Determining True Material Constants of Semisolid Slurries from Rotational Rheometer Data

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Abstract. In this work we revisit the issue of obtaining true material constants for semisolid slurries. Therefore, we consider the circular Couette flow of Herschel-Bulkley fluids. We first show how true constants can be obtained using an iterative procedure from experimental data to theory and vice versa. The validity of the assumption that the rate-of-strain distributions across the gap share a common point is also investigated. It is demonstrated that this is true only for fully-yielded Bingham plastics. In other cases, e.g., for partially-yielded Bingham plastics or fully-yielded Herschel-Bulkley materials, the common point for the fully-yielded Bingham case provides a good approximation for determining the material constants only if the gap is sufficiently small. It can thus be used to simplify the iterative procedure in determining the material constants.

Introduction

The main objective in viscometry is to determine objectively fluid material constants independently of experimental and analysis errors. In principle, these material constants are determined by measuring independently the local stress and the rate of deformation. The latter depends on the velocity distribution that must be available for the analysis of the data. In very few cases the velocity distribution is known a priori, but in most cases the distribution is a priori unknown and must be assumed using a constitutive model. However, once the material constants are determined the true rate of strain usually turns out to be different from the assumed rate. This is because the experimentally determined rheology is not necessarily the same as the initially assumed constitutive model. Therefore only “apparent” and not “true” material constants are predicted. As shown in [1] the introduced error in viscoplastic fluids can be significant. This problem can be corrected for instance by proper iteration between the experimental data and the predicted model parameters [2].

Here we focus on an interesting approach proposed by Schummer and Worthoff [3] where in viscometric flows flow curves can intersect at a common point within the rheometer whose location is independent of the rheology of the fluids. Schummer and Worthoff [3] demonstrated this concept for a number of flows and found approximate locations where the resulting experimental error is minimum. Obviously this is a profound result because one can experimentally get “true” material constants without iteration or other corrections. We investigate this concept for the case of Herschel-Bulkley fluids in a rotational rheometer whose basic flow is represented by the classical circular-Couette flow.
In the following sections, we first summarize already available analytical solutions for the circular Couette flow of Herschel-Bulkley materials and then discuss the existence of common intersection points.

![Figure 1. Schematic of the geometry of the flow.](image)

**Theoretical Framework**

Let us consider the steady flow of a viscoplastic material between two co-axial, infinitely long cylinders of radii $R_1^*$ and $R_2^*$, with $R_2^* > R_1^*$. It should be noted that throughout this work the stars denote dimensional quantities. Symbols without stars will denote dimensionless variables and parameters. The inner cylinder is rotating at a constant speed $\Omega^*$ while the outer cylinder is fixed, as illustrated in Fig. 1. The solution of this flow can be found in the literature (see, e.g., [1]). It is thus conveniently summarized here in order to illustrate the range of validity of the assumption of having a common point independent of the fluid rheology.

Under the assumption of axisymmetric flow only in the azimuthal direction, the conservation of linear momentum for any fluid yields

$$\tau_{\phi}^* = \frac{c^*}{r^*^2}$$

(1)

The rate of strain is defined as

$$\dot{\gamma}^* = r^* \frac{d}{dr^*} \left( \frac{u_\theta^*}{r^*} \right) = -r^* \frac{d}{dr^*} \left( \frac{u_\theta^*}{r^*} \right)$$

(2)

The constitutive equation for a Herschel-Bulkley fluid may be written in scalar form as follows:

$$\begin{cases}
\dot{\gamma}^* = 0, & \tau^* \leq \tau_0^* \\
\tau^* = \tau_0^* + k^* \dot{\gamma}^* n, & \tau^* > \tau_0^*
\end{cases}$$

(3)

where $\tau^* = |\tau_{\phi}^*|$, $\tau_0^*$ is the yield stress, $n$ is the power-law exponent, and $k^*$ is the consistency index. The above model is a combination of the Bingham-plastic model ($n=1$) and the power-law model ($\tau_0^* = 0$). The Newtonian fluid corresponds to $n=1$ and $\tau_0^* = 0$.

In what follows, we will use non-dimensionalized equations. The dimensionless variables are defined by

$$u_\theta = \frac{u_\theta^*}{\Omega R_1^*}, \quad r = \frac{r^*}{R_1^*}, \quad \dot{\gamma} = \frac{\dot{\gamma}^*}{\Omega}, \quad \tau = \frac{\tau^*}{\tau_0^*}$$

(4)
With the above scalings, the dimensionless form of the constitutive equation (3) is

\[
\dot{\gamma} = 0, \quad \tau \leq 1 \\
\dot{\gamma} = 1 + \frac{1}{Bn} \dot{\gamma}^n, \quad \tau > 1
\]

(5)

where

\[
Bn = \frac{\tau_0^*}{k^* \Omega^*}
\]

(6)

is the Bingham number.

Thus the non-dimensional rate of strain is

\[
\dot{\gamma} = Bn^{1/n} \left( \frac{c}{r^2 - 1} \right)^{1/n}
\]

(7)

where \( c = c^* / R_1^2 \tau_0^* \). To obtain the velocity \( u_\theta^* \) one simply needs to integrate and apply the boundary conditions. Below a certain critical Bingham number, \( Bn_{\text{crit}} \), the fluid is yielded everywhere in the gap \( 1 \leq r \leq R_2 \). In this case the boundary conditions are \( u_\theta^*(1) = 1 \) and \( u_\theta^*(R_2) = 0 \) (no-slip boundary conditions), which lead to the following expression for the velocity

\[
u_\theta(r) = r \left[ 1 - Bn^{1/n} \int_1^{r_0} \frac{1}{\xi} \left( \frac{c}{\xi^2 - 1} \right)^{1/n} d\xi \right], \quad 1 \leq r \leq R_2
\]

(8)

where the constant \( c \) is calculated by demanding that

\[
Bn^{1/n} \int_1^{r_0} \frac{1}{\xi} \left( \frac{c}{\xi^2 - 1} \right)^{1/n} d\xi = 1
\]

(9)

Above the critical value \( Bn_{\text{crit}} \), the fluid is yielded only partially in the range \( 1 < r < r_0 \), where \( r_0 < R_2 \) is the radial distance from the inner cylinder to the point where \( \tau = \tau_{r\theta} = 1 \).

From Eq. (7) it is deduced that

\[
c = r_0^2
\]

(10)

and therefore

\[
\dot{\gamma} = \begin{cases} 
Bn^{1/n} \left( \frac{r_0^2}{r^2 - 1} \right)^{1/n}, & 1 \leq r \leq r_0 \\
0, & r_0 \leq r \leq R_2
\end{cases}
\]

(11)

and

\[
u_\theta(r) = \begin{cases} 
r \left[ 1 - Bn^{1/n} \int_1^{r_0} \frac{1}{\xi} \left( \frac{r_0^2}{\xi^2 - 1} \right)^{1/n} d\xi \right], & 1 \leq r \leq r_0 \\
0, & r_0 \leq r \leq R_2
\end{cases}
\]

(12)
where \( r_0 \) is a solution of

\[
Bn^{1/n} \int_1^{r_0} \frac{1}{\xi} \left( \frac{r_0^2}{\xi^2} - 1 \right)^{1/n} d\xi = 1
\]  

(13)

In general, Eqs. (8) and (12) are integrated numerically. However for selected values of \( n \), i.e. \( n=1, 1/2, 1/3 \) etc, these can be integrated analytically [1].

The Bingham Plastic Case \((n=1)\). For a Bingham plastic \((n=1)\) it turns out that for \( Bn > Bn_{crit} \),

\[
u_\theta(r) = r \left[ 1 - Bn \left( \frac{r_0^2}{2} \left( 1 - \frac{1}{r^2} \right) - \ln r \right) \right], \quad 1 \leq r \leq r_0
\]

(14)

and

\[
\dot{\gamma} = Bn \left( \frac{r_0^2}{r^2} - 1 \right), \quad 1 \leq r \leq r_0
\]

(15)

where \( r_0 \) is a root of

\[
Bn = \frac{2}{r_0^2 - 2 \ln r_0 - 1}
\]

(16)

Obviously, the critical Bingham number \( Bn_{crit} \) is:

\[
Bn_{crit} = \frac{2}{R_2^2 - 2 \ln R_2 - 1}
\]

(17)

It is instructive to plot \( Bn_{crit} \) versus the outer radius \( R_2 \), as in Fig. 2. The critical Bingham number increases exponentially with the rheometer gap \((R_2 - 1)\). When the dimensionless gap is 0.01 \((R_2 = 1.01)\) the critical Bingham number is so high \((Bn_{crit} = 10033)\) that one can safely assume that the flow is fully yielded for all non-exotic viscoplastic materials. However, if the gap is big the critical Bingham number is low and the possibility of having partially yielded flow cannot be excluded. For example, \( Bn_{crit} = 103.2 \) and 26.54 when \( R_2 = 1.1 \) and 1.2, respectively.

For \( Bn \leq Bn_{crit} \) (fully-yielded flow) the velocity is given by

\[
u_\theta(r) = r \left[ 1 + Bn \ln r - \frac{1 + Bn \ln R_2}{1 - 1/R_2^2} \left( \frac{1}{1 - (1/r_0^2)} - \frac{1}{r^2} \right) \right], \quad 1 \leq r \leq R_2
\]

(18)
Hence, the shear rate is

$$\dot{\gamma} = r \left[ \frac{d}{dr} \left( \frac{u_\theta}{r} \right) \right] = 2 \frac{1 + Bn \ln R_2}{(1-1/R_2^2)r^2} - Bn, \quad 1 \leq r \leq R_2$$ (19)

It is easily verified that the critical radius $r_c$ at which $d\dot{\gamma}/dBn = 0$ is

$$r_c = R_2 \sqrt{\frac{2 \ln R_2}{R_2^2 - 1}}$$ (20)

By substituting into Eq. (19) one finds that the corresponding rate of strain is

$$\dot{\gamma}_c = \frac{1}{\ln R_2}$$ (21)

At this point the shear rate is independent of the Bingham number. By setting $Bn=0$ in Eqs. (18) and (19) the Newtonian expressions are recovered. It should be noted, however, that in this case a scaling different from that of Eq. (4) should be used for the shear stress.

The variation of $r_c$ with the dimensionless gap $(R_2 - 1)$ is illustrated in Fig. 3. For very small gap sizes, i.e. $(R_2 - 1) < 0.1$ this point essentially coincides with the mean radius in the gap, $r_m$. As the gap size is increased, the common point moves closer to the rotating inner cylinder.
Figure 3. Variation of $r_c$ with the dimensionless rheometer gap $(R_2 - 1)$: (a) logarithmic plot; (b) linear plot; $r_m$ is the radius corresponding to the middle of the gap.
Figure 4. Angular velocities (a) and rates of strain (b) for various Bingham plastics ($n=1$) when $R_2 = 1.01$. The red circle corresponds to the common point that exists for $0 \leq Bn \leq Bn_{\text{crit}}$, where $Bn_{\text{crit}} = 10033$. 
Figure 5. Angular velocities (a) and rates of strain (b) for various Bingham plastics \((n=1)\) when \(R_2 = 1.1\). The red circle corresponds to the common point that exists for \(0 \leq Bn \leq Bn_{\text{crit}}\), where \(Bn_{\text{crit}} = 103.2\).
Figure 6. Angular velocities (a) and rates of strain (b) for various Bingham plastics ($n=1$) when $R_z = 1.2$. The red circle corresponds to the common point that exists for $0 \leq Bn \leq B_{n_{\text{crit}}}$, where $B_{n_{\text{crit}}} = 26.54$. 
The Herschel-Bulkley Case for \(n=1/2\). Even though the solutions for \(n=1/2\) and 1/3 are provided in Chatzimina et al. [1], we repeat the former here for convenience. When \(n=1/2\), the critical Bingham number is found to be given by For a Bingham plastic (\(n=1\)) it turns out that for \(Bn > Bn_{crit}\),

\[
Bn_{crit} = \frac{2}{\sqrt{R_2^4 - 4R_2^2 + 4 \ln R_2} + 3}
\]

(22)

For \(Bn > Bn_{crit}\) (partially-yielded flow),

\[
u_\phi(r) = r \left[ 1 - Bn^2 \left\{ \ln r - r_0^2 \left( 1 - \frac{1}{r_0^2} \right) + \frac{r_0^4}{4} \left( 1 - \frac{1}{r_0^2} \right) \right\} \right], \quad 1 \leq r \leq r_0
\]

(23)

and

\[
\dot{\gamma} = Bn^2 \left( \frac{r_0^2}{r^2} - 1 \right) \quad 1 \leq r \leq r_0
\]

(24)

where \(r_0\) is a root of

\[
Bn^2 = \frac{4}{r_0^4 - 4r_0^2 + 4 \ln r_0} + 3
\]

(25)

For \(Bn \leq Bn_{crit}\) (fully-yielded flow), the velocity and the shear rate are respectively given by

\[
u_\phi(r) = r \left[ 1 - Bn^2 \left\{ \ln r - c \left( 1 - \frac{1}{r^2} \right) + \frac{c^2}{4} \left( 1 - \frac{1}{r^2} \right) \right\} \right], \quad 1 \leq r \leq R_2
\]

(26)

and

\[
\dot{\gamma} = Bn^2 \left( \frac{c}{r^2} - 1 \right) \quad 1 \leq r \leq r_0
\]

(27)

where

\[
c = \frac{2R_2^2}{1 + R_2^2} \left[ 1 + \sqrt{1 + \frac{1 + R_2^2}{1 - R_2^2} \left( \ln R_2 - \frac{1}{Bn^2} \right)} \right]
\]

(28)

Results

In the case of a Bingham plastic (\(n=1\)), the rate of strain distributions within the rheometer gap share a common point that is independent of \(Bn\) as long as the material within the rheometer is fully yielded. Figures 4-6 show velocity and rate of strain distributions for three different gap sizes, i.e. \(R_2 = 1.01, 1.1\) and 1.2, respectively. The corresponding values of \(\dot{\gamma}_c\) are 100.5, 10.49, and 5.485. It should be noted that when \(Bn > Bn_{crit}\) the yield point \(r_0\) eventually becomes less than \(r_c\). In other words, there may not be even flow at the “common” point.
Figure 7. Angular velocities (a) and rates of strain (b) for various Herschel-Bulkley fluids with $n=0.5$ when $R_2 = 1.01$. The red circle corresponds to the common point that exists in the case of fully-yielded Bingham flow.
Figure 8. Angular velocities (a) and rates of strain (b) for various Herschel-Bulkley fluids with $n=0.5$ when $R_2 = 1.1$. The red circle corresponds to the common point that exists in the case of fully-yielded Bingham flow.
Figure 9. Angular velocities (a) and rates of strain (b) for various Herschel-Bulkley fluids with $n=0.5$ when $R_2 = 1.2$. The red circle corresponds to the common point that exists in the case of fully-yielded Bingham flow.
Figure 10. Angular velocities (a) and rates of strain (b) in the fully-yielded regime for various Herschel-Bulkley fluids with $n=0.5$ and $Bn=0, 5, 10, 15, 20, 25, \text{ and } 27.37$ when $R_z = 1.1$. The red circle corresponds to the common point that exists in the case of fully-yielded Bingham flow.
Figure 11. Calculated error versus the Bingham number for $n=1$ (Bingham fluid) and (a) $R_z=1.01$; (b) $R_z=1.1$; (c) $R_z=1.2$. The first vertical line corresponds to the critical Bingham number $B_{n_{\text{crit}}}$ below which the flow is fully yielded. The second vertical line corresponds to the critical Bingham number at which the radius of the yielded region coincides with $r_c$. 
Figure 12. Calculated error versus the gap ($R^2 - 1$) for $n=1$ (Bingham fluid) and (a) $Bn=1$; $Bn=10$; $Bn=100$. 

(a) $Bn = 1$

(b) $Bn = 10$

(c) $Bn = 100$
Figure 13. Calculated error versus the Bingham number for $n=0.5$ and (a) $R_2=1.01$; (b) $R_2=1.1$; (c) $R_2=1.2$. The first vertical line corresponds to the critical Bingham number $Bn_{\text{crit}}$ below which the flow is fully yielded. The second vertical line corresponds to the critical Bingham number at which the radius of the yielded region coincides with $r_c$. 
Figure 14. Calculated error versus the gap ($R_2-1$) for $n=0.5$ and (a) $Bn=1$; $Bn=10$; $Bn=100$. 
The results obtained for a representative Herschel-Bulkley fluid with \( n = 0.5 \) and \( R_2 = 1.01, 1.1 \) and 1.2 are shown in Figs. 7-9. The results are similar to those for the Bingham case \((n=1)\), the only difference being that the rate of strain distributions do not share a common point. This is illustrated in Fig. 10 for the case \( R_2 = 1.1 \).

Let us now consider the relative error when using the common point \( r_c \) for calculating the rate of strain rate. This is defined as

\[
\text{error} = 100 \left| \frac{\dot{\gamma}_a - \dot{\gamma}_c}{\dot{\gamma}_a} \right|
\]

(29)

where \( \dot{\gamma}_a \) is the actual rate of strain, and \( \dot{\gamma}_c \) is the rate of strain evaluated at \( r_c \). In the case of a Bingham fluid \((n=1)\) the relative error is obviously zero when \( Bn \leq Bn_{\text{crit}} \) and non-zero when \( Bn > Bn_{\text{crit}}, \) i.e. when the flow is partially yielded. Moreover, for Bingham numbers greater than a second critical value,

\[
Bn'_{\text{crit}} = \frac{2}{r_c^3 - 2 \ln r_c - 1} > Bn_{\text{crit}}
\]

(30)

the flow is unyielded at \( r_c \) and therefore the relative error defined above becomes infinite.

The effects of \( Bn \) and \( R_2 \) on the relative error are illustrated in Figs. 11 and 12. In Fig. 11, the relative errors for \( R_2 = 1.01, 1.1 \) and 1.2 are plotted versus the Bingham number. When \( Bn \leq Bn_{\text{crit}} \) the error is, of course, zero. As already mentioned, \( Bn_{\text{crit}} \) is a decreasing function of \( R_2 \). For \( Bn > Bn_{\text{crit}} \) the error increases rapidly becoming infinite as the Bingham number tends to \( Bn'_{\text{crit}} \). In Fig. 12, the relative errors for \( Bn = 1, 10 \), and 100 are plotted versus the gap \((R_2-1)\). For relatively small \( Bn \) and \( R_2 \) the error can be very small, well below other experimental errors. \( R_2 \) appears to have a more pronounced effect: for small values of \( R_2 \) the error is fairly small even for large values of \( Bn \).

The relative error becomes bigger for different values of the power-law exponent, as illustrated in Figs. 13 and 14, where results for \( n = 0.5 \) are shown. In this case, there is no common point when the flow is fully yielded and hence the error is non-zero even for \( Bn \leq Bn_{\text{crit}}, \) as illustrated in Fig. 13. In general, the relative error increases as \( n \) is reduced.

**Application.** In an actual experiment the wall shear stress is evaluated using the measured torque on the rotating cylinder. Using Eq. (1) then we get \( c = R_i^2 \tau_w \). Therefore the local stress at the common point \( r_c^* \) is calculated as

\[
\tau_c^* = \tau_w \frac{R_i^2}{r_c^*}
\]

(31)

The significance of having a common intersection point is that the local rate of strain \( \dot{\gamma}_c^* \) can be calculated using the Bingham plastic constitutive relation. The flow curve \((\tau_c^*, \dot{\gamma}_c^*)\) can be constructed by varying the rotational speed on the inner cylinder. The rheological parameters can then be obtained by curve fitting. As long as the material is Bingham plastic and the corresponding flow is fully yielded, the evaluated constants are the “true” material constants and no additional corrections are needed. In all other cases, the relative error in \( \dot{\gamma}_c^* \), is of course, non-zero and additional corrections may be needed.
Conclusions

The circular Couette flow of Herschel-Bulkley fluids has been studied. It has been demonstrated that the rate-of-strain distributions across the gap share a common point only in fully-yielded Bingham plastic flows. However, if the gap is sufficiently small the common point for the fully-yielded Bingham case provides a good approximation for determining the material constants in other cases, e.g., for partially-yielded Bingham plastics or fully-yielded Herschel-Bulkley materials.

References

