Research Article

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Rheological Characterization of Yield-Stress Fluids with Brookfield Viscometer

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Abstract: This paper analyzes various techniques to use viscometers equipped with vane spindles to characterize rheological properties of yield stress fluids. Specifically, application of Brookfield viscometers to this end is discussed. A wide selection of toothpastes and lotions were tested. It is shown that a simple method based on apparent shear rate and stress, commonly referred to as a representative viscosity method, works well for moderately non-Newtonian samples but may significantly underestimate viscosity for samples with a more pronounced yield stress behavior. To get more accurate data an integral equation relating torque to angular velocity needs to be solved which can be easily done numerically to get a good agreement between the data collected on an inexpensive viscometer and the data from high-end rheometers.

Keywords: yield stress, vane, Brookfield viscometer, toothpaste

1 Introduction

Brookfield viscometers (currently manufactured by AMTEK) are ubiquitous in food and consumer product industries as well as many others. They are simple to use, inexpensive and reliable. The most widely used setup in industry is based on spindle immersion technique whereby viscosity is measured by immersing a spindle into a cup with a product. The most used spindles are disks and T-bars (RV and T-types in Brookfield manuals [1]). While there are attachments which allow using these viscometers to perform more rigorously defined rheological measurements (narrow-gap concentric cylinders or cone-plate) this immersion technique remains by far the most widely used in industry. However, one must keep in mind that the internal data processing software implemented in Brookfield viscometers assumes Newtonian properties of the tested fluids. This does not prevent many researchers from applying it wider in which case the viscometer works as an indexer, i.e., the measurement has only comparative meaning, not delivering objective, frame-invariant rheological parameters.

Brookfield viscometers work as any strain-controlled rheometers – since they apply angular velocity (reported in rotation-per-minute units, RPM) and measure torque (reported in percentage of the maximum torque specific for the given type of the instrument). Then they convert thus measured torque into viscosity based on the calibration for a Newtonian standard. Mitschka [2] and Wein et al. [3] suggested a method to modify this approach so as to make it applicable to power-law fluids. The conversion they suggested works for disk spindles. This method (often referred to as “Mitschka method”) has gained some popularity and there were examples of its successful applications in the food industry in particular – (see, e.g., Briggs and Steffe [4]). However, for the fluid which exhibits yield stress, i.e., viscosity going to infinity at a certain stress, parametrization in terms of power laws is clearly insufficient. Besides, disk spindles are not optimal for testing such fluids because of the slippage on the surface and significant disturbance of the sample caused by the disk immersion into the sample. There are two most often used tools available for Brookfield viscometers suitable for characterization of yield stress fluids: T-bar and vane. Anderson and Meeten [5] presented an extensive analysis of how the data obtained with T-bar tools can be converted into meaningful rheological parameters in terms of Herschel-Bulkley equation rather than reporting them as some “viscosities” indexes.

Vanes stand somewhat apart because Brookfield introduced a separate test marketed as “yield stress test” which is essential a step-shear test in which the stress measured at a certain angular velocity is interpreted as the yield stress. This approach has some merit as long as the tests are performed at low RPMs. More generally, one would want to be able to obtain the full flow curve to which end RPM and torque has to be converted into shear rate and shear stress.
Vane geometry was used to characterize yield stress materials for a fairly long time – see Nguyen and Boger [6] and references therein. It is widely used in industry to characterize food, cosmetics, toothpastes and many other products [7–9]. The conversion of angular velocity and torque to shear rate and stress is usually justified by an assumption that the flow pattern around the vane closely follows the one around an encompassing cylinders, the “Couette analogy” as some researches refer to this assumption [10]. It was vigorously defended by some researchers [11, 12] and disputed by others [13–15]. If one takes the Couette analogy for granted, the conversion of angular velocity and torque to shear rate and stress can be done by using one of many approximations for a particular type of fluids (see any of multiple textbooks, such as [16], or recent studies, (e.g., [10, 17])).

All the criticism of vane geometry as a rheological tool notwithstanding, there is no doubt that (1) Brookfield viscometry with the vane tool will continue being used in industry as the main tool of characterization of strongly shear-thinning fluids and that (2) Couette analogy will continue being used for processing the data. Accurate application of the Couette analogy to non-Newtonian fluids, of course, requires multiple-point measurement and the corresponding data processing in which readings at several different angular velocities are used to determine the full flow curve. However, in industry users often prefer to get a single-point reading of viscosity or yield stress to which end a simplified conversion is used, sometimes using built-in conversion factors of the viscometer (the so-called “spindle multiplier constant” or SMC for viscosity and “yield multiplier constant” or YMC for the yield stress in the language of Brookfield manuals [1]).

It should be made clear that the goal of this paper is not to study in detail the accuracy of the Couette analogy in general but rather to see how well it works with respect to the specific example of Brookfield rheometry and what error one can expect to get when using single-point version of it as opposed to the multiple-point one. We will show that the full use of the Couette analogy and appropriate data processing allows getting flow curves on these inexpensive viscometers which are fairly close to what one gets when using high-end rheometers with standard rheometric tools. This will be demonstrated using variety of consumer products, toothpastes and lotions, as examples.

2 Materials and Methods

2.1 Materials

All tests reported in this paper were performed on commercially available products, toothpaste and lotions, listed in Table 1. Hereafter they will be referred to by their numbers, 1 through 15. The first eleven were toothpastes, the other four were lotions.

2.2 Methods

The products were tested on a rheometer, ARG2, by TA Instruments using Couette (concentric cylinders, the so-

| Sample ID | Manufacturer       | Brand                            | Type of product |
|-----------|--------------------|----------------------------------|-----------------|
| #1        | Colgate-Palmolive  | Baking Soda                      | toothpaste      |
| #2        | Colgate-Palmolive  | Clean Mint                       | toothpaste      |
| #3        | Colgate-Palmolive  | Max Fresh                        | toothpaste      |
| #4        | Colgate-Palmolive  | Triple Action                    | toothpaste      |
| #5        | Colgate-Palmolive  | Cavity Protection                | toothpaste      |
| #6        | P&G                | Crest whitening scope            | toothpaste      |
| #7        | GSK                | Sensodyne Repair & Protect       | toothpaste      |
| #8        | GSK                | Sensodyne Max Strength Original  | toothpaste      |
| #9        | GSK                | Aquafresh Extreme Clean          | toothpaste      |
| #10       | Colgate-Palmolive  | Total Advanced                   | toothpaste      |
| #11       | P&G                | Crest 3D                         | toothpaste      |
| #12       | Elta MD            | Moisture Rich Body cream         | lotion          |
| #13       | Elta MD            | Face and Body                    | lotion          |
| #14       | La Roche-Posay     | Daily Repair                     | lotion          |
| #15       | Bioelements        | Emollient Body Moisturizer       | lotion          |
Table 2: Parameters of Brookfield viscometers and vane spindles. Also listed are various conversion constants as explained in the text. Spindle to cup diameter ratio 0.1 is assumed.

| Instrument and spindle | spring torque (mN*m) | vane length (cm) | vane diameter (cm) | SS    | TK    | SRC   | SMC   |
|------------------------|----------------------|------------------|--------------------|-------|-------|-------|-------|
| HB, v72                | 5.7496               | 4.333            | 2.167              | 1.799 | 8     | 0.107 | 9.111 |
| HB, v73                | 5.7496               | 2.535            | 1.267              | 8.995 | 8     | 0.107 | 45.554|
| HB, v74                | 5.7496               | 1.176            | 0.589              | 89.718| 8     | 0.107 | 454.379|
| HA, v72                | 1.4374               | 4.333            | 2.167              | 0.450 | 2     | 0.107 | 9.111 |
| HA, v73                | 1.4374               | 2.535            | 1.267              | 2.249 | 2     | 0.107 | 45.554|
| HA, v74                | 1.4374               | 1.176            | 0.589              | 22.430| 2     | 0.107 | 454.379|
| RV, v72                | 0.7187               | 4.333            | 2.167              | 0.225 | 1     | 0.107 | 9.111 |
| RV, v73                | 0.7187               | 2.535            | 1.267              | 1.124 | 1     | 0.107 | 45.554|
| RV, v74                | 0.7187               | 1.176            | 0.589              | 11.215| 1     | 0.107 | 454.379|

called DIN standard) geometry. Serrated cup and cylinder were used to avoid slippage. Shear rate sweeps were performed starting 30 to 0.1 sec\(^{-1}\), 10 logarithmic steps per decade, 20 sec per step while stress was measured by averaging the last 10 sec of each step. Thus measured downshear flow curve may be thought of as an approximation to the true equilibrium curve although the latter may require much longer pre-shear for thixotropic systems. One of us discussed this issue in greater details in [18] where 5 minutes pre-shear at 30 sec\(^{-1}\) was found necessary to accurately predict flow patterns of similar systems (toothpastes) pumped through long pipes. Generally, tests protocol should be adjusted to fit a particular application. In the context of the current work this simplified protocol was used because it fairly well represents a typical quality control test in industrial environment which usually is not conducive to time consuming tests. Below we will briefly illustrate thixotropic effect by showing a typical stress overshoots in step-shear tests at 30 sec\(^{-1}\) to which end a different rheometer was used (ARES-G2, also of TA Industries) because it is better suited for such transient tests.

The flow curves measured on the rheometer were compared to measurements on Brookfield DV2T viscometers. Two types of viscometers were used, HA and HB which differ in the strength of their spring (see Brookfield manual). Two types of vane spindles were used, V74 and V73. The bigger one, V73, was used with HB viscometer and the smaller one, V74 – with HA viscometer. The dimensions of the spindles and other parameters of the instruments are listed in Table 2 which also contains conversion coefficients defined below. The tests on Brookfield viscometers emulated the tests on the rheometer, i.e., RPM (rotation per minute) was swept from 200 down to 0.5 with 20 sec per point in 25 logarithmic steps. Torque on the spindle shaft is recorded in percentage points with an in-house software (or, alternatively, Brookfield Rheocalc software can be used to this end). Conversion of RPM and torque into frame-invariant rheological parameters, shear rate and stress, is discussed in the next sub-section.

All the aforementioned rheometers and viscometers were calibrated on Newtonian standard fluids by TA Instruments and Amtek/Brookfield specialists respectively following standard protocols, i.e., using cone-plate geometries for the rheometers and disk spindles for the viscometers.

2.3 Data processing

For Couette geometry of concentric cylinders (or any other which fits the ‘Couette analogy” as described in the Introduction) most commercial rheometrical software use a conversion of angular velocity, \(\Omega\), and torque, \(T\), into shear rate, \(\dot{\gamma}\), and stress, \(\sigma\), which can be written for their “apparent” values as follows [19]:

\[
\dot{\gamma}_{\text{app}} = C_s \frac{2\Omega}{1-\kappa^2}, \quad \sigma_{\text{app}} = \frac{C_s}{C_{\text{ee}}} \frac{2T}{\pi LD^2}
\]

where \(L\) and \(D\) are the length and diameter of the internal cylinder, \(\kappa\) is the ratio of the internal to external cylinder. Also, there are two coefficients introduced here: \(C_s\) is the so-called shift factor introduced to account for the fact that shear is shifted deeper into the gap and \(C_{\text{ee}}\) is the coefficient accounting for end effects. For the former the following approximation is often used [20]:

\[
C_s = (1 + \kappa^2)/2
\]

Note that the above equations are identical to those implemented in the software for TA Instruments rheometers (see the definitions of the strain and stress factors in the manuals [19] available for download on TA Instruments site).
while Bingham equation had
\[ C_{ee} = -0.0214N^3 + 0.1339N^2 - 0.2975N + 1.27. \] (3)

Thus, it varies between 1.085 for a Newtonian fluid up to 1.27
for a yield-stress flow. For the case of an isolated cylinder
(or any other tool which can be approximated as a cylinder,
say, vane) Nguyen and Boger [6] suggested a simple estimate
based on an assumption that shear occurs right on the
surface of that cylinder:

\[ C_{ee} = 1 + 2R/3L. \] (4)

Eqs. (1–4) represent a typical single-point data processing
method whereby a measurement at a certain angular velocity
is used to calculate the shear rate and stress approximately
corresponding to the condition of this test. This approximation
becomes less accurate as the flow approaches
the yield point in which case the shear actually occurs on
the surface of the tool or very close to it. In that case a
multiple-point test is required. The most commonly used
ones assume a power law dependence of stress on shear rate
or the Bingham equation for the yield-stress materials
(see, [e.g., 16] or some more recent works [10, 17]). Here
we use the most basic method which relies on the general
equation relating angular velocity to torque [16]:

\[ \Omega = \int_{\max(\sigma_y, \kappa \sigma_v)}^{\sigma_v} \frac{\dot{\gamma}}{2\sigma} d\sigma \] (5)

where \( \sigma_v = 2C_e T/\pi LD^2 \) is the stress on the wall of the
rotating cylinder and \( \sigma_y \) is the yield stress. The simplest
way of using eq. (5) is to directly fit the \( T(\Omega) \) data using it
in combination with an appropriate constitutive equation,
e.g., the generalized Casson equation which may be written
as follows:

\[ \sigma = \left[ \sigma_0^n + \eta_\infty \dot{\gamma}^n \right]^{1/n} \] (6)

where \( \eta_\infty \) and \( n \) are fitting parameters along with the yield
stress. This approach is preferred as long as the postulated
constitutive equation is flexible enough to cover properties of
all fluids which are being tested. Having the exponent \( n \) as a fitting parameter provides the required flexibility.
Indeed, this exponent defines the shape of the flow curve.
While Bingham equation had \( n=1 \) and the original Casson
equation had \( n=0.5 \), most of the actual pastes and lotions
we are dealing with here are better fitted with lower \( n \) values
because their flow curves are less “bended” as will be
clear below. This approach was used in [18] to compare
equilibrium flow curves obtained with vane and DIN
geometries confirming that both are indeed very close (except
that there was a misprint in Eqs. (1), but the calculations
were correct).

The above equations are valid for both the actual concentric
cylinders geometry and the vane-in-cup geometry
as long as the Couette analogy is valid. Thus, it will be
applied here to the narrow-gap (DIN) geometry used on the
TA Instruments rheometer as well as to the immersion vane
gometry of Brookfield tests. Here, before turning to the actual
data analysis, it is worthwhile to recall basic features,
notations and terminology specific to Brookfield viscometers.
As already mentioned above, they report data in terms of
torque in percentage points, \( T\% \), of its maximum torque,
\( T_m \), as a function of the applied angular velocity which is
defined in the units of RPM. Brookfield manual and its internal
software relates rheological parameters to \( T\% \) and RPM
by means of several coefficients tabulated in the manuals.
Thus, the apparent values of the shear stress, viscosity and shear rate are defined as follows:

\[ \sigma_{app} (Pa) = SS \times T\%, \] (7)

\[ \eta_{app} (Pa \times s) = TK \times SMC \frac{T\%}{10 \times RPM}, \]

\[ \dot{\gamma}_{app} (sec^{-1}) = SRC \times RPM \]

Here \( SS \) may be called “stress constant”, \( SRC \) is the “shear
rate constant”, \( SMC \) is the “spindle multiplier constant”
and \( TK \) is a constant which defines the type of the instrument.
Note that here we use SI units for viscosity as opposed
to centipoise used by Brookfield (\( cP=0.001 \) Pa*s). Brookfield
manuals do not actually list SS, but SRC and SMC are
tabulated along with \( TK \). Comparing (7) to (1) one comes to
the following definitions:

\[ SS = 0.01 \frac{C_s}{C_{ee}} \frac{2T_m}{\kappa \pi LD^2}, \] (8)

\[ SRC = C_s \frac{\pi}{15(1-\kappa^2)}, \]

\[ SMC = 10 \frac{SS}{TK \times SRC} \]

Table 2 gives the calculated values of these coefficients
for different combinations of the type of the viscometer and
spindles as they are defined by Brookfield. Spindle-
to-cup diameter ratio, \( \kappa \), is assumed here to be 0.1 which
roughly corresponds to a typical measurement (although
the correction to the “infinite sea” case of \( \kappa=0 \) is only 1% and
negligible). Note that SMC value given in Brookfield
manuals (Table D1) is roughly close to what one would get from eq. (8) if end effects are neglected while other constants are not defined at all (their Table D1 gives SRC=0 presumably implying that shear rate cannot be defined).

3 Results

3.1 Transient step-shear tests

Before comparing the flow curves obtained on different devices and their processing, let us first take a look at the simple transient step-shear test in which a constant angular velocity is applied from rest. ARES-G2 rheometer was used to run this test with a vane. This strain-controlled rheometer is best suited for such tests as it reaches the pre-set angular velocity (or apparent shear rate) in around 0.03 sec (ARG2 would take 10 times longer and Brookfield about 100 times longer than that). The results for the three products of our lists are shown on Figure 1. The stress overshoot represents a typical response of a thixotropic material to shear. As one can see, at this shear rate the overshoot is passed in less than 1 sec and subsequently a gradual decline of stress proceeds for an extended period. In a previous work \[18\] one of us have studied similar systems applying long pre-shear prior to running the down-shear sweep to get the best approximation to the equilibrium flow curve as was necessary to correctly calculate pressure drop in a long pipe. While such tests can be performed and may be important for certain applications, in the context of this work relatively short 20-second-per-point test suffices. This allows to wipe-out the history of the sample loading into the narrow-gap DIN geometry and represents a typical condition for a quality control test like those performed with Brookfield viscometers. In the next sections we will show that the results of such tests performed on a rheometer with the standard DIN geometry agree quite well with those performed on a Brookfield viscometer using immersion vane geometry.

3.2 Flow curves-measurements and processing

Examples of the experimentally determined flow curves as well as theoretical calculations are given in Figure 2 for 6 selected toothpastes and two lotions. Experimental points represent the data obtained with Brookfield (filled symbols) and ARG2 rheometer (empty symbols). All these experimental points are the apparent values calculated from the angular velocity and torque according to eqs. (1) through (4). Brookfield data were obtained with HA/V74 or HB/V73 viscometer/spindle combination. Tests on TA rheometer were performed using the narrow-gap Couette (DIN) geometry.

As one can see, for weakly non-Newtonian samples (Figure 2(a)) apparent data obtained on Brookfield and ARG2 rheometer agreed quite well. In these cases, no further actions would be required - apparent values work just fine. However, for stronger shear thinning samples Brookfield considerably underestimated the shear stress, if one uses the conversion based on apparent values as defined by Eqs. (1–4). In other words, one cannot rely on the apparent values to interpret such data. The multi-point data fitting procedure should be used instead. It works as follows.

Instead of working with the apparent variables, the raw data, \(\Omega\) and \(T\), were used directly (the latter are, of course, proportional to the former according to eq. (1)). Thus, \(T(\Omega)\) is calculated according to eq. (5) with the function (6) defined by three fitting parameters, \(\sigma_y\), \(\eta_\infty\) and \(n\). Then the fitting parameters are screened to minimize the standard deviation from the experimental data. Logarithmic variables, Log\((T)\) and Log\((\Omega)\), were fitted which gives closer fit than when using linear ones. The calculated curves with the best fit parameters are shown as dashed curves on Figure 2. Here, for convenience, \(\Omega\) and \(T\) where converted back into the apparent shear rate and stress by eq. (1). Once the best fit values of the fitting parameters are determined, the “true” flow curves are calculated by substituting them into Eq. (6) – those are the solid curves shown on Figure 2.
Figure 2: Experimental and calculated flow curves for selected samples from Table 1. Data for pastes (a,b,c) were obtained with HA/V74 setup, for lotions (d) – with HB/V73. As an example of how the measurements compare, for one sample, #9, both HB/V73 (in green) and HA/V74 are shown. HA or HB symbols here indicate the type of the viscometer spring, while V74 or V73 – the type of the vane as defined in Table 2. Empty symbols on the plots are ARG2 rheometer data, filled symbols – Brookfield viscometers data. Dashed curves fit the latter to predict the former- solid curves are the predictions.

indeed very well approximate the actual measurements on ARG2 rheometer.

One can see that for the 6 examples on Figure 2 the agreement between the predicted “true” flow curves calculated from Brookfield data and the actual curves obtained on ARG2 rheometer is quite good. Prediction quality can be quantified in terms of the coefficient of determination, $R^2$, defined as

$$R^2 = 1 - \frac{\sum (\sigma_{\text{exp}} - \sigma_{\text{th}})^2}{\sum (\sigma_{\text{exp}} - \sigma_{\text{aver}})^2}$$

where summation is performed over all experimental $\sigma_{\text{exp}}$ points measured with ARG2 rheometer, $\sigma_{\text{th}}$ are predicted values calculated with eq. (6) at the same shear rates with the best-fit parameters $\sigma_y$, $\eta_\infty$ and $n$, and $\sigma_{\text{aver}}$ is the average of all $\sigma_{\text{exp}}$. For all the tested samples, the best-fit parameters are listed in Table 3 along with $R^2$. For most of these samples the agreement is good. Also listed are the percentage of points “in spec”, i.e., those which correspond to torque of 10% of $T_m$ or higher. For some pastes and most of the lotions when measured on HA viscometer this number gets rather low, but, curiously enough, the fit often remains good. This is the case with sample #9 for which both HA/V74 and HB/V73 data are shown on Figure 2(c).
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3.3 Data simulation

To explain better the data processing procedure outlined above an imaginary experiment was performed, *i.e.*, the one in which apparent flow curves were simulated by using Casson equation (6) with a predefined set of parameters. Some of thus “simulated” flow curves are shown on Figure 3. They were fitted as the actual data and the fitted curves are also shown (dashed) on the same plot. Recall that what was fitted was actually torque and angular velocity (which are directly proportional to apparent values) but the curves were plotted in terms of the apparent values so as to make comparison to the “true” flow curves easier. The latter were calculated with the best-fit parameters, again the same way as the actual data were processed above. Both apparent and “true” parameters are shown in Table 4. From that table one confirms (which can be concluded by visually inspecting the plot as well) – that the true curves tend to be more “bended” (higher $n$), have higher yield stress and lower high-shear viscosity limit.

The difference between the two predictions is not that big but the one obtained with HA/V74 with only 44% of data points above 10% of torque provided better agreement with the rheometer data.

3.4 Apparent viscosity against the true viscosity

One can compare apparent viscosity measured on Brookfield (or any viscometer which uses the same data processing method) to the “true” viscosity calculated from the same data. In the case of Brookfield, the apparent viscosity is defined from torque reading at a pre-defined RPM following eqs. (7) and (8). Again, this is essentially a single-point method, *i.e.*, no data at other RPMs are needed. To estimate the true viscosity the whole torque-RPM set of data needs to be fitted as explained above to get the best-fit parameters of the Casson equation (6). The true viscosity is estimated at the same apparent shear rate which is calculated from the given RPM. The comparison is shown on Figure 4. The data are summarized in terms the ratio of the true to apparent viscosity and the flow index (flow curve slope in logarithmic coordinates) which for Casson equation is estimated as follows:

$$N = \frac{d\log(\sigma)}{d\log(\dot{\gamma})} = \left(\frac{\eta_\infty \dot{\gamma}}{\sigma(\dot{\gamma})}\right)^n$$

Both actual data for the 15 samples tested with two different viscometers and the simulated data are shown. There is a general dependence which predicts the two values being close for more Newtonian samples (high flow index $N$). However, apparent viscosity significantly underestimates the true viscosity as the behavior becomes more yield-stress-like (hence, low $N$).
Table 3: Fitting parameters for the flow curves obtained with the two configurations: HA type viscometer with V74 spindle and HB with V73 spindle for all fluids from Table 1. Also shown are coefficients of determination estimated when using Brookfield data as a predictor for the data obtained on ARG2 rheometer as explained in the text and the percentage of points which produced the torque above 10% (usually considered “in-spec” by Brookfield).

| instrument/spindle used | HA/V74 | HB/V73 |
|------------------------|--------|--------|
| Sample ID   | \(\sigma_y\) (Pa) | \(\eta_\infty\) | \(n\) | % in spec. | R² | \(\sigma_y\) (Pa) | \(\eta_\infty\) | \(n\) | % in spec. | R² |
| #1         | 1.43   | 0.27   | 0.14  | 46      | 0.995 | 1.03  | 0.17 | 0.13 | 83      | 0.994 |
| #2         | 0.405  | 0.80   | 0.13  | 52      | 0.988 | 0.781 | 1.34 | 0.15 | 82      | 0.999 |
| #3         | 24.2   | 1.15   | 0.30  | 36      | 0.990 | 28.6  | 2.04 | 0.39 | 72      | 0.956 |
| #4         | 80.8   | 1.39   | 0.41  | 56      | 0.988 | 75.0  | 2.53 | 0.53 | 100     | 0.703 |
| #5         | 10.8   | 0.02   | 0.14  | 64      | 0.951 | 16.7  | 0.15 | 0.19 | 100     | 0.941 |
| #6         | 54.5   | 1.24   | 0.32  | 56      | 0.993 | 57.3  | 3.12 | 0.43 | 100     | 0.928 |
| #7         | 206    | 20.10  | 0.48  | 100     | 0.933 | 174   | 16.60| 0.46 | 100     | 0.992 |
| #8         | 2.53   | 0.00   | 0.10  | 76      | 0.885 | 2.13  | 0.01 | 0.11 | 96      | 0.938 |
| #9         | 91.6   | 0.43   | 0.39  | 44      | 0.842 | 84.7  | 0.74 | 0.43 | 100     | 0.554 |
| #10        | 0.130  | 0.17   | 0.10  | 64      | 0.983 | 1.04  | 1.42 | 0.15 | 90      | 0.997 |
| #11        | 16.2   | 0.03   | 0.15  | 68      | 0.959 | 21.5  | 0.17 | 0.19 | 100     | 0.990 |
| #12        | 150    | 0.14   | 0.46  | 68      | 0.402 | 125   | 0.17 | 0.48 | 100     | 0.649 |
| #13        | 16.7   | 0.06   | 0.30  | 0       | 0.902 | 20.6  | 0.14 | 0.40 | 16      | 0.889 |
| #14        | 36.4   | 0.10   | 0.38  | 0       | 0.938 | 34.4  | 0.08 | 0.39 | 32      | 0.895 |
| #15        | 30.9   | 0.00   | 0.17  | 8       | 0.212 | 47.2  | 0.11 | 0.40 | 60      | 0.923 |

Table 4: Apparent parameters used to generate curves on Figure 2 and the best-fit parameters calculated from the simulated curves to produce the “true” curves on that plot.

| apparent values | fitted values |
|-----------------|---------------|
| \(\sigma_y\) (Pa) | \(\eta_\infty\) | \(n\) | \(\sigma_y\) (Pa) | \(\eta_\infty\) | \(n\) |
| 100             | 0.1           | 0.4   | 166             | 0.077           | 0.5 |
| 10              | 0.1           | 0.4   | 16.6            | 0.070           | 0.5 |
| 3               | 0.1           | 0.4   | 4.98            | 0.066           | 0.5 |
| 1               | 0.1           | 0.4   | 1.66            | 0.065           | 0.5 |
| 0.3             | 0.1           | 0.4   | 0.488           | 0.075           | 0.5 |
| 0.1             | 0.1           | 0.4   | 0.158           | 0.087           | 0.5 |
| 0.03            | 0.1           | 0.4   | 0.0474          | 0.084           | 0.5 |
| 0.01            | 0.1           | 0.4   | 0.0156          | 0.086           | 0.5 |
| 0.003           | 0.1           | 0.4   | 0.00452         | 0.090           | 0.5 |
| 0.001           | 0.1           | 0.4   | 0.00148         | 0.089           | 0.5 |

4 Conclusions

Despite certain criticism of the vane method as a general rheological tool, it remains a widely used technique in industry, especially in its implementation as an immersion technique whereby the vane is immersed into a wide vessel filled with the product. In this paper 15 different products, toothpastes and lotions, were tested and the data analyzed using different techniques. The data collected on different Brookfield viscometers with vane spindles were compared to the data obtained on a high-end rheometer (ARG2 by TA Instruments) using concentric narrow-gap geometry. Two ways to process the data were considered. Both methods assume that the flow around the vane is close to the one around its encompassing cylinder (“Couette analogy”). Firstly, the apparent viscosity method was used-the same method commonly used in many lab rheometers. It estimates apparent values of shear rate and stress from torque readings assuming that shear occurs deep inside the gap between the vane and the vessel. This method works fine for moderately non-Newtonian samples. A more general method is based on numerical solution of the integral equation relating torque to angular velocity. The agreement of thus processed data and the data obtained on ARG2 rheometer for all tested products was found to be very good. This justifies this measurement technique as well as the suggested data processing.

Conflict of interest: Authors state no conflict of interest
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