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Rheological Evaluation of Melt Blown Polymer Melt

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Abstract. In this work, shear and uniaxial extensional viscosities of polypropylene melt blown sample with melt flow rate equal to 450 g/10min have been determined in wide deformation rate range by using capillary rheometry and novel orifice die design and the capability of recently proposed generalized Newtonian model to describe the measured experimental data has been tested.

Keywords: Shear viscosity, Extensional viscosity, Capillary rheometry, Generalized Newtonian law, Rheology, Polyolefines.
PACS: 83.85.Vb, 83.50.Ax, 83.85.Rx, 83.80.Sg, 47.85.md, 47.11.-j

INTRODUCTION

One of the key problem in the polymer processing operations optimization is unknown relationship between equipment design, processing conditions and rheological characteristics of the polymers. In order to assess such relationships, the modelling of the polymer processing is widely utilized for which the rheological characteristics of the polymer melts [1-2] determined at the particular temperature range represents crucially important material input data. However, the selection of the proper constitutive equation for the polymer melt, which is able to represent the measured rheological data mathematically, is required to reach fully predictive capabilities of the performed polymer processing simulations. Thus, the polymer processing operation at which the full rheological polymer sample characterization is very complicated or practically impossible by standard rheological tools is difficult to optimize. The melt blown technology for the polymeric nanofibers production is one of them [3-5]. In more detail, polymer melt viscosity during the nanofiber production has to decrease considerably (melt index 450-1600 g/10 min). As the result, the measurement of basic rheological characteristics at the processing deformation rates for the melt blown polymer samples is challenging task.

With the aim to extend the current knowledge in such a research field, the work is focused on the rheological evaluation of one PP melt blown sample by using capillary rheometer in order to find out the way in which shear as well as extensional viscosities can be determined experimentally at wide range of deformation rates. In the second

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part of this work, the fitting capability of recently proposed generalized Newtonian model [6-7] is evaluated for the tested polymer sample.

EXPERIMENTAL

In this work, melt blown PP Borflow HL504 FB B2-70006 (Borealis Polyolefine) having the following basic characteristics has been used in this work: Melt Flow Rate (230°C/2.16 kg) = 450 g/10min, Melting temperature (DSC) = 161°C, Molecular weight distribution - very narrow. Rosand RH7-2 twin bore capillary rheometer (scheme provided in Figure 1), together with Bagley and Rabinowitsch corrections, has been utilized for the experimental determination of shear and uniaxial extensional viscosities by using a novel orifice die depicted in Figure 2.

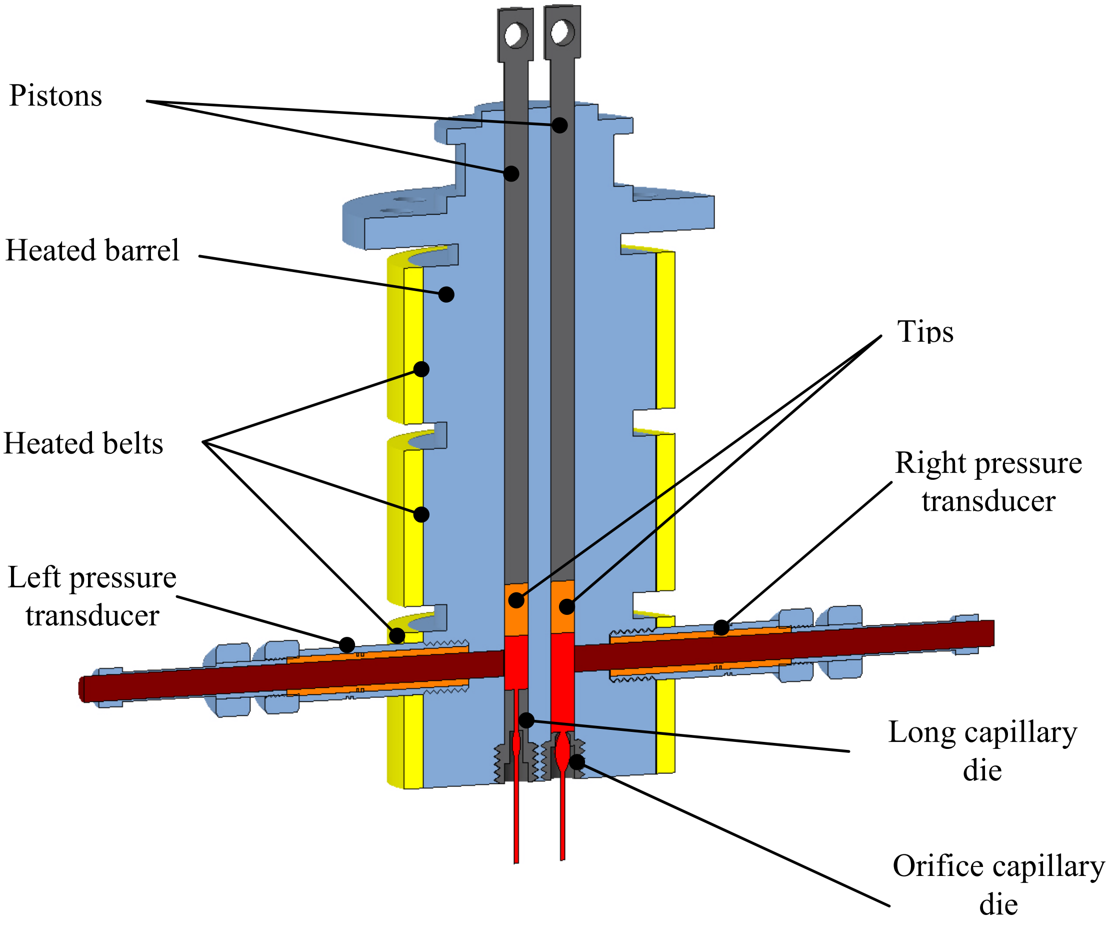

FIGURE 1. Sketch of the RH7-2 twin bore capillary rheometer.

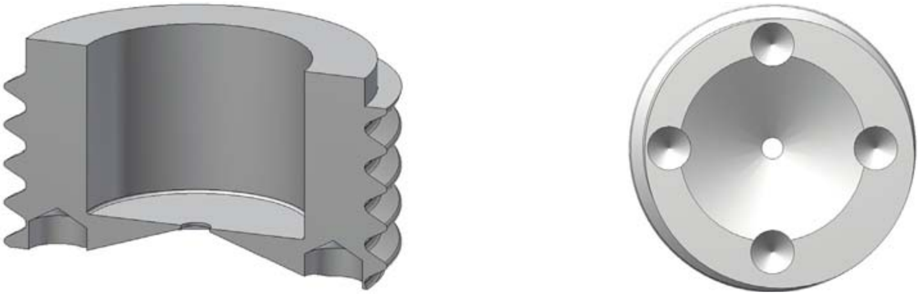

FIGURE 2. Sketch of the circular orifice die (CZ UV 19221) with abrupt entry for uniaxial extensional viscosity measurements developed in [8].

The main advantage of the utilized orifice die is the open downstream region design which eliminates any possibility for artificial pressure increase due to polymer melt touching the downstream wall. The uniaxial extensional viscosity has been determined through entrance pressure drop measurements by using the Cogswell model [9]. Due to the fact that the melt index of the chosen melt blown sample is extremely high, polyether ether ketone (PEEK) piston tips rather than copper ones have been used. The PEEK piston tips have been used in order to prevent any possible polymer melt leakage flow between the piston tips and the barrel due to very low shear viscosity of the melt blown samples. The comparison between the PEEK and copper piston tips for the capillary rheometer is provided in Figure 3.

FIGURE 3. Comparison between conventional copper piston tip (left) and PEEK piston tip (right) for the RH7-2 capillary rheometer.

The measurements were performed in a constant piston speed mode at the shear rate range of (10-80,000) s^{-1}. In our measurements we used pressure transducers (Dynisco, USA) in ranges of (10,000) PSI (68.9476 MPa), (1,500) PSI (10.3421 Mpa), (500) PSI (3.4473 MPa). In order to obtain the most accurate data of extensional viscosity for low extensional strain rates range, the highly sensitive pressure transducer (250) PSI (1.7237 MPa) calibrated into its down resolution limit was used for pressure recording at the entrance to the orifice capillary die.

Constitutive Equation

In order to describe basic rheological data for both tested samples, recently proposed generalized Newtonian fluid model has been utilized [6-7]:

$$\tau = 2\eta\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right)D \tag{1}$$

where τ means the extra stress tensor, D represents the deformation rate tensor and η stands for the viscosity, which is not constant (as in the case of standard Newtonian law), but it is allowed to vary with the first invariant of the absolute value of deformation rate tensor $I_{|\mathrm{D}|} = tr\left(|D|\right)$, (where $|D|$ is defined as the square root of D^2) as well as on the second $II_{\mathrm{D}} = 2\mathrm{tr}\left(D^2\right)$, and third, $III_{\mathrm{D}} = \det(D)$, invariants of D according to Eq. 2

$$\eta\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right) = A^{1-f\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right)}\eta\left(II_{\mathrm{D}}\right)^{f\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right)} \tag{2}$$

where $\eta\left(II_{\mathrm{D}}\right)$ is given by the well-known Carreau-Yasuda model (Eq. 3) and $f\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right)$ is given by Eq. 4.

$$\eta\left(II_{\mathrm{D}}\right) = \frac{\eta_0 a_{\mathrm{T}}}{\left[1 + \left(\lambda a_{\mathrm{T}} \sqrt{II_{\mathrm{D}}}\right)^a\right]^{\left(\frac{1-n}{a}\right)}} \tag{3}$$

$$f\left(I_{|\mathrm{D}|}, II_{\mathrm{D}}, III_{\mathrm{D}}\right) = \left\{\tanh\left[\alpha a_{\mathrm{T}}\left(1 + \frac{1}{4\left(\sqrt{3}\right)^3}\right)^{-\psi}\left(\left|1 + \frac{III_{\mathrm{D}}}{II_{\mathrm{D}}^{3/2}}\right|\right)^{\psi} \frac{\sqrt[3]{4\left|III_{\mathrm{D}}\right|} + I_{|\mathrm{D}|}}{3} + \beta\right]\frac{1}{\tanh(\beta)}\right\}^{\zeta} \tag{4}$$

here A, η_0, λ, a, n, α, ψ, β, ζ are adjustable parameters and a_{T} is temperature shift factor defined by the Arrhenius equation:

$$a_{\mathrm{T}} = \exp\left[\frac{E_{\mathrm{a}}}{R}\left(\frac{1}{273.15 + T} - \frac{1}{273.15 + T_{\mathrm{r}}}\right)\right] \tag{5}$$

where E_{a} is the activation energy, R is the universal gas constant, T_{r} is the reference temperature and T is local temperature.

Results and Discussion

The Role of the Capillary Rheometer Set-up in Rheological Evaluation of Melt Blown Samples

As it has been mentioned, the melt flow index of both tested melt blown PP samples is extremely high (450 g/10min determined at 230°C/2.16 kg) which indicates that the shear as well as extensional viscosities of both samples can be expected to be very low. Thus, the selection of suitable pressure transducers to properly capture rheological responses of such fluids is called for. In this part, utilization of two different pairs of pressure transducers has been investigated in order to find out the optimal testing conditions for precise determination of shear and extensional viscosities.

The Figure 4 shows comparison between strain rate dependent shear viscosities determined on capillary rheometer equipped with two different pressure transducers having different sensitivity: 10,000 PSI (long capillary)/1,500 PSI (short capillary) and 500 PSI (long capillary)/250 PSI (short capillary).

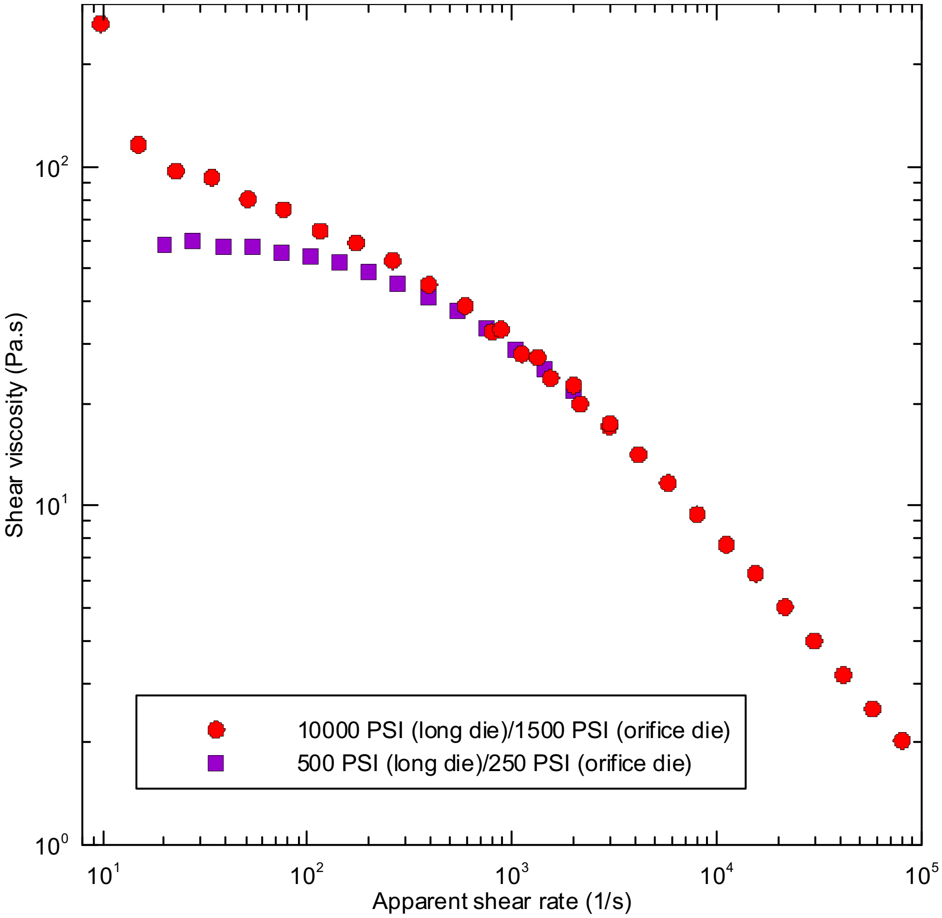

FIGURE 4. Effect of pressure transducer sensitivity on the deformation rate dependent shear viscosity for PP Borflow HL504 FB B2-70006 sample at 190°C.

It is clearly visible that utilization of the low sensitive pressure transducer leads to overestimation of the shear viscosity especially at low share rate in comparison with high sensitive pressure transducer by using which the Newtonian plateau in shear viscosity has been reached as expected. Thus it can be concluded that the 500 PSI (long capillary)/250 PSI (short capillary) pressure transducer set-up should be preferred to measure the basic rheological characteristic on the twin bore capillary rheometer for melt blown polymer samples.

Effect of Approximation Function Type Selection on the Index of Non-Newtonian Behavior Determination

The key problem to properly determine the index of non-Newtonian behavior is the proper evaluation of the term d(log τ_{xy})/d(log $\dot{\gamma}_{APP}$) from discontinues set of τ_{xy} and $\dot{\gamma}_{APP}$ pairs measured experimentally. In fact, there is number of possible ways to determine this term which is widely utilized in the practice. One possibility is to find out the 'proper' approximation function to fit the measured data and then to calculate the particular derivatives for given apparent shear rates by using that function. In this work, the following four approximation functions are selected, tested and evaluated for the index of non-Newtonian behavior determination for the tested polymer sample:

$$\log\left(\tau_{xy}\right) = A\log\left(\dot{\gamma}_{APP}\right) + B \tag{6}$$

$$\log\left(\tau_{xy}\right) = A\left[\log\left(\dot{\gamma}_{APP}\right)\right]^2 + B\log\left(\dot{\gamma}_{APP}\right) + C \tag{7}$$

$$\log\left(\tau_{xy}\right) = A\left[\log\left(\dot{\gamma}_{APP}\right)\right]^3 + B\left[\log\left(\dot{\gamma}_{APP}\right)\right]^2 + C\log\left(\dot{\gamma}_{APP}\right) + D \tag{8}$$

$$\log\left(\tau_{xy}\right) = \frac{A}{\left\{1 + \left[B10^{\log\left(\dot{\gamma}_{APP}\right)}\right]^C\right\}^{\frac{1-D}{C}}} \tag{9}$$

Note, that Eqs. 6-8 represent unphysical polynomial functions widely used in the engineering practice whereas Eq. 8 represents well known Carreau-Yasuda physical model widely used for the flow curve modelling.

In the first step, the measured τ_{xy} and $\dot{\gamma}_{APP}$ dependences have been fitted by all above mentioned equations and the parameters *A*, *B*, *C* and *D* have been estimated for the tested polymer sample at 190°C, and they are summarized in Table 1.

TABLE 1. Approximation function parameters for PP Borflow HL504 FB B2-70006, $T = 190°\text{C}$.

Approximation function name	***A***	***B***	***C***	***D***
Equation (6)	0.57848258	2.58117264	-	-
Equation (7)	-0.11911000	1.31716193	1.57597129	-
Equation (8)	0.01405944	-0.24998443	1.69374102	1.24760671
Equation (9)	62.90000000	0.00170000	-0.88880000	-0.30740000

In the second step, the index of non-Newtonian behaviour has been determined as d(log τ_{xy})/d(log $\dot{\gamma}_{APP}$) utilizing particular approximation function defined by Eqs. 6-9. The effect of approximation function type selection on the index of non-Newtonian behaviour determination for the tested polymer sample is visualized in Figure 5.

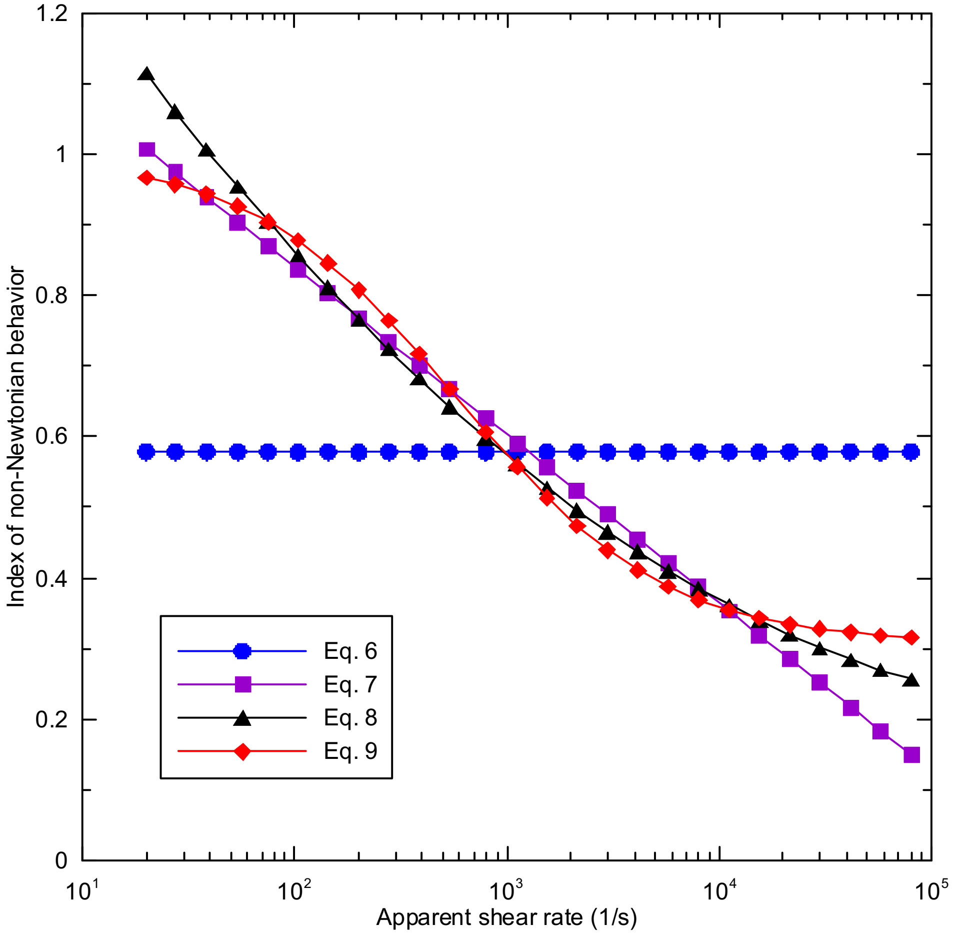

FIGURE 5. Effect of approximation function type selection on the index of non-Newtonian behavior determination for PP Borflow HL504 FB B2-70006 sample at 190°C.

It is clearly visible that the type of the utilized approximation function has significant effect on the index of non-Newtonian behavior determination within whole range of shear rates. Obviously, the utilization of the Carreau-Yasuda model for determination of shear rate dependent index of non-Newtonian behaviour is the most realistic because it gives physically correct values at low and high shear rates, which is not the case of another utilized models. In more detail, first order polynomial function (Eq. 6) leads to shear rate independent index of non-Newtonian behaviour (see Figure 5) which causing artificial shear/extensional viscosity decrease and increase at low and high deformation rates, respectively. This is visible in Figure 6 (note that extensional viscosity was measureable in this case only at middle and high extensional strain rates).

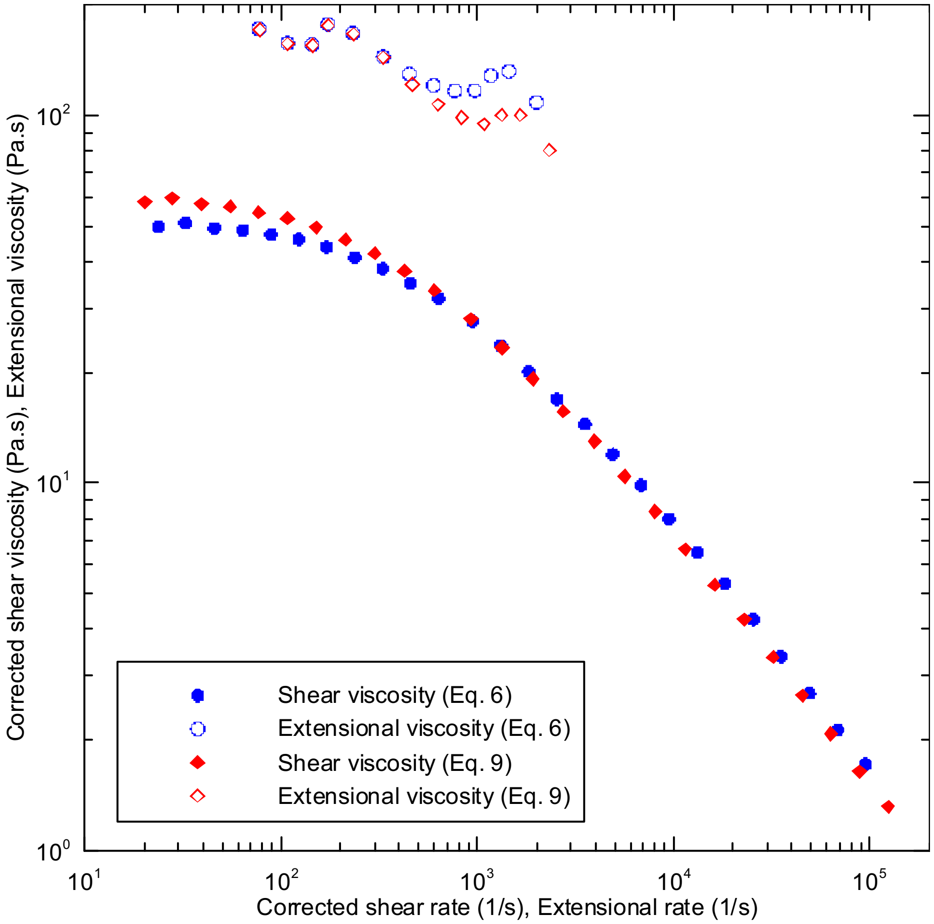

FIGURE 6. Effect of approximation function type selection (Eq. 6 vs. Eq. 9) on the shear and uniaxial extensional viscosity for PP Borflow HL504 FB B2-70006 sample at 190°C.

On the other hand, even if the utilization of higher order polynomial functions (Eqs. 7-8) leads to much precise evaluation of index of non-Newtonian behaviour, there is still tendency to artificially overestimate and underestimate index of non-Newtonian behaviour at low and high deformation rates, respectively, having impact especially on extensional viscosity, as visible in Figure 7. Therefore it can be concluded that the specific care has to be paid during approximation function selection for the index of non-Newtonian behaviour determination and physical rather than unphysical polynomial functions should be preferred.

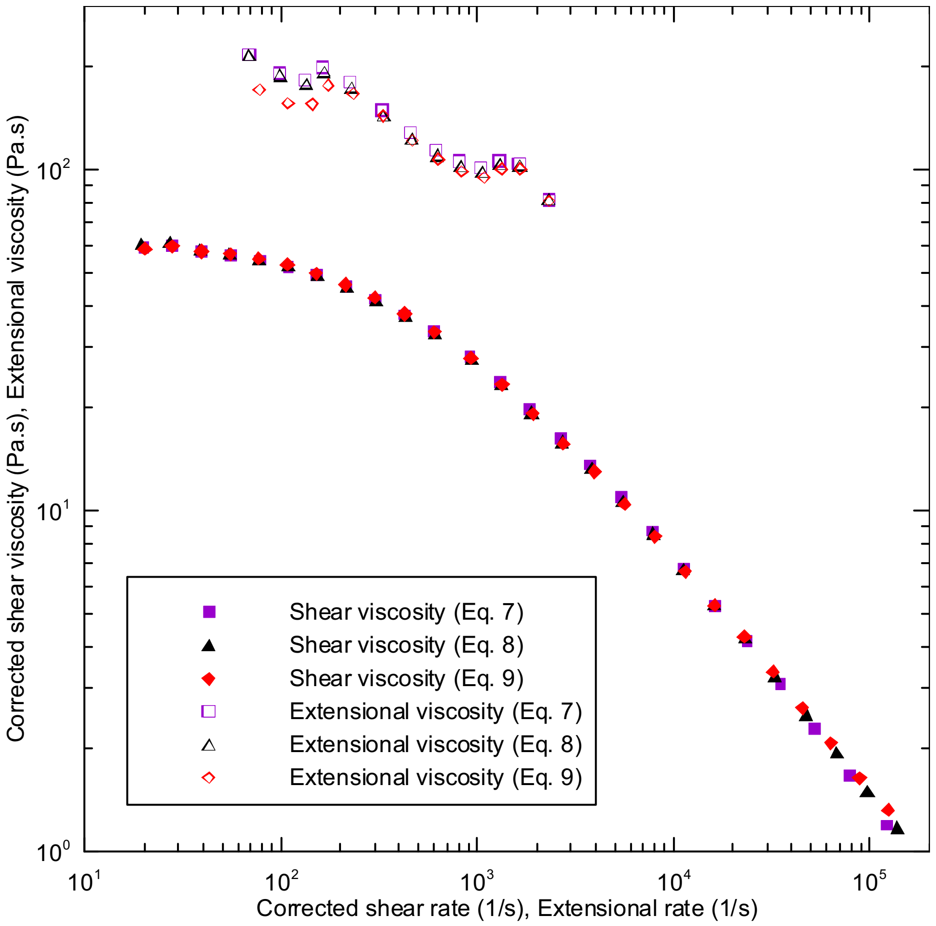

FIGURE 7. Effect of approximation function type selection (Eqs. 7-8 vs. Eq. 9) on the shear and uniaxial extensional viscosity for PP Borflow HL504 FB B2-70006 sample at 190°C.

Evaluation of Generalized Newtonian Model

Deformation rate dependent shear and uniaxial extensional viscosities, determined through utilization of Eq. 9 for index of non-Newtonian behaviour, has been fitted by the recently proposed generalized Newtonian model (Eqs. 1-5). The model parameters are summarized in Table 2 and comparison between the experimental data and model prediction is provided in Figure 8. As it can be seen, the model capability to describe the tested melt blown polymer sample is very high.

TABLE 2. Generalized Newtonian model parameters for PP, Borflow HL504 FB B2-70006.

η_0 (Pa.s)	λ (s)	a (-)	n (-)	α (s)	β (-)	ζ (-)	ψ (-)	A (Pa.s)
62.81	0.0021	0.8861	0.3079	0.00047	0.08	0.0073	20	$2\cdot10^{-16}$

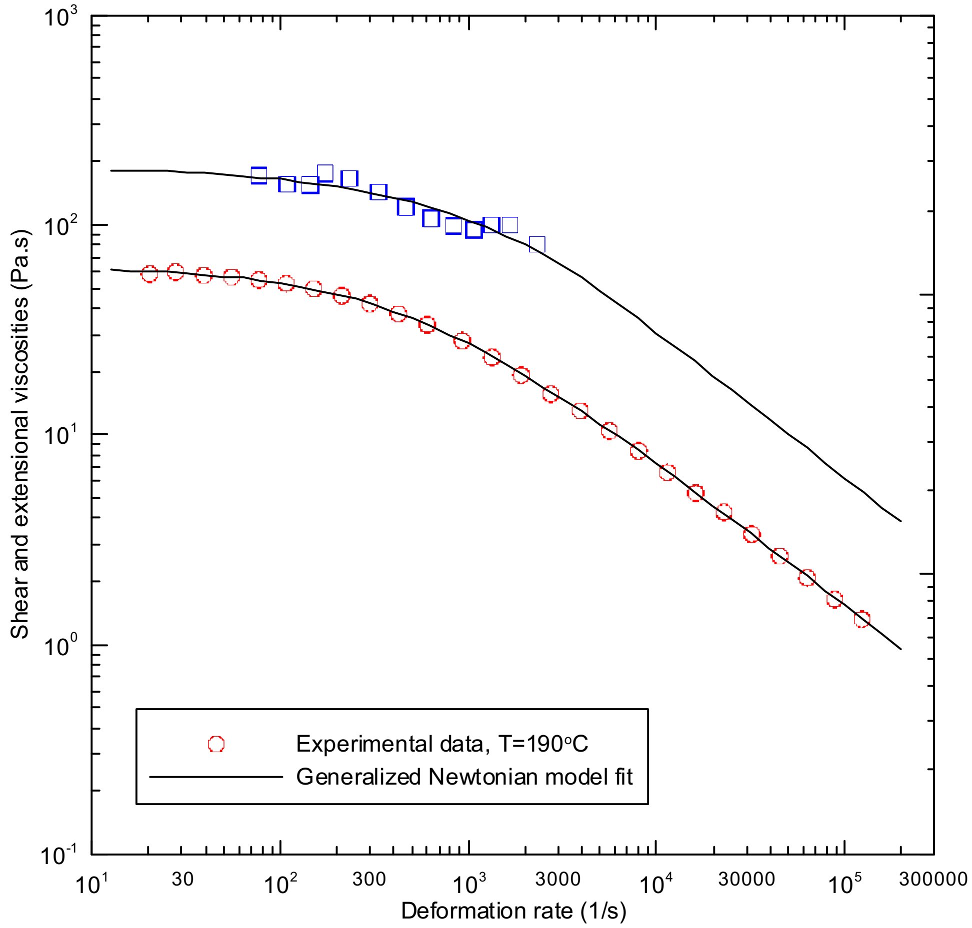

FIGURE 8. Comparison between experimental data for PP Borflow HL504 FB B2-70006 sample at 190°C and generalized Newtonian model prediction given by Eqs. 1-5.

CONCLUSIONS

It has been concluded that the 500 PSI (long capillary)/250 PSI (short capillary) pressure transducer set-up should be preferred to measure basic rheological characteristics on the twin bore capillary rheometer for melt blown polymer samples.

Physical rather than unphysical polynomial functions should be preferred during approximation function selection for the index of non-Newtonian behaviour determination from the measured corrected shear stress vs. apparent shear rate data; otherwise shear as well as extensional viscosities might not be determined correctly, especially at low deformation rates.

It has been found that recently proposed generalized Newtonian model [6,7] can describe the measured shear as well as uniaxial extensional viscosity data of PP melt blown sample very well, thus the model can be considered as good candidates for melt blown process modelling purposes.

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