Thixotropic Behavior of Fresh Cement Pastes from Inclined Plane Flow Measurements

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Abstract:
We show that the rheological characteristics of a fresh cement paste can be determined from inclined plane tests. The apparent flow curve measured from inclined plane flows coincides with the apparent rheogram from classical rheometer tests and the flow curve obtained from local Couette flow measurements with magnetic resonance imaging (MRI). In order to describe the thixotropic properties of these fluids we suggest to use a simple model, the four parameters of which may be determined from specific, practical, inclined plane experiments.

Zusammenfassung:
Wir zeigen, dass sich die rheologischen Eigenschaften frischen Zements aus Experimenten an einer schiefen Ebene bestimmen lassen. Die so gemessene scheinbare Fließkurve stimmt sowohl mit dem aus klassischen Rheometrie-Versuchen erhaltenen Rheogrammen überein, als auch mit der Fließkurve, die mittels Magnetischer Resonanz Mikro-Bildgebung (magnetic resonance imaging (MRI) in lokaler Couette-Strömung erhalten wird. Um die thixotropen Eigenschaften des Zements zu beschreiben, schlagen wir ein einfaches Modell vor, dessen vier Parameter sich in spezifischen, praktischen Experimenten an einer schiefen Ebene bestimmen lassen.

Résumé:
Au cours de cette étude, nous montrons que les propriétés de thixotropie d'une pâte de ciment peuvent être déterminées à partir d'essais sur plan incliné. Les rhéogrammes apparents obtenus à partir d'écoulements sur plan incliné présentent une bonne concordance avec ceux provenant d'essais rhéométriques classiques ainsi qu'avec les rhéogrammes locaux déterminés avec des mesures de vélocimétrie d'imagerie par résonance magnétique (IRM). De plus, les quatre paramètres du modèle de thixotropie utilisé ici peuvent être estimés par l'intermédiaire d'essais pratiques sur plan incliné.

Key words: rheology, cement paste, thixotropy, inclined plane, model

1 INTRODUCTION

In general, viscometric flows (i.e. constant history of the strain rate) are obtained by confining a fluid within different geometries under specific boundary conditions (torque, rotation velocity). For fresh concrete one typically uses geometries such as Couette, vane or parallel disks, with wide gaps appropriate for coarse suspensions [1, 2]. The theoretical background for such flows may be found in Coleman et al. [3] and Bird et al. [4]. Besides, inclined plane flows can be used to determine rheological properties of pasty materials. Indeed uniform, steady flows over an inclined plane are viscometric flows and the thickness of the critical thickness, h_c, for fluid stoppage or incipient flow is directly related to the fluid yield stress, τ_c, through $\tau_c = \rho g h_c \sin\beta$, where ρ is the fluid density, g the gravity, and β the plane slope. This technique is particularly appropriate for determining the yield stress of coarse concentrations [5 - 9] as one may arbitrarily fit the boundary conditions (slope angle) so as to get a critical thickness much larger that the largest particle size for the continuum assumption to be valid. In addition, for the simple relation between the yield stress and the flow characteristics to be valid, the sample thickness must be much smaller than its spreading extent

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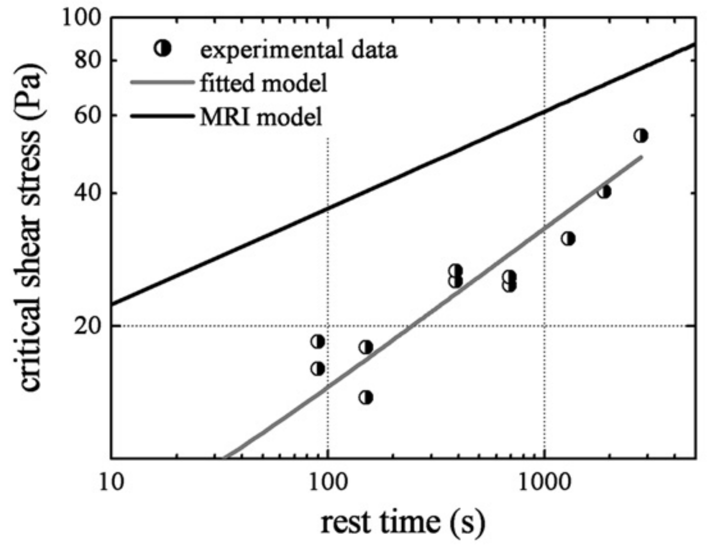

Figure 11: Critical shear stress as a function of the time of rest. Comparison with the predictions of the model with parameters values obtained from MRI measurements [20] (n = 1.22, η_o = 0.269 Pas, θ = 0.15 s, and α = 0.203) and with newly fitted parameters values (n = 1.36, θ = 2.39 s, and η_o/α = 11.54).

sequence, according to Eq. 9 the apparent yield stress of the material is proportional to the time spent at rest. Thus the cement paste is not a simple yield stress fluid which may be characterized with the help of a Bingham, a Herschel-Bulkley or a Casson models, the material restructuration implies that the material yield stress is not unique. In Figure 11 we plotted the theoretical curve predicted from Eq. 10 coupled with Eq. 11 with the parameters values of our simple thixotropic model and deduced from independent MRI experiments with the same material [20] (i.e. n = 1.22, η_o = 0.269 Pas, θ = 0.15 s, and α = 0.203). The theoretical curve is not situated exactly within the experimental data cloud, it is significantly outside and at higher stress levels. Remark in this model the parameters of the structuration kinetics are similar in flow and rest regime. With our procedure (determination of the parameters from MRI measurements after changes in rotation velocity) we in fact focus on the flow characteristics in the liquid regime. Thus we can suggest that other parameters (in fact modifying the model to take into account a true solid regime would be ideally more appropriate) of the model, and in particular concerning the structuration kinetics, might give predictions in better agreement with our data. This is effectively the case, as may be seen in Figure 11. In that case the time θ takes a value sixteen times and the exponent n is only slightly larger than the values of the initial fit. So it seems that our model cannot correctly represent both the structuration at rest and the flow properties of our fresh cement paste.

In fact, our finding of a linear structuration of the material as a function of time is valid only in a limited range of time. It is likely that we would find some kind of logarithmic (or decreasing exponential) variation over a wider range of time. Moreover we have shown that some irreversible aging effects can occur after some time with such a cement paste [20], effects that are not taken into account in our model, and neglected in our experiments. The refinements of the model these effects would require are much too complex with regards to our effective understanding of the problem. In this context the main advantage of this theoretical approach (i.e. with a simple model) is to have a model with only four parameters, which proves useful in practice. Indeed, from critical angle measurements we can determine θ, n, and η_o/α. Then from rheometrical tests we can get the viscosity η_o as it corresponds to the apparent viscosity at high shear rates. Thus from very simple measurements we can already have a good estimate of the thixotropic properties of fresh cement pastes and predict some of their flow characteristics under various (steady or transient) flow conditions.

5 CONCLUSION

From inclined plane measurements we showed that cement pastes exhibit a yielding behavior which differs from that predicted by usual yield stress models. Inclined plane "static tests" show that the apparent yield stress depends on the time spent at rest before the test. A simple thixotropic model, with only four parameters, has been used to represent the experimental data. Theoretical predictions and experimental data are in relatively good agreement although various effects may have affected this comparison (large uncertainty on some measurements, aging effects not taken into account in the model). Also the apparent rheograms deduced from inclined plane "dynamic test" are in good agreement with those obtained from conventional rheometry. Finally, a simple and practical method, based on the analysis of static tests, is described which makes it possible to determine the parameter values of the model. Thus from very simple measurements we can already have a good estimate of the thixotropic properties of fresh cement pastes and predict some of their flow characteristics under various (steady or transient) flow conditions.

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