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Linking yield stress measurements: Spread test versus Viskomat

Robert J. Flatt [a,*], Domenico Larosa [a], Nicolas Roussel [b]

[a] Sika Technolog y AG, CH-8048 Zürich, Switzerland
[b] Laboratoire Central des Ponts et Chaussées, Paris 75732 Paris Cedex 15, France

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Abstract

Two rheological tools widely used in the cement industry are compared: the Viskomat rheometer and the spread test. It is shown that proper calibration of the first, and selection of an adequate physical model for the second yield to a quantitative agreement of yield stress values. Such results are important for industrial labs that may translate existing data in arbitrary units into rheologically relevant parameters.

Keywords: Rheology; Workability; Admixtures

1. Introduction

The rheology of cementitious materials appears to be gaining increasing interest and is a probable result of the growing use of highly flowable concrete such as SCC. An ongoing ACI committee is striving to compare existing concrete rheometers and seems to have concluded all predict the same trends in rheological parameters although there is still debate on the origin of the discrepancy among the values themselves [1].

Obtaining correct numerical values for these rheological parameters is important in several ways. First of all, there is a major interest from industry for reducing the extent of experimental work to predict trends in concrete rheology as a function of mix design. The so-called multiscale or homogenisation approach is the most likely to fulfil this ambition. It is based on using properties of a matrix and solid inclusions to predict the properties of a new material. For example mortar rheology would be predicted from paste rheology and the volume fraction, shape and size distribution (packing resulting from these) of the sand particles. Concrete would be predicted in a similar way from the mortar rheology and the nature and amount of the aggregates [2]. This type of physical approach was not adapted to traditional concretes, the behavior of which was dominated by grain to grain contacts. Any prediction of this behavior had to take into account all the interactions between the particles, even the smallest, to estimate the maximum packing fraction [3]. The behavior of modern concretes without even speaking of SCC is getting closer to the behavior of a suspension. The physical concept of inclusions in a suspending fluid becomes adapted to the description of each of the constitutive phase of the concrete. In the process of validating such approaches, one needs to know quantitatively if the predictions are correct and this cannot be done if exact rheological parameters cannot be determined (agreed on). Furthermore, fluid dynamic simulations are making significant progress and complex flow patterns of form filling are being investigated. An engineering science of the casting phase is appearing. In such cases, the pertinence of the results and their use is strongly dependent on the accuracy of the input. Once again, quantification is needed. Finally, depending on the situation, one of various pragmatic but fast tests might be used on a routine basis. This will function well until additional questions or requirements lead to testing by complementary methods. How to compare the results, how to avoid duplicating experiments that ultimately all give the same information are all questions where quantification will help. Fluid mechanics can bring answers to these numerous questions and help understanding the meaningfulness of an existing test if proper rheological parameters are entered in the analysis. Two examples of such cases are dealt with in this paper. The first concerns flow tests and the second the rheometer called Viskomat.

* Corresponding author.
E-mail address: flatt.robert@ch.sika.com (R.J. Flatt).

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The Viskomat rheometer was initially developed for the bread industry but has since enjoyed a wide success in the cement related industry for measuring cement paste or fine mortar. It is used to obtain flow curves that have units of torque instead of shear stress and sample holder rotation rate instead of shear rate. Flow curves obtained with that instrument are often linear and Bingham fitting is largely used. However since conversion to proper rheological units is close to nonexistent, such results are difficult to compare with other tests and can barely be used to predict properties in concrete. In this paper it is shown that both torque and rotation are probably proportional to an average shear stress and an average shear rate. This facilitates comparison with results from other test methods.

The other method examined is the paste spread. It is also very widely used in cement industry. The reason is because it is extremely quick. It is essentially a variation on the slump test except that in this case one measures the diameter at the base of the sample and that the sample container is much smaller. It is very popular for testing admixtures in cement paste [4]. As for slump, it is well accepted that the spread diameter is related to yield stress if inertia effects can be neglected (i.e., mould slowly lifted). However, different relations can be found in the literature. A limited number of experiments presented in this paper confirm that these relations cannot be used beyond the range of spreads for which their underlying hypothesis hold.

In this paper, it is shown that the yield stress obtained from a calibrated Viskomat and the yield stress obtained from a spread measurement show quantitative agreement. This is a useful result for industrial laboratories that extensively use both methods.

2. Background on yield stress and spread

Slump as well as spread have been shown to be related to the yield stress τ_0. However, what the exact nature of this relation is has undergone some debate. Some correlations are purely empirical, while others are based on a physical model. In the later category one can distinguish two main approaches. They are outlined below with a focus on relating spread rather than slump to yield stress. It will be emphasized that both models do not have the same scaling with sample volume and spread radius. The underlying hypothesis make them relevant in different ranges of yield stress and an interpolation function is proposed for the specific sample volume which is widely used for screening admixture effects in cement paste.

2.1. Murata approach

The first approach used to describe slump as a function of yield stress was from a purely mechanical point of view and was first presented by Murata [5]. With a similar approach, Pashias et al. [6] using the same approach published paper with a very eloquent title about the appeal of using slump-type experiments to determine yield stress: “A fifty cent rheometer for yield stress measurement”. This model was improved by Schowalter and Christensen [7] and more recently by Saak et al. [8] who generalized it for various geometry of sample holder (from cone to cylinder). A similar approach that also involved finite element modelling was presented by Davidson et al. [9].

The model is almost exclusively presented as a way of determining yield stress from slump. However as we will see the underlying hypothesis is very easily used for determining yield stress from spread radius.

The basis principle is that the sample is considered as a stack of layers, which, under the load from the layers above, each deform at constant mass until the radial stress in each layer equals the yield stress. The yield criterion is then not fulfilled anymore and flow stops. There exists a zone in the cone (i.e., the top layers) that are subject to stress smaller than the material yield stress and do not deform. In the initial Murata approach, the shear stress was taken to be one half of the gravity load giving:

$$\tau_0 = \frac{\rho g H}{2} = \frac{\rho V g}{2\pi R^2}. \quad (1)$$

The basic hypothesis of the layer deformation without flow among the layers is clearly unsatisfactory for large spreads. Either there is flow among the layers, giving a uniform load as above, or there is no flow among the layers and stress on the bottom layer is clearly not uniform. At limited spreads however, this hypothesis probably holds. On the other hand, it has to be noted that all the components of the stress tensor are neglected in this approach compared to the shear stress. The sample at stoppage is assumed to be under pure shear. On a theoretical point of view, this assumption is valid only for large spread [10]. This obvious contradiction is due to the oversimplification of the yield criterion to a monodimensional relation linking the shear stress and the yield stress. In the case of limited spreads, the flow is all but monodimensional.

More recent work on the pastes with low spread demonstrates that the deformation of such samples involves extensional flow [11]. For very low spread, there is no shear stress in the sample. The yield criterion is in fact fulfilled by the three principal components of the stress tensor. Using a three dimensional treatment of von Misès yield criterion, a very similar but more rigorous expression is then obtained:

$$\tau_0 = \frac{\rho g H}{\sqrt{3}} = \frac{\rho V g}{\sqrt{3}\pi R^2} \quad (2)$$

where ρ and V are respectively the density and the volume of the sample, R is the radius of the spread and H the height of the collapsed sample. It has to be noted that recent experimental results by Chamberlain [12] showed that this relation holds for initial height to initial radius ratio between 0.8 and 2.

Inclusion of this 3D yield criteria yields, the slump can be recalculated using the Murata approach. Doing so, we find that paste data for a cylindrical mold reported by Saak et al. [8] is slightly better matched at high yield stress or low slump values (Fig. 1).

Other than this illustration, within the scope of this paper, we restrict our selves to mentioning that a rather long demonstration is needed to show that Eq. (1) is not correct [11] and does not represent any existing flow situations.

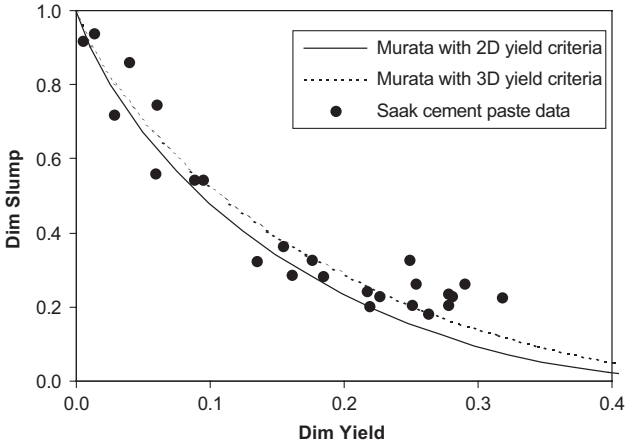

Fig. 1. Data from Saak et al. [8] showing dimentionles slump versus yield stress (for the cylindrical mould). The continuous line represents the Murata approach prediction using the 2D yield criteria, while the discontinuous line is what is obtained with the 3D yield criteria.

However, it is quite easy to show that Eq. (2) is more adapted to describe slump flows.

Let us imagine a cylindrical mould in which we can pour various amounts of a yield stress fluid and let us assume that the Murata approach and the extensional approach are both acceptable flow patterns. The first one is based on a purely shearing flow and the second one on a purely extensional flow.

The imaginary experiment we do here consists in pouring a level H of a given yield stress fluid in the cylindrical mould and then lifting the mould. If H is high enough, there will be a measurable slump; otherwise, H will not vary and will be increased for the next experiment.

The Murata approach predicts that we have to pour a level $H=2\tau_0/\rho g$ to get a measurable slump. This will never happen because the extensional flow pattern will first occur for $H=3^{1/2}\tau_0/\rho g$, which is smaller. As always, Nature will choose the easiest path, which is here the extensional flow pattern.

Finally, in regard to this approach, we point out that this modified version is expected to perform better at small than at large spreads. This theoretical expectation is supported by the following practical experience. Admixture dispersing effects are often tested on cement paste using a cylinder of 50 mm diameter and 50 mm high. In such tests, one typically observes that extremely well flowing pastes with yield stress values no larger than 1–2 Pa do not flow to diameters much larger than 200–250 mm, as can be seen for example in the data presented by Schober and Mäder [4]. In those experiments, water cement ratios were typically 0.23 and 0.25, giving densities 2.25 and 2.20 g/cm^3. Using the average of these densities, Eq. (1) would predict that the yield stress at maximum spread would vary between 38 and 22 Pa. This is much higher than the 1–2 Pa measured experimentally, and the above equation does not hold for large values of spread and the discrepancy cannot simply be accounted for by experimental error.

2.2. Roussel approach

Recently Roussel et al. [13] have presented a convincing treatment of how the diameter of samples exhibiting large spreads can be related to yield stress. The treatment uses the so-called long wave approximation. This allows to express the variation of pressure in the sample according to height or radius as well as the variation of pressure in the sample as a function of height. Successive integrations lead to expressing the sample height as a function of radius. Volume integration for a fixed sample volume then yields the desired expression of yield stress. In fact, for very large spreads another term must be added to account for surface tension effects. The final equation reads:

$$\tau_0 = 1.747\rho V^2 R^{-5} - \lambda\frac{R^2}{V} \tag{3}$$

where ρ and V are respectively the density and the volume of the sample, R is the radius of the spread and λ is a constant linked to

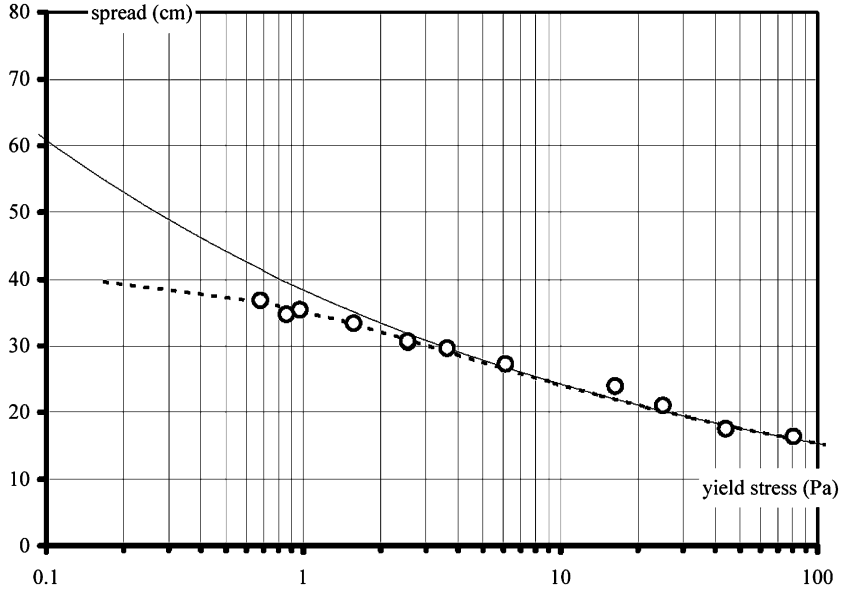

Fig. 2. Data by Roussel et al. [13] showing correlation between measured yield stress and spread of sample in ASTM minicone test.

the liquid vapor interfacial energy and the wetting angle on the plate.

The data Roussel et al. [13] present in support of this expression is given in Fig. 2 The continuous line corresponds to the first term of Eq. (3) thus neglecting surface tension effects. The discontinuous line includes surface tension, but the value of λ is fitted (0.005). However, the authors indicate that the value obtained is reasonably close what could be expected based on water-vapor surface tension and contact angle with the substrate.

Thus this model appears to be reliable for large spreads. In addition in regard to the large spread situation discussed previously, a sample density of 2.25 g/cm^3 that would spread between 200 and 250 mm would have a yield stress between 3.8 and 1.2 Pa. This is now clearly in the range of the values found in a rheometer with parallel serrated plates.

On the other hand, Eq. (3) as stated by the authors really only holds for large values of spread. Thus this expression must be considered as covering a complementary range of yield stress values as the corrected Murata type approach.

It is important to note that both models do not offer the same scaling with either sample volume or spread radius. This was to be expected as the flow patterns vary very much between the low spread regime (three dimensional extensional flow) and the high spread regime (purely shearing flow with a dominating radial velocity). Some limited experiments presented below were done with different sample volumes to test the pertinence of each of these models in the different regimes.

3. Materials and methods

The cement used in the Viskomat experiments is an equal part mixture of three Swiss OPC, which is used to reduce batch to batch variability that can occur with a single supplier. For the spread experiments with different cylinder sizes, the cement was a Korean OPC.

3.1. Sample volume effect

For testing whether the yield stress expressions propose the correct dependence on sample volume and spread radius, experiments were done with different cylinder sizes filled with cement paste from the same batch and measured at the same time. The dimensions of these cylinders are given in Table 1. The surface on which the spread was measured was a dry glass plate.

In the first series of samples, only cylinders 1 and 2 were used. The spreads ranged for the reference cylinder from 128 to 254 mm. One polymer was used at three dosages and measurements were done after 0, 30 and 60 min. In the second series of experiments, the three cylinders were used. For these, two different polymers each at a fixed dosage were measured at 0, 30 and 60 min.

Table 1
Sizes of cylinders used

	Diameter [mm]	Height [mm]
Cylinder 1	50	51
Cylinder 2	30	50
Cylinder 3	50	25

The paste preparation involved adding the cement into a container under mechanical mixing and already containing the water and admixture. The mixing time and the mixing rate come from an internal procedure giving reproducible paste properties with a degree of structural breakdown apparently similar to take place when preparing mortars. However, as the material from the same batch was used to fill the different cylinders, the exact state structural breakdown in the sample is at best only of secondary importance in this case.

3.2. Viskomat rheometer calibration

The Viskomat rheometer is an instrument largely used in the cement industry. It is produced by Schleibinger Geräte Teubert u. Greim GmbH (Buchbach, Germany) and the model used in this study was a Viskomat NT. It consists of a thermostated sample holder (a cup of about 500 mL) which rotates at a variable speed. In the center of the cup is an anchor that is fixed to a torque cell above it. At the cup edge, a fixed knife/spatula scrapes material along the cup wall in an effort to bring it back into the center and avoid excessive radial segregation.

Flow curve on cement pastes or fine mortars typically show linear relations, which are fitted most often using a Bingham model. However, units are given in min^{-1} for the rotation rate and in N.mm for the torque. Conversion to rheological units of s^{-1} and Pa is barely ever presented in the cement literature. The complexity of the flow profile has been considered as a barrier to this and this rheometer is consequently used mainly for comparative purposes.

Tatersall and Banfill [14] have proposed a calibration procedure for the two-point test concrete rheometer (see Appendix A). It involves using a Newtonian fluid and at least two power law fluids. These fluids must be measured in a reference rheometer (giving flow profiles in rheologically relevant units) as well as in the rheometer to be calibrated.

The calibration done for the Viskomat in this paper used a silicon oil (200 fluid—30'000 cps form Dow Corning) as Newtonian fluid and aqueous solutions of high molecular weight MHPC at 2% and 2.5% by mass as the power law fluids. The preparation of those solutions led to significant air entraining and the samples had to be left to rest enough time for before measuring them in each rheometer. Once all foam and apparent bubbles had disappeared, part of the sample was transferred slowly to the measuring cup of the Viskomat. The cup was placed covered in the Viskomat and left to rest, covered, in order to allow any entrapped air to escape and to let the sample reach the measuring temperature of 20 °C. Furthermore to limit air entraining during the measurement, the flow curves were measured with increasing rotation frequencies of (5, 10, 20, 40, 80, 160 min^{-1}). Each step lasted 2 min. The data from the highest step was systematically discarded because of excessive air entraining.

From the rest of the sample not used for that experiment, a small portion was measured in a rheometer by Paar Physica (model MCR 300; Ostfildern, Germany) using a cone plate

geometry. Flow curves were measured by increasing shear rate between 0.01 and 1000 s^{-1} at logarithmically spaced steps, reducing the duration of each step as the shear rate was increased. This method was found to limit problems of sample centrifugation during the test. At high shear rates, when the risk of induced heterogeneity is the highest, the step duration is the shortest. On the other hand, as the characteristic time to reach steady state decreases with strain rate, the shorter step duration does not perturb the test results in the high strain rate range. Anyway, the shear rate range relevant for Viskomat turned to be much lower than the maximum value measured, only about 20–30 s^{-1}. All data above this range of shear rates is therefore not relevant to the calibration and is not presented in this paper.

3.3. Cement paste measurements in Viskomat rheometer

The cement paste that was measured in the Viskomat contained 1% (w.r.t. cement) of a retarder solution (Sika Tard 930). The water cement ratio was varied between the experiments and no superplasticizer was added. The objective of these experiments was to have pastes with varying yield stress and which would only undergo limited irreversible modification of this yield owing to ongoing hydration during the experiment.

Because of the larger sample volume required by the Viskomat, initial mixing was done in a Hobart mixer. The water and retarder mixed together were added into the bowl already containing the cement. The total mixing time was 3 min. For the thickest pastes, the mixing was interrupted after 1 minute to homogenize manually the mix with a spatula (edges were not well mixed initially). After the mixing was complete, the samples were again mixed by hand before placing into the Viskomat measuring pot (about 460 ml).

All samples were then pre-sheared during 6 min at 160 min^{-1}. This reduced substantially the torque, close enough to a plateau value to consider the sample had been well preconditioned and a steady state had been reached. The rotation frequency was then reduced at logarithmically spaced steps (160, 80, 40, 20, 10, 5 min^{-1}), each step lasting 2 min. Flow curves were established with data corresponding to steady state only. Then the samples were sheared once more at 160 min^{-1} for 6 min to return the sample to a degree of agglomeration as close as possible to what it had before the flow curve measurement initiated.

At the end of this procedure the sample holder was removed. Some dry cement paste at the top edge of the sample was removed with a spoon, taking care not to drop any in the sample. The sample was then mixed with a spoon. Any scraping of the edges of the base was avoided in these cases. After this, the cement paste was poured into a reference cylinder (#1) and placed on a dry glass plate. The whole was placed on a scale and the weight determined. At that point the mould was lifted and the remaining mass determined. This allows to correct the spread mass for the amount of the sample remains stuck to the cylinder. It introduces a minor but non-negligible correction for the samples with very low spread (about 15–20%). Once the sample spread was stable, 4 diameters were measured and the average taken. The tube and plate were then washed and the experiment repeated. Spreads of the second measure were systematically lower though only moderately so.

4. Results

4.1. Sample volume effect

For the experiments done with different sample volumes, the yield stress values were calculated from Eqs. (2) and (3). Results are shown in Fig. 3.

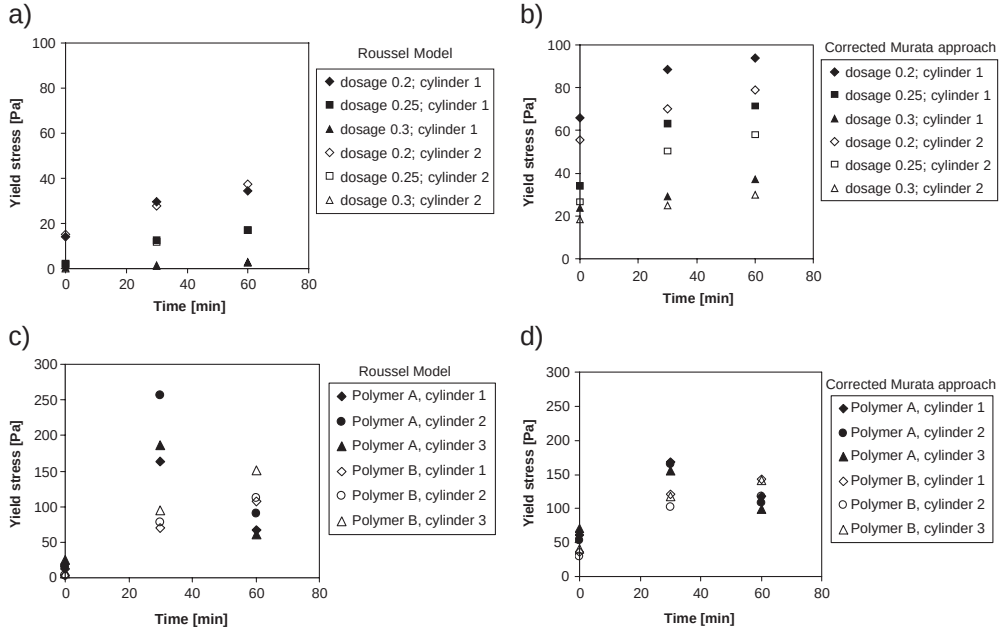

Fig. 3. Comparison of calculated yield results for different cylinder sizes, a & b using Eq. (2), c and d using Eq. (3).

The results show an interesting trend. Data in Fig. 3 a and c for which the yield stress values are lower (larger spreads), show a better agreement between measurements with the different cylinders. This is what should be expected if the model gives the right scaling with volume fraction. On the other hand, for the second series of experiments which gave much higher yield stress (smaller spreads), the situation is the opposite. This confirms our initial statement that the corrected Murata type approach is relevant at low spreads while the Roussel approach holds at large spreads. On the basis of the results presented here, the boundary yield stress between the two solutions application range seems to be around 100 Pa. Of course, this boundary value depends on the tested cylinder volume and this value is only an initial approximation for the three tested cylinders.

4.2. Role of surface tension

It must be noted that the above results were obtained without determining a specific value of the surface tension parameter. To see the role that surface tension can play, the following graph reports yield stress calculated from spread with λ having values of 0.005 and 0. In addition the error that would be made without including surface tension effects in samples where $\lambda = 0.005$ is reported for the reference cylinder #1. Fig. 4 clearly indicates that surface tension effects become important above about 21 cm for this cylinder.

4.3. Interpolation between both models

Despite the problem of the exact determination of the surface tension linked parameter λ, we propose for convenience to use an interpolation function between both models using the value of λ given by Roussel et al. [13] for their experiments. This is done for the reference cylinder (#1) which is widely used for testing admixture effects on cement paste. For other samples volumes the interpolation below will not be valid.

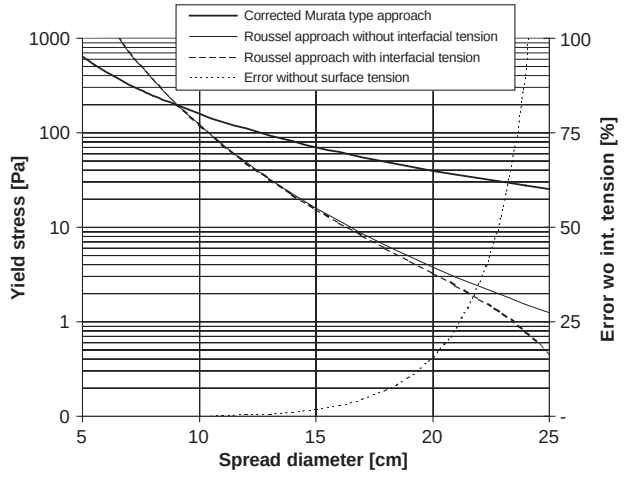

Fig. 4. Relation between yield stress and spread diameter calculated with Roussel's model and the corrected Murata type approach for the cylinder used in spread tests at Sika (50 mm diameter, 50 mm height). The sample density is 2.25 g/cm^3. Each model is plotted in its application range only. In the case of the Roussel's model, discontinuous line is what would be obtained without interfacial tension effects, continuous line includes these effects. The dotted line shows the error that would be committed if interfacial tension effects were neglected.

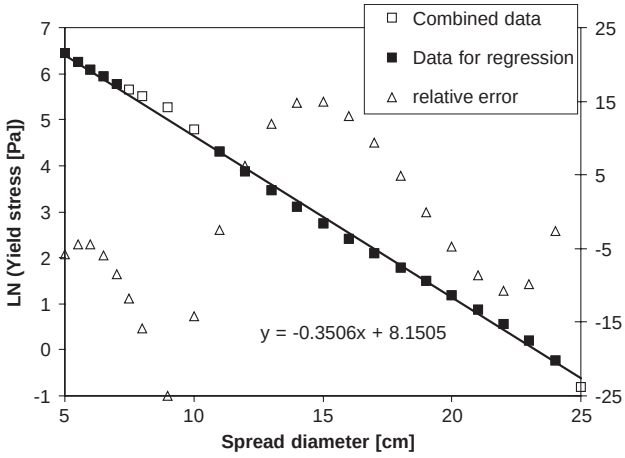

Fig. 5. Interpolation between both models, open squares represent the lowest yield value predicted by either the corrected Murata type approach or the Roussel model including surface tension. The sample density is 2.25 g/cm3 as in Fig. 4 data giving minimum yield predictions and data selected to propose an interpolation function, relative error in % is given for the points chosen in the regression.

The predictions of both models are plotted on Fig. 4. The minimum value predicted by either the modified Murata or the Roussel approach (with surface tension effects included) is

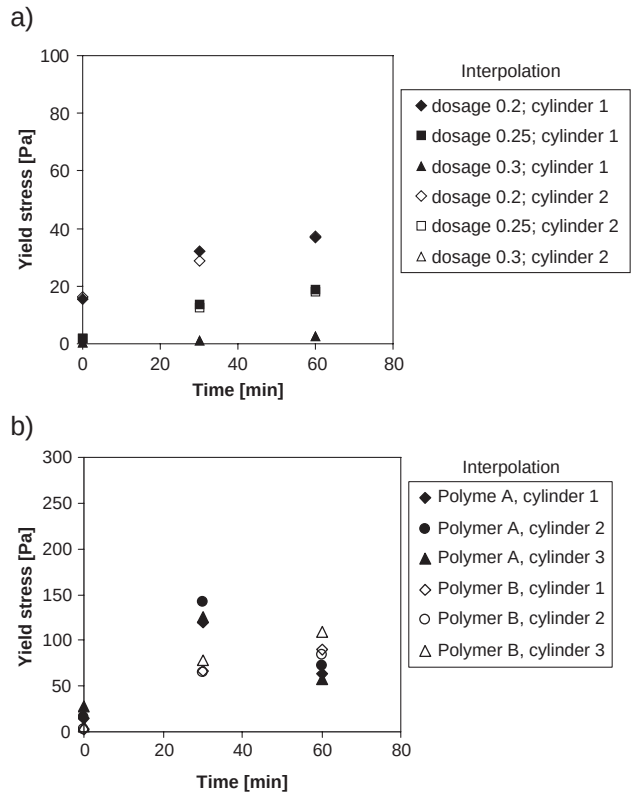

Fig. 6. Comparison of calculated yield results for different cylinder sizes, using exponential functions to interpolate between the limits given by Eqs. (2) and (3). a) Series with 1 admixtures and 3 dosages, b) series with 2 admixtures and 1 dosage.

plotted in Fig. 5 (open squares). From this, data is selected where the prediction of one or the other model is assumed correct (filled squares). An exponential function is found to fit this data quite well. The error with respect to the function giving the lowest yield value is plotted in the secondary axis and is found to be small (open triangles). Typically, it is smaller than 25%. The largest errors occur where neither model is expected to give a proper results (around the boundary yield stress value 100 Pa, $LN(\tau_0) \cong 5$). The error rises again for very large spreads, but this is in the range where surface tension effects become so predominant that the spread test itself for such a low yield stress material becomes quite questionable anyway.

The above results suggest that the yield stress is approximately given by an exponential function of spread diameter over the wide range of diameters of interest. We can thus write:

$$\tau_0 = a\exp(-bR) \quad (4)$$

where a and b are fitting parameters given in Fig. 5.

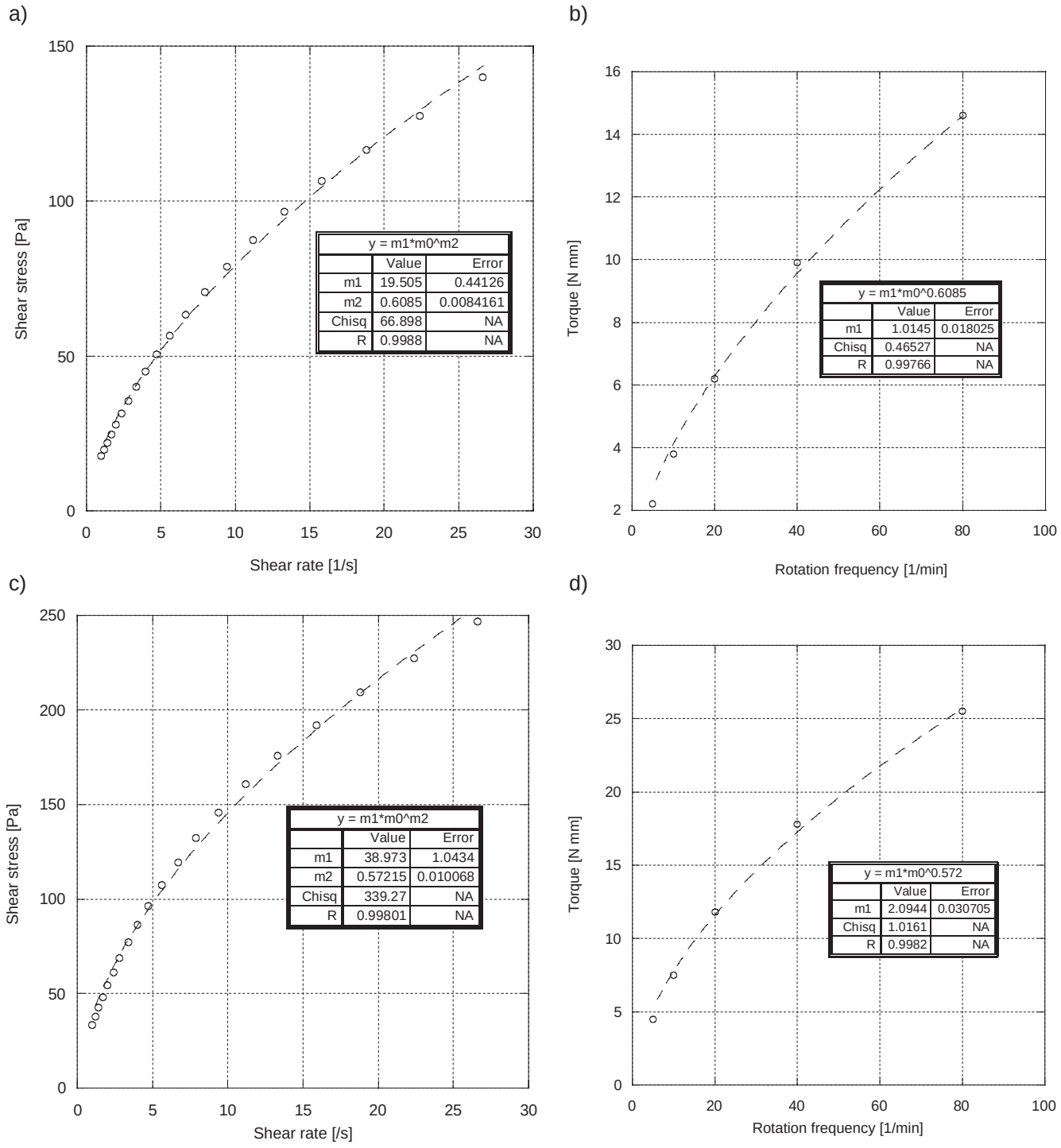

Fig. 7. Flow curves of calibration fluids in cone plate (a and c) and Viskomat (b and d) rheometers. Aqueous solution of MHPC are 2% (a and b) and 2.5% (c and d).

Table 2
Parameters for power law fluids and calibration coefficients

	2.0%	2.5%	Average
Shear rate conversion factor K, [s^{-1}/min^{-1}]	0.329	0.335	0.332
Shear stress conversion factor K/G, [Pa/(N mm)]	9.77	9.95	9.86
Viscosity conversion $1/G$, [Pa s^{-1}/(N mm min^{-1})]	29.7	29.7	29.7

Carrying out this procedure for each cylinder volume, the yield stress estimated from the spread of samples of different volume shows relatively good agreement over the whole range of spreads measured (Fig. 6).

4.4. Viskomat calibration

The Newtonian fluid showed a linear relation in both rheometers. Furthermore, it was found that the power law fluids exhibited similar exponents. This is a critical information that indicates that the torque is proportional to an average shear stress. On a viscous dissipation point of view, there exists a similitude between the local shear stress/strain rate relation obtained with the cone-plate rheometer and the global rotation frequency/torque measured with the viskomat.

With this observation, further data treatment was done using the exponent from the measurement in the rheometer with cone plate geometry because of its higher accuracy. The data from the Viskomat flow curve were then fitted to a power law function with the determined exponent (Fig. 7).

The coefficients are summarized in Table 2. It is clear that both fluids give very close results which may be judged as a confirmation of the relevance of this calibration procedure.

According to these calibration coefficients, (about 1/3 for shear rate/rotation frequency coefficient and about 10 for the shear stress/torque coefficient), the maximum average shear rate and shear stress in the Viskomat are respectively about 67 s^{-1} (200 rpm) and 20,000 Pa (200 N mm). It is important to emphasize that this is for the given anchor used in these experiments and that, locally, in particular at the surface of that anchor, values will be quite different. First of all, the real velocity field is three dimensional and cannot be described by a unique value. Moreover, according to the anchor geometry, we cannot be sure that steady state can exist in such a flow. Finally, the real velocity field may display higher shear rate zones or even unyielded zones. However, the similitude obtained between the cone-plates rheometer results and the viskomat with this anchor allows the use of this calibration procedure. This could not have been true.

4.5. Flow curves of cement paste

The flow curves of the cement pastes all exhibited a Bingham behavior as indicated in Fig. 8 Because of the risk of a plug flow at low rotation velocity, the two lower shear rate data points were systematically rejected for the regression calculation. Results are shown in Fig. 9 where they are compared with values inferred from the spread measurements.

5. Discussion

The first part of this paper is concerned with the comparison between two types of models used to determine yield stress from the spread of a paste on a flat horizontal surface. The two relations show very different scaling of the yield stress with sample volume and spread radius. The yield stress is a material property and should not depend on sample volume. Thus a given tested sample should spread in such a way that its final spread radius should cancel out the volume effect in the yield stress expression. A simple test of this requirement was done by measuring the spread of different sample volumes taken at the same time from the same batch.

Results confirmed that one of the expressions performs well at low spread radius and not at large spread while it is the opposite for the other expression. For convenience, a function interpolating between both zones well predicted by each model was proposed. It gives the yield stress as a decreasing exponential of the spread radius. This function seems to provide a good agreement over a wide range of spread radii for different sample volumes. Such a function is useful for industrial labs that widely use the spread method, but are

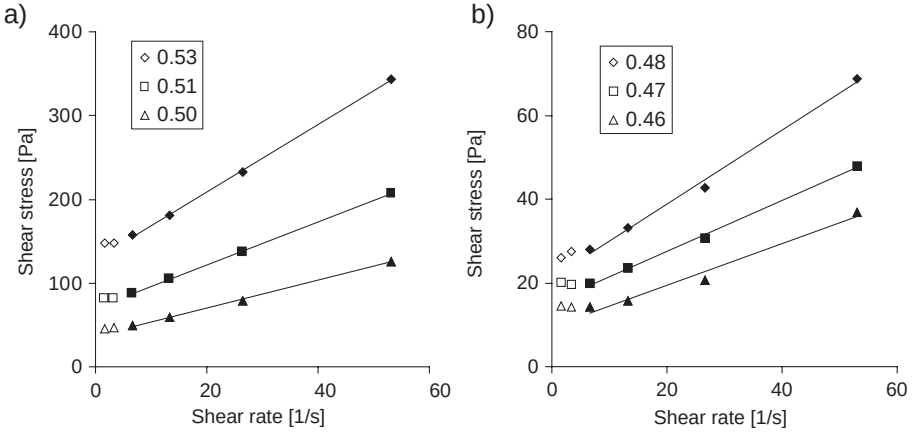

Fig. 8. Flow curves of cement paste with different volume fractions of cement obtained in Viskomat. Torque and rotation rate have been converted to shear stress and shear rate with factors in Table 2, numbers in the insert refer to the volume fraction of solid in the suspension.

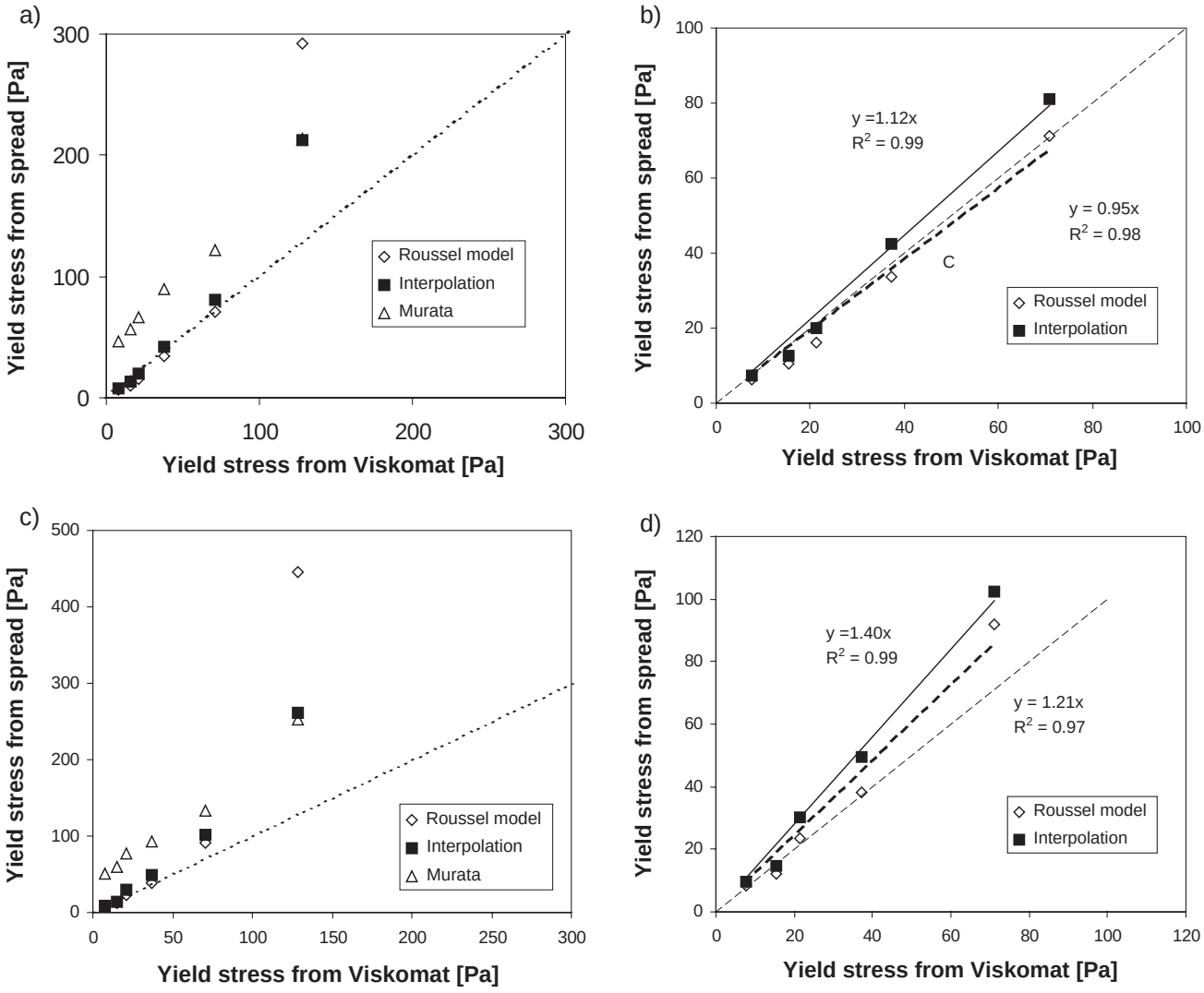

Fig. 9. Comparison of yield stress determined with calibrated Viskomat versus yield stress from various functions based on spread radius. a and c) all models over entire range of measured values for the first and second spread measurement respectively, b and d) Roussel model and interpolating function excluding highest yield stress value, for the first and second spread measurements, respectively. Dotted line is a 1 to one relation. In part b), the linear fit with zero ordinate at the origin are given.

lacking ways to translate results into meaningful rheological parameters.

The second part of the paper is concerned with another issue relevant to rheological investigation often performed in industry. It is a fact that the rheometer Viskomat is very widely spread among industrial labs, particularly in Europe. However, here again, quantification of the results in terms of basic rheological parameters is generally not achieved.

Comparisons among rheometers used to measure cementitious materials is not a trivial matter. At this stage literature results about the use of spread diameter to assess yield stress together with the above results led us to examine whether such results would compare advantageously with data produced by the Viskomat. To free this comparison from as many artifacts as possible, the sample for the spread was taken directly from the viscomat pot at the end of the flow curve measurement. Prior to this, an additional high shear regime was imposed of equivalent duration before the initiation of the flow curve measurement. The addition of a retarder also decreased risks of sample irreversible alteration between both measurements.

Two successive measurements of the spread diameter systematically gave a slightly larger spread for the first than for the second measurement. This indicates an on-going agglomeration or flocculation. Thus despite the precautions taken there is some evolution of the sample and a perfect correlation with the value obtained in the Viskomat cannot be expected.

Overall, results in Fig. 9 show that the corrected Murata model is clearly not adequate at low yield stress values. This was expected. On the other hand, apart for the highest yield stress sample (i.e., more than 100 Pa), the Roussel model (Eq. (3)) shows excellent quantitative agreement for the first series of experiments. The interpolating function also shows a linear relation with zero ordinate at the origin, but the slope departs from unity by about 12%. Which of the two is the most

appropriate is not easy to judge. Clearly the interpolation introduces some error, but the samples with higher yield stress are not well described by Eq. (3).On the other hand, the calibration procedure was done for non-Newtonian fluids with no yield stresses. It must be kept in mind that, although the flow pattern in the viskomat with this anchor seems to leave no zone at rest, the presence of unyielded zones in the measuring pot is always a possible cause of discrepancy at the highest yield stresses. Furthermore as mentioned above, agglomeration is on-going in the system. The second series of measurements, which was conducted about 5 min after the first taken additional material from the Viskomat measuring pot, shows a change of about 30%. Both curves show a high coefficient of correlation, so that it is difficult to reject one rather than the other. Overall, it is clear that the original model will be better in the low yield stress range, while the interpolation function should allow to reach higher yield stress values. This is apparent in Fig. 9.

Overall, it appears that the calibrated Viskomat gives a yield stress that agrees well with the one that can be inferred from a sample having the same shear history. Having said this, it must be emphasized that this article does not deal with the validation of the plastic viscosity obtained by the Viskomat. This is expected to be a more delicate topic owing to segregation in high shear zones inside the rheometer, which might lead to incoherent results.

The yield stress evolution observed over a short period of time clearly states the difficulty in comparing results from different measurements. It appears that thixotropy and/or aging of the paste complicate comparisons among rheometers. The correlations here are very good overall, but they were obtained by taking a lot of care to make such a comparison as pertinent as possible. Such correlations would probably be lost if this type of care were not taken.

6. Conclusion

Two rheological tools widely used in the cement related industry have been examined: spread experiments and Viskomat measurements. The selection of an adequate expression to relate spread to yield stress on one hand and a careful calibration procedure of the rheometer on the other lead to an agreement which is basically quantitative among both measurements.

This constitutes useful practical information for interpreting existing data acquired by such methods in rheological terms. The calibration parameters presented are given as illustration. They must not be used as such. They would be totally irrelevant with another measuring geometry.

Finally, the close link between both types of yield stress determination methods enhances the attractiveness of the spread measurement to collect rapid and now quantitative data on yield stress. Cross correlation with other means of determining plastic viscosity was not undertaken in this paper. This may be left to other enthusiastic industrial scientists who wish to understand the meaning of the measurements their colleagues perform on a daily basis.

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Appendix A. Calibration of viscometers

The calibration procedure is described by Tattersal and Banfill [14].

It involves the measurement of the flow curve of liquids in the rheometer to be calibrated and in a rheometer giving absolute value, in this case a Cone plate rheometer. These rheometers will be denoted as VK and CP, respectively.

The basic assumption is that the average shear rate and the average shear stress in VK are proportional respectively to Torque, T, and rotation frequency, N. We write:

$$\tau = \frac{K}{G} T \tag{5}$$

$$\dot{\gamma} = K\ N \tag{6}$$

where K and G are proportionality constants.

In a first step of the calibration a Newtonian fluid is measured in both devices. We write its viscosity as

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{7}$$

$$h = \frac{T}{N}. \tag{8}$$

From the above equations we get the first proportionality constant G from:

$$\frac{h}{\eta} = G. \tag{9}$$

From Newtonian fluids, it is only possible to get G. To get K, one uses fluids that exhibit a power law dependence between shear stress and shear rate. Solutions of polymers of high molecular weight typically exhibit such behavior. In those case one writes:

$$\tau = r\dot{\gamma}^s \tag{10}$$

$$T = pN^q. \tag{11}$$

Provided values of s and q are similar, we can write:

$$T = pN^s. \tag{12}$$

The ratio between Eqs. (10) and (12) gives:

$$\frac{\tau}{T} = \frac{r}{p}\left(\frac{\dot{\gamma}}{N}\right)^s. \tag{13}$$

Using Eqs. (5) and (6) this becomes:

$$\frac{K}{G} = \frac{r}{p}\mathrm{K}^{s} \tag{14}$$

which leads to the following expression of K:

$$K = \left(\frac{p}{r\ G}\right)^{\frac{1}{s-1}}. \tag{15}$$

For the above equation to hold work the exponents must be the same and the samples must be measured over an equivalent range of shear rates.

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