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Rheology of low carbon fibre content reinforced cement mortar

P.F.G. Banfill *, G. Starrs, G. Derruau, W.J. McCarter, T.M. Chrisp

School of the Built Environment, Heriot-Watt University, Edinburgh EH14 4AS, UK

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Abstract

It is recognised that the addition of carbon fibres to a brittle cement matrix results in a less dense composite with enhanced ductility, improved impact resistance and increased toughness. In addition, the reinforcing effect of fibres in the cement often produces superior flexural strength and marked improvements in post-cracking behaviour. Further, carbon fibres influence the electrical properties of the composite which could, potentially, make it a *smart* material, with a range of applications. Despite attention directed towards the mechanical and electrical properties of carbon fibre reinforced cement (CFRC), there is a dearth of information of the influence of fibre additions on the rheological properties of the resulting composite. To this end, this paper describes an investigation using the Viskomat NT into the influence of carbon fibre additions (fibre length in the range 3–12 mm and volume in the range 0–0.5%) on the rheological properties of CFRC. Within the limitations of the instrument and testing procedure it is shown that CFRC's conform to the Bingham model: increasing fibre volume and fibre length increase both the yield stress and plastic viscosity.

Keywords: Carbon fibre; Portland cement; Mortars; Rheology

1. Introduction

Since the 1960s extensive research has been undertaken on fibre reinforced cement matrices, and they are now firmly established as construction materials. Fibre additions offer a convenient and practical means of achieving improvements in many of the mechanical properties of plain cement and concrete such as enhanced toughness, fatigue resistance, impact resistance, flexural strength, reduced creep and shrinkage; marked improvements in the post-cracking behaviour have also been reported. High performance and ultra-high performance fibre reinforced cement matrices represent a new generation of tough, durable construction materials for the 21st century now being actively researched [1,2].

Although glass, polypropylene and steel fibres have been extensively used in cement matrices, this paper focuses on the addition of carbon fibres. Normal concrete is a poor conductor of electricity, particularly under dry conditions. However, since carbon fibres are electrically conductive, it is possible to use both the ac and dc electrical properties in a number of functional applications, whilst maintaining the enhanced mechanical properties of the material afforded by the inclusion of carbon fibres. The electrical response of carbon fibre reinforced cement (CFRC) could allow such a composite to be exploited as a material with self-monitoring capabilities with respect to deformation and damage [3–6]; for example, as CFRC is deformed or stressed the contact between the fibre and the cement matrix will be affected by crack generation and propagation which, in turn, would affect its electrical response. In other words, the inclusion of carbon fibres could make the material intrinsically *smart* as it senses and reacts to internal and external changes.

CFRC could open up a wide range of self-sensing applications in the detection and (real-time) monitoring of cracking, vibration or fatigue in concrete structures (e.g. bridges, pressure vessels) [7]. Other areas where CFRC could find practical application include: electrically conductive cementitious overlays for heating applications [8,9]; as an effective, durable low-resistance grounding-system

* Corresponding author. Tel.: +44 131 451 4648.
E-mail address: P.F.G.Banfill@sbe.hw.ac.uk (P.F.G. Banfill).

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[10] for earthing electrical installations or preventing static electricity build-up; as an overlay in cathodic protection systems providing a more uniform current distribution, or in structures where electromagnetic shielding or screening is required [11].

Whilst considerable work has now been published on the electrical properties of CFRC matrices, there is a dearth of information on their rheological properties. To fully realise its use as a smart material, CFRC must be capable of being moulded, compacted, extruded or otherwise formed. Additionally, it is recognised that there is a lower limiting concentration of the conductive fibre which needs to be exceeded in order to confer the desired electrical properties on CFRC, referred to as the percolation threshold, and values of 0.3–0.5% fibre volume concentration have been reported [4,12–16]. Appropriate test methods, capable of testing the rheological properties of CFRC up to and above this threshold are required, and it is this aspect which is addressed in the current paper. This will, ultimately, lead to the development of practical mix formulations.

2. Rheology of cement-based materials

There is considerable evidence that the rheology of cement-based materials conforms to the Bingham model [17]:

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

The material is an elastic solid at shear stress $\tau < \tau_0$, (the yield stress), but, at higher stresses, begins to flow. μ is termed the plastic viscosity and $\dot{\gamma}$ the shear rate. The yield stress is a consequence of interparticle forces and can be determined from Eq. (1) by extrapolation, or direct measurement in an appropriate instrument.

In principle, two measurements at different shear rates or shear stresses are the minimum required to characterise a Bingham material [18] and this underpins this study of the rheology of CFRC. Single point tests such as slump or slump-flow are limited because there are an infinite number of combinations of yield stress and plastic viscosity that can produce the same result (effectively a measurement of shear stress at a particular shear rate, or vice versa).

Even though single point tests are limited, previous studies with CFRCs indicate that there is a negative (linear) correlation between the flow spread and the fibre content (expressed as specific surface area) of the fibre reinforced paste [19]. Higher fibre contents require a greater fractional volume of cement paste in the mortar for a given level of workability [20]. Therefore, the maximum amount of fibres that can be uniformly distributed without excessive loss of workability is larger for cement paste and mortar than for concrete [21]. The specific surface of fibre per unit volume of mixture is influenced by the combination of fibre aspect ratio and fibre length [22].

Workability may also be influenced by the fact that carbon fibre tows, consisting of bundled filaments held weakly together by 'size' (a viscous coating applied during manufacture) must disperse into individual filaments during the production of the composite. If this fails to occur, the reinforcing unit may not be a single filament dispersed in a matrix, but a bundle, with each filament having freedom of movement relative to the others [23]. The consequence of this in the hardened state has been described by Bartos as 'telescopic' behaviour, where, under stress, the outermost filaments are strongly bonded to the matrix and may fracture, whilst only the sliding and pullout behaviour shown by the inner filaments provides energy absorption and impact resistance [24]. The use of a water-soluble size generally overcomes this problem. Finally, water-reducing or superplasticising admixtures are also required to ensure adequate workability at realistic water contents.

Fresh cement-based materials undergo structural breakdown during shear [17] and the measured data are sensitive to the shear history of the sample, which includes the test itself. Breakdown manifests itself in two ways:

(1) The material breaks down during the test and hysteresis loops are obtained whereby the down-curve falls to lower stress values than the up-curve and successive hysteresis loops fall to progressively lower values of torque in a rotational viscometer [25].
(2) The material's yield stress decreases, together with reductions in the rest of the curve, as the total amount of shear energy experienced by the sample increases. This can be due to mechanical mixing [26] and the effect can be quantified in terms of the total shear energy received by the sample prior to the test [27,28].

The feasibility of a coaxial cylinder viscometer for mortar testing has been demonstrated [29], but the small-scale mixer geometry of the Viskomat NT [30,31], is much more convenient. In this instrument, as the cylindrical sample container rotates (Fig. 1), the mortar flows through the blades of the impeller and exerts a torque which is measured by a transducer. A series of data points of torque (T) and rotational speed (N) are recorded by computer and output as a spreadsheet file. For a Bingham material T and N are related by the 'straight-line' equation:

$$T = g + hN \quad (2)$$

In this equation, g (the intercept) is proportional to yield stress and h (the gradient) is proportional to plastic viscosity of the material. The constants of proportionality can be determined by a simple calibration procedure [32]. The Viskomat has been established as a sensitive and fundamentally sound test instrument [30], and has been used to investigate the effect of mix composition, admixtures and fine particles on the rheology of mortar [33].

On the basis of this previous work it would be expected that the rheology of CFRC would show increasing yield stress and plastic viscosity with increasing fibre concentration and fibre length and that structural breakdown might

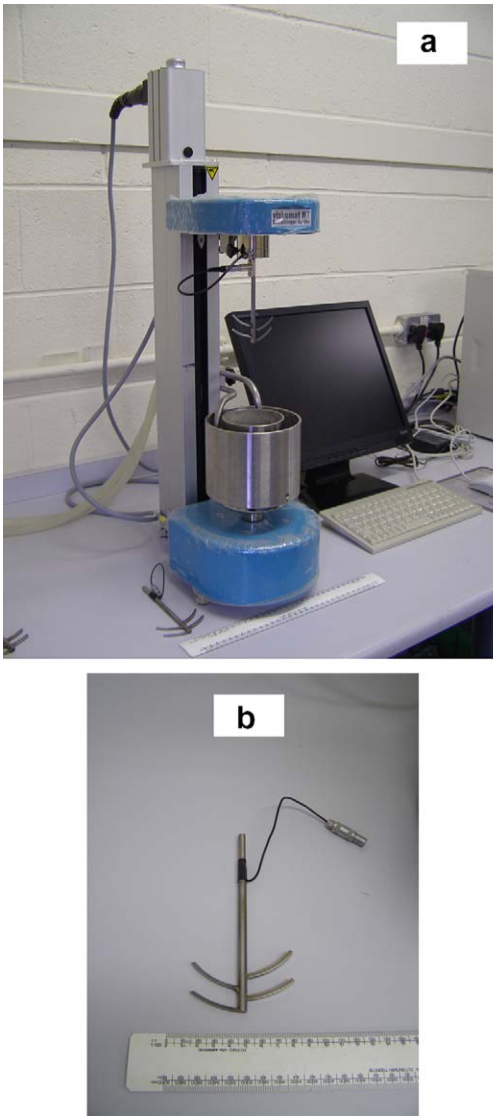

Fig. 1. (a) The Viskomat measuring system; and (b) impeller used in the experimental programme.

be significant. A particular interest would be whether the rheological parameters of cementitious systems at fibre concentrations at or near the percolation threshold fibre concentration could be obtained. Such work would provide the basic information necessary to develop practical mix formulations for CRFC mortars and concretes.

3. Experimental

The experimental programme focussed on an investigation of the rheological properties of CFRC mortars in particular, the influence of fibre dosage and length. A Viskomat NT was used to evaluate the rheological parameters – yield stress and plastic viscosity – of the CFRC.

3.1. Materials

Samples were made with CEM I Portland cement and a siliceous sand of maximum particle size 2 mm (Table 1).

Table 1
Particle size distribution of the sand

Sieve aperture (mm)	2.0	1.18	0.6	0.3	0.15	0.075
% Passing	100	78	61	30	6	1

Water-reducing plasticizer (Conplast P515) was used in all mixes at a dosage of 0.6% (by weight of cement). The carbon fibre used throughout the experimental programme was SIGRAFIL C®. The fibre had a diameter of 7.5 μm and a density of 1800 kg/m^3. Its electrical conductivity was 62,500 S/m, representing a resistivity of 16 μΩ m, and it contained glycerine as a water soluble size (at approximately 4% by weight).

A standard mortar with a sand/cement ratio of 0.5 (by mass) was used in all tests using

(i) water/cement (w/c) ratios of 0.4, 0.45 and 0.5,
(ii) fibre lengths of 3, 6, 9 and 12 mm; and,
(iii) fibre volumes of 0.15%, 0.25%, 0.35% and 0.5% (by volume of sample).

Fibre aspect ratio was not used as an explicit variable in the programme but it can be noted that it increases with fibre length over a fourfold range.

3.2. Experimental procedure

The volume fractions noted above were produced by sequential addition of fibres to a base mortar containing 0.15% fibre up to the maximum value of 0.5%. Using a BS EN196 mortar mixer [34], the sand, cement, fibres and water containing the appropriate quantity of plasticizer were placed in the bowl in that order and mixed at 140 rev/min for 5–8 min. This base mortar was tested in the Viskomat; after testing, fibre was added and mixed (using a Viskomat impeller mounted in the chuck of an electric screwdriver, rotating at 400 rev/min for 60 s) to create 0.25% fibre volume. The resulting mortar was subsequently tested in the Viskomat and the process repeated until all fibre dosages were tested. Strict timekeeping in this procedure ensured that the times at which the up-curve started were 5, 14, 23 and 32 min. An unavoidable consequence of this procedure is that rheological effects due to increasing fibre content and increasing age of the mortar are inseparable, but since the influence of the latter is well known the effect of fibre content can be inferred.

The Viskomat can exert a maximum torque of 200 N mm and is fitted with a torque cut-out device. Stiff mixes, expected to go over this torque, used test cycle 1 (Fig. 2): speed increased to 200 rev/min in 40 s, holding this speed for 40 s and then decreasing to 40 rev/min by 4 min and zero by 7 min. The impeller was progressively inserted into the sample during the up-curve to prevent torque overload, even though this sacrifices the detail of the structural breakdown. Less stiff mixes used test cycle 2 (Fig. 2): speed increased to 10 rev/min in 3 min, followed by an increase to

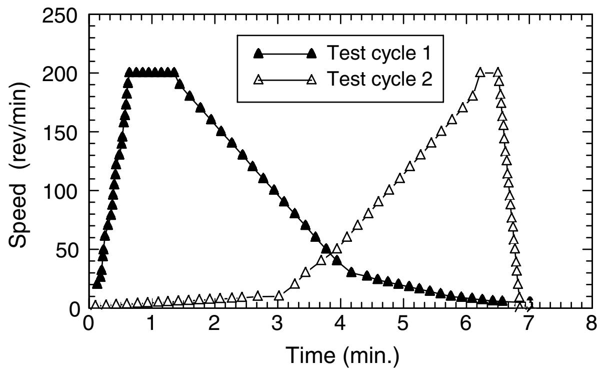

Fig. 2. Test cycles showing the variation of speed with time.

200 rev/min at 6 min and a rapid descent to zero by 7 min. In every case, the down-curve was used for the calculating the Bingham parameters, while only qualitative assessments of the structural breakdown can be gained from the shape of the loop. The results were consistent between the down-curves determined according to both test cycles.

4. Results and discussion

Fig. 3 shows a typical set of torque–speed relationships for a standard mortar with w/c = 0.45 containing 6 mm fibres at a dosage of 0.35% by volume. It can be clearly seen that the mortar conforms to the Bingham model with the down-curve approximating to a straight line where flow is fully developed above approximately 50 rev/min but it is not superimposed on the up-curve and this would confirm that some structural breakdown has occurred. The best straight line can be fitted to Eq. (2) through the points on the down-curve after removing the lowest values where flow is not fully developed. Values of $g = 43$ N mm and $h = 6.4$ N mm s, with a correlation coefficient of 0.988 (based on 20 points), are obtained for this measurement. The constants of proportionality applicable to Eq. (2) have not been determined for the geometry used in this instrument, but previous work [30] suggests that these values correspond to a yield stress of 350 Pa and a plastic viscosity of 5 Pa s. However, for the purposes of the current study, all results are presented in terms of g and h values obtained directly from the experimental data.

In order to establish the repeatability of the test procedures, this test above was replicated three times, giving g values of 43.4, 40.0 and 44.1 N mm and h values of 6.4, 6.7 and 7.4 N mm s. Hence, the error on a single determination is approximately ±9% on g and ±14% on h and the results of the main programme should be considered in this light.

Fig. 4(a)–(c) shows the effect of increasing fibre volume content on g for water/cement ratio 0.5, 0.45 and 0.4 mortars, respectively, while Fig. 5(a)–(c) shows the effect on h for the same mortars. It is clear that both g and h increase with increasing fibre content, increasing fibre length and decreasing water/cement ratio. The histograms in Figs. 6 and 7 convey an impression of the contour surfaces that these effects produce.

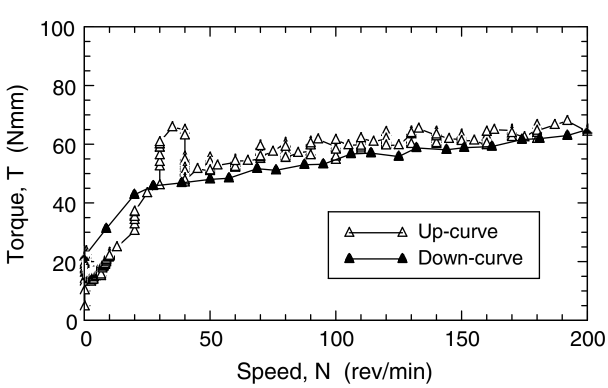

Fig. 3. A typical flow curve output from the Viskomat (0.35% of 6 mm fibre at 0.45 water/cement ratio).

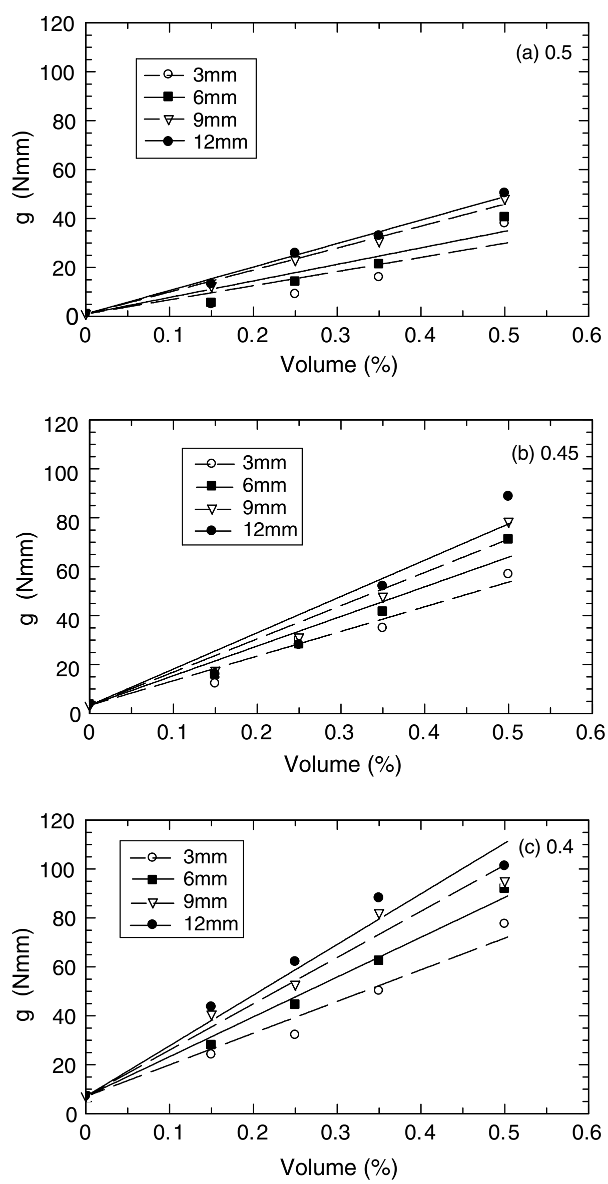

Fig. 4. The effect of fibre volume on g at (a) 0.5, (b) 0.45 and (c) 0.4 water/cement ratios.

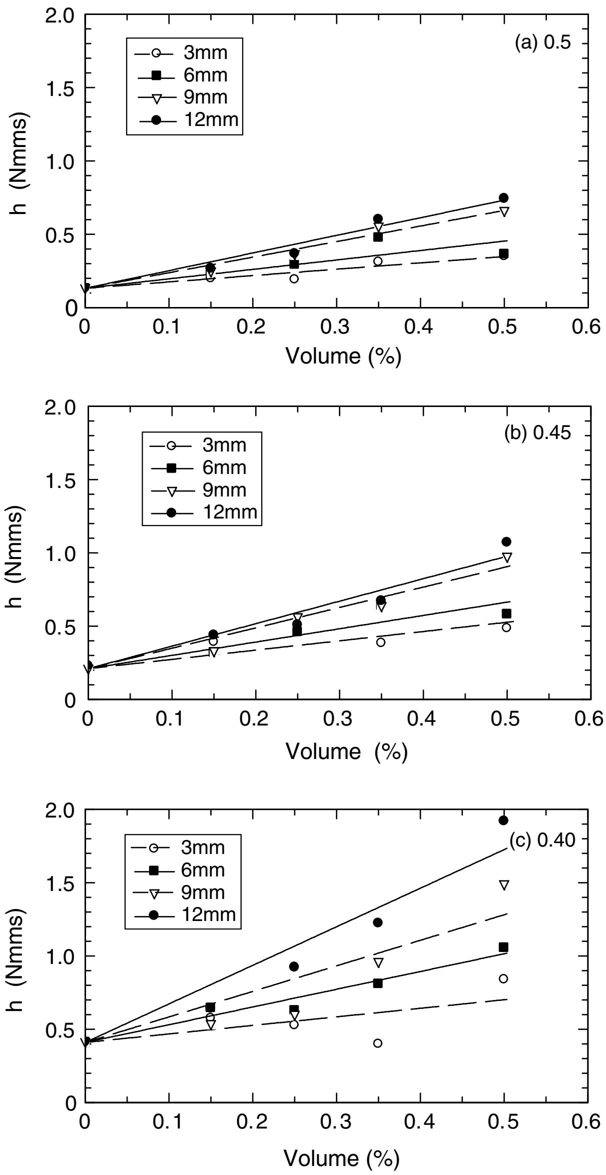

Fig. 5. The effect of fibre volume on *h* at (a) 0.5, (b) 0.45 and (c) 0.4 water/cement ratios.

Fig. 8(a) and (b) presents graphs of *g* versus *h*, which summarise the trends relating to the influence of water/cement ratio, fibre volume content and fibre length. Using this form of presentation makes it relatively easy to identify the range of sensitivity of the instrument. Noting the estimate of reproducibility of testing already mentioned, the trends for 9 and 12 mm fibres are smoother and within experimental error, whereas the data for shorter fibres are more scattered and individual points stray outside the limits of ±9% and ±14% for *g* and *h*, respectively. Nevertheless, the results indicate that decreasing the water/cement ratio, increasing the fibre volume and increasing the fibre length increase both *g* and *h*; furthermore, *g* increases more steeply than *h* up to values of *g* approximately 80 N mm while, above this point, *h* increases more steeply than *g*. This is shown schematically in Fig. 9, which also shows the limit to capacity of the Viskomat discussed below.

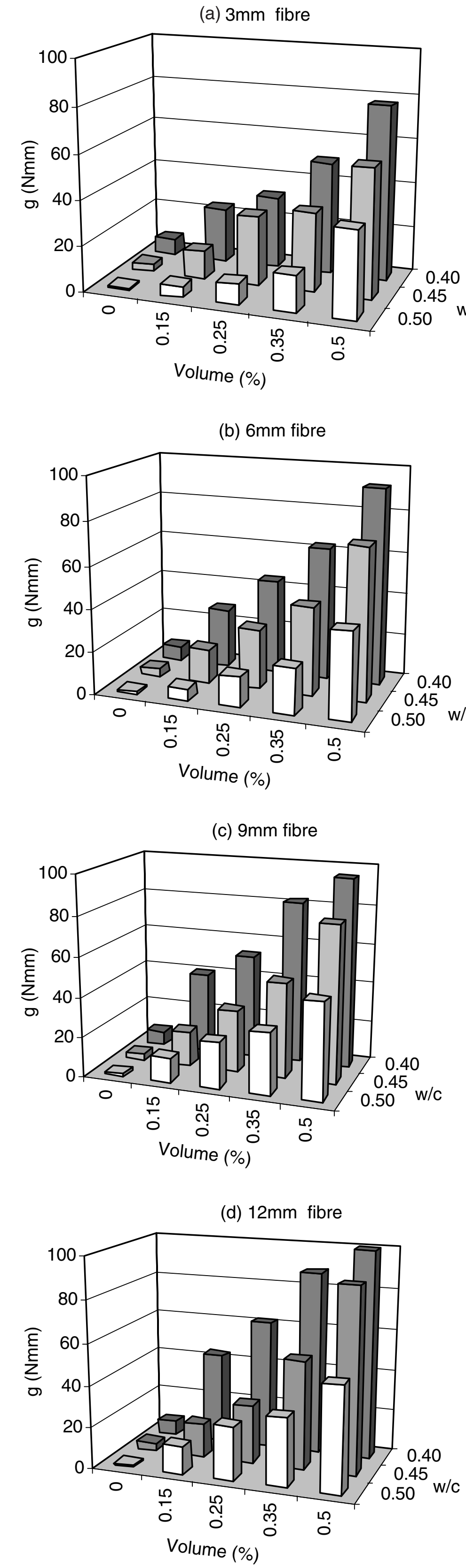

Fig. 6. Summary of results for *g*.

Once the torque overload cut-out on the Viskomat activates at 200 N mm the test in progress is aborted and the

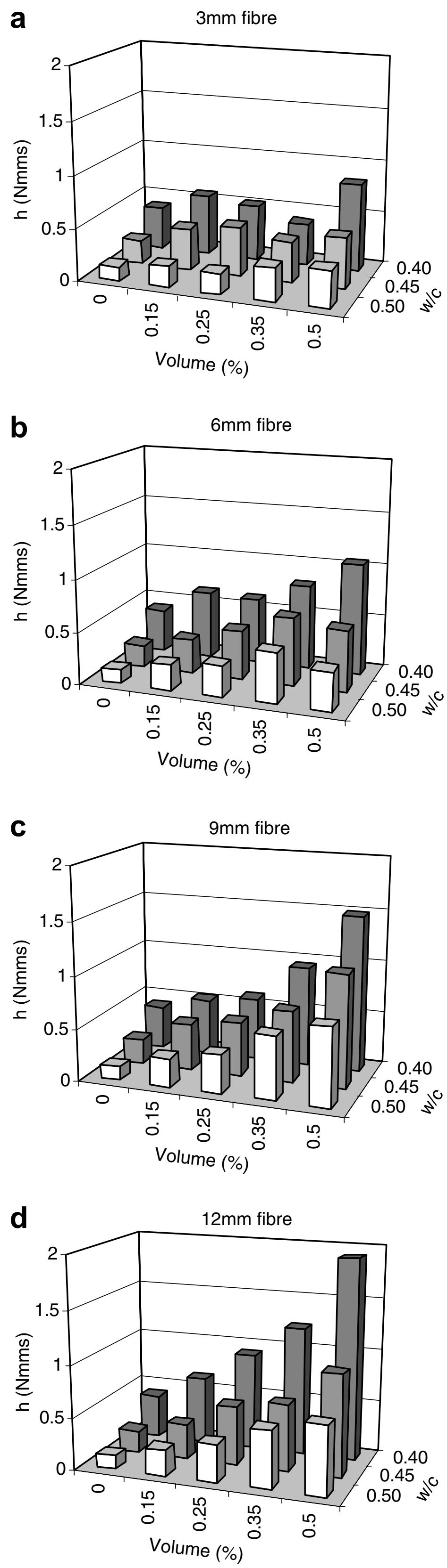

Fig. 7. Summary of results for *h*.

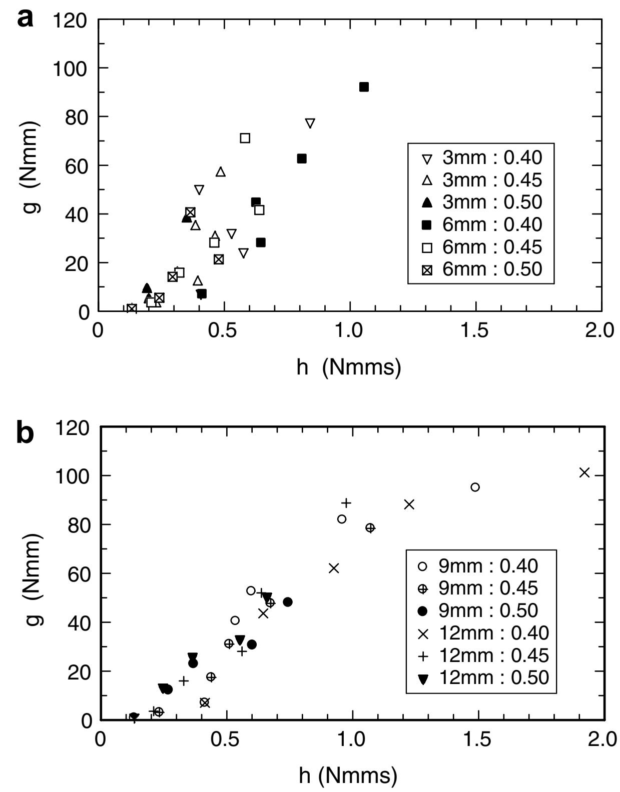

Fig. 8. Variation of *g* and *h* for mortars containing (a) 3 mm and 6 mm fibres, and (b) 9 mm and 12 mm fibres.

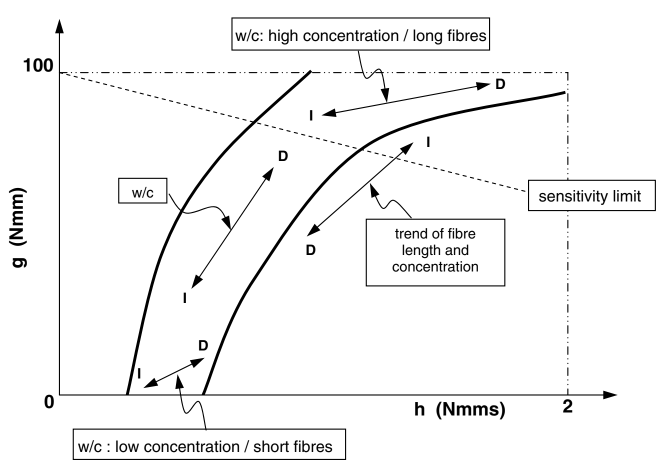

Fig. 9. Summary of the influence of composition on rheology of carbon fibre cement mortar. The arrows denote the direction of the change in rheology in response to an increase (I) or decrease (D) in the parameter indicated.

data are lost. The structural breakdown during a test means that this overload is most likely to happen during the up-curve. The decrease in torque from up-curve to down-curve may be as much as 50% (see Fig. 10) hence, using the results obtained from the down-curve to predict the risk of overload requires an estimate of the conditions giving a torque of approximately half the maximum value. Using Eq. (2), it is a simple matter to identify values of *g* and *h* such that *T* in the down-curve never exceeds

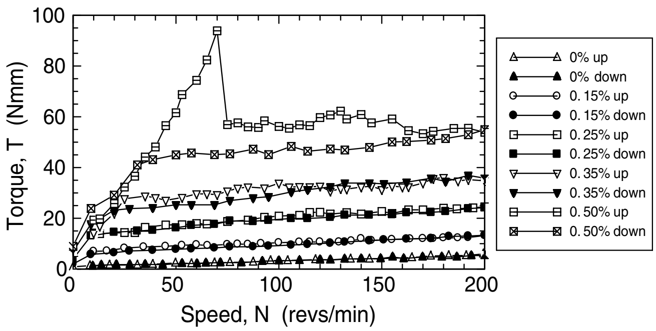

Fig. 10. Variation of hysteresis loop width with fibre content (6 mm fibre at 0.5 water/cement ratio).

100 Nmm during the test over the speed range $N = 0$–3.33 rev/s. The governing inequality is

$$100 \leqslant g + 3.33h \quad \text{or} \quad g \leqslant 100 - 3.33h \tag{3}$$

This line is shown in Fig. 9 and implies that all combinations of g and h falling below the line will be capable of testing without risk of torque overload, whilst those above and to the right of the line will possibly cause torque overload (depending on the width of the hysteresis loop). The high fibre volume, low water/cement ratio mortars, for which test cycle 2 was used, all gave results in the region above and to the right of the line and hence are consistent with this approach. It should be noted, however, that this in no way invalidates the results reported in the region above the line – the fact that they were obtained simply means that the up-curve did not exceed the torque limit during the test because the hysteresis loop was not as wide as the 50% value would suggest. This implies that the 50% used to give Eq. (3) gives a conservative estimate of the capacity of the instrument.

As detailed in Section 1, percolation thresholds for electrical conduction in CRFC are generally 0.3–0.5% carbon fibre (by volume), for fibre lengths in the range 3–15 mm [15,16]. These thresholds may need to be exceeded to ensure that the material can develop 'smart' properties. The results have shown that the rheometer is operating at the limit of its capacity at, or around, 0.5% fibre volume; if higher fibre volumes are required it would then become necessary to redesign the testing configuration to ensure that the required torque can be exerted. Clearly a higher torque range could be used but this would require a different torque sensor to be designed by the manufacturer; alternatively, lower speeds could be used, but this is unreliable because overload usually occurs during the up-curve and at low speeds when the initial structural breakdown occurs. This leaves only the possibility of adjustments to the geometry of the impeller to reduce the resistance to flow and a further investigation is currently underway in this respect.

Finally, based on previous studies [29], structural breakdown would not normally be expected under the test conditions used in the work presented. However, closer inspection of the flow curves reveal a pattern of increasingly significant structural breakdown, made manifest by wider hysteresis loops, at higher fibre concentrations (see Fig. 10 for 6 mm fibres). Further refinements to the test procedure may be able to minimise this apparent structural breakdown.

5. Conclusion

It is feasible to use the current rheometer to evaluate the rheological parameters of CFRC containing fine aggregate up to 2 mm maximum size; however, the current instrument is operating at its limit at 0.5% fibre volume and would need to be modified to extend its range to higher fibre volumes.

It is shown that, at fibre concentrations within the range 0–0.5%, CFRC's conform to the Bingham model, exhibiting a yield stress and plastic viscosity. Increasing fibre volume concentration and fibre length increases both the yield stress and the plastic viscosity. These effects tend to be additive along a broad curve on the yield stress versus plastic viscosity plot. This curve shows a trend towards a more rapid increase in plastic viscosity than in yield stress at higher fibre volume, increasing fibre length and lower water–cement ratio. Associated with this behaviour is evidence of structural breakdown during mixing before the test and during the test itself.

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