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https://doi.org/10.5109/23952

出版情報：九州大学大学院農学研究院紀要. 35 (1/2), pp.73-79, 1990-12. Kyushu University
バージョン：
権利関係：

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J. Fac. Agr., Kyushu Univ., 35 (1•2), 73-79 (1990)

Development and Evaluation of an Extrusion Viscometer for Polymer Melt

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(Received July 30, 1990)

An extrusion viscometer with a closed reservoir-capillary system (a completely air-tight system) was designed in order to determine the flow property of polymer melt containing any substance with high vapor pressure at high temperature. The performance of this developed extrusion viscometer was evaluated using polystyrene melt at 170°C. No quantitative difference was found between a generalized flow curve obtained in this work and that of published results. From the performance rating obtained from the developed extrusion viscometer, its value for practical usage was sufficiently recognized for the determination of the flow property of polymer melt.

INTRODUCTION

Many types of rheometers or extrusion viscometers equipped with a capillary have been developed and used to evaluate the flow property of polymer melt. These viscometers/rheometers can be characterized by the method of pressure loading upon pdlymer melt or highly viscous material in a reservoir : (1) pressure being yielded in an extrusion screw (Jao and Chen, 1978 ; Padmanabhan and Bhattacharya, 1989), (2) mechanically driven plunger (Collins and Krier, 1966 ; McLuckie and Rogers, 1969 ; Kamal and Hyun, 1973 ; Chee et al., 1976), (3) high pressure gas directly on polymer melt (Severs and Austin, 1954 ; Lanieve and Bouge, 1968), (4) high pressure gas on a plunger mounted reservoir (Bagley, 1957 ; Nakajima and Shida, 1966 ; Rudin and Leeder, 1968), (5) hydraulic pressure in a plunger mounted reservoir (Takahashi et al., 1985) and (6) weight upon a plunger (Bartos and Holomek, 1971 ; Fujiyama and Kagiyama, 1972). In the case of such viscometers, the pressurized polymer melt contained in a reservoir, flows through a capillary from an inlet and discharges to an open outlet. The main disadvantage of such viscometer/rheometer is that the polymer melt can pass only once through a capillary and consequently dis-chargs to an open outlet thus the polymer melt containing high vapor pressure substances such as water at a high temperature are not applicable. From a practical point of view it must be necessary to develop a viscometer/rheometer for polymer melt containing any substance with high vapor pressure.

The objective of this work was to design an extrusion viscometer with completely air-tight reservoir-plunger-capillary system, and then to evaluate in detail the performance of the developed extrusion viscometer using polystyrene melt as the standard

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polymer.

MATERIALS AND METHODS

Polystyrene

The polystyrene (additive-free, PS) was purchased from Katayama Chemical Co. Ltd. (Tokyo, Japan). This PS is of number-average molecular weight (Mn) of 97000, weight-average molecular weight (Mw) of 261000, and glass transition temperature (Tg) of 90°C.

Extrusion viscometer and operation

Figure 1 shows the schematic diagram of the developed extrusion viscometer which was designed by our laboratory and assembled by Shinmeiwa Co. Ltd. (Nishinomiya, Japan). Two sample reservoirs (cylinders) with 11.28 mm inner diameter (cross-sectional area, 1 .O cm^2) mounted in a plunger are firmly fixed between both sides of capillary die. Both plungers were synchronously driven by hydraulic cylinders connected in both plungers. The maximum stroke of the plunger was available at 60 mm. Five capillary dies with differrent inner diameter (2R= 1.0, 1.5, 2.0, 3.0 and 4.0 mm ; R=radius) were available and their length (L) were the same at 20 mm. Each capillary die corresponded to L/R ratios of 40.0, 26.7, 20.0, 13.3 and 10.0, respectively. Temperature (up to 200°C) of the sample reservoirs and capillary was adjustable accordingly within ± 0.2℃ by an electric heater with a preset PID controller (model DB-1, Chino Co. Ltd., Tokyo, Japan). The traveling speed of plungers and pressure applied on both plungers can be controlled by the flow rate and the pressure from a hydraulic pressure pump. Traveling speed and the pressure applied on both plungers were also detected and monitored by a displacement trans-

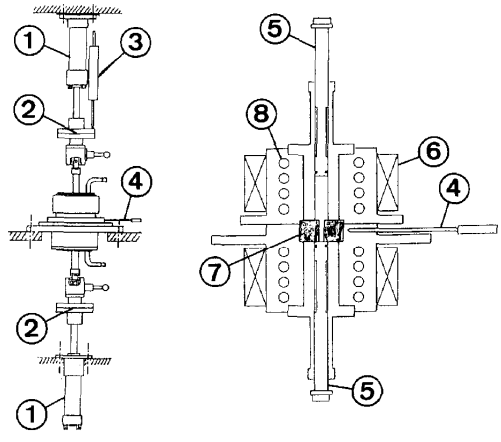

Fig. 1 Assembly of experimental apparatus
1; Hydraulic cylinder 2 ; Pressure transducer 3 ; Displacement transducer
4 ; Thermo-regulator probe 5 ; Plunger 6 ; Electric heater 7 ; Capillary tube
8 ; Cooling water channel

ducer (model DB-100, Toyo Measuring Instruments Co. Ltd., Tokyo, Japan) and a pressure transducer (model LM-2T, Kyowa Electronic Instruments Co. Ltd., Tokyo, Japan), respectively. The responses of both transducers were amplified and recorded against elapsed time on a multi-pen recorder (model 3056, Yokogawa Electric Co. Ltd., Tokyo, Japan). Pressure drop when polymer melt passes through the capillary was measurable up to 100 MPa. Viscometry of PS melt (4. Og in the reservoir) took place at 170℃, using the five capillary dies and by various traveiling speeds of the plunger.

RESULTS AND DISCUSSION

Response of pressure transducer

Extrusion viscometry tests of PS melt at 170°C were done. Figure 2 shows the typical response charts of pressure transducer at the same traveling speed using three capillary dies having 1.0, 2.0 and 3.0 mm diameter. The recorded responses fluctuated against time, particularly in 1.0 mm die. The average pressure difference, AP, then was determined by visual inspection. From repeated measurements (10 times) under the same conditions, the responses were statistically treated and the results for two different flow rates are reported in Table 1. The average value, $\bar{x}$, the

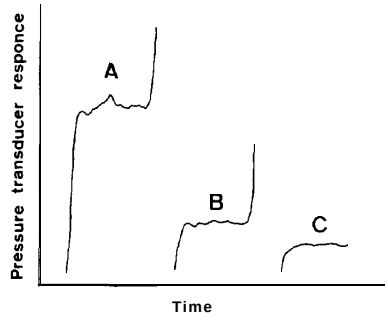

Fig. 2 Typical response chart
Capillary diameter : A ; 1.0mm, B ; 2.0mm, C ; 3.0mm Capillary length ; 20mm

Table **1.** Statistical analysis on meaured pressure drop.

Capillary diameter (mm)	Flow rate (m^3/sec)	Measured pressure drop (MPa) $\bar{x}$[1]	σ[2]	ψ[3]
1.0	8.51×10^{-7}	44.27	1.00×10^{-1}	2.26×10^{-3}
	1.05×10^{-8}	22.44	2.00×10^{-1}	8.99×10^{-3}
2.0	9.40×10^{-7}	14.99	9.61×10^{-2}	6.54×10^{-3}
	9.26×10^{-8}	6.97	6.50×10^{-2}	9.66×10^{-3}
3.0	9.72×10^{-7}	9.92	6.74×10^{-2}	6.79×10^{-2}
	9.18×10^{-8}	3.60	7.84×10^{-2}	2.18×10^{-2}

1) average, 2) standard deviation, 3) coefficient of variation

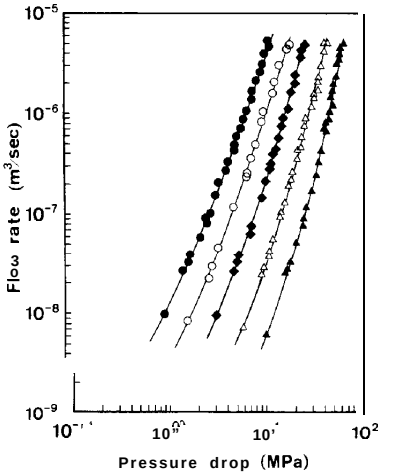

Fig. 3 Relationship between pressure drop and volume flow rate for PS melt through various capillaries at 170°C.
Capillary diameter : ▲;1.0mm, △;1.5mm, ● ;2.0mm, ○;3.0mm, ● ;4.0mm

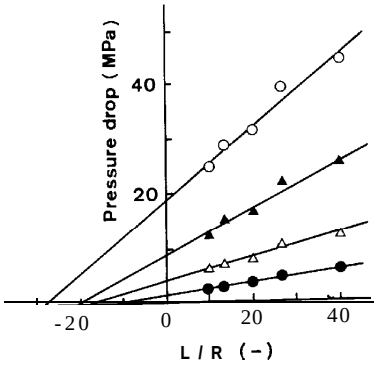

Fig. 4 Pressure drop plotted against L/R at fixed shear rate for PS melt. Temperature was 170°C.
Shear rate [sec^{-1}] : ●;1×10^{1}, △; 1×10^{2}, ▲; 1×10^{3}, ○; 1×10^{4}

standard deviation, σ, and the coefficient of variation, ψ, were calculated out of 10 observations. The observed pressure drop on the present viscometry method are confidently accurate because of the considerably small value of ψ.

AP against flow rate

Figure 3 demonstrates all observed data, namely, volumetric flow rates ranging from 1×10^{-8} to 6×10^{-6} m^3/sec and resultant AP ranging from 1 to 60 MPa, plotted on log-log scale. Flow curves for PS melt gave non-linear curves on a log-log scale and

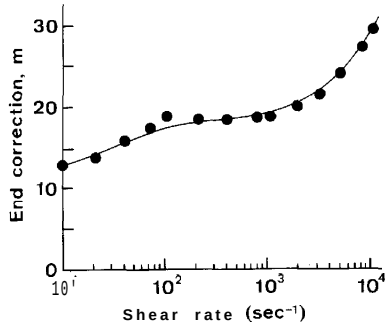

Fig. 5 Dependence of end correction on shear rate for PS melt at 170°C.

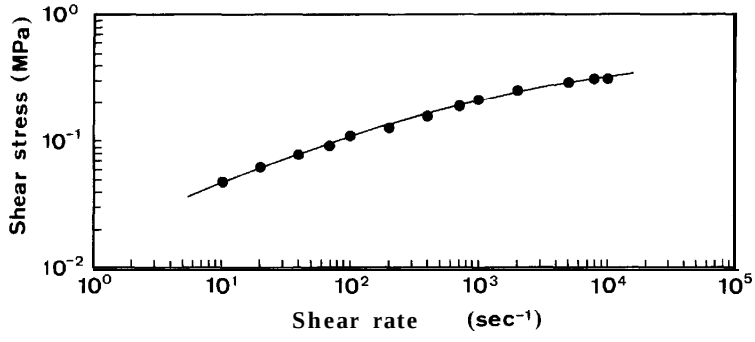

Fig. 6 Bagley corrected flow curve for PS melt at 170°C.

shifted horizontally in proportional order to capillary diameter applied.

Bagley's end effect correction (Bagley, 1957)

The observed AP, as shown in Fig. 3, included an additional AP due to entrance and end effects (Hyun, 1974). In order to obtain net AP passed through a capillary used, end correction was applied to the series of observed data in accordance to Bagley's (1957) theory. Figure 4 represents a series of relationship between AP vs. L/R. By extrapolation of such linear plot to AP=O, the value of m (end correction) for that particular shear rate was obtained. Figure 5 shows the relationship between the value of m and logarithm of the Newtonian shear rate at the wall.

Corrected flow curve for polystyrene melt

Using the appropriate end correction from Fig. 5, the curves of Fig. 3 were replotted by the relationship between shear rate and shear stress as shown in Fig. 6. As a result, the curves corresponding to five capillary dies in Fig. 2 were reduced to a single curve. This result for PS melt is relatively similar to that of polyethylene melt (Bagley, 1957) and polypropylene melt (McHugh, 1972). On the basis of the convexity of the curve in Fig. 6, the flow property of PS melt at 170°C can be characterized as that of a pseudplastic fluid independent of power-low within the limit

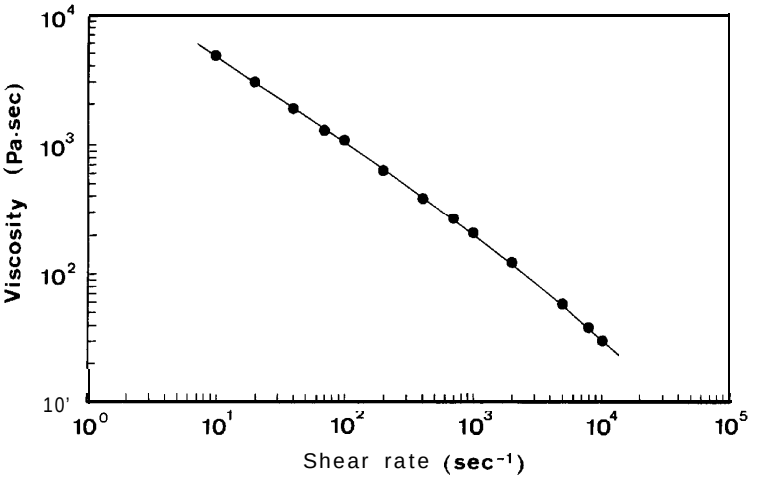

Fig. 7 Relationship between **shear** rate and viscosity for PS melt at 170°C.

of shear rate ranging from 10' to $10^4(\sec^{-1})$.

Dependency of apparent viscosity on shear rate

The fluid property developed during the capillary flow, characterized for the PS melt by the apparent viscosity, can be defined as follows :

$$\eta = \tau_w / \dot{\gamma}$$

where $\eta, \dot{\gamma}$ and τ_w were apparent viscosity (Pa · sec), shear rate $(\sec^{-1})$ and shear stress at wall (Pa), respectively. The apparent viscosity of PS melt can be calculated from the observed τ_w at the corresponding $\dot{\gamma}$ and the results were plotted on a log-log scale. As shown in Fig. 7, the apparent viscosity of PS melt considerably depended on the magnitude of shear rate within the limit of the present experiment. Such findings on the apparent viscosity gives a good agreement with the reported data of PS melt (Hyun and Karam, 1969, Kamal and Nyun, 1973). It was then concluded that the developed extrusion viscometer could be one of the useful tools for viscometry of polymer melt.

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