The effect of mixing on the rheology of cement-based materials containing high performance superplasticisers

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ABSTRACT

Superplasticisers are dispersing agents capable of reducing the water needed to produce workable concrete by 30% to permit higher strength and durability, but their performance interacts with the way the mix is produced. The shear rate imparted to the paste during industrial mixing procedures was modelled by controlled stress rheometry and the results showed that the measured rheology is highly sensitive to the energy imparted during mixing.

INTRODUCTION

It is well known that the measured rheology of a cement-based paste depends upon the type and fineness of the constituent solid materials as well as the water-solids ratio and the type and amount of additive used[1]. While generally characterised by the Bingham model, the previous shear history of the paste has a significant effect on the measured rheological properties and this is probably due to destruction of the bridges formed of hydration products, which link particles together[2]. Orban et al drew attention to the risk of applying conclusions from laboratory tests carried out on cement slurries prepared in high shear mixers to industrial situations where low shear field mixers are used. They introduced the concept of the specific mixing energy[3], subsequently applied to mortars by Banfill[4].

This paper presents the results of recent research concerning the effect of mixing shear on rheology. The first case describes the simulation of a high shear mixing regime which was designed as a basis for the assessment of grout formulations. The second case involves a simulation of the influence of low shear mixing on the water demand for a given level of rheology.

INSTRUMENTATION

All results were obtained using a CSL500 controlled stress rheometer (TA Instruments) fitted with a specially designed impeller. It is an interrupted ribbon helix, which maintains the axial semi profile necessary to permit the well known equations for a concentric cylinder rheometer to be used. It is machined from brass to fit inside the 30mm diameter cylindrical cup and consists of spiral blades with short interruptions which permit the paste to fall back under gravity into the cup and a 1mm gap to minimise the shear rate variation across the sample from blade surface to cup wall. Previous work and calibration by the Metzner-Otto approach confirmed that it performs satisfactorily with very fluid pastes containing superplasticisers and can deliver results in fundamental units[1,5].

HIGH SHEAR MIXING

The mixing regime

Plant A had established a grout mixing regime, based on laboratory scale mixers, for testing the suitability of new grout formulations (using ground granulated blastfurnace slag with Portland cement and a naphthalene sulphonate superplasticiser) in their production line. The challenge was to simulate this mix regime using the CSL rheometer to enable simulation of any other mix regime including the full scale mixers used at Plant A. This would enable, not only the testing of small amounts of materials for their suitability in the grout process, but also mapping of the shear history of the grout during its preparation to gain a better understanding of the dependency of the grout properties on mixing parameters.

The plant A mix procedure begins with the solids in a small Hobart bowl. The required water amount is added over 30 seconds while the mixer is working on setting 1. The mixing is then continued for a total time of 10 minutes. The bowl and contents are then transferred to a Silverson high shear mixer and mixing continued for a further 10 minutes. The bowl and contents are then transferred back to the Hobart mixer and mixing continued for a total time of 150 minutes. The rheological properties of the grout were then assessed using the standard Plant A procedure.

Mixing simulation

Fig. 1 shows the flow curves obtained from the Plant A ‘standard’ grout formulation during an acceptance mix procedure. The grout was sampled for testing in the CSL rheometer after the initial Hobart mixing stage (H10), again after the Silverson mixer stage (S20), during the Hobart ‘hold up’ stage at 60 minutes (M60), and finally after 150 minutes (M150). The sampled grout was mixed for two minutes at a constant shear rate of $50s^{-1}$. This preshear was followed by the measuring stage where the shear rate was raised in a 20 step ramp from 0.15 to $10s^{-1}$ and then a further 20 step ramp from 10 to $200s^{-1}$.

For the purposes of this initial simulation exercise testing was carried out at $25^{o}C$. A ‘standard’ formulation grout was then presheared in the CSL at $50s^{-1}$ for 10 minutes before being tested with the normal two stage ramp routine. When this did not prove adequate, the experiment was repeated with a preshear rate of $100s^{-1}$. The results of these tests are given in Fig. 2, from which it is clear that a close approximation of the H10 situation has been reached by the latter procedure.

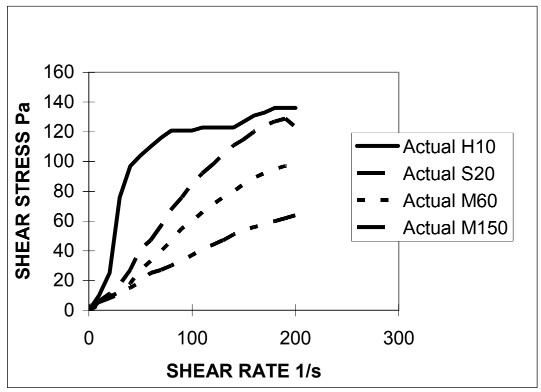

Figure 1. Actual acceptance mix flow curves.

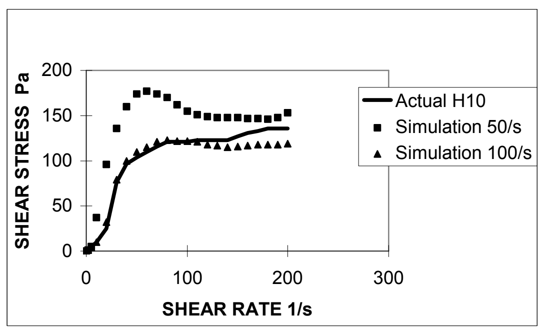

Figure 2. H10 simulation flow curves.

The Silverson phase was simulated by subjecting the grout to 10 minutes at $100s^{-1}$, then the test regime and then 10 minutes at $300s^{-1}$ followed by another test regime. The results of this procedure are shown in Fig. 3. As can be seen the second phase preshear had to be increased to $650s^{-1}$ (the maximum that the CSL can achieve with the helical geometry) before a good simulation was achieved.

The M60 phase of the mix was simulated in a similar fashion. The grout was subjected to 10 minutes at $100s^{-1}$, then a test regime, then 10 minutes at $650s^{-1}$ and then a test regime. At first the next preshear was set at $25s^{-1}$ for 24 minutes so that the total time elapsed at the commencement of the third test regime was 60 minutes. The results of this phase of the simulation are given in Fig. 4.

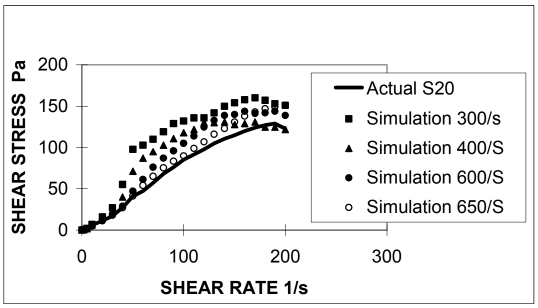

Figure 3. S20 simulation flow curves.

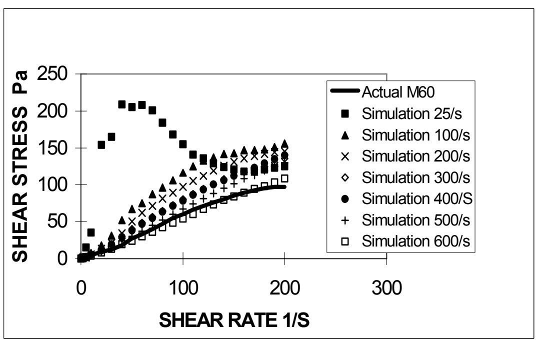

Figure 4. M60 simulation flow curves.

The $25s^{-1}$ preshear produced a most unexpected effect, demonstrating that if the grout is not sheared sufficiently during the M60 hold up stage it begins to rebuild the original structure. The preshear rate had to be increased to $600s^{-1}$ before good agreement with the actual flow curve was obtained.

LOW SHEAR MIXING

The problem

During a recent research project on the development of high performance superplasticisers involving the production of concretes at two plants, problems were experienced replicating the water demand of concretes containing the same materials at the same mix proportions. In almost all cases the Plant C concretes required a higher amount of water to achieve the desired rheological properties.

A possible explanation for the observed difference in the water contents of corresponding concretes made at Plant B and Plant C is that the Plant C mixer is less energetic, i.e., the Plant C mixer does not deliver the same amount of shear energy to the concrete mix. This effect was reproduced using a paste system tested on the CSL rheometer, where the energy input could be varied using a preshear stage.

Experimental details

A Portland cement with a high tricalcium aluminate content was chosen for the test series as a worst case situation. It was dosed with a polycarboxylate superplasticiser at 0.3% sbwc with a w/c ratio of 0.375. The powder, water and polymer were mixed by hand for 30 seconds, poured into the test vessel and mixed for two minutes at a constant shear rate. This preshear stage was followed by the measuring stage where the shear rate was raised in a 20 step ramp from 0.15 to $10s^{-1}$ and then a further 20 step ramp from $10\text{-}200s^{-1}$ as before. The preshear rate was varied from 10 to $500s^{-1}$.

The flow curves showed a relatively linear region extending from a shear rate of 30 s^{-1} up to 120 s^{-1}. Regression analysis of at least 6 points in this region gave slope and intercept values which are equivalent to plastic viscosity and yield stress (Bingham model) and are useful in following the changes in the rheological behaviour of such pastes. The regression results for this test series are illustrated in Fig. 5.

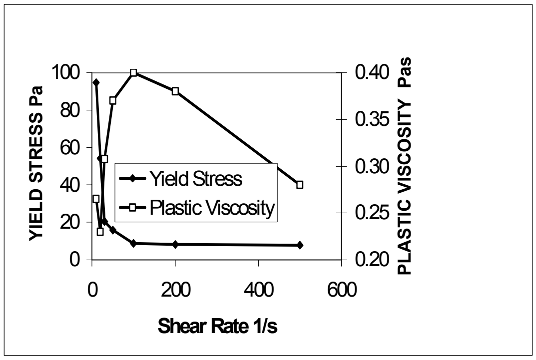

Figure 5. Effect of initial mixing shear rate on yield stress and plastic viscosity.

Practical results

At low shear rates, especially below about $30s^{-1}$, the mixing energy has a large effect on the measured yield stress. Above a shear rate of $100s^{-1}$ the effect is insignificant. In terms of measured plastic viscosity below $100s^{-1}$ increasing the shear rate increases the viscosity, while beyond this shear thinning occurs.

The concretes in Plant B and C were being produced to meet a target slump (i.e. yield stress) so the practical effects manifested themselves as differences in the water content. The mixers being used were at the lower end of the shear rate range (speed of rotation 1-2 rev/sec with and without contra-rotating paddles to give forced action in a mixer vessel of 10-20 litres capacity) where yield stress is highly sensitive to preshear rate. Thus only small differences in the energy input between the two mixers could account for the observed differences in water content.

CONCLUSIONS

The measured rheology of cement based systems is highly dependent on the previous shear history of the sample. It is possible to simulate the shear history experienced by a grout on the different stages of mixing by appropriate choice of shear conditions in a controlled stress rheometer. Differences in the shear rates in concrete mixers can explain differences in the water required to achieve a rheology target.

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