NOVEL RHEOMETER TO MEASURE YIELD STRESS OF SUSPENSIONS

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Abstract

Slump has often been correlated with the yield stress of concrete as defined by the Bingham model. The discussion is still open as to what the yield stress value actually is and how to measure the yield stress of a suspension in general and for a cementitious material in particular. A plate rheometer is a recent development in the measurement of yield stress of suspensions that allows shear rates far below most rotational rheometers. This paper will present the rheometer and the modifications made so that it can be used with suspensions such as welan gum and bentonite in aqueous solution, as well as cement paste. A careful analysis of the yield stress measurements will be discussed.

1. INTRODUCTION

Most suspensions, such as cement paste and concrete, do not behave as a Newtonian material for which the flow behaviour can be characterized entirely by the viscosity. A suspension flow should be characterized by at least two parameters [1, 2, 3] and the most commonly used are the Bingham parameters, yield stress and plastic viscosity. The slope of the shear stress vs. shear rate is the plastic viscosity and the intercept at zero shear rate is the yield stress. The measured yield stress depends on the range of shear rate selected and it is really an extrapolation more than a direct measurement. According to Hackley et al. [3], the physical definition of yield stress is the stress needed to initiate movement; therefore it should be measured by slowly increasing the shear stress until movement occurs. This direct method is not easy to implement in most rheometers because it implies that the shear stress is very well controlled and most rheometers cannot control the stress with step small enough to detect the yield stress. Also, most concrete rheometer are not stress controlled but only shear rate controlled. Other methods [4] were conceived to measure yield stress, such as stress growth.

This method requires the material to be sheared at a very low shear rate. The lower the shear rate, the better the estimation of the yield stress. Amziane et al. [5] showed how this method could be applied to a concentrated suspension such as cement paste. They also concluded that a shear rate lower than 0.01 s^{-1} that is lower than most rotational rheometers could currently provide would be beneficial.

Zhu et al. [6] designed a novel rheometer that allows a controlled and very low shear rate. The shear rate is imparted by moving a plate very slowly edgewise through the suspension. The shear stress is measured as the drag force necessary to pull the plate. In this experiment, the shearing is due to the relative motion of the plate and gravity acting on the material. This set-up is not totally novel as the principle of operation is similar to the ball rheometer. The novelty resides in the fact that the plate is designed to create suspension/suspension shearing and not between the object or plate and the suspension [6]. This will eliminate the boundary effect and lead to the measurement of the "true" yield stress.

2. EXPERIMENTAL PROGRAM

2.1 Materials

Three materials were used: bentonite, welan gum, and cement paste (with a retarder). The bentonite suspension was a 4.3 % concentration by volume in water. This material was selected because it is similar to cement with regards to particle size distribution but the viscous properties are constant in time.

The welan gum suspension is a mixture of welan gum and water at concentration of 5.8 % by volume. As this material is a natural product, it degrades very quickly (in days); therefore to stabilize it, a biocide was added that allows the suspension to be stable for months.

The last suspension was cement paste with water/cement (w/c) ratio of 0.33 by mass. This 48 % concentration by volume in water can be considered to be a high concentration suspension. A retarder admixture was added to allow a longer time to conduct the measurements.

2.2 Description of the Plate device

The instrument set-up as used previously by Zhu et al. [6], shown schematically in Figure 1, was designed for very diluted suspensions. The scope of this study was to apply the technology to concentrated suspensions such as cement paste. It consists of a container with the material to be tested placed on a platform that can move up and down. The movement is controlled by a computer through a step-motor with a speed of 0.05 mm/s. A balance is placed on a support on top of the container. The balance support cannot move and a hook below the balance suspends the plate.

When the plate is immersed in the material and the container is slowly moved downwards, the material in the beaker exerts a force on the plate. The plate in turn exerts a force on the balance, and the resulting force and corresponding displacement are recorded by a computer at near real time.

Various types of plates were used to ensure that the measurement recorded the shear stress within the material. The plates were laser cut from one 1.2 mm thick stainless steel sheet. Sandblasting on both faces was performed to roughen the surface and minimize slippage. The plates were of two types: smooth plane plate and a slotted plate (SL). The smooth plates were of various lengths: 52 mm (short), 75 mm (medium), and 100 mm (long). The slotted plate had, as shown in Figure 2, only one length, medium (75 mm). It is assumed that when the slotted plate moves in the material, the suspension in the slot remains static relative to the plate. Zhu et al. [7] have shown that if the ratio of the height of a slot over the thickness of the plate is smaller than 3, the suspension filling the slots can be considered static, with no secondary flow and with shearing occurring only at the edge of the slots. Also, various sizes and shapes of container were used to establish whether the size of the container had an influence on the measurements. It was determined [9] that if the distance between the plate and the wall of the container is greater than 20 mm, the size and shape of the container have no influence on the results for the three suspensions investigated in this study.

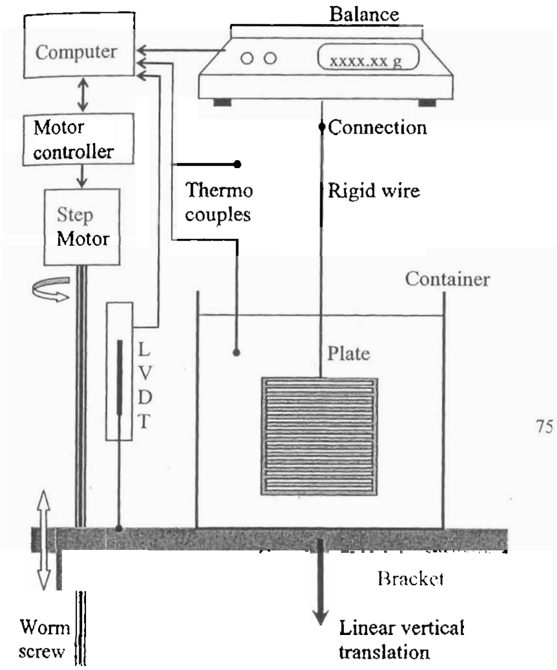

Figure 1: Experimental set-up
(not to scale)

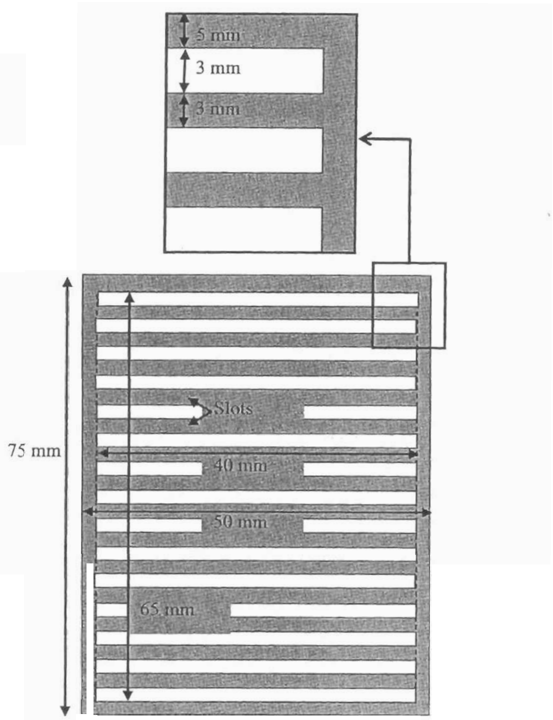

Figure 2: Slotted plate design
(not to scale)

2.3 Parallel plate rheometer

A parallel plate rheometer was also used to compare the results obtained with the plate device. This rheometer is described in detail in Ref. [5, 8]. It consists of two 35 mm, serrated, circular plates. The top plate rotates at a controlled shear rate and the torque generated from the resistance of the material tested was measured on the top plate. The temperature was controlled at 23 °C ± 2 °C.

2.4 Procedure for the plate device

The following procedure was used to place the material. The material was first loaded in the container and consolidated by rodding. Then the container was placed on the platform and the plate selected was attached to the scale and lowered into the material by slowly raising the platform. The material was agitated with a spatula to ensure that the plate was hanging from the balance and was not supported by the material. The balance was then zeroed.

The motion of the platform is schematically illustrated in Figure 3. The 600 s of waiting period was designed to ensure that the material was at rest and always in equilibrium. The measurements were performed by slowly lowering the platform while recording both the position and the balance reading. Figure 3 shows a typical recorded response and is discussed later.

The speed was selected so that the test duration should be short enough to consider the rheological properties as constant during the measurement period, even for cement paste.

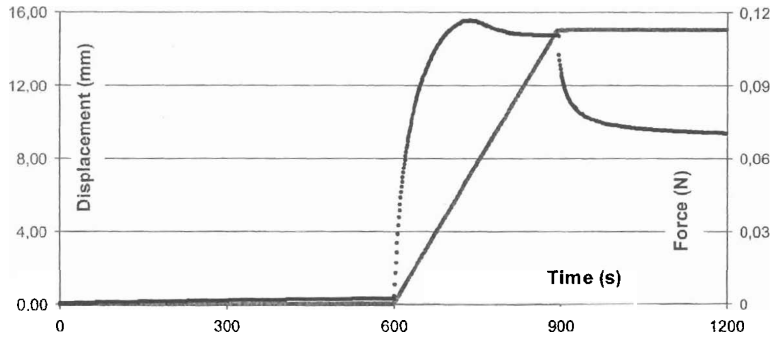

Figure 3: Schematic strain ramp applied and typical force response obtained (case of bentonite suspension)

The combined plate and wire mass less the buoyant force FI must be subtracted from the measured force in order to obtain the net force F_R, due to the displacement of the plate inside the beaker. The smooth plane plates were used to correct the drag force F_D exerted on the plate edges and to evaluate τ_S, the stress from the steel surface. Plotting F_R versus the length of the plates generates a straight line with intercept F_D, and with a slope related to τ_S.

Using the slotted plate, the true suspension yield stress τ_B is measured at the interface of the suspension inside the slots and the adjacent bulk. The mean stress exerted on the plate is the combination of the yield stress τ_B over an area S_{Bulk} and τ_S over an area S_{Steel}. τ_S is assumed to be lower than τ_B due to slip. The mean shear stress τ exerted on the plate is a function of the area ratio $\beta = S_{Bulk}/S_{Steel}$, τ_S, and the parameter to measure, τ_B as shown in equation 1:

$$\tau = [\beta\tau_b - (1-\beta)\tau_s] \quad (1)$$

The drag force depends on the sample as well as on operating conditions such as the plate speed and the test temperature, but it is not a function of the slot ratio β since the suspension in the slots moves with the plate. The slotted plate used has a ratio β = 0.315. It allows one to extrapolate the contribution of the slippage effect and to deduce the yield stress τ_B of the suspension [9].

3. RESULTS AND DISCUSSION

The analysis of the measured net force versus plate length shows a perfect correlation for low concentration suspension. Figure 3 shows that at first, the stress increases almost linearly, confirming the elastic nature of the material before yielding. Just before the stress reach its maximum, a nonlinear elastic behaviour is also observed, the slope of the curves decreases significantly likely associated with structural rearrangements. At the maximum stress, obstructions are overcome collapse, and the suspension flows. Beyond this point, a constant shear rate may induce a decrease in stress and viscosity due to the thixotropic nature of the material. The yield stress value is determined from the maximum stress recorded. This represents a dynamic yield stress. The static yield stress, which is not a function of platform speed, is determined from the force level at departure from linearity on Figure 3 [10] and results in a lower yield stress value. Zhu et al. [6] have shown that the value depends on the strain rate, but that at a sufficiently low speed, around 0.01 mm/s, the yield stress ceases to be a function of platform speed. However, the rheological properties of sample change according to the hydration kinetics of cement paste. To consider constant properties during testing, the platform speed has been selected at 0.05 mm/s. A lower speed would not allow one to clearly observe a maximum stress as shown in Figure 3. If the strain is stopped, a stress relaxation will be recorded (see Figure 3), and the remaining stress recorded at the end of the test is associated with the residual stress, which cannot be used to compute a reliable value of yield stress because of the thixotropic nature of suspensions studied [9].

Yield stress measurements are affected by the time scales of the experiment [6]. The longer the measurement time, the smaller the yield stress value. The yield stress is reached in a few seconds while using a parallel plate test, and in approximately 100 seconds in a plate test. The values obtained for low concentration suspensions (welan gum and bentonite) are thus lower with the plate test.

Table 1: Comparison of the results obtained with the plate device and the parallel plate rheometer*Approximated value

Mixes	Plate device	Parallel plates
Welan Gum 7 %	87 Pa	220 Pa
Bentonite 12 %	6 Pa	25 Pa
Cement paste w/c = 0.33 with retarder	126 Pa	135 Pa
Cement paste w/c = 0.33 neat	130 Pa*	145 Pa

The error of the results shown in Table 1 is estimated to be about 10 % for the measurements with the plate rheometer [2, 5]. For the tests using the plate device from Table 1, the estimation of the error is harder due to the more limited number of tests performed with this device. Nevertheless, it can be assumed that the error is also of about 10 %. More discussion on this topic will be provided in Ref. [9].

4. CONCLUSIONS

Yield stress measurements are difficult to perform, especially when hydration phenomena occur during testing. However, yield stress is a key parameter in flow models concerning fresh cement-based materials. This paper summarized an attempt to develop a novel rheometer to study the yields stress of high concentration suspensions, like cement paste. This novel rheometer is easier to build with off the shelf parts than rotational rheometers, while allowing yield stress measurements at lower shear rates.

Further development and tests are needed to ensure that the results obtained are the true yields stress and are not incorrectly computed due to other phenomena, e.g., thixotropy, mixing procedure before test, and time evolution of the material due to hydration. Also, even without these complicating factors, additional experimentation is needed to determine the static yield stress more accurately.

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