

ORIGINAL ARTICLE



Mechanisms of thixotropy in cement suspensions considering influences from shear history and hydration

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Abstract

The rheological properties of fresh concrete are a direct function of the interaction behaviour of the granular inventory of the concrete (i.e., gravel, sand and cement) and especially of the colloidal fractions of cement. Under low shear stresses, agglomeration of colloidal particles is observed, while at high shear stresses, dispersion of these agglomerates occurs. Besides the agglomeration state, the formation of shear banding, zones with concentrated shear flow, is another controlling mechanism of the flow behaviour of cement suspensions. Rheological creep tests in this study are focused on investigating the influence of shear history and hydration process on thixotropy of cement suspension. In this paper, the meaning of the word thixotropy is slightly extended to additionally encompass rheological aging and hydration effects. Selected samples were analyzed by coupling a rheometer to synchrotron X-ray tomography to gain insight into the shear-induced microstructural changes during shear start-up tests. The observations show heterogeneities in the velocity profile in the shear gap and the development of shear banding.

Keywords

Rheology, Thixotropy, Cement suspension, Hydration, Rheo-Tomography, Shear banding

1 Introduction

A main factor for ensuring high-quality concrete structures are appropriate rheological properties of the fresh concrete tailored to the corresponding placing process [1]. High flowability provides complete filling of complex formworks, prevents the formation of blockages during concrete pumping, and guarantees the escape of trapped air due to good compactibility. However, the flowability must not exceed a certain level in order to prevent sedimentation processes [2].

The rheology of fresh concrete is a direct consequence of the interaction behaviour of the granular inventory (i.e., gravel, sand and cement) of the concrete and here especially of the finest cement fractions suspended in water. Since cement shows a highly polydisperse particle size distribution, especially the colloidal particles coin the rheological behaviour by interparticle interactions [3]. The formation of a percolated particle network due to attractive interactions among the colloidal particles produces a

non-Newtonian flow behaviour with a pronounced yield stress [4], i.e., yielding of the suspension requires a critical shear load below which flow ceases to occur. At rest, agglomerates of particles are formed, while the suspension flow results dominantly from the dispersion of the developed agglomerate structure upon shear [5]. In contrast, Chateau *et al.* postulated that a yielding process is a statistical process, with the onset of flow originating in zones with locally lower solids phase content [4]. Consequently, yielding of colloidal suspensions can be related to the formation of shear banding, which results from coexisting different apparent viscosities and yields in a non-linear velocity profile within the sample [6,7]. Experimental evidence of shear band formation in real cement systems is currently incomplete and would help to develop more accurate models of flow behaviour as knowledge of controlling flow mechanisms is expanded.

For the description of the flow behaviour of cement suspensions, generally, models based on steady-state flow behaviour are used to determine Bingham yield stress (τ_0)

or Bingham plastic viscosity (μ) [8,9]. Whilst highly accurate models are available to predict the rheological properties of dilute suspensions, equivalent models accounting for the rheology of highly-filled suspensions (i.e., cement suspensions) are either completely missing or fail to describe important characteristics such as thixotropy [8,10].

A thixotropic structure breaks up while shearing and rebuilds at rest [11]. Note that thixotropy is defined as a reversible, inelastic, time dependent change of viscosity [11,12], whereas in cementitious systems they will always be superimposed by reversible as well as non-reversible effects due to physical particle interaction, aging, and chemical hydration process [13,14]. For the sake of simplicity and conforming to the conventions in the cement community the meaning of the word thixotropy is extended in this study to describe these additional effects.

For cement suspensions, Roussel *et al.* showed a linear increase in static yield stress with resting time and presented a model for the prediction of thixotropic properties in a linear dependency on measurement time [10]. Perrot *et al.* state that the proposed linear correlation of thixotropy by Roussel is not valid for hydration times higher than 60 min and thus amended the linear approach with an exponential term [14]. The authors of the present study already showed for selected rheological parameters, the closer the hydration time approaches the setting time, the more pronounced the non-linear behaviour becomes [15,16].

Various influencing factors also explain the lack of comprehensive models describing the complex rheological behaviour of cement suspensions. Following water-cement contact, irreversible changes caused by hydration processes occur, affecting the rheological properties. Two main hydration reactions – the silicate and the aluminate phases – proceed in parallel and affect rheology [17]. Roussel *et al.* see the hydration products of the silicate reactions as main controls of thixotropy [18]. In contrast, Barnes & Bensted evaluate the aluminate reaction as dominating mechanism on cement paste rheology [19]. Additionally, cement suspensions are influenced by shear history due to a change in the particle structure [5]. Consequently, shear history should always be considered when determining rheological properties, especially thixotropy. Therefore, in the presented work, the influence of shear history and hydration on the thixotropic properties of cement suspensions is studied to give a precise dependence on the impact of both factors. Further, the possibility of verifying the occurrence of shear banding and corresponding segregation of an initially homogeneous suspension as influencing mechanisms of the flow behaviour provides deeper insight into thixotropy of cement suspensions.

2 Materials and Methods

2.1 Material and preparation of suspensions

The investigated cement suspensions were prepared with an ordinary Portland cement CEM I 42.5 R. The cement was characterized in detail in an interlaboratory study (see [20]). The essential parameters are its density of 3.12 g/cm³, specific surface area of 3600 cm²/g determined by the Blaine method, and particle size distribution

with a d_{50} -value of 14.8 μm [16]. The cement suspensions for rheological testing were prepared with 1778.9 g of dry cement, stored at 20°C, and 728.0 g of demineralised water, pre-tempered at 10°C. The mixing procedure is shown in Tab. 1 and was performed with a standard mixer, as defined in DIN EN 196-1 [21]. The resulting suspensions have water/cement mass ratio of 0.41 and a volumetric content of solids (solids phase content) of 0.44.

Table 1 Mixing procedure for sample preparation.

Procedure	Mixing intensity	Duration (s)
Homogenization of dry mixture	140 rpm	60
Addition of water and mixing	140 rpm	15
Mixing at low speed	140 rpm	45
Rest phase and manual return of caking material	-	90
Mixing at high speed	285 rpm	60
Rest phase and manual return of caking material	-	30
Mixing at high speed	285 rpm	120

After mixing, the sealed cement suspensions were stored in a water bath at 20°C until the rheological investigation with the standard measurement profile started at hydration times $t_{\text{hyd}} = 15, 30, 60, 120,$ and 180 min. The hydration time t_{hyd} starts with water-cement contact and describes the age of the samples, whereas the later mentioned measurement time t starts at the beginning of each rheological measurement. A remixing of the samples at 140 rpm for 30 s was performed one minute before starting the rheological measurements. Each measurement corresponds to a new suspension to prevent influences by pre-shear history. The rheological investigation with the extended measurement profile started $t_{\text{hyd}} = 15$ min after water-cement contact and lasted up to $t_{\text{hyd}} = 180$ min.

2.2 Methods

The hydration kinetics of cement suspensions were investigated by isothermal conduction calorimetry with internal mixing of the sample. The tests were performed on a Ton-Cal Trio (Tonitechnik, Berlin, Germany). The results were adjusted with a mathematical correction of the device-dependent time constant. By assuming complete hydration at 30°C after 96 h, the degree of hydration was calculated based on the ratio of the total released heat at corresponding t_{hyd} in relation to the total released heat at complete hydration (315.2 J/g):

$$\xi = \frac{\text{released heat } \left[\frac{\text{J}}{\text{g}} \right]}{315.2 \frac{\text{J}}{\text{g}}} \quad (1)$$

The rheological measurements were performed with a Haake MARS 60 rheometer (ThermoFisher Scientific, Karlsruhe, Germany) equipped with a building materials cell. The measurement geometry is a two-blade vane without the middle part and the cylindrical sample container is ripped in order to prevent wall slip. For this setup, a sample volume of 301 cm³ is required. A tempering unit

ensured a constant temperature of 20°C during measurements. Further details about the measurement geometry and the rheometer can be found elsewhere [8,22]. The rheological measurement sequence mainly consists of loading phases (LP) characterized by a structural breakup of the particle network in the suspensions due to high shear stress and recovery phases (RP) with subcritical shear stress ensuring a recovery of the particle network.

The first series of rheological measurements were performed with a standard measurement profile (Fig. 1a). Fresh samples were remixed and tested at $t_{\text{hyd}} = 15, 30, 60, 120,$ and 180 min after water-cement contact. The shear stress during LP was varied between 300, 200, 150, and 100 Pa.

Besides different shear stresses during LP, the influence of the resting time between measurements was investigated to probe different aspects of shear history. This second series of investigation was carried out with an extended measurement profile (Fig. 1b). A suspension was continuously investigated between sample ages of $t_{\text{hyd}} = 15$ min and $t_{\text{hyd}} = 180$ min. Different resting times were set by adjustments of the measurement job. The shear stress during LP was constant for the whole measurement (i.e. 300, 200, 150, or 100 Pa). The resting time between RP and LP varied over 1, 5, 10, to 30 min. The sample remained continuously in the rheometer, and no remixing took place.

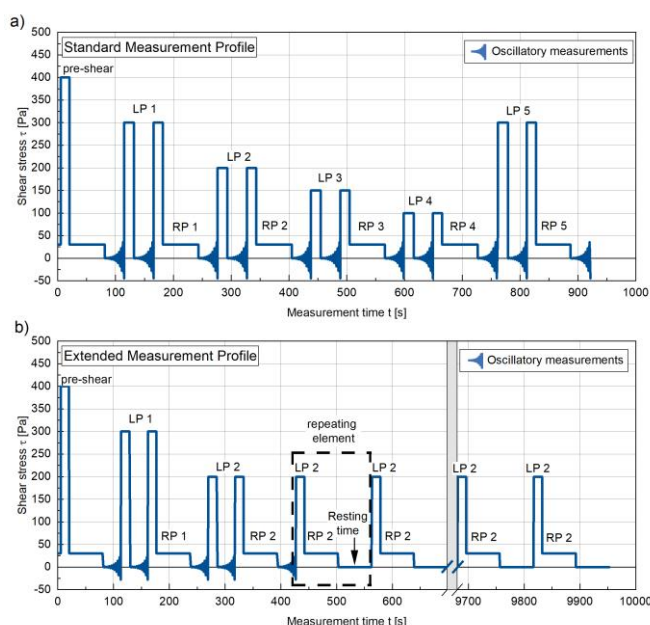


Figure 1 Rheological measurement profiles consisting of loading phases (LP) and recovery phases (RP). (a) Standard measurement profile performed with fresh and remixed samples and (b) extended measurement profile for continuously tested samples (Here: Example of a shear stress of 200 Pa during LP and a resting time of 1 min between RP and LP).

Selected cement suspensions were investigated with time-resolved synchrotron tomography at the TOMCAT-Beamline X02DA of the Swiss Light Source (SLS), Paul Scherrer Institute (Villigen, Switzerland) to quantify the change of microstructure induced by shear. Cement suspensions with a volume of 10 ml and a volumetric content of solids of 0.4 were prepared in a 25 ml glass vial. Water was added to the dry cement powder, and the suspension was manually stirred with a spatula for 60 s

before being mixed on intensity level 7 with a VORTEX 2 laboratory shaker (IKA, Staufen i.Br., Germany) for 60 s. This mixing routine was repeated 11:30 min after water-cement contact to reach an intensive remixing. The samples were investigated 15 min after water-cement contact in a Couette geometry with an outer cylinder of 10 mm diameter and an inner cylinder of 4.9 mm diameter. The height of the outer cylinder was 10 mm. The samples were sheared, and the microstructure was imaged by X-ray tomography with a voxel size of 2.75 μm and a scan time of 0.5 s for the total sample. The technical information on the data acquisition can be found in [23].

3 Results

3.1 Reaction kinetics

The exothermal hydration process generates heat and the associated measurements give deep insight into the mechanisms and progress of the hydration process. The total heat signal is a superposition of several hydration processes of pure clinker phases. Thus, a correlation of characteristic points of the calorimetry results of cement (i.e., extremum of the calorimetry curve) to single reactions of pure clinker phases is rather complex or very challenging. However, it is commonly accepted to correlate the high heat release rate during the induction period with a domination of the aluminate reaction, and the increase during the acceleration period with the onset of C-S-H precipitation by the silicate reaction (Fig. 2a). The determined degree of hydration (Eq. 1) is shown in Fig. 2b, relating the ongoing hydration to its relative progress.

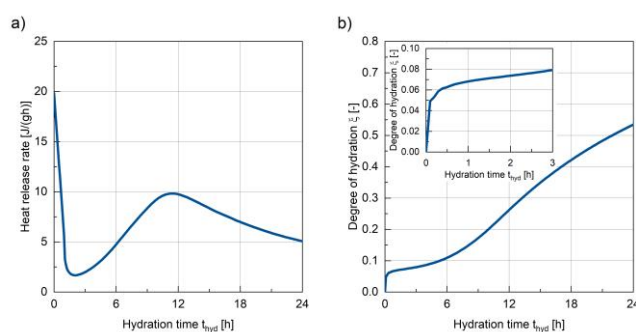


Figure 2 Reaction kinetics during hydration process as (a) heat release rate and (b) calculation of the degree of hydration ξ over hydration time t_{hyd} . Inset: Short-time evolution of ξ , considering t_{hyd} for rheological measurements.

3.2 Influence of shear history and hydration

Quantifying the influence of shear history and hydration on the rheological properties of cement suspensions represents a main goal of this study. The influence of different shear stress at various degrees of hydration during LP on the corresponding recovery behaviour during RP are presented in this section.

Constant high shear stress during LP results in an exponential decrease of the dynamic viscosity η until after approx. 1 s an equilibrium dynamic viscosity η_{eq} is reached. The stress during LP is applied for $t = 15$ s to ensure a steady state for η_{eq} where breakup and recovery of the particle network occur with the same rate. The value of η_{eq} decreases for increasing applied shear stress (Fig. 3a).

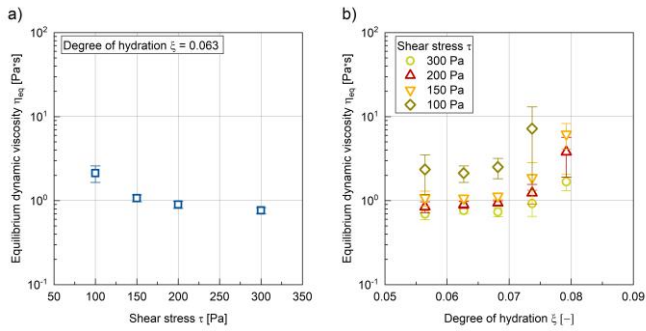


Figure 3 Equilibrium dynamic viscosity η_{eq} during high shear with the standard measurement profile. (a) Dependency on applied shear stress τ and (b) on the degree of hydration ξ .

With an ongoing hydration, η_{eq} increases linearly up to a degree of hydration $\xi = 0.068$ and non-linearly at $\xi > 0.068$ (Fig. 3b). The dependency on the applied shear stress interacts with the effect of hydration. The dependency on different shear stresses remains valid even for an increased degree of hydration. After the LP, the samples are exposed to a subcritical shear stress, a shear stress below the dynamic yield stress τ_0 (i.e., $\tau \ll \tau_0$) as to measure the change in dynamic viscosity during RP $\eta(t)$. The dynamic viscosity is chosen as an indicator of the structural state of the sample. As can be seen in Fig. 4, the restructuring behaviour highly depends on the shear history of the sample (i.e., the shear stress applied during LP; termed “pre-shear stress”; see Fig. 4a), and on the degree of hydration ξ at the onset of recovery (Fig. 4b). Lower pre-shear stress causes faster build-up of the dynamic viscosity during RP.

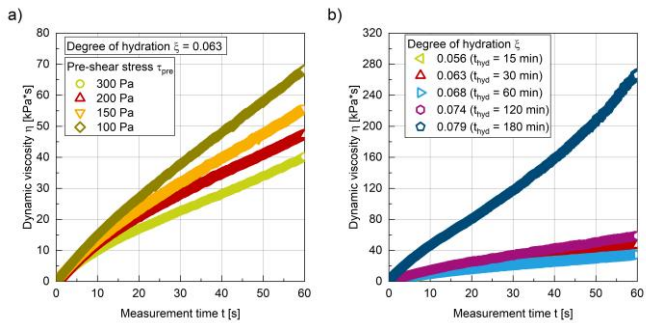


Figure 4 Increase of the dynamic viscosity during the 60 s lasting RP (see Fig. 1a) for (a) various pre-shear and (b) changing hydration states with the standard measurement profile.

Considering the influence of the different degrees of hydration ξ , at the onset of RP, the recovery curves up to $t_{hyd} = 120$ min ($\xi \leq 0.074$) show a concave shape and differ significantly from the recovery exhibited at $t_{hyd} = 180$ min ($\xi = 0.079$), which is characterized by a convex shape (Fig. 4b). Higher hydration states show a significantly faster build-up of the dynamic viscosity and suggests a correlation between the microstructure, affected by the degree of hydration, and the pronounced thixotropic properties.

The results of the continuous rheological measurement with the extended measurement profile shows, for higher resting times, an increase of η_{eq} during LP (Fig. 5a). The higher the degree of hydration, the more pronounced the increase.

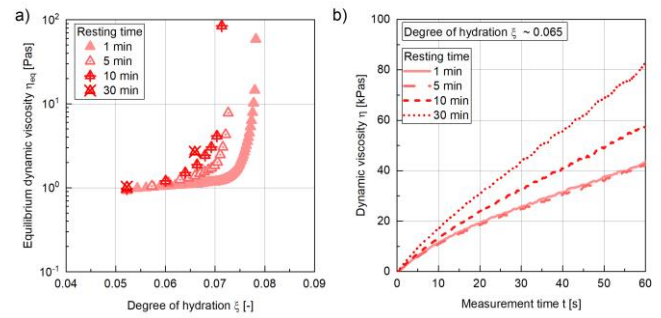


Figure 5 Results of the extended measurement profile. (a) Dependency of the equilibrium dynamic viscosity η_{eq} during LP and (b) the structural recovery with the same degree of hydration on resting time.

The influence of the resting time on the structural recovery during RP for samples with the same degree of hydration is shown in Fig. 5b. It indicates a higher recovery for increasing resting time. Thus, it is demonstrated that the resting time between the measurements as part of the shear history significantly influences the thixotropic behaviour.

Looking into the microstructure, the homogeneous particle arrangement observed at rest (Fig. 6a) having being changed after shear (Fig. 6b & 6c). Indeed, after shear, one can distinguish, in close vicinity of the inner cylinder, a darker grey circular zone in the tomography images (Fig. 6b & 6c). The particles between the inner (rotating) cylinder and the detected circle start translating in the direction of rotation. The particles at the wall of the outer cylinder, however, do not show any movement and remain in their original position relative to the outer wall. The change to darker values of the circular zone is clear proof of a higher water content compared to the neighbouring areas and represents the separation of different shear velocities as a consequence of shear banding.

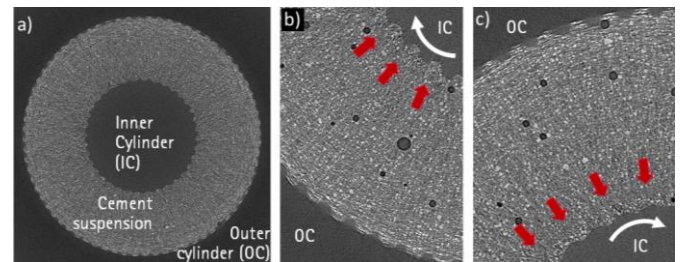


Figure 6 Results of in-situ tomography testing of a cement suspension with a shear gap of 2550 μm . (a) Overview of the unshared sample. (b) & (c) Magnification of the gap of a sheared sample (Rate-controlled start up test from resting with a constant shear rate $\dot{\gamma} = 0.01$ 1/s. The magnifications are taken 10 s after start of shearing). Shear banding and a concentrated shear zone, separating different velocity bands (thick arrows in the suspension), are identified.

The findings from the rheological investigation will be coupled in the following section with mechanisms of the flow behaviour identified by tomography tests.

4 Discussion

The experimental results show an influence of shear history and hydration process on thixotropy of cement suspensions. Lower pre-shear load and higher degree of hydration result in higher restructuring rates of the particle network. The understanding of this rheological behaviour requires access to the microstructural mechanisms.

The rheo-tomography investigation suggested that the loading phase LP applied in the experiments shown in Sec. 3 will, with very high likelihood, result in the formation of shear banding with a concentration of shearing and a non-linear velocity distribution. Between different velocity bands a circular zone occurs which separates different apparent viscosities. In the following, this zone is named as concentrated shear zone and defined as the highest changing rate of the shear gradient within the measurement gap. The agglomeration and dispersion mechanisms used so far to describe flow processes in cement suspension should be supplemented by the phenomenon of development of shear banding.

Consequently, lower viscosities result from both an increased breakup of the particle structure resulting in smaller agglomerates, and the formation of shear banding with a higher lubrication effect, either by a higher number of concentrated shear zones or thicker zones. However, in all tomography tests, only one concentrated shear zone per shear gap was detected.

Immediately at the beginning of the RP (i.e., when the shear stress has dropped to a lower, sub-critical value), the composition and thickness of the concentrated shear zone, and of the rest of the shear gap, will be unaltered. However, the equilibrium state, which has formed during high shear, is now disturbed by the sudden drop in stress. Consequently, during the RP, the concentrated shear zones change in thickness and composition as a function of time. The structural build-up and, thus, the thixotropy of cement suspensions shown in Fig. 4 are pronouncedly influenced by the formation and dissipation of the concentrated shear zones due to shear banding.

For interpreting the results of Fig. 4 the structural recovery $\eta(t)$ is quantified by a mathematical function. Since the rheological recovery phase is a creep experiment – applying a subcritical stress (i.e., $\tau \ll \tau_0$) – the relation between deformation and measurement time matches with a power-law. As the dynamic viscosity is the shear stress divided by the rate of deformation (i.e., the shear rate $\dot{\gamma}$), the power-law behaviour remains valid for the growth of the dynamic viscosity during recovery time. This yields:

$$\eta(t) = \eta_{eq} + \chi \cdot t^{n_\xi} \quad (2)$$

where η_{eq} is the equilibrium dynamic viscosity (a function of the previous shear stress), χ the structural recovery rate, and n_ξ the structural recovery exponent. The structural recovery rate χ strongly depends on the pre-shear stress, reflecting a reversible physical impact on particle structure. In contrast, the structural recovery exponent n_ξ is influenced by the hydration process. For a fixed hydration degree ξ , the structural recovery rate χ is larger for lower pre-shear load (Fig. 7a & 7b). The formation and dissipation of the concentrated shear zone can be correlated with χ , indicating a smaller thickness of the concentrated shear zone and its deviation from the bulk paste composition for lower pre-shear stress τ_{pre} . Thus, re-structuring will occur much faster, as shown in Fig. 7a.

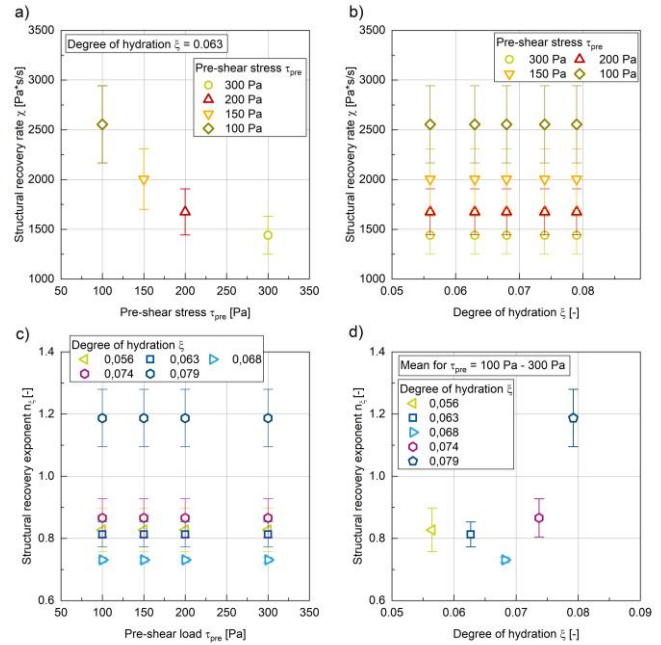


Figure 7 Parameters determine the increase of viscosity during RP (Fig. 4) (derived from the standard measurement profile). Dependency of structural recovery rate χ on (a) pre-shear stress and (b) on the degree of hydration as well as of the structural recovery exponent n_ξ (c) on pre-shear load and (d) on degree of hydration.

The dependency of the structural recovery exponent n_ξ is shown in Fig. 7c & 7d. For a low degree of hydration, n_ξ is almost constant and even shows a minimum for approx. $\xi=0.068$ followed by a sharp increase for higher degrees of hydration. This minimum is in line with results from the literature [7,12]. The remixing and high pre-shear stress destroy the interparticle bonds formed by early hydration products. During the dormant period, the cement reactivity is limited, giving another hint for low recovery of the interparticle network around $\xi \approx 0.068$ ($t_{hyd} \approx 60$ min) and thus, limited recovery and the occurrence of a minimum for n_ξ . The following increase of n_ξ can be correlated to the acceleration period characterized by the onset of C-S-H precipitation. It can be assumed that the development of concentrated shear zones is also affected by the hydration process. Precipitation of hydration products causes an increase in total solids phase content, which could reduce the size of the concentrated shear zone. In parallel, water is irreversibly bound in hydration products [13].

After the influence of previous shear stress and hydration, further effects of different resting times between the measurements (1, 5, 10, and 30 min) were investigated. Samples tested with the standard measurement profile are intensively remixed before the rheological measurement, whereas the samples of the extended measurement profile were not remixed. The remixing seems to cause smaller particle agglomerations and well-developed concentrated shear zones resulting in lower n_ξ . Conversely, the absence of remixing leads to a continuous increase of n_ξ and is related to less destruction of interparticle bonds by early hydration products and less developed concentrated shear zones. The occurrence of a minimum is not valid for samples without remixing. A decline of n_ξ is not occurring for samples without remixing.

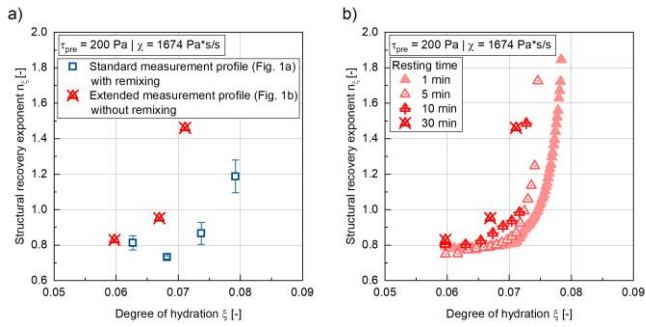


Figure 8 Influence of shear history on the structural recovery exponent n_ξ as (a) with and without remixing of the cement suspension and (b) with different resting times for the extended measurement profile.

Further, the influence of resting time (implemented between RP and LP (Fig. 1)) on n_ξ (Fig. 8b) are evaluated. Increasing the resting time speed up the increase of n_ξ , showing that the hydration process during resting time results in strengthened particle network and a concentrated shear zone with a less lubrication effects.

The influence of shear history and hydration process on development of concentrated shear zones helps better to understand the thixotropic properties of cement suspensions. The flowability of these suspensions is a function of the agglomeration and dispersion behaviour of the particles and the formation of shear banding, whereas the lubrication effect is subject to external influences.

5 Conclusion

The aim of this study was to resolve the influences on thixotropy of cement suspensions resulting from the pre-shear history and the hydration process.

Higher constant stress during the loading phase (LP) causes lower equilibrium dynamic viscosities η_{eq} . The subsequent recovery phase (RP) at subcritical loading shows lower structural recovery rates χ for high pre-shear loads.

Higher degree of hydration ξ result in higher η_{eq} during LP and higher structural recovery exponent n_ξ . Due to changes in the reaction kinetics, n_ξ shows a decline for low ξ and a further incline with higher ξ . This minimum characteristic is linked to intensive remixing before rheological investigation.

Besides the long-standing hypothesis following which the flow properties of cement suspension is controlled by agglomeration and dispersion of particles, we have identified an additional controlling mechanism in the formation of shear banding. Between bands of different velocity, the shear gradient decreases significantly in areas named concentrated shear zones. These zones are characterized by lower solids phase content and formed by shear-induced particle migration. The formation of the shear banding seems to be dependent on the level of shear stress during LP. With the transition to a subcritical stress during RP, the concentrated shear zones vanish again. This vanishing process controls the structural recovery and thus, thixotropic properties of cement suspensions. These findings help better to understand the mechanisms for precise prediction of the flow behaviour of concrete.

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