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Abstract

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ORIGINAL PAPER

In-Process Measurements of Flow Characteristics of Wood Plastic Composites

Valentina Mazzanti[1] · **Francesco Mollica**[1]

Abstract The flow characteristics of polypropylene filled with high percentages of wood flour (50 and 70 wt%) are investigated at a feasible processing temperature (195 °C). Rotational rheometry in oscillatory mode in conjunction with the Cox–Merz rule is ineffective at this temperature due to insufficient linear viscoelastic region (LVR) size and ease of fibres degradation. An in-line rheometer, directly attached to a single screw extruder, is used to avoid these problems: this method allows measurements in real processing conditions with no LVR issues and with reduced degradation risks. The 70 wt% displays plug flow, thus it has been impossible to obtain the flow curve but only the slip velocity as a function of shear stress. The 50 wt% could be corrected for slip using the Mooney procedure. Comparing the flow curve of this material with the ones of the 30 wt% and the unfilled polypropylene matrix, a single master-curve can be obtained and modelled with a Carreau–Yasuda equation.

Keywords Wood plastic composites (WPCs) · Natural fibres · Rheology · Extrusion

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Introduction

Wood Plastic Composites (WPCs) are thermoplastic polymers filled with wood flour. In recent years, the growing sensibility about environmental issues has pushed the WPC market along an increasing trend [1, 2]. WPCs can be processed like polymers but can also be worked in the same way as wood, hence they can substitute wood especially in outdoor products, where the presence of the hydrophobic polymer increases durability. Moreover, natural fibres are very convenient as fillers: they are cheap, lighter than ceramic fillers, biodegradable, less abrasive against processing machineries, capable of high filling levels and relatively easy to obtain if coming from local waste production [3, 4].

High percentages of wood flour determine a lesser usage of the more expensive polymeric matrix, thus leading to a reduction in material cost and an increase in environmental sustainability. This justifies the interest towards highly filled WPCs, such as 50 wt% or more. However, the presence of large quantities of natural fibres shows a number of drawbacks. First, the hydrophilic natural fibres are incompatible with hydrophobic polymers, thus a coupling agent is necessary. Further, the presence of hydrophilic fillers leads to water absorption, and this may cause swelling and distortion in the finite products. The major problem of materials filled with natural fibres, though, is their ease of oxidative degradation at relatively low temperatures [5]: only polymers that can be processed at temperatures below 200 °C are suitable as WPC matrices. Among these, polypropylene (PP) is interesting since PP based—WPC (from now on PP-WPC) have the advantage of possessing higher mechanical properties [6]. However, the PP melting temperature of about 165 °C makes the processability range rather narrow, i.e. only about 20 °C

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wide (180–200 °C). Moreover, as the processing temperature is close to the melting temperature, viscosity is relatively high and the advantage of incorporating high amounts of filler is thwarted by an additional viscosity increase. Therefore, although high natural fibre content is desirable, processing may become a very challenging task. An accurate knowledge of the rheological properties is necessary for successful control of WPC processing.

A common method for characterizing the flow properties of materials is rotational rheometry in oscillatory mode, equating complex and shear viscosity using the Cox–Merz rule. On the other hand, this holds only in conditions of linear viscoelasticity and WPC does not display a sufficiently wide linear viscoelastic region (LVR) at processing temperatures [7–10]. Moreover, in the literature there is no clear agreement over Cox–Merz rule applicability in the case of concentrated suspensions [11]. These drawbacks can be overcome by using a process rheometer, which allows to obtain accurate rheological data on polymeric fluids in processing conditions. Another advantage comes from the reduced oxygen content inside the extruder barrel, that makes it easier to avoid thermo-oxidative degradation during testing. On the other hand, an accurate temperature control may be very difficult to achieve [12].

Process rheometers have already been used for unfilled materials [13]: the slit is the preferential geometry, with two or more flush mounted pressure transducers to measure the pressure drop along the channel directly, thus without the need to perform the Bagley correction. Moreover, relatively thick slits can be used conveniently to reduce some of the problems related to particle filler size [14]. A 30 wt% PP-WPC has been studied in [8] using an in-process slit die equipped with a variable slit height, for performing the Mooney wall slip correction procedure [15], since it is well known that molten WPCs exhibit wall slip [16].

The aim of this work is to study the flow characteristics of PP-WPCs with high percentages of filler, i.e. 50 and 70 wt%, at a feasible processing temperature (195 °C). The same in-line slit rheometer described in [8] is used in this paper. For completeness, the PP matrix is also studied, using a parallel plate rheometer in oscillatory mode.

Materials and Methods

Materials

The materials used in this study are two commercial PP-WPCs purchased from PlasticWOOD S.r.l., Mazzantica di Oppeano (VR), Italy. The commercial names are PP CO 68/BZ, a 70 wt% composite, and PP 50 SCD, a 50 wt% composite. The producer has also supplied the matrix used for the composites, which is a polypropylene block copolymer. The average fibres length, diameter, and *L*/*D* ratio are 166.1, 16.0 µm and 10.2, respectively, as reported in [8]. The mechanical and thermal characterization of these materials are in [7].

Measurement Apparatus

The rheological measurements have been performed using a slit die in-line rheometer connected to a single screw extruder (P.R.T. SERVICE & INNOVATION s.r.l., Sant' Agostino (FE), Italy). The details of this instrument are described in [8].

The slit die (length 105 mm, width 50 mm) is made of AISI 4317 steel, case hardened and tempered to HRC 61. In this study, slit thicknesses of 1.95, 3.31, 4.04 mm have been used. With the slit die width to thickness ratio greater than 10 it is customary to neglect edge effects and the flow may be assumed as to occur between infinite parallel plates. A further thickness of 5.35 mm, slightly oversized, has been used only for the 70 wt% WPC.

Three flush mounted pressure transducers (GEFRAN M32 type mercury–filled transducers, ±0.25 % full scale accuracy) allow pressure drop measurements along the die. In order to reduce the risks that measurements are done in non-fully developed flow conditions, the first pressure transducer (200 bar full scale) is placed 40 mm after the slit entrance. The remaining two transducers are located 25 mm apart, hence at 65 and 90 mm from the entrance (100 and 50 bar full scale, respectively), thus making the overall measurement length $L = 50$ mm.

The slit die is equipped with a thermostat for temperature control. To increase reliability, the polymer temperature is also checked with thermocouples (J-type, ±0.1 C accuracy) contained inside the pressure transducers and capable of measuring the melt temperature directly. Analogic signals are conditioned and interfaced to a personal computer using the NI 9237 module for pressure and the NI 9211 module for temperature. LabVIEW 2013 has been used to acquire and record data.

Experimental Protocol

In-Process Rheometry

In order to reduce the moisture content, the WPC pellets have been dried at 80 °C for 24 h in a drying system before being flood fed into the extruder to perform the rheological measurements. During extrusion, a uniform temperature distribution of 195 °C has been maintained along the extruder barrel and the slit die. The volumetric flow rate Q, controlled through the extruder screw speed, has been determined by dividing the mass flow rate $\dot{m}$ by the known

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density of the fluid at the testing temperature ($\rho = 1125\ \mathrm{kg/m^3}$ for the 70 wt% and $1025\ \mathrm{kg/m^3}$ for the 50 wt%). The mass flow rate is the ratio of the throughput weight (in the present work around 100 g, weighed with a precision scale) over the time needed to extrude it.

Parallel Plate Rheometry

The PP matrix rheological characterization has been performed with 25 mm diameter smooth surface parallel plate rheometer (ARES,TA Instruments) in dynamic oscillation and strain controlled mode. Strain and frequency sweep tests have been done at 195 °C. The strain sweep test allows to determine the linear viscoelastic region (LVR) of the material, which is essential for performing the frequency sweep test properly. Three different frequencies, $\omega = 1, 3, 10$ rad/s, have been evaluated in a shear strain range of 0.02–5 %. The 4 % strain value has been chosen for the frequency sweep test. The frequency has been varied between 0.1 and 100 rad/s. The storage modulus G' and the loss modulus G'' have been measured as a function of frequency. These values have been used to calculate the complex viscosity:

$$\eta^* = \sqrt{\left(\frac{G'}{\omega}\right)^2 + \left(\frac{G''}{\omega}\right)^2}. \tag{1}$$

Using the Cox-Merz rule, the complex viscosity at a given frequency is numerically equal to the shear viscosity evaluated at a shear rate which equals the frequency.

Results and Discussion

The melt temperature in the extruder metering zone has always been stable at 195 °C. Along the slit die, temperature has remained within 2 °C from the target value (195 °C). It should be pointed out that temperature non-uniformity is a usual problem in process rheometers [12]. Examples of the temperature profiles that have been recorded during measurements are shown in Fig. 1. It can be seen that the highest deviation from the set point occurs in the last temperature reading (located at 90 mm from the entrance, or 15 mm from the exit), where temperature drops down by about 2 °C with respect to the adjacent reading. This effect is a consequence of the proximity of the slit die exit, but it can be speculated that, even if the last thermocouple is in close contact with the melt, its measurement may be influenced by the local temperature of the steel die, which is lower in that location due to the presence of the free surface at the exit. Thus it is possible that the melt temperature may actually remain more uniform, probably closer to the target temperature, in spite of the thermocouple reading. Such temperature profiles are thus considered to be acceptable.

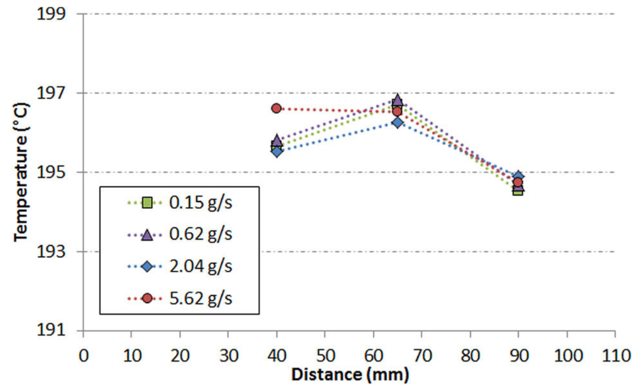

Fig. 1 Some typical temperature profiles along the die for the 50 wt% WPC, parameterized as a function of the mass flow rates

Pressure profiles from the three flush mounted transducers are linear, thus the pressure gradient grad$p$ can be promptly estimated from the slope of the best fitting straight line. Thus, the absolute value of the wall shear stress τ_w is

$$\tau_w = -\frac{H}{2}\,\mathrm{grad}p, \tag{2}$$

with H being the slit die thickness. The apparent Newtonian shear rate $\dot{\gamma}_{app}$ can be calculated from the volumetric flow rate Q and the geometry of the die (thickness H and width W) as follows:

$$\dot{\gamma}_{app} = \frac{6Q}{WH^2}. \tag{3}$$

In Fig. 2 the shear stress versus apparent shear rate diagram is shown for the 70 wt% WPC for the slit thicknesses that have been used. The curves do not superimpose: at a given shear stress, the apparent shear rate is higher for smaller slit thicknesses, a clear indication of wall slip. The curves have to be post-processed in order to subtract the contribution of wall slip. This can be done through the

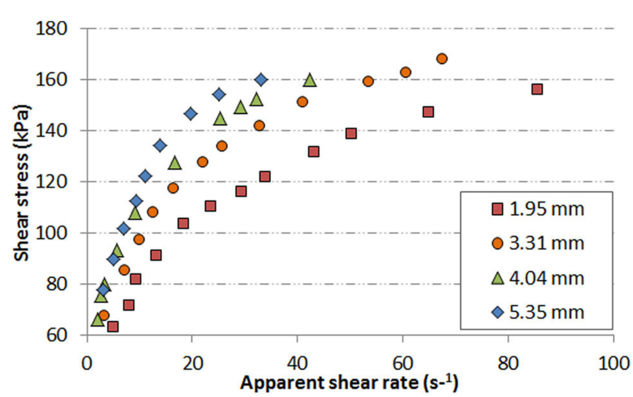

Fig. 2 Shear stress versus apparent shear rate for 70 wt% WPC for various slit heights

Mooney procedure [15], i.e. using the following decomposition of the apparent wall shear rate $\dot{\gamma}_{app}$ into a part due to wall slip and another due to viscous shearing at constant wall shear stress:

$$\dot{\gamma}_{app} = \frac{6v_s}{H} + \dot{\gamma}_{app\ no\ slip}, \tag{4}$$

where v_s is the slip velocity and $\dot{\gamma}_{appnoslip}$ is the apparent wall shear rate corrected for slip.

The results of the Mooney procedure are shown in Fig. 3: the apparent shear rate versus the reciprocal of slit height $1/H$ at constant wall shear stress is reasonably linear, in agreement with (4), and the slip velocities could be obtained from the curves slope. This notwithstanding, the intercepts on the apparent shear rate axis are not positive, thus the procedure is in fact inapplicable. The Mooney procedure is not always successful, for example if the Mooney plot shows negative intercepts on the apparent shear rate axis [13] or if the constant shear stress lines are not straight [17]. It is easy to fall into one of these possibilities when highly filled materials are tested [16]. In these cases, as it is impossible to subtract the slip contribution from the measurement of the apparent shear rate, the rheological characterization cannot be performed.

The negative intercept is a frequent problem in case of fluids that display relatively high wall slip, thus one should check for prevailing plug flow behaviour. This can be done, as shown in Fig. 4, by plotting the shear stress as a function of the average flow velocity $\bar{V}$ that is defined as

$$\bar{V} = \frac{Q}{WH}. \tag{5}$$

Indeed, as the four curves collapse into a single one, it can be concluded that this material undergoes plug flow. This result is alike to the one obtained by Li and Wolcott [16], who used a polyethylene based WPC with a concentration of filler of 60 wt% in a capillary rheometer.

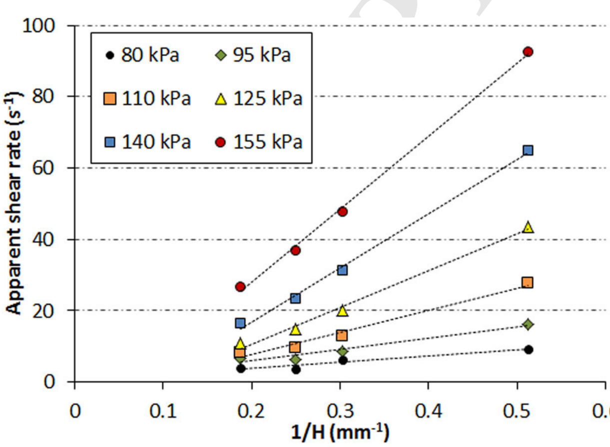

Fig. 3 Mooney plot of the 70 wt% WPC for six levels of shear stress

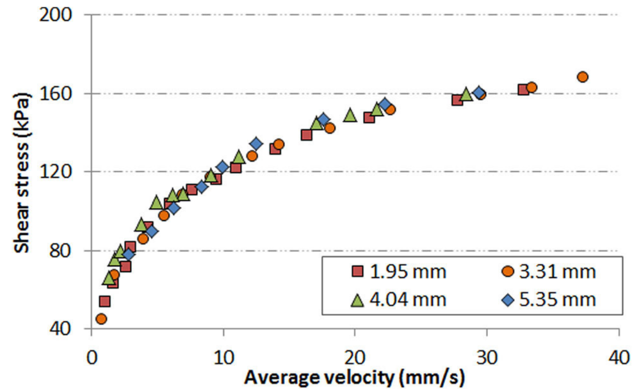

Fig. 4 Wall shear stress as a function of average flow velocity for 70 wt% WPC

In Fig. 5 the shear stress versus apparent shear rate diagram of the 50 wt% WPC is presented for the slit thicknesses that have been used. The curves show dependence on geometry, but it can be observed that the three curves are closer than the ones of the 70 wt% WPC. The Mooney analysis, shown in Fig. 6, has been performed successfully: the curves are reasonably linear and the intercepts are positive for all levels of shear stress. Next, the apparent shear rate has been corrected for non-Newtonian effects using the Rabinowitsch procedure. The viscosity versus true shear rate plot shows a shear thinning behaviour, as pictured in Fig. 7. For completeness, the viscosity values of 50 wt% WPC are shown together with the viscosity curves of the 30 wt% WPC taken from [7] and the neat PP flow curve, obtained with the rotational rheometer at 195 °C using the Cox–Merz rule.

The oscillatory testing used for characterizing PP has the advantage of exploring a wider shear rate range, especially at lower values. For this reason, it is possible to observe the Newtonian plateau at low shear rates, which does not appear in the WPCs flow curves. As shown in Fig. 7, a higher content of natural fibres increases viscosity.

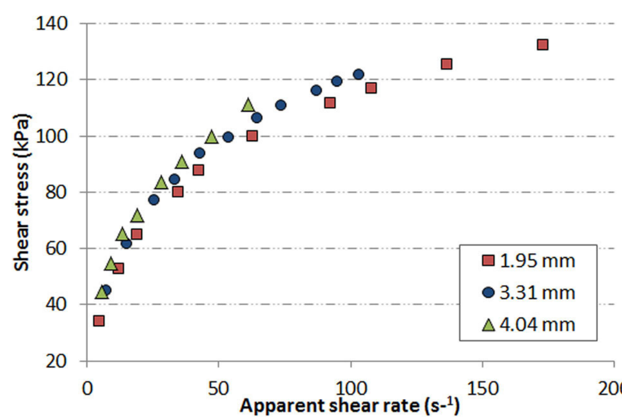

Fig. 5 Shear stress versus apparent shear rate for 50 wt% WPC for the various slit heights

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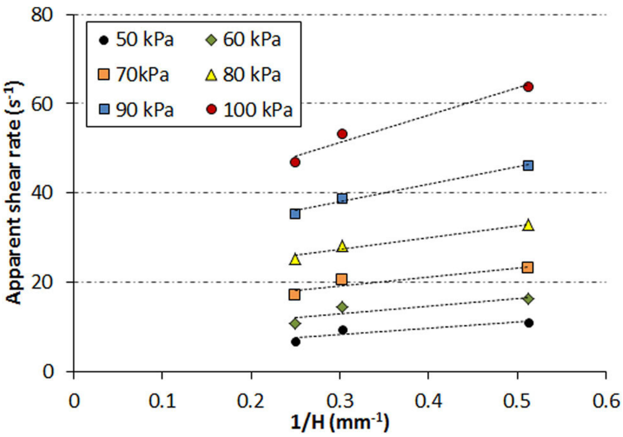

Fig. 6 Mooney plot for 50 wt% WPC at different levels of stress

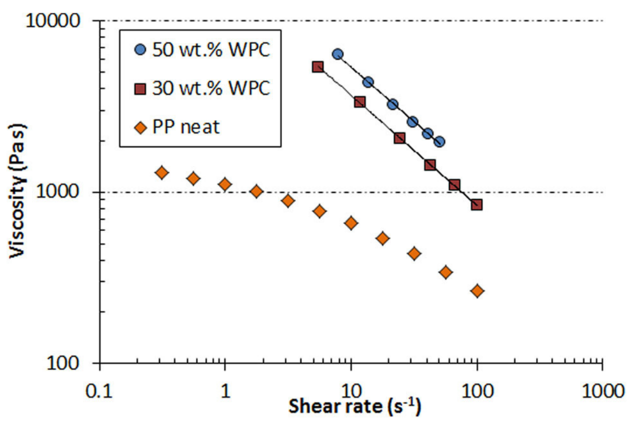

Fig. 7 Shear viscosity versus true shear rate for the 50, 30 wt% WPCs and complex viscosity of neat PP versus frequency at 195 °C. Interpolating line corresponds to the power law model

The viscosity curves are fitted with a power law model

$$\eta = K\dot{\gamma}^{n-1} \tag{6}$$

where n is the exponent of the power law and K is the consistency index. In the case of neat PP, the curve fitting has been performed sufficiently far from the Newtonian plateau (between 30 and 100 rad/s). All the fitting parameters are listed in Table 1. The consistency index increases with the percentage of wood, as viscosity generally increases with filler amount. On the other hand, the exponent of the power law essentially keeps the same value (around 0.4), irrespective of the filler content, and this shows that the shear thinning behaviour depends principally on the polymeric matrix, not on the filler, in agreement with Ares et al. [18].

Table 1 Fitting parameters for the power law model

Compound	K (Pa s^n)	n
Neat PP	4258	0.44
30 wt%	16,191	0.36
50 wt%	23,092	0.37

As introduced by Highgate and Whorlow [19] and reported also by Barnes [9], it is possible to obtain a single master-curve by shifting the flow curves in a log–log viscosity versus shear rate plot. The shift must be done in a 45° diagonal direction, i.e. comprising a horizontal shear rate shift and a vertical viscosity shift of the same value but opposite sign. The master-curve is shown in Fig. 8 and describes the viscosity of the unfilled matrix at 195 °C. Indicating this viscosity with η_{PP}, it can be fitted with a Carreau–Yasuda model:

$$\eta_{PP} = \frac{\eta_0}{\left(1 + (\lambda\dot{\gamma})^c\right)^{\frac{1-n}{c}}}, \tag{7}$$

where η_0 is the viscosity value at the Newtonian plateau for neat PP, $\dot{\gamma}$ is the shear rate, λ and c are fitting parameters. The remaining value, n, is the slope of the shear thinning portion of the curve in a log–log plot, thus the same symbol as the power law exponent (Eq. 6) has been chosen. From Fig. 8, it can be seen that the match of the fitted curve to the experimental set of data is very good, the fitting parameters are listed in Table 2.

The diagonal shift factors $a(\phi)$ are listed in Table 3 and plotted as a function of the filler volume fraction in Fig. 9. These data can be fitted with a modified Eilers model [20]:

$$a(\phi) = \left[1 + \xi \frac{\phi}{1 - \frac{\phi}{\phi_{max}}}\right]^2, \tag{8}$$

where the fitting parameters are $\xi = 9.85$ and $\phi_{max} = 0.79$. In particular, ϕ_{max} is the maximum volumetric loading of wood fibres in the PP matrix. With this procedure, the

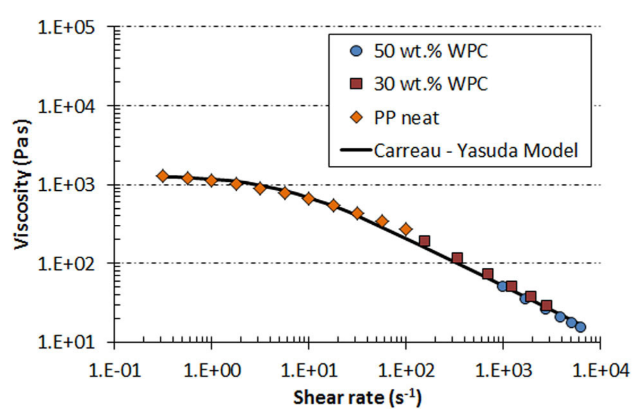

Fig. 8 Viscosity master-curve at 195 °C with the neat PP as the reference material fitted with a Carreau–Yasuda model

Table 2 Fitting parameters used in the Carreau–Yasuda equation

λ	0.171
c	1
n	0.38
η_0 (Pa s)	1300

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Table 3 Diagonal shift parameter as a function of the filler volume fraction

WF mass fraction wt%	ϕ WF volume fraction % vol.	Diagonal shift factor log(a)	Shift factor a
0	0	0	1
30	27	1.46	29
50	44	2.1	126

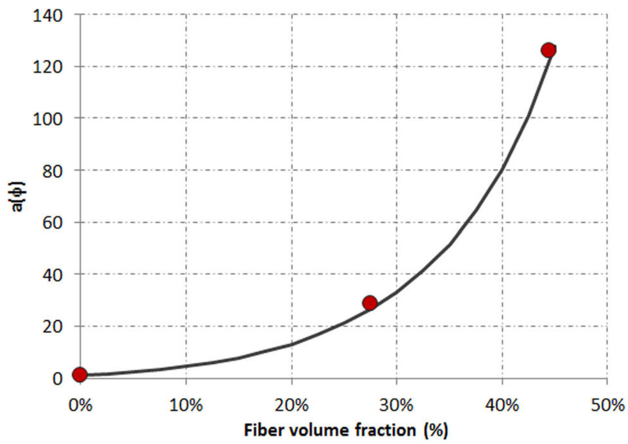

Fig. 9 Shift factors as a function of filler volume fraction and curve fitting using Eq. 8

viscosity curve at 195 °C of PP–WPCs at any percentage of filler between 0 up to 50 wt% can be interpolated using the correspondent diagonal shift factor with the following equation:

$$\eta(\phi, \dot{\gamma}) = a(\phi)\eta_{PP}(a(\phi)\dot{\gamma}). \quad (9)$$

The wall slip velocity versus wall shear stress curves are presented in Fig. 10. Slip occurs in the processing of WPCs, and in the case of the 70 wt%, this is the sole contribution to flow. This is not surprising, as external lubricants are typically added to the material composition in order to facilitate flow. Slip velocity varies in a range between 0.8 and 37.3 mm/s depending on the filler content and increases with the shear stress for all materials. In particular, at a fixed wall shear stress, materials with higher wood fibre content have smaller slip velocity in agreement with the results of [21] for HDPE-based WPC.

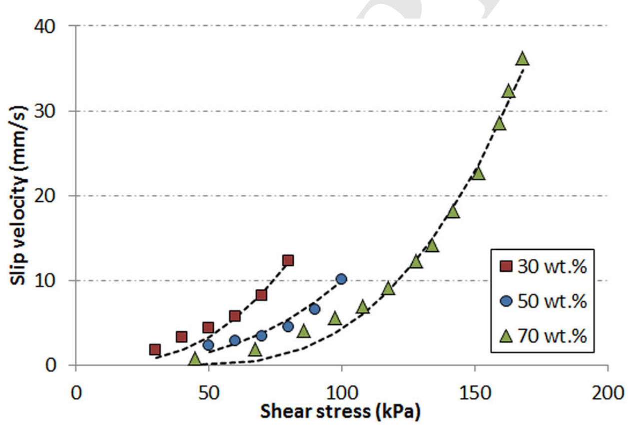

Fig. 10 Wall slip velocity of wood filled composites as a function of shear stress. *Dotted line* corresponds to the model indicated in Eq. 10

Table 4 Fitting parameters for the wall slip curves for 30–50–70 wt% WPCs

Compound (wt%)	h (mm/(s kPa^m))	τ_c (kPa)	m
30 %	8.0E−05	0	2.72
50	4.1E−05	0	2.69
70	6.0E−05	41	2.74

Within the examined range of shear stress the curves can be fitted with:

$$v_s = h(\tau - \tau_c)^m, \quad (10)$$

where τ_c is a critical shear stress at which wall slip starts. The other fitting parameters, h and m, are listed in Table 4. Notice that both the 30 and 50 wt% WPCs can be modelled without requiring the critical stress for slip activation, which is needed only for the 70 wt%. Interestingly, it is found that for each formulation the exponent m is nearly independent of the filler content.

Conclusions

In this paper we have studied the flow behaviour of two polypropylene based WPC compounds, filled with 50 and 70 wt% wood fibres, together with the neat PP matrix. These materials have many advantages in terms of cost and environmental sustainability but are very difficult to process because of their high viscosity and ease of oxidative degradation. For these reasons, knowledge of their rheological properties is important. Despite oscillatory rheometry is not suitable to characterize PP-WPCs at processing temperatures [7], in-line rheometry has been found to be appropriate at 30 wt% filler loading [8]. In the present paper, the 50 and 70 wt% PP-WPC have been characterized through in-line rheometry with the same methods described in [8]. The results show that the 70 wt% WPC flow curve cannot be obtained because the material shows plug flow behaviour, while the 50 wt% WPC viscosity curve has been obtained and compared to those of 30 wt% WPC and neat PP. The materials are found to be shear thinning, their viscosity increases with the wood flour percentage, but the slope of the viscosity curves remains approximately the

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same. This allows to scale the flow curves with respect to the filler content and obtain a single master-curve, representing the neat PP viscosity at 195 °C. The master curve can be modelled with a simple Carreau–Yasuda equation and the shift factor allows to estimate the viscosity curve for different wood flour concentrations.

The wall slip velocity has been determined for all WPCs and can be modelled with a simple power law relation for the 50 wt%, while the 70 wt% requires a shear stress threshold. The viscosity values for wood flour filled materials at high fibre concentrations are quite remarkable, nevertheless processing of these materials is made possible by extensive wall slip.

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