Data analysis from capillary rheometry can be enhanced by a method that is an alternative to the Rabinowitsch correction

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ABSTRACT

The following article compares a method (presented by Salas-Bringas et al[1]) using a flow rate equation with the Rabinowitsch method to estimate the consistency and flow behavior index of a Non-Newtonian fluid in capillary flow. The differences is that the method presented by Salas-Bringas et al[1] gives the uncertainty of the measurements and requires less number of experiments when different flow rates are measured.

INTRODUCTION

The simplicity of capillary rheometers[2, 3] has resulted in its wide usage within the process industries for applications as diverse as iron or slurries, foams, distilled water, glycerol and polymer solutions[3]. Due to their inherent similarity to many process flows, which typically involve pipes, capillary rheometers are widely employed in process engineering applications[2].

Measurements of pressure drop in the capillary are required to estimate absolute values of viscosity. Pressure estimations are currently performed by measuring the pressure in the reservoir or barrel above the capillary. Pressure losses occur at the capillary entry, and thus corrections must be taken to avoid wrong estimations of shear stresses[1, 4, 5]. Salas-Bringas et al[1] is developing a capillary rheometer that uses direct pressure measurements in the capillary. The increased developments in sensor technology will soon allow commercial capillary rheometers to account with pressure measurements in the capillary, and thus pressure corrections will no longer be required.

Today, the most important method to estimate the shear rate at the capillary walls ($\dot{\gamma}_w$) for Non-Newtonian fluid behavior is the often termed Weissenberg - Rabinowitsch or Rabinowitsch - Mooney correction[2, 4].

Salas-Bringas et al[1] presented an alternative method based in the flow rate information that does not uses the Rabinowitsch correction to estimate the consistency (K) and the flow behavior index (n) of Non-Newtonian fluids.

How the method presented by Salas-Bringas et al[1] performs when comparing to the Rabinowitsch correction is a question that has not been analysed yet and thus, there is no reference from where to make the decision of selecting one method over the other.

This article presents a study that analyses and compares the performance of both methods using the same capillary data.

Table 1. Capillary data for dough (34.7%) at room temperature made from defatted soy flour treated to cause protein denaturation. Using a capillary of $D =$ 0.00318 m (data from Morgan[6] and used by Steffe[5], the first and the last two columns where calculated for this study).

Q (m3/s)	$4Q/(\pi R^3)$ (1/s)	$Q/(\pi R^3)$ (1/s)	L/D (-)	ΔP_m* (MPa)	ΔP_{en}** (MPa)	ΔP*** (MPa)	τ_w (kPa)	L (m)	$\Delta P/L$ (MPa/m)
1.50E-07	47.4	11.9	2	4.21	3.58	0.63	0.079	0.00636	104.82
1.50E-07	47.4	11.9	5	5.21	3.58	1.63	0.082	0.01590	97.48
1.50E-07	47.4	11.9	8	6.14	3.58	2.56	0.080	0.02544	101.15
2.99E-07	94.8	23.7	2	5.46	4.63	0.83	0.104	0.00636	141.51
2.99E-07	94.8	23.7	5	6.81	4.63	2.18	0.109	0.01590	126.83
2.99E-07	94.8	23.7	8	8.02	4.63	3.39	0.106	0.02544	134.17
5.99E-07	190.0	47.5	2	5.25	4.38	0.87	0.109	0.00636	162.47
5.99E-07	190.0	47.5	5	6.80	4.38	2.42	0.121	0.01590	157.23
5.99E-07	190.0	47.5	8	8.30	4.38	3.92	0.123	0.02544	159.85
2.99E-06	948.0	237.0	2	7.68	6.17	1.51	0.189	0.00636	255.77
2.99E-06	948.0	237.0	5	10.12	6.17	3.95	0.198	0.01590	229.56
2.99E-06	948.0	237.0	8	12.31	6.17	6.14	0.192	0.02544	242.66

* Measured pressure drop
** Entrance loss pressure correction
*** Corrected pressure drop

MATERIALS AND METHODS

To compare how both methods performs to determine K and n values in a fluid without yield stress, K and n will be estimated using the same set of capillary data which is given in literature[5, 6] (Table 1), where: Q is the volumetric flow rate (m^3/s), R the capillary radius (m), L the capillary length (m), D the capillary diameter (m), ΔP the pressure drop (MPa) and τ_w the shear stress at the capillary walls (kPa).

Each method will be presented describing step by step the solving procedure.

Consistency and flow behavior index by Rabinowitsch-Mooney equation

The Rabinowitsch-Mooney equation is used to determine the shear rate at the capillary walls as follows:

$$\dot{\gamma}_w = f(\tau_w) = \left(\frac{3Q}{\pi R^3}\right) + \sigma_w \left[\frac{d(Q/(\pi R^3))}{d\tau_w}\right] \quad (1)$$

The experimental data shown in Table 1 is plotted in Fig. 1.

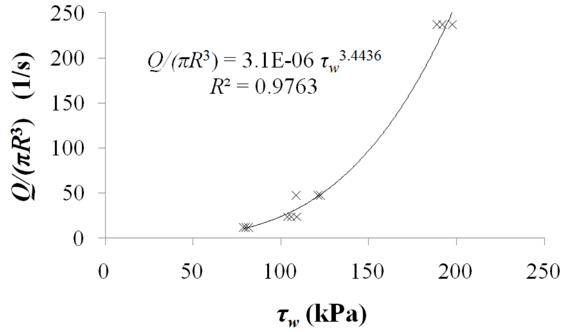

Figure 1. Capillary data for soy dough corrected for pressure loss.

The derivative of the equation created from the power law curve fitting (Fig. 1) is expressed as follows:

$$\frac{d(Q/(\pi R^3))}{d\tau_w} = 1.06 \cdot 10^{-5} (\tau_w)^{2.44} \quad (2)$$

This equation can be incorporated into the Rabinowitsch-Mooney equation (Eq. (1)) to find out the shear rate at the capillary walls:

$$\dot{\gamma}_w = \left(\frac{3Q}{\pi R^3}\right) + \tau_w \left[1.06 \cdot 10^{-5} (\tau_w)^{2.44}\right] \quad (3)$$

Once these calculations are performed, a shear rate versus shear stress plot can be made.

Consistency and flow behavior index by a flow rate equation

To calculate the flow rate (Q) in capillaries, for fluids without yield stress that satisfy the power law model, the following equation can be used[1, 2, 5]:

$$Q = \pi \left(\frac{\Delta P}{2 l K}\right)^{1/n} \left(\frac{n}{3n+1}\right) R^{(3n+1)/n} \quad (4)$$

Since Q, R, l ΔP and are known from Table 1, it is possible to estimate K and n values by solving Eq. (4). To obtain the two unknown variables (K, n), at least two equations are needed. The data from Table 1 includes four different Q, each of them repeated three times. K and n can be estimated by re-arranging Eq. (4) as: $Q_1 - f(R, \Delta P, l, K, n) = 0$ and iteratively find the K values for a number of n values. These values can be plotted positioning n in the independent axis and K on the dependent axis, the result will be a curve. By doing the same procedure for a new Q_2 and plotting the new curve together with the one generated using Q_1, it will produce an intercept in a point (n, K) indicating the two unknown values.

Statistical analysis (e.g. standard deviations, standard errors, etc) can be performed if more values (different Q) are taken. The number of crosses or intersections (J_N) can be calculated by:

$$J_N = \sum_{i=1}^{I-1} i \quad (5)$$

where I is the number of curves at different Q. Averages of K and n can be estimated with their errors around a mean.

Since three repetitions were done by Morgan[6] for a single Q, the crosses cannot be plotted in one figure as the plot demands to be built using different Q. This will result in a large number of plots (27) that cannot be shown in this article. However one of the plots is included (Fig. 2) as an example.

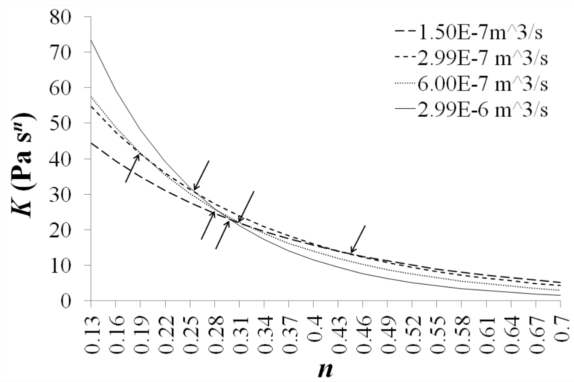

Figure 2. Six intersections for four different flow rates. The intersections are indicated with an arrow.

RESULTS

Consistency and flow behavior index by Rabinowitsch-Mooney equation

From the plot presented in Fig. 3, is possible to obtain the consistency K, and flow behavior index n.

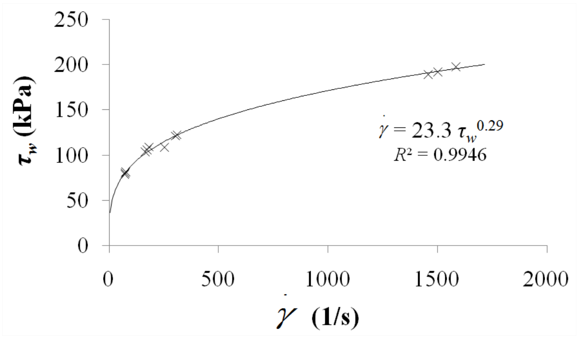

Figure 3. Rheogram for soy dough showing the *K* (23.3 Pa s^n) and *n* values (0.29).

As shown in Fig. 3, the Rabinowitsch-Mooney equation resulted in a *K* value of 23.3 Pa s^n and a *n* value of 0.29.

<u>Consistency and flow behavior index by a flow rate equation</u>

Fig. 4 indicates the distribution of intersections for the different *K* and *n* values for all data (four flow rates repeated three times). The average of these points will represent the *K* and *n* values of the soy dough.

All intersections together (Fig. 4) presented the same trend as the one shown in Fig. 3 for the six intersections.

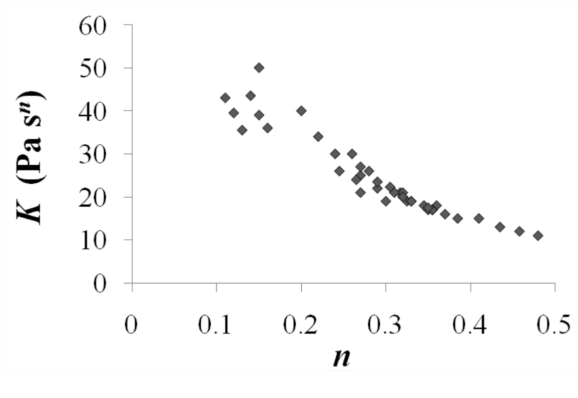

Figure 4. Distribution of intersections (*K* and *n*) for all plots. Each point represents one intersection.

The data points or crosses displayed in Fig. 4 where re-plotted in a frequency plot to observe how the data were distributed for the *K* and *n* values (see Fig.5 and Fig. 6).

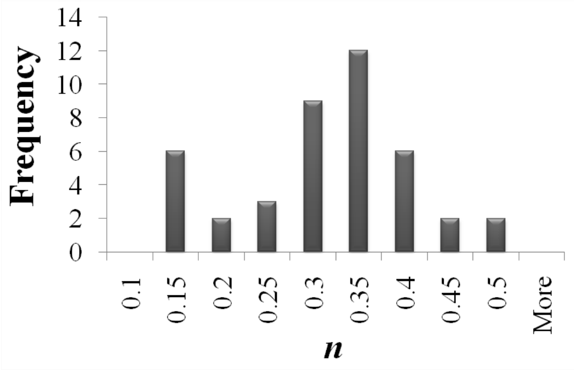

Figure 5. Frequency plot for the distribution of *n* values.

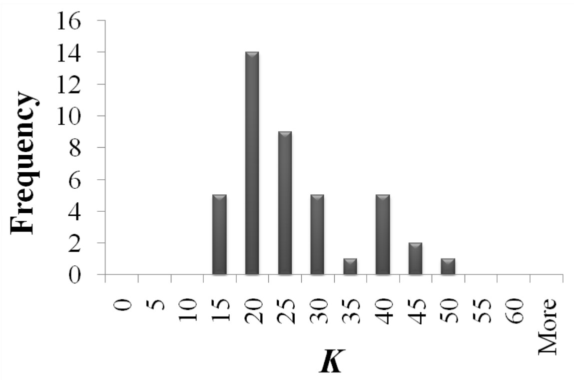

Figure 6. Frequency plot for the distribution of *K* values.

The averages of *K* and *n* values with their standard deviations are: $K = 24.28 \pm 9.56$ and $n = 0.29 \pm 0.09$.

DISCUSSIONS

Similar assumptions have to be made when using the method using the flow rate information[1] compared to the Rabinowitsch-Mooney correction[1, 2, 5]: flow is laminar and steady, fluid is incompressible, properties are time and pressure independent, temperature is constant, no slip occur at the wall of the capillary, radial and tangential velocity components are zero and the pressure drop should be linear in the capillary. Therefore both methods demand the same experimental conditions.

The method from Rabinowitsch demanded less plots using Morgan's data[6], because each experiment was repeated three times. The Rabinowitsch method delivers only one n and K value without informing the absolute uncertainty of the measurements. Instead the method using the flow rate equation demanded more plots for these specific data, but is able to give absolute values of the uncertainty of the measurements and thus comparisons with more confidence is possible when using different fluids.

If uncertainty is to be estimated from the Rabinowitsch method, additional statistical analyses have to be made like the Root Mean Square Error of Prediction (*RMSEP*). This will result in a prolonged calculation.

With Morgan's data and using *RMSEP*, the uncertainty can be estimated based on 12 data points (see Fig. 3). Instead, when using the method based on the flow rate equation, the uncertainty (e.g. standard deviation, standard error, etc) can be estimated based on 42 data points (see Fig. 4).

The large number of plots using the flow rate equation was due to Morgan[6] repeated three times his experiments at each flow rate. Instead, a minimum of two non-repeated flow rates are required to obtain a single n and K value using the method based on the flow rate equation, but the uncertainty of the measurement can be done using three or more flow rates. Therefore, to obtain uncertainty is preferable to do three or more experiments at different flow rates without been repeated.

Both methods gave the same n values (0.29), but not the same K values. Rabinowitsch method resulted in a K of 23.3 Pa s^n, and the flow rate method in a K of 24.28 Pa s^n. According to the estimated uncertainty of measurements, the consistency of the fluid can be found in an interval $14.72 \le K \le 33.84$ which cover the value obtained by the Rabinowitsch method.

The flow behavior index of the fluid can be found in an interval $0.2 \le n \le 0.38$.

CONCLUSIONS

Using different flow rates, the method based on the flow rate equation requires a minimum of two flow rates to obtain the n and K values which is less than the minimum necessary to build a curve when using the Rabinowitsch method.

For the set of data given by Morgan[6] that uses a flow rate that is repeated three times, the Rabinowitsch method is faster to perform because it requires only one plot.

Rabinowitsch does not give information about the uncertainty of the measurements. If uncertainty is wanted, other statistical methods have to be made in additionally (e.g. *RMSEP*).

Using the same number of experiments at different flow rates, the method based on the flow rate equation gives the uncertainty based on more data points compared to the Rabinowitsch method.

The method based on the flow rate equation gives both the K and n values together with the uncertainty in the same calculative step.

The Rabinowitsch method and the method using the flow rate equation resulted in the same n value and a similar K value.

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