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Viscoelastic properties of fresh cement paste: measuring procedures and influencing parameters

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Abstract

Fresh cement pastes behave as viscoelastic materials below the flow onset. The measurements of viscoelastic properties of fresh cement paste provide valuable insight into the dispersion of solid particles as well as the hydration kinetics at early age and its influence on the structural evolution and solidification behavior at quasi-static conditions. Monitoring the development of viscoelastic properties of fresh cement paste using dynamic oscillatory shear measurements can also elucidate the working mechanisms of chemical admixtures. These properties are efficient indicators to guide mixture proportion design and are necessary to understand the rheology and stability of concrete. In this paper, the most common techniques, including dynamic oscillatory measurements, used to assess the viscoelastic properties of fresh cement paste are presented and discussed. The measurement challenges and their effects on the accuracy of the obtained properties are highlighted. On the other hand, the effects of high-range water-reducer, viscosity-modifying admixture, and supplementary cementitious materials are discussed. Furthermore, the use of viscoelastic measurements to assess yield stress and structural build-up of cement paste is presented.

Keywords: Viscoelastic properties; Fresh cement paste; Rheology; Measuring techniques; Rheometers

1 Introduction

Fresh cement pastes behave as viscoelastic materials. Viscoelastic materials exhibit both elastic and viscous behaviors when undergoing strains or stresses. When fresh cement paste is subjected to an external force, it responds in a manner intermediate between an elastic material and a viscous material, although for a majority of situations, the purely elastic or purely viscous assumptions are sufficiently accurate. This behavior is mainly determined by the solid content, the dispersion state of the particles, the hydration process, and the presence of admixtures and supplementary cementitious materials (SCM). Under a critical strain of the order of 10^{-4} [1,2], fresh cement paste behaves as an elastic solid-like material, as (non-linear) visco-elastic when this critical strain is exceeded, and as a viscous fluid once the strain associated to the yield stress is surpassed. This small critical shear strain ensures the integrity of the internal microstructure, i.e. it can be used as a non-destructive testing method. The critical strain accounts for the extent of the linear viscoelastic domain (LVED), indicating the deformation capacity of the connections between solid particles, which are due to the bridges of early hydration products, as shown in Figure 1 [3].

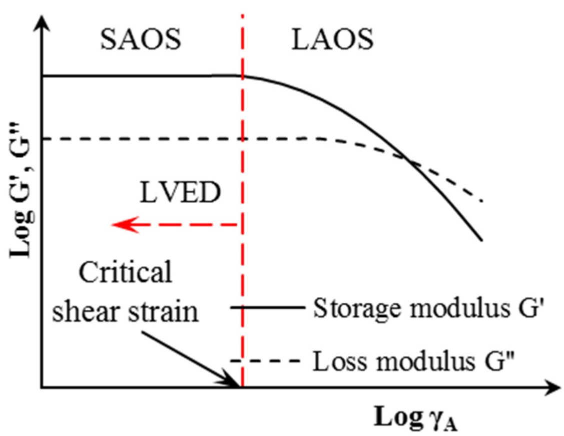

Figure 1. Schematic illustration of the variations of G' and G" in a strain sweep test.

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Similarly, if the applied shear stress is sufficiently low, fresh cement paste exhibits a viscoelastic solid-like behavior, resulting in an undisturbed material. When the applied stress is higher than the critical stress corresponding to yield stress, the fresh cement paste shows viscoelastic behavior and starts to flow, but predominantly viscous, exhibiting a decrease in viscosity. This viscoelastic behavior is time-dependent and changes during the induction period of the cement hydration stage, as the visco-elastic properties continuously increase due to flocculation and hydration [4-7] and is a useful index of the structural build-up state of the cementitious system.

The viscoelastic properties are closely related to many engineering challenges. Initially motivated by a need to understand the behavior of vibrated concrete, the oscillatory shear response of fresh cement pastes showed that it can provide thorough insight into the solid-like properties of the matrix below the yield stress. Indeed, the viscoelastic properties of fresh cement paste are of great importance in guiding mixture proportioning, evaluating flowability, simulating and evaluating pumpability, and assessing constructability during 3D printing processing. Indeed, solid state properties of fresh cement paste and their evolution in time are related to formwork pressure [8,9] and buildability in 3D printing [10, 11]. In additive manufacturing applications, the structural kinetics should be properly controlled to balance buildability and interlayer bonding [12-16], and ensure good shape retention. These properties are also important for the stability and homogenization of granular particles and fibers in the fresh cement paste matrix, especially in flowable concrete. After casting, the fresh cement paste should develop enough rigidity to resist gravity-induced sedimentation or creep of the granular particles and ensure stable concrete. Understanding the viscoelastic properties of fresh cement paste can be helpful to better understand the behavior of concrete and control its stability. Several measurement techniques can be used to investigate the viscoelastic properties of fresh cement paste. This includes creep, stress relaxation, and oscillatory tests [1, 2, 15-17]. However, special attention has been given to oscillatory shear tests to study the rheology of fresh cement paste.

2 Theoretical background for viscoelastic measurements

Ideal elastic (solid) materials deform instantaneously when subjected to a stress, where the energy invested in deforming the material is fully stored. In this case, the elastic strain is related to the stress applied. If the relationship between stress and strain is linear, Hooke's law can be applied. The deformation of a viscoelastic material is not proportional to the applied stress because of energy dissipation. The viscoelastic properties of suspensions arise from particle interactions [4, 5]. At rest, a preferred arrangement of the particles to fulfill the minimum energy requirement prevails. Subjecting the material to very small perturbations around the rest state can allow the measurement of its elastic response. Also, under these conditions (i.e. very small strains), the behavior can be assumed linear and can be analyzed using the linear viscoelasticity theory.

In general, two different methods can be used to determine linear viscoelastic properties of fresh cement paste: static and dynamic rheology. The static methods are either creep tests at constant stress or relaxation tests at constant strain. This involves applying a step change in stress (or strain) to capture the resulting strain (or stress). On the other hand, the dynamic methods consist of applying a strain that is varied sinusoidally with time at a certain frequency and observing the resulting stress. Dynamic rheology can allow the application of micro shear strains to monitor the viscoelastic properties and the structure's recovery in a non-destructive manner. The use of oscillatory methods increased considerably thanks to the development of sophisticated rheometers available for processing the input and output signals at different frequencies ranging between 10^{-3} to 10^{3} s^{-1}, hence a time spectrum from about 10^{3} to 10^{-3} s can be covered [18, 19]. The principles of the oscillatory tests can be illustrated using a two-plates-model in as shown in Figure 2 [3].

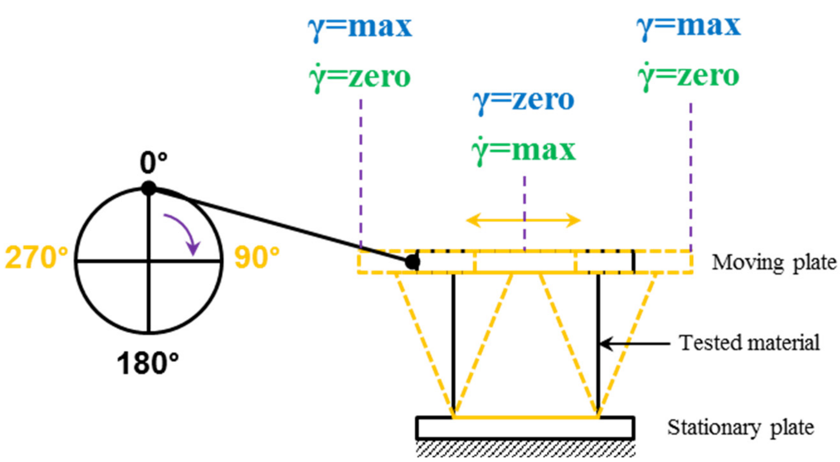

Figure 2. The two-plates-model and oscillatory shear, adapted from [3].

In this system, the tested material is located between two plates where the lower plate is stationary while the upper one is oscillating by means of a drive wheel and a pushing rod. One complete rotation of the driving wheel from 0º to 360º corresponds to a single shearing cycle. The performed number of shearing cycles per unit time represents the frequency. The deformation of the material between the two plates is maximum (i.e. maximum shear strain) and the speed is zero (i.e. zero shear rate) at positions of 90° and 270°. In the case of a perfect solid material, Hooke's law applies, as follows:

$$\tau = G \cdot \gamma \tag{1}$$

When fresh cement paste is subjected to oscillatory shear in a parallel plate rotational rheometer, small amplitude oscillations (γ_0) imposed on one plate are transmitted to the other as a torque, which is generally out of phase with the applied oscillations by a phase angle δ, which is between 0 and 90°. The shear stress response will be a function of both *G'* and *G"*, as follows:

$$\tau(t) = G^{'}\gamma_0\, sin(\omega_0 t) + G^{''}\gamma_0\, cos(\omega_0 t) \tag{2}$$

where *G'* is the storage modulus (Pa) and *G"* is the loss modulus (Pa). The storage modulus represents the elastic component of stress and the loss modulus represents the viscous component of stress.

The phase angle δ is the ratio of the viscous and the elastic portions and it can be calculated as follow:

$$\tan(\delta) = G^{''}/G^{'} \tag{3}$$

In the case of viscoelastic materials, both moduli are nonzero, and the phase angle lies between 0 and 90°.

In the case of purely elastic materials, the stress and strain waves are in phase, resulting in a phase angle δ = 0° and G" = 0. For purely viscous materials, the stress and strain are fully out of phase by an angle of 90° and G' = 0. Therefore, dynamic rheology methods can help distinguish between the elastic and viscous properties of fresh cement paste. Subjecting a viscoelastic material to stress which is varying sinusoidally with time at a certain frequency may result in strain that is not in the same phase as the applied stress, hence resulting in a phase lag between strain and stress [3].

3 Viscoelasticity versus thixotropy

Both thixotropy and viscoelasticity refer to reversible time effects [20-22]. The distinction between them can be highlighted in the stress response due to a sudden decrease in shear rate from the initial state to equilibrium, as shown in Figure 3. A normal viscoelastic fluid would react by a decrease in stress to the equilibrium plateau (i.e. stress relaxation). During stress relaxation, the microstructure recovers to its new steady state level. In contrast, under similar conditions, the shear stress of an inelastic thixotropic material would drop instantaneously to a lower value and evolves gradually in the opposite direction to its new steady state. The most general response would be a combination of the two types, i.e. a sudden drop followed by a gradual stress relaxation and finally a slow and gradual increase in viscosity. Because of the time-dependent viscosity recovery, these two cases are considered thixotropic [20].

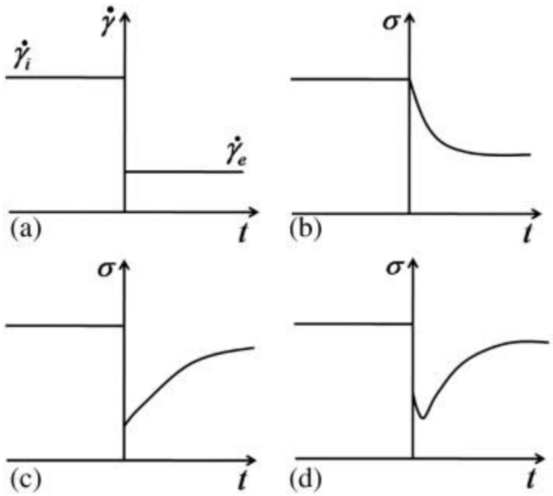

Figure 3. Responses to a sudden reduction in shear rate (a): b) viscoelastic; c) inelastic thixotropic; d) most general. Reproduced with permission from [20].

4 Viscoelastic properties of fresh cement paste

The amplitude oscillatory test is carried out by increasing the induced strain while keeping the frequency at a constant value (Figure 4) to determine the critical shear strain defining the limit of the LVED. It is worth noting that frequency sweeps are not usually carried out for cement paste systems. The frequency sweep test provides information about the stability of the system. Viscoelastic measurements have to be carried out on stable suspensions and frequency of the order of 1 Hz [1,32] has been shown to provide reliable measurements when performing strain sweep or time sweep measurements.

Fresh cement paste exhibits a linear viscoelastic behavior until a certain critical shear strain. Within the LVED the storage modulus (G') is independent of the shear strain. The determined critical shear strain ranges between 0.0025% and 0.2% depending on the mixture formulation [23]. Indeed, two domains could be identified from the obtained variation of *G'* and *G"* with applied shear strain, as shown in Figure 1 [3].

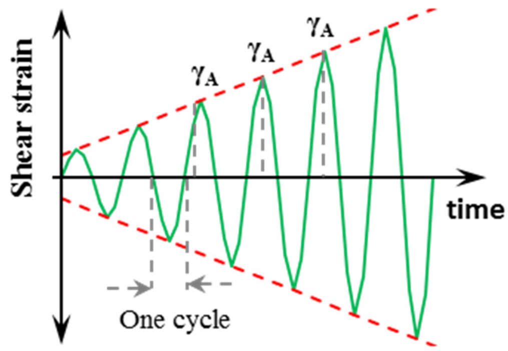

Figure 4. Schematic illustration of the strain sweep test at a fixed frequency [3].

In the first domain, which corresponds to low strain values, the storage and loss moduli are constant and independent of the value of applied strain (i.e. linear viscoelastic domain, LVED). On the other hand, for strain values higher than the critical strain value, a nonlinear domain appears where *G′* and *G″* become functions of the applied strain indicating a large amplitude oscillatory shear (LAOS). Preforming the oscillatory tests at strain values within the LVED (i.e. small amplitude oscillatory shear, SAOS) can ensure the microstructure is not disturbed, enabling non-destructive measurements. The critical strain value defining the LVED corresponds to the strain value at which the *G′* or *G″* values begin to deviate noticeably from the preceding constant values.

5 Applications of viscoelastic measurements in cementitious materials

5.1 Static yield stress

The widely used approaches to assess the structural build-up of fresh cementitious materials include creep recovery test and SAOS test. Whereas, the static yield stress and creep recovery tests are destructive measurements, the SAOS test is a non-destructive method. The application of shear stress below a certain critical value ensure viscoelastic solid-like behavior. Indeed, the critical stress is considered to mark the solid-liquid transition of the material. The critical stress corresponding to this viscosity bifurcation, i.e. where viscosity decreases, can be assumed as the static yield stress. It is well established that measures of critical strain by LAOS are found to support this statement. This value is reported to be in good agreement with the static yield stress determined using the creep recovery test [24, 25].

The shear rate-controlled method, i.e., constant shear rate method or stress growth test, is the most popular testing mode used to evaluate the static yield stress. The commonly used shear rate ranges from 0.001 s^{-1} to 0.05 s^{-1} for cement paste [26,27]. In general, lower static yield stress and less time to reach the peak value were recognized for higher shear rates. The selection of an appropriate shear rate is essential to ensure successful measurement. Similar evolutions of static yield stress and storage modulus are generally obtained [28].

5.2 Structural build-up

The conventional SAOS test, referred as the time-sweep test, can be used to monitor the structural build-up of cement paste. The application of a continuous sinusoidal shear strain within the LVED allows for the determination of the responses of the material, including storage modulus (*G*′), loss modulus (*G*″) and phase angle (δ). Based on the principle of the SAOS test, Mostafa and Yahia [17] defined two parameters, i.e., percolation time and rigidification rate, to quantitatively characterize the structural build-up of cementitious paste. These two parameters provide reliable indicators to describe the structural build-up of cementitious paste considering the physical and chemical points. Furthermore, the authors [29] proposed a semi-empirical model to correlate these parameters with the microstructural characteristics. It is reported that different trends of evolution of structural build-up of cement paste can be concluded by stress growth test and SAOS test.

Roussel et al. employed dynamic rheology to isolate the structuration due to the colloidal network from the one due to chemical hydration of 0.40 *w/c* suspensions [30]. For this, a shear strain of 0.50%, which is larger than the smallest critical strain value (0.03%), was applied to break the C-S-H bridges between cement particles. In addition, it was found that the frequency of the applied oscillations should be sufficiently high to reduce the influence of C-S-H nucleation when the system reaches a zero-strain rate configuration. This critical frequency is 0.2 Hz. Below this value, the complex modulus increases because there is no equilibrium between destruction and nucleation of CSH. Between 0.2 Hz and 5 Hz, a steady state is reached which depends on the frequency showing that CSH contributes to the strength of the system. Above 5 Hz, the complex modulus does not depend on frequency: the oscillations frequency is high enough to prevent CSH formation.

6 Influencing mixture parameters

6.1 Water-to-cementitious ratio

The water-to-cementitious materials ratio (w/cm), which is inversely related to the solid content, is a significant parameter affecting the rheology of cementitious materials. Lower w/cm indicates higher solid particle volume fraction, hence reflecting lower interparticle distance. Accordingly, the critical strain associated with the CSH network decreases, i.e. shorter LVED (i.e. less dispersed) while the storage modulus increases [17, 28, 31]. The variation of storage G′ and loss G″ moduli determined from strain sweep measurements for different cement mixtures containing silica fume and HRWR are shown in Figure 5. As can be observed, below a critical strain, all mixtures exhibited a LVED characterized by a constant G′ and G″. At shear strain values lower than this critical strain, the suspension can recover elastically and acts as a solid structure (i.e. undisturbed). However, the increase in shear strain beyond the critical value resulted in decreasing shear moduli, reflecting some destruction of the network between cement particles [12-17]. Furthermore, increasing w/cm ratio reduces the storage modulus at LVED and viscoelastic yield stress of cement paste, whereas higher w/cm increases the critical strain. However, the increase in w/cm from 0.35 to 0.40 did not show a significant change, while a relatively high critical strain is observed for the cement paste with w/c of 0.45 [32]. The value of this critical strain depends on the mixture composition. Its value is around 0.0025% for flocculated suspensions and increases with the presence of HRWR (almost 0.20% with the case of saturation dosage).

For a given rest time, the increase in solid fraction resulted in higher rigidity, i.e. higher G’ [17]. Furthermore, the partial replacement of cement with 4% SF resulted in a higher nucleation effect of SF and lower inter-particle distances, leading to an increase in storage modulus. On the other hand, incorporating HRWR at a dosage corresponding to 100% of the optimum dosage resulted in a reduction in static yield stress values of the 0.35 w/c suspensions. Higher storage modulus and lower phase angle indicate higher stiffness of the material. Moreover, higher solid fraction allowed a faster formation of the elastic percolated network reflected by lower percolation time [17]. The percolation time reflects the formation of internal microstructure due to the colloidal interactions, and the rigidification rate indicates the structural evolution because of the chemical hydration.

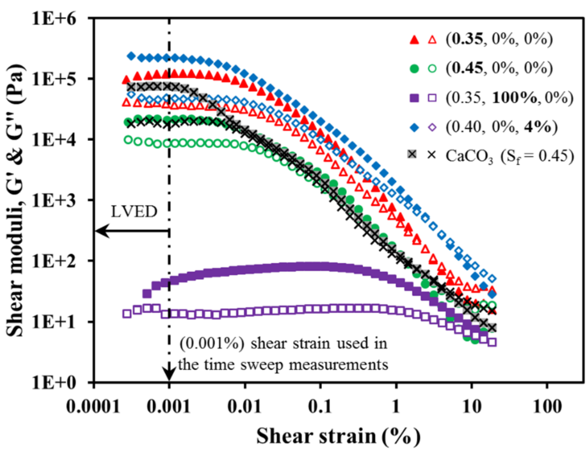

Figure 5. Variations of shear moduli with shear strain between 0.0001% and 20% and at angular frequency of 10 rad/s. (G’: full symbol, G”: empty symbol), the two % values indicate the HRWR and silica fume percentages. Reproduced with permission from [17].

6.2 Cementitious material

Presently, there are limited reported studies on the influence of SCM replacement rate and type on storage modulus evolution, which presents a knowledge gap. The partial replacement of cement by silica fume (up to 4%) was found to increase G’ and reduce percolation time, hence reflecting higher kinetics of structuration [17]. This was attributed to the presence of higher number of colloidal particles with high specific surface. Another study found similar effects with silica fume, as well as zeolite – at additions up to 10% they were both found to increase G’ evolution due to their increased reactivity [33]. In contrast, limestone at the same addition was found to have very little effect on G’ due to its inert nature. Fly ash and ground granulated blast furnace slag at 30% cement replacement were found to decrease the value of G’ overall but exhibited comparable rates of G’ increase at various early stages of hydration (i.e. during the induction period and beginning of the acceleration period) compared to the control [34]. However, Jiao and De Schutter reported that the replacement of cement by fly ash decreased the storage modulus at LVED and viscoelastic yield stress because of the increase in the interparticle distance. On the other hand, the replacement of cement by fly ash resulted in the same critical strain of pure cement paste [32].

Apart from SCMs, the effect of mineral admixtures on the viscoelastic properties of cement have been investigated via SAOS, most commonly nanoclay. Nanoclays are found to decrease critical strain measured by amplitude sweeps, which provide evidence that nanoclays are increasing the interconnectivity of the fresh-state microstructure [35]. However, conflicting results have been reported on cement pastes modified with attapulgite nanoclay, where some studies have measured an overall decrease in G’ [36] while others have measured higher G’ initially before eventually falling below the control over time [29, 37-39]. These discrepancies may be due to various factors, including shear history condition and dispersion/processing, and the type of clay will also have an impact. More SAOS investigation is needed to further understand the effect of nanoclay, and other mineral admixtures and binders, on G’.

6.3 Admixture

The critical strain of cement paste gradually increases with the concentration of polycarboxylate ether (PCE) superplasticizer, which possibly can be attributed to the interactions and entanglement of PCE molecules adsorbed onto the solid particles [32]. By contrast, cement pastes with low PCE additions exhibit an increase in the viscoelastic yield stress, while higher PCE additions significantly decrease the storage modulus at LVER and viscoelastic yield stress of cement paste.

Viscosity-modifying agents (VMA) are widely used to improve the viscosity and stability of fresh cement paste. Typical organic VMAs include cellulose ether and Welan gum, while nano-silica and nano-clay are two kinds of representative inorganic VMAs. Kappa carrageenan has been used as a new biopolymer viscosity admixture for cement-based materials. The addition of VMA considerably decreases the critical strain and increases the storage modulus of fresh cement paste [25, 38, 39]. The long chains in VMA molecules have a great potential to physically absorb the free water in the interstitial solution, hence increasing the viscosity of the system [29, 38, 39]. On the other hand, attractive forces develop between the VMA chains and promote the formation of gel structure and inter-particle links. However, Ma et al. reported that the use of diutan gum significantly decreased the storage modulus G’[36].

6.4 Temperature

The ambient temperature has a significant influence on rate of chemical hydrations, and thus the thixotropic structural build-up of cement-based materials [7, 17, 20, 21]. Indeed, increasing temperature promotes the dissolution of clinker minerals, hence increasing ionic concentration and the production of a large number of fine particles and higher attractive forces between solid particles.

Mostafa and Yahia reported that the increase in temperature can result in higher kinetics of structural build-up at rest of cement suspensions [17]. This is reflected by shorter percolation times due to the enhancement in the frequency of Brownian collisions, as well as higher rigidification rates due to the higher rate of hydrates formation and stronger chemical stiffening of the formed elastic network [17]. The increase in temperature can also enhance the rate of diffusion and decrease the solvent viscosity, which eventually increases the rate of collisions. This can explain the shorter percolation time measured at higher temperature [17, 39-43].

6.5 Smart materials / Electrical and magnetic field

Recently, the development of smart cementitious composites has introduced stimuli-responsive additives that can be used to actively control the rheological behaviour of cement paste and can either trigger the stiffening of the concrete or ease its flow. These additives can be activated by a stimulus that can be either electrical or magnetic and can be redox-responsive polymers [44], magnetic nanoparticles such as Fe_3O_4 [45 - 49] or the combination of both [44]. The effect of magnetic field on rheology of fresh cement paste containing nano-Fe_3O was indeed investigated [45 - 52]. Jiao and De Shutter found that the incorporation of nano-Fe_3O_4 particles results in an increase in the storage modulus at LVER, critical strain, and viscoelastic yield stress [32]. This is probably due to the magnetic field-induced structures of the iron particles, hindering motion of the cement particles, and the improvement of cohesive bonding between cement particles by nanoparticle. For those stimuli-responsive cement pastes, the description of the effect of the stimulus on the fresh-state rheological behaviour is needed in order to assess the set-on-demand / fluid-on-demand properties of these smart materials. Nair and Ferron [48, 49] reported that the storage modulus of fresh cement paste containing carbonyl iron particles showed a significant field sensitivity to an external magnetic field. This can be a useful approach in controlling the evolution of structural build-up of cement-based materials. The study of the visco-elastic properties of these cement pastes under specific stimulus (i.e. electrical current or magnetic field) have been performed using dedicated devices that can be adapted on rheometers to apply different stimulus during an oscillatory rheological test carried on with parallel plates geometry. These devices are generally called magneto-rheological device for magnetic responsive paste where a magnetic field (generally ranging from 0 and 1T) [48, 49] and electro-rheological device where an electric potential is applied on the upper plate for redox responsive paste [44]. Commonly, these devices have been used to carry out small amplitude oscillatory shear measurements. Therefore, strain sweep measurements are performed to obtain the linear visco-elastic region or time sweep tests can be carried out to determine the structural build-up of the active cement pastes under applied stimulus.

7 Concluding remarks

This paper discusses the viscoelastic properties of fresh cement paste mixtures based on the measurement of the critical strain and the storage modulus at LVED from SAOS and other types of rheometry tests. The critical strain is the end of the LVED, indicating the deformation capacity of the connections between solid particles, which are due to the bridges of early hydration products. These properties are mainly influenced by the solid content (w/cm), the presence of admixtures and dispersion state of the particles, the hydration process, and supplementary cementitious materials (SCM). Under a critical strain, fresh cement paste displays elastic solid-like behavior. This small shear strain ensures the integrity of the internal microstructure and SAOS is a non-destructive testing method. Increasing w/cm reduces the storage modulus in the LVED. Higher critical strain is observed for cement paste with relatively high w/cm or incorporating high-range water-reducer admixtures. The addition of HRWR PCE type increases the critical strain of cement paste and reduces the rigidity of the matrices. On the other hand, the effect of supplementary cementitious materials depends mainly of the type and reactivity of SCM. The critical strain of fly ash cement paste is in the same order to that of pure cement paste. Nanoclays are found to decrease critical strain, while conflicting results have been reported on the storage modulus. However, the addition of nano-Fe_3O_4 particles increase of critical strain, which can possibly be attributed to the modification of cohesive bonds between cement particles by the nanoparticles.

Authorship statement (CRediT)

Ammar Yahia: Conceptualization, Methodology, Writing – original draft, Writing – review and editing, Supervision.

Arnaud Perrot: Conceptualization, Methodology, Writing – original draft, Writing – review and editing.

Dimitri Feys: Conceptualization, Methodology, Writing – original draft, Writing – review and editing.

Kamal Khayat: Writing – original draft, Writing – review and editing.

Mohammed Sonebi: Conceptualization, Writing – original draft, Writing – review and editing, Project administration.

Shiho Kawashima: Writing – original draft, Writing – review and editing.

Wolfram Schmidt: Conceptualization, Writing – original draft, Writing – review and editing.

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