

Application of the Falling Needle Viscometer

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Introduction

The Falling Needle Viscometer (FNV) [1] was originally developed to measure the rheological properties of Newtonian and non-Newtonian fluids. However it is a versatile instrument and its functions have been expanded to experimentally determine other physical properties. These include, in addition to the liquid viscous properties, liquid density, zero shear rate viscosity and yield stress, mixture concentration, relative particle settling rates, intrinsic viscosity and molecular weight, high pressure and high temperature viscosity, and time constant measurements of visco-elastic fluids.

The operating principle of the FNV is quite simple and is described in detail elsewhere [2, 3, 4 and 5] so only a general description will be presented here.

Figure 1 shows a schematic of the instrument which consists of an inner cylinder containing the liquid whose properties are to be measured. A constant temperature liquid is circulated between the measuring cylinder and the coaxial outer cylinder to specify the temperature of the test liquid.

A slender hollow cylinder (needle) with hemispherical ends and a weighted tip or a controlled needle with hemispherical ends and extension bar plus external weights falls through the test liquid with the longitudinal axes of the needle and the measuring cylinder parallel to the gravity vector. The needle quickly reaches its terminal velocity, which is then measured either visually or electronically. A knowledge of the terminal velocity, the fluid and needle densities, and the needle geometry is sufficient to determine the viscous properties of the fluid.

The manufacturer (Stony Brook Scientific, Ltd.) makes available three models of the falling needle viscometer. The FNV-200 is a general laboratory model where the needle velocity is measured by placing a small magnet in the needle tip which activates a pair of Hall sensors located outside of the outer cylinder and which turn on and off a digital clock (see Fig. 1). Partial specifications are: viscosity range, 0.5 to 10^6 cP, absolute accuracy, better than $\pm 1\%$.

Model DV-100 is shown in Fig. 2 and has several additional features than the FNV-200. An important feature is the fact that the measuring cylinders are smaller and thus require less experimental fluid (4.5ml). In addition, the measuring cylinders of the DV-100 are disposable which does away with cleaning problems. Partial specifications are: viscosity range, 1 to 10^8 cP, absolute accuracy, better than $\pm 1\%$.

For field work with a viscometer that doesn't require the accuracy of the other models, the PDV-100 is available. The absolute accuracy is better than

$\pm 5\%$ and the needle velocity depending on the needle density is measured either visually or electronically.

In the following section, a number of possible FNV measurement techniques are discussed.

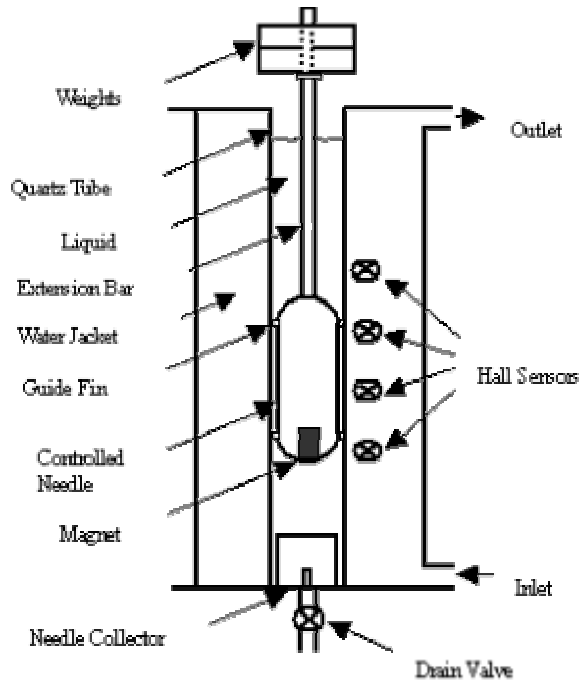


Figure 1 Schematic of falling/controlled needle viscometer



Figure 2 Photo of a DV-100 disposable viscometer with microprocessor console

Applications

Newtonian fluid viscosity measurements

Figure 3 shows the results of Newtonian fluid dynamic viscosity measurements using the DV-100. The process is described in Reference [3]. The fluid is a standard viscosity fluid from the Cannon Instrument Co. In the figure, the ordinate is the measured viscosity and the abscissa is a number identified with a single measurement run. From the figure, the accuracy (as compared to the standard fluid) is better than $\pm 1\%$ and the reproducibility is also better than $\pm 1\%$.

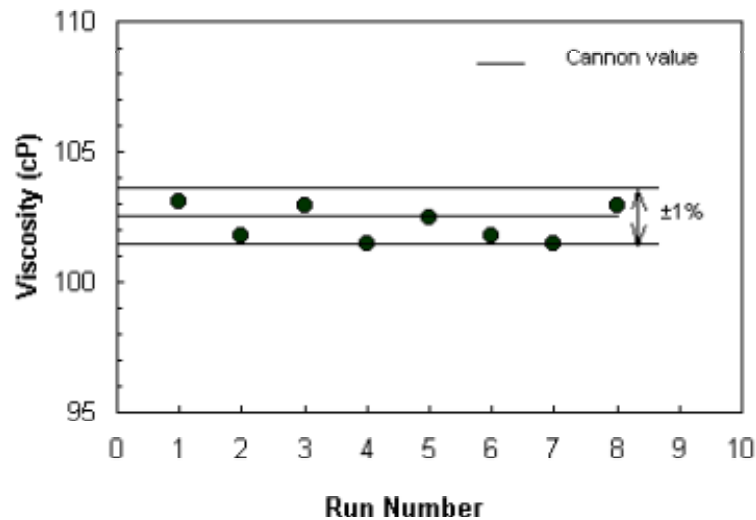


Figure 3 Viscosity measurement for Cannon S-60 fluid with DV-100 disposable viscometer

Non-Newtonian fluid viscosity measurements

The Falling Needle Viscometer can also be used to measure the rheological properties of purely viscous non-Newtonian fluids. The process is described in Ref. [4]. Figure 4 shows the results of using the FNV to measure the apparent viscosity vs. the shear rate for AP 273 Separan. Also of interest in Fig. 4 are apparent viscosity measurements of the same fluid using a commercial rotating cylinder viscometer (RCV). It is seen that the agreement between the two methods is quite satisfactory. It can also be noted that the FNV can operate at much lower shear rates than the RCV (lowest shear rate measured at 0.05RPM of the RCV, which is the low limit of the RCV). This is important in measuring yield stresses, which will be discussed below.

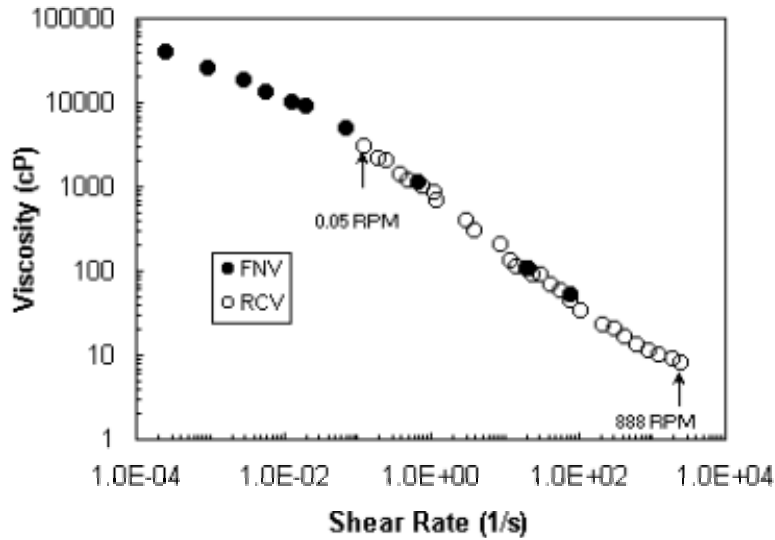


Figure 4 Typical flow curve measured with the Falling Needle Viscometer (FNV) and Rotating Cylinder Viscometer (RCV), AP 273 Separan 2000 wppm at 25°C

Density and viscosity measurements

One of the advantages of the FNV is that for Newtonian and power-law non-Newtonian fluids it is possible to measure the fluid density at the same time as measuring the fluid viscosity. This technique is described in Refs. [4], [6] and [7]. Essentially it involves dropping two needles of the same geometry but of different densities. Figure 5 and 6 show the results of such measurements made on S-600 standard fluid along with a comparison line taken from Cannon specifications. It is seen from the figure that the accuracy of the density measurements is of the order of $\pm 0.27\%$.

In addition to the above technique for measuring liquid densities, an alternative method has recently been developed [7]. This method involves dropping three or more needles and has the possibility of greater accuracy than the former method. With the same data for calculating the liquid density, the viscosity can also be obtained.

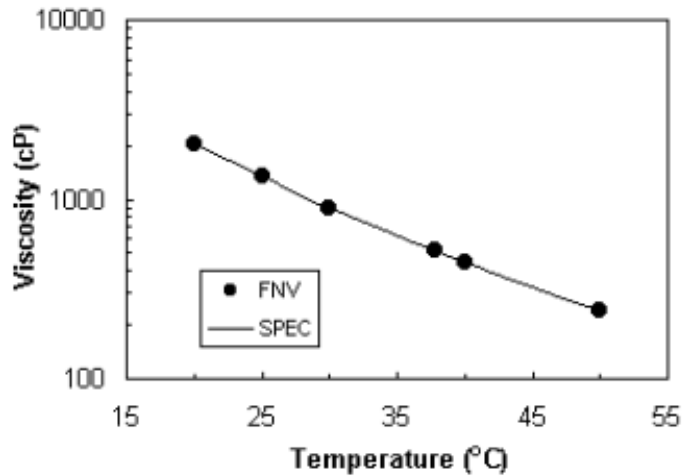


Figure 5 Viscosity measurement of Cannon S-600 standard fluid

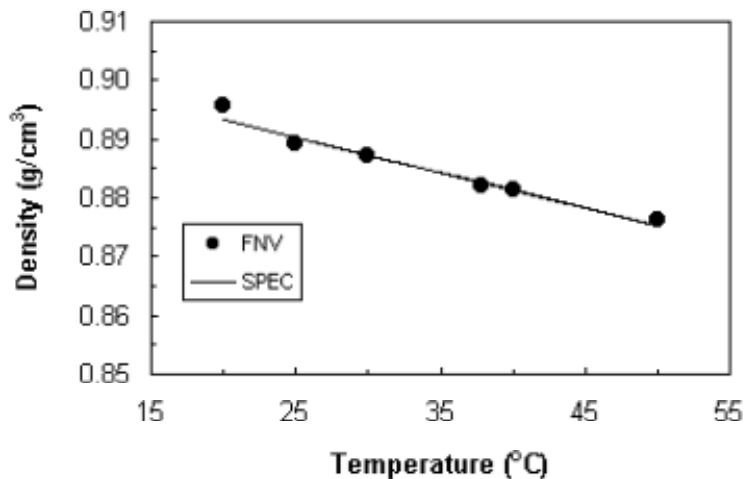


Figure 6 Density measurement of Cannon S-600 standard fluid

Zero shear rate viscosity measurements

One of the important properties of non-Newtonian fluids is the zero shear rate viscosity. Strictly speaking, this is the value of the viscosity when the fluid is at rest and therefore cannot be measured by any viscometer. The method generally used is to measure the viscosity at lower and lower shear rates and then extrapolate the resulting curve to zero shear rate. Clearly, in using such a technique, the lower the shear rate at which viscosities can be measured, the more accurate the extrapolation process. As mentioned above, because the FNV is capable of viscosity measurements at very low shear rates, it is particularly suitable for these zero shear rate viscosity determinations. Examples of such measurements are given in Ref. [4].

Yield stress measurements

There is a class of fluids that when subjected to a stress do not go into motion until a certain stress is applied. This critical stress is called yield stress. It is an important property for such liquids as paints which need to remain without motion on a vertical surface until they dry, or medical ointments so that they remain in place on the skin.

The FNV can be used to measure a yield stress as follows: Consider a falling needle when the fluid has a yield stress [8]. If the density difference driving force (weight minus buoyancy) is exactly equal to the yield stress force, the needle velocity will be zero and the needle will be stationary. Under these conditions, a simple force balance can be made between the yield stress, weight and buoyancy force

$$\tau_y \pi d L + \tau_y \pi^2 d^2 / 4 = (\rho_n - \rho_l)_s g (\pi d^2 L / 4 + \pi d^3 / 6) \quad (1)$$

Where τ_y = yield stress, d = needle diameter, L = total needle length minus one diameter, $(\rho_n - \rho_l)_s$ = static density difference between needle (ρ_n) and liquid (ρ_l), and g = acceleration of gravity. The second term on the left hand side is the net vertical shear force around the two hemispherical ends.

Equation (1) states that if we can get the density difference just right, the needle will be suspended at zero velocity. This density difference is difficult to determine and an extrapolation process is called for. The process is illustrated in Fig. 7 where a number of needles of different densities were dropped and their velocities measured. If the solid line is extrapolated to zero velocity then the density difference at that point is the proper one to insert into Equation (1) to calculate the yield stress τ_y .

It should be noted that the lower the measured velocity in Fig. 7, the more accurate the extrapolation process to obtain the critical density difference. That is why the FNV is especially appropriate for such measurements. Also, yield stresses are often quite small (less than 0.1 N/m^2) which makes direct measurements very difficult.

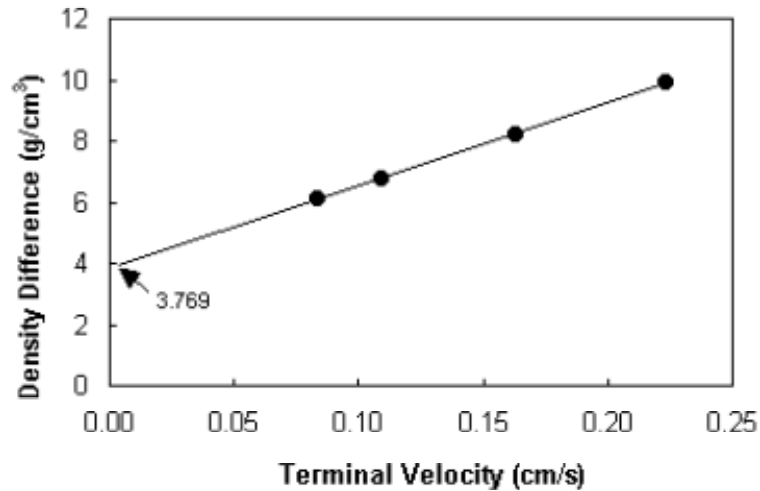


Figure 7 Yield stress measurement of ointment (density difference: needle density minus liquid density)

Mixture concentration measurements

The dynamic viscosity of liquid-liquid mixtures is sensitive to mixture composition. This suggests that viscosity measurements can be used to determine liquid concentrations for homogeneous liquid-liquid mixtures. As an illustration of such concentration measurements, Ref. [9] shows viscosity measurements of small amounts of kerosene or gasoline mixed with motor oil at different temperatures.

A careful inspection of the figure in Ref. [9] would reveal that a 5% concentration by weight of gasoline in oil at 20°C results in a 40% change in viscosity as compared to pure motor oil. These measurements were made with the FNV which can therefore serve as a concentration measuring device once properly calibrated for a given liquid-liquid mixture.

Relative particle settling rate measurements

Dynamic viscosity changes are most sensitive to changes in liquid composition. This makes it possible to test for contaminants in a liquid or to study the sedimentation process of particles in a liquid. In a study of the latter [9], viscosity measurements were made for cornstarch, particles falling in distilled water. Figure 8 shows the results of these measurements and illustrates the technique for measuring the local needle velocity during its decent. On the right side of Fig. 8 is a schematic of the FNV inner cylinder. The H symbols stand for Hall sensors of which there are four. Thus it is possible to measure their local needle velocities. These local falling velocities are plotted against the falling distance in Fig. 8 with the parameter being the elapsed time from when the aluminum particles-water mixture was made homogeneous by shaking. For example, the horizontal line marked 0 minute indicates that just after shaking the

decent velocity was constant along the needle's path. The upper line marked solvent (distilled water) indicates the decent velocities before the introduction of any aluminum particles.

The result of such an experiment is that one can deduce that all of the significant settling has occurred during 100 minutes. Another interesting conclusion is that the needle velocity distribution never reaches that of the pure solvent (complete settling). This possibly indicates that the smallest particles, which do not settle because of Brownian motion, continue to exert a significant influence on the needle decent velocity.

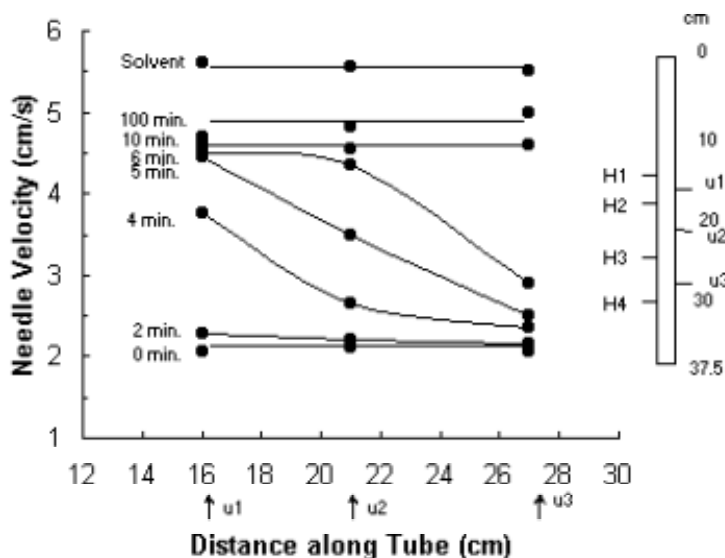


Figure 8 Needle velocity vs. distance along tube for 2% aluminum particles in distilled water

Intrinsic viscosity and molecular weight determination

Another FNV measurement technique is the measurement of the molecular weight of a polymer by measuring the intrinsic viscosity of polymer solutions at lower and lower concentrations. Such a technique consists of measuring the viscosity of polymer solutions at lower and lower polymer concentrations [10].

Such measurements have been made with the FNV and they compare favorably with other techniques.

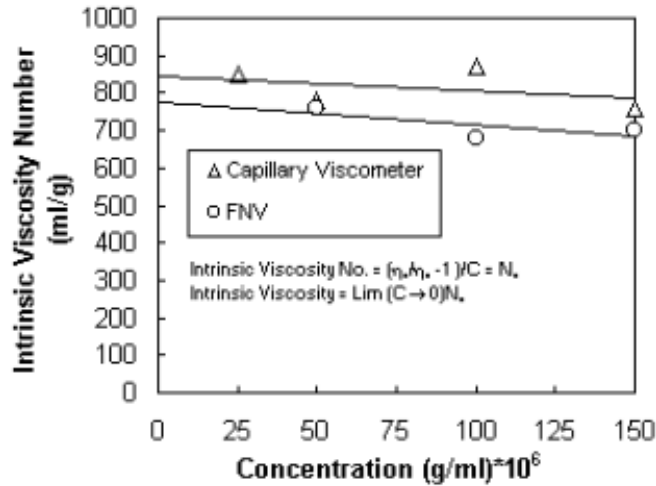


Figure 9 Intrinsic viscosity measurement of CMC polymer solution

High pressure viscosity measurements

Viscosity measurements at pressure higher than ambient are of interest in a number of fields such as oil recovery, polymer processing, etc. Such measurements are difficult however since the experimental fluid must be isolated from its surroundings to allow independent control of the fluid pressure level.

An atmospheric pressure falling needle viscometer has been converted to a high pressure dynamic viscosity instrument. The conversion is relatively straight forward since the only information that must be transferred from the high pressure region to the surroundings is a time signal which is transmitted via a magnetic pulse.

Using the FNV modified for high pressure operation, high pressure viscosity measurements were made on Cannon S-600 oil over a pressure range of 1-110 atmospheres, as shown in Fig. 10 and described in Ref. [11].

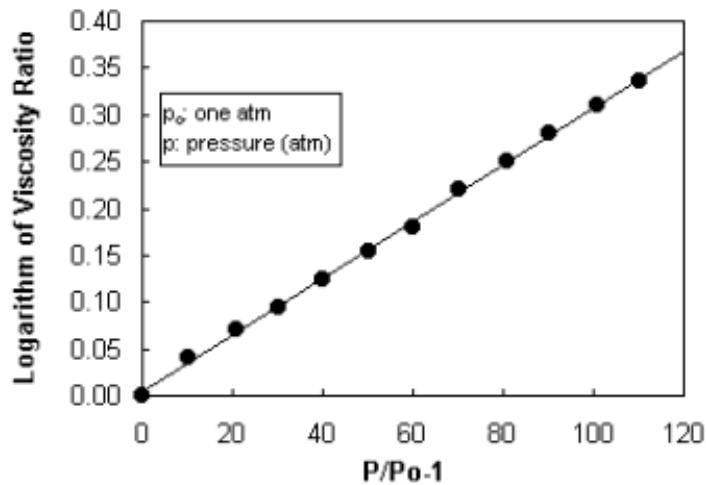


Figure 10 Variation of viscosity with pressure for Cannon S-600 oil

High temperature viscosity measurements

Rheological property measurements at high temperature are very important to characterize the molten plastics, pitches, asphalts, etc. Such measurements are difficult however since the experimental sample must be isolated from its surroundings to allow independent control of the sample temperature.

A falling/controlled needle viscometer has been converted to a high temperature viscosity instrument. The conversion is relatively straight forward since the only information that must be transferred from the high temperature region to the surroundings is a time signal which is transmitted via a magnetic pulse.

Using the falling/controlled needle viscometer modified for high temperature operation, high temperature viscosity measurements were made on Low Density Polyethylene at 190°C as shown in Fig. 11.

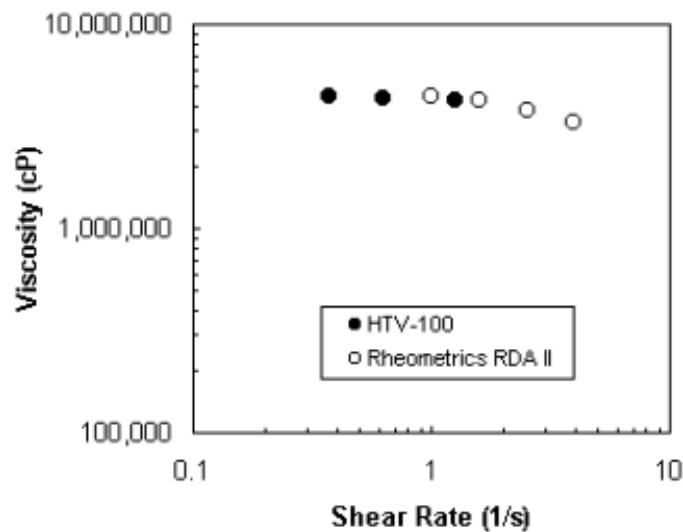


Figure 11 Viscosity measured by the high temperature viscometer compared to the data measured by the Rheometrics RDA II for Low Density Polyethylene at 190°C

Time constant measurements of viscoelastic fluids

It is well known that viscoelastic fluids possess a memory. If such a fluid is disturbed by changing its shear field from equilibrium, only after a finite time will the fluid return to its original equilibrium state. This time can be characterized as a time constant for the fluid.

In a recent study [12], time constant measurements were made on a highly viscoelastic fluid (Polyacrylamide Gel) using the falling needle viscometer (model: DV-100). The procedure was as follows. The viscoelastic fluid was placed in the viscometer and temperature equilibrium was established. Then, a needle was dropped and the time was measured for the needle to fall a specified distance. This time called t_0 was the reference time for a needle to fall in an undisturbed fluid. Following this measurement of t_0 , after a specified time, t_p , had passed, the same needle was dropped and the time, t , was measured. This sequence was repeated with fresh fluid until a series of values of t/t_0 were obtained. The same series of measurements were also made with different needle densities to study the relaxation time of different stress fields.

The above measurements were displayed on a graph such as Fig. 12. The figure shows that when the specified time interval, t_p , is small. The needle velocity is smaller than the t_0 run because of the flow disturbance caused by the first needle drop. As t_p increases, t approaches t_0 as the flow disturbance disappears.

In Fig. 12, the time constant for the heaviest needle is of the order of 65 minutes while it is only of the order of 15 minutes for the lighter needles. A knowledge of these time constants make it possible to measure certain fluid properties for viscoelastic fluids such as the steady shear viscosity without running into elastic effects.

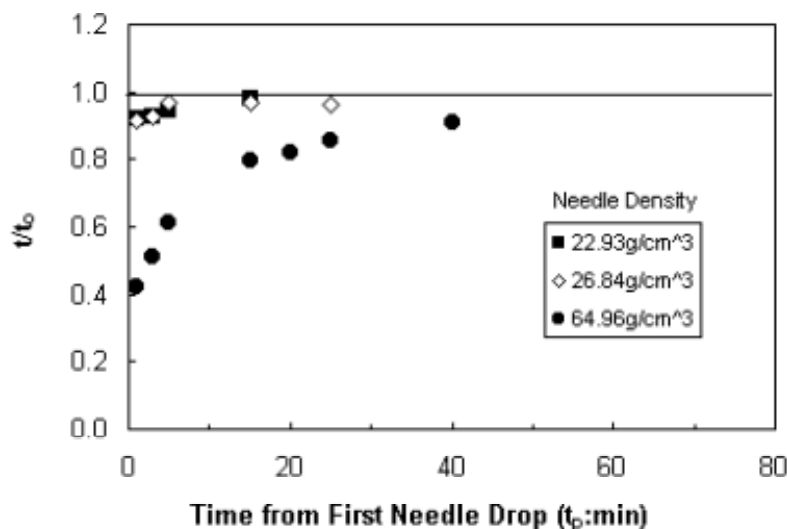


Figure 12 Time interval effects of three falling needles for Polyacrylamide Gel at 25°C

Summary and Conclusions

A brief discussion was presented of the operating principle of the Falling Needle Viscometer. Then, a series of viscometer applications were discussed including viscosity measurements of Newtonian and purely viscous non-Newtonian

fluids, liquid density, zero shear rate viscosity and yield stress, mixture concentration, relative particle settling rates, intrinsic viscosity and molecular weight, high pressure viscosity, high temperature viscosity, and time constant measurements of viscoelastic fluids.

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