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Experimental Evaluation of the Pressure Sensitivity of Molten Polymer Viscosity with a Triple-stage Capillary Rheometer

Xiang Lin[1], Jiong Liu[2], Changqing Wu[2], Dongyun Ren[2] and Jun Zhang[3]

[1]School of Chemistry and Biological Engineering, University of Science and Technology Beijing, 30 Xueyuan Road, 100083 Beijing, China
[2]College of Mechanical and Electrical Engineering, Beijing University of Chemical Technology, 15 Beisanhuan East Road, 100029 Beijing, China
[3]Xi'an Modern Chemistry Research Institute, 168 Zhangba East Road, 710065 Xi'an, China

*Corresponding author: xiang003.buct@163.com

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Abstract:
A triple pressure-stage capillary rheometer was individually developed for providing an insight of pressure effect on polymeric melts viscosity during steady and continuous flow. Three capillary dies with identical/varied diameters and aspect ratio were assembled in series along the flow direction, relying on which the flow was divided into three zones with varied pressures under the same flow rate. Several polymeric melts, such as low density polyethylene (LDPE), polystyrene (PS), polypropylene (PP) as well as its nanocomposites of PP/$CaCO_3$, PP/$Mg(OH)_2$, and PP/ halloysite nanotubes (PP/HNTs) were taken as the experimental samples. The principles for calculating the pressure sensitivity of shear viscosity in capillary flow were discussed, including methods based on constant shear rate (CSR), constant shear stress (CSS), and curve superposition (CSP). For the several polymer melts adopted in this work, a sequence of pressure dependence of viscosity was revealed as PS > PP > LDPE, which is typically acknowledged.

Key words:
Polymer, rheology, viscosity, pressure

1 INTRODUCTION

Principles of capillary viscosity measurement generally include two modes: constant flow rate (CFR) and constant flow pressure (CFP), which are only dependent on the squeezing piston movement. The former is the most adopted and controlled by stage-by-stage piston velocity, under which the liquids, generally polymer melts, are flowing with a constant apparent shear rate. As to the CFP mode, the shear rate is varied with the unstable flow rate and the shear stress is kept constant during each stress-stage. A general principle based on the CFR mode for a capillary rheometer is described as below. With the hypothesis of no wall slip, the shear stress can be given as

$$\tau_w = \frac{R\Delta P}{2L} \tag{1}$$

The Bagley correction should be considered in the correction of entrance effect, correcting the pressure drop with an entry pressure. The corrected shear stress $\tau_w{}^c$ is then given as

$$\tau_w^c = \frac{R\Delta P}{2L + 4Rn_B} \tag{2}$$

where n_B is the correction coefficient for entry effect. And the apparent shear rate under ideal flow state is

$$\dot{\gamma}_w = \frac{4Q}{\pi R^3} \tag{3}$$

As the non-Newtonian effect, the shear rate should be corrected as

$$\dot{\gamma}_w^N = \frac{(3n+1)\,\dot{\gamma}_w}{4n} \tag{4}$$

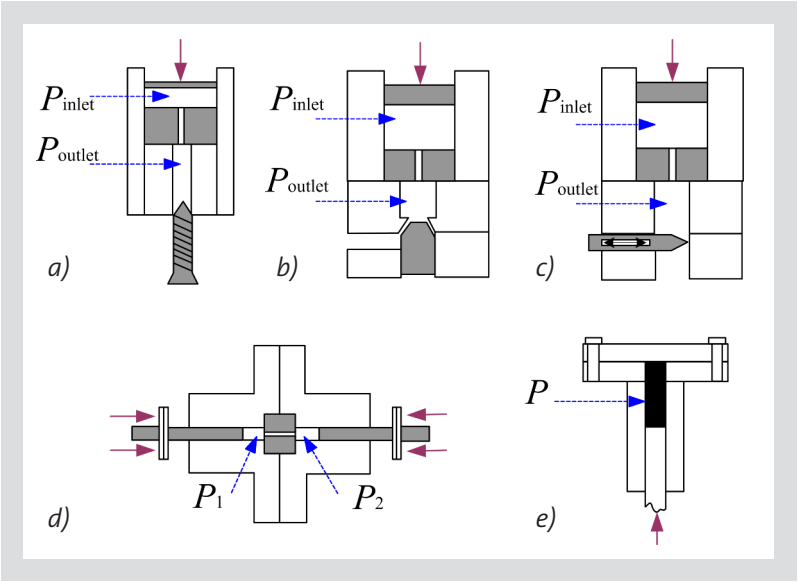

Figure 1: Prototypes of capillary rheometer with pressurizing effect: a) Baker and Thomas [28], b) Couch and Binding [14, 15], c) Sedlacek et al. [1], d) Westover et al. [29], and e) Foltz et al. [31].

where the non-Newtonian coefficient n is calculated by

$$n = \frac{d\ln \tau_w^c}{d\ln \dot{\gamma}_w^N} \tag{5}$$

Therefore, the shear viscosity is

$$\eta = \frac{\tau_w^c}{\dot{\gamma}_w^N} \tag{6}$$

where R is the die diameters [m], L the length of capillary die [m], ΔP the pressure drop [Pa] between the entrance and the exit, and Q the volume flow rate [m³/s].

Many constitutive equation, were developed in the past decades. Each of them was mainly focusing on certain factors which could significantly determine the viscosity. The effect of melt pressure, which plays the crucial role in high-precision injection or extrusion processes [1, 2] had been attracting researchers [1–13]. The first study of pressure effect on viscosity could be traced back to 1957, in which Maxwell and Jung [9] found that the viscosity of polyethylene melt increased by fourteen times when the pressure increased to 168 MPa from atmospheric environment. Similar works were also reported by Choi [10], Ito et al. [11], Lin et al. [12, 13], Couch et al. [14, 15], Cogswell and McGowan [16], Laun [17, 18], and Mackley and Spittler [19]. The coefficient of pressure sensitivity of viscosity β was proposed for estimating the dependence degree. Couch et al. [14, 15] and Kadijk and van den Brule [7] indicated that the coefficient β decreased with the increase of temperature, while Cogswell and McGowan [16] proposed that there is no dependence between β and temperature. Similarly, some researchers [20, 21] suggested that the coefficient β was determined by shear rate, while Kadijk [7], Couch & Binding [14, 15], Pantani and Sorrentino [22], and Carreras et al. [23] claimed that the β should be independent on shear rate at all. It can be revealed that the effect of pressure on viscosity of polymeric melts is rather complicated and a consistent conclusion is still not obtained. Experimental measurement is still taking over the responsibility to solve this problem. For similar investigations based on polymeric nanocomposites, the importance of these data is out of question that injection molding process of polymer-based nanocomposites would imply pressures above of 100 MPa, at which the pressure sensitivity of viscosity should be significant [24, 25]. As compared with those considering pristine polymer liquids, the viscosity dependence on melt pressure should be much more complicated owing to interfacial behavior as well as dispersion state [26, 27].

Experimental approaches were reported for evaluating the pressure sensitivity of viscosity such as enhancing outlet-pressure by a valve [14, 15, 28, 29] in which the melt pressure was increased by controlling the area of flow channel through a valve or switch (Figure 1a–c), opposite-extrusion with twin-piston [1, 19, 29, 30] in which the flowing melt was squeezed based on a good control of the pressing speed (Figure 1d), and ambient pressurizing by liquid medium [31–33] such as air and inert oil (Figure 1e). The most used method is to increase the melt pressure by adding a plug at the exit of capillary die, forming a chamber to hold the melt from outlet. This can be obtained by improving the conventional rheometer. Baker and Thomas [28] designed an improved capillary rheometer to detect the pressure dependence on viscosity. A valve with screw channel was set at the bottom of melt chamber, in which the melt from capillary die outlet was reserved with a certain pressure(Figure 1a). Couch and Binding [14, 15] studied the pressure sensitivity of polymer viscosity by a valve, by which the outlet pressure was mainly controlled (Figure 1b). Similar work was also reported by Sedlacek et al. (Figure 1c) [1]. As to the opposite-extrusion with two pistons, Westover et al. [29] developed the first device in 1960s (Figure 1d). This capillary rheometer contained two pistons and two barrels. The capillary die was set in the middle and shear behavior of melt was controlled by the opposite movement of pistons. The shear stress and shear rate were obtained by modulating moving velocity of pistons, inducing pressure drop between inlet and outlet of capillary die. Other improved prototypes based on this principle were further developed by Mackley et al. [19] and Son

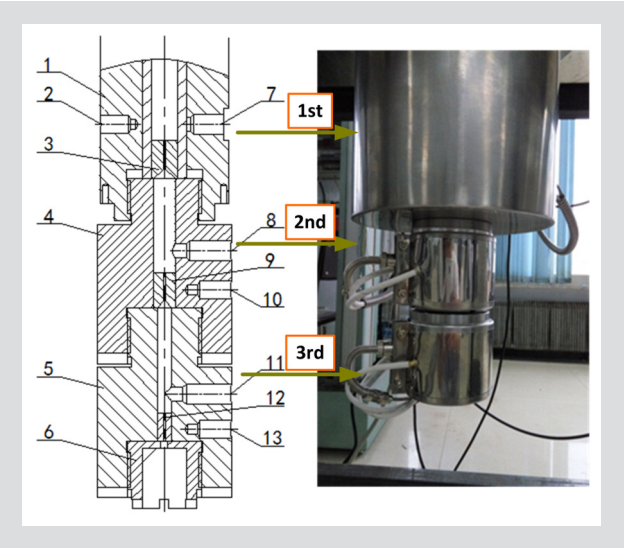

Figure 2: Schematic diagram of the combination of capillary dies in series.

et al. [30]. Another method was also adopted for enhancing the pressure of melts during capillary flow. Within a closed surrounding, the ambient pressure was enhanced by introducing inert liquid medium, such as air. The pressure was mainly dependent on the amount of injected medium. Bridgman et al. [32] considered the influence of pressure on viscosity of several polymers by using a falling sphere viscometer. The falling speed and time was the main indexes for calculating the viscosity. Such method was totally different from the capillary rheometer, which the viscosity was estimated by the flow behavior of melts. Improved devices were developed based on the same principle by Foltz [31] (Figure 1e).

For reducing repeat measurements and obtaining the viscosity data under a continuous flow state, prototype of a triple-stage-pressure capillary rheometer was individually developed based on single-bore capillary rheometer and to evaluate the pressure effect on capillary shear viscosity in this work. With three chambers being assembled in series, the melt pressure would be divided into three separated level with a single testing, thus the pressure effect of shear viscosity of polymers as well as their nanocomposites could be experimentally evaluated for the pressurizing melt. Meanwhile, the difference of calculating principles was also discussed for giving an overview on the evaluation of such behavior. Although the topic of the pressure effect on polymer viscosity seems to be a little platitude, these data in improving and controlling the precise processing technology never get old.

2 EXPERIMENTAL DETAILS

For pristine polymer melts, low density polyethylene (LDPE, CSPC 2624H) with a melt flow index (MFI) of 1.9 g/10 min and a density of 0.92 g/cm^3, polypropylene (PP, LCY 7533) with a MFI of 5.0 g/10 min and a density of 0.90 g/cm^3, and polystyrene (PS, GP 5250) with a MFI of 7.0 g/10 min and a density of 1.04 g/cm^3 are selected as the samples. In addition, several inorganic nanoadditives purchased from domestic companies, including spherical $CaCO_3$ (average diameter ca. 40 nm), $Mg(OH)_2$ flakes (average thickness ca. 50 nm) and rod-like halloysite nanotubes (HNTs, average diameter ca. 100 nm, average length ca. < 500 nm) were introduced into the PP matrix by melt blended with the help of a twin screw extruder with a combined static flow mixer. The detailed geometries of these adopted nanoparticles are provided by the manufacturers. Silane coupling agent was used for improving the compatibility between PP matrix and nanoadditives aiming to a better dispersion state.

Geometrical structure of the three-pressure-stage capillary dies is shown in Figure 2. Three capillary dies 3, 9, and 12 with three reservoirs (i.e. barrels) were assembled in series along the melt flow direction. The samples were melted well in the first barrel 1 and then were extruded from the third-stage capillary die 12. Three pressure sensors 7, 8, and 11 and three temperature thermal couples 2, 9, and 13 were axially sited in subsequence. The second barrel 4 and the third barrel 5 also acted the role of die supports as same as the support 6. The melt temperature was well controlled by the thermal couple 6 and its feedback system. With a single piston extrusion, therefore, the melt flowed passing three capillary subsequently under the same flow rate but under different melt pressure. The capillary dies with diameters of 0.5, 0.8, and 1.0 mm and aspect ratio L/D = 16 were chosen for each die. Barrel diameter of 15.0 mm was selected for barrels 1 and 4 and diameter of 10.0 mm was used for barrel 5, providing a pressure range of 10 – 200 MPa.

3 RESULTS AND DISCUSSIONS

The pressure effect on steady shear viscosity of LDPE, PP, and PS was measured by using the self-developed capillary rheometer. An example of the experimental data of LDPE at 190 °C, PP at 200 °C, and PS at 210 °C were illustrated in Figure 3. Under the same shear rate, the influence of hydrostatic pressure on shear viscosity was strongly revealed, especially for the PS melt. With a higher melt pressure during capillary flow, a higher viscosity was obtained, weakening the shear-thinning behavior. Different from the previous works [12, 13], the characterized pressure here is only up to ca. 80 MPa, limited to the third stage pressure loss. It can be seen that pressure fluctuation is revealed due to the flow instabilities, which is attributed to the last stage outlet and then contributes to next stage. The pressure data used for evaluating viscosity data were average values,

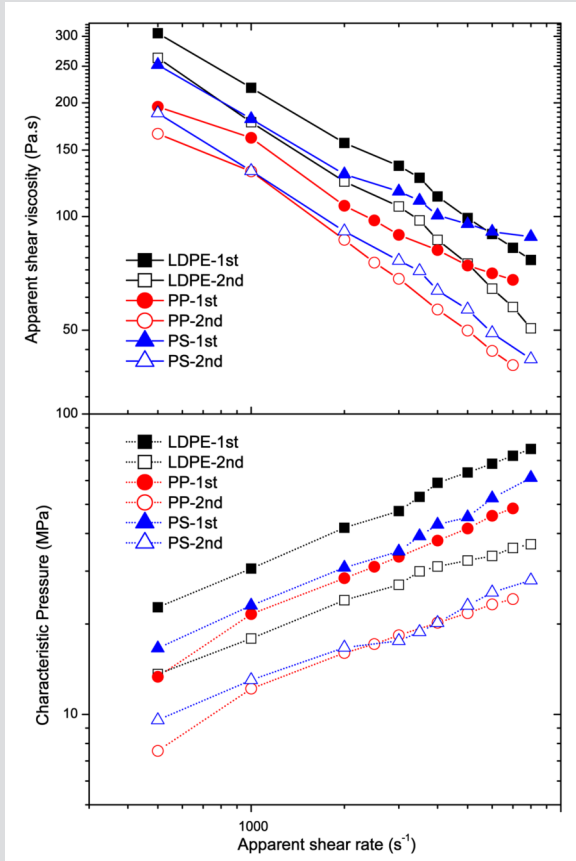

Figure 3: Viscosity curves of the three polymers affected by melt pressure.

calculated from the inlet and the outlet pressures. The pressure gradient in each chamber can neglected which was confirmed by finite element simulation. The pressure effect on viscosity follows the so-called Barus equation as:

$$\beta = \left(\frac{d\ln\eta}{dP}\right)_{T,i} \tag{7}$$

where β is the pressure sensitivity coefficient [GPa^{-1}], η the viscosity [Pas], P the characteristic pressure, i.e. average pressure [Pa], T the considered temperature [K] and i represents the applied shear. It should be noticed that for a certain shear rate or shear stress, the β is related to the slope of curves P versus lnη. This leads to three different methods for calculating β, including constant shear rate (CSR), constant shear stress (CSS), and curve superposition (CSP) principles. With a constant shear rate consideration, for instance, the viscosity as a function of melt pressure was given according to

$$\beta_{\dot{\gamma}} = \left(\frac{d\ln\eta}{dP}\right)_{T,\dot{\gamma}} \tag{8}$$

where $\beta_{\dot{\gamma}}$ is the pressure sensitivity coefficient under CSR principle [GPa^{-1}] and $\dot{\gamma}$ the shear rate [s^{-1}]. A comparison of $\beta_{\dot{\gamma}}$ for the three polymers is illustrated in Figure 4. This pressure sensitivity of steady shear viscosity can be simply explained by Doolittle equation as:

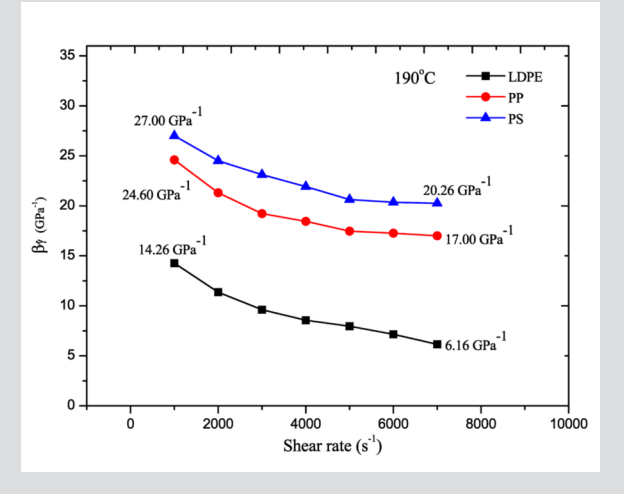

Figure 4: $\beta_{\dot{\gamma}}$ as a function of $\dot{\gamma}$ at 190 °C.

$$ln\eta = B\frac{V_o}{V_f} + lnA \tag{9}$$

where V_o is the substantial volume of molecules, V_f the free volume, and A, B are constants. For a certain amount of polymer melt, the V_o is kept as constant and V_f should be decreased with an increasing pressure, leading to an increase of the ratio of V_o/V_f. For a computation or theoretical calculation of flow, although the melt is frequently considered to be incompressible this is only acceptable when the pressures are relatively low usually less than 30 MPa. The considerable difference of $\beta_{\dot{\gamma}}$ should be attributed their varied molecular structures, which is largely relative to the flexibility and the polarization of molecular chains as particularly discussed in our previous work by molecular dynamics simulation [12, 13]. The lowest $\beta_{\dot{\gamma}}$ of LDPE is mainly attributed to its linear molecular chains without branches or benzenoid-structure. With the shear rate increasing, enhanced orientation makes the molecular chains stretched with less entanglement along flow direction and packed more densely, causing a reduction of $\beta_{\dot{\gamma}}$ at higher shear rate. When the calculation principle comes to CSS, the pressure sensitivity coefficient β_τ can be given as

$$\beta_\tau = \left(\frac{d\ln\eta}{dP}\right)_{T,\tau} \tag{10}$$

where β_τ is the pressure sensitivity coefficient under constant shear stress principle [GPa^{-1}] and τ the characteristic shear stress [Pa]. The Ellis viscosity model was used for viscosity curve fitting as

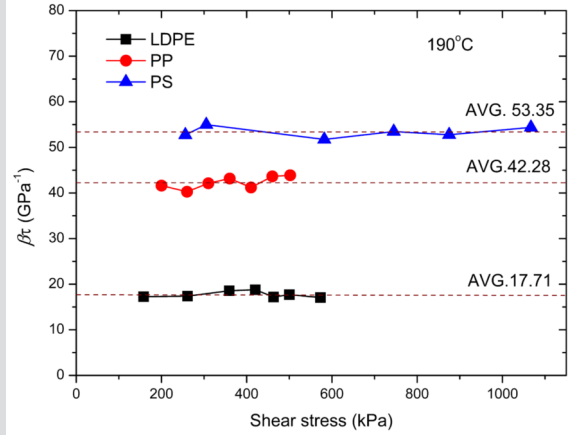

Figure 5: β_τ as a function of τ at 190 °C.

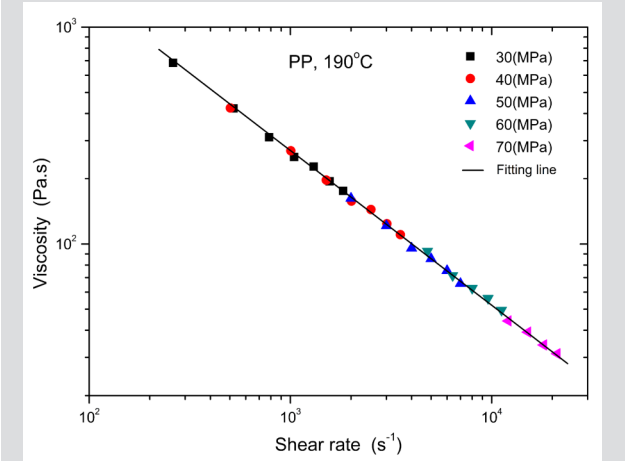

Figure 6: Example of viscosity curves shifted by ap at 190 °C.

$$\frac{\eta_o}{\eta(\tau)} = 1 + \left(\frac{\tau}{\tau_a}\right)^{\alpha-1} \tag{11}$$

where η_o is the zero shear viscosity [Pas], τ_a the shear stress [Pa] at $\eta = \eta_o/2$, and α a constant. The viscosity curves of the three polymers are well fitted by Ellis model at 190 °C based on variation of shear stress. Thus, data points under the same shear stress can be selected from these fitted curves. Then viscosity curves of lnη versus P were plotted for calculating β_τ according to the slope of these fitted curves.

It can be found that, comparing with the CSR principle, a relative stable β_τ value was observed for each polymer based on the CSS principle as shown in Figure 5. Identical result of subsequent order of β_τ (PS) > β_τ (PP) > β_τ (LDPE) was revealed. The β_τ was found to stay constant without changing with the shear stress, while β_γ was varied with shear rate. As for the CSP principle, a shift factor a_{TP} was proposed when the effects of shear, temperature and pressure were simultaneously considered. The shift factor a_{TP} was given as

$$\eta(\lambda, P, T) = \eta\left(a_{TP}\gamma, P_{ref}, T_{ref}\right) a_{TP} \tag{12}$$

where T_{ref} and P_{ref} are the referenced temperature [°C] and pressure [Pa], respectively. With the help of this shift factor, effects of shear rate, temperature, and pressure on viscosity can be separated by shifting the viscosity curves, keeping one of these effects constant. Example of an proposed a_{TP} was developed by Couch and Binding [14, 15] by combining Barus equation and Arrhenius equation as

$$a_{TP} = a_P a_T = exp\left[\beta_\theta\left(P - P_{ref}\right)\right] exp\left[\frac{E_s}{\left(R/T - R/T_{ref}\right)}\right] \tag{13}$$

where a_P is the pressure shifting factor, a_T the temperature shifting factor, β_θ the pressure sensitivity coefficient [GPa^{-1}] calculated under CSP principle, E_s the viscous flow activation energy [J/mol], and R the gas constant of 8.31 J/(molK). Under the same melt temperature, curves of shear viscosity versus shear rate under a series of constant melt pressures can be shifted onto the unique viscosity curves under the referenced pressure, e.g. 50 MPa. An example of PP viscosity curves was presented with superposition manipulation as shown in Figure 6. It can be observed that the curves are perfectly superposed onto a single fitted curve with an enlarged shear rate range. This indicates that the CSP principle can be well applied in the calculation of pressure sensitivity. Based on Equation 13, the pressure effect on viscosity can be eliminated by shifting viscosity curves according to ap, which revealed from the slope of plotted curves lna_p versus ΔP. The calculated β_θ is given in Figure 7, in which the pressure sensitivity coefficient was also found to be independent on pressure, similarly to that calculated from CSS principle in Figure 5.

The comparison above of the three calculation principles for evaluating pressure sensitivity of viscosity by using the triple-stage capillary rheometer shows that the coefficient β_γ, obtained from CSR is largely dependent on the shear rate, while β_τ and β_θ, which are calculated by CSS and CSP principles are independent on shear stress and pressure respectively and kept as

Polymers	T [°C]	β [GPa^{-1}]
HDPE	150 - 210	10.36 [1], 10 [14], 59 [15]
LLDPE	150 - 190	11.72 [1], 10.9 [35]
LDPE	150 - 200	18.33 [1], 17 [36], 17.71*,
PP	190 - 230	21.03 ± 4.1 [1], 22 [14], 42.28*,
PC	280 - 300	31.12 [1]
PS	190 - 230	43.45 ± 12.1 [1], 39.4 ± 10.3 [7], 53.35*
PMMA	230 - 250	43.57 [1], 24.6 [14], 36.5 [35][1], 48 [15][2]

*Table 1: Examples of the experimental β values for different polymers (1 T = 150 - 200°C, 2 T = 200°C, * average data in this work).*

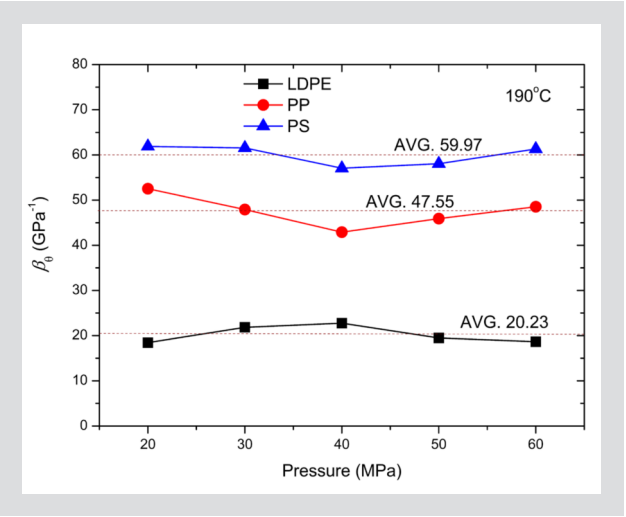

Figure 7: β_θ as a function of pressure P at 190 °C.

relative constant. This provides the potential to compare the pressure effect in the whole shear rate range, making them more convenient to use in practical applications. For a certain polymer, the pressure coefficients calculated by CSS (β_τ) and CSP (β_θ) methods are closed to each other, showing small differences. Compared the pressure coefficient β_γ from CSR method with β_τ or β_θ, however, big differences are revealed. Similar results were also reported in literature [29, 30]. Thus, the developed triple-stage capillary rheometer can be effectively used for experimental investigation. Although these experimental measurements are difficult to give a consistent conclusion, relative comparisons are still present clear understandings on the pressure dependence of polymeric viscosity. Varied β values from references are illustrated in Table 1, which several typical polymers are only given.

As to the polymeric nanocomposites, pressure effect on their viscosities was also studied by using this triple-stage capillary rheometer. A twin-screw extruder combined with a static flow mixer were adopted in samples preparation, in which the PP was adopted as the matrix with varied nanoparticles (NPs), including spherical $CaCO_3$, Halloysite nanotubes (HNTs) and Nano-$Mg(OH)_2$. The β_γ values of these polymeric nanocomposites were presented as shown in Figure 8. It can be found that not only the volume fraction of the additives but also the NPs themselves affect the pressure dependence of shear viscosity. The intrinsic attribution to these results are rather complicate, including surface morphology, compatibility with matrix, dispersion state as well as chemical features. But the pressure effect gradually decreases with the increasing shear rate. Based on a good dispersion, a relative high concentration of these nanoadditives is generally contributed to a reduction of compressibility, unless the porosity of NPs themselves is significant. This will introduce large number of micro-cavities which can be compressed under a certain pressure and lead to flow instability, causing false appearance on the pressure dependence of shear viscosity. Well dispersed NPs can reduce the free

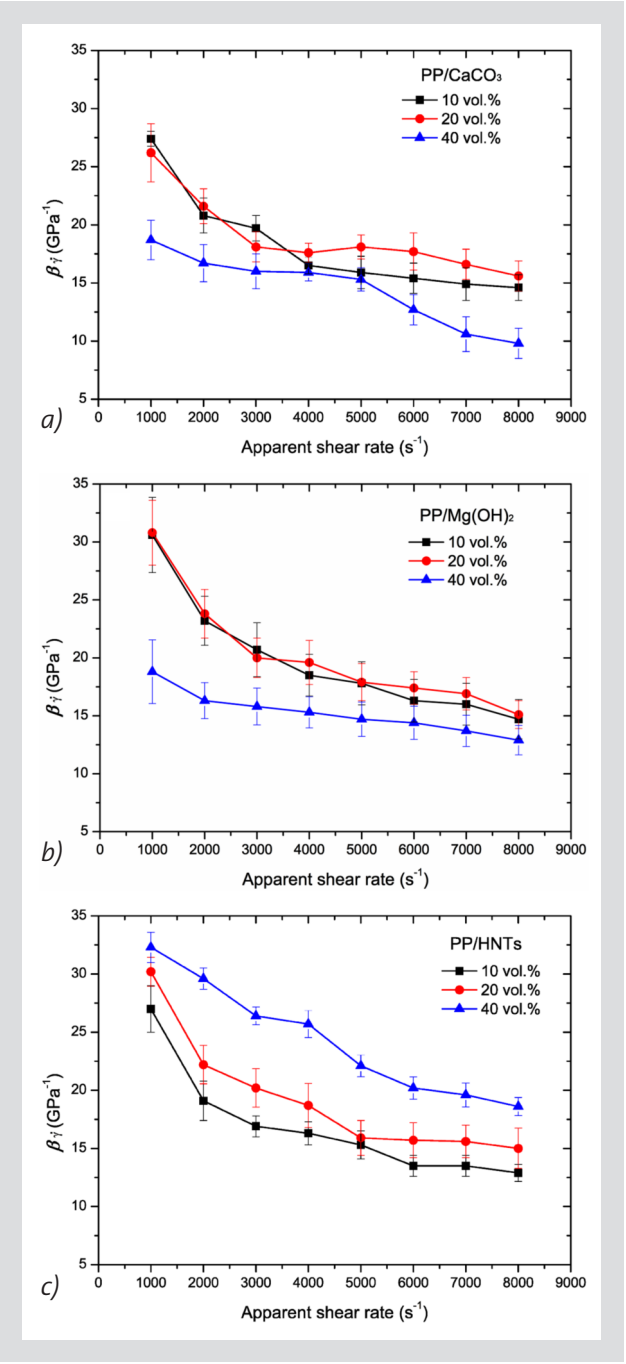

Figure 8: β_γ of PP-based nanocomposites: a) PP/CaCO3, b) PP/Mg(OH)2, and c) PP/HNTs.

space of the matrix and confine the flow ability of molecular chains, naturally leading to reduced compressibility.

4 CONCLUSIONS

In this work, the role of the hydrostatic pressure in viscosity determination of polymeric melts in capillary flow was emphasized. A triple-stage-pressure capillary rheometer prototype was manufactured for providing a pressurized environment. The melted polymers flowed subsequently passing through the combined capillary dies in series. Several polymeric melts, includ-

ing LDPE, PS, and PP as well as their nanocomposites, were used as experimental samples. Although the adopted calculation principles would cause results deviation, the typical sequence of pressure dependence of viscosity as PS > PP > LDPE was still obviously revealed. Principles for estimating the pressure sensitivity, including CSR, CSS and CSP, were comprehensively discussed in calculating the pressure coefficient of viscosity β. Results showed that the coefficient β_γ was dependent on the shear rate, while β_τ and β_θ were independent on shear stress and pressure, respectively. This provided an overview in considering the pressure effect when pressure effect played an important role in practical processing. Furthermore, pressure effect on the shear viscosity of the polymeric nanocomposites was also considered. Owing to the introduction of nanoparticles effectively filled the free volume of matrix, a reduction of compressibility was obviously observed. The β_γ of PP/NPs decreased with the increase of shear rate. Meanwhile, the pressure effect on viscosity of PP/NPs was also found to be dependent on their additives content. A higher particles introduction usually decreased the compressibility and naturally led to a decreased pressure coefficient.

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