MEASUREMENT OF THE RHEOLOGICAL PROPERTIES OF CEMENT PASTE: A NEW APPROACH

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Abstract
This paper presents a new approach for predicting the rheological properties of concrete from rheological measurements on cement paste and from concrete composition. This approach requires measurement of the cement paste rheology under the same conditions as those experienced in the concrete. It is known that the rheology of concrete is affected by the cement paste content, by contributing to the gap between the aggregates. A parallel plate fluid rheometer was used to simulate the gap between the aggregates. It is demonstrated that the distance between the plates affects the rheological parameters of cement paste. Some preliminary results are presented and discussed. As the mixing procedure affects the values of the rheological parameters of the cement paste, special precautions were taken to mix the paste under the same conditions as those experienced in the concrete.

1. Indroduction

The purpose of this paper is to present a new approach for predicting the rheological properties of concrete. The relationship between cement paste rheology and concrete rheology has never been completely established. It is clear that changes in the rheology of cement paste affect the concrete. For instance, to increase workability, water or high range water reducing admixtures (HRWRA) are added. These materials mainly affect the cement paste rheology as they have almost no effect on the aggregates. Therefore, it is assumed that a correlation between cement paste and concrete rheology should be possible. The main reason that such a correlation has never been successful is that cement paste rheology is always measured under conditions that are never experienced by the cement paste in concrete. The values

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usually reported in the literature for cement paste correspond to bulk values and do not take into account the contribution of the aggregates [1]. A method is being developed to predict concrete rheology from constituent properties that will include measurements on cement paste using a novel procedure and a simulation model to simulate the concrete being sheared. The cement paste measurement methodology will be presented here. The details of the simulation model, which is under development, will be presented elsewhere [2].

Determining the correct method for measuring cement paste rheology requires examination of the conditions that cement paste experiences in concrete. Various factors were addressed in designing the proposed test procedure:

- Cement paste is "squeezed" between the aggregates. The distance between the aggregates, the gap, depends on the paste content of the concrete considered [3]. Therefore, the rheometer geometry must be a variable geometry in which the gap can be changed. A parallel plate rheometer is a suitable device.
- The mixing of cement paste must imitate the shear stresses experienced in concrete. Portland Cement Association (PCA) [4] developed a methodology and identified the hardware required.
- As the purpose is to correlate paste rheology with concrete, the parameters of the cement paste tests should be selected to correspond to the parameters of the concrete tests [5].

This paper presents the methodology to measure cement paste rheology and give some results that show the influence of the gap and the addition of admixtures on the rheological properties of cement paste.

2. Background

The rheological behavior of a fluid such as cement paste, mortar or concrete is most often characterized by at least two parameters, τ_0 and μ, as defined by the Bingham equation [7]:

$$\tau = \tau_0 + \mu \dot{\gamma} \qquad (1)$$

where τ is the shear stress applied to the material (in Pa), τ_0 is the yield stress (in Pa), μ is the plastic viscosity (in Pa·s), and $\dot{\gamma}$ is the shear strain rate (also called the strain gradient) (in s^{-1}). The yield stress and the plastic viscosity are the Bingham parameters that characterize the flow properties of the material. For special concretes, such as self-leveling concretes, a third parameter might be necessary to correctly represent the shear rate-shear stress relationship. Other equations have been used for describing the concrete flow, because in certain circumstances concrete flow does not obey the Bingham equation [5, 6]. The cement paste on the other hand is either described as a Newtonian fluid ($\tau_0 = 0$, $\mu \neq 0$) or a Bingham fluid ($\tau_0 \neq 0$, $\mu \neq 0$).

Figure 1 explains why if only one of the two parameters is determined, prediction of a material field performance might not be correct. To determine the yield stress and the viscosity of cement paste, mortar or concrete, the instrument must be able to measure stresses generated at a minimum of two different shear rates [7]. Nevertheless, most of the instruments or methods in use for concrete are either not able to shear the concrete at various shear rates, or the shear rates are not measured or controlled [8]. Therefore only one of the two Bingham parameters can be estimated. Tattersall [7] was the pioneer in developing a concrete rheometer with controlled shear rates capable of measuring the stresses in concrete. Presently, three commercially available rheometers exist capable of varying $\dot{\gamma}$: IBB [9], BML viscometer [7, 10] and the BTRHEOM [11, 12]. The results of research presented in [5], were obtained using the BTRHEOM because it gives the rheological parameters in fundamental units, i.e., Pa and Pa·s for yield stress and viscosity respectively. The instruments mentioned above can be used for mortar, although some tests specifically designed for mortar do exist, i.e., the flow table [13].

The situation for cement paste is different, because the largest particle size is still small enough to allow the use of a fluid rheometer designed for other materials. Most

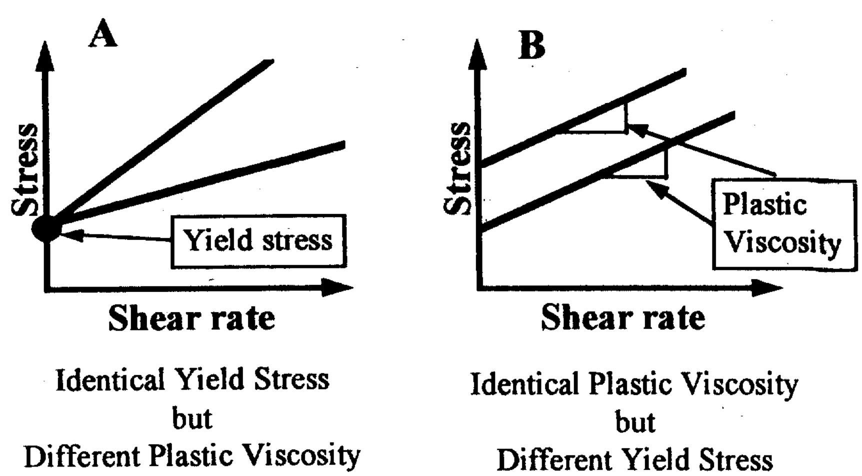

Figure 1: Definition of the Bingham parameters for concrete flow

researchers use a concentric cylinder geometry [4, 14], because it is the most common geometry available, but great precautions need to be taken to avoid sedimentation and slippage. Also, the gap or distance between the two cylinders is fixed. Therefore, the values obtained are those of bulk cement paste, not of the cement paste confined between two aggregates, preventing a possible correlation with concrete. The only geometry that allows a variable gap is the parallel plate (see Figure 2). Few researchers [3, 15, 16] use this geometry for cement paste. Unfortunately, the only part that the three researchers from [15, 16, 3] have in common is the geometry of the instrument. The data and the scope of their research are all different. It is beyond the scope of this paper to discuss their different approaches.

The shear history of a cement paste affects its flow parameters, therefore it is important to handle the cement paste with the same shear rate as it will experience in concrete. The shear rate of cement paste in concrete during mixing and placement was established by Helmuth at 70 s^{-1} [4]. Also the shear rate applied to the cement paste in concrete during a rheological test is from 3 to 24 s^{-1}, as determined when the calculation are based on the shear rate applied to concrete in a BTRHEOM rheometer [5].

The mixing method affects the rheological response of the cement paste [15], therefore a controlled speed mixer is the best method to insure that the cement paste is always mixed in the same way. The method was designed by PCA [4]. It will be shown how this new method improves the reproducibility of the results.

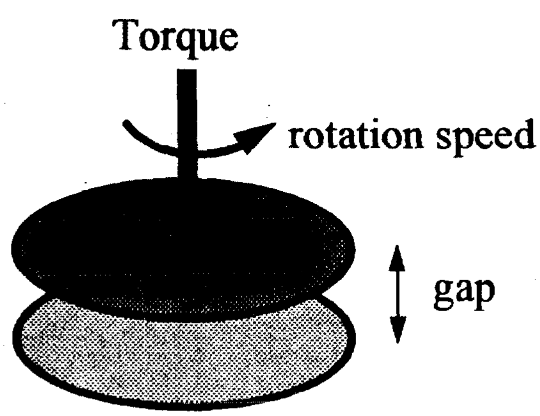

Figure 2: Parallel plate geometry

Depending on the cement paste content of a given concrete, the gap between the aggregates can vary significantly. To estimate the average gap between aggregates we used a mathematical method developed by Garboczi and Bentz [17] based on equations developed by Lu and Torquato [18]. The aggregates are treated as spheres suspended in a cement paste matrix. The volume of paste contained in a shell of thickness r around each aggregate is accurately given by the equations, even allowing for overlaps between shells. The value of r is computer where 99 % of the paste is contained in the shells, and the gap is taken to be twice this value. The mathematical calculation has been shown to be very accurate for a wide range of concretes using numerical analysis [19]. As examples of concretes to calculate the gap, concretes described in ref. [5] were used. The gaps were computed to be between 0.16 mm and 0.22 mm. These gaps are much smaller than gaps used by most researchers in concentric cylinders geometries, which are in the range of 0.7 mm to 1 mm. The gaps calculated to be in concrete are of the order of 2-3 times the maximum particle size of the cement, while the gap used by most researchers is 20-25 times the particle size. These large gaps lead to measurements of the bulk values of the cement paste rheological parameters and not the "correct" value to be used to correlate cement paste rheology with concrete rheology.

In summary, a test for cement paste that is designed to be used to predict concrete rheology should have a mixing pattern that reproduces the shear history of concrete, a gap between the plates of the rheometer that is related to the cement paste content of the concrete, and a shear rate sweep range that corresponds to the shear rate range of the concrete tested.

3. Experimental Program

To mix cement paste, the methodology adopted was that developed by PCA [4]. The system consists of a blender mixer with a capacity of 1 liter connected to a speed controller and to a temperature controlled water bath. The speed controller allows the speed of the blades in the mixer to be held constant regardless of the load. The cement paste resistance to mixing can vary with the viscosity or yield stress of the paste. The more viscous or higher the yield stress, the higher will be the load for the blades. The controlled temperature bath is set to 15 °C. This temperature allows the paste to be about 20 to 22 °C at the end of the mixing cycle, which is the temperature that is measured in concrete just after mixing. If the base of the mixer was not cooled, the temperature would be much higher and, therefore, the rheological parameters will correspond to a material at a different stage of the hydration process. In concrete the aggregates are a heat sink that replaces the role played by the cooled base of the mixer.

The mixing regime adopted was:

- Add water and any admixture to the mixer
- While the mixer blades rotate at a speed of about 4000 rpm the cement is introduced in 30 sec
- The speed of the blades is increased to 10000 rpm and kept constant for another 30 s
- The mixer is stopped and the walls of the mixer scraped
- After 2.5 min. of rest the mixer is turned on again at a speed of 10000 rpm for 30 s.
- The temperature is measured just after this cycle.

A precise volume of cement paste is transferred to the rheometer, using a disposable syringe (without the needle) having a maximum capacity of 1 ml. Depending on the gap the correct volume is selected. The amount of paste needed for the rheometer should uniformly fill the volume between the two plates with very little excess.

Once an amount of paste is placed on the lower plate of the rheometer, the two plates are automatically driven to the preset gap by the computer. The measurements are then initiated. To homogenize the paste between the plates, the top plate of the rheometer is rotated by predetermined steps up to a shear rate of 80 s^{-1} for 160 s. The torque measured is stable after this treatment. Then the shear rate is again swept between 0 and 24 s^{-1} (up curve) followed by the down curve with the same shear rate range as the up curve. Only the down curve is considered for the calculation of yield stresses and viscosities.

To avoid slippage during the measurements of the torque at varying shear rates, the plates of the rheometer are covered with an abrasive paper. The paper had a self adhesive backing and had a grit size of 240. This insured that the grit particles had the same average size as the cement particles.

4. Results and discussion

To determine the validity of the methodology adopted, the following tests were performed:
- Scatter of data with and without the controlled speed mixer
- The influence of addition of HRWRA on the yield stress and viscosity
- The influence of the gap

To determine that the controlled mixer was necessary and that no other cheaper method could be used, some tests were made using an adjustable voltage controller to set the blade speed. This simple system does not allow a feedback loop to keep the speed constant if the load from the cement paste changes from mix to mix. Six batches were preparesd of an identical cement paste design and tested them according to the procedure described above. Three specimens were mixed using the controlled voltage mixer and three using the controlled speed mixer. The cement paste had a w/c of 0.4 and no admixtures were used. It is clear from Figure 3 that the controlled speed mixer gives a better reproducibility of the data.

To determine the influence of the gap and of the dosage of HRWRA on the yield stress and viscosity, six batches of cement paste at a constant w/c of 0.36 and three dosages of a melamine based HRWRA were prepared. The dosages were 0, 1%, and 2% by mass of the cement. The cement was of type I and its complete characteristics are reported in Ref. [5]. Two gaps were selected: 0.16 mm and 0.26 mm. A full factorial design using the dosages and the gaps was used as described above. All measurements were performed twice using a new batch of cement paste each time. The yield stress and the plastic viscosity were calculated according to a Bingham model. Figure 4 and Figure 5 show, respectively, the yield stress and the plastic viscosity. The error bars in the figures represent one standard deviation.

The yield stress shows, as expected, a dependence on the dosage of the HRWRA. With the higher dosages giving lower yield stresses. The plastic viscosity is also reduced by the addition of the HRWRA but a higher dosage does not seem to significantly reduce it. These statements need to be verified further because the standard deviation is relatively high in some of the data.

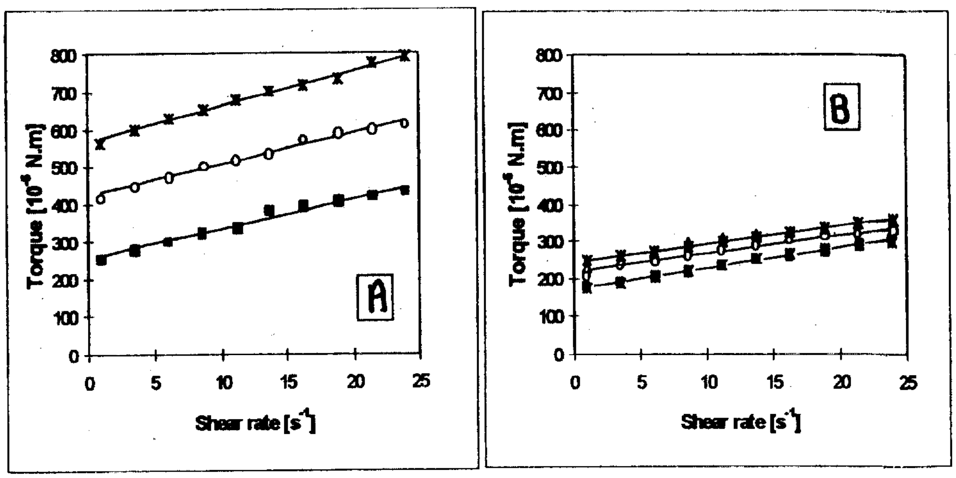

Figure 3: Torque vs. shear rate using two methods for controlling the speed of the blades during mixing. A) Adjustable voltage controlled mixer with no feedback; B) Controlled speed mixer with feedback. The error in the measurement of the torque is about 10 %.

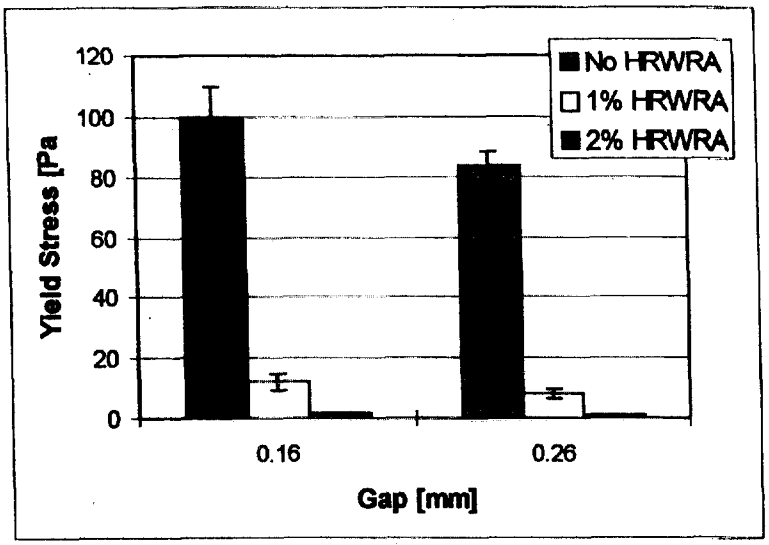

Figure 4: Yield stress vs. gap for various dosages of HRWRA. The error bars represent one σ.

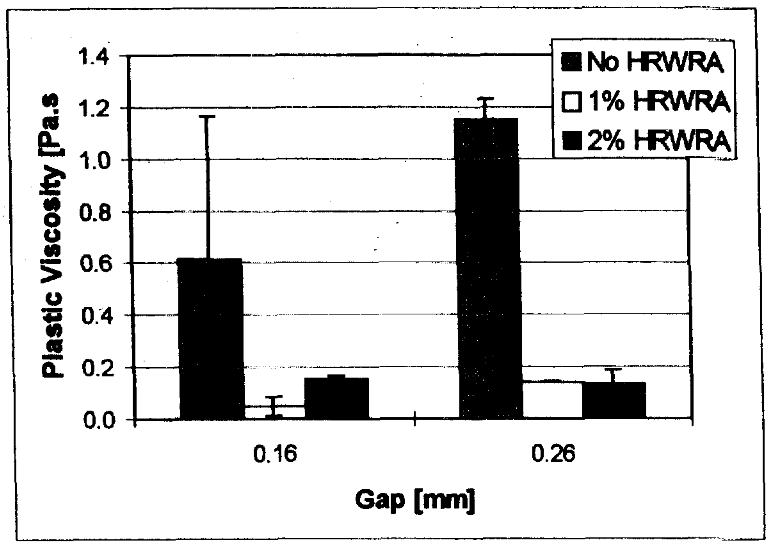

Figure 5: Plastic Viscosity vs. gap for various dosages of HRWRA. The error bars represent one σ.

The influence of the gap showed an interesting result. The yield stress increases with a decreasing gap size. This was expected because HRWRA is designed to reduce the yield stress. In concrete the yield stress is the most commonly measured value because it is related to the slump cone test. On the other hand, the viscosity shows either the opposite influence or no influence at all. The explanation of the strange behavior of the viscosity needs to be further investigated especially because the error (showed in Figure 5 by the bars) is very large for the measurement done at 0.16 mm gap with no HRWRA. The non-influence of the gap on the viscosities for the cement paste containing HRWRA is an interesting discovery. It could imply that if the yield stress is overcome then the paste is not influenced by the proximity of the aggregates, represented here by the gap.

5. Conclusion

To obtain reliable measures of cement paste rheology several precautions need to be taken to insure reproducibility. Also, for possible prediction of concrete rheology from cement paste, the measurements should be made in a variable geometry rheometer, i.e., parallel plate. The gap should be carefully selected to reflect the true value of the distance between the aggregates. The method developed by Garboczi and Bentz is deemed adequate to predict the gap in concrete from the granulometry and the amount of cement paste selected. The limited results show that the gap greatly influences the yield stress while having practically no influence on the viscosity. Further measurements and studies need to be performed to confirm these results.

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