

INFLUENCE OF VISCOSITY-MODIFYING ADMIXTURES ON THE THIXOTROPIC BEHAVIOUR OF CEMENT PASTES

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ABSTRACT:

Water soluble polymers such as cellulosic or starch ethers are often included in the mix-design of Self Compacting Concretes (SCCs) in order to improve their stability and robustness. The stability, including resistance to liquid-solid separation and sedimentation, may be attributed to the increase of the viscosity of the liquid phase due to the thickening effect of the polymer. The later is then referred to as a Viscosity-Modifying Admixture (VMA). In the present study, we consider the influence of VMAs on the rheological properties of the material at cement scale level. In particular, the change in the thixotropic properties of the cement paste due to the inclusion of VMA is investigated. It is found that addition of VMA significantly enhances rebuild-up kinetics at rest following shearing at high shear rate. The influence of VMA on the steady state rheological properties is also considered. As reported in the literature, the yield stress is found to monotonically increase with VMA content, while the consistency presents a minimum indicating the existence of an optimum value of the VMA for which the workability of the cement paste is maximum.

ZUSAMMENFASSUNG:

Wasserlösliche Polymere wie Zellulose- oder Stärkeether werden häufig in die Herstellung von Mischungen mit selbstverfestigendem Beton miteinbezogen, um deren Stabilität und Robustheit zu verbessern. Die Stabilität, einschließlich des Widerstandes zur Flüssigphasen-Feststoffphasentrennung und zur Sedimentation, kann der Viskositätssteigerung der flüssigen Phase aufgrund des Verhärtungseffektes des Polymers zugeschrieben werden. Dieser Zusatz für den Verhärtungseffekt wird als viskositätsmodifizierende Beimischung (VMA) bezeichnet. In dieser Arbeit wird der Einfluss von VMAs auf die rheologischen Eigenschaften des Materials im Zementzustand betrachtet. Insbesondere wird die Änderung der thixotropen Eigenschaften des Zements aufgrund der VMA-Inklusionen untersucht. Die Zugabe von VMA erhöht wesentlich die Wiederherstellungskinetik im Ruhezustand nach einer Scherung mit einer hohen Schergeschwindigkeit. Der Einfluss von VMA auf die stationären Eigenschaften wird ebenfalls untersucht. Im Einklang mit Literaturangaben nimmt die Fließspannung monoton mit dem VMA-Inhalt zu. Die Konsistenz nimmt dabei ein Minimum an, was die Existenz eines optimalen VMA-Wertes für die optimale Verwendung der Zementpaste andeutet.

RÉSUMÉ:

Les polymères solubles dans l'eau tels que les éthers cellulosiques, sont souvent utilisés dans la formulation des bétons autoplaçants (BAP) afin d'améliorer leur stabilité et leur robustesse. La stabilité, la résistance à sédimentation et la résistance à la séparation de phase (liquide solide) peut être attribuée à l'augmentation de la viscosité de la phase liquide qui est due à l'effet viscosifiant du polymère. Ce dernier est alors considéré comme un agent de Viscosité ou viscosifiant (A.V.). Dans la présente étude, nous considérons l'influence des ajouts de l'agent de viscosité sur les propriétés rhéologiques des pâtes de ciment. En particulier, le changement des propriétés thixotropiques de la pâte de ciment, due à l'ajout de l'agent de viscosité (AV). Il a été constaté que l'AV améliore de façon très significative la reprise de thixotropie après un cisaillement à un taux élevé. L'influence de l'AV sur les propriétés rhéologiques est également pris en compte. Comme cela a été déjà rapporté dans la littérature, le seuil d'écoulement est proportionnel avec l'AV, tandis que la consistance présente un minimum indiquant l'existence d'une valeur optimale de l'AV pour laquelle l'ouvrabilité de la pâte de ciment est maximale.

KEY WORDS: cement paste, self-compacting concretes, thixotropy, viscosity-modifying admixture

cement (g)	filler (g)	water (g)	SP (g)	VMA (g)
1000	330	300	7	2

Step	Mixing Time (min)
Cement + Filler	5
Water + SP + VMA addition	0.5
Mixing at low speed (95 rpm)	4
Mixing at low speed (165 rpm)	2

Table 1 (left):
Composition of the cement
paste [6 - 7].

Table 2:
Mixing procedure.

1 INTRODUCTION

Viscosity-modifying admixtures (VMAs) are often used in mix-design of highly fluid cementitious materials, including self-compacting concretes (SCCs), pumpable concretes, etc. to avoid solid-liquid separation and to improve the robustness of the formulation. That is the primary objective when using these admixtures. However, one can expect some influence on the rheological behaviour since addition of VMA will change the rheology of the aqueous phase and the interactions between the solid particles. This problem has been investigated in the literature, in particular by considering the combination of VMAs and Superplastizers (SPs) [1 - 7]. Phan et al. [7] showed that the influence of VMA on the rheological properties of cement pastes is actually small compared to that of SPs. This has been explained by the fact that VMAs act mainly on the liquid phase, while SPs modify the granular configuration (deflocculation), which actually dominates the rheological behaviour in such a highly concentrated suspension. The previously reported results concerning the influence of VMAs on the rheological properties of cementitious materials (the studies concerned in general cement pastes) converge to the conclusion that addition of this admixture decreases the fluidity (increases both yield stress and plastic viscosity) of the material (see for instance [5] and references therein).

A number of authors pointed out that cement pastes show thixotropic properties [8 - 12]. Moreover, even mortars and concretes can show significant thixotropy. This is particularly the case for self-compacting concretes [13-15]. Thixotropy is often a crucial property for building materials. Thixotropy may be advantageous, and sometime necessary, for the application process of the material. This is the case for instance for paints or rendering mortars. On the other hand, thixotropy may be undesirable for instance when dealing with the process of successive concret-

ing. Thixotropy may also have important consequences on the evolution of the pressure formworks [16]. The influence of mix-design of such materials on their thixotropic behaviour is then important.

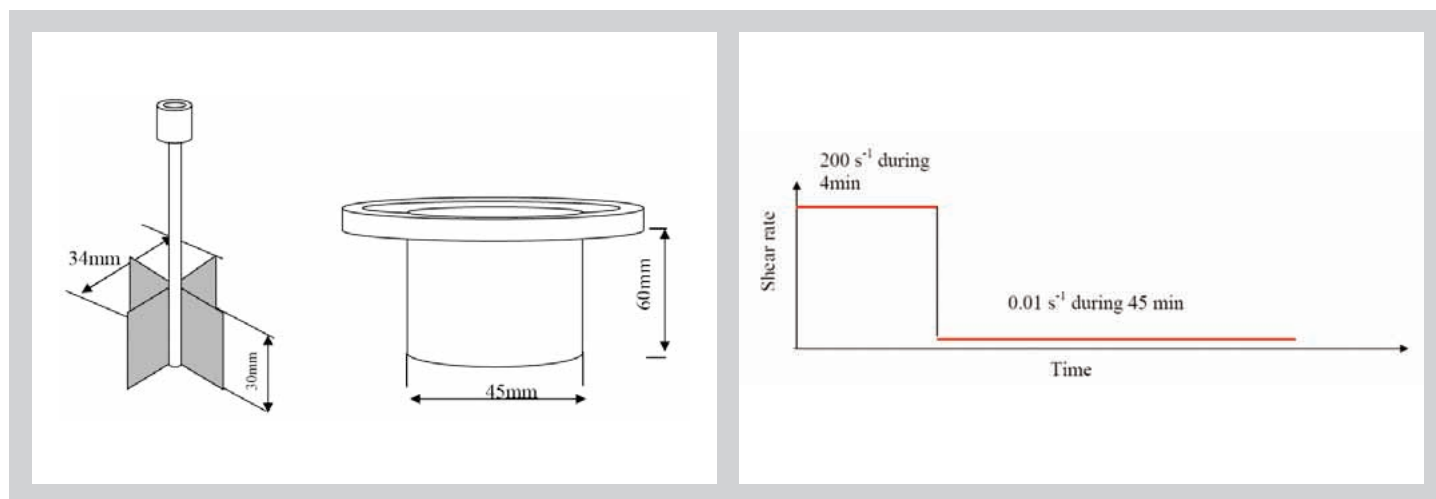
In the present study, we consider the influence of VMAs on both thixotropic and steady-state rheological properties of cement pastes. When dealing with thixotropy we consider both microstructure breakage at a high shear rates and rebuild up at rest. The results presented here show that the influence of VMA on the rheological parameters is actually more complex than reported previously, due to the fact that this admixture may play a dual role: on one hand it may increase the liquid phase viscosity, which would lead to an increase of the paste's viscosity, and on the other hand, it may lubricate the contacts between the solid particles, which would lead to a decrease of the viscosity.

2 EXPERIMENTAL PROCEDURE

2.1 MATERIALS

The cement pastes are composed of tap water, Portland cement (CEM I 52,5 PM ES CP2 from Teil in France), fillers (Ground limestone with a similar granular size distribution than that of the cement), a Polycarboxylate-type Superplastizer (Glenium 27 from Degussa) and a polysaccharide-type viscosity-modifying admixture (Foxcrete from AVEBE). The role of the SP is to disperse the cement particles that are prone to aggregation due to colloidal interactions. In the case of the SP used here the dispersion is due to both steric and electrostatic effects of the adsorbed polymer. It is shown here that the cement pastes (even without VMA) are shear thinning, indicating that we do not have complete dispersion of the aggregation for the SP dosage rate used.

The composition of the cement paste is reported in Table 1. The composition reported in this table corresponds to the reference paste,



which is used in practice to proportion selfleveling concretes. Here we consider the change in the rheological properties when varying the concentration by weight of the VMA. Then, four other pastes are prepared by increasing or decreasing the VMA dosage rate. The five cement pastes considered are then: REF (reference paste), REF-50 (obtained by dividing the VMA dosage rate by 2), REF-100 (without VMA), REF+50 (obtained by increasing the reference dosage rate by 50%) and REF+100 (obtained by increasing the reference dosage rate by 100%).

The mixing procedure consists of the same set of steps for all the pastes considered in order to improve the reproducibility of the tests. A laboratory paddle-pan mixer is used. The total duration of mixing is quite high (11.5 min) to insure homogeneous suspensions. The mixing procedure is described in Table 2.

2.2 RHEOLOGICAL MEASUREMENTS

2.2.1 APPARATUS

The rheological measurements were performed using a stress-controlled shear rheometer (AR2000 from TA Instruments) equipped with the vane geometry (Figure 1). In such 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 get a simple relationship between the measured torque/rotational velocity and the shear stress/shear rate. Vane geometry is nevertheless recognized to be appropriate for granular suspensions such as mortars [19 - 20] since slippage can be avoided and the material is sheared in volume.

The gap (distance between the periphery of the Vane and the outer cylinder) is 5.5 mm, which is more than an order of magnitude larger than the maximum size of the cement or filler particles (about 0.1 mm). The shear rate and the shear stress are inferred from the torque and the rota-

tional velocity of the vane by calibrating with a Newtonian fluid. The temperature was regulated at 25°C (to within 0.1°C) thanks to a circulating water system. In order to prevent evaporation of the paste's water the measurement system was sealed.

2.2.2 MEASUREMENT PROCEDURE

All the rheological measurements were undertaken during the induction period during which the hydration rate of the cement is very low and may have non-significant influence on rheology. In order to check that this was actually the case, two successive and same rheological measurements with the same sample were performed, indicating that there was no irreversible transformation (hydration) of the material up to 2 hours. The fact that the induction period is so long is due to the presence of the organic phases, including the SP and the VMA.

Before starting measurements, the samples are pre-sheared at 200 s⁻¹ and then held at rest during 1 min. The steady state rheograms (shear stress versus shear rate) are determined by subjecting the material to cycles of increase-decrease of shear rate. An approximate steady state is obtained within 4 - 5 loops. The flow curves reported here correspond to the descendant branch of the last loop. For each paste, the rheological parameters reported here correspond to average values over at least five tests undertaken using a freshly prepared sample.

The transient behaviour is considered separately in details. Figure 2 represents the flow path used to investigate the break down at high shear rates and rebuild up at rest. In order to mimic rebuilding at rest (to fit the precise definition of thixotropy), the material is subjected to a very low shear rate (0.01 s⁻¹) during this period.

Figure 1 (left): Rheological measurement system: a) vane, b) inner cylinder.

Figure 2: Flow path used to consider transient properties (thixotropy), including breakdown at a high shear rate and rebuild up at rest (very low shear rate).

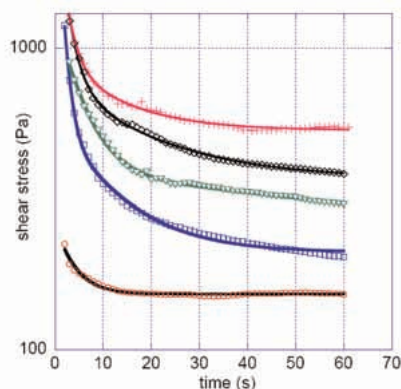


Figure 3 (left): Evolution of the stress versus time for different VMA dosage rates at a relatively high shear rate (200 s^{-1}), representing the microstructure break down. The continuous lines correspond to the best fit with the sum of two exponentials ($\circ$: REF-100, $\square$: REF-50, ∇ : REF, $\diamond$: REF+50, $+$: REF+100).

Table 3: Characteristic times for breakdown and that of rebuild up for different dosage rates of VMA.

3 RESULTS AND DISCUSSION

3.1 THIXOTROPIC BEHAVIOUR

Figure 3 represents the temporal evolution of the stress when the paste is subjected to a relatively high shear rate (200 s^{-1}), corresponding the breakdown of the microstructure. As it can be seen in Figure 3, the breakdown kinetics can be fairly well fitted by the sum of two exponentials in the case of pastes containing VMA. The correlation coefficient of the fit is in all cases very close to 1 (0.999). For pastes without VMA a simple exponential decay turned out to be enough to account for the breakdown kinetics.

The breakdown kinetics of the pastes devoid of VMA can be understood in terms of a competition between shear-induced breakage of cement and filler aggregates and Brownian induced aggregation of the colloidal part of the particles. In the presence of VMA, the polymer is subject, on one hand, to alignment and disentanglement under flow and, on the other hand, to relaxation towards maximum entropy due to Brownian motion. Due to the very high molecular weight of the VMA polymer (order of millions), its average relaxation time would be quite high. One can then assume that the breakdown dynamics is governed by two main characteristic times. Then it is straightforward to show, using a basic thixotropy model, that the temporal evolution of the stress can be modelled by the sum of two exponentials in agreement with the experimental results reported in Figure 3.

A large number of more or less sophisticated models for thixotropy have been reported in the literature [17 - 19, 22, 23]. To deal with thixotropy the simplest model has to contain at least a parameter λ that characterises the degree of interconnection of the material's microstructure at a given time and shear rate. The precise physical meaning of λ depends upon the actual microstructure evolving under flow and that has significant effects on the measured rheological

	g (s)	p (s)	r (s)
REF-100	3.77	-	2382.1
REF-50	1.3	11.64	1062.2
REF	0.77	7.5	760.3
REF+50	2.2	17.95	642.3
REF+100	1.95	12.43	555.1

property (yield stress, apparent viscosity, etc.). In general it is assumed that $\lambda = 0$ for a fully broken down microstructure and $\lambda = 1$ when the microstructure is fully built up. The evolution of the parameter λ is governed by the competition between the microstructure rebuild up, that takes place with a characteristic time τ , and the microstructure breakdown whose kinetics can be assumed to be proportional to the shear rate.

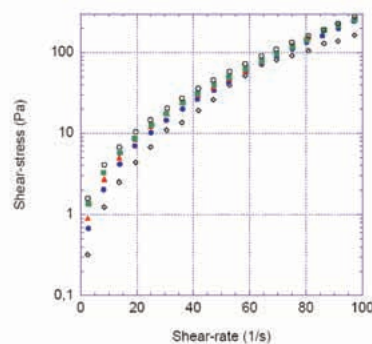
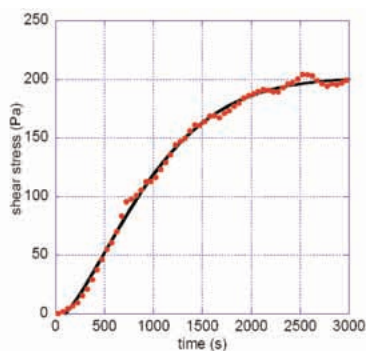
Our experimental results (Figure 3), as discussed above, strongly suggest that the rebuild up kinetics would be governed by at least two different characteristic times τ_g and τ_p , corresponding respectively to the granular and the polymer (VMA) average relaxation times. The fact that we have only one characteristic time to account for breakdown kinetics of the paste without VMA supports this hypothesis. We can then write down the following kinetic equation for the structural parameter λ :

$$\frac{d\lambda}{dt} = \frac{1}{\tau_g} + \frac{1}{\tau_p} - \lambda \dot{\gamma} \quad (1)$$

This corresponds to the simplest model for thixotropy as proposed many years ago by Moore [23]. In particular; this simple model does not take into account eventual shear-induced aggregation. More sophisticated model can be used [18, 23], however Equation 1 is enough for a qualitative interpretation of our experimental results.

For a given shear rate, the stress σ can be assumed to scale to a first approximation with the parameter. That is $\sigma = \alpha \lambda = \alpha (\lambda_g + \lambda_p)$ where α where $\cdot$ is a constant and the indexes g and p refer respectively to the granular and polymer contributions to the degree of microstructure interconnection. Solving Equation 1 leads then to the transient behaviour of the stress:

$$\sigma = \sigma_g + \sigma_p = \sigma_g^0 + \left(\sigma_g^0 - \sigma_g^\infty \right)^{-t/\tau_g} + \sigma_p^0 + \left(\sigma_p^0 - \sigma_p^\infty \right)^{-t/\tau_p} \quad (2)$$



where the superscripts o and ∞ refer respectively to the initial and steady state values of the stress.

The best fit of the experimental stress relaxation leads to the values of the relaxation times for the different pastes considered. They are reported in Table 3. One can notice that the polymer relaxation times are an order of magnitude higher than the granular ones. This can be expected since the molecular weight of the polymer is very high. The evolution of the relaxation times for microstructure breakage is not monotonous. One can observe a minimum value for the reference paste. The reason of such evolution of microstructure breakdown kinetics as a function of VMA content is not clear. This point deserves more investigation. Figure 5 represents a typical temporal evolution of the stress (representing the microstructure rebuild up) when the paste is held at rest (very low shear rate) following shearing at a high shear rate (200 s^{-1}). The example in Figure 4 corresponds to the reference paste. The results for the other pastes are similar.

In contrast with breakage the rebuild up kinetics cannot be described by the sum of two exponentials. In this case a stretched exponential is more suitable to fit the stress growth curves. A stretched exponential is akin to processes involving a whole distribution of relaxation times. One can understand physically such a behaviour by the fact that since the paste is subjected to a very low shear rate a full distribution of relaxation times are mobilized in the paste, including those corresponding to the grains and the polymer chains. During the break down process only relaxation times on the order of the characteristic time of flow (inverse of applied shear rate) may be mobilized. Stretched exponential response of thixotropic fluids has already been reported in the literature [18]. Once again a simple model of thixotropy, including an infinite set of independent relaxation times, can be used to account for a stretched exponential behaviour of the structure rebuild up. The best fit

of the experimental curves with stretched exponentials leads the characteristic times τ_r of the rebuild up process:

$$\sigma(t) = \sigma_{\infty} + (\sigma_o - \sigma_{\infty})^{-(t/\tau_r)^{\alpha}} \quad (3)$$

Where σ_{∞} and σ_o are respectively the equilibrium and the initial stresses.

The evolution of rebuild up characteristic time for different dosage rates of VMA is reported in Table 3. Our experimental results clearly show that the VMA speeds up the rebuild up properties of the paste. The physical interpretation of this result is not straightforward and needs further investigation. In practise VMAs are sometimes referred to as thixotropic agents, our experimental results confirm that in a more quantitative way.

3.2 STEADY STATE BEHAVIOUR

Figure 5 represents the steady state flow curves of the different pastes considered. The influence of VMA dosage rate is quite small. This has been attributed (see [7]) to the fact that the rheological behaviour of such a highly concentrated granular suspension is dominated by the granular phase, whereas VMA has influence mainly on the liquid phase. The effect of VMA dosage rate depends qualitatively upon the shear rate interval considered, in agreement with the results reported by other authors [5]. This will be discussed in more details below when considering the rheological parameters.

Figure 6 represents the evolution of the plastic viscosity, which is defined as the derivative of the shear stress with respect to the shear rate, in function of shear rate for different dosage rates. It is to be noted that we use here plastic viscosity instead of apparent viscosity (shear stress divided by shear rate) because it is the slope of the flow curve that determines the sensitivity of the stress to the variation of shear rate. Since we

Figure 4 (left): Typical temporal evolution of the stress at a very small shear rate (0.01 s^{-1}) representing the microstructure rebuild up after break down at a high shear rate (200 s^{-1}). The continuous line corresponds to the best fit with a stretched exponential.

Figure 5: Flow curves for different VMA dosage rates ($\diamond$: REF-100, $\bullet$: REF-50, $\blacktriangle$: REF, $\blacksquare$: REF+50, $\circ$: REF+100).

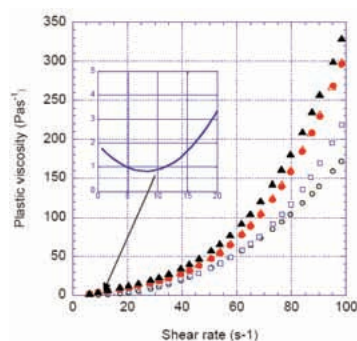


Figure 6 (left): Evolution of the plastic viscosity for different VMA contents (○: REF-100, □: REF-50, ●: REF, △: REF+50, ▲: REF+100). The inserted graph zooms in the behaviour of REF-100 at low shear rates.

Table 4: steady state rheological parameters of pastes.

	Yield Stress	Consistency	Fluidity Index
REF-100	0.09	0.062	1.72
REF-50	0.15	0.012	2.15
REF	0.35	0.013	2.16
REF+50	0.39	0.014	2.13
REF+100	0.72	0.029	1.98

deal with yield stress fluids the two types of viscosity are different. Figure 6 shows that the cement pastes containing VMA are shear-thickening (plastic viscosity increases with shear rate) throughout the whole considered shear rate interval. On the other hand the inserted zoom-in graph in Figure 6 shows that the rheological behaviour of the cement paste without VMA is not monotonous. This paste is shear-thinning at low shear rates and shear-thickening at high shear rates.

Shear-thinning behaviour is akin to flocculated suspensions and entangled polymer solutions or melts. Shear-thinning is generally attributed to shear-induced deflocculation in the former case and to polymer-chains disentanglement and alignment in the later. In our case, the shear thinning may be attributed to both phenomena. Shear-thickening is generally attributed to repulsive interactions between both colloidal and non-colloidal particles in the case of suspensions [28 - 31] and shear-induced structures in polymer solutions [30 - 31]. Both phenomena may contribute to shear thickening in our case since aqueous solutions of polysaccharide are known to exhibit shear thickening at sufficiently high shear rates [32]. The steady state rheological parameters can be determined by the best fit with a Herschel-Bulkley (H-B) model:

$$\sigma = \sigma_y + k\dot{\gamma}^n \quad (4)$$

where σ is the shear stress, $\dot{\gamma}$ the shear rate, σ_y the dynamic yield stress, k the consistency, and n the fluidity index.

In the case where the behaviour is not monotonous (paste without VMA) the shear thinning and shear thickening zones of the flow curve are fitted separately. The evolution of the dynamic yield stress as a function VMA dosage rate is reported in Table 4. The cement paste yield stress monotonically increases with VMA content in agreement with previous studies report-

ed in the literature [2, 4, 5]. Such behaviour is expected and may be attributed in particular to entanglement and intertwining of the VMA polymer chains at low shear rates.

In contrast to the yield stress, the evolution of the consistency (in the shear-thickening zone) when increasing VMA dosage rate is non-monotonous (see Table 4). The consistency first decreases when adding VMA and then increases. Such behaviour has already been reported in the literature in the case of another type of a cementitious material [33] and attributed in particular to air-entraining effects of the polymer admixture. One can also invoke the fact that the polymer may actually play an ambiguous role. On one hand, it may increase the liquid phase viscosity leading to an increase of the paste viscosity, and on the other hand, it may lubricate the contacts between the solid particles leading to a decrease of the paste viscosity. The competition between the two effects would lead to the behaviour observed here. A similar discussion has already been reported in the case of silica suspensions and cement pastes whose composition is close to ours [34].

Similarly to the consistency, the behaviour of the fluidity index (in the shear thickening zone) is non monotonous, presenting a maximum value around the reference paste (see Table 4). This may also be attributed to a double role that may be played by the VMA: on one hand polysaccharide aqueous solutions are known to exhibit shear-thickening at high shear rates [25] and, on the other hand, its lubricating effect would decrease the contribution of the granular contacts to shear-thickening.

4 CONCLUSION

The rheological behaviour of cement pastes involved in mix-design of highly fluid concretes was investigated experimentally. In particular the influence of VMAs on the rheological properties was considered. Both transient (thixo-

tropy) and steady state behaviours were investigated. Without VMA the pastes exhibited complex steady-state flow-curves, including a shear-thinning branch at low shear rate and a shear-thickening one at high shear rates. It was found that the shear-thinning branch was absent for pastes with VMA. The evolution of the steady state rheological parameters was found to be complex: the dynamic yield stress monotonically increases with VMA content, while the consistency and the fluidity index present an extremum value around the reference dosage of VMA (the one used to mix-design self-levelling concretes).

The influence of the VMA on thixotropic behaviour was investigated by considering breakdown kinetics under high rates and rebuild up at rest or very low shear rate. It was found that the breakdown kinetics was governed by two main characteristic times (the relaxation curves could be fitted with the sum of two exponentials) which differ by an order of magnitude. This was attributed to the two different constituents of paste, namely the VMA polymer and the granular phase, whose relaxation dynamics would take place at quite different timescales. On the other hand, the rebuild up kinetics was found to follow a stretched exponential-like process. This was attributed to the fact that, at rest or very low shear rate, a large set of relaxation times, including those corresponding to the polymer and the grains may be mobilized.

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