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Rate of thixotropic rebuilding of cement pastes modified with highly purified attapulgite clays

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ABSTRACT

This study investigates the influence of highly purified, nano-sized attapulgite clays on the rate of structural rebuilding of cement pastes. A shear rheological protocol is implemented that measures the rate of rebuilding of pastes after being broken down under shear and maintained under stress corresponding to the weight of the material. This simulates a real casting situation during which the concrete is initially in motion, then cast in place and measures how quickly it gains green strength immediately after placement. The rate of recovery for different resting times and preshear conditions are considered. The strain rate decay curves are fitted with a compressed exponential model to obtain relaxation time. The results show that the purified attapulgite clays significantly accelerate rate of recovery of pastes, especially at early ages. However, this accelerating effect diminishes at longer resting times as hydration mechanisms begin to dominate.

1. Introduction

Self-consolidating concrete (SCC) improves constructability through its high flowability and superior segregation resistance. But despite its benefits, major issues include high transfer of lateral pressure to the formwork wall by SCC during casting and overall poor understanding of SCC formwork response, both of which can contribute to the possibility of formwork failure. Due to these reasons American Concrete Institute Committee 347, Formwork for Concrete, recommends that formwork be designed to withstand hydrostatic pressure. This raises the cost of formwork, which makes up a significant portion of the cost of construction. Therefore many studies have explored methods to tie the rheological behavior of SCC to formwork pressure response and strategies to reduce SCC formwork pressure through mix design [1–4].

It has been shown that it is possible to reduce formwork pressure through the use of mineral admixtures [5,6]. In one study by Kim et al., it was found that small additions of highly purified attapulgite clay can reduce the formwork pressure of SCC concrete mixes over time [7]. However, the physical origin of this effect is still not fully understood. A major factor affecting SCC formwork pressure is its level of thixotropy — rate of structural breakdown under shear followed by a recovery upon the removal of shear [8,9]. High rate of structural rebuilding indicates a rapid development of green strength (fresh-state stiffness) and subsequently greater reduction in lateral pressure on the formwork wall. Past studies have shown that clays can increase the flocculation strength and floc size of cementitious materials [10,11], which can explain its effect on the rheological properties. To tie it to formwork pressure, it is necessary to measure the effect of clays on the level and rate of thixotropic rebuilding. Many methods have been implemented to measure the thixotropic structural breakdown and recovery of cementitious materials [9,12–17]. For formwork pressure, it is of interest to determine the rate of rebuilding after shear and under an applied stress, which is the equivalent of casting during construction. Also, it is critical to know the change in the rate of flocculation at different ages of the material, as casting occurs over time.

In the present study, the effect of a highly purified attapulgite clay on the rate of structural rebuilding of cement pastes is investigated. A shear rheological protocol, adopted from a separate study [18], is implemented that measures the rate of structural rebuilding of pastes after being broken down under shear and maintained under stress corresponding to the weight of the material. This simulates a real casting situation during which the concrete is initially in motion, then cast in place and measures how quickly it gains green strength immediately after placement. This allows determination of the influence of clays on rebuilding when the material is essentially at rest but under stress corresponding to the weight of the material above the sample considered. Due to the effects of hydration, cementitious materials are both shear-history (thixotropy) and age dependent,

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particularly within the first few minutes. Therefore the effect of clays on the rate of recovery for different resting times and preshear conditions is considered. The strain rate decay curves are fitted with a compressed exponential model to obtain relaxation time, which will describe the influence of the various parameters on rate of structural recovery.

The present study is limited to the paste phase. Although there is a marked difference between paste and concrete rheology due to the presence of larger, inert particles in the latter, the time evolution of the rheological properties due to hydration mechanisms and thixotropic rebuilding are primarily tied to the paste phase. Therefore, how attapulgite clays alter the rheology of pastes will reflect how they will alter the fresh-state properties of concretes. And such results can subsequently help to further elucidate their effect on the formwork pressure response of SCC.

2. Materials and experimental methods

2.1. Materials

Tap water and type I Portland cement with a Blaine fineness of 385 m^2/kg are used in all mixes. A commercially available, highly purified form of the mineral attapulgite, or palygorskite, is the clay chosen for the study. They are chemically exfoliated from bulk attapulgite to remove impurities such as smectite, bentonite and other swelling clays, making them effective rheology-modifiers for various materials, including concretes. It is a rod-like nanoclay — 1.75 μm in average length and 3 nm in average diameter [19]. Given its high aspect ratio, it can form a gel even at small solid concentrations. Herein, the purified attapulgite clay will simply be referred to as “clay” or “nanoclay,” but the results are for this specific type of clay and not necessarily representative of other types.

Cement paste mixes with a water-to-cement ratio of 0.43 by mass are tested. They are each prepared by hand-stirring for 60 s, then loaded in the rheometer and tested immediately after. Pastes with 0 and 0.5% nanoclay addition by mass of cement (labeled 0NC and 0.5NC, respectively) are tested. For the clay-modified pastes, the nanoclays are introduced as an aqueous suspension — the nanoclay is blended with the mixing water in a blender for 5 min to disperse.

2.2. Experimental methods

All rheological tests are performed on a Paar Physica MCR rheometer with a parallel-plate geometry. The top plate has a diameter of 50 mm and the bottom plate is temperature-controlled with a circulating water bath set to 20 °C. The surfaces of the plates are covered with 150-grit adhesive sandpaper to prevent slip. The measuring gap is 1 mm. All the measurements are performed at least three times in order to ensure the reproducibility of the results. Details on the experimental procedures can be found elsewhere [20].

2.2.1. Rate of structural recovery

To measure the effect of clays on rate of structural rebuilding, a rheological protocol is applied where the sample is initially sheared at a constant shear rate to break down its structure and then a fixed shear stress lower than its yield stress is applied. The strain rate decay provides a measure of the rate at which the material regains enough structure to resist the applied stress. The faster the rate of decay, the higher the rate of rebuilding and vice versa. The protocol is shown in Fig. 1. It is initially strain rate controlled, where a preshear is applied for 60 s. Then it switches to shear stress control and the strain rate decay is measured. The shear stress is applied until the strain rate reaches zero. A break criterion is defined in this step: when the shear rate becomes less than 0.01 s^{-1} (essentially zero) it skips to the next step or to the end of the test.

During the creep step, constant shear stresses of 20 and 30 Pa are applied, both of which are lower than the yield stress of the mixes tested. Due to hydration mechanisms, the rate of recovery will vary depending on the age of the sample and the shear condition. Therefore resting times of 0, 120, and 1800 s and shear rates of 50 and 300 s^{-1} are considered. For the cases of 120 and 1800 s resting time, the preshear at the beginning of the test is always set to 50 s^{-1} (to ensure all mixes start with the same shear history before the rest period) and only the preshear in the step prior to the creep step is varied.

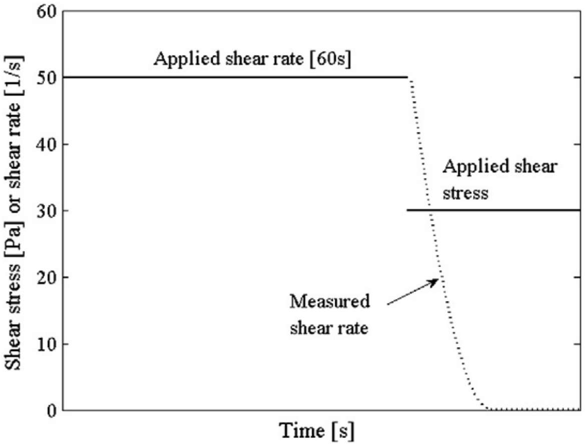

Fig. 1. Shear rheological protocol to measure strain rate decay during creep.

2.2.2. Yield stress

In the shear rheological protocol for obtaining relaxation time, the applied shear stress must be less than the yield stress. Therefore it is necessary to determine the yield stress of the paste samples. This is done through the protocol shown in Fig. 2. The applied shear stress is incrementally increased (by 10 Pa) until the shear rate no longer reaches zero (the material cannot rebuild enough structure to resist the applied stress). When shear rate diverges away from zero, the yield stress has been exceeded.

2.2.3. Low-amplitude oscillatory shear rheometry

The structural evolution of the pastes is obtained through low-amplitude oscillatory shear rheometry, a method that provides a measure of the viscoelastic properties of suspensions. It has been demonstrated to be applicable to fresh cement paste and the details of the test can be found elsewhere [21].

Oscillatory strain is applied as a sine function:

$$\gamma = \gamma_0 \sin \omega t \tag{1}$$

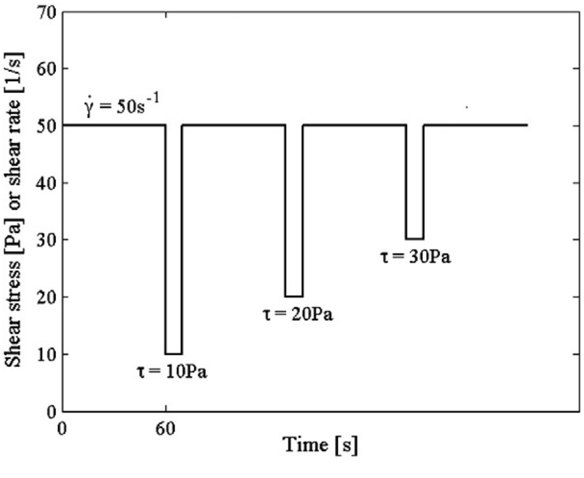

Fig. 2. Shear rheological protocol to measure yield stress.