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Steady and transient flow behaviour of fresh cement pastes

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Abstract

Fresh cement pastes behave as non-Newtonian viscous fluids. During steady flow, their apparent viscosity depends on the applied strain rate. During transient flow, the apparent viscosity is a function of time. In this work, a thixotropic model is presented. Its four parameters are identified experimentally for a tested cement paste using coaxial viscometer test. Viscometer flow simulations are then carried out. The model proves to be able to predict the trends of the fresh behaviour of cement pastes in various flow situations.

Keywords: Rheology; Cement paste; Thixotropy

1. Introduction

Cement-based suspensions, such as cement pastes and grouts, are colloidal suspensions consisting of fine particles dispersed in a liquid. Interactions among the particles may lead to microstructures formation in the suspension at rest. Depending on how the structure responds to an applied shear stress, one can observe different types of macroscopic flow behaviour, such as yield stress behaviour, thixotropy and visco-elasticity [1–3]. According to the "dictionnaire de Rhéologie" [4], shear thinning bodies are said to be thixotropic if (i) after a long rest when a shear stress or strain rate is applied suddenly and then held constant, the apparent viscosity is a diminishing function of the time of flow; (ii) the body recovers its initial state following a long enough interval after the cessation of the flow. This time-dependant decrease in the viscosity may be explained by a reversible change of the fluid microstructure during shear. In the absence of shear, the damaged structure rebuilds. Thus, yield stress or shear-thinning behaviour might be considered as a particular case of thixotropy where both structural breakdowns under shear and structure rebuilding take place simultaneously. Although it is still often considered as a single physical parameter of the fluid in the sake of simplicity [5,6], the yield stress as a true property of a suspension is one controversial subject. Numerous experimental methods coexist (Couette Viscometer test, Vane test, etc.) for different measurements (dynamic yield stress, static yield stress, equilibrium yield stress, etc.). Barnes [7–9] has gathered most of this literature.

A large number of experimental and theoretical studies of the rheological characteristics of fresh cement pastes have been published. In the case of steady state flow, several non-Newtonian models have been proposed and tested with success. However, in the case of transient flow, the attempts to quantify and predict the behaviour of cement pastes are not as numerous.

This paper is organised as follows: we start by reminding the existing literature about the steady state and transient behaviour of fresh cement pastes. Then, we present the thixotropic model used in this work. Its parameters are identified using experimental Couette viscometer results obtained for one cement paste. Finally, Couette viscometer simulations are carried out. They show the influence of the strain rate history on the shear stress/strain rate curve and are compared to experimental results.

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2. A thixotropic model

2.1. Literature study and proposed model

In this work, the aging phenomenon due to the hydration process will not be studied. Only the reversible part of the transient behaviour of cement pastes will be described using the proposed model.

2.1.1. Steady state

It is now accepted that the viscosity of cement pastes depends on strain rate. Shear thinning or shear thickening behaviours may be observed. Bingham or modified Bingham, Hershel Bulkley, Ellis, Casson or Eyring models are more or less suitable to describe steady state behaviour of fresh cements pastes. Atzeni et al. [10] tested these models on Portland cement pastes and concluded that Hershel Bulkley and Eyring models are as acceptable to describe the nonlinear strain rate/shear stress curve of cement pastes. Yahia and Khayat [11] studied the influence of the choice of one model in this list on the identification of a yield stress.

The viscosity of cement pastes may also depend on both strain rate and its application time. That is why thixotropic behaviour may be observed while studying the transient flow of fresh cement pastes.

2.1.2. Transient flow

For a constant shear rate, Lapasin et al. [12] measured the difference between the maximum shear stress τ_{max} needed to initiate flow and the equilibrium value τ_e at different viscometer rotating speeds. A logarithmic evolution of the shear stress was obtained.

$$\tau = \tau_e(\tau_{max} - \tau_e)\exp(-Bt) \tag{1}$$

with B a constant depending on the viscometer rotating speed. The thixotropic behaviour was quantitatively characterised by the authors by the area between the maximum shear stress and the equilibrium shear stress in the shear stress–rotating speed diagram. The authors demonstrated a link between the thixotropic behaviour and the specific surface of the cement powder. Otsubo et al. [13] studied the time dependence of the apparent viscosity of cement pastes submitted to a constant shear rate after a rest period of 1 min. In a first phase, the apparent viscosity decreases with time until it reaches a minimum. Then it starts to increase. The authors using these minimum shear stress values plotted the steady state flow curve. They stated that the behaviour once this minimum is reached is rheopectic but they did not check that the viscosity increase was reversible. Indeed, if the measured phenomenon was due to aging or hydration process, it was not reversible and as such could not be characterised as rheopexy. In fact, other authors [12,14] demonstrated that the first phase is dominated by a destructuration phenomenon under constant shear rate (thixotropic behaviour). Once this phenomenon has reached equilibrium, the behaviour keeps on evolving because of the hydration process. This second phenomenon is not reversible. Geiker et al. [15] studied recently the characteristic time or relaxation period needed to reach steady state in the case of concrete. Concrete, of course, containing thixotropic cement paste, displays also thixotropic behaviour. For the tested concrete, for the apparatus used and for the studied strain rates, they found that this relaxation time was about 10 s.

2.1.3. Hysteresis loop

Flow curves are usually obtained by applying a succession of constant strain rates for short times. Shauhgnessy and Clark [16] or Atzeni et al. [10] pointed out that the shape of the hysteresis loop is directly related to the experimental duration of the measuring cycle. In fact, if the strain rate is applied for a time shorter than the relaxation period, steady state is not reached. If the flocculation in the sample is higher than the equilibrium state of flocculation, the measured shear stress is higher than the equilibrium shear stress. The measured flow curve is then above the equilibrium flow curve. The deflocculation process does not have enough time to bring the structure to its equilibrium state. On the other hand, if the flocculation in the sample is lower than the equilibrium state of flocculation, the structure rebuilding process does not have enough time to bring the structure to its equilibrium state and the measured flow curve is below the equilibrium flow curve as shown on Fig. 1(a). For very long measurement cycles as demonstrated by Banfill and Saunders [14], the hydration process and the long-term increase in viscosity perturb the previous patterns and hysteresis loops similar to the one on Fig. 1(b) may be obtained.

2.1.4. Structuration at rest

Thixotropy is by definition a reversible phenomenon. If a cement paste is left at rest, its flocculation will increase. This flocculation is however reversible by mixing the sample strongly enough. This is not the case with aging phenomena. These phenomena affect in an irreversible way the rheological behaviour.

The thixometer is an apparatus developed by Kalousek [17]. It allows the measurement of both the thixotropic behaviour and the paste aging effects. A first set of test is carried out on vibrated samples. This imposed vibration of the sample prevents the sample from flocculating. A second set is carried out on properly resting samples. The load needed to initiate flow is measured in both sets for several resting times. The load needed to “break” the flocculation is higher than the load needed to initiate flow in the vibrated samples. This relative difference increases with the resting time. This thixometer is an interesting way to quantify the structuration regime and to separate it from the aging phenomenon. However, because of the complex shape of the rotating apparatus generating the flow, it is not possible to link the measured load with any intrinsic properties such