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Influence of bentonite clay on the rheological behaviour of fresh mortars

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ABSTRACT

Fine mineral additives are often used in the formulation of ready-mix mortars as thickeners and thixotropic agents. Yet, these attributed fresh state properties are not clearly defined from the rheological point of view. In the present study, we consider the influence of bentonite (montmorillonite-based clay mineral) on the rheological behaviour of mortars, including in particular creep and thixotropy. The mortar pastes are subjected to different shear-rates and then allowed to creep under fixed shear stresses until reaching steady state, which corresponds to either rest if the applied stress is smaller than the yield stress or permanent flow otherwise. The evolution of the creep strain is investigated depending on shear history for different contents of bentonite. The microstructure rebuilding kinetics after shear (thixotropy) is considered by analysing the temporal evolution of the creep strain for different applied shear stresses (lower than the yield stress). As expected, bentonite is found to enhance the mortar creep (or sag) resistance. This enhancement consists of both an increase of the yield stress recovered after shear, and a diminution of the characteristic time for yield stress recovery (related to microstructure rebuilding).

1. Introduction

Ready-mix mortars, including among others tile adhesives, rendering and repairing mortars, are often characterised by a highly complex formulation. In particular different types of organic and mineral admixtures are included in their mix-design in order to meet a number of requirements related to their placement processing (pumpability, workability, sag-resistance, etc.), their hardening properties (open-time, cracking-resistance, etc.) and their long-time behaviour (water-proofing, mechanical properties, durability, etc.).

Ready-mix mortars are generally provided to the construction site in a dry power form. A given dosage rate of water is then added and the mix is kneaded to obtain a homogeneous mortar paste that can be mechanically or handily placed on vertical or horizontal supports. The rheological behaviour of the mortar paste will clearly determine its placement properties. Yet, the relationship between the rheological properties of these materials and their on-site placement behaviour are far to be clearly established [1,2]. Most of the rheological studies devoted to fresh mortars, which are usually assumed to behave as Bingham fluids, have focused essentially on two rheological parameters, namely the yield stress and the plastic viscosity [3,4]. These investigations are related in particular to the self-compacting/self-levelling issue. The transient rheological properties of cementitious materials, including mortars, are much less considered [5–7]. Moreover these investigations are mostly related to the practical issue of successive concreting and pressure on formworks. Paiva et al. [8,9] reported some rheological studies concerning specifically render mortars, considering in particular the influence of water-retaining agents [8], without however considering the issue of thixotropy.

When dealing with the problem of pumpability, as in the present study, a more extensive rheological characterisation is required since in such a process the material experiences a highly complex flow. During a machinery application process the mortar is sheared under varying rates, leading to an irreversible evolution of its microstructure, and subsequently its rheological properties. The crucial practical question is then: Does the mortar have enough thixotropy to recover a sufficient yield stress once on the support to avoid creeping under its own weight?

In the present study, to reproduce approximately the pumping process from the rheological point of view, the mortars are subjected to different shear-rates in a rheometer and let to creep under a given applied shear stress. This stress may correspond in practise to the one exerted by gravity, which intensity can be estimated as (one assumes a vertical wall): $\rho g a$, where ρ is the mortar density, a the applied layer thickness and g the acceleration of gravity. Once on the wall the material must quickly recover a yield stress higher than the gravity stress in order to avoid sagging. For a typical render mortar application ($\rho = 1800$ kg/m^3, $a = 2$ cm, $g = 10$ m/s^2), the yield stress must exceed 360 Pa once on the wall in order to avoid sagging.

We consider both the level of the yield stress recovered after shear and the dynamics of the microstructure recovering (thixotropy). We focus in particular on the influence of a bentonite clay mineral on these rheological properties.

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2. Materials and experimental procedure

2.1. Materials

The base mortar considered in the present investigation includes Portland cement (CEM I 52.5 N CE CP2 NF BLANC), hydraulic lime (NHL 3.5Z), siliceous sand with a controlled granular size distribution and an air-entraining admixture (NANSA LSS 495 / H) (see Table 1). This mortar composition is a very simple version of commercially available render mortars. The only variable mix parameter is the dosage rate of a commercially available sodium bentonite. Bentonite is composed essentially of montmorillonite which is a clay mineral [10]. The Bentonite grade used here is commercialised as additives for drilling fluids.

The recommended dosage rate of kneading water for render mortars in field applications is around 15% by weight of the dry mix, and will be retained in all the mixes considered.

In order to minimise the granular skeleton's porosity, several sands and charges with different granulometries are generally used in practise. For the present simple formulation, rolled siliceous sand is sieved to obtain two contrasted granulometries, designated as (d1) and (d2), whose diameter sizes are respectively within the intervals [0.16 mm, 0.315 mm] and [1.25 mm, 2.5 mm]. The percentages of (d1) and (d2) in the sand part are set to 30% and 70% respectively. The latter values are based on an on-going study on the influence of sand granulometry on the properties of fresh mortars, which results are intended to be submitted for publication in the future.

2.2. Rheological measurements

2.2.1. Apparatus

The rheological measurements were performed using a stress-controlled shear rheometer (AR-G2 from TA Instruments) equipped with a four-blade vane geometry (Fig. 1). Using this geometry, the tested material is not subjected to a uniform shear rate. This condition is usually required in rheological experiments in order to measure actual material properties, and to have an analytical relationship between the measured torque/rotational velocity and shear-stress/shear-rate. Nevertheless, vane geometry has been retained since it is appropriate for high yield stress fluids such as dense granular suspensions, including mortars [11,12], as slippage can be avoided and the material can be sheared in volume. The shear-rate and shear-stress are inferred from the measured torque and rotational velocity of the vane using a calibration method described in details in Refs. [13,14].

The gap, which represents the distance between the periphery of the vane and the outer cylinder, is equal to 8.3 mm, which is only about three times the maximum grain size. As a result, the influence of the discrete aspect of the suspensions on the rheological measurements should be considered. However, this will not change fundamentally the results reported here. The temperature has been regulated at 25 °C (to within 0.1 °C) thanks to a circulating water system. In order to minimise water evaporation the cup of the measurement system was sealed.

2.2.2. Measurement procedure

All the mortars have been mixed (using a Perrier laboratory mixer) using the same procedure, that comprises the following steps:

i) mixing of the dry components at low speed (60 rpm) for 30 s
ii) addition of the required quantity of kneading water
iii) mixing at low speed for 30 s

Table 1
Mix design of mortar.

Constituent	Cement	Hydraulic lime	Sand	Air entraining	Bentonite clay	Water
% (by weight)	15	5	80	0.01	Varied: 0→1%	15

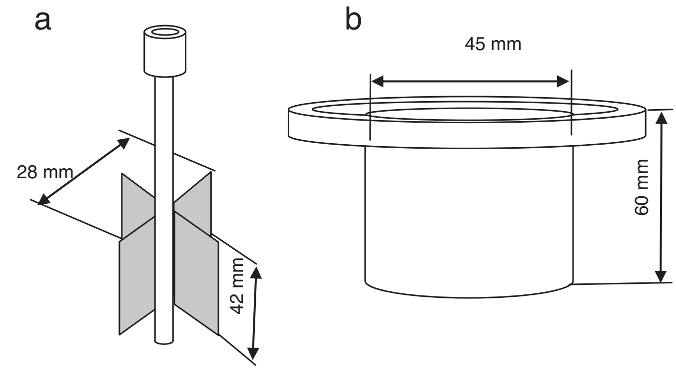

Fig. 1. Rheological measurement system: a) vane tool and b) outer cylinder (cup).

iv) scraping down the sides of the mixer bowl for about 30 s
v) mixing at low speed for 60 s.

The rheological measurements started after 5 min resting from the end of mixing.

The measurements were undertaken during the induction period, characterised by a very low hydration rate, which may not influence the rheological response of the material. In order to check that this was actually the case, two successive and identical rheological measurements were performed on the same sample, indicating that no mechanical irreversible transformations (hydration) of the material were detected within two hours after mixing.

The sample was subjected to the solicitation history illustrated in Fig. 2. It was first sheared at a given controlled shear-rate $\dot{\gamma}$ during T_s (shearing time). This was followed by the application of a relatively small controlled stress σ_1 during T_c (creeping time). The value of σ_1 was chosen to be smaller than the yield stress, which was *a priori* inferred from the flow curve determined at controlled shear-stresses. The sample was sheared again at $\dot{\gamma}$ during T_s and a higher stress σ_2 ($>\sigma_1$) was applied during the same creeping time T_c. This procedure was repeated for increasing applied stresses. To account for the influence of the value of the applied shear-rate before creep, freshly prepared samples were used and the same procedure was repeated.

The shearing time T_s and creeping time T_c were taken to be long enough to reach steady state with all the shear rates and shear stresses considered. In order to fulfil this criterion, we took $T_s = 60$ s and $T_c = 30$ s.

In a typical rendering mortar application using a plastering machine the maximum flow rate (Q) generally encountered is around 10 l per 30 s, that is $Q = 3.33\times10^{-4}$ m^3/s. The product is subjected to varying shear-rates throughout the pumping circuit. For a given flow rate the shear rate is the highest in the regions where the duct section is the smallest. The duct would be the tightest at the spray gun level. In this region the minimum duct radius (r) is about r = 0.5 cm. The maximum shear-rate experienced by the product during a typical application can be then estimated as: $\dot{\gamma}_{max} = (Q/\pi r^2)\times 1/r \approx 850\,\mathrm{s}^{-1}$ In this estimation, it is assumed that the material is sheared all through the duct section (Poiseuille flow). Yet, since it possesses a yield stress, one has rather a plug flow in which it is sheared only near the duct surface. The value above of the maximum shear rate may be then underestimated. We cannot apply such a high shear rate with our rheometer equipped with a measurement system with a gap of 8.3 mm. The maximum shear rate that can be applied with our apparatus is estimated to be 600 s^{-1}.

3. Results and discussions

3.1. Steady state rheological behaviour

Similar to other types of fresh cement-based materials, mortars are often modelled approximately as Bingham fluids [4]. For theses fluids

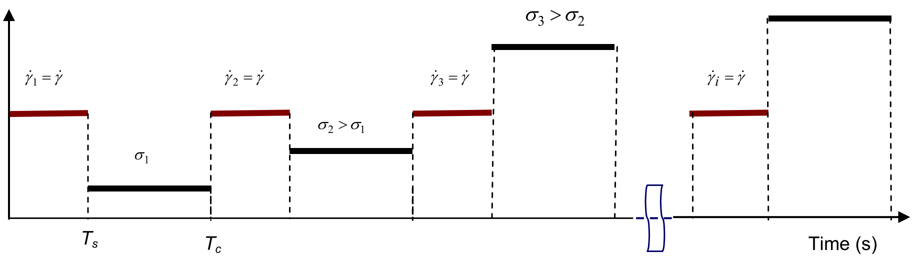

Fig. 2. Solicitation sequence used to consider creep.

the steady-state stress (σ) is related to the steady-state shear rate ($\dot{\gamma}$) through the following relationship:

$$\sigma = \sigma_0 + \mu\dot{\gamma}, \quad (1)$$

where σ_0 is the yield stress and μ is the plastic viscosity. Yet, in the case of mortars comprising high contents of polymer admixtures a third rheological parameter is needed in order to model the flow curves (Herschel–Bulkley's model) [5,15–18]. That is:

$$\sigma = \sigma_0 + \mu\dot{\gamma}^{\mathrm{n}}, \quad (2)$$

where n designates the fluidity index.

A typical flow curve (for a mortar with 0.1% bentonite), obtained by increasing and then decreasing the shear stress, is represented in Fig. 3. The mortar was presheared at 300 s^{-1} before starting the upward flow curve determination. For each applied stress the temporal evolution of the shear-rate is recorded until an approximate steady state value is obtained (see the creep experiments). The flow curve is determined by plotting the applied stresses versus average values of the corresponding shear-rate plateaus. It takes less than a second to obtain the steady state as it can be seen in Figs. 9–10. For each applied stress the shear-rate is recorded during 60 s which is then long enough to obtain steady state. It takes 90 s to obtain each point of the flow curve. The duration of the upward and downward flow curves determination is 22.5 min, which is much lower than the duration of the induction period (about 2 h).

The upward branch of the flow curve is lower than the downward one, indicating that the material is rheopectic. The shear-induced thickening of the material can be explained as the following. The mortar is mainly composed of coarse solid particles (sand and partly cement) and more or less fine particles (partly cement, lime and bentonite). The mortar can be considered as a suspension of the coarse grains in a suspension (or dispersion) of colloidal particles. The very fine particles are expected to have lubricating effects on the coarser ones. Without a dispersing admixture (such as a super-plastizer) the colloidal particles are flocculated in equilibrium. Under shear flow the flocs can be broken and the suspension of the fine particles is expected to be shear-thinning. Then when subjected to shear flow, the suspension of coarse particles (mortar) will thicken since the lubricating effects of the fine particles will decrease.

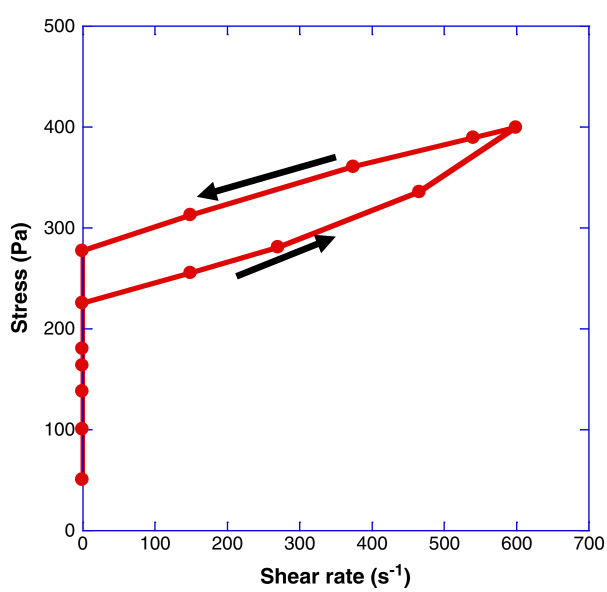

Fig. 3. Typical flow curve of mortars with 0.1 % bentonite.

Fig. 3 shows that the downward flow curve follows approximately the Bingham's model, while the upward branch rather follows the Herschel–Bulkley's shear-thickening model. Nevertheless, at present the interest lies in the determination of the yield stress value associated with the onset of fluid flow, independently from which model is considered. The latter value is required in order to determine the shear stress interval to consider for the creep experiments. The yield stress is determined by extrapolating the upward flow curve to zero shear-rate. This value of yield stress corresponds to the microstructure reached by the material after mixing, resting for 5 min, and loading into measurement system. As will be shown below, the value of the yield stress is highly dependent upon the solicitation history.

3.2. Creep test results

Fig. 4 represents the time evolution of the strain (angular displacement of the periphery of the vane tool divided by the gap thickness) for different applied stresses, in the case of the base mortar with 0% content of bentonite. The value of the pre-shear rate in this example is 600 s^{-1}. The origin of the time axis corresponds to the end of pre-shear and beginning of stress application.

The creep curves can be divided into two different categories. From Figs. 4 and 5, for relatively low applied stresses (lower than 380 Pa), strain increases at first during a certain transient period and then reaches a steady state corresponding to flow stoppage. Above a certain critical value of the applied stress (comprised between 350 and 380 Pa), a permanent flow with a finite shear-rate is obtained after a certain transient period. The transient period will be discussed further in this article in relation with thixotropy. Such "viscosity" bifurcation effect has already been reported in the literature in the case of other kinds of thixotropic/yield stress fluids [19].

Fig. 5 represents the time evolution of the apparent shear rate during the creep period for different applied stresses. Two different bentonite contents (0% and 1%) have been employed. Below the yield stress the shear rate goes to zero in less than a second. Above the yield stress value, the resulting flow is characterised by a highly fluctuating shear-rate. The amplitude of these fluctuations decreases as we move away from the yield stress. This phenomenon may be related to the avalanche behaviour of pasty-like fluids as discussed by Coussot et al. [20] and deserves further investigation in the case of

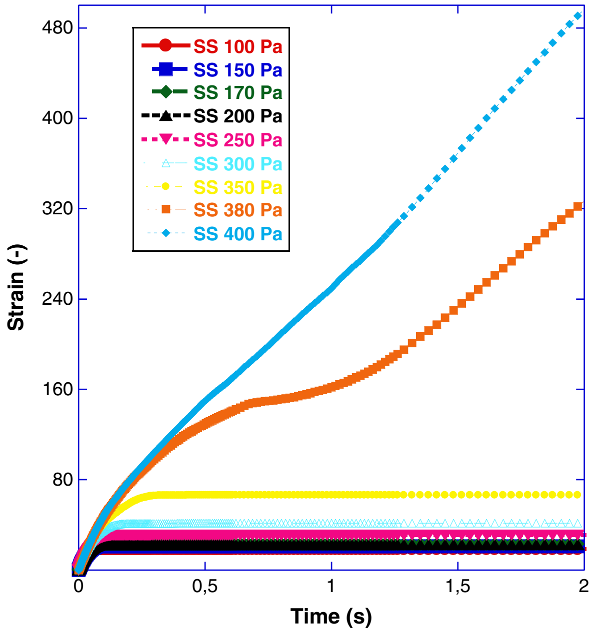

Fig. 4. Time evolution of strain for different applied shear stresses, in the case of mortar without bentonite.

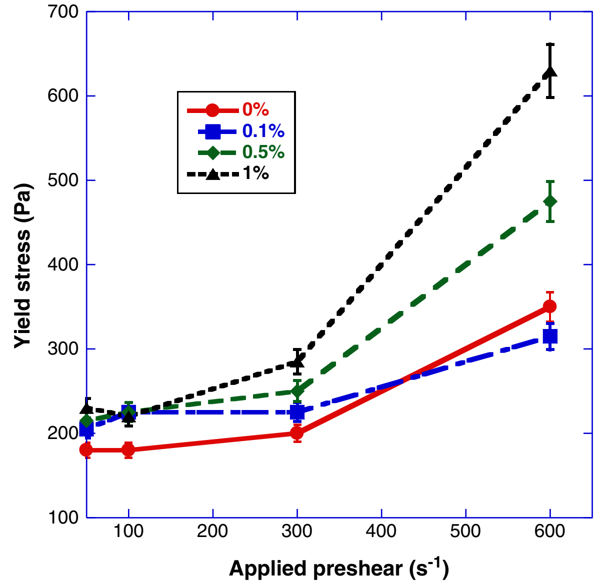

Fig. 6. Yield versus pre-shear rate for different bentonite contents.

mortars. The transient part of the curves is analysed in more details below.

Fig. 6 represents the evolution of the yield stress recovered during the creep period as a function of the applied pre-shear rate for different bentonite contents. All the formulations display rheopexy, in the sense that the yield stress increases with the level of pre-shear. The rheopexy aspect increases with bentonite content. It is to be noted that bentonite suspensions in water are actually thixotropic [21], as their yield stress decreases with pre-shear rate. The rheopexy of the mortar may be considered as an indirect consequence of the thixotropy of the aqueous bentonite suspension. Indeed, when the mortar is sheared, the yield stress of the bentonite-water phase is decreased, which may induce more inter-granular contacts. This will result then in an increase of the mortar yield stress.

From a practical point of view, it is important to assess the total displacement due to creep for a given applied stress (related to the applied mortar thickness). Indeed, a certain amount of creep is always present, depending upon the flow history of the mortar, which is related to the placement method (by hand or mechanically).

Fig. 7 represents the evolution of the total creep, in terms of strain as a function of the applied stress, for different bentonite contents. As expected, the total creep increases with the applied stress and diverges at yield stress. The experimental results show that under any given applied stress, bentonite addition decreases the total creep. This effect increases with the applied stress.

3.3. Relationship with thixotropy

During the creep period the material undergoes a constant shear stress. Under shear stresses higher than the material's yield stress and

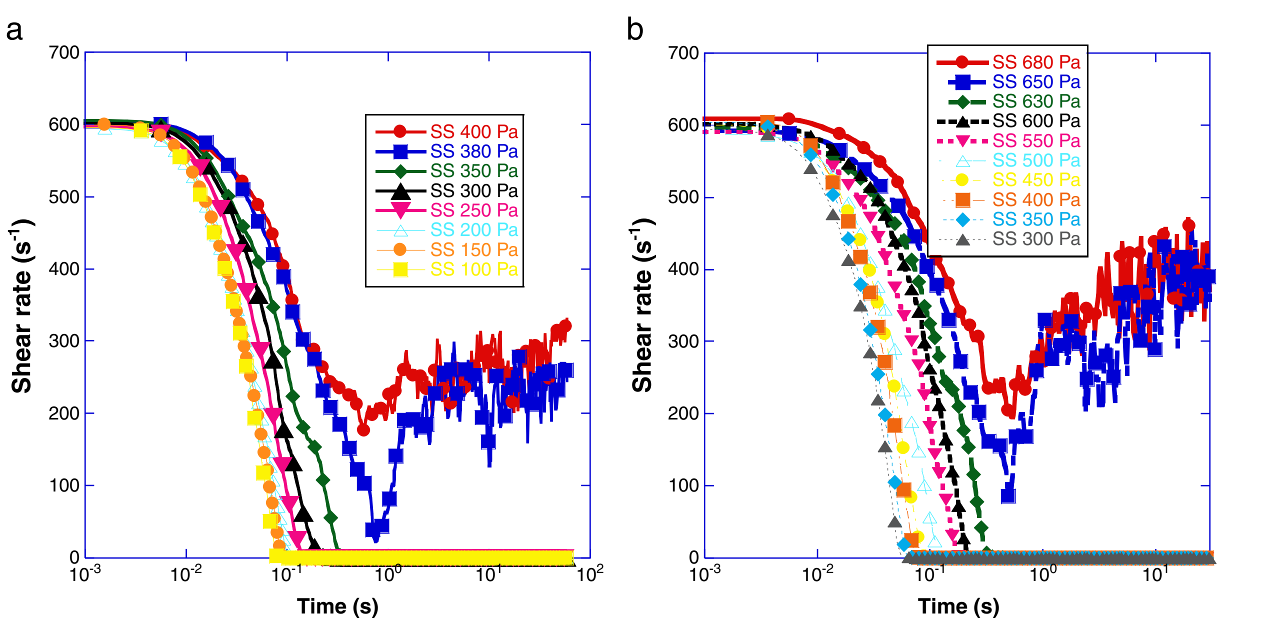

Fig. 5. Time evolution of shear rate during the creep period under varying applied shear stresses. (a) Formulation without bentonite; (b) Formulation with 1% bentonite.

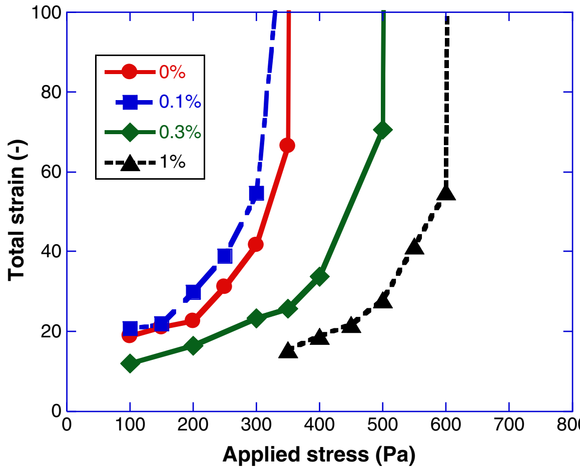

Fig. 7. Total creep versus applied shear stress for different bentonite contents.

after a certain period of time, a permanent flow is obtained (Fig. 5) with a shear rate depending upon the applied shear stress. For stresses smaller than the yield stress, flow stoppage is obtained after a certain transient time. The temporal evolution of the shear rate for a given applied stress indicates that the material's rheological properties are evolving with time. To take into account this phenomenon a thixotropy model can be used. Several models, of varying complexity, have been reported in the literature to describe thixotropy (see for instance the review articles [22,23]).

In the present study, we propose to relate in a very simple manner creep and thixotropy. In the following discussion, it is not necessary to make distinction between thixotropy and rheopexy, as both cases are concerned with the temporal evolution of rheological properties with time.

The simplest thixotropy model must comprise at least a structural parameter λ, which characterises the connectivity of the microelements making up the material. Rigorously speaking, to deal with thixotropy the connectivity that determines λ must be reversible. For instance mechanical contacts among the sand grains are not reversible. Then, they do not contribute to thixotropy. The interactions giving rise to thixotropy are those related to the colloidal scale particles, meaning in our case bentonite, polymer admixtures and partly cement and lime.

In general one considers $\lambda = 0$ for a fully broken material's microstructure and $\lambda = 1$ when the connectivity in the material is maximum. During creeping, the evolution of the parameter λ may be determined by the competition between microstructure rebuilding, that takes place with a characteristic time τ, and breakdown whose kinetics depend essentially on the instantaneous shear rate $\dot{\gamma}(t)$. Then, one can assume, as a first approximation:

$$\frac{d\lambda}{dt} = \frac{1}{\tau} - \lambda \dot{\gamma}(t). \tag{3}$$

This corresponds to the simplest possible thixotropy model, which has been proposed by Moore [24].

To go further, a functional form must be introduced for the relationship between the structural parameter λ and the rheological properties. The rheological property that can be directly inferred from creep measurements is the plastic viscosity μ (ratio between the applied stress and the instantaneous shear-rate). The plastic viscosity must diverge when the structural parameter goes to unity and has a finite value for a completely broken structure (for $\lambda = 0$). The simplest relationship between μ and λ that fulfils these conditions is $\mu(\lambda) = \mu_0(1/1-\lambda(t))$. μ_0 is the Newtonian viscosity corresponding to a fully broken down microstructure. When the material is subjected to a fixed creep stress σ, the shear-rate can be written as follows (assuming a Binghamian behaviour for simplicity):

$$\dot{\gamma}(t) = \frac{\sigma - \sigma_y}{\mu_0}(1 - \lambda(t)), \tag{4}$$

where σ_y is the yield stress corresponding the pre-shear conditions considered.

By combining Eqs. (3) and (4), we obtain:

$$\frac{d\lambda}{dt} = \frac{1}{\tau} - \frac{\sigma - \sigma_y}{\mu_0}\lambda + o(\lambda^2). \tag{5}$$

To solve Eq. (5) the nonlinear term is ignored assuming that λ remains small compared to unity.

Solving Eq. (5) and using Eq. (4), we obtain the relationship giving the evolution of the shear-rate during the initial stage of the creep:

$$\dot{\gamma}(t) = \left(\frac{\sigma - \sigma_y}{\mu_0} - \frac{1}{\tau}\right) + \left(\dot{\gamma}0 - \left(\frac{\sigma - \sigma_y}{\mu_0} - \frac{1}{\tau}\right)\right) exp\left(-\frac{t}{\mu_0 / (\sigma - \sigma_y)}\right), \tag{6}$$

$\dot{\gamma}_0$ is the initial shear rate.

From Eq. (6), the characteristic time of the creep flow is given as:

$$\tau_{creep} = \frac{\mu_0}{\sigma - \sigma_y}. \tag{7}$$

Eq. (7) predicts that the creep characteristic time increases when the applied stress approaches the yield stress. When the applied stress is equal to the yield stress, Eq. (7) indicates a divergence of the creep characteristic time. This corresponds to the appearance of a permanent flow.

The best fit between Eq. (6) and the experimental results is illustrated in Fig. 8. We can see that the fit is quite satisfactory at the beginning of creep, while a significant divergence between model predictions and measurements is obtained when approaching flow stoppage. This may be attributed to the fact that the above model is strictly valid for small structural parameters, at the beginning of creep.

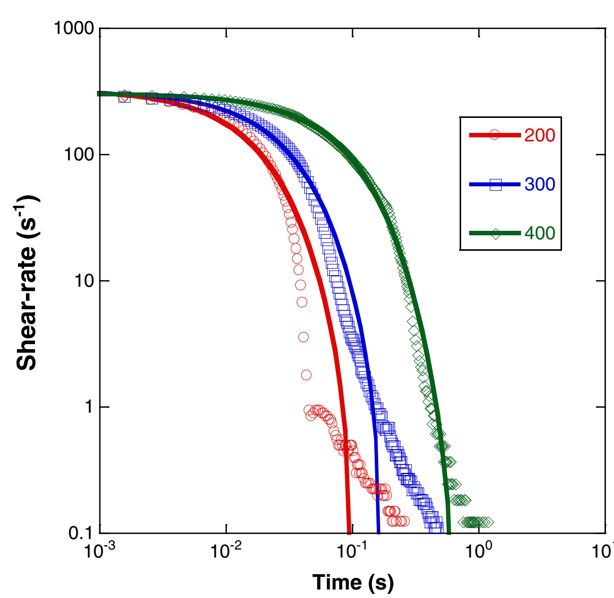

Fig. 8. Illustration of the fit between the simple thixotropy model and experimental creep results in the case of a mortar with 1% bentonite.

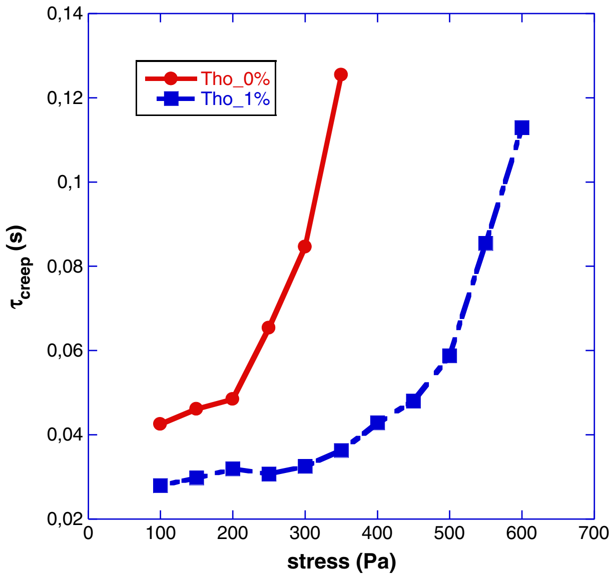

Fig. 9. Evolution of the rebuilding characteristic time versus applied stress for different contents of bentonite.

A more complete creep model bringing into play more than one relaxation time, for instance a distribution of relaxation times, is needed in order to fit the whole experimental curves.

From a practical point of view, it is important to know the maximum load that the material can withstand for a given flow history, which is related to the yield stress value. Therefore one may wish to know the time needed for the material to reach its yield stress. The best fit between Eq. (6) and creep curves can lead to an estimation of this creep characteristic time.

The evolution of the creep characteristic time as a function of the applied stress is reported in Fig. 9 in the case of two different formulations (without bentonite and with 1% bentonite).

In agreement with Eq. (7), the creep characteristic time increases with applied stress. That is, increasing the applied stress (remaining of course below the yield stress) leads to a slow down of the creep flow.

Fig. 10 represents the evolution of the characteristic time of yield stress recovering with bentonite content. It can be seen that bentonite

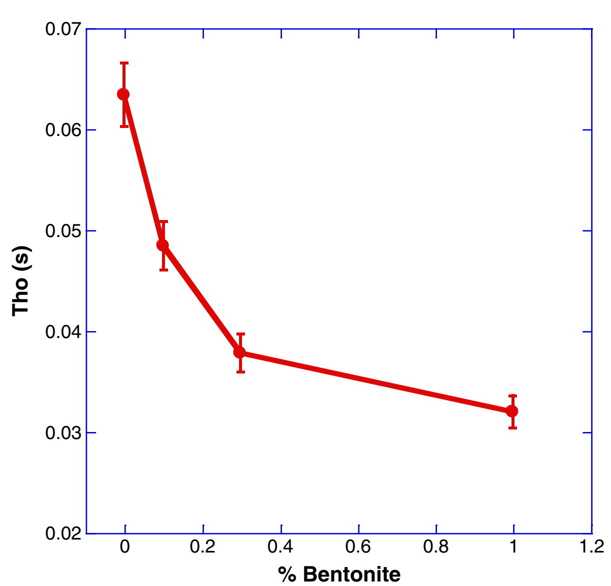

Fig. 10. Characteristic time of rebuilding as function of bentonite content.

speeds up the rebuilding kinetics. These results highlight the beneficial effect of such additives on the thixotropy of the mortar.

4. Conclusions

We have investigated the influence of bentonite clay addition on the rheological behaviour of fresh mortars, and more particularly on the ability of the material to recover yield stress after being sheared at high rates (i.e. thixotropy). By determining the flow curves at controlled stresses, the mortars were first shown to display a rheopectic behaviour. This rheopectic aspect was attributed to the diminution of the lubricating effects of the fine particles suspensions due their shear-thinning property.

It was found that bentonite increases both the level of yield stress recovered after shear and the kinetics of the microstructure rebuild up. The yield stress recovered after shear increases with pre-shear rate, which is related to the rheopectic aspect of the mortar's rheological behaviour.

A simple thixotropy model was used to interpret, at least qualitatively, the experimental creep curves.

Addition of bentonite enhances the shear-induced thickening effect (i.e. rheopexy). On one hand, this phenomenon may prove advantageous regarding the problem of sagging during machinery application of mortars on vertical masonries. On the other hand, the shear-induced enhancement of the yield stress, which may exceed the critical yield stress of a pumpable mortar, can lead to flow stoppage.

Acknowledgments

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