A calibration method for a new type of rheometer

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ABSTRACT

Developments of rheometers with uneven geometries of the rotating shaft demand the elaboration of different or new calibration procedures. The present article is based on describing an empirical calibration procedure that enables the prediction of viscosity by using torque and rotational speed.

INTRODUCTION

A new on-line process rheometer for highly viscous food and animal feed materials[1,2] was developed with a headed shaft with helical flights to produce a continuous flow of the tested material (Fig. 1).

The headed shaft with helical flights of the rheometer is assembled inside a barrel that has an upper and a lower end, a die hole is present at the lower end of the barrel to produce a pressure gradient, which enables the continuous measurement of highly viscous compositions.

This research centers its attention on describing a calibration procedure that makes possible the prediction of viscosity by using a rheometer having a shaft with an uneven geometry of the Searle type.

The prediction of viscosity from a rheometer having a shaft with an uneven geometry (Fig. 2) demands an empirical rather than an analytical approach. This is because the analytical assessment of shear rate (s^{-1}) from the speed of the shaft (rpm) and shear stress (Pa) from the torque (Nm) gets complicated owing to the complexity of the geometry along the shaft.

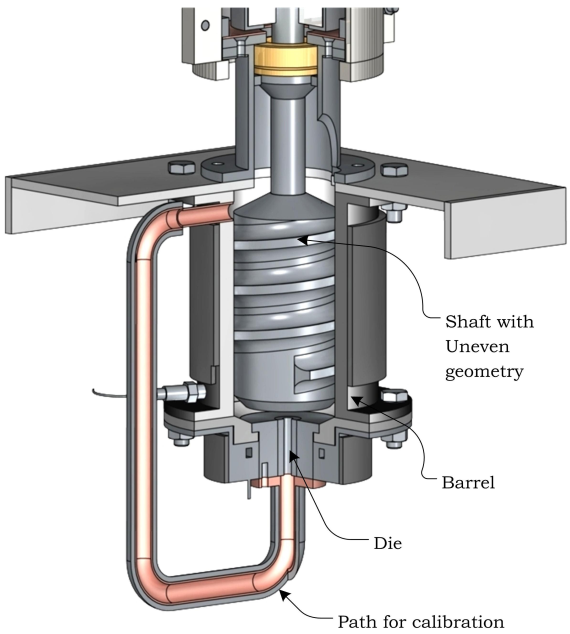

Figure 1. On-line process rheometer for highly viscous materials.

Since rheometers are instruments that perform rheological measurements at different shear rates, the calibration method should contemplate the running of the apparatus at different speeds and at different viscosities.

MATERIALS AND METHODS

The complex geometry of the shaft plus the pressure gradient along the rheometer barrel make it difficult to analytically determine the shear stress and shear rate by means of torque and speed. However, from the relationship between torque and shear stress and speed and shear rate, it is possible to predict viscosity throughout a calibration experiment using a Newtonian standard with known viscosities.

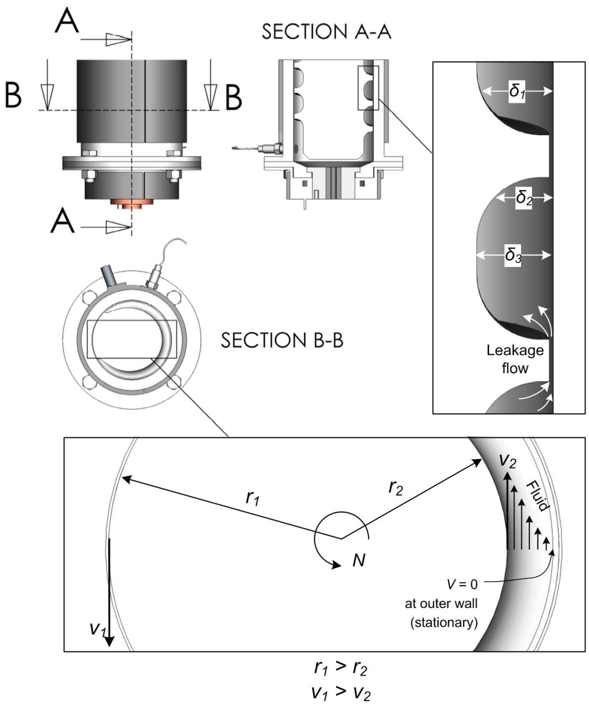

Figure 2. Description of the uneven shaft. Section A-A shows the different clearances (δ) which are associated to different shear rates along the barrel. Section B-B shows different peripherical speeds of the shaft that are also associated to different shear rates. [2]

Experimental procedure to gather data in the rheometer

Since rheometers are instruments that can perform rheological measurements at different shear rates[3], the calibration will contemplate the use of the rheometer from a maximum speed of 75 rpm down to approximately 0 rpm, running the instrument every 5 rpm for about half minute, gathering 10 data points per second. Then, averages of torque (related to shear stress) and rotational speed (related to shear rate) will be calculated, ensuring a high population of data (n > 150 at least). As the viscous standard is a Newtonian fluid, a linear regression will be applied between rotational speed and torque. This procedure is made in duplicate, and repeated every 5 °C between 40 °C to 85 °C to cover a wide range of viscosities presented by Polybuthene-1.

Development of a model to predict viscosity

The local shear stresses (τ) and local shear rates ($\dot{\gamma}$) depend on position (Fig. 2). In the shaft, the torque is the integral of the shear stress times radius, over the surface of the shaft.

$$M = \int \tau \cdot r \, dA \tag{1}$$

In addition, the local shear rate for Searle flow[3,4] is shown in Eq. 2.

$$\dot{\gamma} = \frac{dv}{dx} = \frac{v}{x} = \frac{\omega \cdot r}{\delta} \tag{2}$$

Where δ is the distance between the rotating shaft and the stationary barrel which varies along the shaft, v is the peripherical velocity of the shaft which consequently varies. ω is the angular speed of the shaft and r is the radius of the shaft which vary as well (Fig. 2).

In concentric cylinders with constant radius and gap size, the angular speed ω (rad s^{-1}) can be calculated by:

$$\omega = \frac{2 \cdot \pi \cdot N}{60} \tag{3}$$

Where N is the rotational speed (rpm), thus:

$$\dot{\gamma} = \frac{2 \cdot \pi \cdot N}{60} \cdot \frac{r}{\delta} \quad (4)$$

Then, the local shear rate is represented by:

$$\dot{\gamma} = \frac{2 \cdot \pi \cdot r}{60 \cdot \delta} \cdot N \quad (5)$$

Where *r* and *δ,* depends on position. Eq. 6 comes from the definition of viscosity (η) as a function of shear stress (τ) and shear rate ($\dot{\gamma}$).

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (6)$$

In this rheometer, the torque comes from the material resistance to flow, which is proportional to the shear stress. This resistance is present near the barrel walls and along the channels in the helical flights, also the die of the rheometer indirectly affect the torque by the pressure gradient. Therefore to simplify this estimation the mean shear stress is used. Something similar occurs with the estimation of the shear rate. In this way, a constant which should characterize the system is needed to extract the average shear rate and average shear stress from the speed and torque, and thus to predict viscosity. Eq. 7 describes this situation:

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{M \cdot k_1}{N \cdot k_2} \quad (7)$$

Here, *M* is torque (Nm) and *N* is the number of revolution per minute of the shaft. k_1 is the constant needed to convert torque to mean shear stress, and k_2 is the constant to convert the rotational speed to an average shear rate. To reduce the number of constants to estimate, k_1 and k_2 can be expressed in terms of another constant *K* where:

$$\eta = \frac{M \cdot k_1}{N \cdot k_2} = \frac{M}{N} \cdot K \quad (8)$$

In consequence, to predict viscosity it will be necessary to find the constant *K*. To find this value, a Newtonian Certified Viscosity Standard (Polybuthene-1 – 100%) was introduced into the system. This is a temperature dependent polymer and three of its viscosities were documented by the manufacturer. Consequently it was necessary to use a calibrated rheometer (Physica UDS200, Germany) to determine a wider range of viscosities according to different temperatures.

Using a Newtonian fluid, a proportional relation is expected between the rotational speed and torque, therefore applying the best curve using the equation of a linear fit (Eq. 9) should result in:

$$M = a \cdot N + b \quad (9)$$

Where *a,* is the slope of the curve and *b* is the constant where the curve intersect the axis of the dependent variable. Using Newtonian fluids, the curve intersects the origin and consequently *b* is set to zero. Then, Eq. 9 in Eq. 8 results in:

$$\eta = \frac{M}{N} \cdot K = a \cdot K \quad (10)$$

The viscosity of Polybuthene-1 changes at an exponential rate when the temperature changes; this behavior was assessed using both rheometers (Fig. 3, 4). Thus *K* varies as a function of the slope, *a* (Nm/rpm), due to the changes in temperature that affect viscosity.

Therefore, the model which will come from Eq. 10 will be used to predict viscosity, and it is specific for the geometry and behaviour of the developed rheometer.

Prediction of viscosity

The model that will be used to predict viscosity from a slope, *a* (Nm/rpm), given by the new type of rheometer, will be built from the empirical result coming from the plot of slope, *a* (Nm/rpm), versus the viscosities obtained in the calibrated rheometer (Physica USD 200, Germany).

RESULTS AND DISCUSSIONS

Experimental procedure for rheological measurements

Due to the large number of experiments (rotational speed v/s torque) that were repeated twice every 5 °C between 40 °C to 85 °C, the results will not be represented in a graph. However, the slopes coming from the linear regressions between rotational speed and torque are presented and used as a base to build the model to predict viscosity.

Development of a model to predict viscosity

As commented earlier, it is necessary to document experimentally the wide range of viscosities of Polybuthene-1. Fig. 3 shows the results.

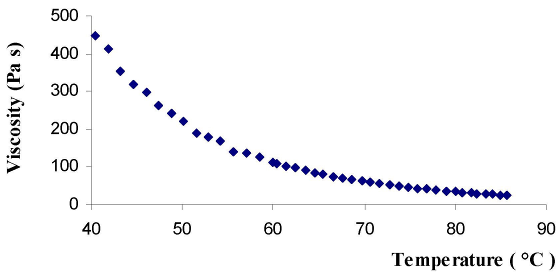

Figure 3. Viscosities of Polybuthene-1 at different temperatures measured in a calibrated rheometer (Physica UDS 200, Germany).[2]

It was necessary to perform a similar assessment on the new type of rheometer. As explained earlier, the slope, *a* (Nm/rpm), at different temperatures (°C) is used.

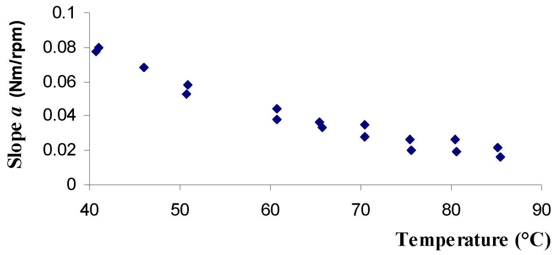

Figure 4. Slopes responses measured in the rheometer for highly viscous materials at different temperatures (two repetitions), which are related with the viscosities of Polybuthene-1. Slope *a* is described in Eq. 9.[2]

Each data point in Fig. 4, comes from a linear regression of 16 averages (e.g. avg. for 75, 70, 65 rpm, etc. which has $150 < n < 270$ and with a standard error of the mean < 0.02) obtained between rotational speed and torque.[2]

By comparing Fig. 3 and 4, we see that both rheometers present similar behaviour, but have different levels of curvature.

The comparative analysis of behaviour between both rheometers is presented in Fig. 5.

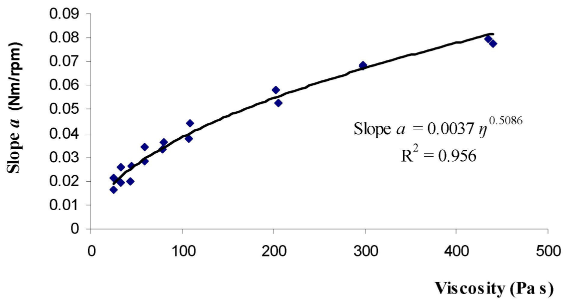

Figure 5. Viscosities of Polybuthene-1 (Pa s) at different temperatures, versus the slope *a* (Nm/rpm) from the rheometer with uneven shaft.

From Fig. 5 it is possible to distinguish how the slope changes when viscosity changes. The assessment given by Fig. 5 allowed us to build the predictive model where:

$$\text{Slope } a = 0.0037\,\eta^{0.5086} \qquad (11)$$

Thus, from Eq. 11 that was theoretically expressed in Eq. 10, comes Eq. 12.

$$\eta = \left(\frac{a}{0.0037}\right)^{1/0.5086} \qquad (12)$$

To assess the suitability of using this calibration procedure, the values from the predicted viscosities given by Eq. 12 are plotted with the viscosities of Polybuthene-1 given by Fig. 3. This results in Fig. 6.

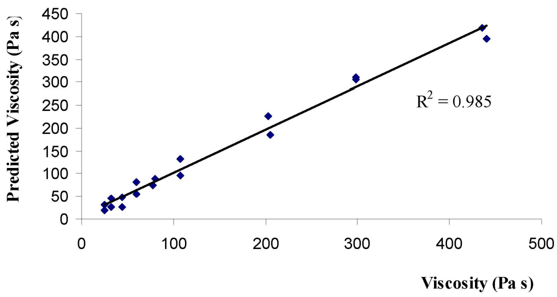

Figure 6: Viscosities (measured in the Physica UDS 200 rheometer), versus predicted viscosity (measured in the new type of rheometer) based in the slope *a* (Nm/rpm).

CONCLUSIONS

The calibration of a rheometer with a rotating shaft of uneven geometry can be simplified by using an empirical calibration experiment with a known viscosity standard fluid, this to build a prediction model for viscosity.

The resulting prediction model includes all the phenomena in the rheometer as backward leakages and pressure gradients, which affects the relation between shear stress and shear rate. A final plot of viscosities of Polibuthene-1 versus predicted viscosities can reveal the suitability of the presented calibration method.

ACKNOWLEDGMENTS

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REFERENCES

1. Salas-Bringas, C. (2004), "Development of an On-line Rheometer and Die Tester for Feed/Food Quality Control", Dep. Mathematical Sciences and Technology. MSc. Thesis Norwegian University of Life Sciences, Ås. pp. 84.

2. Salas-Bringas, C., Jeksrud, W.K., and Schüller, R.B. (2006), "A New On-line Process Rheometer for Highly Viscous Food and Animal Feed Materials", *Journal of Food Engineering*., **In Press**.

3. Steffe, J.F. (1996), "Rheological Methods in Food Process Engineering". 2nd ed., East Lansing, MI, USA: Freeman Press.

4. Tabilo-Munizaga, G. and G.V. Barbosa-Canovas. (2005), "Rheology for the food industry", *Journal of Food Engineering*., **67,** 147-156.