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Simple Method for Determining Stress and Strain Constants for Non-standard Measuring Systems on a Rotational Rheometer

John J. Duffy*, Adrian J. Hill, and Shona H. Murphy

Malvern Instruments Ltd, Enigma Business Park, Malvern, WR14 1XZ, United Kingdom

* Corresponding author: john.duffy@malvern.com

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Abstract:
There is often a necessity to measure, or at least estimate, true viscosity values using non-standard measuring systems on a rotational rheometer. This may be to replicate a mixing or manufacturing process on a lab scale, to keep a sample dispersed and uniform during a measurement or to measure some rheological property that would be difficult or impossible with a standard configuration. Such measurements can be made easily enough, but without a process for converting torque to shear stress and angular velocity to shear rate only these raw data variables can be reported. In this paper a simple and novel empirical method for determining strain/strain rate C_1 and stress C_2 constants for non-standard measuring systems on a rotational rheometer is presented. This method uses relative torque measurements made with a Newtonian and non-Newtonian material and their corresponding power law fitting parameters to determine C_1 and C_2 using a non-linear regression analysis. Equilibrium flow curves generated for two non-Newtonian fluids using two non-standard mixing geometries show very good agreement with data generated using a standard cone and plate configuration, therefore, validating the approach.

Key words:
Measuring system constant, true viscosity, torque, mixer, angular velocity, shear rate factor

1 INTRODUCTION

To measure shear viscosity accurately using a rotational rheometer it is important that the flow field is homogenous and laminar and the shear rate is well defined. For this to be the case the shear gap needs to be small (based on the sample being measured) and linearly dependent on the velocity at the shearing surface. Such conditions are met in the case of 'cone and plate' measuring systems, so long as the angle between the plate and cone is small, because any increase in linear velocity with cone radius correlates with an equivalent increase in shear gap. The working equations for cone and plate are shown below (Equations 1 and 2) with ω the angular velocity, θ the cone angle, τ the torque, and R the radius of the cone.

$$\dot{\gamma} = \frac{\omega}{\theta} \tag{1}$$

$$\sigma = \frac{3\tau}{2\pi R^3} \tag{2}$$

For a 'parallel plate' system the linear velocity increases with radius, but since the gap remains constant the shear rate then varies across the radius of the plate [1] as illustrated in Figure 1. This does not matter too much for Newtonian liquids since both shear stress and shear rate are linearly related, and hence viscosity is constant, however, for non-Newtonian liquids the shear stress has a non-linear dependence and the viscosity will thus vary at different radial locations. This can be largely corrected for by applying a non-linear correction based on the local power law index n [1] or by calculating the viscosity at ¾ of the plate radius instead of the edge, since the shear rate for non-Newtonian and Newtonian materials have comparable values close to this point [2]. The working equations for parallel plates are given below (Equations 3 and 4) based on shear rate calculation at the plate edge and with non-linear corrections applied for shear stress. To implement the single point correction, one just assumes a Newtonian response (n = 1) with the resultant values or equations for $\dot{\gamma}_R$ and σ multiplied by a factor of ¾.

$$\dot{\gamma}_R = \frac{R\omega}{h} \tag{3}$$

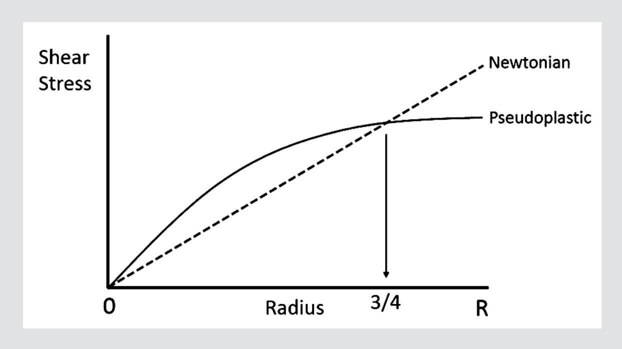

Figure 1: Schematic showing stress distribution across a parallel-plate for Newtonian and pseudoplastic fluids having equivalent viscosity at ¾ R.

$$\sigma = \frac{\tau}{2\pi R^3}[3+n] \tag{4}$$

Both the single point method and power law correction methods can give slight errors compared with measurements made on a cone-plate configuration, especially in the transition region between Newtonian and non-Newtonian behavior. For the single point correction the radial position at which measured stresses are equivalent for Newtonian and non-Newtonian materials can shift slightly from the ¾ position, while for the power law method, estimation of *n* by local differentiation of the torque vs angular velocity data can be another source of error. Similar issues exist for 'coaxial cylinders' since shear stress and shear rate decrease with radial distance from the surface of the rotating inner cylinder, with shear rate showing a non-linear dependence for non-Newtonian materials. In this case errors are minimised by using a small gap and assuming a linear profile across the gap, while for large gaps a non-linear correction needs to be made [1, 4]. An additional 'end-correction' also has to be made to account for any additional shear at the base of the moving inner cylinder [4].

For each of the above measuring systems a strain constant C_1 and stress constant C_2 can be assigned based on the dimensions of the measuring system and the size of the shearing gap. By multiplying by the respective constant the applied or measured torque can be converted into shear stress, the angular displacement in to shear strain and the angular velocity in to shear rate, with any non-linear corrections requiring the power law index *n* usually applied afterwards. The situation gets more complicated for non-standard geometries such as mixers and even regular geometries submersed in a sea of fluid, since the shear rate varies in both the axial and radial directions and the shear stress may not be well defined. For such systems the best one can hope to attain is an average shear rate and a corresponding shear stress such as to give similar viscosity shear rate profiles to those attained with standard geometry configurations such as narrow gap coaxial cylinders or cone and plate.

Various methods exist for estimating stress and strain constants for these geometries some of which have a theoretical basis and others empirical. One empirical method referred to as the 'viscosity matching method' is based on a procedure designed for estimating the viscosity in mixing vessels. Here power measurements or the corresponding torques can be used to estimate the viscosity of Newtonian and non-Newtonian materials at a particular mixing speed, corresponding to an average shear rate. The viscosity is then compared with a flow curve generated on a rotational rheometer to determine the shear rate at which the viscosity is of equivalent magnitude. This method was utilised by Otto and Metzner [5] to determine the shear rate range in a mixing vessel and by Wood and Goff [6] to estimate the average shear rate in the Brabander Viscograph. In a later paper Rao and Cooley [7] referred to this method as the Metzner-Otto-Wood-Goff (MOWG) method. Another method developed by Rieger and Novak [8] uses the relationship between the power number *P* and power law index *n* associated with a mixer to determine a value for the rpm-shear rate conversion factor k_1 since $P \sim k_1^{(n-1)}$. By measuring the power input required to agitate several power law fluids, k_1 can be determined graphically. This method was used by Rao [9] to determine the effective shear rate of a flag impeller and later referred to this method as the RN method to distinguish from the MOWG method described previously.

Castell–Perez and Steffe [10] showed that a constant value of the mixer proportionality constant k_1 was not valid for all types of rheological fluids and was dependent on relative dimensions of the cup and mixer as well as the rotational speed. In a later paper Castell–Perez et al. [11] used a mathematical approach based on the analogy of a power-law fluid in a concentric cylinder system to determine the shear rate and shear stress for a paddle system without the need for standard calibration fluids, which showed good agreement with measurements made on a rotational rheometer. Bousimina et al. [12] derived a model based on the empirical method of Goodrich and Porter [13] for determining the shear rate and viscosity in a batch mixer using an equivalent cylinder principle. This requires estimating the internal radius of an equivalent concentric cylinder system that gives the same torque as the mixer system at an equivalent rotation rate. This principle has also been used by Aerts and Verspaille [14] to produce flow curves from a Brabander Viscograph and both studies showed good agreement with rheometer data. A separate numerical method was derived by Williams [15] for determining true viscosity-shear rate data for a rotating disk in a sea of fluid based on a Brookfield viscometer.

There is clearly a necessity to measure, or at least estimate, true viscosity values using non-standard measuring systems as evidenced from the studies and applications cited previously. This may be to replicate a mixing or manufacturing process on a lab scale, to keep a sample dispersed and uniform during a measurement or to measure some rheological property that would be difficult or impossible with a standard configuration. Such measurements can be made easily enough, but without a method for converting torque to shear stress and angular velocity to shear rate only these raw data variable can be presented. The purpose of this paper is to propose and discuss a simple empirical method that can be performed with a rotational rheometer to estimate the shear stress and shear rate for non-standard measuring systems. The proposed method is not too dissimilar to some of those cited previously but is much simpler and requires fewer experimental steps than either the RN or MOWG methods. The procedure is based on Equation 5 which has previously been used to estimate power law parameters for a simple mixer using measured torques and angular speeds [16]. This same equation can, however, be used to determine the proportionality constant between angular velocity and shear rate C_1 if the power law parameters K (consistency) and n (power law index) are known for a standard Newtonian fluid (denoted by the subscript N) and non-Newtonian fluid (denoted by the subscript P). The power law index n is 1 for a Newtonian liquid so does not appear in Equation 5.

$$\frac{\tau_P}{\tau_N} = \frac{\sigma_P}{\sigma_N} = \frac{K_P\left(C_1\omega\right)^n}{K_N\left(C_1\omega\right)} \tag{5}$$

By making torque measurements with a non-standard geometry at a defined angular velocity in both the Newtonian and non-Newtonian fluid fluids (under laminar flow conditions), the relative stress value σ_P/σ_N can be estimated from the torque ratio τ_P/τ_N. Inputting this value along with the power law parameters K_N, K_P, and n into Equation 5 leaves just one unknown, which is C_1. This equation can then be solved using a non-linear regression analysis with the objective to minimise the residual between the calculated relative stress (σ_P/σ_N) and the measured relative torque (τ_P/τ_N) by iteratively changing C_1 until an optimum solution is found. A key benefit of this approach is that the value determined for C_1 is optimised for both Newtonian and non-Newtonian materials since the optimization method uses data generated for both fluid types to find a solution. This is effectively the same as locating the radial position on the parallel plate where the shear stress is equivalent for a Newtonian and non-Newtonian material at a given shear rate. Once C_1 has been determined this value can be substituted in to Equations 6 and 7 to determine individual values for σ_P and σ_N.

$$\sigma_P = K_P\left(C_1\omega\right)^n \tag{6}$$

$$\sigma_N = K_N\left(C_1\omega\right) \tag{7}$$

The corresponding proportionality constant C_2, required to convert torque to shear stress, can then be determined from Equation 8 by inputting σ_P or σ_N and their corresponding torques τ_P and τ_N. Both sets of values should yield similar values of C_2 if an optimised value for C_1 was found from the non-linear regression analysis, however, an average of the two values should be used to give a single optimum value of C_2.

$$C_2 = \frac{\sigma}{\tau} \tag{8}$$

It should be noted that constants generated by this method will only be relevant for the particular measuring configuration and fluid volume used to determine them. The measurement process can be summarised as follows:

- Determine power law constants for a Newtonian oil (1 Pas oil) and a power law fluid (hair gel or body lotion) by making measurements with a suitable cone and plate geometry and fitting a power law model to the data. A higher viscosity fluid will minimise turbulence in the proceeding step.
- Measure the steady state torque for each fluid at an equivalent angular velocity (1 rad/s) using the uncalibrated geometry in the appropriate vessel, ensuring the correct gap and same fill volume are used for both measurements.
- Perform a non-linear least square analysis with the objective to minimise the residual between the calculated relative stress σ_P/σ_N and the measured relative torque τ_P/τ_N by iteratively changing C_1. This can be performed with the Solver Tool in Microsoft Excel, employing the Generalized Reduced Gradient (GRG) Nonlinear analysis function [16] or another technical computing software package.
- Calculate σ_P and σ_N by substituting in to Equations 6 and 7 and divide by τ_P and τ_N respectively as indicated by Equation 8 to give two values for C_2 and take the mean.

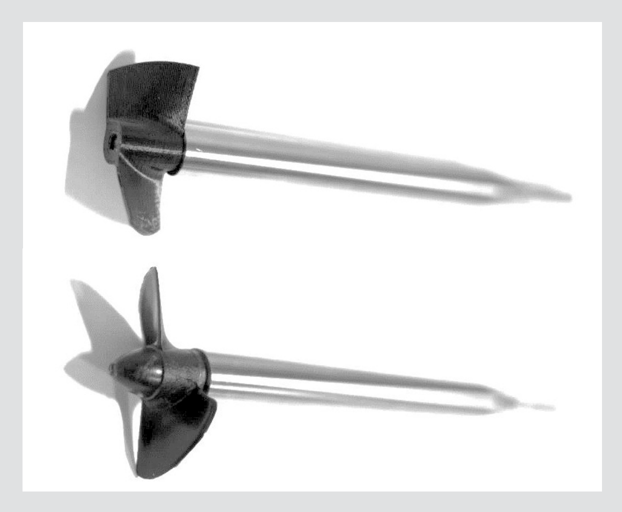

Figure 2: Photographs of the Mixer A (above) and Mixer B (below) used in this study.

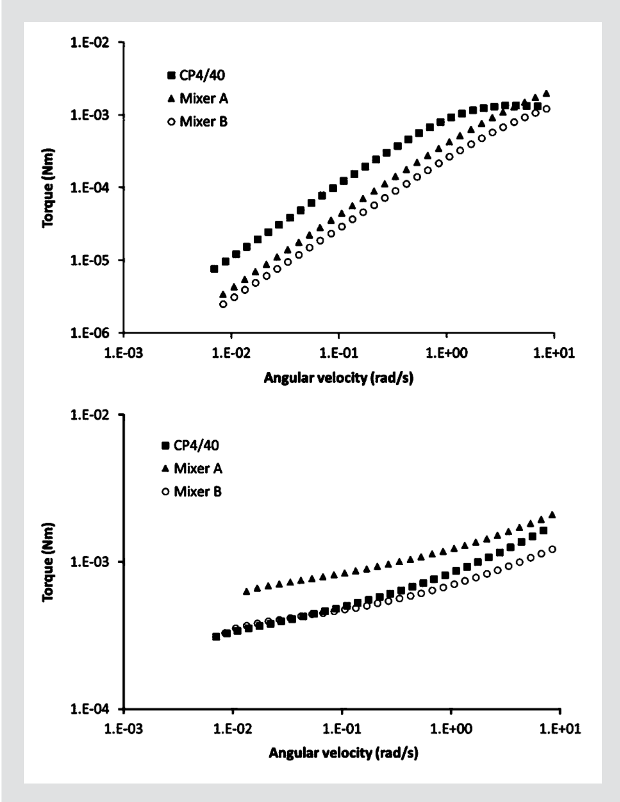

Figure 3: Torque-angular velocity curves measured with Mixer A, Mixer B, and a cone-plate geometry on a body lotion (above) and body wash (below).

2 MATERIALS AND METHODS

Two non-standard mixing geometries Mixer A and Mixer B were used in this study, both connected to a stainless steel shaft as shown in Figure 2. To determine the stress and strain constants for these systems a commercial body lotion was employed as a standard power law fluid and 1 Pas oil as a standard Newtonian fluid. All measurements were made using a Kinexus Pro+ rotational rheometer (Malvern Instruments) at a temperature of 25 °C. The power law parameters for each of these fluids were determined using an equilibrium table of shear rates test with a 4°/40 mm cone and plate measuring system then fitting a power law model to the resultant data to determine values of K and n. For the body lotion K was determined to be 28.7 and n was 0.23. Steady state torque measurements were then made with both the body lotion and 1 Pas oil in a 37.5 mm cup at an angular velocity of 1 rad/s. Non-linear analysis was performed using the Solver Tool in Excel, employing the Generalized Reduced Gradient (GRG) Nonlinear analysis function [16, 17]. The calculated values of C_1 and C_2 were validated by performing an equilibrium table of shear rates tests on a surfactant structured body wash and a body lotion, and comparing the data with that generated using a cone and plate measuring system.

3 RESULTS AND DISCUSSION

Torque versus angular velocity plots are shown in Figure 3 and demonstrate clear differences in the torque-angular velocity profiles obtained with the various measuring systems, making any meaningful comparison difficult at best. This approach therefore is really only valid where relative comparisons are required between similar samples. To get the curves to overlay requires shifting the data both vertically and horizontally, with the shift factors being the stress constant C_2 and strain constant C_1, respectively. For Mixer A the calculated strain constant C_1 and the average stress constant C_2 were determined to be 2.82 and 29802. The individual calculated stress constants used to determine this average value were 29787 for the Newtonian oil (n = 1) and 29817 for the non-Newtonian fluid (n = 0.23) so very little difference between the two.

Equilibrium flow curves for the two non-Newtonian fluids measured with the Mixer A and a cone and plate geometry are shown in Figure 4. For the body lotion the agreement between the standard and non-standard geometries is excellent across the measured shear rate range, validating the approach for generating the constants. Good overlap is also seen for the body wash product with the onset of shear thinning occurring at a similar shear rate for both geometries. There is some slight variation in observed curvature in the transition region between Newtonian and power law behavior, which is to be expected, since the flow field is most complex and variable in this region and hence it is difficult to generate values for C_1 and C_2 that work across all shear rates and materials. This is an inherent limitation of the method but is also an issue with the parallel plate and wide gap concentric cylinder configurations albeit to a lesser extent. For Mixer B the calculated shear rate constant C_1 and the average shear stress constant C_2 were determined to be 3.126 and 53317, respectively. The individual stress constants used to determine this average were 53284 and 53350, respectively for the Newtonian and non-Newtonian

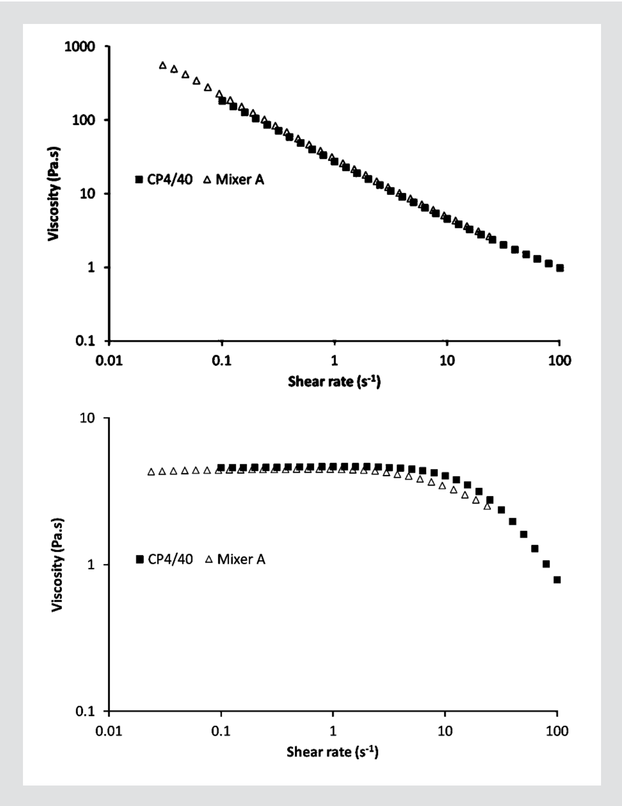

Figure 4: Flow curves measured with Mixer A and a cone-plate geometry on a body lotion (above) and body wash (below).

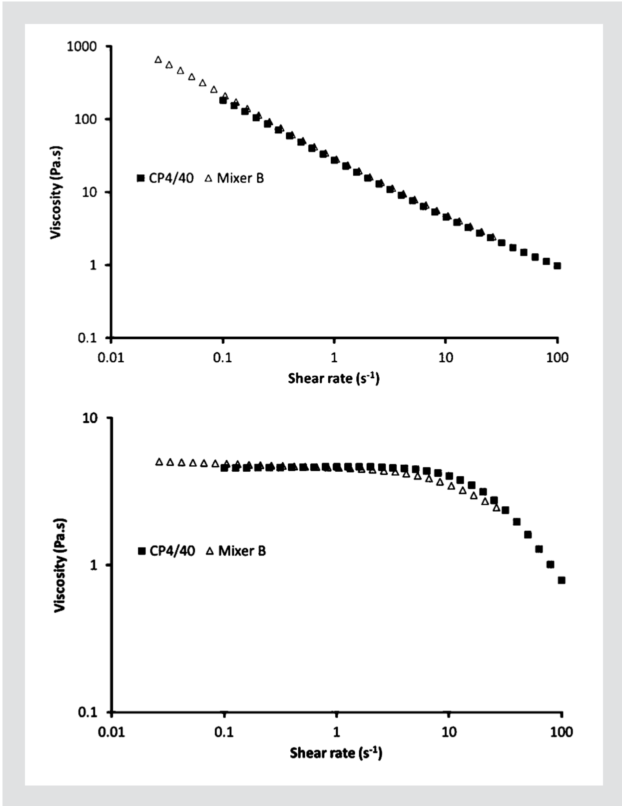

Figure 5: Flow curves measured with Mixer B and a cone-plate geometry on a body lotion (above) and body wash (below).

fluid, so again similar values. Equilibrium flow curves for the two non-Newtonian fluids measured with Mixer B and the cone and plate geometry are shown in Figure 5. As with Mixer A the agreement between the two configurations is very good particularly for the body lotion. For the body wash product the same discrepancy in the transition region observed with Mixer A is observed which is again attributed to the complex and variable flow field in the vicinity of the mixer.

This study clearly demonstrates the feasibility of the approach taken to estimate stress and strain constant for non-standard measuring systems and one that is relatively quick and easy to perform. As stated in the introductory section, there are clearly benefits of being able to generate comparable rheological data to that obtained with a standard geometry configuration using non-standard measuring systems or mixers and/or non-standard vessels. This may be to replicate mixing, for keeping a sample dispersed during a measurement or to measure some rheological property that would be difficult or even impossible with a standard measurement configuration. In theory it should also be possible to generate C_1 and C_2 constants for larger scale mixing vessels using this approach if mixer torques and mixing speeds are known. If the power input is linearly dependent on torque then it may also be possible to replace the torque with power input in Equation 5.

4 CONCLUSION

A simple and novel empirical method for determining strain/strain rate C_1 and stress C_2 constants for non-standard measuring systems on a rotational rheometer is proposed. This method uses relative torque measurements made with a Newtonian and non-Newtonian material and their corresponding power law fitting parameters to determine C_1 and C_2 using a non-linear regression analysis. Equilibrium flow curves generated for two non-Newtonian fluids using two non-standard mixing geometries showed very good agreement with data generated using a standard cone and plate configuration, therefore, validating the approach.

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