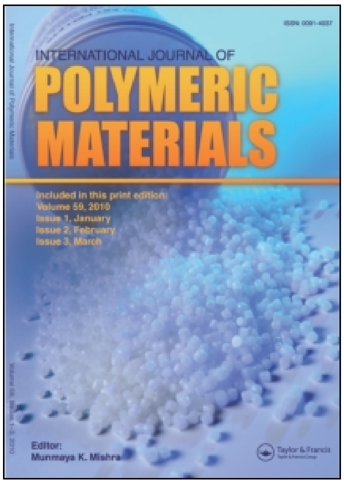

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Rheological Behavior of Polyethersulfone (PES)

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Rheological Behavior of Polyethersulfone (PES)

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The rheological behavior of Polyethersulfone (PES), was studied using a Shimadzu Universal Materials Testing Machine and an Haake Torque Rheometer. It is found that in an industrially relevant range of shear rate and temperature PES was highly non-Newtonian. Its shear dependence and temperature dependence of viscosity were unexpected, and the power law index and activation energy were determined. A phenomenon of thickening of melt during processing of PES was found.

KEY WORDS Polyethersulfone (PES), rheological property, Haake Plasticorder, thickening of melt.

1. INTRODUCTION

Polyethersulfone (PES) is perhaps one of the most important engineering thermoplastics that displays excellent properties.[1,2] The use of this material requires that it be melt processed for injection and extrusion molding, etc. In previous papers there are a lot of brief introduction about the processing conditions of PES[3,4] and seldom deals with the rheological properties.[5] But no study has been conducted to investigate the rheological properties of PES relevant to real processing conditions. The knowledge of the rheological behavior of PES over an industrially relevant range of shear rate and temperature is essential for the assessment of the material processability, process design optimation and troubleshooting. It is to this end that this work was undertaken.

The rheological behavior of PES over an industrially relevant range of shear rate

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and temperature has studied by means of a capillary instrument attached to a Shimadzu Universal Material Testing Machine. As a torque rheometer (plasticorder), this rheological device is useful for studying the rheological behavior of plastics. It produces a flow curve that is a measure of the resin's processibility. A torque-temperature-time curve of PES, measured by Haake microcomputer-controlled torque rheometer model SYS 40, is presented in this paper and can be directly related to the real world of molding.

2. EXPERIMENTAL

2.1 Materials

The PES resin used in this study was kindly provided by Jilin University, Changchun, P.R. China. Its molecular Weights is $Mn = 30000$ (data supplied by supplier of PES resin). Prior to use in experiments, PES pellets (diameter ≤ 3 mm) were dried in a vacuum furnace at 150°C for 4 h to remove any absorbed moisture.

2.2 Tests and Measurements

Melt rheological properties of PES were evaluated on a capillary instrument attached to a Shimadzu Universal Materials Testing Machine model AG-10TA. A capillary die of 1 mm in diameter and 10 mm in length was used. The applied pressure forces, F, were recorded on the Shimadzu at loading speeds, V, of 5, 10, 20, 50, 80, 100, 150, 200, 300 and 500 mm/min and at temperatures 315, 330 and 350°C.

Kneading of PES performed on an Haake Torque Rheometer model SYS 40, fitted with a RHEOMIX 600 mixer, at a set temperature of 335°C and at a rotor speed of 15, 25 and 40 rpm for 1 h. The torque required to turn the Haake and the material temperature were recorded and processed automatically by a microcomputer attached to the Haake as a function of time.

2.3 Rheological Analysis

The capillary rheological data were processed as follow:

The apparent shear rate, γ'_w, were expressed by

$$\gamma'_w = 4Q/\pi R^3$$

where R is the capillary die radius ($=0.5$ mm) and Q is the volumetric flow rate which can be expressed as

$$Q = \pi R_p^2 V$$

where R_p is barrel radius ($=5$ mm) and V is the loading Speed (mm/min).

Thus, the apparent shear rate could be calculated from

$$\gamma'_w = 4\pi R_p^2 V/\pi R^3 = 40V/3 \text{ (l/s)}$$

The shear stress at the wall of capillary were calculated by

$$\tau_w = R\Delta P/2L = 318.3F \text{ (Pa)}$$

where F is the applied pressure force (N), L is the capillary die length ($=10$ mm) and ΔP is the pressure differential on the end of capillary die. So, the apparent viscosity, η_a, were calculated with the value of the apparent shear rate, γ'_w, and shear stress, τ_w, as follow

$$\eta_a = \tau_w/\gamma'_w = 23.873F/V \text{ (Pa·s)}$$

and the equation used for calculation of the power law index, n, is expressed as

$$n = \delta \log \tau_w/\delta \log \gamma'_w$$

The Bagley and Rabinowitsch corrections were not applied to the capillary rheometry data.

3. RESULTS AND DISCUSSION

3.1 Shear Rheometry of PES Melt

3.1.1 Flow curves. Viscosity curves of PES measured at 315, 330 and 350°C and for shear rates from 10 to 10000 l/s are presented in Figure 1. A typical pseudoplastic behavior can be seen. That is the melt viscosites of PES decrease with the increase of apparent shear rates.

The shear stress vs. rate plots of molten PES are shown in Figure 2. As expected, due to the strong non-Newtonian behavior, the variations of log (shear rate) with log (shear stress) are not linear (Figure 2). It suggests that the power law index, n, will change with the shear rate. The power law index values calculated are presented in Table I.

The power law indices, n, in Table I are close to the value ($n = 0.53$) reported by Saini.[5] As can be seen, the values of n decrease with decrease in temperature and increase in shear rate. In other words, the lower the temperature and the higher the shear rates were, the more non-Newtonian behavior of the molten PES would be. It is well consistency with Saini.[5]

3.1.2 Factors affecting rheological properties of PES. The temperature dependence of the viscosity was expressed in terms of an Arrhenius equation

$$\eta_a = A \exp(E/RT) \quad \text{or} \quad \log \eta_a = \log A + (E/2.303R) * (1/T)$$

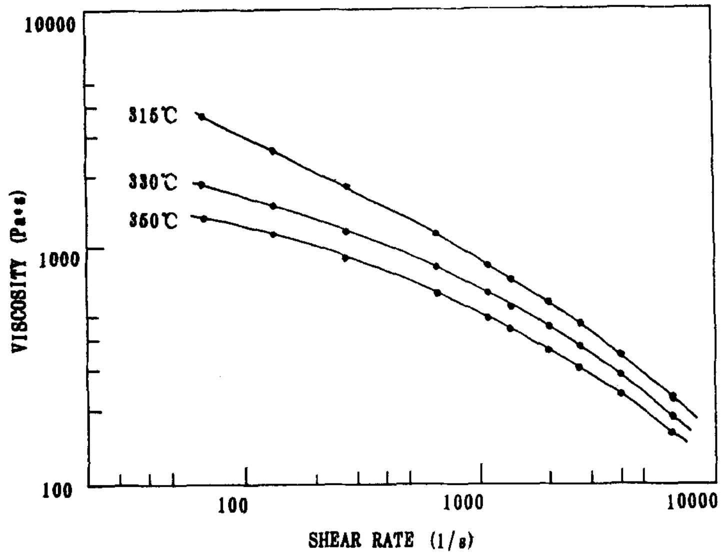

FIGURE 1 Viscosity vs. shear rate for PES.

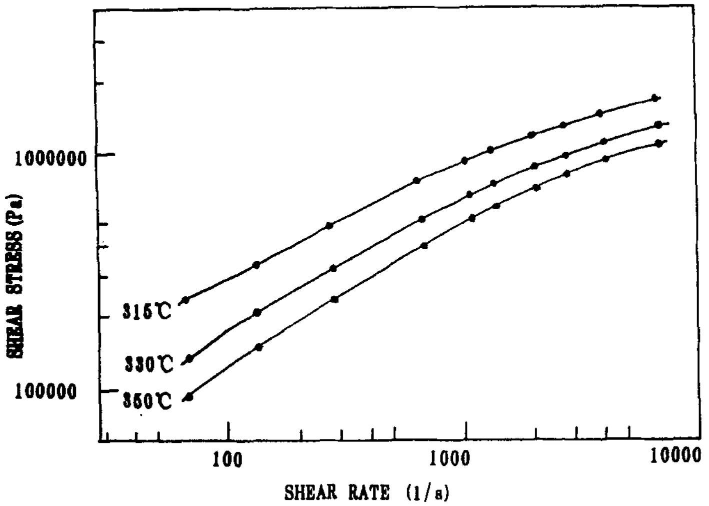

FIGURE 2 Shear stress vs. shear rate for PES.

The flow activation energy (J/mol), E, for viscous flow can be measured from log (apparent viscosity) vs. $1/T$ plots, where T is the absolute temperature (K) and R is a constant ($=8.314$ J/K·mol). The activation energy of viscous flow is found to decrease with increase in shear rate (Figure 3). But it can be seen that viscosity of molten PES has a weak sensitivity to temperature, because of its fairly low viscous flow activation energy.

As seen above, the viscosity of PES decreases with increase in shear rate. PES consists of semi-rigid molecular chains which has strong effect on its rheological behavior. With a semi-rigid-chain PES, the decrease in apparent viscosity with

TABLE I

Flow index values of PES

Temperature (℃)	Flow index values at shear rates of	
	10—999 1/s	1000—10000 1/s
315	0.50	0.33
330	0.58	0.41
350	0.63	0.40

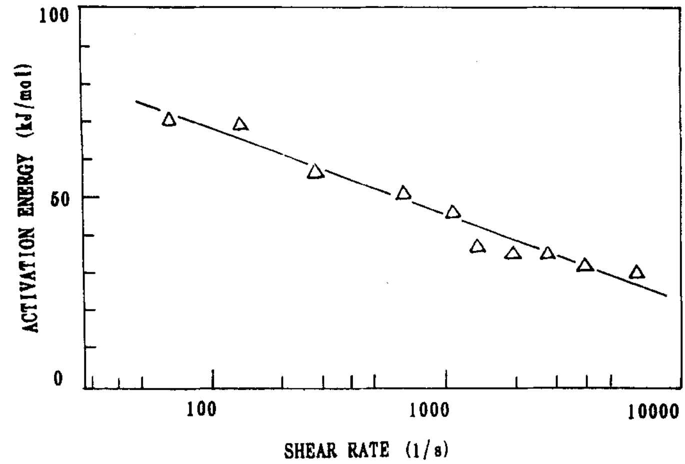

FIGURE 3 Variation of viscous flow activation energies with shear rate.

increasing shear rate, is less than that of flexible-chain polymer, but higher than that of a rigid-chain polymer.

3.1.3 Effect of rheological behavior on molding. In plastics processing, adequate melt viscosities are required to make products using different processing methods. By adjusting the molding conditions, the processing flexibility can be enhanced.

Because of its molecular chain structure, the viscosity of PES is not very sensitive to changes in temperature or shear rate. So the flow performance cannot be increased effectively by simple increases in shear rate (screw rpm) or increases in barrel temperature.

3.2 Torque Rheological Property of PES Melt

The rheological characterization of the PES resin was made by using capillary and torque rheometer. The torque-temperature-time diagram of PES at a set temperature of 335°C are presented in Figure 4. The results of kneading test are summarized in Table II.

Torque rheometer produces a flow curve that is a measure of the polymer's processability.[6,7] In Figure 4, the curve of TQ and T2 represent the relationship between torque and temperature with time. The point of L, S and D were the times of loading, stabilization and degradation of PES, recorded by Haake, during kneading. It can be seen from Figure 4, that the torque upon loading of PES is quite large, but stabilized to a relative small value. So, the energy consumption of molding machine during processing of PES would not be very large. This has been proved by our extrusion and injection molding tests.[7,8] The time of relative high torque (about point L) is very short.

At the present condition, the torque and temperature stabilize to a constant value in about 5 min (Figure 4). This suggests that in order to obtain a well molten PES, the molding machine with a high length to diameter ratio, *L*/*D*, twin-screw and/or high pressure should be used.

At time (point D) of over 40 min, a considerable increase of torque is observed. The color of PES products turns black and energy consumption of machine arises

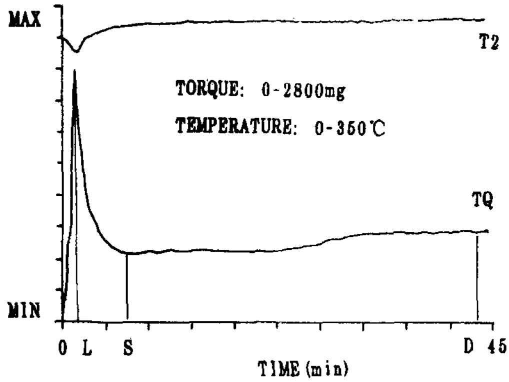

FIGURE 4 Record torque (in meter-gram) vs. the time at a set temperature 335°C and at 15 rpm.

TABLE II

Results of kneading tests

Set Temperature (℃)	Kneading time (min)	Equil. torque (mg)*	Ultimate torque (mg)*
335	50	552	906
335	50	752	996
335	50	968	1329

* Unit conversion: 1mg=9.807×10^{-3}N

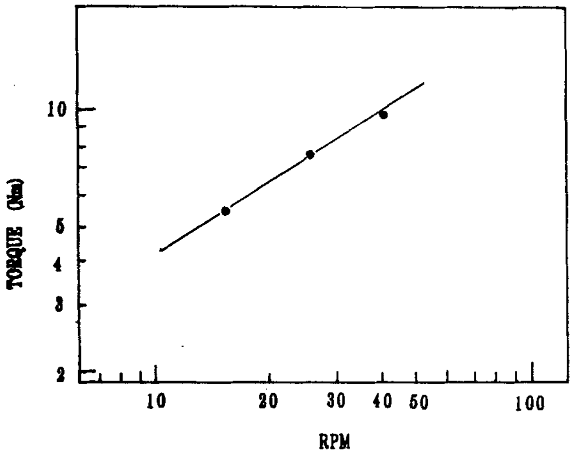

FIGURE 5 Variation of torque with rpm at 335°C.

during injection and extrusion molding, if the melt has a long residence time in the barrel.[8] The increase in melt viscosity with the increase in processing time, caused by prolonged shear, heat and oxidation is different from the thermal degradation. This phenomenon had been named "thickening of melt during processing of PES" by present authors.[7]

There is a relationship[9] between torque (Nm), M, and rotor speed (rpm), S,

$$M = CS^{\alpha}$$

where C and α are constants. This relationship resembles the familiar power law and can be used for calculation of the power index, n. Figure 5 shows variation of equilibrium torque (torque at point S as in Figure 4) vs. rpm at 335°C. A power index obtained from Figure 5 is $n = 0.58$, which is consistent with the data of Saini[5] and capillary rheological results presented above.

4. CONCLUSIONS

Capillary rheological experiments show that molten PES, as a typical pseudoplastic fluid, has a small non-Newtonian index ($n = 0.33$–0.63) and a rather low viscous flow activation energy ($E = 30$–70 kJ/mol). And hence, the shear rate dependence and the temperature dependence of the viscosity are both not obvious.

According to the torque-temperature-time plots measured under industrial relevant processing condition, the energy consumption during molding would be acceptable. But the residence time of resin in the barrel must be less than 40 min. A thickening of melt during processing of PES takes place.

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