Measurement Errors in Yield Stress Rheometry that Arise from Torque Auto Zero

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Abstract:
The measurement of the shear rheology of concentrated particulate suspensions is important to a range of mixing, pumping and flow operations. The use of a four or six bladed vane attached to a rheometer in an open cup is a popular technique to achieve a rheological characterisation. A problem occurs in the use of automated software with a number of rheological devices for yield stress materials. A torque auto zero default causes the torque at the start of a test to be ignored, and can result in significant errors and underestimation of the yield stress or rheological response of the suspension. The potential effect of using a torque auto zero default is demonstrated for a concentrated particulate suspension of nickel laterite.

Zusammenfassung:
Die Messung der Scherrheologie von konzentrierten partikulären Suspensionen ist für viele Misch-, Pump- und Fließprozesse von Bedeutung. Die Anwendung von 4 oder 6 Rührschaufeln in einem offenen Behälter bei einem Rheometer ist eine beliebte Methode zur rheologischen Charakterisierung. Ein Problem tritt bei der Anwendung der automatisierten Software bei einer Vielzahl von rheologischen Geräten bei Materialien mit einer Fliessgrenze auf. Ein automatisches Nullsetzen des Drehmoments führt dazu, dass das Drehmoment bei Testbeginn ignoriert wird, und verursacht signifikante Fehler und eine Unterschätzung der Fließgrenze oder der rheologischen Antwort der Suspension. Der potentielle Einfluss des automatischen Nullsetzens wird für eine konzentrierte, partikuläre Suspension aus Nickellaterit vorgeführt.

Résumé:
La mesure de la rhéologie de cisaillement de suspensions concentrées de particules est importante pour toute une gamme d'opérations comprenant un écoulement telles que le mixage et le pompage. L'utilisation d'une géométrie de type vane équipée de 6 à 4 lames attachée à un rhéomètre dans un cylindre ouvert est une technique populaire pour effectuer une caractérisation rhéologique. Un problème émerge lorsque un logiciel automatisé est utilisée avec un nombre d'appareils rhéologiques pour la caractérisation de matériaux à contrainte seuil. L'absence d'une mise à zéro automatique du couple a pour conséquence le fait que le couple au démarrage du test est simplement ignoré, et entraîne des erreurs significatives et la sous estimation de la contrainte seuil ou de la réponse rhéologique de la suspension. L'effet potentiel de l'absence de mise à zéro automatique du couple est démontré pour une suspension concentrée de particules de latérite de nickel.

Key words: Yield stress, suspension rheology, vane rheology, concentrated suspensions, particles

1 INTRODUCTION

There are a number of routes to the measurement of the yield stress of concentrated particulate suspensions [1]. These include, for example, extrapolation of shear stress-shear rate data to zero shear rate, commonly referred to as the Bingham yield stress [2 - 4], using the point of deviation from linear viscoelastic behaviour in a oscillatory measurement for increasing angular offset [5] and measurement of the maximum torque response in the initial rotation of a roughened bob or multi-bladed vane immersed in a suspension [6, 7]. The latter technique has become increasingly popular and a large number of rheometers are now supplied with a vane attachment. Despite the apparent simplicity of the measurement, researchers have shown that it is not just applicable to quality control and if used properly, can provide useful information as to the state of dispersion and role of additives in manipulating the flow behaviour of suspensions [8 - 10].

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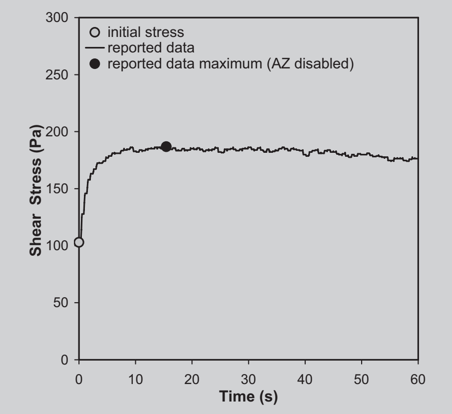

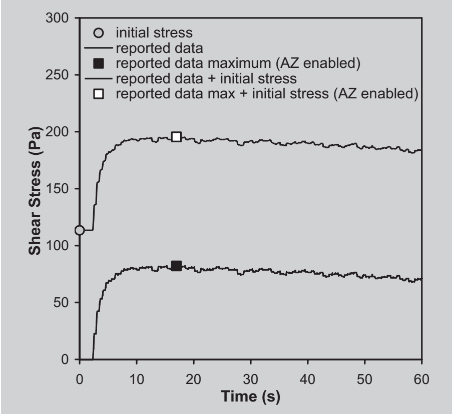

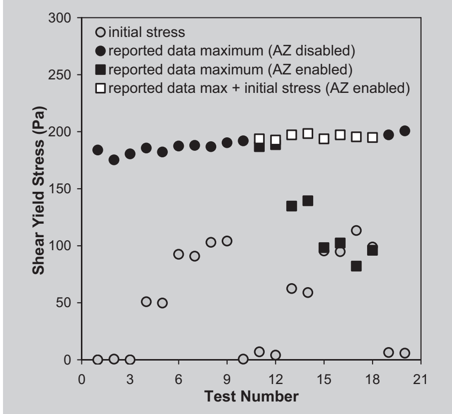

Figure 1 (left):
A single vane yield stress measurement for a 0.56 w/w nickel laterite suspension using a Haake rheometer with the torque auto zero (AZ) disabled (chart shown is that of Test 8 in Fig. 3).

Figure 2 (middle):
A single vane yield stress measurement for a 0.56 w/w nickel laterite suspension using a Haake rheometer with the torque auto zero (AZ) enabled (chart shown is that of Test 17 in Fig. 3). The dashed curve was obtained by shifting the curve reported by the software (solid line) vertically by a magnitude equivalent to the initial stress.

Figure 3:
Vane yield stress measurement results for a 0.56 w/w nickel laterite suspension using a Haake rheometer with the torque auto zero (AZ) both enabled (squares) and disabled (circles). The respective initial stress for each test (crosses) relates to the stress induced on the vane fixture immediately prior to the commencement of the yield stress test. 'reported stress' is that given by the software. Yield stresses were determined by the maximum torque response at a constant vane rotation rate (see Figs. 1 and 2 for examples).

A large sample was homogenised with a laboratory overhead stirrer and then sub-sampled for rheological measurement. Samples were rested for a period of at least one hour prior to measurement.

Yield stress measurements were made on a Haake Model VT550 using the vane yield method [6, 7]. The four bladed vane had dimensions D_v = 20.2 mm and L_v = 20.2 mm. The vane was fully immersed into the sample, then the cup was slightly rotated to induce an actual initial stress on the vane before the test was started. In reality, initial stresses could be caused during the sample loading or would be present from a prior test if a sequence of tests were being performed on a single sample without reloading. The initial stress present was read off the rheometer digital display immediately prior to commencing the test using the software interface. For the yield stress testing, the vane was rotated at 0.2 rpm for 60 seconds and the stress response was recorded. A typical torque response without using a torque auto zero is shown in Fig. 1.

Shear stress-shear rate sweeps were made using the same rheometer and Haake MV1P bob and MVP cup, both of which are profiled to minimise slip. The MV1P bob had the dimensions D_b = 40.08 mm and L_b = 60 mm and the MVP cup was of diameter D_c = 42 mm. Tests were performed in a measurement mode whereby the shear rate was applied for 12 seconds at each point. All tests were performed at ambient temperature.

3 RESULTS AND DISCUSSION

Figure 1 shows a typical torque response from a yield stress test with an initial torque present, and the torque auto zero (torque offset correction) function disabled. The yield stress is taken as the maximum stress measured during the test (filled circle data points). To contrast this Fig. 2 shows a measurement on the same sample with the torque auto zero enabled. This is where the initial torque present immediately prior to the commencement of the test is automatically zeroed and not taken into account during the test and hence the reporting of the sample yield stress.

Figure 3 shows the yield stress results for a lateritic nickel suspension for 20 consecutive measurements. Tests 1 - 10 and 19 - 20 were performed with the torque auto zero disabled. That is the torque acting on the vane immediately before the start of the test was not zeroed. The grey filled data points show the initial stress (directly proportional to the initial torque) acting on the fixture immediately before each test was started. The initial stresses in tests 1-10 ranged from 0 to 104 Pa. Despite this variation in initial stress, the yield stress results reported (shown as filled circle data points) were very consistent. Tests 11 - 17 were performed with the torque auto zero enabled. That is the torque acting on the vane immediately before the start of the test was offset back to zero. The initial stresses in tests 11 - 17 ranged from 4 to 113 Pa. As the results clearly show, the greater the initial stress on the vane, the lower the yield stress reported by the software (shown as filled squares data points). The results using the two methods were brought into coincidence when the initial stresses for tests 11 - 17 were accounted for by adding them to their respective reported yield stress results (shown as unfilled square data points). The slight increase in the sample yield stress from the start to the finish of testing was consistent with an increase in solids fraction of the sample of 0.002 due to drying (increasing from 0.56 to 0.562 w/w). It is clear that despite the ambiguity of the software package explanation, the correct approach is to disable the torque auto zero (no torque offset correction).

For shear rate and shear stress sweeps, the use of a torque auto zero will subtract the initial stress present on the fixture from every measurement in the sweep, again creating erroneous results and hence difficulty in reproducibility. Figure 4 shows shear stress, shear rate sweeps performed with the torque auto zero disabled. The initial stresses induced on the fixture (shown as grey filled data points) ranged from 1 to 42 Pa.

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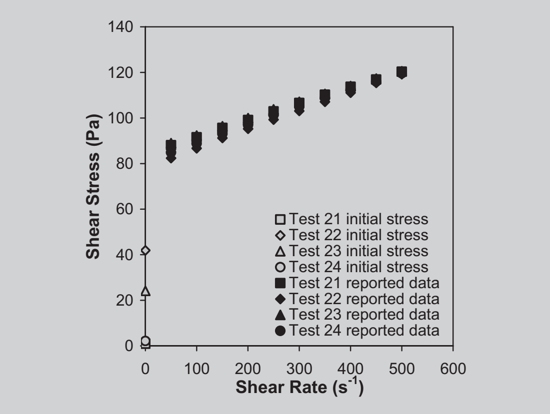

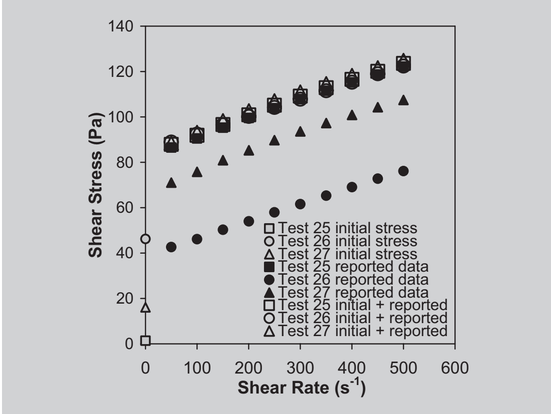

Figure 4 (left): Shear stress-shear rate sweeps for a 0.56 w/w nickel laterite suspension using a Haake rheometer (MV1P bob and MV1 Cup) with a measurement mode of 12 seconds per point and the torque auto zero disabled. Grey filled data points indicate the stress acting on the fixture immediately before the start of the test.

Figure 5: Shear stress-shear rate sweeps for a 0.56 w/w nickel laterite suspension using a Haake rheometer (MV1P bob and MV1 Cup) with a measurement mode of 12 seconds per point per point and the torque auto zero enabled.

These initial stresses have little detectable effect on the final test results reported (shown as black filled data points) as a torque auto zero function was not used, hence the initial torque was taken into account when the test began.

In testing a yield stress material, if the initial stress induced on the fixture before the start of the test is zeroed, the errors can be significant. Figure 5 shows shear stress-shear rate sweeps with the torque auto zero enabled. This gives very different results depending on the initial stress present. These initial stresses ranged from 1 to 46 Pa (grey filled data points in Fig. 5). If this initial stress is simply added to each data point reported by the software in each respective test (black filled data points in Fig. 5) the results show good reproducibility (unfilled data points in Fig. 5, "initial + reported") and consistency with the results presented in Fig. 4.

CONCLUSIONS

With modern rheometers and more automated software packages, it is still important to have a good understanding of good rheological procedure to obtain meaningful results. The testing of yield stress materials is a good example, where the rheometer torque should be manually zeroed before a sample is loaded and an automatic torque zero function (torque offset correction) should not be used. If a sample is loaded perfectly and no stress is induced on the fixture (the initial stress present is zero), zeroing the torque will make no difference, however if a stress is acting on the fixture due to loading or as a residual from a previous test in a sequence, using an automatic zeroing function can cause significantly erroneous results and difficulty with reproducibility.

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