KSME International Journal, Vol. 16 No. 2, pp. 255~261, 2002

Continuous Viscosity Measurement of Non-Newtonian Fluids over a Range of Shear Rates Using a Mass-Detecting Capillary Viscometer

Sehyun Shin*, Do-Young Keum
School of Mechanical Engineering, Kyungpook National University, Daegu 702-701, Korea

A newly designed mass-detecting capillary viscometer uses a novel concept to continuously measure non-Newtonian fluids viscosity over a range of shear rates. A single measurement of liquid-mass variation with time replaces the flow rate and pressure drop measurements that are usually required by capillary tube viscometers. Using a load cell and a capillary, we measured change in the mass flow rate through a capillary tube with respect to the time, $m(t)$, from which viscosity and shear rate were mathematically calculated. For aqueous polymer solutions, excellent agreement was found between the results from the mass-detecting capillary viscometer and those from a commercially available rotating viscometer. This new method overcomes the drawbacks of conventional capillary viscometers meassuring non-Newtonian fluid viscosity. First, the mass-detecting capillary viscometer can accurately and consistently measure non-Newtonian viscosity over a wide range of shear rate extending as low as $1\ s^{-1}$. Second, this design provides simplicity (i. e., ease of operation, no moving parts), and low cost.

Key Words: Viscosity, Non-Newtonian Fluids, Shear Rates, Capillary Viscometer

Nomenclature

h : Height
Lc : Capillary length
m : Mass
$\dot{m}$: Mass flow rate
n : Power-law index
P : Pressure
Q : Volume flow rate
t : Time

Greek Symbols

ρ : Density
Φ_C : Capillary diameter
Φ_F : Falling tube diameter
η : Non-Newtonian viscosity
μ : Newtonian viscosity
γ : Shear rate
τ : Shear stress

Subscripts

C : Capillary tube
e : Entrance & and exit
F : Falling tube
i : Initial
w : Wall
∞ : Final or infinite

1. Introduction

The measurement of viscous flow by capillary viscometry is firmly established both theoretically and experimentally (Middleman, 1968, Bird et al., 1987 and Macosko, 1993). In fact, the capillary viscometer was the first viscometer, and this device remains the most commonly used for measuring viscosity for polymer solutions and other non-Newtonian fluids because of features such as simplicity, accuracy, similarity to process flows like extrusion dies, and no free surface. However, most existing capillary viscometers

---

* Corresponding Author,
E-mail : shins@knu.ac.kr
TEL : +82-53-950-6570; **FAX** : +82-53-956-9914
School of Mechanical Engineering, Kyungpook National University, 1370 Sankyuk-dong Buk-gu Daegu 702-701, Korea. (Manuscript **Received** July 31, 2001; **Revised** November 5, 2001)

produce viscosity measurement one shear rate at a time. For Newtonian fluids, the flow rate for a giver pressure drop is sufficient to define the flow behaviour. However, for non-Newtonian fluids viscosity measurements should be performed over a range of shear rates.

To measure viscosity over a range of shear rates, one needs to repeat measurement by varying either driving pressure head or capillary tube diameter, which leads to a time-consuming process requiring intensive labor. Hence, currently available capillary viscometers are not suitadle for measuring the rheology of polymer fluids that may exhibit shear-dependent viscosities. Furthermore, application of such techniques often requires relatively large volumes of the test fluids. Therefore, there has been a need to develop a simple and labor-free viscometer, which can measure the viscosity of fluids over a range of shear rates in a short period of time.

Recently, Cho and his co-workers (Cho et al., 1999 and Kim et al., 2000) introduced a new scanning-capillary-tube viscometer that uses a charge-coupled-device sensor array to measure the viscosity of Newtonian and non-Newtonian fluids. This scanning-capillary-tube viscometer can produce viscosity data for a low shear range by extending the shear rate range as low as 5 s^{-1} for water and diluted glycerine solutions (Cho et al., 1999), 0.1 s^{-1} for EDTA-anticoagulated bovine blood at room temperature and, 1 s^{-1} for unadulterated human blood at body temperature (Kim et al., 2000).

The present study introduces an innovative approach to capillary-tube viscometry. A single measurement of liquid-mass variation with time provides the ability to measure fluid viscosity continuously over a range of shear rates. Throughout the development of this technique emphasis has been placed on the simplicity of design that would result in ease of operation, no moving parts, and low cost while allowing measurements to be made over a broad range of shear rates. In order to demonstrate the validity of the this new mass-detecting capillary viscometer, its viscosity data were compared with data obtained from a rotating viscometer. Also, the accuracy of the new instrument was demonstrated by measuring the viscosity of mineral oil and comparing the results with its reference value.

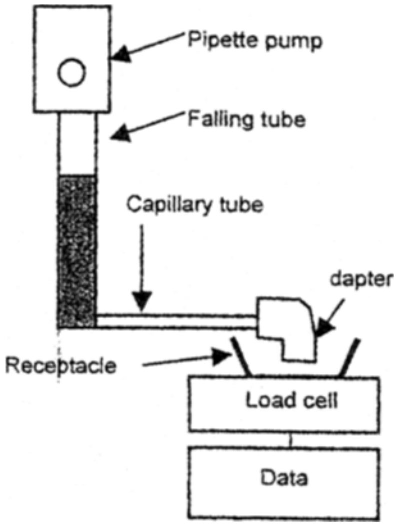

Fig. 1 Schematic diagram of the mass-detecting capillary viscometer system

2. Method

2.1 Description of instrument

Figure 1 is a schematic diagram of the mass-detecting capillary viscometer, which consists of a pipette pump, a falling tube, a glass capillary tube, a glass adapter, a receptacle, a load cell, and a computer data acquisition system. The inside diameter of the falling tube was $\Phi_F==8$ mm. The inside diameter and length of the capillary tube were $\Phi_C=1.08$ mm and $L_C=100$ mm, respectively. The diameter and length of the capillary tube were chosen to ensure that the friction loss in the capillary tube was significantly greater than the loss in the other parts of the system (Cho et al., 1999). Capillary end effects were accounted for during data reduction by adjusting the values of the length of the capillary tube. In addition, the capillary tube dimensions were designed to finish one test with water within a minute.

The essential feature in the mass-detecting capillary viscometer is the use of a precision mass balance to measure the fluid collected in the receptacle, m(t), every 0.04s with a resolution of 0.01 gram. The instantaneous fluid weights were recorded in a computer data file through an analog-to-digital data acquisition system with respect to time.

2.2 Testing procedure

Typical tests are conducted as follows: The pipette pump sucks up the test fluid from the receptacle so that the initial height of the fluid in the falling tube reaches a preset position. Once this condition is achieved, the pipette pump stops the fluid flow until the start of the test. At time t = 0, the data acquisition system is enabled and the pipette pump is opened to atmosphere, allowing the fluid to flow through the capillary and to be collected in a receptacle as. When the fluid level in the falling tube approaches the bottom of the falling tube, the test fluid stops flowing.

Typically, it took approximately a minute for the fluid level in the falling tube to reach settle with water. The time to complete a run varies depending on type of liquid and size of falling and capillary tubes. If a larger-diameter falling tube is used, longer run time is required. For a fixed capillary tube, for example, it took 30 minutes for water with a 30 mm diameter falling tube, but only 4 minutes with a 10 mm diameter falling tube. However, using a falling tube with a larger diameter resulted in more accurate data.

2.3 Test fluids and comparison instrument

Aqueous solutions of commercial polyacrylic acid (Carbopol-934, Dow Chemical Company, Midland, Michigan) and polyacrylamide (Separan AP-273, Dow Chemical Company, Midland, Michigan) were chosen as test fluids because they are commonly used thickeners in the chemical industry and related fields (Shin and Cho, 1993). Only one concentration of 1, 000 wppm solutions was studied.

Both polymers were well dissolved into distilled water. First, approximately half of the required amount of distilled water was poured into a beaker. A predetermined amount of polymer was then mixed with water, while the water was gently stirred with a paddle. Then, the remainder of the required water was added to the beaker. In order to make homogeneous solutions, the solutions were mechanically stirred at low speed. To increase its viscosity the Carbopol solution was neutralized with a 10% sodium hydroxide solution, as recommended by the manufacturer. The sodium hydroxide solution was added to the Carbopol solution by drop from a calibrated burette until a pH of 7±0.2 was obtained.

Mineral oil, purchased as a viscosity standard fluid (4.9 cP at 25 ℃) from Brookfield Engineering Laboratories, Inc., was used for calibration. For comparison, the viscosities of the mineral oil and the polymer solutions were also measured by using a rotating viscometer (Physica UDS-200, Paar Physica USA, Inc. , Glen Allen, Virginia).

2.4 Data analysis

The fluid mass data from the receptacle were analysed in the following way to determine viscosity. The mathematical model of the flow analysis began with application of energy conservation between the fluid level in the falling tube and datum. Assuming a quasi-steady flow behavior, one may write the governing equations as follows (Munson et al, 1998):

$$\left\{ P_1+\frac{1}{2}\rho V_1^2+\rho g h_1(t) \right\} - \left\{ P_{datum}+\frac{1}{2}\rho V^2{}_{datum}+\rho g h_{datum} \right\} = \Delta P_c(t)+\Delta P_e(t)+\rho g \Delta h_\infty \qquad (1)$$

where P is the static pressure, ρ the density of the test fluid, V the fluid velocity, g the acceleration due to gravity, h the fluid level, ΔP_c the pressure drop across the capillary tube, ΔP_e the pressure drop occurring at the entrance and exit of the capillary tube, and the subscripts 1 and datum are in the falling tube and at datum, respectively. The third term in the right hand side in Eq. (1) represents the residue height at the falling tube at t = ∞ due to surface tension effect.

With this experimental set-up, the pressure drop (ΔP_e) caused by secondary flow patterns or eddies in the entrance and exit of the capillary tube may appear to be significant in a high shear zone. One accurate method for determining ΔP_e is to make a Bagley plot with at least two shot capillaries of the same diameter (Macosko, 1993). The contribution from the second term on the right hand side in Eq. (1) is less than 0.5%; and this term can be neglected for all practical purposes (Cho et al., 1999).

Since $P_1 = P_{datum}$ (static ambient pressure), $|V_1| = |V_{datum}|$, and $h_{datum} = 0$, Eq. (1) can be simplified as

$$\Delta P_C(t) = \rho g[h_1(t) - h_\infty] = \rho g[h_i - h_\infty - \Delta h_1(t)] \quad (2)$$

where h_i is the initial fluid level at t = 0, h_∞ is the final fluid level at t = ∞, and $\Delta h_1(t)$ is the fluid level difference between h_i and $h(t)$. In addition, Eq. (2) can be expressed as a function of fluid mass collected in the receptacle as follows:

$$\Delta P_C(t) = \frac{4g}{\pi \Phi_F^2}[m_\infty - m_i - m(t)] \quad (3)$$

where Φ_F is falling tube diameter, mi the fluid mass at t = 0, and m_∞ the fluid mass at t = ∞. It is of note that the volume flow rate is proportional to the rate of change of the mass of the fluid collected on the load cell. Hence, the corresponding flow rate in the capillary tube can be expressed as

$$Q(t) = \frac{1}{\rho}\frac{dm}{dt} \quad (4)$$

2.4.1 Newtonian fluids

The Hagen-Poiseuille flow may be used to derive the following relationship for the pressure drop at the capillary tube as a function of capillary tube geometry, fluid viscosity, and flow rate (Kim et al., 2000):

$$\Delta P_C(t) = \frac{128\mu L_C Q(t)}{\pi \Phi_C^4} = \frac{128\mu L_C \dot{m}(t)}{\pi \rho \Phi_C^4} \quad (5)$$

where Φ_C is the diameter of the capillary tube, L_C is the length of the capillary tube, $\dot{m}$ is the mass flow rate, and μ is the viscosity of the fluid. Subsequently, the shear rates (Eq. 6) and the shear stress (Eq. 7) at the capillary tube wall and viscosity (Eq. 8) can be determined for Newtonian fluids (Macosko, 1993).

$$\dot{\gamma}_w(t) = 32\frac{Q(t)}{\pi \Phi_C^3} = 32\frac{\dot{m}(t)}{\pi \rho \Phi_C^3} \quad (6)$$

$$\tau_w(t) = \frac{\Delta P_C \Phi_C}{4L} = \frac{g\Phi_C}{\pi L \Phi_F^2}[m_\infty - m_i - m(t)] \quad (7)$$

$$\mu = \frac{\rho g \pi \Phi_C^4}{32 L_C \Phi_F^2}\left(\frac{m_\infty - m_i - m(t)}{\dot{m}(t)}\right) \quad (8)$$

2.4.2 Non-Newtonian fluids

The shear rate dependent viscosity for a non-Newtonian fluid flowing in the capillary tube is obtained from experimental data with some mathematical treatment, and the necessary equations can be found in any standard handbook (Bird et al., 1987 and Macosko, 1993). The shear rate at the capillary tube wall is obtained from the classical Weissenberg-Rabinowitsch equation for generalized non-Newtonian fluids (Macosko, 1993),

$$\dot{\gamma}_w(t) = -\left.\frac{dV_z}{dr}\right|_{r=R} = \frac{1}{4}\dot{\gamma}_{aw}\left[3 + \frac{d\ln Q}{d\ln \tau_w}\right] \quad (9)$$

where $\dot{\gamma}_{aw}$ is the apparent or Newtonian shear rate at the wall.

$$\dot{\gamma}_{aw}(t) = \frac{32\dot{m}(t)}{\rho \pi \Phi_C^3} \quad (10)$$

The shear stress at the wall is given by

$$\tau_w(t) = \frac{g\Phi_C}{\pi L \Phi_F^2}[m_\infty - m_i - m(t)] \quad (11)$$

Thus, the viscosity corresponding to the wall shear rate is calculated in the form of a generalized Newtonian viscosity:

$$\eta = \frac{\tau_w}{\gamma_w} = \frac{\rho g \Phi_C^4}{8 L_C \Phi_F^2}\frac{[m_\infty - m_i - m(t)]}{\dot{m}(t)}\left(3 + \frac{d\ln Q}{d\ln \tau_w}\right)^{-1} \quad (12)$$

Equation (2) is the stating point of most of the existing methods of processing capillary viscometry data. A key step in these methods is the differentiation of the log-log plot of Q against τ_w. Due to the limited experimental data and unavoidable noise, the numerical value of the derivative can be very sensitive to the differentiation method e.g a local least-squares polynomials or an interpolating spline (Nguyen et al., 1999)). However, if there are enough data near the point of interest, it is possible to evaluate

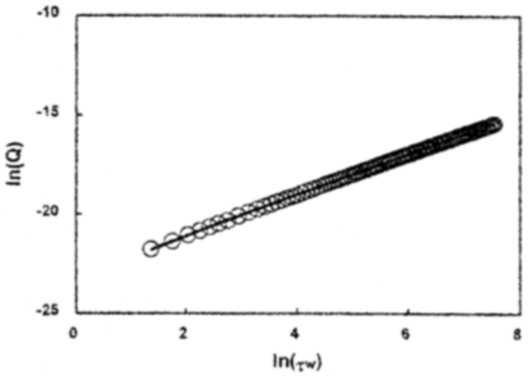

Fig. 2 Log-log plot of volume flow rate(Q) against wall shear stress(τ_w) for mineral oil

the derivative $d\ln Q/d\ln \tau_w(=1/n')$ where n' is simply the exponent of the power law constitutive equation.

Figure 2 shows a typical log-log plot of volume flow rate (Q) against wall shear stress (τ_w). The typical number of data points in the mass-detecting capillary viscometer is about 1,000 over a range of shear rates. Therefore, even if there is unavoidable noise in the data, the numerical value of the derivative can be stable regardless of the differentiation method. Even though the power-law exponent is used in the above equations, this does not limit the capability of the present measurement for power-law fluids. The rigorous approach can still be taken for obtaining a viscosity versus shear rate relationship for any fluid (Macosko, 1993). Thus, Eq. (12) can be described in terms of the mass measured in the mass-detecting capillary viscometer as follows:

$$\eta=\frac{\rho g \Phi_C^4}{8 L_C \Phi_F^2}\frac{[m_\infty-m_i-m(t)]}{m(t)}\left(\frac{n'}{3n'+1}\right) \quad (13)$$

The viscosity versus shear rate information can be obtained from Eqs. (9)-(13) by measuring the mass of the collected fluid with respect to time, from which the pressure drop and flow rate can be calculated. The values of Φ_C and L_C must be obtained by calibration. Since Eq. (13) is non-linear, the procedure to calculate the shear rate and the corresponding viscosity is not straightforward. One of the approaches to obtain the viscosity from the general equations presented above is to adopt a finite difference technique for differentiation of Eq. (13).

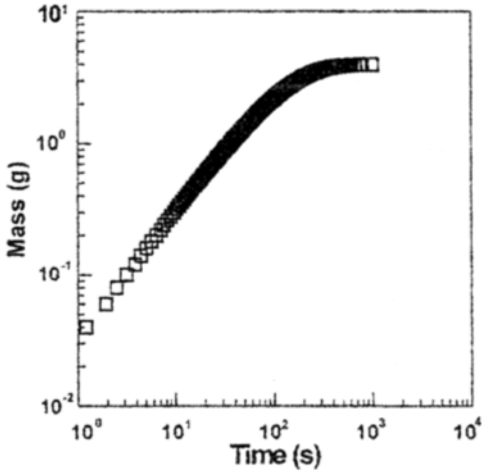

Fig. 3 Mass variations of fluid vs. time for mineral oil measured with mass-detecting capillary viscometer

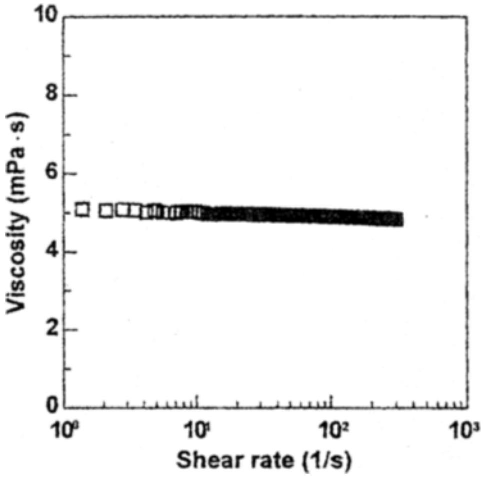

Fig. 4 Viscosity measurement for mineral oil with mass-detecting capillary viscometer

3. Results and Discussion

Figures 3 and 4 provide test results obtained with mineral oil measured at room temperature (approximately 25℃) with the mass-detecting capillary viscometer. Figure 3 shows the mass variation of the collected fluid as a function of time: $m(t)$. As time passed, the collected fluid mass gradually increased and reached m_∞. It is

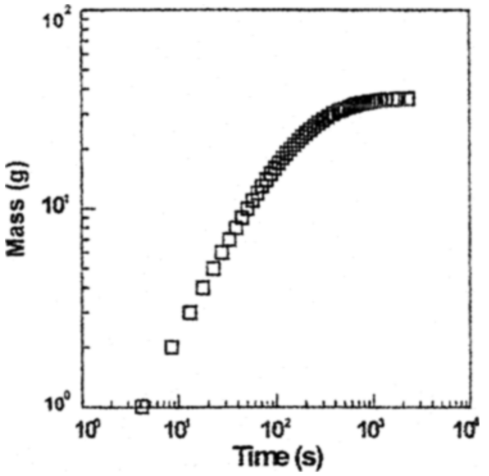

Fig. 5 Mass variations of fluid vs. time for aqueous polyacrylic acid solution (Carbopol-934; 1, 000 wppm) measured with mass-detecting capillary viscometer

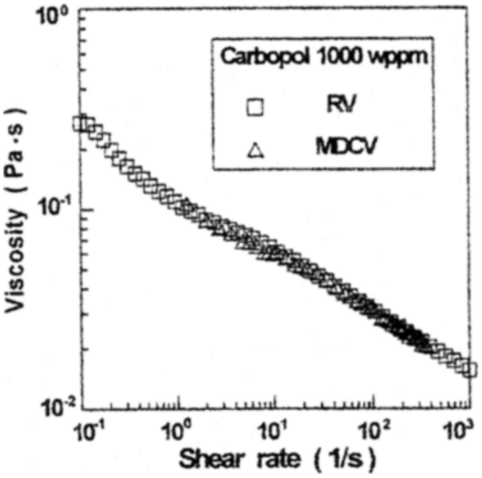

Fig. 6 Viscosity measurement for Carbopol solution with mass-detecting capillary viscometer (MDCV) and rotating viscometer(RV)

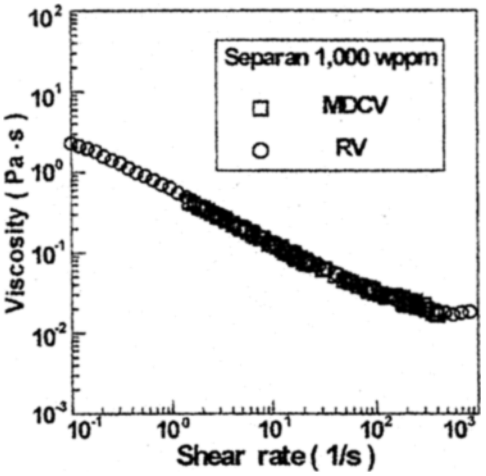

Fig. 7 Viscosity measurement for aqueous polyacrylamide solution (Separan AP-273; 1, 000 wppm) with mass-detecting capillary viscometer(MDCV) and rotating viscometer (RV)

important to note that the rate of change in the collected mass decreased with time. This was associated with the pressure differential decrease caused by the column of fluid falling toward the datum, subsequently resulting in a decreased volume flow rate. The viscosity of mineral oil was calculated from $m(t)$ using Eq. (7).

Figure 4 shows the viscosity of the mineral oil at room temperature obtained with the mass-detecting capillary viscometer. Based on our viscosity measurement method, the viscosity of the mineral oil was found to be between 4.88 and 4.92 cP at 25℃, which was within 0.5% difference in the whole range of shear rates ($1.38 \sim 300\ s^{-1}$) from the standard viscosity of 4. 9 cP at the same temperature.

Figures 5 and 6 provide test results obtained with an aqueous Carbopol solution (1,000 wppm) at room temperature. Figure 5 shows the carlopol solution mass variation as function of time. The trends are similar to those for mineral oil. The test run took less than 30 min. The test duration can be shortened by adjusting the capillary tube dimensions. Figure 6 shows the viscosity results for the aqueous Carbopol solution at room temperature. For comparison, the test fluid viscosity was also measured by a rotating viscometer. The test results from the mass-detecting capillary viscometer were in very close agreement (less than 2%) with those from the rotating viscometer in a range of shear rates ($10^0 \sim 10^3\ s^{-1}$).

Figure 7 presents conesponding viscosity data for a Separan solution (1, 000 wppm) at room temperature. Once again, the viscosity data from the mass-detecting capillary viscometer showed excellent agreement (less than 1. 5%) with those from the rotating viscometer over a range of shear rates.

4. Conclusion

This study introduced a new method to continuously measure liquid viscosity over a wide range of shear rates (as low as 1 s^{-1}). The feasibility and accuracy of the new viscosity measurement technique has been demonstrated for a standard-viscosity oil and aqueous polymer solutions by comparing the new results against those using an established viscosity measurement technique (i.e. a rotating viscometer). Among the advantages of this new viscometer are simplicity (i. e., ease of operation and no moving parts) and the ability to make accurate measurements over a relatively broad shear rate range. Both limits of viscosity and shear rate in the MDCV measurements can be varied by the dimensions of the falling and capillary tubes which are directly related with driving force and resultant pressure drop. In addition, the MDCV can be used for blood viscosity measurements.

References

Bird, R. B., Amstrong, R. C., Hassager, O., 1987, *Dynamics of Polymeric Liquids*, Vol. 1, Wiley, New York.

Cho Y. I., Kim, W. T., and and Kensey, K. R., 1999, "A New Scanning Capillary Tube Viscometers," *Review of Scientific Instruments*, Vol. 70, pp. 2421~2423.

Kim, S., Cho, Y. I., Jeon, A. H., Hogenauer, B., and Kensey, K. R., 2000, "A New Method for Blood Viscosity Measurements," *J. Non-Newtonian Fluid Mech.*, Vol. 94, pp. 47~56.

Macosko, C. W., 1993, *Rheology: Principles, Measurements, and Applications*, VCH, New York.

Middleman, S., 1968, *The Flow Flow of High Polymers, Interscience*, New York.

Munson, B. R., Young, D. F., and Okishi, T. Hl, 1998, *Fundamentals of Fluid Mechanics*, Wiley, New York, (Chapter 8).

Nguyen Y. T., Vu, T. D., Wong, H. K. and Yeow, Y. L., 1999, "Solving the Inverse Problem of Capillary Viscometry by Tikhonow Regularization," *J. Non-Newtonian Fluid Mech.*, Vol. 87, pp. 103~116.

Shin, S. and Cho Y. I., 1993, "Temperature Effect on the Non-Newtonian Viscosity of an Aqueous Polyacrylamide Solution," *Int. Comm. Heat Mass Transfer*, Vol. 20, pp. 831~844.

Shin, S., Lee, S. and Keum, D., 2001, "A New Mass-Detecting Capillary Viscometer," *Review of Scientific Instrument*, Vol. 72, pp. 3127~3128.