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ABSTRACT:

The rheological behaviour of a cement paste used in Self-Compacting Concretes (SCC) formulations is compared to that of an "ordinary" cement paste (OC) devoid of organic admixtures. In order to mimic the flow conditions experienced by the cement paste in the inter granular space of concretes, the rheological behaviour is investigated in a squeeze flow geometry. By considering the evolution of the squeeze force for different velocities as a function of the instantaneous distance between the discs, it is found that the behaviors of the two cement pastes are qualitatively different. For the OC pastes, the force decreases with increasing squeeze velocity for any given discs separation, indicating that the material is undergoing fluid-solid separation due to filtration of the fluid phase through the porous media made up by the grains. Such behaviour reflects the very poor flowability of the OC paste. The behaviour of the SCC paste is qualitatively different. Above a certain critical value of the speed U_c , the force is an increasing function of the speed for any given disc separation. Under these flow conditions the rheological behaviour of the material is that of a viscous, although highly non-Newtonian, fluid which corresponds to the flowability conditions of the material. For squeeze speeds smaller than U_c , the rheological behaviour of the SCC paste is similar that of OC, indicating that below this critical velocity the material undergoes solid-fluid separation corresponding then to its non-flowability zone.

ZUSAMMENFASSUNG:

Das rheologische Verhalten einer Zementpaste, die in selbstverfestigenden Betonformulierungen (SCC) verwendet werden, wird mit dem Verhalten einer „gewöhnlichen“ Zementpaste (OC) ohne organische Beimischungen verglichen. Um die Fließbedingungen, die von einer Zementpaste im Zwischenraum zwischen den Betonkörnern herrschen, nachzuahmen, wird das rheologische Verhalten in einer Quetschflusströmung untersucht. Bei der Analyse der Entwicklung der Quetschkraft für verschiedene Geschwindigkeiten als Funktion des momentanen Abstands zwischen den Scheiben wurde gefunden, dass sich das Verhalten der beiden Zementpasten qualitativ unterscheidet. Für die OC-Paste nimmt die Kraft mit zunehmender Quetschgeschwindigkeit für einen gegebenen Scheibenabstand ab. Dies deutet darauf hin, dass in dem Material ein Flüssig-Fest-Übergang stattfindet aufgrund der Filtration der flüssigen Phase durch das poröse Medium, das aus den Körnern besteht. Dieses Verhalten reflektiert die sehr geringe Fließeigenschaft der OC-Paste. Das Verhalten der SCC-Paste ist qualitativ verschieden. Oberhalb einer kritischen Geschwindigkeit U_c ist die Kraft eine steigende Funktion der Geschwindigkeit für einen gegebenen Plattenabstand. Unter diesen Fließbedingungen entspricht das rheologische Verhalten des Materials dem einer viskosen, obwohl sehr nicht-newtonischen, Flüssigkeit, was dem Fließverhalten des Materials entspricht. Für Quetschgeschwindigkeiten, die kleiner als U_c sind, ähnelt das rheologische Verhalten der SCC-Paste dem der OC-Paste. Dies deutet darauf hin, dass das Material unterhalb dieser kritischen Geschwindigkeit eine Fest-Flüssig-Trennung durchführt entsprechend seiner Nichtfließzone.

KEY WORDS: self-compacting concrete, rheological behaviour, squeeze flow, filtration

1 INTRODUCTION

Self-Compacting Concretes (SCC) are characterized by their high fluidity so that they can be processed without vibration, fill easily small interstices of formworks and be pumped through long distances. On the other hand, the SCC cement paste has to be viscous enough to avoid gravitational or flow-induced segregation. Since these two types of required properties are appar-

ently contradictory, the formulation of SCC turns out to be critical and is not well-controlled. The link between the flow (rheological) properties and the formulation is actually one of the key-issues for the design of SCC. If we consider the rheological properties that characterize SCC, the yield stress must be zero or very low and the behaviour of its effective viscosity as a function of the flow field must be controlled. The range of

viscosities needed to obtain good consolidation without vibration and without segregation has been the topic of various studies [1 - 3]. Most of them used semi-empirical tests such as the filling ability test to characterize concrete flow behavior. The rheological properties of the cement paste of SCC were found to be very important to avoid segregation. If the viscosity of the paste is high enough, the coarse aggregates will be supported by the paste, thus avoiding segregation. Often, viscosifiers such as welan gum or mineral admixtures are added to increase the viscosity of the paste, without significantly increasing the yield stress [2, 3].

Segregation may also be induced by the flow, and this problem is much less considered in the literature in the case of SCC. Actually, in most of the reported studies the material is considered to remain homogeneous during rheological measurements [4 - 6]. Nevertheless concentrated suspensions, such as a cement paste, often become heterogeneous in complex flows. For example, it is well known that non-uniform shear flows lead to particle migrations from high to low shear regions in concentrated suspensions [7]. The origin of this phenomenon is relatively well understood and attributed to irreversible interactions (collisions) among the particles [8]. This process is diffusive [7] and is actually negligible in the timescale of our experiments.

In the present study, we consider the rheological behaviour and flow-induced heterogeneities of SCC pastes in a squeezing geometry. Such geometry is expected to roughly mimic the flow experienced by the cement paste in inter granular space of a concrete or mortar. Moreover, squeeze tests are often utilized in practice, and are considered to be more appropriate than usual shear-rheometers, to determine the rheological properties of highly viscous materials [8] such as cement [9], polymer composites [10], ceramic pastes [11], etc. The material is squeezed out between two parallel surfaces at either controlled normal force or squeezing speed, and assuming a rheological model, the flow parameters of the material are inferred by fitting the model with the experimental measurements. For small gaps a lubrication-type approach can be used to determine the relationship between the applied force (or velocity) and the rheological parameters of the material. In an early study, Stefan solved the case of a Newtonian fluid and later

Scott considered the case of power law fluids [14]. More recently, Adams *et al.* [15] and Sherwood and Durban [16] considered the more general case of a Herschel-Bulkley fluid. In these studies the fluid was generally considered to remain homogeneous during the squeeze flow, with a predefined flow law. However, in the case of a highly concentrated suspension or a paste, the material can become heterogeneous under squeeze flow. Sherwood [17] reported recently a theoretical and numerical investigation of this problem. Poitou and Racineux [18] considered the rheological behaviour of highly concentrated ceramic pastes in both capillary and squeeze geometries. They showed that their experimental results could be modeled only if assuming a phase separation in which the particle concentration increased in the axial regions. Chaari *et al.* [19] considered the case of squeeze-induced heterogeneities in sewage sludge, while Delhaye *et al.* [20] reported a similar study in the case of model concentrated suspensions of spheres in a Newtonian liquid. In most industrial suspensions the liquid phase is not Newtonian. This is the case for SCC pastes, in which the addition of polymer admixtures gives to the fluid phase a highly shear-thinning aspect. To consider the effect of polymer additives we also investigate squeeze flow of ordinary cement paste containing only a granular phase and water, denoted hereafter as ordinary cement (OC).

2 EXPERIMENTAL

2.1 SQUEEZING SET-UP

The cement pastes were squeezed out between two parallel and rough (to minimize wall-slip) discs mounted on a compression-traction machine. The upper disc could be displaced at controlled velocities, while the lower one was maintained stationary. The normal force exerting on the former was recorded as a function of time for each fixed velocity. In all the experiments, the maximum initial disc separation was taken to be 6 mm, which was much smaller than the disc diameter (40 mm), insuring lubrication-type flow conditions.

2.2 MATERIALS AND FORMULATION PROCEDURE

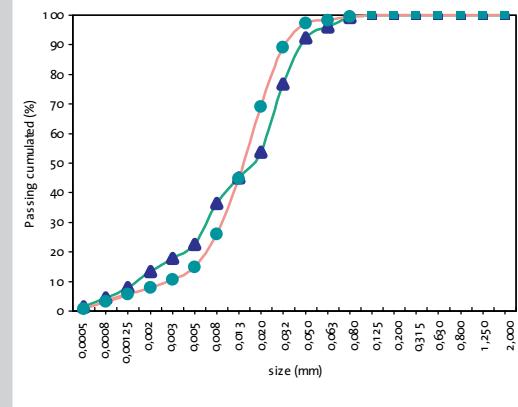
The composition of the two cement pastes considered in our study is reported in tables 1a and

Figure 1:
Granular size distribution of the cement grains and the fines.

	Cement [g]	Fine [g]	Water [ml]	SP [g]	VA [g]
OC	1000	330	500		
SCC	1000	330	300	20	10

Table 1:

a) Composition of the ordinary cement paste (OC),
b) Composition of the self-compacting concrete paste (SCC).



	Mineralogical and chemical composition (%)	Physical tests	
C_3S	71	Specific gravity	3.15
C_3A	4	Specific surface blaine (cm^2/g)	3400
C_4AF	6	Beginning of catch (min)	185
CaO/SiO_2	2.88		
MgO	0.78		
Al_2O_3	2.99		
Gypse	2.7		

Table 2:
Mineralogical, chemical and physical properties of the cement used in the present study.

	Mineralogical and chemical composition (%)	Physical tests	
$CaCO_3$	94 ± 3	Specific gravity	2.17
Ca	37.6 ± 1.2	pH	ca 9
Al_2O_3	< 1	Index pore rigidem	$35 \pm 1\%$
Fe_2O_3	< 1	Specific surface blaine (cm^2/g)	2900 ± 650
MgO	< 1		

Table 3:
Mineralogical, chemical and physical properties of the used fines.

Operations	Cement+fines mixing	Water+SP+AV mixing	Mixing low speed	Mixing fast speed
Time (mn)	5	0.5	4	2

Table 4: Mixing procedure.

1b. For the OC paste the water to cement ratio (W/C) is 0.5, and for the SCC paste W/C is 0.33. Detailed description of the different components of the pastes is reported in the following.

2.2.1. Cement

The cement used in this study is an ordinary Portland cement (CEM I 52,5 PM ES CP2) from Teil in France. It has a specific gravity of 3.15 and Blaine fineness of 3400 (cm^2/g). Its Mineralogical/chemical (as measured using X-ray diffraction by Bonneau [23]) and physical properties are shown in Table 2.

2.2.2. Additional fines

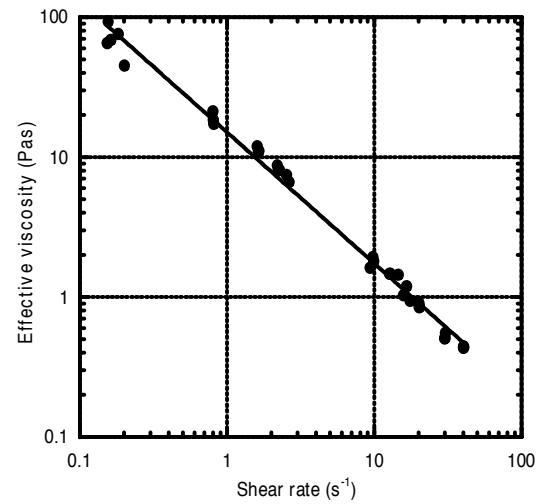
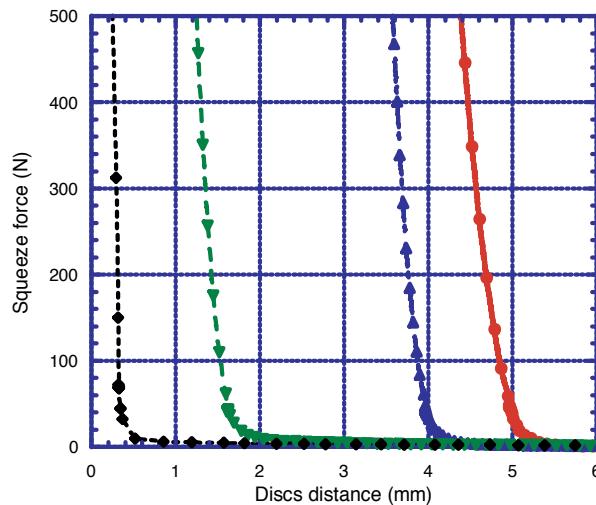
The additional fines are type PIKETTY A with ground limestone of specific gravity 2.7 and Blaine fineness of 2900 ± 650 (cm^2/g). Chemical and physical properties of the used fines are shown in Table 3. The granular size distribution (in terms of the cumulative distribution function) of the cement grains and the fines is represented in Fig. 1. We can observe some continuity in the grain size of the cement and the fines, which should give the corresponding concrete some type of high performance properties.

2.2.3. Polymer additives

CHRYSO Fluid Optima 100 is used here as superplasticizer (SP). Its properties are the following: density 1.06 ± 0.01 , pH 4 ± 0.5 , point of coagulation $-3^\circ C$, content of ion $Cl^- \leq 0.1\%$, Na_2O equivalent $\leq 0.3\%$, and dry extract $30\% \pm 1.5\%$. The viscosity agent (VA) used in the present study is diluted modified starch type with 20% dry extract.

2.3. MIXING PROCEDURE

The procedure of the paste preparation (using a laboratory mixer with paddle) is summarized in Table 4.



3 RESULTS AND DISCUSSION

3.1 SQUEEZE FLOW OF THE OC PASTE

In Fig. 2, we represent the evolution of the normal force as a function of the instantaneous discs separation for different squeezing velocities. Each run (corresponding to a given velocity) is started 30 mn after the preparation of the paste. We checked out that the squeeze flow behaviour was almost independent upon age up to 70 mn, which corresponds to the duration of the runs at the smallest speed involved in our experiments.

The squeeze behaviour of the OC paste is qualitatively different from what one expects for such material. Indeed, the rheological behaviour of the OC paste determined using a shear rheometer can be approximately modeled as a power-law fluid (see Fig. 3). For such fluids, the squeeze force is expected to follow the Scott's law [13]:

$$F = 2\pi \left(\frac{2m+1}{m} \right)^m \frac{1}{m+3} \frac{A}{\sqrt{2}} \frac{U^m}{h^{2m+1}} R^{(m+3)} \quad (1)$$

Where R is the disc radius, h the instantaneous disc separation, m the shear-thinning index of the suspension and A its consistency. The shear-thinning index and the consistency are defined through the relationship $\mu = A \dot{\gamma}^{m-1}$, where μ is the effective shear viscosity $\dot{\gamma}$ and the shear rate. For the OC paste, we have $A = 15 \text{ Pas}^m$, and $m = 0.1$ (see Fig. 3). The value of the fluidity index is quite low, indicating that the paste is highly shear-thinning.

Equation 1 states that, for any given disc separation, the squeeze force is an increasing function of the speed for a power law fluid, for both

shear-thinning ($m < 1$) and shear-thickening ($m > 1$) fluids. This is opposite to what is observed in our experiments as it is illustrated in Fig. 2. Actually, the velocity dependence of the squeeze force for a given instantaneous discs separation can be better seen in Fig. 4. This figure shows that the force is a decreasing function of the speed for any value of the gap, in contrast with the prediction of Eq. 1.

A way to understand such behaviour of the OC paste is to assume that its squeeze flow is associated with fluid-solid separation that can take place by fluid filtration through the porous media made up by jammed solid particles (cement and fines). This phenomenon has already been reported in the literature for other types of pastes [17 - 20]. The fluid filtration would lead to the increase of local particle concentration and then the effective viscosity of the paste. This is discussed in more detail farther. It is to be noted that solid-fluid separation may also take

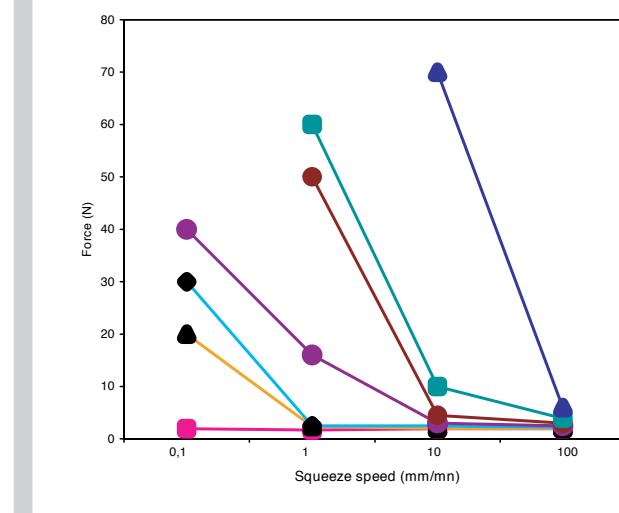


Figure 2 (left above): Evolution of the squeeze force F as a function of discs separation h for different velocities U for an OC paste:
 ◆ 100 mm/mn;
 ▼ 10 mm/mn;
 ▲ 1 mm/mn;
 ● 0.1 mm/mn.

Figure 3 (right above): Rheological behaviour of the OC paste as determined using a shear-rheometer with a Couette geometry:
 ● Experimental data and – Power law fit.

Figure 4 (right below): Evolution of the squeeze force as a function of speed for different instantaneous discs separations h for the case of an OC paste:
 □ 5.4 mm; △ 5.16 mm;
 ◇ 5 mm; ○ 4 mm; ● 3 mm;
 ■ 2 mm; ▲ 1 mm.

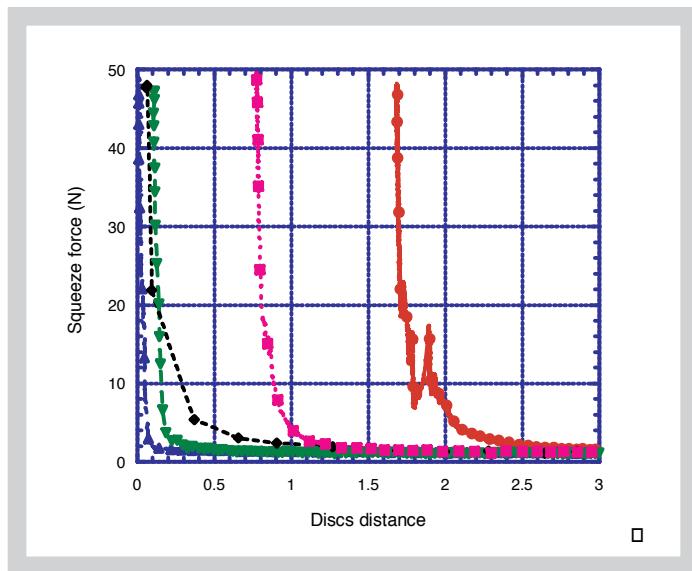
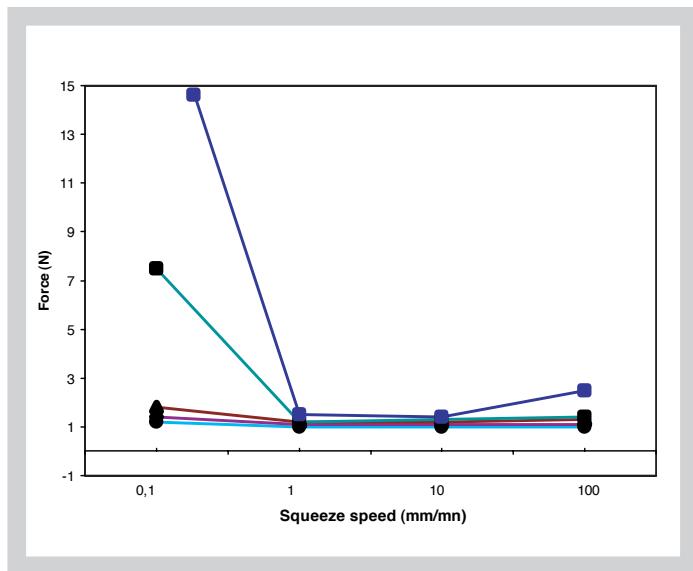


Figure 5 (left):
Evolution of the squeeze force F as a function of discs separation h for different disc velocities U , for a SCC paste: \blacklozenge 100 mm/mn;
 \bullet 10 mm/mn; \blacktriangle 1 mm/mn;
 \circ 0.3 mm/mn;
 \blacksquare 0.1 mm/mn.

Figure 6:
Evolution of the squeeze force as a function of speed for different instantaneous discs separations h for the case of a SCC paste:
 \square 5.4mm; \triangle 5.16 mm;
 \diamond 5 mm; \circ 4 mm; \bullet 3 mm;
 \blacksquare 2 mm; \blacktriangle 1 mm.



place through particle settling. However, sedimentation effects may be significant only at the beginning of the experiments. The sedimentation rate would be vanishingly small after few minutes of settling, at the moment when we have a significant increase of the force. Moreover, we undertook experiments in which the initial gap, and subsequently the duration of the run, was varied, and we found no significant influence on the squeeze force behavior.

3.2 SQUEEZE FLOW OF THE SCC PASTE

Fig. 5 represents the evolution of the normal force as a function of the instantaneous discs separation for different squeezing velocities in the case of a SCC paste. Figure 6 represents the force as a function the velocity for different instantaneous discs separations. The squeeze behaviour of the SCC paste is qualitatively different from that of an OC paste. First, we can note that the involved normal forces for SCC pastes are an order of magnitude smaller than those for OC. This can be attributed to the effect of the SP. More importantly, the squeeze behaviour of the SCC paste corresponds to two different regimes. At small velocities (and/or small gaps) the normal force increases when decreasing the squeeze speed. This would correspond to a situation where we have fluid-solid separation, similarly to the case of the OC pastes. Above a certain value of the velocity (depending upon the instantaneous gap), the force becomes an increasing function of the velocity. This would correspond to a situation in which the squeeze behaviour is dominated by the paste flow, remaining more or less homogeneous. We can note large fluctuations of the force for the smallest velocity involved here (see Fig. 5). These fluctuations are akin to dry granular type behaviour, and this indicates that we actually have solid-fluid separation.

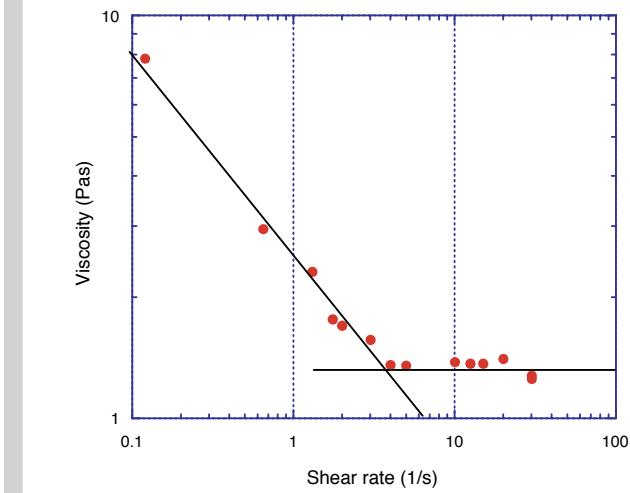
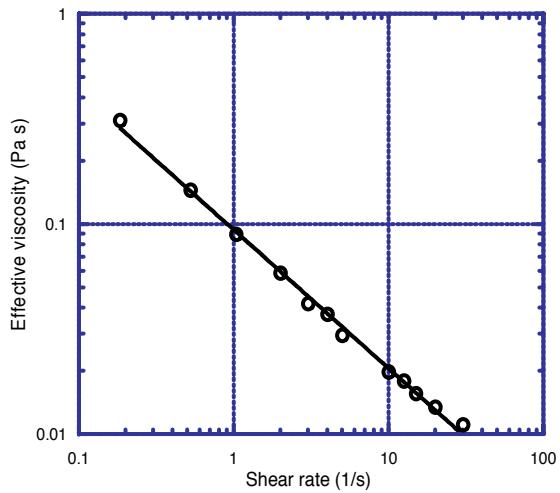
Similarly to previously reported studies [22], the main parameters defining the frontier between the two regimes can be determined by comparing the timescales of two flow phenomena that would be involved in a squeeze experiment of a granular paste; namely the fluid filtration and the paste deformation. The characteristic time of the paste flow (τ_{flow}) is determined by the imposed squeeze speed U . That is $\tau_{flow} \sim h/U$. To define the characteristic times we choose here as characteristic length scale the instantaneous discs separation and not the discs radius because in the discussion developed here we assume $h \ll R$ (lubrication-type approximation).

The characteristic time of the fluid filtration can be determined using the modified Darcy's law for power-law fluids. Indeed, the polymer solution (Water + SP + VA) used here is shown to be shear-thinning (see Fig. 7) and behaves as a power-law fluid in the shear rate interval involved in our squeeze experiments (between $U/d \sim 0.1 \text{ s}^{-1}$ and 10 s^{-1} , where d is the average grain size). The Darcy's law for power-law fluids writes [21, 22]:

$$F = |v_f - v_s| \propto \left[\frac{k^{n+1/2}}{\mu_0} \Delta p \right]^{1/n} \quad (2)$$

Where v_f and v_s are respectively the average fluid and solid velocities, k the Darcy permeability (for a Newtonian fluid, which scales as the grain size squared) and Δp the pressure. n is the shear-thinning index of the polymer solution (here $n = 0.34$) and μ_0 is its consistency (here $\mu_0 = 0.1 \text{ Pas}^n$).

Since $n < 1$, the above equation takes into account the fact that, for a given filtration veloc-



ity, the pressure drop is higher for a shear-thinning fluid than for a Newtonian fluid, which has been demonstrated experimentally. The rheological behaviour of the SCC paste can also be modeled as a power-law fluid up to a shear rate of 5 s^{-1} as it is shown in Fig. 8. That is $\sigma = A \dot{\gamma}^m$, where σ is the shear stress, A the paste consistency and m its shear-thinning index. Here, we have $A = 2.6 \text{ Pas}^m$ and $m = 0.52$. We can note that above 5 s^{-1} the paste becomes Newtonian. Since we are interested here only on the squeeze behaviour at small speeds, the paste can be considered to have a power-law fluid behavior. The driving force of the pressure drop is the stress due to the suspension flow. Then, under quasi-static conditions, we can estimate a typical value of the pressure gradient as: $\Delta p \sim A(U/h)^m/h$. This leads to the following estimation of the characteristic time for the relative solid-fluid motion:

$$\tau_{filtration} \propto \frac{h}{|v_f - v_s|} = h \left(\frac{hk}{\mu_o A} \right)^{1/n} \left(\frac{h}{U} \right)^{m/n} \quad (3)$$

Since the fluid filtration is a diffusive phenomenon and the suspension flow is a convective one, we can define a Peclet number, Pe , by taking the ratio of the two corresponding characteristic times. That is:

$$Pe = \frac{\tau_{filtration}}{\tau_{flow}} = \left(\frac{\mu_o h^{m+1} U^{n-m}}{Ak^{n+1}} \right)^{1/n} \quad (4)$$

The velocity for which we expect a transition between a homogenous flow of the suspension to a situation in which we have significant relative solid-fluid motion can be estimated by setting $Pe = 1$. This leads to:

$$U_c = \left(\frac{Ak^{n+1}}{\mu_o h^{m+1}} \right)^{1/n-m} \quad (5)$$

Equation 5 shows that the critical velocity increases when n decreases. Since in our case the fluid phase is shear-thinning ($n < 1$), the shear-thinning aspect of the fluid is expected to promote solid-fluid separation. This is in apparent disagreement with our experimental results. Actually, such effect of the shear-thinning index is expected. In our experiments, the pressure gradient in the fluid is fixed through the imposed disc's speed. Thus, since for a given pressure gradient the filtration rate increases when the shear-thinning index decreases (see equation 2), the solid-fluid relative motion effects would appear at higher disc speeds for low shear-thinning index fluids. The effect of the shear-thinning would be then to decrease the extension of the flowability domain of the suspension. The influence of the polymer consistency is opposite to that of the shear-thinning index (see Eq. 5). Then, it can be concluded from our analysis that the origin of the increase of the extension of the flowability domain of the paste by addition of a polymer is rather due to the increase of the fluid consistency. The shear-thinning aspect would have the opposite effect. However, this aspect would be benefic in the placing process of SCC. Then, similarly to other types of industrial fluids such as drilling mud, the effect of polymer addition is to give SCC stability relative to fluid-solid separation at small solicitation rates (mechanical or gravitational) or at rest, and workability at relatively high solicitation rates. The influence of the rheological parameters (A , μ_o , n and m) on the 'critical velocity' U_c will be considered in more details in a future publication.

Figure 7 (left): Rheological behaviour of the 'fluid phase' (polymer solution) of the SCC paste:
○ Experimental data and – power-law fit.

Figure 8: Rheological behaviour of SCC paste in shear flow (determined using a Couette geometry).

4 CONCLUSION

We have presented an experimental investigation into the behaviour of a SCC paste compared to that of an OC paste in squeeze flow geometry. The OC paste was suspected to undergo flow-induced heterogeneity due to the phase separation between the fluid and the particles. This gave rise to a velocity dependence of the squeeze force opposite to that usually expected for a concentrated suspension under these flow conditions. This reflects the very poor workability of the OC paste as formulated here. In contrast, the SCC paste was found to show a more complex squeeze behavior. At high squeeze speeds and/or large paste thicknesses, the normal force is an increasing function of the speed, as expected in the case of purely viscous flow of the paste. Under these circumstances, the paste is suspected to remain more or less homogeneous. At small speeds and/or small paste thicknesses, the squeeze behaviour of SCC paste is similar to that of OC paste. We then determined a qualitative diagram of the flowability of the SCC paste. From scaling analysis, we argued that the consistency of the fluid phase (polymer solution here) was the key parameter to obtain a stable suspension under squeeze flow conditions (and/or at rest). However, in practice we need the shear-thinning aspect of the fluid phase (small shear-thinning index) to increase the fluidity of the paste under processing conditions (such as pumping). Then, similarly to other types of industrial fluids (such as drilling mud), the SCC would be stable relative to fluid-solid separation at small solicitation rates (mechanical or gravitational), but flowable enough at high solicitation rates.

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