CHARACTERISATION OF NON-NEWTONIAN FLUIDS
USING A BACK-EXTRUSION TECHNIQUE

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ABSTRACT
At present standard rheometers provide sufficiently precise measurements characterising behaviour of non-Newtonian materials. In practice, this accuracy is not always necessary, and the methods providing relatively cheap, fast and sufficient measurements of the rheological characteristics are fully acceptable. Back extrusion - representing one of these methods - is based on plunging of a circular rod into an axisymmetrically located circular cup containing the experimental sample. Formerly this method was applied for a characterisation of power-law, Bingham and Herschel-Bulkley fluids. The aim of this contribution is to present a sufficiently simple user-friendly procedure how to determine the individual rheological parameters appearing in the Vočadlo model (sometimes called Robertson-Stiff one) - yield stress, consistency parameter and flow behaviour index.

INTRODUCTION
Back extrusion (see Fig.1) represents a method providing relatively cheap and sufficient measurements of the rheological characteristics, see Steffe and Osorio [1]. This method is often used in food industry, e.g. for characterisation of tomato concentrate (Alviar and Reid [2]), mustard slurry (Brusewitz and Yu [3]), caramel jam (Castro et al. [4]), wheat porridge (Gujral and Sodhi [5]), corn starch (Singh et al. [6]), rice (Sodhi et al. [7]), raspberry (Sousa et al. [8]), blackberry (Sousa et al. [9]), etc.

The principle of a back-extrusion technique consists in penetrating of a circular plunger into an axisymmetrically placed circular container with a material studied. For a determination of rheological parameters appearing in the individual empirical rheological models, knowledge of a relation between pressure gradient $P$ and volumetric flow rate $q$ through an annulus formed by a plunger and a container is substantial. This relation is possible to derive from the relation for an axial velocity profile of the material studied in an annulus.

This problem was already solved for a determination of the parameters appearing in the following empirical constitutive equations:

- Osorio and Steffe [10] derived an analytical solution for a determination of consistency index $K$ and flow behaviour index $n$ in the power-law model

$$
\tau = K \gamma^{n-1} \dot{\gamma} \quad (1)
$$

- The same authors (Osorio and Steffe [11]) generalised their approach for the case of the Herschel-Bulkley model (for $n=1$ we obtain the Bingham model as a special case)

$$
\tau = \tau_0 + K \gamma^{n-1} \dot{\gamma} \quad (2)
$$

This enables to take into account viscoplastic materials exhibiting a plug-flow region, nevertheless in this model a yield stress $\tau_0$ represents a strict singular term.

The aim of this contribution is - using a back-extrusion technique - to derive a procedure how to determine the parameters in the case of the Vočadlo model. This (sometimes called Robertson-Stiff) model (Parzonka and Vočadlo [12]; Robertson and Stiff [13]) seems to be more user-friendly viscoplastic model involving a term with a yield stress in a more appropriate form

$$
\tau = \left[ K \gamma^\frac{1}{n} + \left( \frac{\tau_0}{\gamma} \right)^\frac{1}{n} \right] \gamma \quad \text{for} \quad |\gamma| \geq \tau_0 \quad , \quad (3)
$$

$$
\dot{\gamma} = 0 \quad \text{for} \quad |\gamma| \leq \tau_0 \quad (4)
$$

where $K$ and $n$ represent consistency and flow behaviour indices, respectively; $\tau_0$ stands for a yield stress.
SOLUTION FOR THE VOČADLO MODEL

The Vočadlo model rewritten in the form corresponding to the flow situation in a back extrusion (see Fig.1) is of the form

\[ \tau_n = \left[ K_n \left( \frac{d\psi}{dr} \right)^{\frac{n-1}{n}} + \tau_0 \left( \frac{d\psi}{dr} \right)^{\frac{1}{n}} \right]^n \quad \text{for} \quad |\tau_r| \geq \tau_0 \quad , \quad (5) \]

\[ \frac{d\psi}{dr} = 0 \quad \text{for} \quad |\tau_r| \leq \tau_0 \quad (6) \]

Introducing the following dimensionless transformations

\[ \xi = \frac{r}{R} \quad , \quad \varphi = \frac{\psi}{V} \quad , \quad T = \frac{2\tau_n}{\left| P[R] \right|} \quad , \quad T_0 = \frac{2\tau_0}{\left| P[R] \right|} \quad , \]

\[ \Lambda = \left[ \frac{P[R]}{2K (V)} \right]^n \quad , \quad Q = \frac{q}{2\pi R^2 V} \quad (7) \]

the problem of flow within an annulus can be reformulated in the form

\[ T = \frac{\lambda^2}{\xi} - \xi \quad , \quad (8) \]

\[ \varphi(\kappa) = -1 \quad , \quad \varphi(1) = 0 \quad , \quad (9) \]

\[ T = \left[ \Lambda^{1-n} \frac{d\varphi}{d\xi}^{1-n} + T_0 \frac{d\varphi}{d\xi}^{-n} \right]^n \quad \frac{d\varphi}{d\xi} \quad \text{for} \quad |T| \geq T_0 \quad , \quad (10) \]

\[ \frac{d\varphi}{d\xi} = 0 \quad \text{for} \quad |T| \leq T_0 \quad (11) \]

where \( \lambda^2 \) is a dimensionless constant of integration, \( s = 1/n \).

If \( \lambda_i, \lambda_o \) denote the dimensionless boundary values of the plug flow region (see Fig.2), then from Eq.(8) it follows that

\[ \lambda^2 = \lambda_i \lambda_o \quad , \]

\[ \lambda_i = \lambda_o - T_0 \quad . \quad (13) \]

For simplification the following notation will be used in the further analysis

\[ H(\xi) = \left[ \xi - \frac{\lambda_i (\lambda_i + T_0)}{\xi} \right]^n \quad . \quad (14) \]

The solution of the above stated problem provides the following expressions for the inner, plug-flow region and outer velocity profiles

\[ \frac{d\varphi}{d\xi} = -\Lambda \left[ \left( \xi - \frac{\lambda^2}{\xi} \right)^n - T_0^n \right] \quad \text{for} \quad \lambda_i < \xi < 1 \quad \text{(where} \quad \frac{d\varphi}{d\xi} < 0 \text{)(17)} \]

From the condition of continuity of the velocity profile

\[ \varphi(\lambda_i) = \varphi_o(\lambda_o) \quad (18) \]

it follows that \( \lambda_i \) is a solution of the equation

\[ \int_{\lambda_i}^{\lambda_i + T_0} \Lambda' H(\xi) d\xi + \int_{\lambda_i + T_0}^{\lambda_o} \Lambda' H(\xi) d\xi = -\left( 2\lambda_i + T_0 - \kappa - 1 \right) \Lambda' T_0^n - 1 = 0 \quad (19) \]

If we compare a volumetric flow rate \( q \) through an annulus as given by rel.(7) and visually in Fig.1, we get

\[ 2\pi R^2 V = \pi (\kappa R)^2 V \quad . \quad (20) \]

From here it follows that

\[ Q = \kappa^2 / 2 \]
As the determination of dimensionless flow rate \( Q \) is basically similar to that derived in Malik and Shenoy [14] for power-law fluids, in the following we only introduce the final result

\[
Q = -\frac{1}{2} \left(1 - s \right)^2 \left( \lambda^2 - 2 \kappa^2 \right) - \frac{1 + \kappa^3 - \lambda^3 - \lambda^3}{2(3 + s)} \left( \lambda^2 \left(1 + \kappa - \lambda - \lambda_n \right) \right) + \frac{\Lambda \cdot \tau_0}{2(3 + s)} \left( \lambda^2 \left(1 + \kappa - \lambda - \lambda_n \right) \right) + \frac{\Lambda \cdot \tau_0}{2(3 + s)} \left(1 + \kappa - \lambda^3 - \lambda^3 \right) + \frac{\Lambda \cdot \tau_0}{2(3 + s)} \left( \lambda^2 - \lambda^2 \right) + \frac{\Lambda \cdot \tau_0}{2(3 + s)} \left( \kappa^3 - \kappa^3 \right) \left( \lambda^2 - \kappa^2 \right) \left( \lambda^2 - \kappa^2 \right) \right]
\]

Comparing rels.(21,22) we obtain

\[
-\frac{1 - s}{2(3 + s)} \lambda^2 \left( \lambda^2 \left(1 + \kappa - \lambda - \lambda_n \right) \right) + \frac{1}{2(3 + s)} \left( \lambda^2 \left(1 + \kappa - \lambda - \lambda_n \right) \right) = 0
\]

Prior to a determination of the empirical constants \( \tau_0, K \) and \( n \) it is useful to eliminate the member \( \Lambda \) from the relations (19) and (23). Using rel.(19)

\[
\Lambda = \int H(\xi) d\xi - \int H(\xi) d\xi \left(2\lambda_0 + T_0 - \kappa - 1\right) T_0
\]

and inserting this relation into rel.(23) we obtain a rather cumbersome but algebraically and numerically simple equation enabling in the following the determination of flow behaviour index \( n \)

\[
-\frac{1 - s}{2(3 + s)} \lambda^2 \left[ \int H(\xi) d\xi - \int H(\xi) d\xi \left(2\lambda_0 + T_0 - \kappa - 1\right) T_0 \right]
\]

Consequently combining rel.(7) for \( \Lambda \) and rel.(24) we get

\[
K = \frac{\rho c}{2} \left[ \int H(\xi) d\xi - \int H(\xi) d\xi \left(2\lambda_0 + T_0 - \kappa - 1\right) T_0 \right]
\]

In the next section this relation will complete a determination of the empirical constants \( \tau_0, K \) and \( n \) appearing in the Vocadlo model.

**PROBLEM SOLUTION**

First, out of three empirical parameters appearing in the Vocadlo model, a yield stress \( \tau_0 \) will be determined. This step is more or less identical to that introduced by Osorio and Steffe [11] for the determination of yield stress in the Herschel-Bulkley model, for illustration see Fig.6 in Osorio and Steffe [11].

Let us denote \( F_T \) a force recorded just before the plunger is stopped formed successively by a friction force along the plunger \( F_f \), force responsible for fluid flow in the upward direction \( F_u \), and buoyancy force \( F_b \)

\[
F_T = F_f + F_u + F_b
\]

i.e. after expressing the individual force contributions

\[
F_T = 2\pi \kappa R L \rho \left(\kappa R \right)^2 \Delta p + \rho g L \pi \left(\kappa R \right)^2
\]
where \( L \) represents the length of a plunger penetrated into liquid; \( \Delta P \) is a difference between pressures \( p_0 \) at the entrance to annulus and \( p_L \) at the plunger base; \( \rho_F \) stands for fluid density; \( g \) is the gravity acceleration.

When the plunger is stopped (i.e. \( \varphi=0 \)) a static force \( F_T \) attain an equilibrium value \( F_{Te} \).

\[
F_{Te} = 2\pi R L \tau_0 + \rho_F g L \pi (\kappa R)^2 .
\]  
(29)

From here it follows that

\[
\tau_0 = \frac{F_{Te} - \rho_F g L \pi (\kappa R)^2}{2\pi R L} ,
\]  
(30)

force \( F_{Te} \) is experimentally recorded after the plunger is stopped.

For a determination of flow behaviour index \( n \) and consistency parameter \( K \) the following iterative procedure has to be used:

1. choice of a pressure gradient \( P \) (from rel.(7) for \( T_0 \) it follows that \( P > \frac{2\tau_0}{(1-\kappa)R} \));
2. determination of \( T_0 \):
   A value for dimensionless yield stress \( T_0 \) follows from rel.(7)
3. determination of \( T_w \):
   From rels. (27),(28) we obtain
   \[
   \frac{F_T - \rho_F g L \pi (\kappa R)^2}{\pi L \kappa^2} = T_w + \kappa
   \]  
(31)

From the experimental data we know a value for \( F_T \) (force recorded just before the plunger is stopped) and hence rel.(31) provides a value for \( T_w \).
4. determination of \( \lambda^2 \):
   Consequently we determine \( \lambda^2 \) from rel.(8) written at the point \( \zeta=\kappa \):
   \[
   \lambda^2 = \kappa (\kappa + T_w)
   \]  
(32)
5. determination of \( \lambda_i, \lambda_o \):
   Eqs.(12),(13) provide the values for \( \lambda_i, \lambda_o \) as \( T_0 \) and \( \lambda^2 \) are already known.
6. determination of \( n \):
   Flow behaviour index \( n \) is a solution of Eq.(25) (one equation for the one unknown).
7. determination of \( K \):
   Consistency parameter \( K \) is given by rel.(26).
8. comparison of a value for \( T_w \) given in step 3 with that given by the Vočadlo model, rel.(10):

If a difference of these values exceeds chosen accuracy the whole procedure (steps 1-8) is necessary to repeat.

The whole procedure is concluded after attaining the chosen accuracy of \( T_w \) values when - to an a priori calculated yield stress \( \tau_0 \) - knowledge of two remaining parameters in the Vočadlo model, \( n \) and \( K \), completes its full determination.

**APPLICATION**

As an example the experiment presented in Osorio and Steffe [11] is used. They employed 2% aqueous solution of Kelset (sodium-calcium alginate) from Kelco Co. (at 24°C) for which they determined experimental points shear rate vs. shear stress using a Haake RV-12 viscometer.

Application of a procedure introduced in the preceding section results in the following values characterising the Vočadlo constitutive equation for the aqueous solution of Kelset under investigation:
- \( \tau_0=8.53 \) Pa, determination of this value is independent on a choice of the constitutive equation and subjects to a course of force vs. plunger penetration function, this is a reason why this value was taken over from Osorio and Steffe [11];
- \( n=0.4, K=36.2 \) Pa\(^1/\)s, these values were optimised by the procedure introduced above (using the entry (geometrical and kinematical) data introduced in Osorio and Steffe [11]).

Fig.3 provides a correspondence between experimentally determined points using a Haake viscometer and a flow curve predicted by the Vočadlo model with the parameters determined by a back extrusion process. The agreement seems to be good and satisfactory from the practical viewpoint, on average there is a 12% systematic discrepancy.

Figure 3 Comparison of the experimental data (measured by a Haake RV-12 viscometer, see Osorio and Steffe [11, Fig.9]) with the Vočadlo model
a) solid line - optimised parameters \( \tau_0=8.53, n=0.394, K=42.3 \);
b) dashed line - parameters determined by a back-extrusion method \( \tau_0=8.53, n=0.4, K=36.2 \)
CONCLUSION

The Vočadlo model in its form eliminates a singularity appearing e.g. in the Herschel-Bulkley model. ‘Smoothness’ of the Vočadlo model results in better application to the numerical procedures as e.g. a semi-analytical one in back-extrusion characterisation of rheological behaviour of various materials.

Acknowledgement

The authors are grateful to the Grant Agency CR, Grant Project 103/06/1033, for the financial support of this work.

REFERENCES