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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

$$\tau = K\left|\dot{\gamma}\right|^{n-1} \cdot \dot{\gamma} \tag{1}$$

– 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)

$$\tau = \tau_0 + K\left|\dot{\gamma}\right|^{n-1} \dot{\gamma} \quad . \tag{2}$$

This enables to take into account viscoplastic materials exhibiting a plug-flow region, nevertheless in this model a yield stress τ_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

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

$$\dot{\gamma} = 0 \qquad \text{for} \quad \left|\tau\right| \le \tau_0 \tag{4}$$

where K and n represent consistency and flow behaviour indices, respectively; τ_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

$$\tau_{rz} = \left[K^{\frac{1}{n}} \left| \frac{dv_z}{dr} \right|^{\frac{n-1}{n}} + \tau_0^{1/n} \left| \frac{dv_z}{dr} \right|^{-\frac{1}{n}} \right]^n \frac{dv_z}{dr} \quad \text{for} \quad \left| \tau_{rz} \right| \geq \tau_0 \quad , \qquad (5)$$

$$\frac{dv_z}{dr} = 0 \qquad \text{for} \quad \left| \tau_{rz} \right| \leq \tau_0 \qquad (6)$$

Introducing the following dimensionless transformations (for notation see Figs.1,2 and rels.(3,4))

$$\xi = \frac{r}{R} \quad , \quad \varphi = \frac{v_z}{V} \quad , \quad T = \frac{2\tau_{rz}}{|P|R} \quad , \quad T_0 = \frac{2\tau_0}{|P|R} \quad ,$$
$$\Lambda = \frac{|P|R}{2K} \left(\frac{R}{V} \right)^n \quad , \quad Q = \frac{q}{2\pi R^2 V} \qquad (7)$$

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

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

$$\varphi(\kappa) = -1 \quad , \quad \varphi(1) = 0 \quad , \qquad (9)$$

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

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

where λ^2 is a dimensionless constant of integration, $s=1/n$.

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

$$\lambda^2 = \lambda_i \lambda_o \quad , \qquad (12)$$
$$\lambda_i = \lambda_o - T_0 \quad . \qquad (13)$$

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

$$H(\xi) = \left| \xi - \frac{\lambda_i (\lambda_i + T_0)}{\xi} \right|^s \quad . \qquad (14)$$

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

$$\frac{d\varphi_i}{d\xi} = \Lambda^s \left[\left(\frac{\lambda^2}{\xi} - \xi \right)^s - T_0^s \right] \text{for } \kappa \leq \xi < \lambda_i \text{ (where } \frac{d\varphi}{d\xi} > 0 \text{)} \qquad (15)$$

$$\frac{d\varphi_p}{d\xi} = 0 \text{ for } \lambda_i \leq \xi \leq \lambda_o \qquad (16)$$

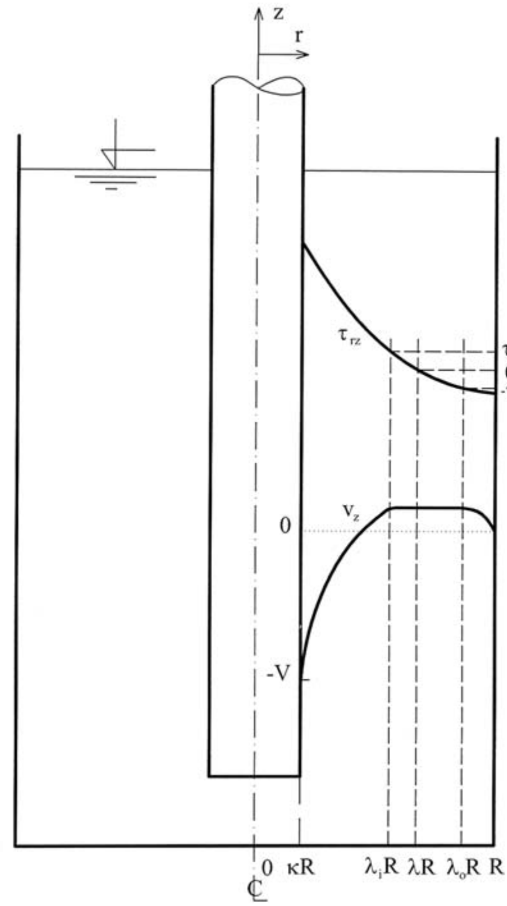

Figure 1 Definition sketch of a back extrusion

$$\frac{d\varphi_o}{d\xi} = -\Lambda^s \left[\left(\xi - \frac{\lambda^2}{\xi} \right)^s - T_0^s \right] \text{for } \lambda_o < \xi \leq 1 \text{ (where } \frac{d\varphi}{d\xi} < 0 \text{)} \qquad (17)$$

From the condition of continuity of the velocity profile

$$\varphi_i(\lambda_i) = \varphi_o(\lambda_o) \qquad (18)$$

it follows that λ_i is a solution of the equation

$$\int_{\kappa}^{\lambda_i} \Lambda^s H(\xi) d\xi + \int_{\lambda_i + T_0}^{1} \Lambda^s H(\xi) d\xi -$$
$$- (2\lambda_i + T_0 - \kappa - 1) \Lambda^s T_0^s - 1 = 0 \qquad (19)$$

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

$$2\pi R^2 V Q = \pi (\kappa R)^2 V \quad . \qquad (20)$$

From here it follows that

$$Q = \kappa^2 / 2 \quad . \qquad (21)$$

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

$$Q=-\frac{1}{2}\left(\frac{1-s}{3+s}\lambda^2-\kappa^2\right)-\left[\begin{array}{l}\frac{1+\kappa^3-\lambda_i^3-\lambda_o^3}{6}-\\-\frac{1-s}{2(3+s)}\lambda^2\left(1+\kappa-\lambda_i-\lambda_o\right)\end{array}\right]\Lambda^s T_0^s+\frac{\Lambda^s}{2(3+s)}\left[\begin{array}{l}\left(1-\lambda^2\right)^{1+s}-\lambda_o^{1-s}\left(\lambda_o^2-\lambda^2\right)^{1+s}\\+\lambda_i^{1-s}\left(\lambda^2-\lambda_i^2\right)^{1+s}-\kappa^{1-s}\left(\lambda^2-\kappa^2\right)^{1+s}\end{array}\right] \tag{22}$$

Comparing rels.(21,22) we obtain

$$-\frac{1-s}{2(3+s)}\lambda^2-\left[\begin{array}{l}\frac{1+\kappa^3-\lambda_i^3-\lambda_o^3}{6}-\\-\frac{1-s}{2(3+s)}\lambda^2\left(1+\kappa-\lambda_i-\lambda_o\right)\end{array}\right]\Lambda^s T_0^s+\frac{\Lambda^s}{2(3+s)}\left[\begin{array}{l}\left(1-\lambda^2\right)^{1+s}-\lambda_o^{1-s}\left(\lambda_o^2-\lambda^2\right)^{1+s}+\\+\lambda_i^{1-s}\left(\lambda^2-\lambda_i^2\right)^{1+s}-\kappa^{1-s}\left(\lambda^2-\kappa^2\right)^{1+s}\end{array}\right]=0 \tag{23}$$

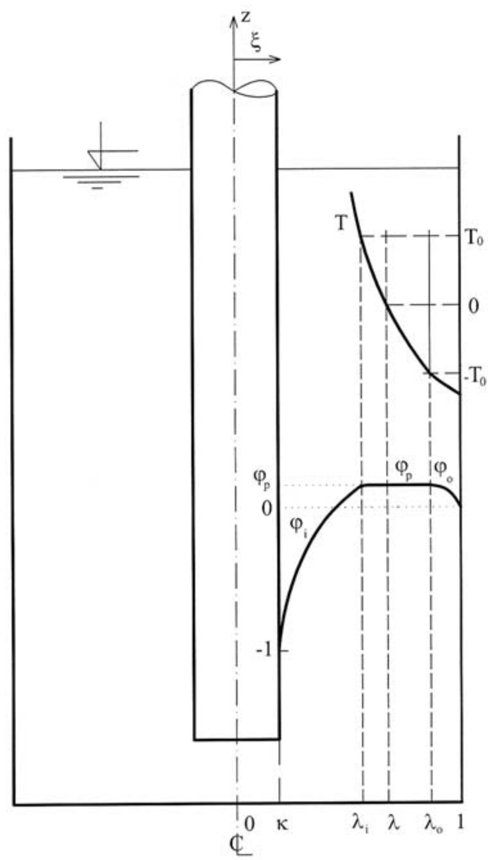

Figure 2 Definition sketch of a back extrusion after dimensionless transformations

Prior to a determination of the empirical constants τ_0, K and n it is useful to eliminate the member Λ^s from the relations (19) and (23). Using rel.(19)

$$\Lambda^{-s}=\int_{\kappa}^{\lambda_i}H(\xi)d\xi-\int_{\lambda_i+T_0}^{1}H(\xi)d\xi-\left(2\lambda_i+T_0-\kappa-1\right)T_0^s \tag{24}$$

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

$$-\frac{1-s}{2(3+s)}\lambda^2\left[\int_{\kappa}^{\lambda_i}H(\xi)d\xi-\int_{\lambda_i+T_0}^{1}H(\xi)d\xi-\left(2\lambda_i+T_0-\kappa-1\right)T_0^s\right]-\left[\frac{1+\kappa^3-\lambda_i^3-\lambda_o^3}{6}-\frac{1-s}{2(3+s)}\lambda^2\left(1+\kappa-\lambda_i-\lambda_o\right)\right]T_0^s+\frac{1}{2(3+s)}\left[\begin{array}{l}\left(1-\lambda^2\right)^{1+s}-\lambda_o^{1-s}\left(\lambda_o^2-\lambda^2\right)^{1+s}+\\+\lambda_i^{1-s}\left(\lambda^2-\lambda_i^2\right)^{1+s}-\kappa^{1-s}\left(\lambda^2-\kappa^2\right)^{1+s}\end{array}\right]=0 \tag{25}$$

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

$$K=\frac{|P|R}{2}\left(\frac{R}{V}\right)^n\left[\begin{array}{l}\int_{\kappa}^{\lambda_i}H(\xi)d\xi-\\-\int_{\lambda_i+T_0}^{1}H(\xi)d\xi-\left(2\lambda_i+T_0-\kappa-1\right)T_0^s\end{array}\right]^n \tag{26}$$

In the next section this relation will complete a determination of the empirical constants τ_0, K and n appearing in the Vocadlo model.

PROBLEM SOLUTION

First, out of three empirical parameters appearing in the Vočadlo model, a yield stress τ_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

$$F_T=F_f+F_u+F_b \tag{27}$$

i.e. after expressing the individual force contributions

$$F_T=2\pi\kappa RL\tau_w+\pi\left(\kappa R\right)^2\Delta P+\rho_F gL\pi\left(\kappa R\right)^2 \tag{28}$$

where L represents the length of a plunger penetrated into liquid; ΔP is a difference between pressures p_0 at the entrance to annulus and p_L at the plunger base; ρ_F stands for fluid density; g is the gravity acceleration.

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

$$F_{T_e} = 2\pi\kappa RL\tau_0 + \rho_F gL\pi\left(\kappa R\right)^2 \quad . \tag{29}$$

From here it follows that

$$\tau_0 = \frac{F_{T_e} - \rho_F gL\pi\left(\kappa R\right)^2}{2\pi\kappa RL} \quad , \tag{30}$$

force F_{T_e} 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

$$\frac{F_T - \rho_F gL\pi\left(\kappa R\right)^2}{\pi L\kappa R^2 \left|P\right|} = T_w + \kappa \tag{31}$$

   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 λ^2:
   Consequently we determine λ^2 from rel.(8) written at the point $\xi = \kappa$:

$$\lambda^2 = \kappa\left(\kappa + T_w\right) \tag{32}$$

5. determination of λ_i, λ_o:
   Eqs.(12),(13) provide the values for λ_i, λ_o as T_0 and λ^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 τ_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 24^0C) 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:

- τ_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 $\text{Pa}^{1/n}$.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.

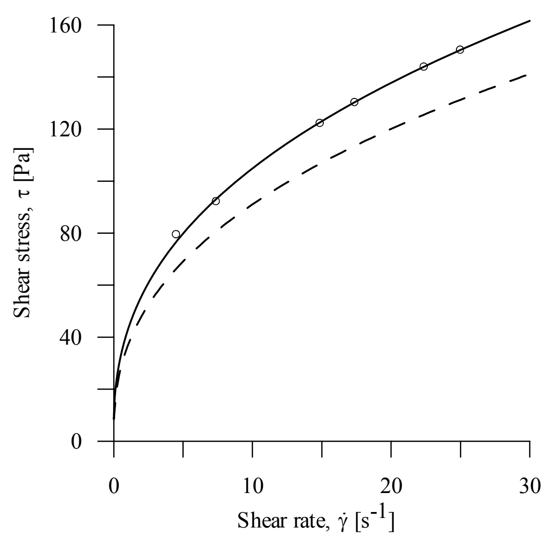

Figure 3 Comparison of the experimental data (measured by a Haake RV-12 viscometer, see Osorio and Steffe [11, Fig.9]) with the Vočadlo model

a) solid line - optimised parameters τ_0=8.53, n=0.394, K=42.3;

b) dashed line - parameters determined by a back-extrusion method τ_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.

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