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Parallel-plate Rheometer Calibration Using Oil and Computer Simulation

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Abstract

Fluid flow patterns in traditional rotational rheometers are generally well known and rheological parameters such as viscosity can be easily calculated from experimental data of single phase fluids and analytical solutions of the patterns. However, when the fluid is a suspension, where some of the particles are as large as 2 mm in diameter, these rheometers need to be modified. The distance between the shearing planes needs to be increased, which necessitates additional physical confinement of the fluid. This causes the flow pattern to be not analytically soluble leading to an inability to correctly compute the viscosity. This paper presents a modified parallel plate rheometer, and proposes means of calibration using standard oils and numerical simulation of the flow. A lattice Boltzmann method was used to simulate the flow in the modified rheometer, thus using an accurate numerical solution in place of the intractable analytical solution. The simulations reproduced experimental results by taking into account the actual rheometer geometry. The numerical simulations showed that small changes in the rheometer design can have a significant impact on how the rheological data should be extracted from the experimental results.

1. Introduction

Traditional rheometers designed for polymers and oil are not always applicable without modifications for suspensions containing relatively large particles. If the suspension contains particles larger then 1 mm or 2 mm, the usual rotational rheometer geometries cannot be used, since the gap between the shearing surfaces is required to be several times the maximum particle size, so as to treat the suspension as a continuum. At such a gap size in a parallel plate rheometer, for example, surface tension cannot hold the material in between the parallel plates. Also, particles should not settle in the time frame of the measurements due to the viscosity of the suspending medium; settling may be restricted due to a reasonable density agreement between the particles and the medium; and a high concentration of particles (up to 56 % solids by volume).

Several researchers have measured the rheology of such suspensions in small laboratory size rheometers, e.g. (Yang *et al*. 1995, Nachbaur *et al*. 2001, Struble *et al*. 1997, Banfill 1990, Ferraris *et al*. 1992, Ferraris 1999). But with the exception of rheometers used for suspensions with particles in the micrometer range the geometry of the rheometer does not allow for an analytical solution of the flow patterns. Therefore, in order to calculate the viscosity, some assumptions or simplifications are necessary. This led, for instance, to discrepancies between the absolute value of viscosities obtained with various rheometers by nominally identical materials (Ferraris and Brower ed. 2001 and 2004).

An example of a suspension would be cement paste (50 um particles), mortar (2 mm particles) and concrete (20 mm particles). For measurements of cement paste and mortar, Ferraris and Gaidis (1992) argued that a parallel plate geometry offers the most flexibility. This geometry is the only rotational rheometer configuration that allows for continuous variation of the distance between the two shear planes, which facilitates the measurement of the cement paste in the same shearing conditions that occur in concrete. In concrete, the shearing surfaces for cement paste are the sides of aggregates, either sand or coarse aggregates. The lower the cement paste content in a mortar or concrete, the smaller the distance between the aggregates, and thus the smaller the gap in the rheometer should be when measuring the cement paste alone. The ability to adjust the gap is paramount if the rheological measurements done on cement paste or mortar are used to predict or to make a comparison with measurements obtained on mortar or concrete, respectively.

The distance or gap between the shearing plates needs to be at least five times the particle size to allow for a continuous fluid approximation (Ferraris *et al*., 2001b). Thus, a parallel plate rheometer, modified to accommodate the measurements of mortars or any suspension with particle sizes up to 2 mm in diameter, requires gap from 10 mm to 15 mm. As the material cannot be contained between the two plates by capillary forces at gaps larger than about 1 mm, some kind of "retaining wall," such as a cylindrical wall around the plates, is needed to hold the material in place during the measurement. This

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cylindrical wall unfortunately invalidates the known analytical solution in the parallel plate geometry, since the no-slip boundary condition and the gap between the outer part of the plate and the cylindrical wall does not allow for an analytical solution of the flow patterns.

A powerful tool for understanding and interpreting fluid flow in a rheometer can be through computer simulation. Computer model also permits a systematic variation of flow conditions in order to study the effect of geometry, shear rate and boundary conditions on measurements (e.g. Hsieh *et al*. 1997). Indeed, numerical solution of the Navier-Stokes equations should allow for extraction of the actual viscosity given experimental input. In an earlier study, Hu *et al*. (1995) simulated the flow of concrete in a rheometer of such geometry using finite elements and determined from the simulation that pure slippage occurs at the confinement walls and that therefore they can be ignored. This simulation needs to be validated by more detailed computation and experimentation.

This paper is a first attempt to analyze the response of modified parallel plate rheometers. Experimental measurements and fluid computations have been combined to identify the important geometrical factors. Using a standard oil having known viscosity, the experimental measurements are compared against obtained from computer simulation. The oil viscosity should be independent of the rheometer configuration, which was used to determine a calibration function related to the geometry of the modified rheometer. This paper represents the first part of a full calibration of modified parallel plate rheometers. A material containing particles having a wide range of sizes should also be tested. Ideally this suspension should be of known viscosity, so it would serve as a reference material. For now, such a reference suspension does not exist.

2. Experimental set up

2.1 Materials

Two oils were used to determine the influence of the rheometer geometry on the viscosity measured:

- Cannon RT 1000 (silicone 100 %); nominal viscosity = 1 Pa s at 20 °C
- Cannon S8000 (poly(1-butene) 100 %); nominal viscosity = 34 Pa s at 20 °C

The oils selected had a viscosity, closely approximating the viscosities of mortar or cement paste. These oils are sold for calibration of rheometers. They were calibrated using a Master Viscometer technique (Swindells *et al*. 1954).

2.2 Equipment

A parallel plate rheometer was modified by redesigning the plates to reduce slippage. A conventional configuration of a parallel plate rheometer with a gap below 1.5 mm could be used to measure suspensions of particles less than or equal to 50 μm. The texture of the shearing planes needs to be carefully selected when measuring suspensions to avoid slippage. If slippage occurs, lower torques would be measured and an incorrect calculation of viscosity and yield stress would result. The design of the serration used here is shown in **Fig. 1**. The serration of the plates was made in a pattern of size 1 mm by 1 mm with a depth of 0.381 mm. This pattern is essentially a grid of truncated pyramids. For a suspension with particle diameters of the order of 2 mm, the serrated plates shown in **Fig. 1** are not enough to avoid slippage. Therefore a different top plate was designed (**Fig. 2**). The pattern was inspired from the BTRHEOM, the only parallel plate concrete rheometer (Hu 1995, de Larrard *et al*. 1996). Four configurations of plates were used: 1) PP35-S: 35 mm diameter serrated plates; 2) PP35-P: 35 mm diameter smooth plates; 3) PP60: 60 mm diameter plates with the bottom plate serrated as shown in **Fig. 1** and the top plate as shown in **Fig. 2**; and **4**) PP60/W: 60 mm diameter plates with the bottom and top plate as shown in **Fig. 2**.

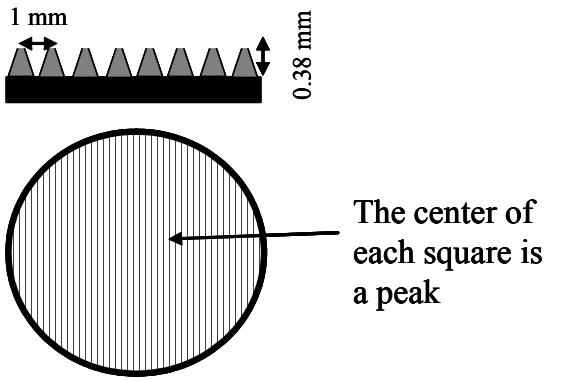

Fig. 1 Schematic of the serration on the plate. Peak –to-peak distance is 1 mm, and depth is 0.38 mm. The bottom schema shows the grid layout of the peaks. Note that for the PP35 configuration both top and bottom plate have this pattern. For the PP60 configuration, only the bottom plate is serrated. (Upper Figure not to scale)

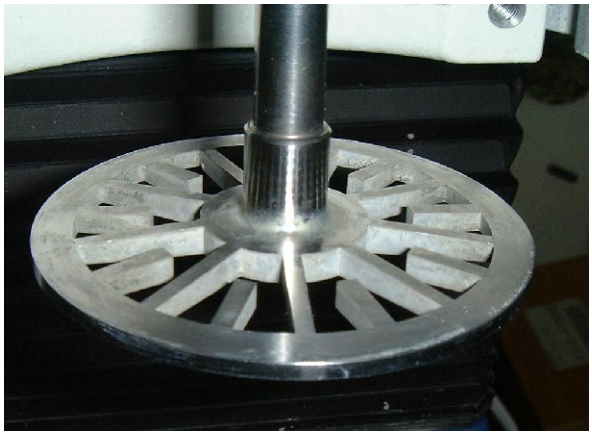

Fig. 2 Top plate used for PP60. This wheel is designed for suspensions with large particles (up to 2 mm in diameter).

With a gap between the plates larger than 1.5 mm, the material being measured will not be held between the plates by capillary forces alone - a confinement wall is necessary. The diameter of the confinement wall used for a particulate suspension needs to be chosen so that the suspended particles do not wedge themselves between the confinement wall and the plate. Such wedging prevents the top plate from freely rotating, resulting in high torques that do not reflect the response of the bulk material. In this paper, as all measurements are done with oil, the confinements were varied to determine the influence of their diameter on the viscosity. **Fig. 3** shows one of the confinement rings. The internal diameter of the confinement rings used in this study were 37.3 mm (suffix-C), 62.2 mm (suffix -M), 64.0 mm (suffix -M2), 67.9 mm (suffix -M4) or 76.7 mm (suffix -M8) (**Table 1**). The uncertainty of the measurement of the diameter of the confinement was ± 0.2 mm. The PP35-C confinement ring was used with the 35 mm diameter plates only, while the other confinement rings were used with the 60 mm diameter plates (**Table 1**).

Fig. 3 Confinement geometry for PP60. A smaller inner diameter was used for the PP35 set-up.

The mode of operation of the rheometer was designed to shear the material at increasing shear rates from 1 s^{-1} to 10 s^{-1} followed by a decrease of the shear rates from 10 s^{-1} to 1 s^{-1}. Ten measurements were made during both increasing and decreasing shear rate regimes. The torque measurement at a given shear rate was taken when steady state was achieved, i.e. variation of less than 0.05 % in the torque, or when a time of 20 s was elapsed (even if steady state had not been reached, to avoid a test that last too long). This time might be too short for steady state in some cases, but a longer time would length the total measurement time. This extra time is not critical for oils but it is very important for suspensions that are time sensitive, such as cement paste. In both cases, the value reported was the average of five measurements of torque taken over 5 seconds when steady state was achieved or 20 s were elapsed. The plastic viscosity is calculated as the slope of the shear rate vs. shear stress curve on the decreasing shear rate branch. The rheometer was temperature controlled, with an accuracy of ± 0.2 °C, by a re-circulating water bath connected to the base below the bottom plate of the rheometer.

2.3 Experimental design

The geometrical parameters to be evaluated were: diameter of plate, type of confinement ring and surface structure of plate.

To make sure that the influence of all parameters was considered, it was necessary to calculate calibration function values with the various geometries, so that a full factorial experimental design was used. **Table 1** shows the series of tests done with the oil S8000.

In each case, the oil was allowed to sit in the rheometer, with the top plate raised away from the material, long enough to make sure that the oil was free of air bubbles caused by pouring the material into the rheometer plate and in equilibrium with the temperature selected. All experiments were performed at 23.1 °C ± 0.2 °C. The temperature should also be checked at the top plate especially in larger gaps or at temperature very different from the ambient.

Table 1 Experimental design.

Name of set-up (figures below)	**Plate diameter [mm]**	**Plate Type**	**Confinement present** ID: Inner diameter
PP35-P	35	Plane	No
PP35-P-C	35	Plane	Yes, ID 37.3 mm
PP35-S	35	Serrated	No
PP35-S-C	35	Serrated	Yes, ID 37.3 mm
PP60-M	60	Serrated bottom and special design top	Yes, ID 62.2 mm
PP60-M2	60	Serrated bottom and special design top	Yes, ID 64.0 mm
PP60-M4	60	Serrated bottom and special design top	Yes, ID 67.9 mm
PP60-M8	60	Serrated bottom and special design top	Yes, ID 76.7 mm
PP60	60	Serrated bottom and special design top	No
PP60-M4/W	60	special design top and bottom	Yes, ID 67.9 mm

3. Computational technique

Because the presence of a confining wall makes and analytical solution of the fluid flow intractable, a computer model was used to define a numerical solution. This simulation was used only for the case of a confinement; it was not able to determine the influence of serration and other parameters. The simulation method used was based on the lattice Boltzmann method (LB). While a detailed explanation of LB is beyond the scope of this paper, let it suffice to say that the LB method is a computationally efficient approach that recovers solutions of the Navier-Stokes equations accurate to second order in velocity gradients. Details of the simulation method relevant to this paper are given elsewhere (Martys and Hagedorn 2002).

The geometry of the rheometer was reproduced in a 3D digital image, with a voxel resolution of 0.125 mm. To simulate shearing the fluid, a local force, $\vec{F}(\vec{r})$, where $\vec{r}$ is a radial vector in the plane of the top rotating plate, was applied on the fluid adjacent to the top plate. This force was adjusted such that the fluid steady state velocity was the same as the rotating plate. The final total torque applied to the fluid is given by $\Sigma \vec{r} \times F$, where the sum is over all the fluid pixels at which the shear force was applied. This approach parallels the experimental method where the fluid was sheared at a controlled rate while the torque was measured. Both the calculated and experimentally measured torque depend on the fluid viscosity, shear rate and rheometer geometry, e.g., gap between plates or confinement, as well as the boundary conditions (slip or no-slip on the walls). The resulting torque may be written as shown in equation 1:

$$T = \eta \cdot f(Geometry, Boundary\_conditions) \cdot \dot{\gamma} \quad (1)$$

where: T = torque
η = viscosity
$\dot{\gamma}$ = shear rate
f = function that takes into account geometrical factors and boundary conditions

Equation 1 is basically the definition of the viscosity with an added parameter with the function f and the viscosity, η, would be constant at all shear rates if the fluid is Newtonian. In both the experiment and the simulation, the product $\eta \cdot f$ (Geometry, Boundary conditions) is calculated. In either case, the function f cannot be calculated unless the viscosity is known. The advantage of simulations over experimental investigations is the ability to easily change the geometry and boundary conditions without cumbersome rebuilding of the rheometer. This allows us to readily determine the geometrical factors that have the most influence on the results.

The configuration of the model rheometers used in the simulations is shown in **Fig. 4**. The model rheometers were designed to closely match the experimental set up. To better understand the rheometer's sensitivity to design parameters, the following three cases are considered, where the boundary conditions on selected parts of the rheometer were varied. In all cases, it was assumed a no-slip boundary condition on the bottom plate and confinement walls and that the bottom plate is fully immersed in the fluid.

- Case 1: The space between the outer edge of the top plate and the confinement wall was approximately 1.0 mm. A slip boundary condition was applied on the rim of the top plate, as if the top plate were not immersed in the fluid. (This case does not reflect the experimental set up where the top plate was partially immersed in the fluid to a depth of 1 mm, i.e.

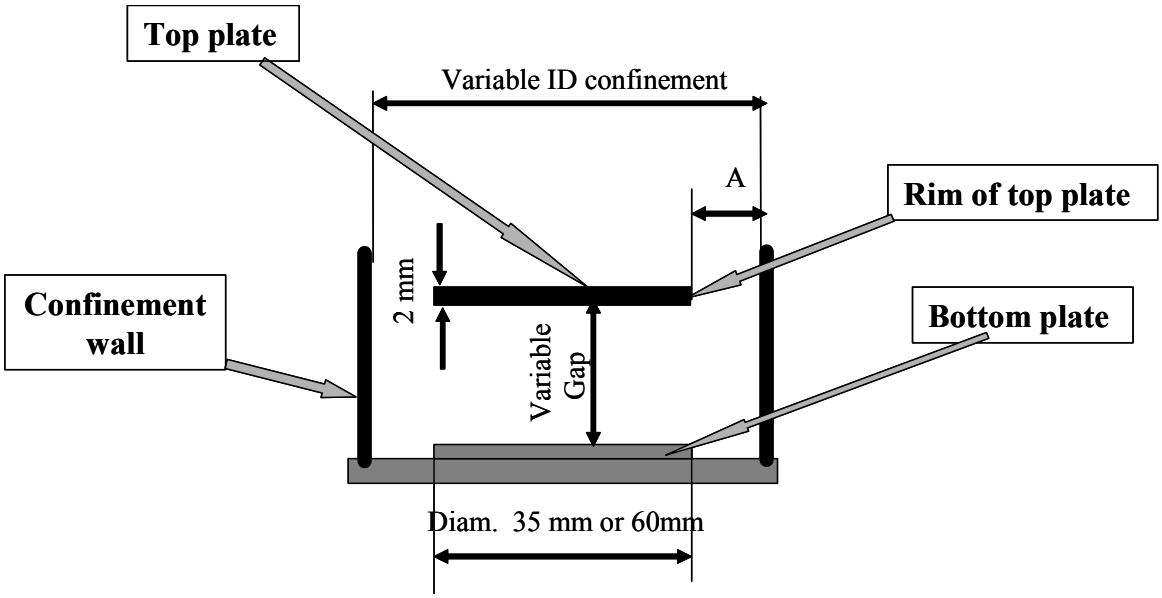

Fig. 4 Schematic of the rheometer geometry used in the numerical simulation (not to scale). The value A depends on the diameter of the confinement as the diameter of the plates do not change with the confinement.

the lower surface was covered, but no fluid flowed trough the opening to rest on top of the top plate.)

- Case 2: In this case, there was a narrower gap between the rim of the top plate and the confinement wall (of order 0.125 mm) than in the experiment (about 1.0 mm). A no-slip boundary condition was applied between the outside rim of the top plate and the confinement wall. The top plate was partially immersed in the fluid to a depth of about 1.0 mm.
- Case 3: This case most closely modeled the geometry of the rheometer PP35-C. The space between the rotating plate and the confinement wall was 1.0 mm. A no-slip boundary condition between the outside rim of the top plate and the confinement wall was considered. The top plate was partially immersed in the fluid to a depth of about 1.0 mm as in case 2.

Table 2 summarizes how the design parameters were varied for the three cases for which the numerical simulation was applied.

4. Results and discussion

The value of the gap was used to calculate the shear rate (in s^{-1}) from the measured speed as shown in equation 2. Uncertainty in the measurement of the gap distance will propagate in the shear rate calculation, affecting the calculation of the viscosity.

$$\dot{\gamma} = \frac{R}{h} \cdot 2\pi \cdot n \tag{2}$$

where: $\dot{\gamma}$ = shear rate [1/s]
R = radius of shear [mm]
h = gap or distance between the plates [mm]
n = speed of rotation of the top plate, revolution/s [1/s]

The viscosity was calculated as the slope of the shear rate-torque curve, using the equation below:

$$\eta = \frac{2 \cdot \Gamma}{\pi \cdot R^3} \tag{3}$$

where: η = viscosity [Pa·s]
Γ = slope of shear rate – torque curve [N·m·s]
R = radius of the shearing area [m], i.e., the plates or the confinement.

Table 2 Parameters used in the simulation (See Fig. 4). The PP35 plates were used.

	Case 1	Case 2	Case 3 (PP35-C)
Slip at the outer edge of top plate	Yes	No	No
Space between the top plate and the confinement [mm]	1.0	0.125	1.0
Thickness of the top plate [mm]	<<1.0	1.0	1.0

Equation 3 is the analytical solution for the case where there is no confinement ring and smooth plates. **Fig. 5** shows the experimental results obtained for the oil S8000 using a combination of serrated plates and confinement rings (**Table 1**). The line on **Fig. 5** indicates the nominal value of the oil viscosity at the temperature of the experiments. The viscosity value obtained using any of the configurations of **Table 1** should be identical to the reported value in the certificate of the standard oil used at the same temperature, if the geometry of the rheometer had no effect. This is, however, clearly not the case. The analytical solution for the case with no confinement wall is of no use for these different geometries.

Fig. 6 shows a normalized viscosity for PP35 (no confining ring) and two types of plate surfaces, plane (P) and serrated (S). The normalization of the viscosity was achieved by dividing the value measured (using equation 3) of the oil with the nominal value of the oil (as given by the certificate of the standard oil) at the same temperature. For a pure parallel plate rheometer, the value of the gap does not influence the calculated value of the viscosity; therefore, the normalized viscosity should be equal to 1 for all gaps. In other words the function *f* of eq. [1] should be 1 for all gap values. This is clearly not the case here especially for the serrated plate surface (**Fig. 6**). Therefore, calibration functions need to be determined not only for the confined geometries but also the non-confined plate serrated geometries.

4.1 Influence of zeroing and serration

The first rheometer configuration to consider is the unconfined geometry. The values measured with the plane plate surface (PP35-P) are not exactly equal to 1 (**Fig. 6**) and the discrepancy is noticeable for the small gap but not for the large (≥ 1 mm) gaps. This phenomenon was also observed by other researchers [Davies and Stokes (2005), Sanchez-Perez *et al*. (2003)]. Davies and Stokes attributed the discrepancy to an error in the zeroing of the plates. They stated that when the two plates are

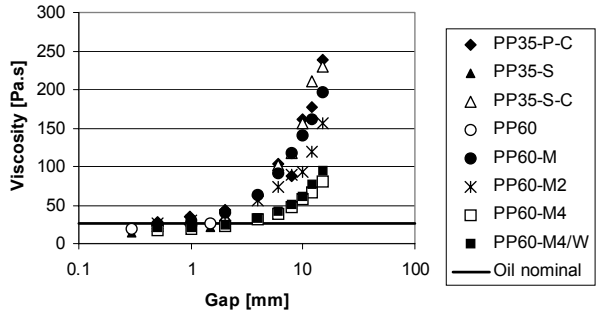

Fig. 5 Viscosity obtained as a function of the gap between the plates for oil S8000. The line shows the nominal value of the oil at the temperature of the experiments. The error bars are not shown here for the sake of clarity.

brought together some entrapped air prevents the plates from completely touching. This will result in the gap used for the measurement being actually larger than the true gap. They developed a method to estimate the error. Using this method and fitting our data for the small gaps of 0.3 mm and 0.5 mm the gap should in our case be corrected by 0.022 mm or 22 μm. This value seems a little high compared with the value obtained by Davies and Stokes of 7 μm. Davies and Stokes suggest verifying this calculation by using spacers. This was done on the rheometer used for this paper. The PP35-P was used and once the automatic zero was reached by the machine, spacers were slid between the plates. It was found that a spacer of 12.5 μm could be slide easily between the plates, while a spacer of 25 μm could not fit. This implies that the error could be as calculated (22 μm).

Sanchez-Perez *et al*. (2003) assumed that for all configurations there was always a slippage at the interface between the plates and the material. They derived an equation to calculate the thickness, b, of that layer and use it to correct the effective gap to be used to calculate the viscosity of the material.

$$b = \frac{(\dot{\gamma}_{g1} - \dot{\gamma}_{g2}) g_1 g_2}{2(g_1 \dot{\gamma}_{g1} - g_2 \dot{\gamma}_{g2})} \tag{4}$$

where g_1 and g_2 are the two gaps in [m] and $\dot{\gamma}_{g1}$, $\dot{\gamma}_{g2}$ are the shear rates measured for the two gaps in s^{-1}

Thus, b can be calculated for any two gaps and any two rotational speeds. When applied to our case of smooth PP35, a value of 11 μm was obtained.

Assuming an error in the zeroing leads to a correction value of 22 μm, while assuming slippage leads to a correction value of only 11 μm. The data obtained and showed in **Fig. 6** will allow the determination of the predominant corrections that should be adopted. **Fig. 6** shows the normalized viscosities as measured using either PP35-P or PP35-S and the same data with the various corrections factors included. A normalized viscosity of 1 implies that the nominal standard viscosity of the oil was measured. There are two figures to allow a better visualization of the correction influence. In **Fig. 6A** shows PP35-S values and the correction (see discussion below on how it is obtained) and the PP35-P with the correction as given by the zeroing correction. **Fig. 6B** shows the data obtained with PP35-P and the two possible corrections, zeroing or slippage. From this figure, it seems that the best correction is for the zeroing correction. The error on computed viscosity could, however, be due to a combination of zeroing and slippage as even the best correction still gives a value below 1 (**Fig. 6B**). It could also be argued that even the smooth surface would appear serrated for the oil molecules and therefore no slippage occurs. Nevertheless, we have no way to verify this hypothesis.

The viscosities measured using the serrated plates are always significantly lower than the values obtained with the smooth/plane plates (**Fig. 6A**). The discrepancy observed is attributed to the gap on the serrated plate not being correctly determined as seen in **Fig. 7**. The two possible uncertainties in the gap measurements are: 1) the zero gap reference; 2) the oil volume that is contributing to the measurements. This random phenomenon is difficult to quantify. The computer controlling the rheometer determines the gap for the measurement by first zeroing the distance between the two serrated plates. This is achieved by raising the bottom plate until it touches the top plate. In the case of the serrated plates (both top and bottom serrated), it is possible that the zero is not always at a peak-to-peak separation of the serration, but a peak to valley position. The height of the serration was measured to be 0.38 mm. In the calculation of the viscosity the gap is assumed to be peak-to-peak and also that all the oil is contained in that gap. One other consideration would be that bubbles could be trapped in the serration and therefore changing the torque measured.

In **Fig. 7A**, the results for PP35-S are shown, with the effective gap assumed to be measured peak-to-peak. **Fig. 7B** shows the same data when the gap is assumed to be larger than the peak-to-peak distance by a fitted value of 0.27 mm. In **Fig. 6A**, the normalized viscosity (ratio of the measured value and the nominal value) was reported as a function of the gap. It is clearly seen that the non-

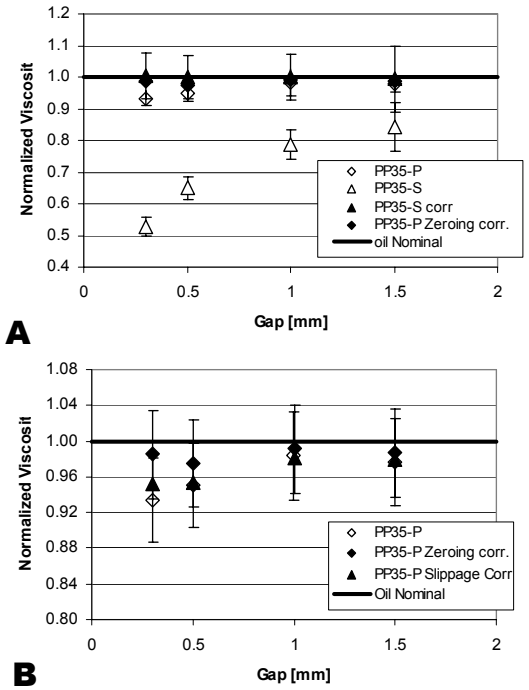

Fig. 6 Normalized viscosity for oil S8000 as a function of the gap between the plates PP35-P and PP35-S, i.e., smooth (P) and serrated (S) plates. The error bars represent one standard deviation. A) all the data are shown; B) shows only the data for PP35-P (scale for the normalized viscosity is narrower). For description of the correction (zeroing and slippage) refer to the text.

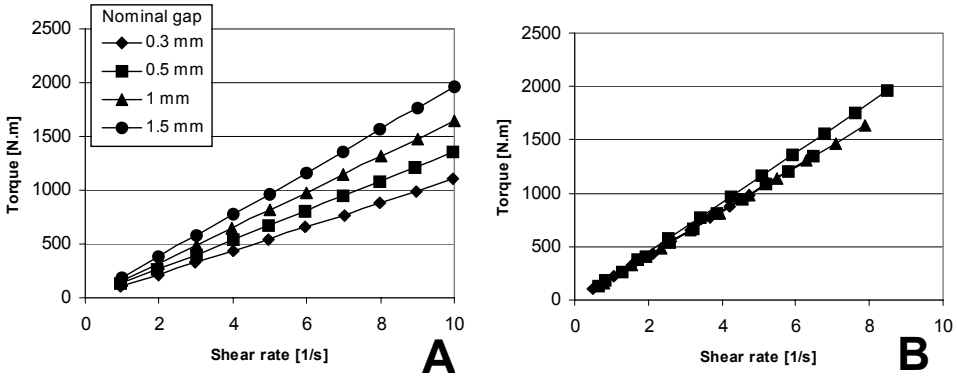

Fig. 7 Torque versus shear rate for oil S8000 for varying gap size, 35 mm diameter, serrated plates and un-confined geometry. A) Measured values, B) gap values corrected by the value of 0.27 mm.

corrected normalized viscosities ("PP35-S" in **Fig. 6A**) are increasing with the gap, while the corrected values ("PP35-S corr" in **Fig. 6A**, gap corrected by + 0.27 mm) are equal to 1. Therefore, the effect of the serration can be accounted for by assuming a larger effective gap size. This value was determined by trial and error and corresponds to about a 2/3 of the serration height. If the calculation of b, as described above, is applied to serrated configuration, a value of 0.26 mm ± 0.006 mm is obtained by using data obtained with the gaps of 0.3 mm and 1.0 mm. The two values experimental and calculated are in very good agreement. Thus, at this stage, it is not possible to determine whether the discrepancies between assumed and true gaps are due to incorrect zeroing or slippage. Therefore, both corrections need to be used in conjunction.

4.2 Influence of the confinement wall

In the case of measurements done with a confinement wall, the viscosities measured are larger than when no confinement was present. Several factors could contribute to this discrepancy, such as friction with the walls, shearing of oil between the rotating top plate and the confinement, and others which the authors are not aware at this time. Therefore, experimental results were combined with simulation to determine the most important geometrical parameters that affect the measurement of the viscosity. Equation 1 is also applicable to these experimental data.

Fig. 8 compares the simulation data with experimental data in the case of the PP35 confined geometries. The agreement is excellent, when the geometry used in the experiment (Case 3) was reproduced accurately. The two critical geometrical parameters are the space between the outer-edge of the top plate and the inner surface of the confinement ring, and the top plate thickness (**Fig. 4** or **Table 2** Case 3). If either of these two geometrical factors are smaller than the experimental values (**Table 2**: case 1 and 2), the agreement between the experimental and simulation data is poor (**Fig. 4**: Cases 1 and 2). In Case 2, the virtual rheometer was modified by decreasing the distance by a factor of 8 between the top plate and the confinement. In this case, it was found a normalized viscosity significantly higher (**Fig. 8**) than that found experimentally. This can be understood as resulting from the higher shear stress due to forcing the fluid velocity to go from the equilibrium velocity of the rotating top plate outer-rim to zero velocity along the confinement wall over a much shorter distance then in Case 3. Obviously, this requires a much higher torque. In Case 1, the original rheometer design was used but allowed for slip on the outside cylindrical part of the rotating top plate. Here the applied torque needed was smaller because the rotating plate was not required to shear the fluid between the top plate and the confinement. As a result, the normalized viscosity appears to be smaller.

The lattice Boltzmann simulation helps us determine the important geometrical features but at this point the

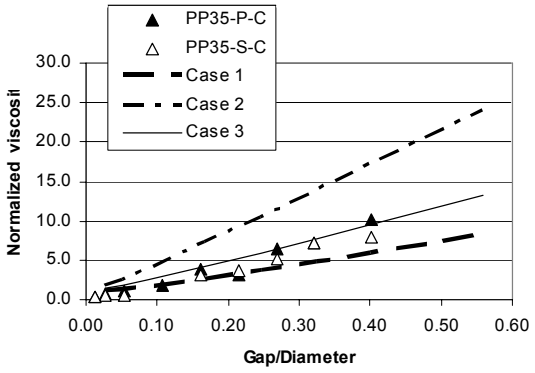

Fig. 8 Simulation and data for the PP35 confined geometry. The three solid lines are simulations with some variation of the geometry shown in Fig. 5, and the symbols represent the experimental data with the gap corrected accordingly (see Fig. 6 and text). Table 2 gives the description of each case.

real viscosity need to be calculated from our measurements by estimating the function *f* from equation 1. From the simulation plot in **Fig. 8** and the data in **Fig. 9**, it was observed a linear relationship between the viscosity measured and the ratio of the gap (distance of the shearing plates) to the diameter of the confinement This linear relationship can be used to calculate a calibration function that will allow the calculation of the true viscosity of the fluid (equation 5). The values calculated for the geometry tested are shown in **Table 3**. A linear relationship could be calculated between confinement diameter and the function *f* for confinement diameters smaller than 68 mm (or M4) as used for PP60; the correlation is 0.998. But for confinements larger than 68 mm it seems that the linear correlation between the function *f* and the diameter of the confinement is not maintained. The calibration function *f* seems not to significantly change once the confinement becomes large enough. This could be interpreted that the influence of the confinement decreases with the diameter of the confinement. Therefore, one possibility to reduce the influence of the confinement is to use a very large diameter and then the calibration function *f* is constant.

$$\eta_r = \frac{\eta_m}{\frac{h}{D} \cdot f + 1} \tag{5}$$

where: η_r = corrected or real viscosity
η_m = measured viscosity
f = value of the calibration function
h = gap
D = diameter of the confinement

5. Conclusions

This paper examined the measurements of viscosity using a modified parallel plate rheometer. The modification involved serrating the plates to allow for suspensions to be tested and adding a confinement wall so that gaps larger than 1.5 mm could be used. This modification was necessary to allow the measurement of viscosities for suspensions where some of the particles are as large as 2 mm in diameter. The main application of this instrument will be for mortar made with portland cement and sand.

The use of a parallel plate geometry instead of coaxial cylinder or others for measuring rheological properties of suspensions offers flexibility as this is the only geometry available, in which the gap between the shearing plates can be easily varied. Nevertheless, modifications of the rheometer for use with suspensions need to be done carefully to avoid systematic errors. The following steps should be taken to ensure proper calibration:

- With smooth plates the zero (or contact point) needs to be assessed carefully using either spacers or Davies and Stokes (2005) method.
- The influence of serration should also be determined to ensure that the gap used for the calculation is correct; calibration using a standard oil is recommended
- A correction factor needs to be established for taking into account the confinement.

Numerical simulations reproduced the experimental results by taking into account the rheometer geometry. These simulations showed that small changes in the rheometer design can have a significant impact on the rheological results. By combining experimental and numerical results, a methodology to correct the measurement using an offset value for the gap and a calibration function related to the confinement was successfully developed. The necessary correction is likely to depend on the size of particles in suspensions.

Clearly, great care must be taken when interpreting experimental results on rheology of fresh cementitious materials and also when comparing results derived from different rheometers (Ferraris *et al*. 2003). In this paper all the experimental work and the simulation was performed using an oil and not a suspension. Therefore, further modifications of the rheometer and of the calibration presented here might be needed to correctly

Table 3 Calibration function for the confined geometries examined. Note not all tests were repeated enough times to allow the calculation of a standard deviation of the function *f*. This was not judged necessary as the standard deviation seemed similar for all diameters.

Geometry	Values of the Calibration function *f*
PP35-S-C	19 ± 1
PP35-P-C	24 ± 1
PP60-M	30 ± 1
PP60-M2	23
PP60-M4	10
PP60-M8	8 ± 1
PP60-M4/W	8

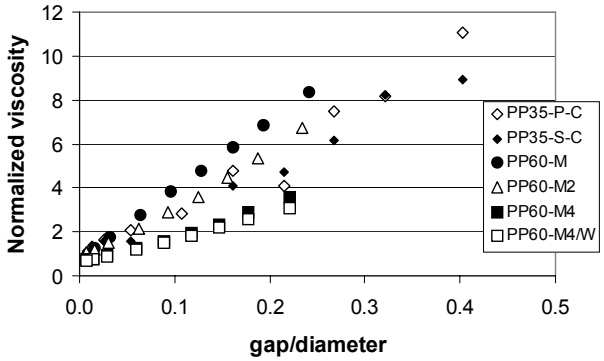

Fig. 9 Normalized viscosity for oil S8000, confined geometry, versus the ratio of confinement diameter and gap. The best fit lines were omitted for the sake of clarity.

measure the viscosity of a suspension. NIST is planning to investigate spherical and crushed aggregates in cement paste and oil, as well as monodisperse spheres using this experimental and numerical setup (Martys *et al*. 2007).

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