput, which is calculated as the ratio between throughput and screw speed, and this data was found to be a quality measure for extruded WPC profiles [5].

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**Table 1.** Process parameters (barrel set temperature  $T$  and throughput  $m$ ) for composite production and data of mechanical testing (elastic modulus  $E$ , tensile strength  $\sigma_{\text{Max}}$ , unnotched Charpy impact strength  $a_{\text{Cue}}$  with according standard deviations  $s$ )

No.	$T/^\circ\text{C}$	$m/\text{kg h}^{-1}$	$E/\text{MPa}$	$s/\text{MPa}$	$\sigma_{\text{Max}}/\text{MPa}$	$s/\text{MPa}$	$a_{\text{Cue}}/\text{kJ m}^{-2}$	$s/\text{kJ m}^{-2}$
1	190	8	3353	15	29.41	0.09	10.93	1.05
2	190	12	3402	36	29.59	0.23	11.23	1.44
3	190	16	3343	16	29.58	0.15	12.23	1.23
4	210	8	3436	23	29.28	0.11	10.31	0.79
5	210	12	3399	26	29.27	0.09	11.07	1.44
6	210	16	3394	32	29.33	0.13	11.51	0.68
7	230	8	3464	10	29.05	0.07	11.08	1.18
8	230	12	3446	31	29.40	0.09	11.46	1.69
9	230	16	3431	22	29.47	0.11	12.02	1.89

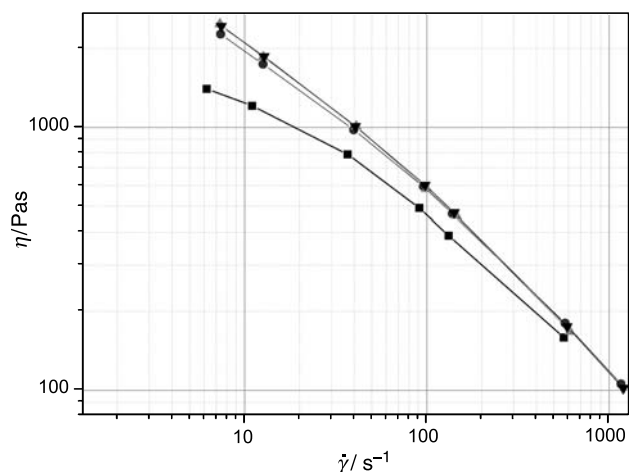
**Fig. 1.** Thermogravimetric analysis of wood particles ( $\text{N}_2$ , heating rate  $5 \text{ K min}^{-1}$ )**Fig. 2.** Actual ( $T_{\text{act}}$ ) and set ( $T_{\text{set}}$ ) temperatures while compounding for the different barrel zones of the compounder (wood particles are incorporated into the melt at zone  $T_4$ ); ■ –  $T_{\text{set}}$   $190^\circ\text{C}$ , □ –  $T_{\text{act}}$   $190^\circ\text{C}$ , ▲ –  $T_{\text{set}}$   $210^\circ\text{C}$ , △ –  $T_{\text{act}}$   $210^\circ\text{C}$ , ◆ –  $T_{\text{set}}$   $230^\circ\text{C}$ , ◇ –  $T_{\text{act}}$   $230^\circ\text{C}$ 

Thermal degradation of the wood particles starts at about  $200\text{--}220^\circ\text{C}$  under nitrogen atmosphere (Fig. 1) and increases rapidly for temperatures above  $250^\circ\text{C}$ . This atmosphere was chosen because while compounding the wood into the polymer melt air is mainly excluded.

A closer look at the actual temperature at the barrel shows that the temperature is increasing from zone 5 to zone 7 up to  $10^\circ\text{C}$  higher than the temperatures were set (Fig. 2). This is due to the energy input by the kneading and mixing elements. Further, the thermocouples in the barrel are measuring the temperature near the surface, and taking into account the low thermal conductivity of polymers in general, this leads to the conclusion that the temperature in the bulk can be considerably higher. From this point of view it is explainable why the composites are getting darker as shown with the lightness from the colour measurements (Table 2). Even the ones which were produced at  $210^\circ\text{C}$  set temperature, where degradation should not happen, as we could see from the thermogravimetric analysis (Fig. 1), show sig-

**Table 2.** Colour measurements (Lab-sphere), results from fibre length scans (length weighted length  $L_{1w}$  and width  $W_{1w}$  of the wood particles), calculated aspect ratio ( $a_r$ ), and interfacial shear strength ( $\tau_c$ ) for the different composites

No.	$L$	$a$	$b$	$L_{1w}/\mu\text{m}$	$W_{1w}/\mu\text{m}$	$a_r$	$\tau_c/\text{MPa}$
1	62.0	7.2	24.5	420	190	2.20	3.13
2	63.1	7.4	24.9	519	265	1.96	3.62
3	63.0	7.1	24.5	555	254	2.18	3.24
4	61.1	8.0	23.5	426	219	1.94	3.49
5	60.9	7.9	24.1	472	193	2.45	2.76
6	64.9	7.1	23.9	517	250	2.07	3.31
7	56.2	8.2	21.9	417	219	1.91	3.44
8	61.6	7.3	23.0	465	228	2.04	3.39
9	64.5	7.2	23.4	521	281	1.86	3.75



**Fig. 3.** Viscosity vs. shear rate of wood plastic composites produced at different processing temperatures (recorded on high pressure capillary rheometer at 210°C); ■ – PP HD120MO, wood plastic composites produced at ● – 190°C, ▲ – 210°C, ▼ – 230°C

nificant darkening, because the actual temperature is beyond 220°C, and degradation of the wood is already started and accelerated with this temperature increase.

To quantify the amount of damage resulting for the polymer, rheological measurements were carried out. In a prior, unpublished work at the author's institute it was shown that the wood particle geometry has no influence on the viscosity at shear rates higher than 100 s<sup>-1</sup> (for composites with the same amount of wood in the formulation). Therefore these measurements can be used to evaluate the differences in polymer damage through the compounding step. From Fig. 3 it can be seen clearly that there is no difference between the compounds, produced at 8 kg h<sup>-1</sup> and different temperatures, namely 190, 210, and 230°C. This means that the amount of damage to the polymer is the same in all three cases, although the absolute amount cannot be determined.

When comparing the mechanical properties, the only difference between the compounds can be found for the maximum tensile strength, in the case of the sample produced at high temperature (230°C) and low throughput (8 kg h<sup>-1</sup>). One possible cause could be the decrease of the interfacial shear strength through the degradation of the wood. This interfacial shear strength  $\tau$  can be calculated from the wood particle geometry, *i.e.* length and width, the wood content, the polymer content, and maximum tensile

strength [6]. Calculating this value for each sample (Table 2) one yields values for the interfacial shear strength of  $3.45 \pm 0.3$  MPa. This is in good accordance with the results from a work of *Sanadi et al.* [7], who have found interfacial shear strengths of about 3 MPa for the system polyethylene-wood with dowel pull-out tests and a further work from *Rogers et al.*, who also have used this setup and have reported interfacial shear strengths for the system polypropylene-wood in the same range [8]. Due to the circumstance, that the polymer damage is the same for all samples, as shown before with rheological measurements, and the same wood type has been used, the only explanation can be given with the wood particle geometry itself. In the case for the sample with the lowered maximum tensile strength, the aspect ratio (the ratio between length and diameter) of the particles is the lowest of all produced samples (Table 2). This means that the wood particles took the most mechanical damage during the compounding step, and therefore the composite strength is lowered. This is also in good accordance with the colour measurements, where this sample also shows the lowest value for the lightness and therefore the highest thermal degradation of the wood.

In conclusion, wood plastic composites show a wide processing window for compounding. The only major differences were found for composites produced at high temperature in combination with low throughput, where composite strength was lowered and strong darkening occurred. Nevertheless, the extrusion is suitable for producing wood plastic composites with good overall properties, especially when choosing the right processing parameters.

## Experimental

Wood plastic composites containing 30 wt% wood particles Lignocel BK40/90, supplied by J. Rettenmaier and Sons, Germany, and 70 wt% polypropylene Daplen HD120MO, supplied by Borealis, Austria, were produced on a parallel co-rotating twin screw extruder (Thermo Haake TSE24HC), equipped with a gravimetric dosing system and with a length-to-diameter ratio of 28 at a constant screw speed of 500 rpm. For getting different composites, the throughput was varied between 8 and 16 kg h<sup>-1</sup>, as well as the barrel temperature, which was set between 190 and 230°C. The different melt strands were cooled *via* a waterbath and subsequently cut to granules with a strand cutter. These granules were dried in a circulating air oven at 80°C for at least 4 h prior to injection moulding.

Injection moulding with an Engel ES80 injection moulding machine was carried out complying with ISO 3167 to retrieve universal test specimens. The barrel temperature was set to 210°C and the dwell pressure was set at 40 bar. These factors were held constant for the whole set of samples to suppress the influence of the injection moulding step on the properties of the composites.

Tensile properties, *i.e.* elastic modulus and maximum tensile strength, were tested by means of a Zwick-Roell universal testing machine (Z020) complying ISO 527, with a crosshead speed of 2 mm/min. Impact properties have been recorded on a Zwick-Roell 5113.300 in accordance with ISO 179eU, as well as heat distortion temperature (HDT-A, ISO 75) has been measured with a Coesfeld HDT/Vicat Tester. Thermogravimetric analysis of the wood particles has been carried out according to ISO 11358 on a Mettler Toledo TGA under N<sub>2</sub>, with a heating rate of 5 K min<sup>-1</sup>.

Rheological measurements have been carried out on a Göttfert Rheograph 2000, equipped with a slit die (10 mm width, 0.65 mm height) at 210°C, in shear rate range of 10–1000 s<sup>-1</sup>. Colour measurements of the composites were carried out according the Lab colour sphere.

A xylene extraction [9] was carried out to regain the wood particles from the injection moulded composite for fibre length analysis. The wood particles were filtered off, dried, and prepared on a microscope slide for measurement of the particle size. To get a satisfactory number of particles in the measurement, multiple image alignment was carried out with the automated stage of the Olympus BX61 microscope and these micrographs were reassembled with the AnalySIS software. At least 1000 particles were measured per sample to get reliable values for the length, which was calculated length weighted to eliminate the influence of dust particles on the average values.

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