



PII S0008-8846(96)00012-9

## EFFECT OF VIBRATION ON THE RHEOLOGICAL CHARACTERISTICS OF MAGNESIA PHOSPHATE AND ORDINARY PORTLAND CEMENT SLURRIES

P. Frantzis and R. Baggott

Department of Surveying, University of Salford, Salford, UK

(Refereed)

(Received October 2, 1995; in final form January 2, 1996)

### ABSTRACT

The effects of vibration on the rheological characteristics of rapid strengthening magnesia phosphate based slurries and an ordinary Portland cement based reference slurry determined with a rotating viscometer incorporating an interrupted helical impeller are reported. The test procedure adopted comprised measuring the effect of vibration on the stress response to three repetitive shear strain rate cycles undertaken over a 15 minutes period immediately after slurry preparation. The results are assessed in the context of the use of the slurry to infiltrate a steel fibre reinforcing array. The data presented indicates that vibration does not change significantly the rheological characteristics of either type of cement. Possible explanations of these apparently anomalous observations are discussed.

### Introduction

High performance fibre reinforced cement composites are a relatively new type of composite with considerable potential for application as bridge deck overlays, security concrete and seismic resistant structural material. Composite flexural and tensile strengths can be increased by up to 10 times that of the unreinforced matrix with even greater increases in toughness. Composite fabrication by infiltration, for example into a steel fibre array, is greatly assisted by means of accompanying vibration or applied pressure. In order to optimise the composition of the slurry from the point of view of effective infiltration it is necessary to understand the process of infiltration. The rheology of the slurry, the nature of the array interstice and the contribution of additional driving forces are the three major factors determining the depth and efficiency of infiltration. The influence of vibration on the viscosity of ordinary Portland cement based slurries has not been widely reported and no work has been reported on its influence on magnesia phosphate viscosity.

The use of magnesia phosphate (1,2,3) as the binder instead of ordinary Portland cement produces useful properties one hour from casting, for example 1 hour compressive strengths of up to 25 MPa can be obtained with magnesia phosphate concretes. However the very rapid strength development is accompanied by even more rapid setting which limits the type of rheological measurement that is possible.

The purpose of the work reported in the paper was to determine the influence of vibration on the viscosity of magnesia phosphate slurries as measured with a rotating interrupted helix

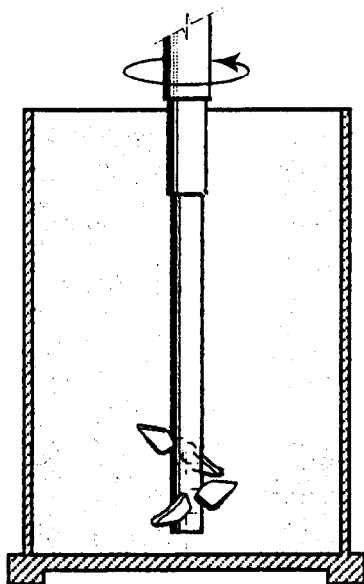


FIG. 1.

Schematic diagram of interrupted helical impeller and sample container.

viscometer. The slurry performance was compared with that of an ordinary Portland cement based slurry of proven good infiltrability into densely packed steel fibre arrays, tested under the same viscometric regime.

### Experimental Procedure

**Materials.** The two types of cement used comprised a rapid strengthening magnesia phosphate, ASR-1, supplied by FEB and an ordinary Portland cement supplied by Blue Circle Ltd. The ASR-1 contained a proportion of very fine aggregate and required the addition of a retarder to increase the setting time from 4 minutes to up to 15 minutes ie sufficient to obtain measurements. Two different retarders were assessed, both at 2% weight of the cement, namely sodium tetraborate, ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) and boric acid ( $\text{H}_3\text{BO}_3$ ). The water to solids ratio was 0.17 as recommended by the manufacturers. In the case of the OPC system a blend of 60 parts by weight of cement and 40 parts by weight of silica flour (BM500) was used at a liquid-solids ratio of 0.3. The liquid included 11% volume of superplasticiser (Conplast 430).

### Equipment and Experimental Procedure.

The viscometer used was the Haake Rotovisco RV2 with MK 500 torque measuring head (4,5). The normal rotating internal cylinder was replaced by an extended interrupted helical impeller designed for viscosity determinations in large open vessels (as shown in Fig. 1), thus enabling measurements to be made while the sample container was attached to a vibrating table. The sample container had an internal diameter of 82 mm providing a 28 mm annular gap between the extremities of the impeller blades and the internal wall and a height of 110 mm. The sample

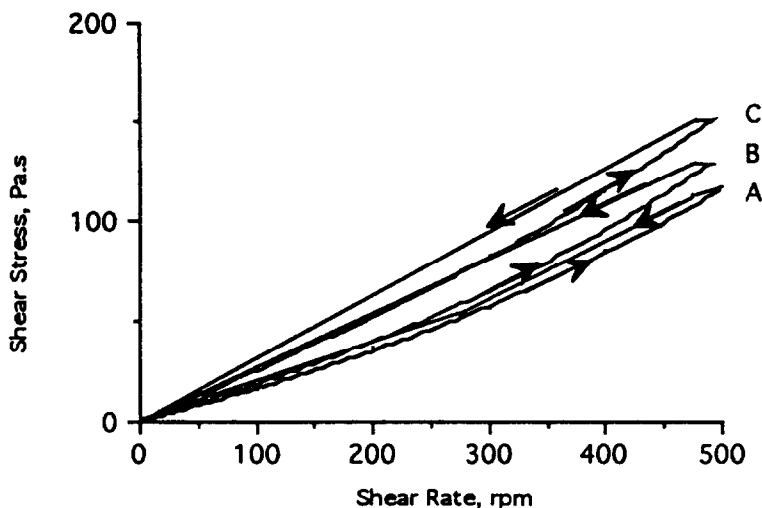


FIG. 2.

Successive hysteresis loops for the ASR-1 2% borax cement system within the 15 minutes time interval, identical with or without vibrations.

container was fixed to the surface of a vibrating table capable of vertical vibrations between 12 and 100 Hz with amplitude of vibrations peak to peak between 1.4 and 7.0 mm approximately. It was not possible to vary the amplitude at a given frequency. For every cycle the rotational speed was increased linearly to 500 rpm and then decreased linearly to zero by means of the PG 142 external programmer. The time to complete one cycle was set to 4 minutes. A Bryans 2500 x/y recorder was used to plot the loops obtained from each cycle.

Individual batches of slurry were prepared in a standard fashion. The water (including the superplasticiser in the case of the ordinary cement) was placed in the dry mixer bowl and the cement (incorporating the appropriate amount of the powder retarder or silica in the case of the ASR-1 and OPC respectively, in both instances the dry mixes having been previously thoroughly dry mixed) was added and mixed for 30 seconds at low speed followed by 1 minute at high speed. The slurry was next transferred to the sample container which was filled to the top ensuring a standard quantity of slurry. The extended interrupted helical impeller of the viscometer was then lowered into the sample container.

Each fresh slurry sample was subjected to three cycles with 0.5 minutes rest time between them. The tests commenced 1.5 minutes after mixing and the total time to complete a test, ie three cycles, was about 15 minutes. The average viscosity was calculated from the slope of the linear parts of the hysteresis loops. All tests were repeated three times using fresh samples.

## Results

**Hysteresis Loops.** Typical results of the rheology tests performed using the three cement systems either with or without the addition of external vibrations are shown in Fig. 2 and 3. These figures display the variation of shear stress with shear rate. All hysteresis loops started

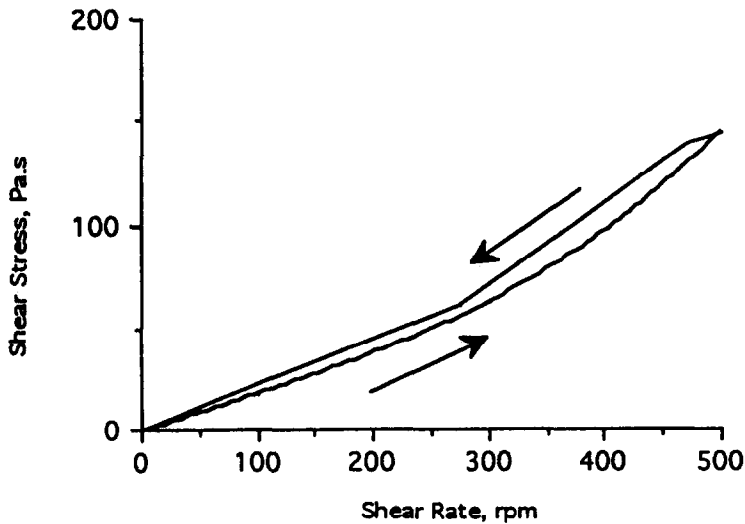


FIG. 3.

Hysteresis loop for the OPC/BM500/Conplast 430 cement system within the 15 minutes time interval, identical with or without vibrations.

and ended at the origin whether or not vibration was applied. The loops exhibited a non-linear curve of increasing slope at increasing shear rates and a linear relationship at decreasing shear rates i.e hysteresis loops were obtained with all cycles.

**Magnesia Phosphate with Borax/Boric Acid Retarder.** Fig. 2 shows the hysteresis loops obtained with time using ASR-1 with 2% addition of borax or 2% boric acid as retarder. Similar families of loops were obtained for both retarders. The stress levels at decreasing shear rates were found to be higher than those at increasing shear rates and the upward going curve was concave towards the stress axis. The downward going part of the curves was linear but the slope of the line changed at about 275 rpm in the case of loop A whereas there was no change in gradient with loops B and C. The curves from tests with vibration were essentially the same as those without.

**OPC Based Slurry.** The same three cycles were applied to the OPC/BM 500/Conplast 430 system but only one loop is shown in Fig. 2 as the three loops were identical, the stress values measured at decreasing shear rates were greater than those at increasing shear rates. The upward going curve was concave towards the stress axis. The downward going curve for decreasing shear rates was linear but the slope of the line changed at about 275 rpm. The loop was essentially the same as loop A with the borax and boric acid retarders. Again there was no major difference between curves from tests with vibration and without.

**Viscosity Values.** Average viscosity values were calculated from the slopes of the linear sections of the hysteresis loops on the chart recordings of the x/y plotter and the calibration factor of the instrument (4,5). There were only marginal differences between the values from tests with and without vibration.

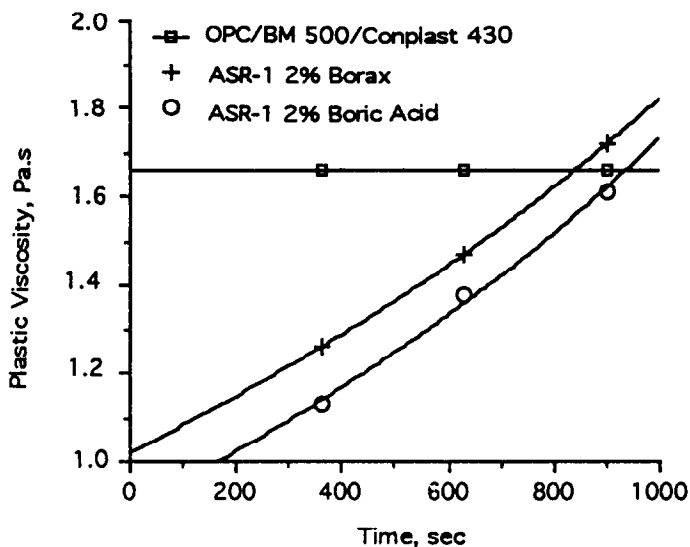


FIG. 4.  
Time dependence of viscosity without vibrations.

### Discussion

There are two aspects of the results to consider, firstly the relevance of the rheological observations regarding the effect of vibration on flow characteristics and secondly the significance of the data with reference to infiltration.

**Rheological Phenomena.** The various features of the data on tests without vibration have been referred to elsewhere (6) and only a brief summary will be made here. Overall it is clear that given the nature of the rapidly changing structure of the magnesia phosphate slurry and the imposition of substantial shear, both by the test instrument and the vibration, the data is test specific and limited in general application. However it provides useful indicators to phenomena associated with cement slurry behaviour with and without vibrations.

**Absence of Yield Stress.** The one common feature of every curve recorded was the absence of a yield stress for both magnesia phosphate and OPC with or without vibration. In the case of OPC this observation is in agreement in principle with data in the literature which shows that the presence of superplasticisers reduces the yield value to essentially zero (7,8,9,10) and in these circumstances no effect of vibration would be expected and this was confirmed across a range of vibration frequencies and amplitudes.

The present data shows no yield occurring with magnesia phosphate without vibration even as setting was approached. The expectation was that yield should have occurred eventually but it did not and it follows again that vibration would not be expected to change this whatever its frequency.

Neither type of material behaved as a Bingham solid.

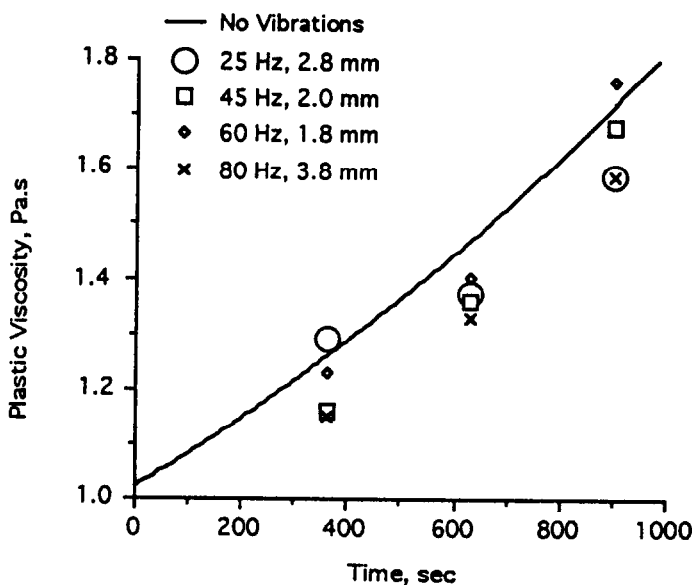


FIG. 5.  
Time dependence of viscosity with and without vibrations.

**Influence on Apparent Viscosity.** Examination of Fig. 2 and 3 indicates that to a first approximation there is a linear relationship between stress and strain rate albeit with two different gradients in some instances. In order to make quantitative comparisons within the data the average gradient of the linear regions of the various curves was used to calculate average apparent viscosity.

Fig. 4 illustrates the time dependencies of the average apparent viscosity of the cement systems without vibrations. The viscosity is plotted against time where time is measured from the completion of mixing to the completion of a shear strain cycle. It can be seen that the viscosity values of the ASR-1 systems increase with increasing time with a dependency of the form

$$\eta = A10^{kt}$$

where  $A = 1.02$  and  $k = 0.00025$  for the 2% borax and  $A = 0.9$  and  $k = 0.00028$  for the 2% boric acid systems,  $\eta$  = viscosity and  $t$  = time.

Fig. 5 and 6 illustrate the time dependencies of the average apparent viscosity of the cement systems with and without the application of external vibrations for magnesia phosphate retarded by borax and boric acid respectively. It can be seen that the results indicate a slight decrease in the viscosity values compared to those obtained without vibrations without any correlation between the frequency and amplitude of vibrations and the viscosity change. To a first approximation the same empirical relationship is applicable for all four systems.

In the case of the OPC system there was no systematic change with time over the 15 minutes period of the tests with and without vibration. The overall average viscosity for the four frequencies employed was 1.65 Pa.s compared to 1.66 Pa.s from tests without vibration, see Table 1.

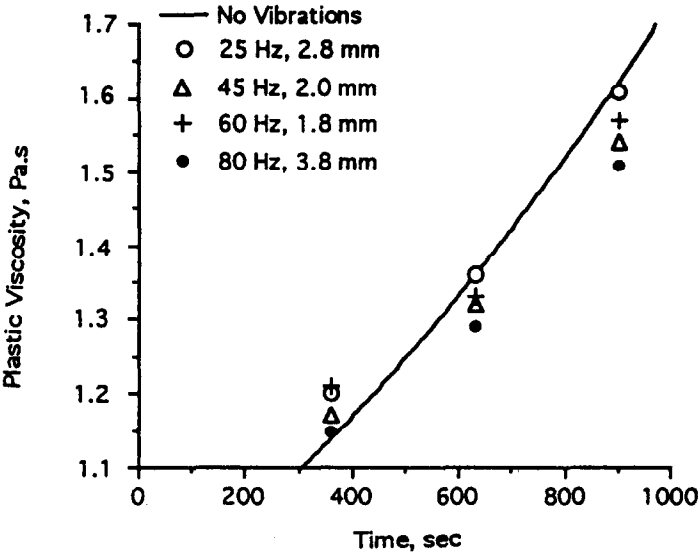


FIG. 6.  
Time dependence of viscosity with and without vibrations.

Comparison of Data with Literature Values. Although it has been common practice for many years to use vibrations for increasing the compaction of fresh concrete, the physical processes involved during vibration are still not well understood. Only a limited amount of work has been reported on the influence of vibration on the rheology of ordinary Portland cement pastes. Tattersall (11) identified a transition from non-Newtonian behaviour to Newtonian with reductions in apparent viscosity dependent upon the velocity of the vibration induced. Lasalle and Legrand (12) have reported studies on the rheological performance of various suspensions with increasing distance from a source of vibration. Borgesson and Fredrikson (13) have shown a vibration induced change to Newtonian behaviour with decreased viscosity for superplasticised cement/silica fume grouts.

TABLE 1  
Average Viscosity Values for the OPC/BM 500/Conplast 430 Cement System  
for the Four Vibrating Frequencies

Vibration frequency and peak to peak amplitude	OPC/BM500/Conplast 430 /Pas
No Vibration	1.66
25 Hz. 2.8 mm	1.50
45 Hz. 2.0 mm	1.81
60 Hz. 1.8 mm	1.59
80 Hz. 3.8 mm	1.70
Average from the four vibration frequencies	1.65

However, no significant effect of vibration on rheological properties has found in the present investigation with either the OPC/BM500/Conplast 430 cement system or the ASR-1 rapid strengthening cement. The explanation for the apparent discrepancy lies in the observed behaviour of the non-vibrated system since the vibrated data on its own is in broad agreement ie behaviour approaching that of a Newtonian fluid. The test regime adopted avoids significant static slurry time as well as imposing high rates of shear during the middle of each cycle and the choice of an interrupted helical impeller ensures effective agitation of the sampled volume, together these three factors minimise structural development until the quantity of reaction product and the magnitude of formation of structural bonding overcomes the breakdown forces of agitation.

**Implications of Results Regarding Slurry Behaviour on Infiltration.** The viscosity values of the OPC system can be considered as a minimum requirement for effective infiltration since it has been used successfully to infiltrate a fine steel fibre array of 15% fibre volume fraction (14). The viscosity values with and without vibrations of the ASR-1 borax and boric acid systems were initially considerably lower than those of the OPC system increasing with to time to a cross over point occurring at about 15 minutes after mixing beyond which the viscosity was greater than OPC. In principle therefore successful infiltration should be possible into the same arrays as infiltrated by OPC and effective fluidity can be maintained by imposed vibration to almost the onset of setting.

The results draw attention to the point made by many workers in the cement slurry rheology field that, given the inevitability of disturbance inherent in most rheometers, characterisation of flow curves should be made at shear rates appropriate for the nature of the application. The present data provides the right characteristics for infiltration assisted by vibration but the unvibrated data is not directly applicable to infiltration under simple gravitational forces.

### Conclusions

The results indicate that vibration did not influence significantly the rheological behaviour of either OPC slurries or those of magnesia phosphate. Similar viscosity values and zero yield stresses were observed with and without the addition of external vertical vibrations at various frequencies and amplitudes. The results provide clear evidence of the effectiveness of the test procedure without vibrations in minimising the contribution of structural development thereby providing base line rheological data for characterising cementitious slurries.

It was also shown that the early viscosity of the rapid strengthening cement based slurry was lower than that of a reference OPC based slurry and hence should effectively infiltrate the same fibre array of appropriate thickness.

Effective fluidity can be maintained for general infiltration to almost the onset of setting by means of vibration and an empirical relationship based on the experimental observations enables the time available for successful infiltration to be determined.

### Acknowledgments

The authors gratefully acknowledge the financial support of the Engineering and Physical Science Research Council. We are also grateful to Prof. P.F.G. Banfill for his professional



advice and assistance. We also like to thank Mr. P.B. Unsworth, Senior Technician, and Mr. I. Hanbridge, Technician, of Salford University.

### References

1. B. El-Jazairi, *Concrete* **16** (9), (1982).
2. D. F. Orchard, *Concrete Technology*, Applied Science Publishers Ltd., **1**, 15 (1979).
3. A. Zurz, I. Odler and B. Dettki, *Symposium Proceedings of the Materials Research Society*, **179**, 69 (1991).
4. P.F.G. Banfill and D.C. Saunders, *Cement and Concrete Research*, **11**, 363 (1981).
5. J.I. Bhatti and P.F.G. Banfill, *Cement and Concrete Research*, **12**, 69 (1982).
6. P. Frantzis and R. Baggott, to be published.
7. D.M. Roy and K. Asaga, *Cement and Concrete Research*, **9**, 731 (1979).
8. K. Asaga and D.M. Roy, *Cement and Concrete Research*, **10**, 287 (1980).
9. D.M. Roy and K. Asaga, *Cement and Concrete Research*, **10**, 387 (1980).
10. P.F.G. Banfill, *Magazine of Concrete Research*, **33** (114), 37 (1981).
11. G.H. Tattersall, *Workability and Quality Control of Concrete*, Spon London, 1991.
12. A. Lasalle and C. Legrand, *Materiaux et Constructions*, **13**, 115 (1980).
13. L. Borgesson and A. Fredriksson, *Proceedings of the International Conference of the University of Liverpool*, edited by Banfill, P.F.G., March 16–29 (1990).
14. A.E.S. Abdel-Monem, Ph.D Thesis, University of Salford, 1990.