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Rheological behaviour of fresh cement pastes formulated from a Self Compacting Concrete (SCC)

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Abstract

Self Compacting Concrete (SCC) has a high flowability and can be placed without vibration. It is defined as a concrete that exhibits a high deformability and a good resistance to segregation. This kind of concrete is of great interest and has gained wide use especially in the case of difficult casting conditions such as heavily reinforced sections. From a rheological point of view, the use of a Viscosity Enhancing Admixture (VEA) along with an adequate superplasticizer content enables to ensure high deformability and stability. However, little is known about the interactions between superplasticizer and viscosity agent. Hence, we propose to study several cement pastes formulated from the original paste of a typical SCC mix. Depending on their rheological behaviour, these pastes will be used later to study the stability of coarse aggregates. The major aim of this paper is to show that empirical tests such as spread and flow time are suitable to characterise the rheological behaviour of cement pastes instead of more complex ones. Rheological properties, i.e. viscosity and shear yield stress, are well correlated with empirical test results in the range of flowable mixes. Moreover, the experimental program leads to emphasize the effects of the mixing procedure on the rhelogical properties of cement pastes. Finally, test results enable to underline the interactions between superplasticizer and Viscosity Enhancing Admixture used in designing Self Compacting Concrete.

Keywords: A. Rheology; D. Cement paste; Admixture; Self Compacting Concrete (SCC)

1. Introduction

Self-Compacting Concrete (SCC) is a highly fluid concrete that does not require any vibration during the placement process. Thus, this kind of concrete is of great interest, especially according to the economical, technical and environmental points of view [1–3]. Although SCC can be used on most construction sites, its rheological characterisation must be improved to better control its

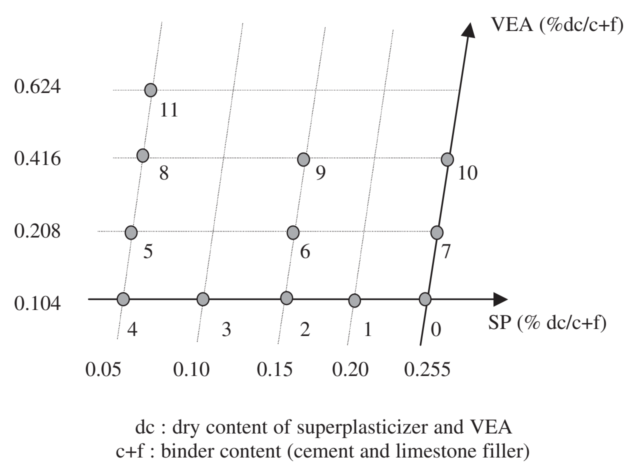

Fig. 1. Mix design of cement pastes.

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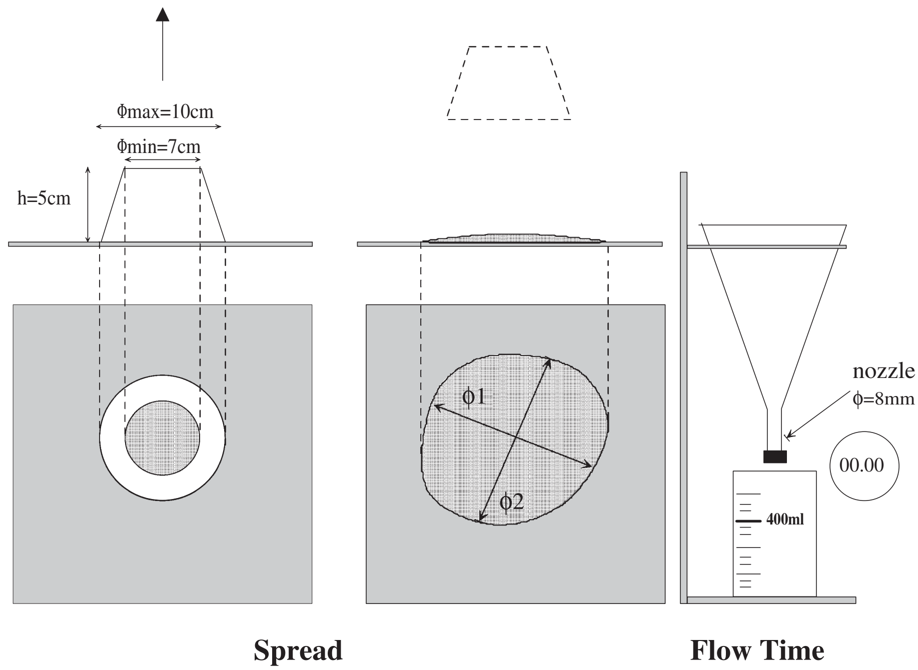


Fig. 2. Characterization of cement pastes: empirical tests.

placement. Fresh SCC must be stable to ensure the homogeneity of the mechanical strength of the structure. However, several problems like bleeding, settlement or segregation can occur, sometimes simultaneously on construction sites. Segregation can appear during placing (referred to as "dynamic segregation") or afterwards, during the dormant stage (referred to as "static segregation"). "Static segregation" consists of the sedimentation of the coarsest aggregates of the suspension under gravity forces. Several empirical tests are proposed to estimate the segregation risk of SCC [4]. Nowadays, none of them can be really considered satisfactory. More knowledge is needed to better understand segregation and to improve existing tests. Avoiding segregation is a matter of cement paste rheology and granular skeleton. The cement paste has to be sufficiently fluid to ensure the fluidity of the concrete itself and sufficiently "viscous" to support the coarse aggregates. In fact, whereas a non-zero shear yield stress enables to avoid the initiation of segregation, the viscosity and the thixotropy enable to limit its effects. According to this point of view, a better characterisation and understanding of the rheological behaviour of fresh SCC pastes is a first step to study the basic phenomena at the origin of SCC stability. In this paper, the rheological characterisation of several cement pastes is reported. The main objectives are to show that, first, a good correlation between empirical test results and rheological ones can be established in the

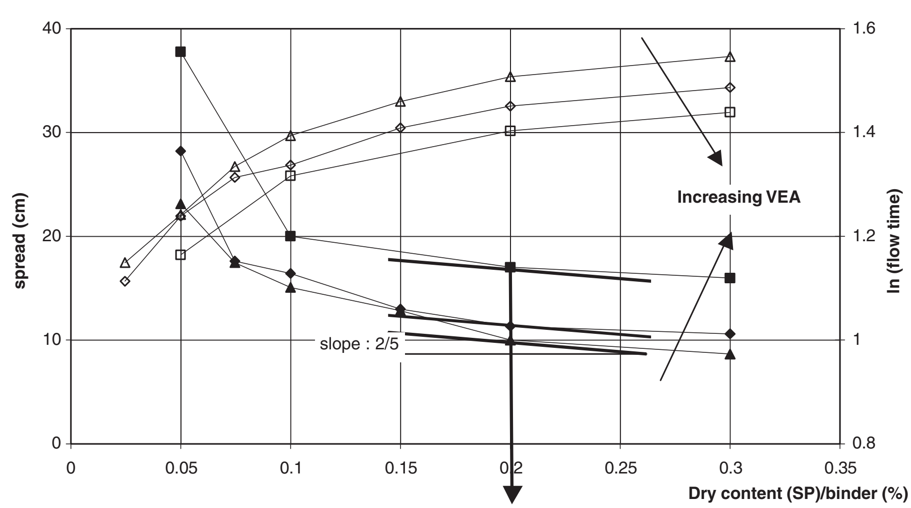


Fig. 3. Determination of the saturation dosage of the SP according to the VEA dosage.

range of flowable mixes, and second, the respect of a given mixing procedure is required as far as rheometric measurements are concerned. Besides, the adopted mix design approach that consists in modifying the Superplasticizer (SP) and the Viscosity Enhancing Admixture (VEA) contents of a typical SCC paste, led to underline the possible interactions between superplasticizer and viscosity agent.

2. Experimental program

2.1. Cement paste mix design

Cement pastes are designed from one of the SCC mixes studied in the framework of the French national project BAP. The constituents of this typical SCC mix are the followings: Portland cement (CEMI 52.5 N CE CP2 NF according to the EN 197-1 [5]), limestone filler, SuperPlasticizer (SP) and Viscosity Enhancing Admixture (VEA). Twelve cement paste mixes are formulated. The cement paste referred to as “mix 0”, is the same paste as in the original SCC mix. Whereas the solid fraction and the filler to binder ratio are kept constant (0.45 and 0.27, respectively), several amounts of superplasticizer and viscosity agent were defined from “mix 0” (cf. Fig. 1). The cement pastes have been characterised by their viscosity and shear yield stress (ranging from 0 to about 100 Pa).

The SP dosage has been limited to the «saturation value». Beyond this value, an increase in the SP dosage does not significantly improve the flowability of the material. The saturation value is determined from flow time results as follows [6]:

- flow time is measured on cement paste for several SP dosages,
- the logarithm of flow time is plotted against the SP dosage (expressed in terms of dry content to binder ratio),
- the saturation dosage is the point of tangency with a straight line whose slope is 2/5.

By using this method, the saturation dosage is independent of the testing procedure, especially of the paste volume. It only depends on the binder and on the superplasticizer.

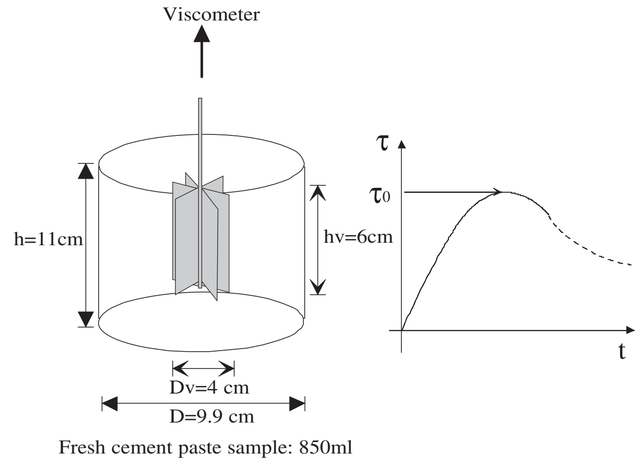


Fig. 5. Determination of rheological parameters: Vane Geometry.

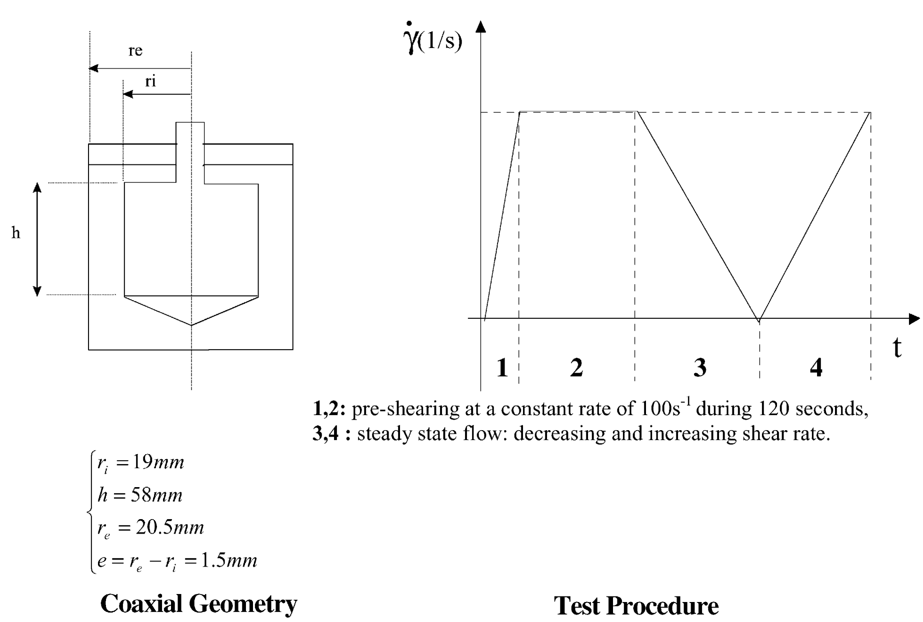


Fig. 4. Determination of rheological parameters: Coaxial Geometry.

Table 1
Evaluation of measurement accuracies

	Empirical tests		Rheological parameters		
			Vane geometry	Coaxial geometry	
	Spread (cm)	Flow time (s)	τ_0 (Pa)	v (mPa s)	τ_0^{HB} (Pa)
Average value $\bar{X}$	29.8	16.47	8.35	749	12.05
Standard deviation S	3.20	1.02	1.29	44.6	3.14
Accuracy ($\pm 1.96\ \sigma$) for $p=1-\alpha=0.95$	$\pm$0.63	$\pm$2.00	$\pm$2.53	$\pm$87	$\pm$6.15

In this study, tests have been performed on cement pastes for several VEA dosages. The results led to the conclusion that the saturation value is not affected by the VEA dosage, whereas the rheological behaviour is modified. The greater the VEA dosage, the lower the fluidity. Moreover, spread measurements are in good agreement with flow time ones. Beyond the saturation value, both spread and flow time, do not change significantly (cf. Figs. 2 and 3).

2.2. Rheological characterisation of the cement pastes

2.2.1. Empirical tests

Empirical tests, namely spread and flow time, are performed on the 12 cement pastes to characterise their rheological behaviour just after mixing.

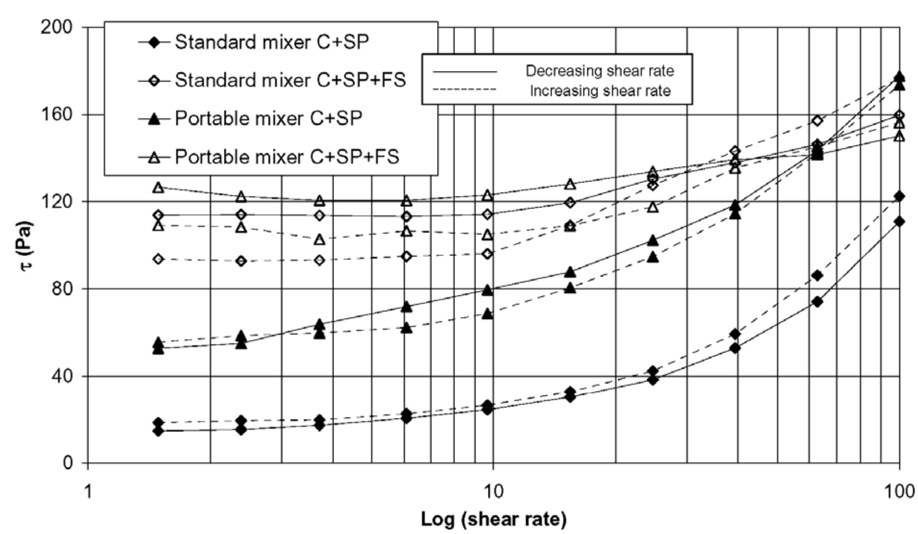
2.2.1.1. Spread measurement (mini cone). This test is carried out by using a mini cone (diameters: 10 and 7 cm, height: 5 cm). The truncated cone mould is placed on a glass plate, filled with paste and lifted. The resulting final diameter of the fresh paste sample is the mean value of two measurements made in two perpendicular directions (cf. Fig. 2).

2.2.1.2. Flow time (Marsh cone). This test consists of measuring the time required for a given volume of paste (400 ml) to flow trough the nozzle (diameter: 8mm). The initial paste volume poured into the cone is 800 ml. The longer the flow time, the lower the fluidity (cf. Fig. 2). The geometry and the dimensions of the Marsh cone are specified in the European standard EN 445 [7].

2.2.2. Determination of rheological parameters

Apparent viscosity and shear yield stress are measured by using a rate-controlled viscometer Haake VT 550.

2.2.2.1. Apparent viscosity (referred to as "viscosity" in what follows). The concentric cylinder geometry is used for the viscosity measurements. Both, the inner and outer cylinders are covered with a rough paper to prevent any slip-surface to occur (roughness 100 µm). A gap of 1.5 mm between the cylinders is larger enough in comparison with the maximum particle size (100 µm) (cf. Fig. 4). Cement pastes usually exhibit a shear-thinning behaviour, that is to say viscosity decreases with increasing shear rate during steady shear flow. For this investigation, the viscosity v was measured at



C+SP: cement and superplasticizer,
C+SP+FS: cement, superplasticizer and silica fume.

Shear yield stress τ_0 (Pa) (Herschel Bulkley model)	Standard Mixer		Portable mixer with a Cowles-blade impeller	
	C+SP	C+SP+FS	C+SP	C+SP+FS
Increasing $\dot{\gamma}$	13.8	110	48.5	102.2
Decreasing $\dot{\gamma}$	16.1	96.3	51.1	119.5

Fig. 6. Flow curves obtained on pastes made with two types of mixer.

the end of a pre-shearing phase of 120 s at a shear rate of 100 s^{-1}. It could be also defined for other shear rate values.

$$v(\dot{\gamma}) = \frac{\tau}{\dot{\gamma}} \quad (1)$$

τ, $\dot{\gamma}$: Shear stress and shear rate.

Each measurement has been carried out under steady state flow to avoid any bias [8].

2.2.2.2. Shear yield stress. Two methods are used to determine the shear yield stress of each paste. The first one consists of fitting the Herschel Bulkley model (Eq.(2)) on flow curves. Hence, the same test enables to determine both viscosity and shear yield stress. The testing procedure is shown in Fig. 4. The Herschel Bulkley model is a three-parameter, one typically used to describe the behaviour of cementitious materials [9]. This viscoplastic material exhibits a yield response with a power law relationship between shear stress and shear rate above the yield stress τ_0.

$$\tau = \tau_0 + K\dot{\gamma}^n \quad (2)$$

τ_0, K, n: Parameters of the model.

The second method consists of a mould filled with paste and a vane that rotates at a constant and extremely small speed. The generated torque evolution is measured and can be related to the shear stress. The results exhibit similar trends and are shown in Fig. 5. The maximum value of the shear stress corresponds to the shear yield stress. Before the peak has been reached, the sample deforms elastically. Then, as far as the structure of the fresh material breaks down a maximum stress is obtained. The relevance of the testing procedure and the definition of the experimental constraints (dimensions of the vane compared to those of the sample) have been already studied by Dzuy and Boger [10,11]. This procedure has also been applied to cement pastes [12]. Measurements have been carried out for several times of rest (ranging from 0 to 30 min) and hence, enable to characterise the evolution of the yield stress τ_0. The thixotropy of the material can be quantified through the evolution of the shear yield stress with the time of rest and the possible interactions between SP and VEA can be focused on. Moreover, both methods of shear yield stress evaluation can be compared.

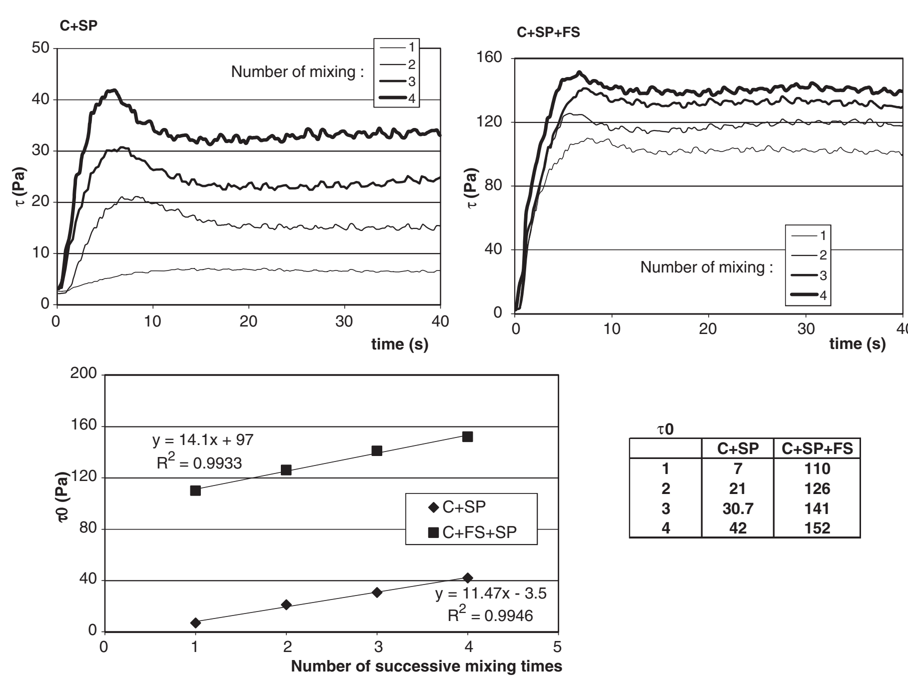
Table 2
Introduction of superplasticizer: empirical test results

Portable mixer with a Cowles-blade impeller	Spread (mm)	Flow time (s) (400 ml)
Mode 1	337	5.5
Mode 2	302	6.7

Mode 1: The superplasticizer is introduced at the end of the mixing procedure.
Mode 2: The superplasticizer is introduced together with the water.

2.2.3. Accuracy of measurements

Repeatability has been carried out on a given cement paste in order to determine the accuracy of each type of measurement. A set of at least 5 tests has been performed in



τ0	C+SP	C+SP+FS
1	7	110
2	21	126
3	30.7	141
4	42	152

C+SP: cement and superplasticizer,
C+SP+FS: cement, superplasticizer and silica fume.

Fig. 7. Shear yield stress of two cement pastes after several mixing times (Vane geometry).

Table 3
Empirical test results obtained on the pastes formulated from the SCC mix

Mix	Flow time (s)	Spread (cm)
0	9.8	36.8
1	10.7	35.4
2	11.1	33.4
3	11.6	29.6
4	16.3	23.9
5	20.4	21
6	11.6	30.6
7	10.6	34.7
8	*	17.5
9	14.7	27.3
10	14.1	30.2
11	No flow	16.3

* A continuous flow was difficult to obtain for mix 8. The measured value is not significant in this case.

each case. The calculated accuracies are summarized in Table 1 for a unique test performed. These values enable to give a better confidence to the analysis of results. For example, one can notice that the accuracy of the shear yield stress measured by the Vane is about two times greater than that extrapolated by the Herschel Bulkley model on flow curves. Both kinds of results are in good agreement. However, the slight difference between Vane and coaxial results can be attributed to the shearing conditions. Yield stress is measured at rest with the Vane and under steady shear flow with concentric cylinders.

3. Experimental results

3.1. Definition of the mixing procedure

The rheological parameters of two typical cement pastes have been measured in order to quantify the influence of several mixing parameters such as:

– type of mixer,
– successive mixing times,
– introduction of the SP.

3.1.1. The type of mixer

The flow curves showed that the mixer influence on the rheological parameters could depend on the cement paste composition. That is why two typical types of cement pastes were tested. One of them contains silica fume. For both pastes, the standardized mixer (NF EN 196-1) [13] leads to lower values of yield stress than the portable mixer with a Cowles-blade impeller. However, the difference is not significant in the case of the paste containing silica fume (cf. Fig. 6). Lumps were observed when using the standardized mixer. One can assume that the portable mixer is more efficient to ensure the unpacking of extremely small particles. Hence, it has been used to make all the pastes. However, as it was mentioned by Ferraris [14], when cement paste is tested instead of concrete, the choice of the mixer has to be guided by the shearing state of the paste in the concrete itself.

3.1.2. Successive mixing times

Several mixing times of two minutes were applied to the same fresh cement paste sample. Both types of pastes were also tested. About two additional minutes were required to measure the shear yield stress (Vane geometry) between two successive mixing times. The evolution of the shear yield stress in relation to the number of mixing is shown in Fig. 7. For both mixes, that is to say whatever the type of cement paste or whatever the yield stress level, the shear yield stress increases with the number of mixing. This result leads to the following conclusion: the cement paste does not recover its initial rheological state after an additional mixing time of two min. However, we cannot assert if this is due to an insufficient energy of mixing or to a delayed action of the superplasticizer, or to both phenomena. Hence, according to our results, the changes in shear yield stress cannot be followed on the same fresh sample. A new batch has to be made for each measurement to ensure the same initial rheological state of the fresh cement paste.

3.1.3. The introduction of the SP

The superplasticizer can be introduced at the end of the mixing procedure or can be added together with the water.

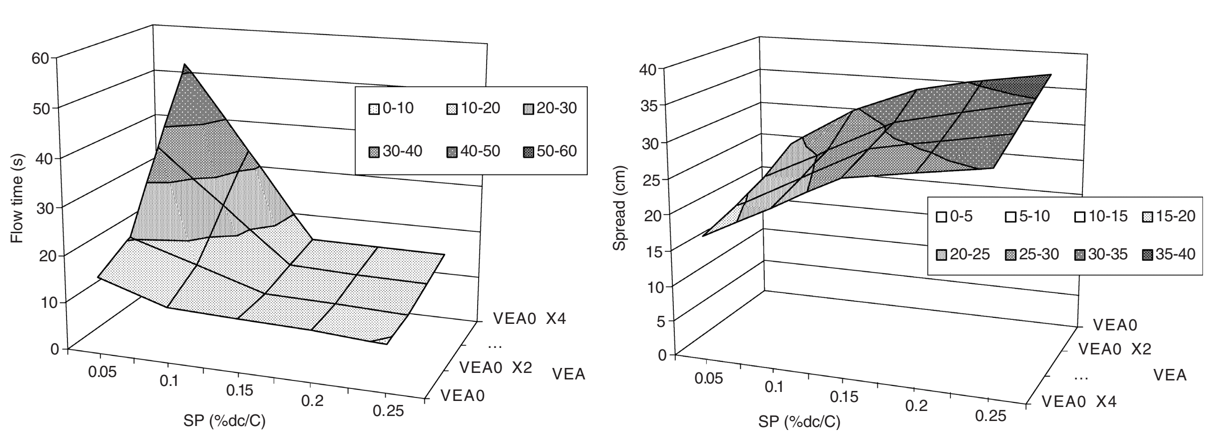


Fig. 8. Flow time and spread of pastes formulated from the SCC mix.

The way to introduce the superplasticizer has an influence on the empirical test results as shown in Table 2. These results are in good agreement with those already published [15]. The introduction of the superplasticizer at the end of the mixing procedure leads to a more flowable paste. One can assume that the superplasticizer molecules do not interact with the calcium sulphate ones and are available to separate the cement particles. Hence, the efficiency of the superplasticizer could be improved when it is introduced at the end of the mixing procedure.

These three main results dealing with the definition of a mixing procedure enable to underline the necessity to respect a given mixing procedure when carrying out rheometric measurements. The mixing procedure adopted in this study is characterized by the following points: portable mixer with a Cowles-blade impeller; volume of the batch: 1.2 litres; total duration of mixing: 6 min with increasing power. The superplasticizer is introduced after 4 min.

3.2. Empirical test results

The empirical test results: spread and flow time, are summarized in Table 3 and presented in Fig. 8. Tests have been performed just after mixing. As expected, the lower the VEA dosage and the higher the superplasticizer dosage, the greater the fluidity, i.e. the shorter the flow time and the greater the spread. However, the fluidity is all the more reduced by an increase in VEA, since the superplasticizer dosage is low. This can be more especially observed on flow time results (cf. Fig. 8).

3.3. Rheological parameters

The rheological parameters: viscosity ($\dot{\gamma}$=100 s^{-1}) and shear yield stress (Vane geometry) are presented in Figs. 9 and 10. The results are summarized in Table 4.

One of the main results is that the shear yield stress values measured by the Vane just after mixing are in good agreement with those extrapolated from flow curves

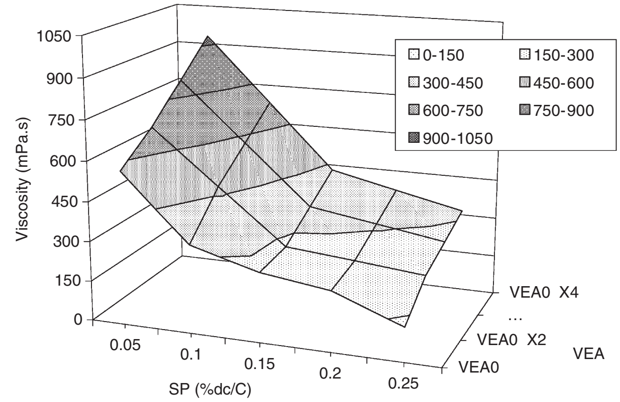


Fig. 10. Viscosity of pastes ($\dot{\gamma}$=100 1/s).

by the Herschel Bulkley model (cf. Fig. 11). This can be attributed to the particular testing procedures and more especially to the shear history. However, one can assume that, whatever the testing procedure, both types of values are well correlated. However, for shear yield stress values greater than 60 Pa, the Herschel Bulkley model gives lower yield stress values than the Vane. As it was planned, the shear yield stress values are ranging from 0 to 80 Pa just after mixing and from 0 to 150 Pa after 30 min of rest.

The other main results can be summarized as follows:

- Whatever the admixture contents, shear yield stress increases with time of rest. This is due to the structuration of the material.
- Whatever the VEA dosage and whatever the time of rest, the higher the superplasticizer dosage, the lower the rheological parameters. Moreover, an increase in the VEA dosage leads to higher values of the rheological parameters. Hence, both viscosity and shear yield stress are affected by the VEA.
- Whatever the time of rest, an increase of the VEA content has a greater effect for the lowest values of the SP content.

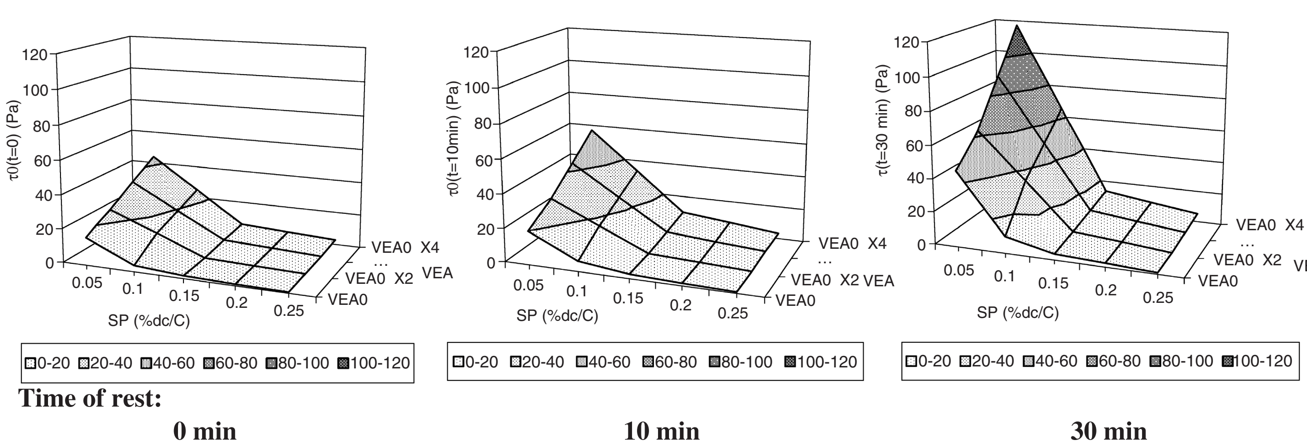


Fig. 9. Shear yield stress evolution according to the time of rest (Vane Geometry).

Table 4
Rheological parameters obtained on the pastes formulated from the SCC mix

Mix	Shear yield stress: τ_0 (Pa)						Viscosity: ν (mPa s) $\dot{\gamma}$=100 s^{-1}
	Time of rest:						
	0 min			5 min	10 min	30 min	
	Vane	HB Decreasing phase	HB Increasing phase	Vane	Vane	Vane	
0	0.7	–	–	0.7	0.7	1	123
1	1	1.1	1.1	1	1.3	2.4	225
2	1.6	2.3	1.5	2.2	2.5	3.8	260
3	3.6	3.6	4.2	4	6	10.4	331
4	16.3	16.5	17	18.6	20	46.3	575
5	25	23.8	23.4	–	30	62.9	676
6	2.5	2	1.8	–	3.7	6.7	274.2
7	0.9	1.6	1.3	–	1.2	1.4	226.2
8	28.6	35.5	41.8	–	26.5	117	930
9	6.1	9.1	9.6	–	9.8	13.6	429.9
10	2.7	2	2	–	3.3	5.3	315.3
11	80.5	59.7	56.5	–	86	150	1170

– For a given quantity of SP, the influence of the VEA content is more important for a greater time of rest.

According to the rheological point of view, the two latest main results enable us to conclude that the SP has an inhibitor effect on the VEA action. Whereas a SP dosage close to the saturation one enables to ensure a high fluidity for a given time, the VEA does not significantly modify the rheological behaviour of the cement paste. The VEA affects the rheological behaviour of the cement paste for the lowest SP contents and the highest time of rest, i.e. when the action of the SP is reduced or when it comes to an end more rapidly. Hence, SCC concrete that usually contains a great SP content can only be stabilized by VEA. VEA does not really affect its initial rheological behaviour.

3.4. Correlation between empirical test results and rheological parameters

The correlation curve between spread and shear yield stress just after mixing (Vane Geometry) is an exponential one. This can be verified by plotting the logarithm of spread against yield stress. The correlation does not

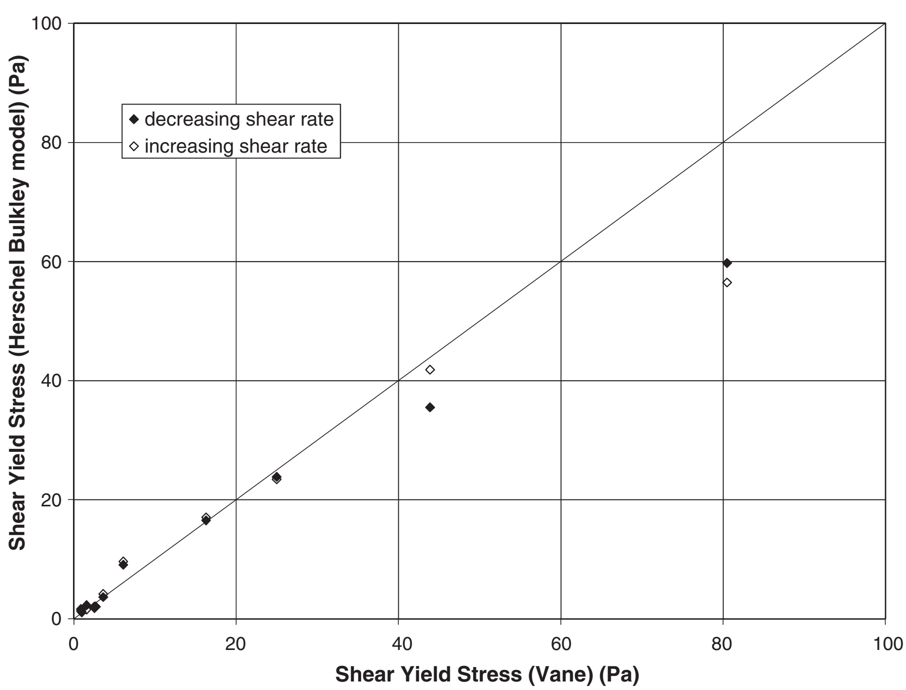


Fig. 11. Comparison between the shear yield stress values determined on flow curves (Herschel Bulkley model) and those measured by the Vane.

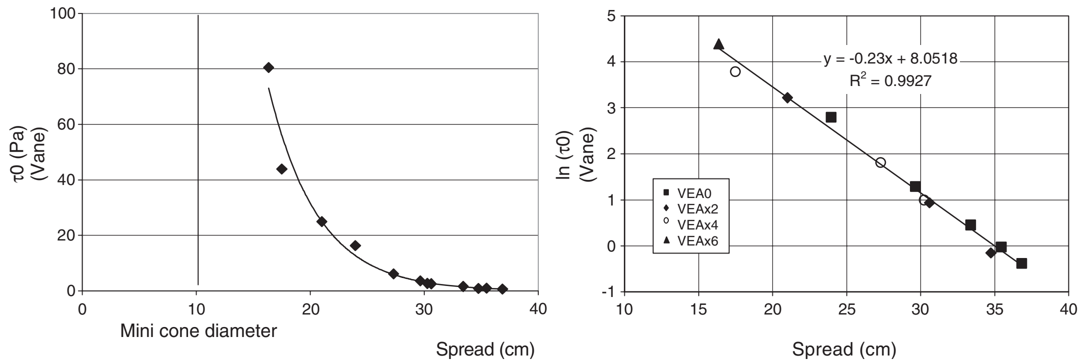


Fig. 12. Correlation between shear yield stress (Vane Geometry) and spread.

depend on the VEA dosage (cf. Fig. 12). The relation between spread and shear yield stress can be written as follows:

$$\begin{cases} \ln(\tau_0) = -0.23\ S + 8.0518 \\ \text{or } \tau_0 = \alpha \exp\left(-\frac{S}{\beta}\right) \text{ with } \begin{cases} \ln\alpha = 8.0518 \\ \frac{1}{\beta} = 0.23 \end{cases} \end{cases} \tag{3}$$

τ_0: Shear yield stress (Pa), S: Spread (cm).

A similar analysis was applied to flow time and viscosity results. A linear correlation is obtained and does not depend on the VEA dosage (cf. Fig. 13). Linear correlations were also obtained between flow time and viscosity defined at other shear rates ranging from 15 to 100 s^{-1} (cf. Fig. 14). The parameters are shear rate-dependent and a power law function enables to describe their evolution in relation to shear rate (cf. Fig. 15). A general linear relation between viscosity and flow time can be given:

$$v(\dot{\gamma}) = A(\dot{\gamma})\text{Flt} - B(\dot{\gamma}) \quad \text{with :} \begin{cases} A(\dot{\gamma}) = 2082.6\dot{\gamma}^{-0.81} \\ B(\dot{\gamma}) = 31454\dot{\gamma}^{-1} \end{cases} \tag{4}$$

v: viscosity at a given shear rate (mPa s), Flt: Flow time (s).

The Bingham model can also be used to roughly describe the rheological behaviour of cement pastes:

$$\tau(\dot{\gamma}) = \tau_{0B} + v_B\dot{\gamma} \tag{5}$$

τ_{0B}, v_B: Bingham parameters: Shear yield stress and plastic viscosity.

The simplicity of this two-parameter model enables to develop a physical modelling more easily. According to Eq. (4), the plastic viscosity given by the Bingham model is also the apparent viscosity obtained for an equivalent shear rate of about 550 s^{-1} (cf. Fig. 14). Hence, the experimental results presented in this paper and described by the more relevant Herschel Bulkley model, are in good agreement with the physical modelling of the flow process in the Marsh cone test reported in [16] where a Bingham model is used.

Finally, these correlations lead to the main conclusion that easier empirical tests can be performed instead of more complex rheological ones, when determining the rheological behaviour of flowable cement pastes. They support the points of view of several authors about the modelling of empirical tests [16,17]. However, according to other published papers, weak correlations are obtained between empirical tests and rheological parameters [14,18]. The only way to explain that good correlations

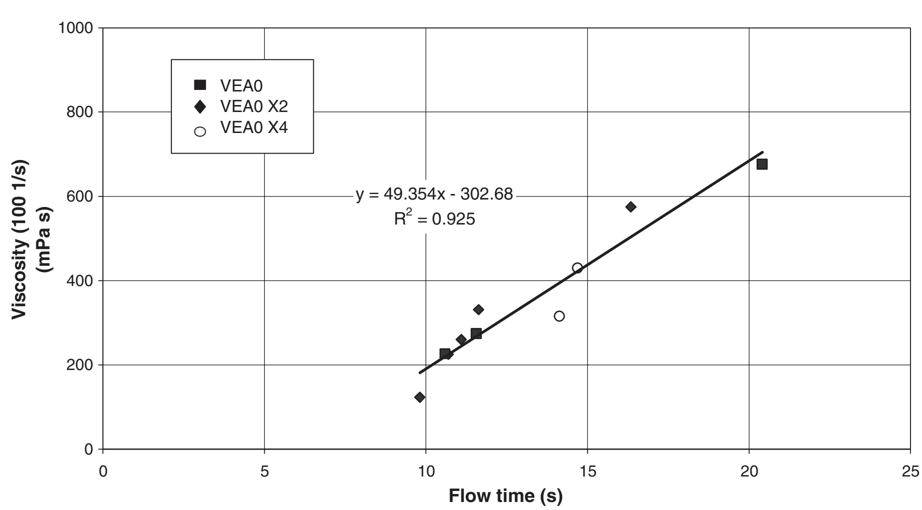


Fig. 13. Correlation between flow time and viscosity (shear rate 100 s^{-1}).

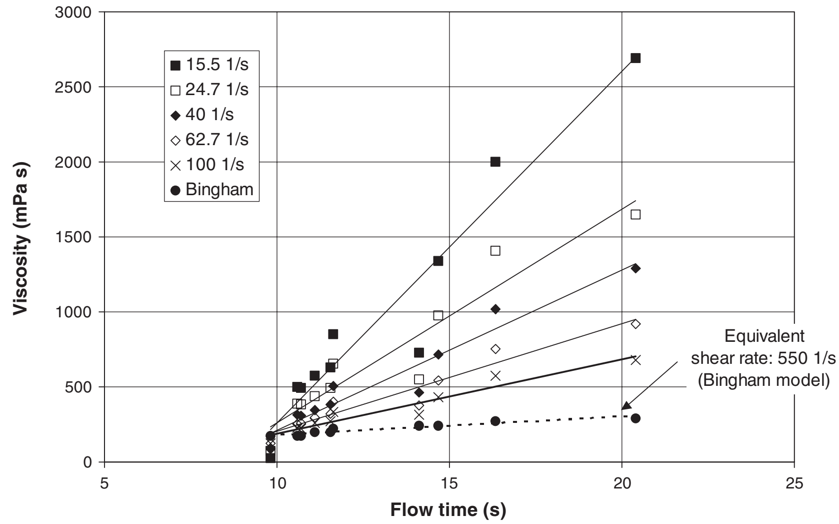


Fig. 14. Correlation between flow time and viscosity for several shear rates ranging from 15 to 100 s^{-1} and in the framework of a Bingham description.

have been established in this study is to assume that the parameters of the models probably depend on the studied binder, SP and VEA.

4. Conclusions

The results enable to underline the necessity to respect a given mixing procedure when carrying out rheometric measurements. This includes the following points:

- Pastes must be done with the same mixer,
- The SP must be added at the same step of the mixing procedure,
- The duration of the mixing procedure and the volume of the batch must not be changed.

In this study, 12 cement pastes have been designed from the paste of a typical SCC by modifying the superplasticizer and the VEA dosages. Their rheological behaviour has been characterised and four main conclusions can be focused on. Two of them deal with the interactions between SP and VEA. The other two enable to simplify the mix design of cement pastes by performing easier empirical test such as spread and flow time, instead of more complex ones. As far as the parameters of the models given by Eqs. (3) and (4) are determined on several preliminary tests, empirical ones can be performed more rapidly.

1. The VEA affects both viscosity and shear yield stress. An increase of the VEA content leads to an increase of the rheological parameters. However, the saturation dosage of SP is not modified by the VEA dosage.

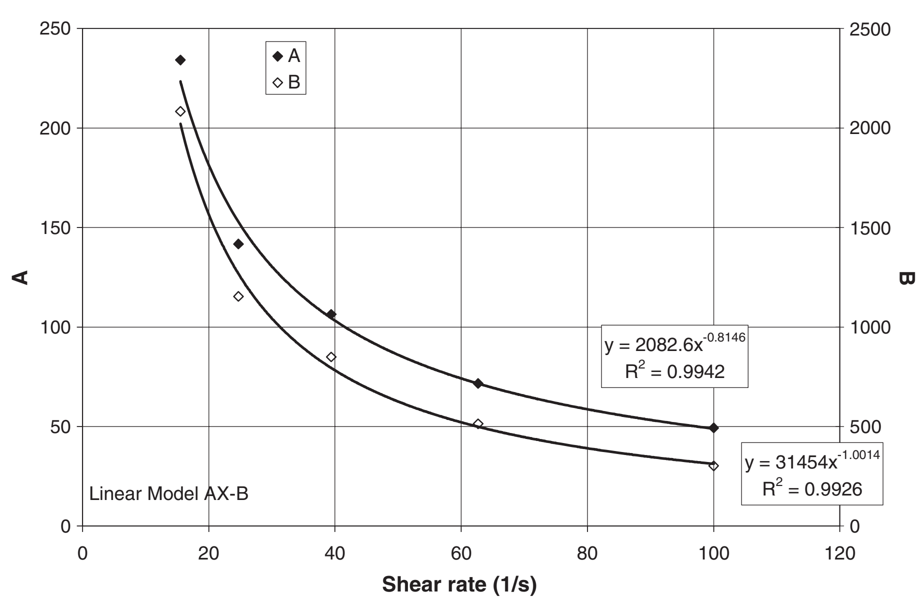


Fig. 15. Shear rate dependence of the parameters A and B of the linear relation between flow time and viscosity.

2. For a SP dosage close to the saturation one, the VEA does not modify the rheological behaviour of the paste. However, it is used to bring stability.
3. The shear yield stress values obtained from the Vane Geometry are in good agreement with those obtained from flow curves (extrapolation by the Herschel Bulkley model).
4. According to the statistical point of view, significant correlations are obtained between spread and yield stress, and between flow time and viscosity. Hence, easier empirical tests can be performed to characterise the rheological behaviour of cement pastes for a given set of constituents.

Besides, a great number of systematic measurements allows for precision and accuracy of each type of measures (spread, flow time, shear yield stress: Vane measurement or extrapolation from flow curves, viscosity). A better confidence can be given to the main conclusions of this experimental study.

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