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In-line rheological properties of rubber toughened Wood Polymer Composites

V Mazzanti[1,3], L Malagutti[1], M Blanchard[1], S Yi[2] and F Mollica[1]

[1]Department of Engineering, University of Ferrara, Via Saragat 1, 44122 Ferrara, Italy
[2]Department of Wood Science and Engineering, Northeast Forestry University, China

[3]E-mail: valentina.mazzanti@unife.it

Abstract. Wood Polymer Composites are materials that are more and more used and studied by both academic and industrial research. Although such materials are successfully substituting natural wood in many relevant applications, still some of their drawbacks limit their widespread diffusion. One of these is their brittleness, which can be limited by blending with a toughening agent. In this study we investigated the rheological properties, in terms of the flow curve and the wall slip characteristics, of a toughened Wood Polymer Composite. Testing was performed using an in-line slit rheometer attached directly to a single screw extruder. The material used for the experiments was a blend of polypropylene-based wood polymer composite compound, containing 35 wt.% of wood flour and a suitable thermoplastic elastomer used as a toughening agent. The results showed a shear thinning behaviour and a significant slip velocity of the material that is beneficial during processing.

1. Introduction

Wood Polymer Composites (WPCs) are materials composed of thermoplastic polymers and wood flour. They can be used as substitutes for wood, thanks to their better resistance to moisture absorption and a reduced environmental impact [1,2]. For these reasons, important applications are in outdoor structures such as decking, fencing and flooring, possibly close to wet environments. Another important benefit of these materials is that increasing the wood content, the cost of the compound decreases [3]. On the other hand, one of their main drawbacks is their brittleness, which is especially relevant at high wood content and this is often at the origin of defects, surface failure and fractures in the final component after a relatively short period of usage.

Usually, reduced toughness problems are tackled by adding a thermoplastic elastomer in blend. On the other hand, the addition of another component into the compound could introduce complexity in transformation process and raise numerous additional issues. In order to better control the many processing variables in such a way that a material with adequate mechanical properties can be obtained, it is important to characterize the composite from the rheological point of view [4].

An in-line viscometer can be used to avoid problems that often affect off-line viscometry of this type of materials [5,6]. For example, rotational rheometry in oscillatory mode in conjunction with the Cox–Merz rule is often ineffective at the processing temperatures [6,7], also due to an insufficiently large linear viscoelastic region (LVR), while the capillary rheometer is frequently difficult to use due to the WPC high viscosity, especially at high filler volume fractions [3].

The aim of this study is thus to characterize the rheology of a rubber toughened polypropylene based-WPC at 185°C for optimizing processing conditions.

2. Material

A commercial wood-polypropylene composite (PPCO68/BZ from Plasticwood, Oppeano (VR), Italy) has been mixed with Santoprene 201-55, an EPDM based thermoplastic elastomer that was purchased from ExxonMobil. Mixing was done in equal proportions, as to obtain a 35 wt.% wood content in the final composite and the compound was been obtained the same extruder used for the in-line rheological measurements, which is described next.
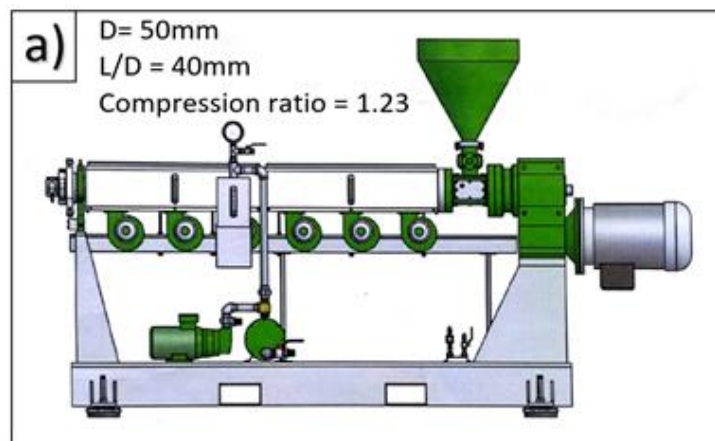
3. Methods

3.1. Measurement apparatus

For measuring the flow properties, an in-line slit viscometer has been used. This device consisted of an instrumented slit die attached to a single screw extruder (P.R.T. SERVICE & INNOVATION s.r.l., Sant'Agostino (FE), Italy) (Fig.1). More design details can be found in [3].

In this study, three slit thicknesses (1.33, 1.95, 4.07 mm) have been used in order to perform the Mooney wall slip correction procedure [8], which consists in performing measurements at different slit heights but at constant wall shear stress values, so that the slip contribution to flow rate could be evaluated and subtracted out. Three flush mounted pressure transducers (GEFRAN M32 type, with 100 bar full scale) allow pressure drop measurements along the die. The slit die is equipped with a thermostat for temperature control, and the polymer temperature is also checked with thermocouples. Analogic signals are conditioned and interfaced to a personal computer using the NI 9237 module for pressure and the NI 9211 module for temperature measurement (National Instruments).

3.2. Experimental protocol

In order to reduce the moisture content, the WPC pellets have been dried at 80 °C for 24 hours in an oven before testing. A uniform temperature distribution of 185°C has been fixed along the extruder as well as the slit die. The volumetric flow rate Q, controlled through the extruder screw speed, has been calculated by dividing the mass flow rate $\dot{m}$ by the density of the fluid ρ at the tested temperature.
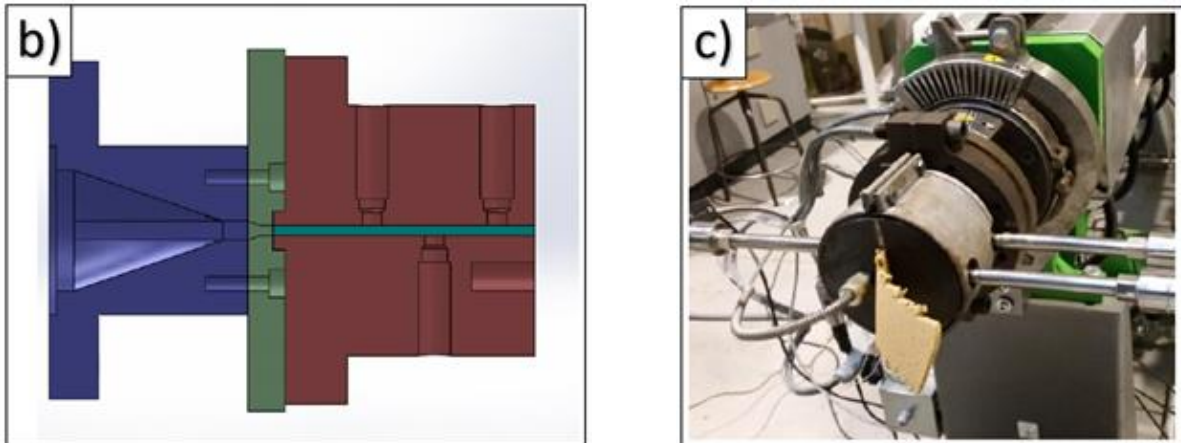
Figure 1. a) Single screw extruder, b) in-line slit viscometer scheme and c) a picture of the measurement apparatus.

4. Results

Pressure profiles measured by the transducers are linear and the pressure gradient p can be obtained from the slope of the straight line. The value of the shear stress τ_w is

$$\tau_w = -\frac{H}{2} grad\ p \tag{1}$$

where H is the slit thickness. The apparent shear rate, i.e. the shear rate the fluid would have if it were Newtonian, is defined as

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

where Q is the volumetric flow rate and W is the width of the slit die.

In Fig. 2, τ_w vs. $\dot{\gamma}_{app}$ is shown for various slit heights. The curves can be fitted with the following power law

$$\tau_w = C\dot{\gamma}_{app}^{\ n} \tag{3}$$

where C and n are fitting parameters (Tab.1). It is interesting to note that both fitting parameters are almost independent of the die height.

At all slit thicknesses tested, τ_w increases with $\dot{\gamma}_{app}$ and the curves do not superimpose. Moreover, the curves place themselves from left to right in going from the largest to the smallest slit height, thus the Mooney procedure can be successful for correcting wall slip effects. This procedure works by decomposing $\dot{\gamma}_{app}$ into two contributions

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

where v_s is the wall slip velocity and $\dot{\gamma}_{app_{no\ slip}}$ is the apparent shear rate without the contribution of wall slip.

The results obtained from the Mooney plot are shown in Fig. 3. The curves are not linear, similarly to what is reported in [5] for the case of the rheological properties of PVC. Nevertheless, an estimate of the slope can be inferred from all curves and the intercepts are positive for all levels of shear stress. In Tab. 2, the apparent shear rate corrected for slip and the wall slip velocity for the four levels of shear stress are listed. Interestingly, the wall slip velocity increases significantly with the wall shear stress.

Next, the apparent shear rate has been corrected for non-Newtonian effects using the Rabinowitsch correction [9]. The viscosity η versus true shear rate plot is pictured in Fig. 4.
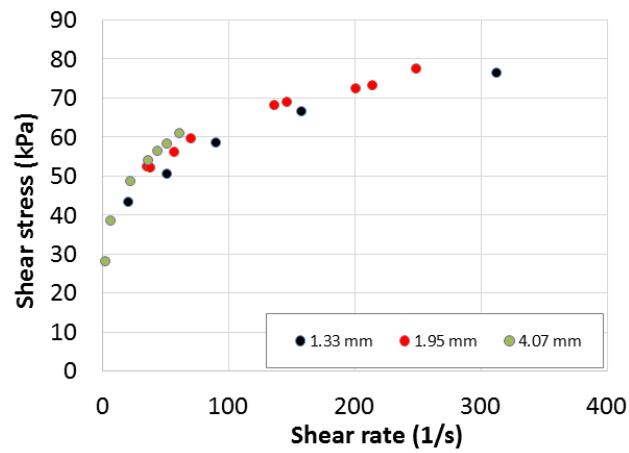
Figure 2. Shear stress vs. apparent shear rate for various slit heights.

Table 1. Fitting parameters for the power law model.

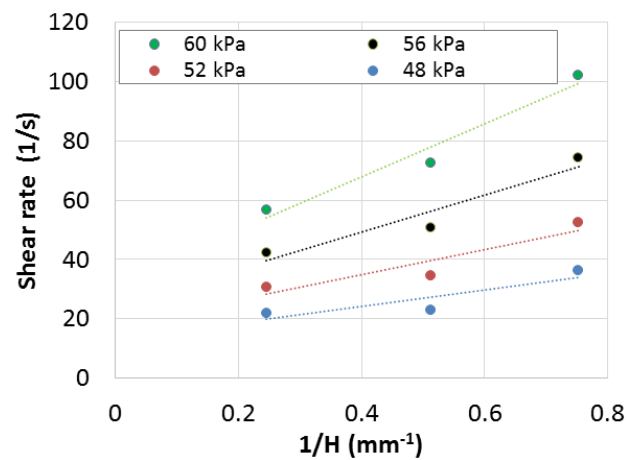| Die height (mm)	C (kPa s^n)	n
1.33	22.14	0.21
1.95	26.13	0.19
4.07	23.27	0.23

Figure 3. Apparent shear rate versus the reciprocal of slit height for four levels of shear stress.

Table 2. Apparent shear rate corrected for the slip and wall slip velocity for the four levels of shear stress.

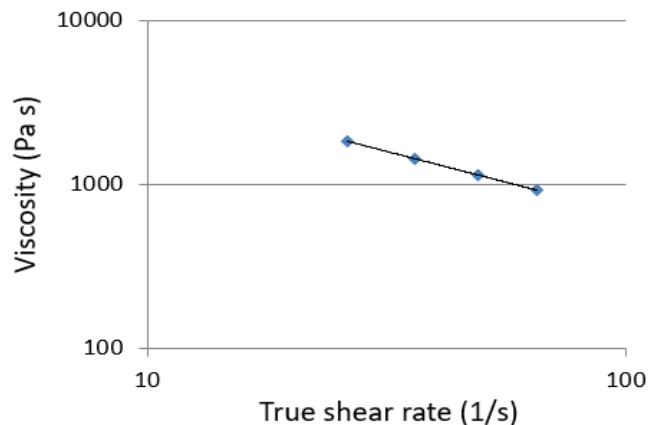| τ_w (kPa)	$\dot{\gamma}_{\text{app}_{no\ slip}}$ (1/s)	v_s (mm/s)
48	12.95	4.69
52	17.93	7.09
56	24.24	10.43
60	32.13	14.90

Figure 4. Shear viscosity versus true shear rate.

5. Discussion and conclusion

Mixing a thermoplastic elastomer with a wood plastic composite allows to obtain a material whose appearance is that of wood, but varying the content of elastomer in its formulation, the properties may lie in between those of a toughened plastic up to a very flexible material. This opens interesting applications in several fields, such as coatings that may resist impact, without affecting the usual WPC properties, like moisture resistance coupled to environmental friendliness.

In fact, the material at the molten state exhibits typical WPC rheological properties, such as shear thinning behavior and wall slip [2,3,10,11]. In particular, viscosity can be modeled through a power law defined as

$$\eta = K\dot{\gamma}^{(m-1)} \tag{5}$$

where K = 21.5 kPa s^m is the consistency and m = 0.25 is the power law index.

As a consequence, despite the wood content of the material is only about 35% by mass, thus typical of an injection molding grade, the rubber toughened WPC possesses a relatively high viscosity, more typical of an extrusion grade. Notice that, for this material, viscosity can hardly be lowered by significantly increasing the processing temperature. The wood flour that is used as a filler, in fact, undergoes thermo-oxidative degradation when the material is kept for too long times at temperatures close to 195°C – 200°C. Processing, though, is made easier by the relatively high slip velocity. This is possible thanks to the high content of external lubricants that are inside the Santoprene formulation [12]. The presence of external lubricants coupled to solid filler may induce pressure dependent wall slip phenomena [13] that may complicate the modeling of the transformation process.

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