CONFERENCE: SCC 2007 GHENT

CALIBRATION OF MODIFIED PARALLEL-PLATE RHEOMETER USING STANDARD OIL AND LATTICE BOLTZMANN SIMULATION

Chiara F. Ferraris (1), Mette Geiker (2), Nicos S. Martys(1) and Nicholas Muzzatti(1)

(1) National Institute of Standard and Technology (NIST), Gaithersburg MD 20899, USA

(2) Department of Civil Engineering, Technical University of Denmark, DK_2800 Kgs. Lyngby, Denmark

Keywords: Rheology, rheometer calibration, mortar rheometer

Proc. of the 5th International RILEM Symposium on Self-Compacting Concrete, Ghent, Belgium, Sept 2007, pp. 297-302

CALIBRATION OF MODIFIED PARALLEL-PLATE RHEOMETER USING STANDARD OIL AND LATTICE BOLTZMANN SIMULATION

Chiara F. Ferraris (1), Mette Geiker (2), Nicos S. Martys (1), Nicholas Muzzatti (1)

(1) National Institute of Standard and Technology (NIST), Gaithersburg MD 20899, USA

(2) Depart. Civil Eng., Tech. Univ. of Denmark, DK_2800 Kgs. Lyngby, Denmark

Abstract

Fluid flow patterns in traditional rotational rheometers are generally well known so that rheological parameters such as viscosity can be easily calculated from experimental data of single phase fluids and analytical solutions of the flow patterns. However, when the fluid is a suspension, where some of the particles are as large as 2 mm in diameter, these rheometers may need to be modified. The distance between the shearing planes needs to be increased, which necessitates additional physical confinement of the fluid. The inclusion of a confining wall makes current analytical solutions inapplicable here. This paper presents the analysis of a modified parallel plate rheometer for measuring cement mortar and propose a methodology for 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

Fresh cementitious materials are complicated suspensions, consisting of a wide range of particle sizes. Therefore, traditional rheometers designed for polymers and oil are not always applicable without modifications. Without considering the need for control of slippage, the viscosity of cement paste can be measured in usual rotational rheometers geometries, such as parallel plate or coaxial cylinder rheometers. But for mortar (up to 2 mm diameter sand grains suspended in cement paste), the usual rotational rheometer geometries cannot be used, since the gap between the shearing surfaces is required to be several times the maximum sand particle size, so as to treat the suspension as a continuum. Several researchers have measured the rheology of cement paste and mortar in small laboratory size rheometers, e.g. [1, 2, 3, 4, 5]. But with the exception of rheometers used for cement paste, the geometry of the rheometer does not allow for an analytical solution of the flow patterns. Therefore, in order to calculate the viscosity alternative methods, e.g. in form of calibration factors, are necessary.

For measurements of cement paste and mortar, Ferraris and Gaidis [4] argued that a parallel plate geometry offers the most flexibility, as it 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. 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. 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. Unfortunately, known analytical solutions for parallel plate geometry cannot properly account for the cylindrical wall since the no-slip and the gap between the outer part of the plate and the cylindrical wall play an important role in controlling flow. Computer simulation can be a powerful tool for understanding and interpreting fluid flow in a rheometer. A 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. 6].

Experimental measurements and fluid computations were combined to identify the important geometrical factors for the measured values. Using a standard oil having known viscosity, the experimental measurements were compared against obtained from computer simulation. Ideally, a suspension of known viscosity should serve as a reference material. Unfortunately, for now, such a reference suspension does not exist. This paper, a summary of the work presented in [7], will be concentrated on the influence of the confinement walls on the flow of single phase fluids with no suspended particles.

2. EXPERIMENTAL SET UP

An oil was used to determine the influence of the rheometer geometry on the viscosity measured: Cannon S8000 (poly(1-butene) 100 %); nominal viscosity = 34 Pa s at 20 °C. A plate rheometer was used with plates of 35 mm in diameter (PP35). The plates were serrated (-S) or smooth (-P) and the confinement was 37.3 mm in diameter (Figure 1). Other configurations were considered but are not discussed here [7]. 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} [7]. The plastic viscosity was calculated as the slope of the shear rate vs. shear stress curve on the decreasing shear rate branch. All experiments were performed at 23.1 °C ± 0.2 °C.

3. RESULTS AND DISCUSSION

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. 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 [8].

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) as shown in equation 1:

$$T = \eta \cdot f(Geometry, Boundary\_conditions) \cdot \dot{\gamma} \qquad \textbf{[1]}$$

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 η ·*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. The configuration of the model rheometers used in the simulations is shown in Figure 1. To better understand the rheometer's sensitivity to design parameters, we considered the following three cases. In all cases, we 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. 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.

4. RESULTS AND DISCUSSION

Figure 2 shows the experimental results obtained. The line on Figure 2 indicates the nominal value of the oil viscosity at the temperature of the experiments. The viscosity value obtained using any of the configurations 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.

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 we are not aware at this time. Therefore, we combined experimental results 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.

Figure 3 compares the simulation data with experimental data. 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 (Figure 1). If either of these two geometrical factors are smaller than the experimental values (Case 1 and 2), the agreement between the experimental and simulation data is poor. In Case 2 we modified the virtual rheometer by decreasing the distance by a factor of 8 between the top plate and the confinement. In this case, we found a normalized viscosity significantly higher (Figure 3) 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, we used the original rheometer design but allowed for slip on the outside cylindrical part of the rotating top plate. Here the applied torque needed was smaller because we were not requiring the rotating plate to shear the fluid between the top plate and the confinement. As a result, the normalized viscosity appears to be smaller.

While the lattice Boltzmann simulation helps us determine the important geometrical features, we also need a way to calculate the real viscosity from our measurements by estimating the function *f* from equation 1. From the simulation plot in Figure 3 and the data obtained, a linear relationship between the viscosity measured and the ratio of the gap (distance of the shearing plates) to the diameter of the confinement[1] was observed. This linear relationship can be used to calculate a calibration function that will allow the calculation of the true viscosity of the fluid (equation 2). The values of *f* obtained for PP35-S-C and PP35-P-C are 19 ± 1 and 24 ± 1, respectively.

$$\eta_r = \frac{\eta_m}{\frac{h}{D} \cdot f + 1} \qquad \textbf{[2]}$$

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

[1] It should be kept in mind that the shearing plate diameter is fixed and that the only way to change distance, *A*, (Figure 1) is to change the diameter of the confinement.

5. CONCLUSIONS

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. For instance, 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.

In this paper all the experimental work and the simulation was performed using an oil and not a suspension. Further modifications of the rheometer and of the calibration presented here might be needed to correctly measure the viscosity of a suspension.

ACKNOWLEDGEMENTS

Financial support from the Knud Højgaard Foundation, Denmark, for funding Mette Geiker's visit to NIST is gratefully acknowledged. The authors would like also to thank Edward Garboczi (NIST), Vince Hackley (NIST) and Chris White (NIST) for their numerous suggestions on this study.

REFERENCES

[1] Yang, M., Jennings, H.M., "Influences of Mixing Methods on the Microstructure and Rheological Behavior of Cement Paste" *Advn. Cem. Bas. Mat*. **2** (1995) 70-78

[2] Struble L.J. , Sun G.-K.. 'Rheological Changes Associated with Cement Setting." *Proceedings of the 2nd RILEM Workshop on Hydration and Setting*, Dijon (1997).

[3] Banfill P.F.G, "Use of the ViscoCorder to Study the rheology of fresh concrete" Mag. of Concrete Research, vol.42 #153, pp.213-221 (1990)

[4] Ferraris C. , Gaidis J.M., "The Connection between the Rheology of Concrete and the Rheology of Cement Paste" ACI Materials Journal vol. 88/4, 388-393 (1992)

[5] C. F. Ferraris, "Measurement of the rheological properties of cement paste: a new approach", Int. RILEM Conf. "The role of Admixtures in High Performance Concrete", ed. by J.G. Cabrera and R. Rivera-Villareal, Monterrey (Mexico) March (1999).pp. 333-342

[6] Hsieh G., Mason T.O., Garboczi E.J., and Pederson L.R., "Experimental limitations in impedance spectroscopy: Part III--Effect of reference electrode position", Solid State Ionics **96**, 153-172 (1997)

[7] Ferraris C.F, Geiker M., Martys N. S. and Muzzatti N., "Parallel-plate Rheometer Calibration Using Oil and Lattice Boltzmann simulation", Submitted to ACT February 2007

[8] Martys N.S. and Hagedorn J.G., "Multiscale modeling of fluid transport in heterogeneous materials using discrete Boltzmann methods" Materials and Structures, Vol. 35, pp 650-659 (2002)

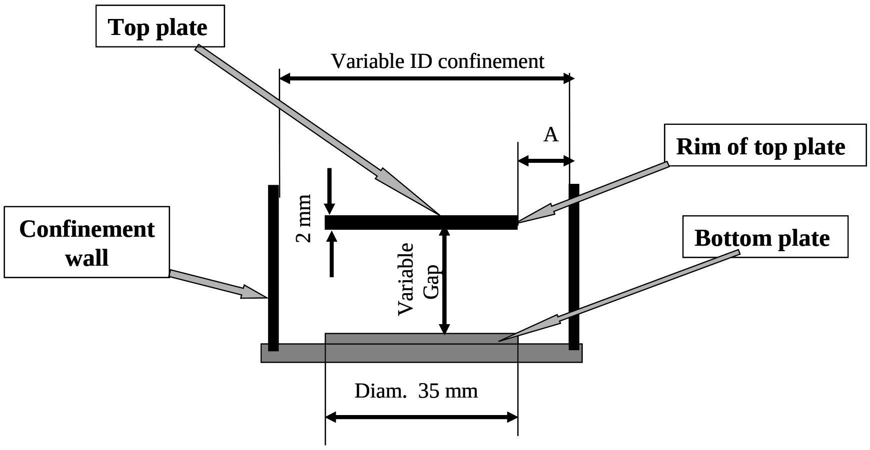

Figure 1: Schematic of the rheometer geometry used in the numerical simulation (not to scale).

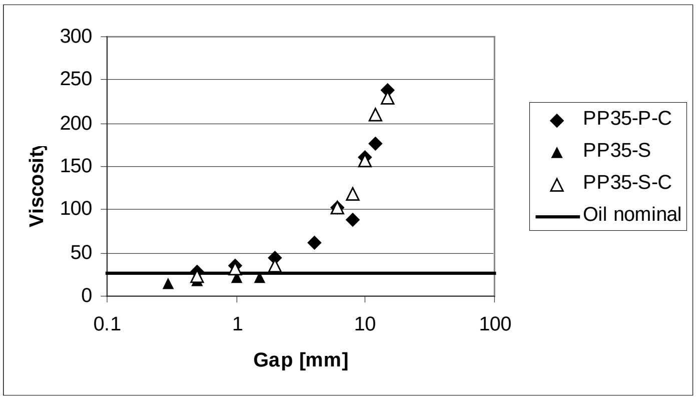

Figure 2: Viscosity obtained as a function of the gap between the plates. The line shows the nominal value of the oil at the temperature of the experiments. The error bars are not shown here for the sake of clarity.

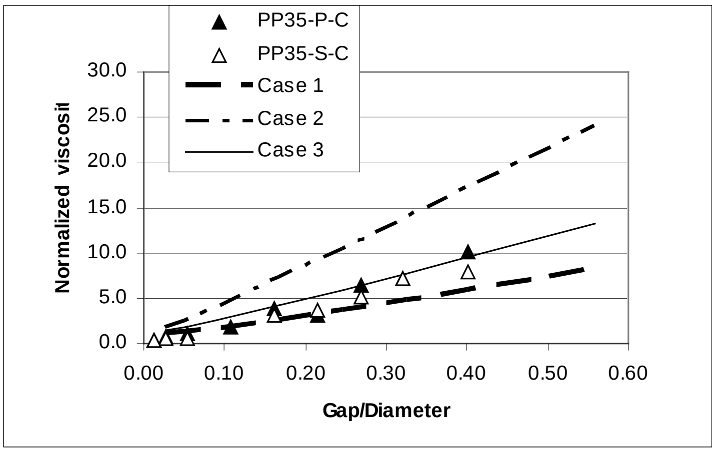

Figure 3: Simulation and data for the PP35 confined geometry. The three solid lines are simulations, and the symbols represent the experimental data (see text).