Effect of Temperature and Pressure on Rheological Measurements of Cement Slurries

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ABSTRACT

The rheological properties of well cement slurries subjected to elevated pressure and temperature were investigated using a rheometer equipped with a pressure cell and a temperature control mechanism. Simultaneous measurements were performed at ambient conditions for comparison purposes. Two different cement types were included in the investigation, as were three different water cement ratios. Extreme pressure was found to have a negligible effect on workability loss compared to elevated temperature. The effect of particle migration is pronounced in the measurements of mixes at high temperature and pressure.

INTRODUCTION

For certain cementitious material applications there is a need for specific flow properties in extreme conditions. This is the case for the well cement slurries, used to support the steel casings in geothermal wells. These slurries need to retain pumpability at high temperature and pressure. It is therefore important to be able to gauge how the rheological properties of these mixes are influenced by extreme conditions. In this work, two rheometers are used to investigate how cement slurries are affected by such conditions. One is a coaxial cylinders viscometer equipped with a pressure cell and a temperature control mechanism. The name of the device is the HAAKE MARS and it is produced by Thermo Scientific. The other rheometer is the ConTec Viscometer 6, a coaxial cylinders viscometer which has been used extensively at the Innovation Center Iceland to measure cementitious paste and mortar mixes[1].

THEORY

Both rheometers in this work are based on a coaxial cylinders geometry. To evaluate yield value (τ_0) and plastic viscosity (μ), one can employ the Reiner-Riwlin equation (Eq. 1)[2].

$$\hat{T} = \frac{4\pi\mu h}{[1/R_\mathrm{i}]^2 - [1/R_\mathrm{s}]^2}\,\omega + \frac{4\pi\tau_\mathrm{o} h}{[1/R_\mathrm{i}]^2 - [1/R_\mathrm{s}]^2}\ln\left(\frac{R_\mathrm{s}}{R_\mathrm{i}}\right) \tag{1}$$

If a linear fit of torque and angular velocity data is available, where the slope is *H* and the point of intersection is *G*, then the rheological properties τ_0 and μ can be calculated using Eq. 2 and Eq. 3.

$$\mu = \frac{H\,(1/R_\mathrm{i}^2 - 1/R_\mathrm{o}^2)}{4\pi h} \tag{2}$$

$$\tau_\mathrm{o} = \frac{G\,(1/R_\mathrm{i}^2 - 1/R_\mathrm{o}^2)}{4\pi h\,\ln(R_\mathrm{o}/R_\mathrm{i})} \tag{3}$$

R_i is the radius of inner cylinder, R_o is the radius outer cylinder and h is the height of the sample in the cylinders (see Fig. 1).

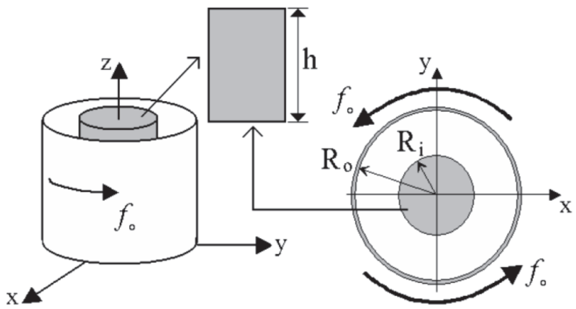

Figure 1. Schematic representation of a coaxial cylinders viscometer[3].

EXPERIMENTAL APPROACH

Two cement types were used in this investigation. Cement A is an API well cement class G (HSR). Cement B is classified as CEM II / A-M and contains 6% silica fume. The cement slurries additionally contain 40 parts silica flour and 2 parts bentonite to every 100 parts cement. This is analogous to the mix design of well cement slurries used geothermal casing applications in Iceland.

A Hobart mixer (NCM 10-30) is used when mixing the cement slurries. The agitator rotates clockwise and the attachment rotates counter clockwise. The attachment is blade shaped, like an open shield. Water is added to the premixed dry materials in the mixing bowl. In this work, when time is referenced, it is the time that starts at this water addition. The mixing procedure for the cement slurries is a five-step procedure: (1) Mixing of dry materials and water at speed 1 for 30 *s*. (2) Hand-mixing for 30 *s*. (3) Mixing at speed 3 for 60 *s*. (4) Hand-mixing for 30 *s*. (5) Mixing at speed 3 for 60 *s*.

After mixing, the slurry is transferred to the two rheometers. The Contec Viscometer 6 is used to obtain reference values, as already mentioned. It is a coaxial cylinders viscometer. The inner cylinder is stationary and registers torque (*T*), while the outer cylinder rotates at a predetermined angular velocity that can be seen in the work by Wallevik[3]. Shearing from the bottom part of the viscometer is filtered out by special means[4]. To avoid slippage between the cylinders and the cementitious material, both the inner and outer cylinders are serrated[5,6]. The sample is agitated between measurements.

The HAAKE MARS viscometer also employs a coaxial cylinders setup. The bob, which is also the inner cylinder, is placed inside a sealed pressure cell and it is rotated by use of magnetic interaction with a rotor which also registers torque (*T*). The temperature in the measurement cell can be set to <180°C while the pressure can be increased to 400 bar, which is the equivalent to the hydrostatic pressure in typical cement paste at 2 km depth. The surface of the inner and outer cylinders is smooth in this rheometer. The stepwise-down angular velocity profile used in the measurements with the HAAKE MARS rheometer can be seen in Fig. 6. The shear rate range was chosen to best resemble the rate of shear expected at the bottom of the well, where the material goes from flowing in the inner casing to flow in the opposite direction in the outer casing. No agitation was performed between measurements due to the sealed nature of the pressure cell.

Different temperature and pressure settings were used in this work. Measurements performed at ambient conditions are indicated by the use of STP (i.e. MARS STP). For mixes that experience temperature and pressure, the notation can be deducted from the following examples: MARS T50iP is a mix that is subjected to 50°C as soon as it is placed in the pressure cell and at 150 bar pressure, MARS T60fP is a mix that is heated to 60°C over 30 minutes in the pressure cell and is subjected to 150 bar pressure immediately, MARS P is a mix that is subjected to ambient temperature and 150 bar pressure.

RESULTS

To compare the two rheometers without the influence of temperature and pressure, measurements were performed at ambient conditions. Fig. 2 shows the rheological properties yielded by the two rheometers for Cement A mixes with three w/c ratios. The results indicate that the MARS rheometer underestimates the yield value and plastic viscosity of the w/c=0.6 mix. There is better correlation for the mixes containing more water.

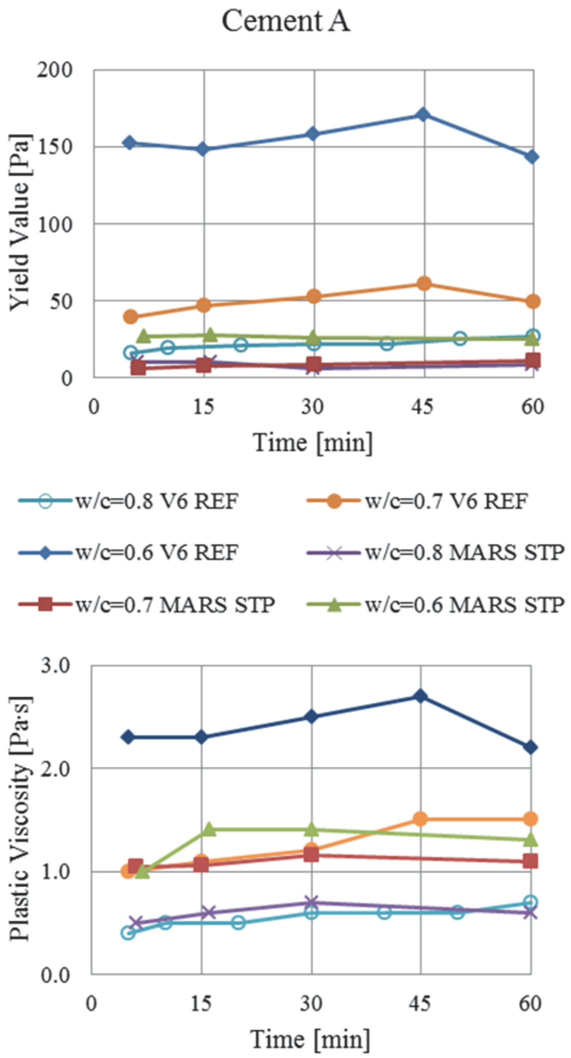

Figure 2. Comparison of rheological properties and workability loss measured at ambient conditions using the two rheometers.

Different temperature and pressure conditions were investigated for the Cement A mix with w/c=0.8. Fig. 3 shows the results for these measurements. The figure reveals that the elevated pressure does not affect the measured rheological properties nor affect the workability loss a significant amount. The MARS T85iP and T60fP mixes show how elevated temperature affects the workability loss of the mix. Adding the mix to a measuring system preheated to 85°C results in near instant stiffening of the mix. The yield values increases fourfold while the viscosity of the system is reduced to almost zero. This reduction in viscosity, albeit less pronounced, is also seen for the mix that is heated up to 60°C over 30 minutes.

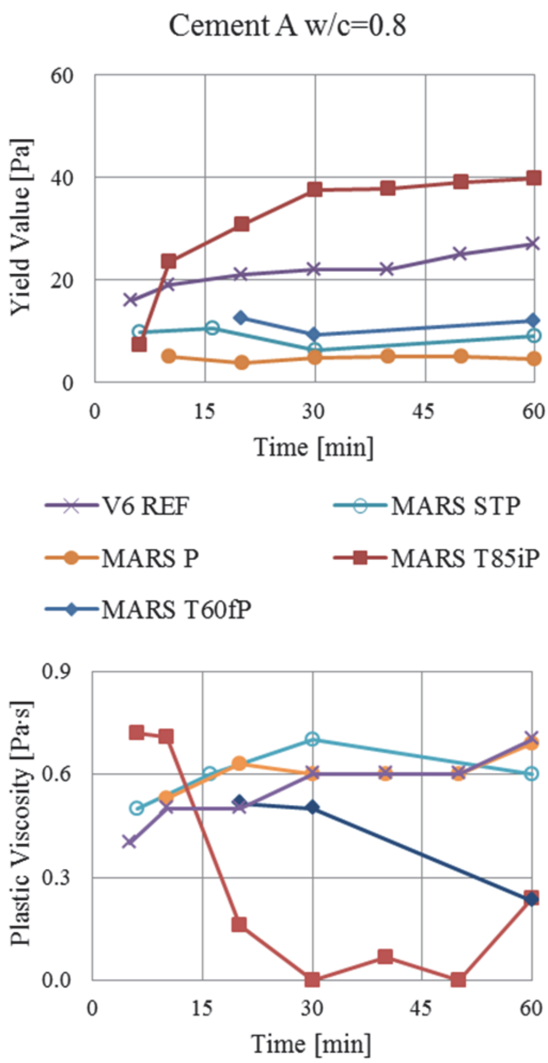

Figure 3. Rheological properties as a function of time for mixes experiencing elevated temperature and pressure.

Mixes based on Cement B were investigated in a similar manner to that which can be seen in Fig. 4, but this time the pressure cell of the MARS rheometer was set to 150 Bar and to reach 42°C after 30 minutes. Fig. 4 indicates that the yield value is underestimated by the MARS rheometer for these conditions for all mixes, while the viscosity results are better correlated. The MARS results show a reduction in viscosity similar to that which was seen for the Cement A mixes in Fig. 3. The viscosity seems to drop as soon as the first measurement is taken and the temperature begins to rise.

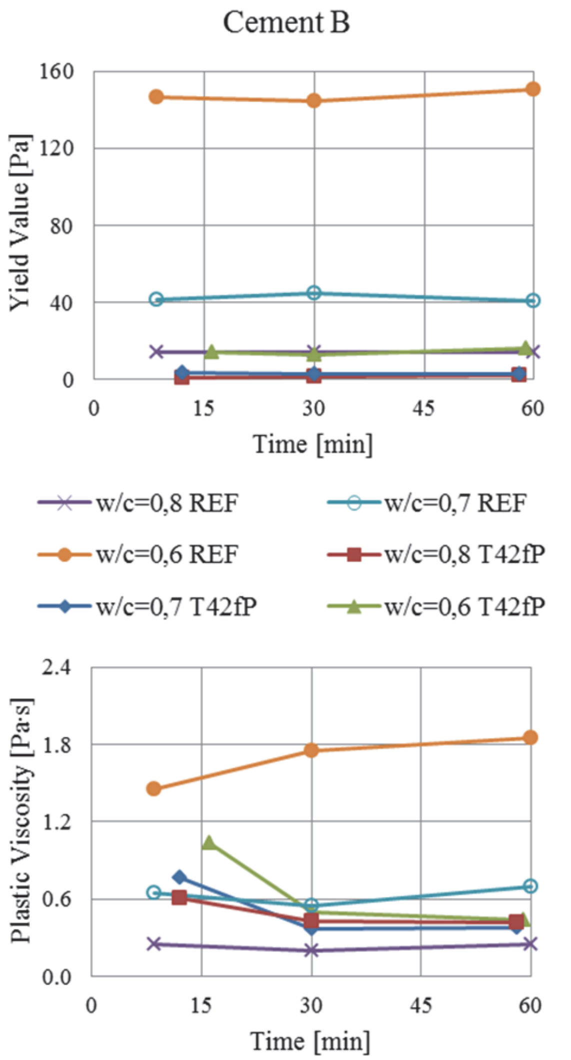

Figure 4. Rheological properties as a function of time for mixes based on Cement B experiencing elevated temperature and pressure.

Figure 5. The bob of the MARS rheometer after it has been removed from the pressure cell after measurement at elevated temperature over one hour.

A reduction in plastic viscosity such as this is unexpected as one would expect overall workability loss as a function of time and increasing temperature. This reduction in plastic viscosity can seemingly be attributed to a number of things. The lack of agitation between measurements is undoubtedly a factor, however, in this case, as was seen for the STP mixes, it is not significant. The smooth nature of both the inside and outside cylinders of the measuring system may also be causing unwanted slippage. Fig. 5 shows the bob of the MARS rheometer after it has been removed from the pressure cell after measurements of a Cement B mix at elevated temperature. A dry layer can be seen on the surface of the bob. This

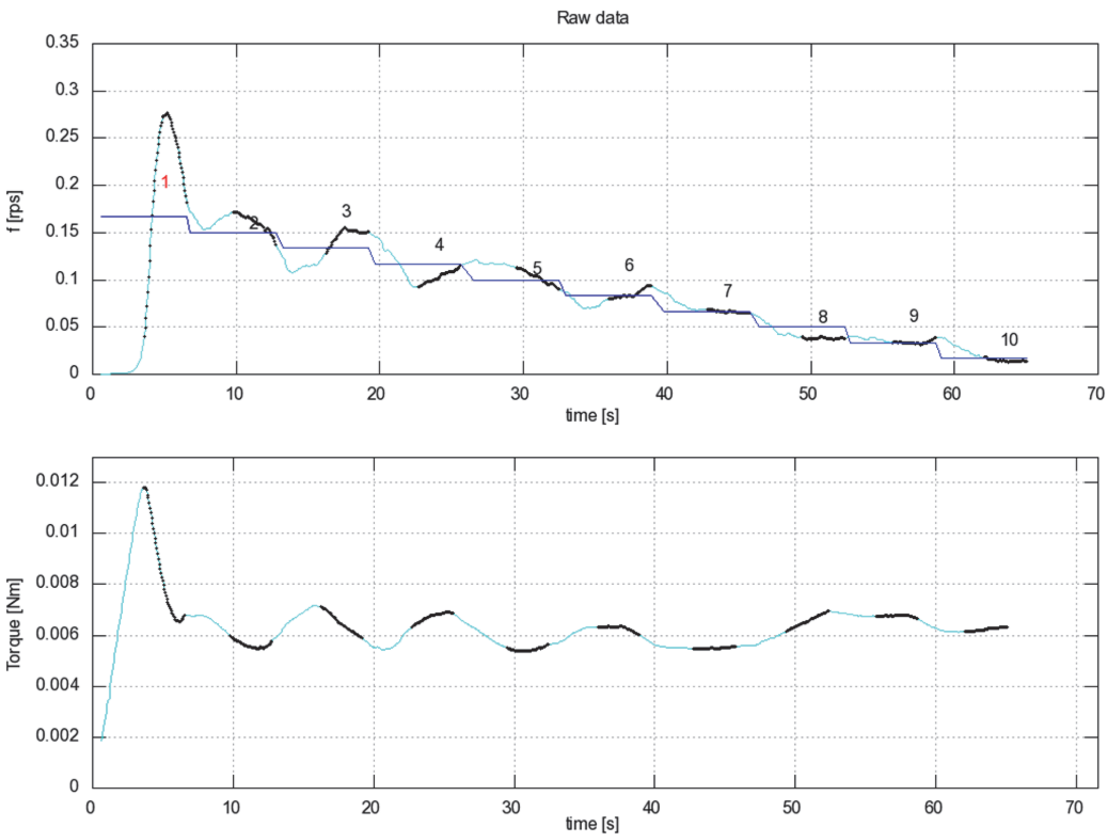

Figure 6. Raw data from a measurement performed at 50 minutes for a Cement B mix, w/c=0.8, at 150 Bar and 42°C.

phenomenon can be attributed to particle migration. This results in the shearing zone of the sample not being representative of the material under investigation.

Fig. 6 shows the raw data from a measurement performed at 50 minutes for a Cement B mix at 42°C and 150 Bar. Although the data shown there is particularly poor, data such as this is fairly characteristic of the high temperature and pressure measurements. Instead of a stepwise reduction in the required torque, the torque oscillates and steady state is not achieved in the rotational frequency. The data points to the material being in a non-homogenous state.

CONCLUSION

The work herein suggests that temperature has a much larger effect on the workability loss of cement slurries than pressure. Elevated temperature accelerates the hydration reaction and also results in increased particle migration in the measurement setup used in this work. The MARS rheometer underestimates the rheological properties of the stiffer mixes investigated in this work. However, the authors are sure that modification of the surface of the inner cylinder of the system would improve this considerably.

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REFERENCES

1. Wallevik, O.H., Gjørv, O.E. and Wierig, H.J. (Ed.) (1990), "10th Proc. of the Rilem Colloquium: Properties of Fresh Conrete, Hanover, 3 - 5 October 1990", Chapman & Hall, London, pp. 213 - 224.

2. Reiner, M. (1949), "Deformation and Flow; An Elementary Introduction to

Theoretical Rheology", H. K. Lewis & Co. LTD, Great Britain, pp. 43.

3. Wallevik, J.E. (2004), "Microstructure-Rheology: Thixotropy and Workability Loss", Nordic Concrete Research, Publication No. 31, The Nordic Concrete Federation 1/2004, Norsk Betongforening, Oslo, Norway, ISBN 82-91341-82-6.

4. Wallevik, J.E. (2004), "Minimizing end-effects in the coaxial cylinders viscometer: Viscoplastic flow inside the ConTec BML Viscometer 3", J. Non-Newtonian Fluid Mech., 155, pp.116-123.

5. Saak, A.W., Jennings H.M. and Shah, S.P. (2001), "The influence of wall slip on yield stress and viscoelastic measurements of cement paste", Cem. Concr. Res., 31, pp. 205-212.

6. Dzuy, N.Q. and Boger, D.V. (1983), "Yield stress measurement for concentrated suspensions", J. Rheol., 27(4), pp. 321-349.