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Yield stress and viscosity equations for mortars and self-consolidating concrete

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Abstract

The rheological behavior of flowable concrete, such as self consolidating concrete is closely influenced by concreting temperature and the elapsed time. The variation of the plastic viscosity and the yield stress with the elapsed time and temperature must be accurately quantified in order to forecast the variation of workability of cement-based materials. A convenient method to study the variation of these rheological parameters is proposed, using the mortar of the concrete. This latter is designed from the concrete mixture, taking in account the liquid and solid phases with a maximum granulometry of 315 μm. Different SCC and mortars proportioned with two types of high range water reducing admixtures (HRWRA) were prepared at temperatures ranging from 10 to 33 °C. Test results indicates that the yield stress and the plastic viscosity of the mortar mixtures vary in a linear way with the elapsed time while an exponential variation of these rheological parameter is seen on SCC. In order to enhance robotization of concrete, general equations to predict the variations of the yield stress and plastic viscosity with time are proposed, using the corresponding mortar initial yield stress and plastic viscosity. Such equations, derived from existing models, can easily be employed to develop concrete design software. Experimental constants which are related to the paste fluidity or the aggregates proportioning can be extracted from a database created with either mortar or aggregates test results.

Keywords: Temperature (A); Fresh concrete (A); High range water reducers (D); Rheology (A); Calorimetry (A)

1. Introduction

Self consolidating concrete (SCC) is now being studied worldwide and progressively used for experimental jobs and actual projects. This type of concrete must be having excellent flowability and adequate segregation resistance at the same time. On sites, delivery delays are frequents and it was found that ambient temperatures influence their workability [1]. Forecasting the evolution of rheological parameters is becoming more and more important as new generations of concretes were proved to be dependent on concreting conditions. Several approaches were attempted to model the rheological behavior of cementitious materials [2–6]. Nevertheless, most of the proposed models describe the variations of the initials parameters. They either aim at obtaining the yield stress (τ_0) and the plastic viscosity (μ) from in-situ tests results [2,5], or expressing these parameters with more complex equations taking in account the cement paste and the packing density of the granular mixture [3,4,7]. Few of existing models take into account the elapsed time and the effect of temperature, minimizing the role of the hydration kinetic on the flow behavior of SCC.

Data presented herein were obtained on SCC and mortar mixtures formulated with polynaphtalene sulfonate (PNS) or polycarboxylate polymers (PCP) high range water reducing admixtures (HRWRA). It was found on mixtures proportioned with *W*/*B* of 0.52 and PCP HRWRA that the flow behavior was dependent on concreting temperature [8]. For temperatures above 15 °C (threshold temperature for the tested mixtures), the mortars made with PCP HRWRA behave the same way as mixtures proportioned with PNS.

The yield stress and plastic viscosity of mortars were continuously increasing in a linear fashion with the elapsed

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time. Thus, to avoid complex behaviors, mixtures made with PCP HRWRA were tested only above their threshold temperature, enabling general equations to be proposed.

The first objective of this study is to investigate the influence of time and temperature on yield stress (τ_0) and plastic viscosity (μ) of highly flowable mortar mixtures and SCC with either PNS or PCP HRWRA. In a second time, general equations are proposed to describe the variations of τ_0 and μ with temperature and the elapsed time from mixing to the end of the dormant period. These equations are proposed in order to forecast the variations of the rheological parameters to enhance concrete batching robotization. Instead of developing new models, the choice was made to partially adapt existing models to predict the variations of τ_0 and μ on concrete from values obtained on mortars. Thus, due to changes in concrete design, materials or temperature could be predicted through the proposed equations and data obtained on mortars. As this latter is easier to batch and test; it will facilitate concrete design by reducing the number of expensive concrete tests and replacing them with lower cost mortar testing. In addition, the proposed equations could easily be computerized to adjust the mix design of the concrete depending on aggregates size distribution, on mortar fluidity and on the temperature of the materials.

The methodology presented herein employs the normalized time (t'), parameter defined as the ratio between the elapsed time and the duration of the dormant period. This t' value is obtained using isothermal calorimetry. These heat flux measurements are conducted to evaluate the kinetics of formation of hydrates during the dormant period given the coupled effect of the HRWRA and temperature variations.

Finally, the effectiveness of a new proposed method to estimate the yield stress and the plastic viscosity for highly flowable concrete was evaluated according to the elapsed time and the temperature by using the rheological properties of their corresponding mortar.

2. Investigations

2.1. Research significance

The potential benefit of forecasting the behavior of the SCC depending on the HRWRA used and the concreting temperature to enhance concreting conditions is not well documented, especially for a range of time as long as the dormant period. This work proposes an easier method to study SCC through their mortars. From data presented, a mathematical approach of the variations of rheological parameters is proposed to highlight the materials behavior taking into account the effect of the temperature. It aims at doing the right SCC mixture proportioning for each site without a costly testing campaign on concrete through the knowledge of the variations of rheological parameter of the corresponding mortar. Interactions between the HRWRA, the materials of the concrete, temperature and time can be predicted and problems of concrete stiffening avoided.

2.2. Scope of investigation

The experimental program presented herein consists of three phases. Phase 1 describe the methodology to access to variations in rheological parameters of the various mixtures according to temperature at the same relative scale of time. The mortar and SCC mixtures were evaluated using co-axial rheometers to monitor variations in yield stress and plastic viscosity with time. The composition of mortars is based on mixture proportioning derived from SCC mixtures, except for the presence aggregates coarser than 315 μm. The cut-off of sand fraction at 315 μm for mortar was chosen to enable the assessment of rheological properties of the system without the risk of blockage or slippage inside the bowl [9] using a co-axial rheometer for which the difference between the inner and the outer radius was only of 1 mm.

Phase 2 presents the coupled effect of temperature and elapsed time on the variation of rheological parameters for two SCC (B1 and B2) and the corresponding mortar (M1 and M2) for temperature between 10 °C and 33 °C (Tables 1 and 2). Mixtures M1 and B1 are based on a commercially available SCC used in Northern France. During constructions, such concrete was cast at ambient temperatures varying between 5 and 30 °C, with no special provisions to adjust for the fresh concrete temperature to site conditions. In some cases, concrete needed to be cast 90 min after mixing and still had to show a minimal slump flow of 550 mm to permit casting without the need for mixture adjustment at the job site. The SCC was proportioned with relatively high water to binder ratio (*W*/*B*) of 0.52, in compliance with the European EN 206-1 Standard for ready-mix concrete in non-aggressive environment. Mixture M2 is based on the same mix design as the M1 mixture, except for the use of PCP-HRWRA instead of PNS. Thus, the derived SCC mixture, B2, is proportioned as the previously described concrete except for the PCP-HRWRA employed. From these first results, models are proposed to estimate the rheological properties of SCC from their corresponding mortar.

Table 1
Summary of experimental program

Series	Mortars					SCC				
	M1 [a]	M2 [a]	M3	M4	M5	B1 [a]	B2 [a]	B3	B4	B5
Temperatures field										
10–12 °C			X					X		
15–18 °C	X			X		X			X	
20–23 °C	X	X	X	X	X	X	X	X	X	X
25–27 °C	X	X	X	X		X	X	X	X	
30–32 °C		X	X	X	X		X	X	X	X
Response obtained for each temperature studied										
Calorimetry	X	X	X	X	X	X	X	X	X	X
Temperature rise	X	X	X	X	X	X	X	X	X	X
Rheology with co-axial rheometer	X	X	X	X	X	X	X	X	X	X

X refers to tested combinations.
[a] Mixture used to calculate experimental constants employed in yield stress and viscosity equations.

Table 2
Mortar and SCC mixtures

Materials	SCC					Mortar				
Name of the series	B1	B2	B3	B4	B5	M1	M2	M3	M4	M5
Cement CEM II B 32.5 R, kg/m^3	400	400	–	350	350	837	837	–	732	732
Cement CEM I 52.5 N, kg/m^3	–	–	400	–	–	–	–	837	–	–
Fly Ash Class F, kg/m^3	–	–	–	36.5	–	–	–	–	76.3	–
Silica fume, kg/m^3	–	–	–	–	36.5	–	–	–	–	76.3
W/B	0.52	0.52	0.52	0.52	0.52	0.52	0.52	0.52	0.52	0.52
Sieved sand (≤315 μm), kg/m^3	–	–	–	–	–	728	728	728	728	728
Sand, kg/m^3	940	940	940	940	940	–	–	–	–	–
Coarse aggregate, kg/m^3	850	850	850	850	850	–	–	–	–	–
Naphthalene-based HRWRA, % by dry mass of cement	0.28	–	0.28	–	–	0.28	–	0.28	–	–
Polycarboxylate-based HRWRA, % by dry mass of cement	–	0.27	–	0.27	0.27	–	0.27	–	0.27	0.27

The adequacy of the analytical model proposed is examined in phase 3. Table 1 summarizes the scope of our investigation reported herein. The effect of temperature between 10 and 33 °C on changes in rheological parameters, heat flux, and temperature rise was investigated on five sets of mortars (mixtures M1 to M5) and the corresponding SCC mixtures (B1 to B5). Mixture M3 is based on the same mix design as the M1 mixture, except for the use of a different type of cement. The derived SCC mixture, B3, is proportioned as B1. All mixes are summarized in Table 2.

M4 and M5 mixtures were designed using either Class F fly ash (M4 and B4 mixtures) or silica fumes (M5 and B5 mixtures) as replacement of 12.5% of the cement volume, without altering the paste volume. This choice was made to keep the paste-to-aggregate volume ratio constant, thus enabling comparison between the tested cementitious materials in terms of rheology. Otherwise, the M4 and M5 mixtures had the same mixture proportioning and materials as that of the M2 mortar.

Heat flux was determined using isothermal calorimetry [10]. This calorimetric technique was used as it enables to determine the variations of heat flux with time at curing temperatures of 10 to 33 °C (Table 1). Since the thickness of the structures cast with this concrete is limited, and the thermal exchange surface of commonly used wall formworks elevated, it can be assumed that the temperature rise due to the heat of hydration of cement is rapidly lost. Thus, the temperature of the concrete inside the formwork can undergo limited variations. Consequently, the simulation of heat conditions in thin walls can be achieved with greater effectiveness using isothermal calorimetry than semi-adiabatic or adiabatic calorimetry.

3. Experiments

3.1. Materials and mixture proportioning

In order to assess rheological parameter of concrete from measurements made on mortars, a concrete equivalent mortar (CEM) [11] could have been done. This equivalent mortar is designed taking into account the liquid phase, the binder, and an equivalent amount of sand. The specific areas of the sand and the coarse aggregate fraction of the concrete are calculated. The aggregates are hence replaced into the mortar by standardized sand only in order to get the same specific area as the one calculated with the aggregates of the original concrete mixture. The behavior of both concrete and its CEM were proved to be easily correlated [11]. Nevertheless, when fillers are added to the aggregates of the concrete (specific area of filler is important), this mortar mixture design calculation leads to the replacement of fillers by a huge quantity of standardized sand, hence artificially stiffening the mortar. As the original SCC mixture was proportioned with a sand containing about 7% in mass of limestone fillers, a CEM would have lead to an over dosage in standardized sand since the specific surface of the fillers is important. Consequently, mortars designed from the original SCC mixtures taking into account the binder, the liquid phase (water and admixtures), and the fraction of sand retained on the 315 μm sieve, were used in this investigation. Table 2 summarizes the mixture proportioning of the SCC and the mortar mixtures.

The B1 SCC was cast with an average slump flow of 600 mm. It had a relatively high W/B of 0.52 and was proportioned with blended cement complying with the French NF P 15-301 Standards (CEM II B 32.5 R). Such cement contains approximately 68% clinker, 21% blast furnace slag, and 6% limestone filler. The SCC was prepared with crushed limestone aggregate of 12 mm nominal size and crushed limestone sand with a fineness modulus of 2.48. A PNS-HRWRA was used in the mortar and SCC mixtures of the B1 and B3 mixtures at a dosage corresponding to 0.28% of the binder mass, expressed on dry basis.

In the case of the B2 mixtures, the SCC from which the mortar was designed had the same slump flow of 600 mm. A polycarboxylate-based HRWRA was used at a total dosage of 0.27% by active mass of cement. The dosage rates for HRWRA were determined using the Grout method [12], targeting the same Marsh cone time on mortars of 78 s for a Marsh cone opening of 8 mm of a capacity of 1.2 L to achieve a flow of 1.0 L of the mortar.

The B3 SCC have the same characteristics and formulation as B1 except for the use of the cement CEM I 52.5 N instead of

CEM II B 32.5 R. Characteristics of these two cement are presented in the Table 3.

From the B2 mix design, two sets of SCC were prepared using either Class F fly ash (M4 and B4 mixtures) or silica fumes (M5 and B5 mixtures) as replacement of 12.5% of the cement volume, without altering the paste volume. As previously said, this choice was made to keep the paste-to-aggregate volume ratio constant, thus enabling comparison between the tested SCC in terms of rheology.

3.2. Test methods

The mortar mixtures were prepared in batches of 6 L and mixed in a Hobart-like mixer with paddles rotating at successive speeds ranging between 140 and 285 rpm. The temperature of the raw materials was adjusted to the targeted temperature of the fresh mixtures. In order to avoid energetic transfers during the mixing, the mixer, the mixing bowl, the pan and the co-axial rheometer used for the mortar investigation were also maintained at the targeted temperature. The mixing procedure consisted of adding the water and HRWRA into the mixer, then introducing the binder gradually over 30 s with the mixer rotating at 140 rpm. The sand was gradually introduced during 30 s, while the mixer was still turned on. Mortar was then mixed during 30 s at a speed of 285 rpm. After a rest period of 90 s, the mixing was resumed for an additional 60 s at 285 rpm.

The SCC mixtures were prepared in batches of 40 L in a mixer equipped with a helicoïdal mixing paddle. The temperature of the raw materials was adjusted to be similar to those of the targeted fresh concrete. The mixing procedure consisted of homogenizing the dry aggregate in the mixer during 30 s. Part of the mixing water was gradually introduced over 30 s with continuing mixing action. A rest period of 10 min was required for the dry aggregate to absorb some of the mixing water. The cement was then introduced gradually over 30 s, and HRWRA was finally introduced with the remaining water. The concrete was then mixed for 2 min.

At the end of mixing, samples were taken to monitor heat generation during the dormant period of cement hydration, and rheological measurements were determined at set intervals over the dormant period. In order to avoid evaporation in between tests, the material was kept in the mixing bowl, covered with a plastic sheet and at an isothermal temperature matching that of the targeted test temperature. Mortar was remixed at low speed of 140 rpm during 60 s in order to ensure homogeneity before the evaluation of the rheological parameters. Such limited mixing intensity should not lead to further defloculation of cement grains of the suspension, thus influencing the duration of the dormant period [13]. SCC were remixed before test during 30 s. This shorter remixing time was chosen for SCC as a unique rotational speed was imposed by the concrete mixer. Consequently, as mixing intensity could not have been chosen, mixing time was lowered.

For isothermal calorimetry measurements, the curing temperature of a prismatic sample measuring 90 × 90 × 160 mm was maintained through the testing period, and the heat flux (Φ) required by the apparatus to cool down the sample during cement hydration is monitored using fluxmeters [10]. The temperature of the water system used to cool down the cementitious sample is controlled by six thermocouples on each side of the formwork and one maintained in the middle of the prismatic sample. A maximal variation of temperature between the core and the surface of the sample of 0.5 °C is obtained for a test temperature of 30 °C. Since the heat flux is measured on each surface of the prismatic sample (S_i, m^2), the variation of the heat of hydration, $Q(t)$, is obtained by integrating the heat flux (Φ, W/m^2) data over time. The $Q(t)$ function is normalized by the cement mass (m_c) used in fabricating the test sample, as follows (Eq. (1)):

$$Q(t) = \frac{1}{m_c} \sum_{\text{surface}} \int_0^t \Phi(t).\mathrm{d}t.Si. \quad (1)$$

As shown in Table 1, tests were done at temperatures varying from 10 to 33 °C, depending on series, to determine the effect of temperature on the hydration kinetics.

A co-axial cylinder rheometer was employed to evaluate the rheological properties of mortars. A 350-ml sample was used for such measurement. As the surfaces of the inner and outer cylinder of the viscometer were not roughened and the materials tested in this investigation were highly flowable, slippage could occur at smooth walls, resulting in an under evaluation of the yield stress [14,15]. Nevertheless, for the sake of this investigation, the cylinders were not serrated in order to keep a sufficient gap value. Moreover, the proposed methodology presented herein employs relative yield value and viscosity [16], and data obtained on all mixes showed few deviations in terms of relative rheological values.

The apparent viscosity was determined, after a stabilization time of 20 s, at five rotation speeds varying between 3 and 300 rpm, which correspond to shear rates of 5.1 to 510 s^{-1}. The apparent viscosity is calculated as the ratio between the shear stress (τ) and shear rate (γ) at a given shear rate. The

Table 3
Chemical analysis and physical properties of cements

	Cement CEM II B 32.5 R		Cement CEM I 52.5 N	
	Chemical analysis	Physical characteristics	Chemical analysis	Physical characteristics
SiO_2	22.4	BET: 320 m^2/kg	20.4	BET: 410 m^2/kg
Al_2O_3	6.2	Specific gravity: 3.05	5.0	Specific gravity: 3.10
Fe_2O_3	2.7		3.2	
CaO	58.8	Initial setting time (Vicat): 180 min	63.8	Initial setting time (Vicat): 150 min
MgO	2.2		0.9	
Na_2O eq.	0.73	Compressive strength	0.24	Compressive strength
C_3A	9.0	2 d: 21 MPa 28 d: 48 MPa	8.0	2 d: 35 MPa 28 d: 62 MPa
Clinker	68%		97%	
Blast furnace slag	21%			
Limestone filler	6%			
Others			3%	

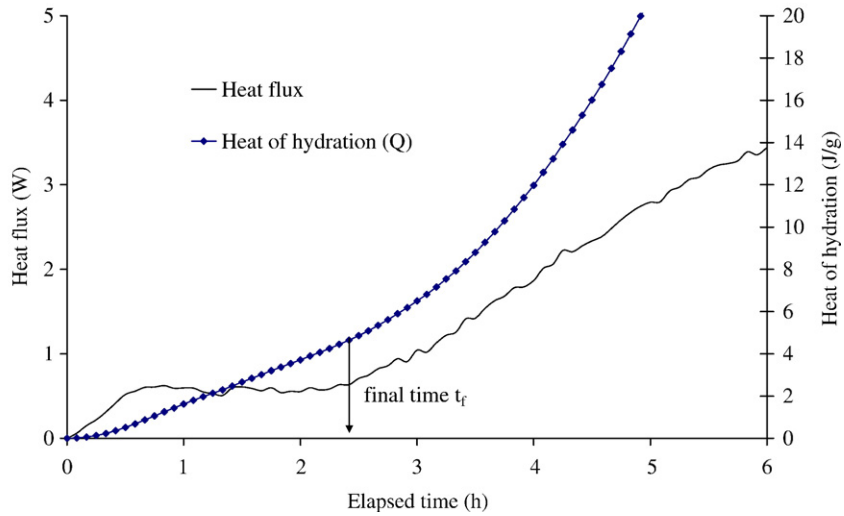

Fig. 1. Example of variations of heat flux and hydration determined from isothermal calorimeter according to the elapsed time.

yield stress refers to the resistance of the material to undergo initial flow, and the plastic viscosity (μ) refers to the slope of the shear stress-shear rate relationship. As the up and down curves coincided, τ and μ values are derived by regression analysis using the shear stress-shear rate data assuming a polynomial response. The second order value is considered as insignificant and is suppressed. The resulting expression can be expressed as $\tau = \tau_0 + \mu$. γ representing the behavior of a Bingham fluid. Repeatability experiments showed little dispersion of the results. The maximal relative deviation was of 6% for both yield stress and plastic viscosity for the mixtures tested on data presented herein. After each rheological test, the sample was discarded in order to ensure confidence in test results.

A BML 4 rheometer was employed to evaluate the rheological properties of the SCC. A 20-L sample of SCC was used for such measurement. The design of the BML concrete rheometer, using blades and serration, avoid any risk of slippage while testing the SCC. The apparent viscosity was determined at seven shear rates. For the SCC mixtures, the shear stress and shear rate data are used to determine the yield stress and plastic viscosity by assuming a Bingham flow model. As hydrates are broken down while processing each rheological test, the sample employed was discarded after having being sheared. This protocol was chosen in order not to mix normally hydrated material with deteriorated cement paste.

No segregation was observed on mortar and concrete samples. Nevertheless, a stability criterion was employed for both mortar and SCC mixtures [17]. The minimal value of the yield stress can be expressed as $\tau_0 \geq \frac{(\rho_a - \rho_f) g . D_{max}}{K_0}$ where ρ_a and ρ_f are the specific gravities of the aggregates and the paste respectively (kg/m^3), g the gravity constant, D_{max} the maximal size of the aggregates, and $K_0 = 18$ a constant suggested by ref [17]. As all experimental data obtained during this investigation were above these thresholds (0.1 Pa for mortar mixture and 67 Pa for SCC), it is assumed that no segregation occurred during the tests, in accordance with what was observed.

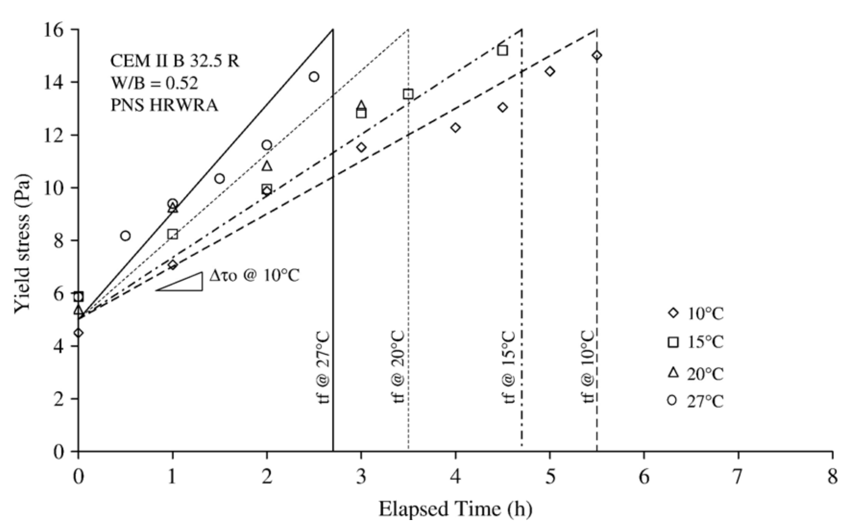

Fig. 2. Variations of yield stress for M1 mortars made with PNS HRWRA according to the elapsed time and temperatures between 10 and 27 °C.

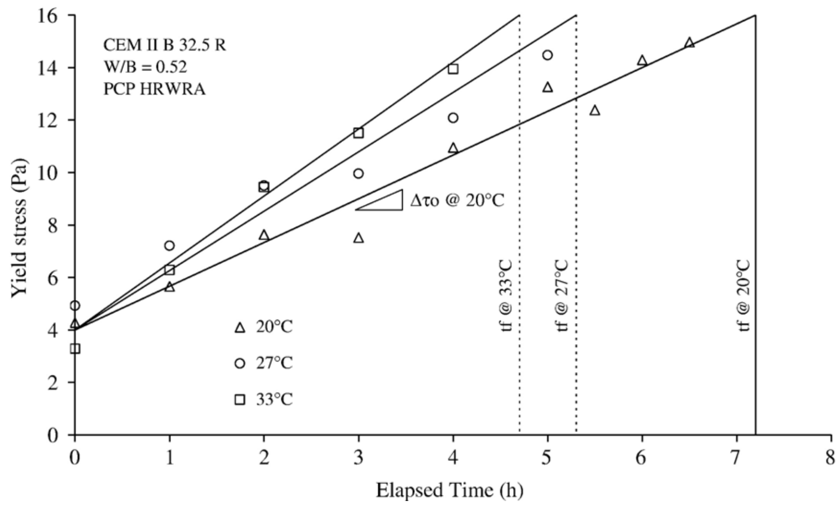

Fig. 3. Variations of yield stress for M2 mortars made with PCP HRWRA according to the elapsed time and temperatures between 20 and 33 °C.

The rheological parameters were evaluated during the dormant period of cement hydration detected from heat evolution measurements using a calorimetric approach. As shown in Fig. 1, a change in slope on the heat flux curve (Φ expressed in W or W/m^2) or a net increase in heat Q (J/g) can be easily detected for the M1 mixture tested at 27 °C. This elapsed time corresponds to the end of the dormant period and is referred as the final time or final elapsed time of the dormant period (t_f). The t_f value is established when an increase in heat flux superior to the standard variation calculated on data collected digitally during the steady period of heat flux is observed. This reflects the beginning of the induction period where an evolutive flux due to the acceleration of the rate of cement hydration takes place. It is important to note that the end of the dormant period is usually considered as the point where the tangents of the slopes of heat curves during the dormant period and acceleration period of cement hydration intersect [13,18]. However, this approach was not retained, rather the elapsed time t_f was taken.

4. Results and discussion on mortars and SCC

4.1. Coupled effect of temperature and elapsed time on the variation of rheological parameters of mortars

The normalized time t', used herein as new time parameter, enables the comparisons of variations in rheological parameters of the various mixtures at the same relative scale of time, regardless of the length of the dormant period affected by temperature. Therefore, the normalized t' value is a non-dimensional parameter that varies from 0 to 1. The parameter t' can be calculated as the ratio of any given elapsed time within the dormant period to the final time t_f ($t'=t/t_f$). As defined earlier, the final time (t_f) corresponds to the end of the dormant period, which is taken here as the first deviation from linearity established from the calorimetric approach. This reflects the beginning of the induction period where an evolutive flux due to the acceleration of the rate of cement hydration takes place. It is considered that the hydration reaction is mainly kinetic, thus

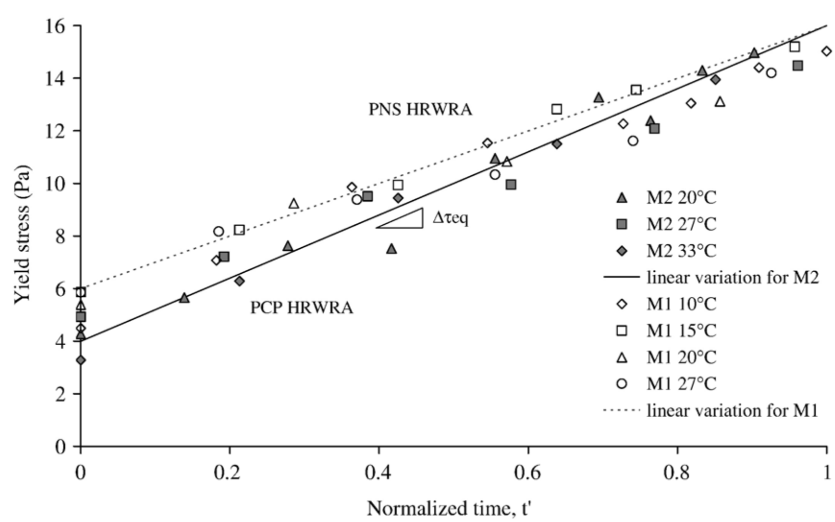

Fig. 4. Variations of yield stress for M1 and M2 mortars made with PNS and PCP HRWRA according to the normalized time for temperatures between 10 and 33 °C.

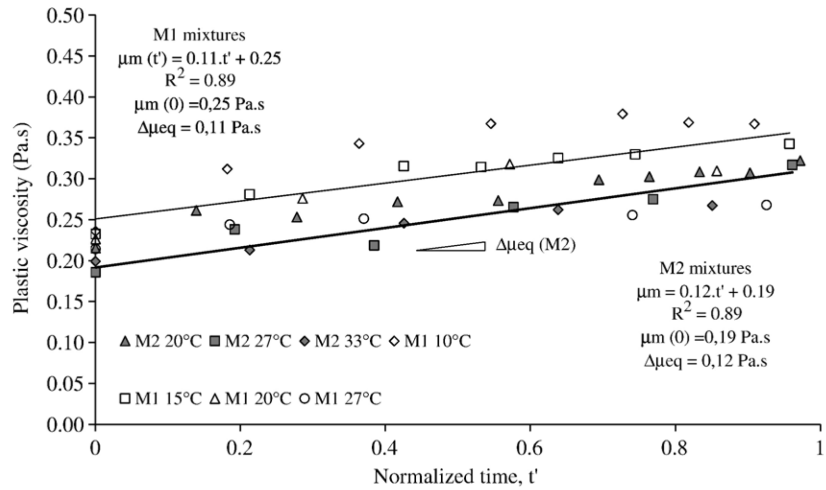

Fig. 5. Variations of plastic viscosity for M1 and M2 mortars taking in consideration the normalized time for temperatures between 10 and 33 °C.

thermo-activated, up to t_f. As a consequence, the t' value permits not to take in account the stiffening or delaying of the cement paste mainly due to temperature.

As shown on Figs. 2 and 3 for M1 and M2 mixtures respectively, the variation of yield stress ($\tau_{o,m}$) in time on mortar mixtures can be expressed as a linear relationship for a given test temperature (T). The slope ($\Delta\tau_o$) corresponds to the increase in yield stress with time at a given temperature and is a function of the temperature of the test. In order to eliminate the effect of temperature, the increase in yield stress is plotted as a function of the normalized time t'. Since the degree of hydration of cement is quite low during the dormant period, the use of the normalized time t' can enable direct comparison among various mixtures at similar relative duration within the dormant period. The relationships between $\tau_{o,m}$ and t' of mortars are given in Fig. 4. The yield stress is shown to increase with the increase in the normalized time period (t'), independently of the mixture temperature. It means that, regardless of temperature, and regardless of the initial adsorption of the HRWRA, the rate of formation of hydrates is constant which translates a kinetic global reaction that leads to the same amount of formed hydrates for each of the tested mixtures. Temperature only affects the reaction kinetics, lengthening the dormant period and thus t_f.

At t' of 0, the initial difference in yield stress of mortar mixtures tested on temperatures varying between 10 and 30 °C is limited to 1.5 Pa. The effect of temperature on the initial adsorption of the HRWRA [19] is hidden by the flowability of mortars, although it exists. As a consequence, the initial yield stress $\tau_{o,m}$, which should be highly dependant on temperature, varies in a restricted interval of 1.5 Pa (Figs. 2–4). Thus, the linear evolution in yield stress with normalized time can be written as (Eq. (2)):

$$\tau_{o,m}(t') = \tau_{o,m}(0,T) + \Delta\tau_{eq}.t' = \tau_{o,m}(0,T) + \Delta\tau_{eq}\left(\frac{t}{t_f}\right). \qquad (2)$$

The term $\Delta\tau_{eq}$ corresponds to the slope of the $\tau_{o,m}$ vs. t' plot and can be used to express the increase in yield stress with normalized time for each mixture, and $\tau_{o,m}(0,T)$ is the initial yield stress at the test temperature. As the modus operandi was to target a same Marsh flow time to get the dosage of HRWRA, the

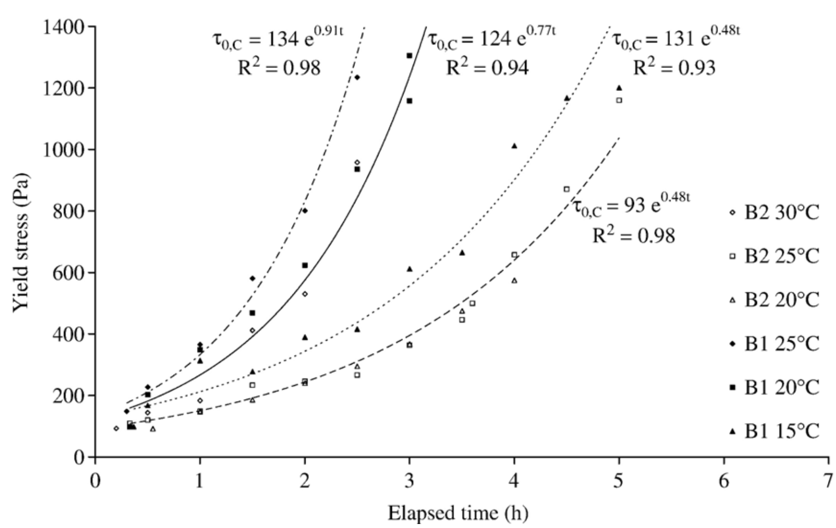

Fig. 6. Variations of yield stress for B1 and B2 concretes according to the elapsed time and temperatures between 15 and 30 °C.

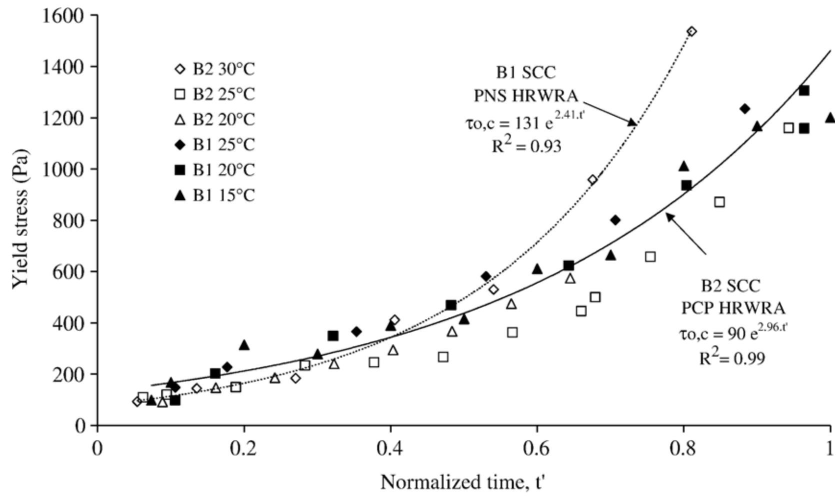

Fig. 7. Variations of yield stress for B1 and B2 concrete according to the normalized time for temperatures between 15 and 30 °C.

obtained mixtures are drastically different in terms of composition (Table 2). As a consequence, having a $\Delta\tau_{eq}$ parameter depending on the mixture proportioning was expected.

Based on the results presented in Fig. 5, conclusions similar to those presented for the evolution of yield stress with the normalized time t' can be made for the evolution of plastic viscosity. The variations of μ_m of mortars with normalized time present a linear and unique trend for each mixture, independently of the test temperature. The slopes of the linear regressions ($\Delta\mu_{eq}$), which refers to the relative increase of plastic viscosity, are found to be close for all tested mixtures. For the M1 and the M2, the $\Delta\mu_{eq}$ parameter reflects the steadiness of the rate of formation of hydrates, translating a kinetic global reaction. For each tested mixtures and at the same relative time, the same amount of hydrates is formed. Consequently, the same decrease of the paste fluidity (defined as the inverse of viscosity) at equivalent t' value is observed. However, dispersion is higher on the data presented for the variations of μ_m with t' than with the yield stress. It is mainly due to the imprecision of the measurement apparatus.

The following equation (Eq. (3)) is proposed to describe the variations of μ with either elapsed time or t':

$$\mu_m(t) = \mu_m(0,T) + \Delta\mu_{eq}.t' = \mu_m(0,T) + \Delta\mu_{eq}.\left(\frac{t}{t_f}\right) \quad (3)$$

μ_m (0, T) is the initial plastic viscosity at the test temperature.

4.2. Coupled effect of temperature and elapsed time on the variation of rheological properties of SCC

As shown as raw results on Fig. 6, the yield stress ($\tau_{o,c}$) of the tested SCC mixtures varies exponentially with the elapsed time for each test temperature. Based on these results, a model for the evolution of $\tau_{o,c}$ with elapsed time can be expressed as follows (Eq. (4)):

$$\tau_{o,c}(t) = \tau_{o,c}(0,T)e^{p(T).t} \quad (4)$$

where $p(T)$ is an experimental constant depending on the test temperature, and $\tau_{o,c}(0, T)$ is the initial yield stress expressed as a function of temperature.

The evolution of $\tau_{o,c}$ with the normalized time, t', is shown in Fig. 7. The resulting exponential variation of $\tau_{o,c}$ with the t' value can be written as (Eq. (5)):

$$\tau_{o,c}(t') = \tau_{o,c}(0,T)e^{\alpha.t'} \quad (5)$$

where α is an experimental constant that depends on the type of concrete. The α values for the tested mixtures at 20 °C are reported in Table 4. Compared to the variations in yield stress with the normalized time t' for the mortar mixture shown in Fig. 4, the increase in yield stress with t' is rather exponential for the concrete mixture. This can be due to the coupled effect of the increase in cohesion of the cement paste with cement hydration and the increase in internal friction among aggregate particles [16].

The evolution of plastic viscosity for SCC mixtures (μ_c) with elapsed time is illustrated as raw results in Fig. 8, and using and normalized time (t') in Figs. 9 and 10, respectively for B2 and

Table 4
Experimental constants for mixtures tested in this investigation: reference values obtained at 20 °C

Mortars	μ_m (0, 20 °C) (Pa s)	$\Delta\mu_{eq}$ (Pa s)	R^2	$\tau_{o,m}$ (0, 20 °C) (Pa)	$\Delta\tau_{eq}$ (Pa)	R^2
M1	0.25	0.11	0.89	5.7	12.4	0.97
M2	0.19	0.12	0.89	4	9.8	0.98
M3	0.27	0.17	0.87	10	12	0.95
M4	0.44	0.28	0.87	16	4.5	0.97
M5	0.39	0.11	0.95	13.8	8.06	0.89
SCC	μ_c (0, 20 °C) (Pa s)	β	R^2	$\tau_{o,c}$ (0, 20 °C) (Pa)	α	R^2
B1	23.32	0.94	0.99	130	2.41	0.93
B2	18.65	1.31	0.86	90	2.36	0.96
B3	25.19	2. 2	0.95	225	2.28	0.92
B4	41.9	3.15	0.9	355	1.44	0.9
B5	27.4	0.93	0.72	315	1.58	0.90

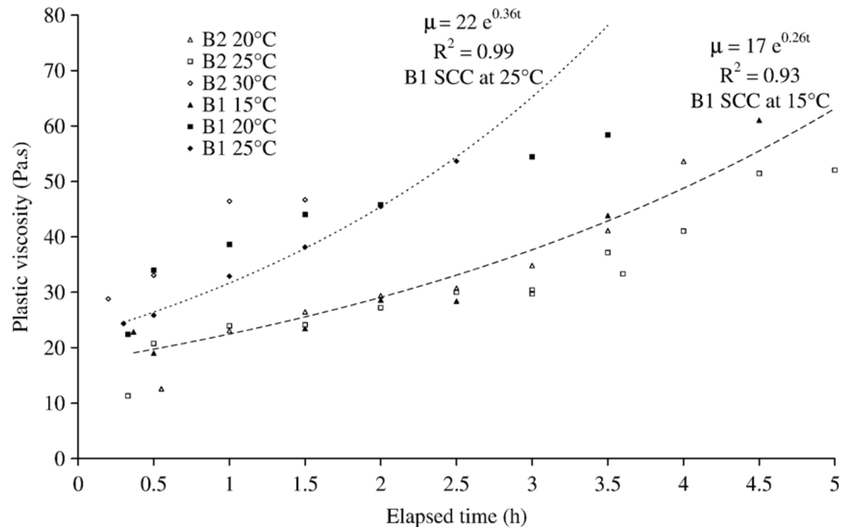

Fig. 8. Variations of plastic viscosity for B1 and B2 concrete according to the elapsed time and temperatures between 20 and 30 °C.

B1 SCC mixtures. Results found on B3 and B4 were comparable. As for the yield stress, an exponential equation is derived to describe the evolution of plastic viscosity with elapsed time (Eq. (6)) and the normalized time (Eq. (7)):

$$\mu_c(t) = \mu_c(0,T)e^{\varepsilon(T).t} \tag{6}$$

$$\mu_c(t') = \mu_c(0,T)e^{\beta.t'} \tag{7}$$

where ε and β are experimental constants and $\mu_c(0, T)$ the initial plastic viscosity depending on test temperature. The β values for the tested mixtures are reported in Table 4. As reported by Ref. [2] for conventional concrete, the plastic viscosity is shown to vary exponentially with the increase in time or temperature on the tested SCC mixtures. Previous research showed that the plastic viscosity decrease with temperature (i.e. fluidity increase with an increase of temperature) [19], however, results found in this investigation show some deviation with this theory. The μ curve for B2 SCC at 30 °C (Fig. 9) was expected below the ones obtained at 20 and 25 °C, and the variation of plastic viscosity for B1 SCC at 15 °C (Fig. 10) should be above the curve obtained at 20 °C. This can be due to an offset current affecting the rotational speed instruction given by the automaton to the rheometer, leading to an overestimation or an under evaluation of the shear rate, creating an offset value of μ. Nevertheless, this phenomena does not affect the proposed equations (Eqs. (6) and (7)) since this offset is included in the initial plastic viscosity value $\mu(0, T)$.

5. Mathematical approach to predict the variation of rheological parameters of SCC from data obtained on mortar

5.1. Viscosity equations

More dispersion is observed on data presented on the variation of μ than on yield stress. This is mainly due to the

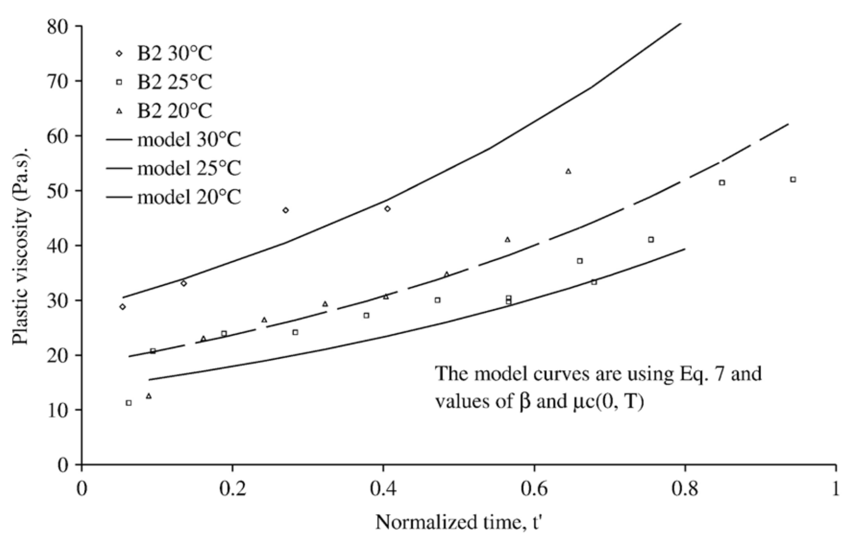

Fig. 9. Variations of plastic viscosity for B2 according to the normalized time t' and temperature between 20 and 30 °C.

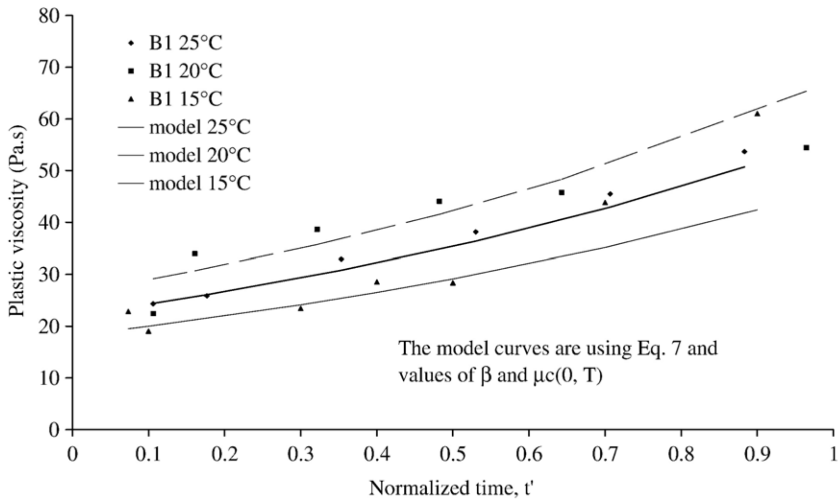

Fig. 10. Variations of plastic viscosity for B1 according to the normalized time t' and temperature between 15 and 25 °C.

sensitivity of the rheometers employed. Anyway, a general linear trend is shown on results presented for mortars, and exponential for concrete. Thus, the general equations to describe the variation of plastic viscosity with t' on mortars (Eq. (3)) and SCC (Eq. (7)) can be taken as granted. The experimental data, $\mu_m(0, T)$ (Pa s), $\Delta\mu_{eq}$ (Pa s), $\mu_c(0, T)$ and β, are summarized in Table 4.

It is observed that the ratio between initial plastic viscosity on mortar mixtures ($\mu_m(0, T)$) and on concrete ($\mu_c(0, T)$) is quite constant on all sets of tested mixtures (Eq. (8)). This ratio μ_{re} can be defined as the relative viscosity between SCC and its corresponding mortar [16].

$$\mu_{re} = \frac{\mu_c(0,T)}{\mu_m(0,T)}. \tag{8}$$

While the μ_{re} parameter seems to depend on temperature (Table 5), it has been chosen to take the average value of 93.3 to develop a mathematical approach as previous investigations showed that the relative viscosity only depends on mixtures composition [2,16]. The following equation, given by Ref. [2] expresses the relative viscosity using the volumetric concentration of differential aggregates (V), the solid volume ratio of differential aggregates (C), the aggregates fineness modulus of differential aggregates f, and two experimental constants, a and b (Eq. (9)):

$$\mu_{re}(t') = \mu_c/\mu_m = (1-V/C)^{-(a.f+b)}. \tag{9}$$

The differential aggregates are made with the fraction of sand and coarse aggregates retained on the 315 µm sieve or the granular portion of SCC removed to obtain the corresponding mortar.

From Eqs. (7) and (8), the variations of plastic viscosity on SCC with normalized time ($\mu_c(t')$) can be expressed, using μ_{re} and the initial plastic viscosity on mortar, as (Eq. (10)):

$$\mu_c(t') = \mu_m(0,T).\mu_{re}\exp^{\beta t'}. \tag{10}$$

A logarithmic correlation was found between the β value obtained on SCC and $\Delta\mu_{eq}$ obtained on the corresponding mortars. The β value can be calculated, for tested mixtures, as (Eq. (11)):

$$\beta = 2.3\ln(\Delta\mu_{eq}) + 6.2. \tag{11}$$

A correlation coefficient (R^2) of 0.98 was calculated for Eq. (11). Thus, for tested mixtures, the variation of plastic viscosity with normalized time on SCC could be described by an equation from which all experimental constants depend on the corresponding mortar (Eq. (12)):

$$\mu_c(t') = \mu_m(0,T).(1-V/C)^{-(a.f+b)}.\exp[[2.3\ln(\Delta\mu_{eq}) + 6.2].t']. \tag{12}$$

As the only constant parameters between the tested mixtures were the aggregates type and proportioning and

Table 5
Experimental values for relative viscosity and relative yield stress

Mixtures	Temperature (°C)	μ_m (0, T) (Pa s)	μ_c (0, T) (Pa s)	μ_c (0, T)/μ_m (0, T)	μ_{re}	$\tau_{o,m}$ (0, T) (Pa s)	$\tau_{o,c}$ (0, T) (Pa s)	$\tau_{o,c}/\tau_{o,m}$ (0, T)	τ_{re}
M1 and B1	15	0.23	18.2	79.1	93.3	5.9	125.4	21.3	22.6
	20	0.22	26.4	120.0		5.4	115.8	21.4	
	25	0.21	22.1	105.2		5.8	133.6	23.0	
M2 and B2	20	0.2	13.8	69.0		3.7	71.3	19.3	
	25	0.25	18.2	72.8		4.2	92.5	22.0	
	30	0.25	28.4	113.6		3.4	97.4	28.6	

Table 6
Correlation coefficients (R^2) calculated between experimental and predicted values for yield stress and plastic viscosity on mixtures tested in this investigation

Concrete proportioned with PNS-HRWRA

	B1			B3		
Temperature, °C	15	20	25	13	20	27
R^2 on yield stress	0.97	0.97	0.99	0.94	0.93	0.98
R^2 on plastic viscosity	0.94	0.90	0.99	0.93	0.83	0.95

Concrete proportioned with PCP-HRWRA

	B2			B4				B5	
Temperature, °C	20	25	30	15	20	25	30	20	30
R^2 on yield stress	0.95	0.99	0.97	0.95	0.99	0.98	0.92	0.83	0.99
R^2 on plastic viscosity	0.97	0.95	0.93	0.99	0.98	0.93	0.91	0.99	0.95

the *W*/*B* ratio, the model can be generalized as follows (Eq. (13)):

$$\mu_c(t') = \mu_m(0,T).(1-V/C)^{-(a.f+b)}.\exp[[\gamma\ln(\Delta\mu_{eq})+\varepsilon].t'] \quad (13)$$

where γ and ε are experimental constant that should only depend on aggregates and the *W*/*B* ratio. Investigations are under way to provide evidence of this theory and the results will be presented in a future paper.

5.2. Yield stress equations

As for the plastic viscosity, the relative yield stress, defined as the ratio between initial yield stress on mortars ($\tau_{o,m}(0, T)$) and on concrete ($\mu_{o,c}(0, T)$), is quite constant. Such relationship can be expressed as (Eq. (14)):

$$\tau_{re} = \frac{\tau_{o,c}(0,T)}{\tau_{o,m}(0,T)}. \quad (14)$$

For the sake of this investigation and as previous researchers showed that this parameter depends on the mixtures composition [3], the relative yield stress has been taken as the average value of τ_{re} calculated at various temperatures on M1 and B1 and M2 and B2 mixtures (Table 5). To enable a mathematical approach, a τ_{re} value of 22.6 was used afterwards.

From the previous equation, and using Eq. (5) to describe the variations of the yield stress on SCC, the following model is proposed to reflect $\tau_{o,c}(t')$ (Eq. (15)):

$$\tau_{o,c}(t') = \tau_{o,m}(0,T).\tau_{re}.\exp^{\alpha t'}. \quad (15)$$

Ref. [3] proposed the following equation to calculate τ_o with granular compacities (Eq. (16)):

$$\tau_o = \exp\left(2.537 + \sum_{i=1}^{n} a_i.K_i'\right). \quad (16)$$

The term K_i' is the contribution of the considered fraction (*i*) to the mixture compaction indicia. Therefore, a_i can be

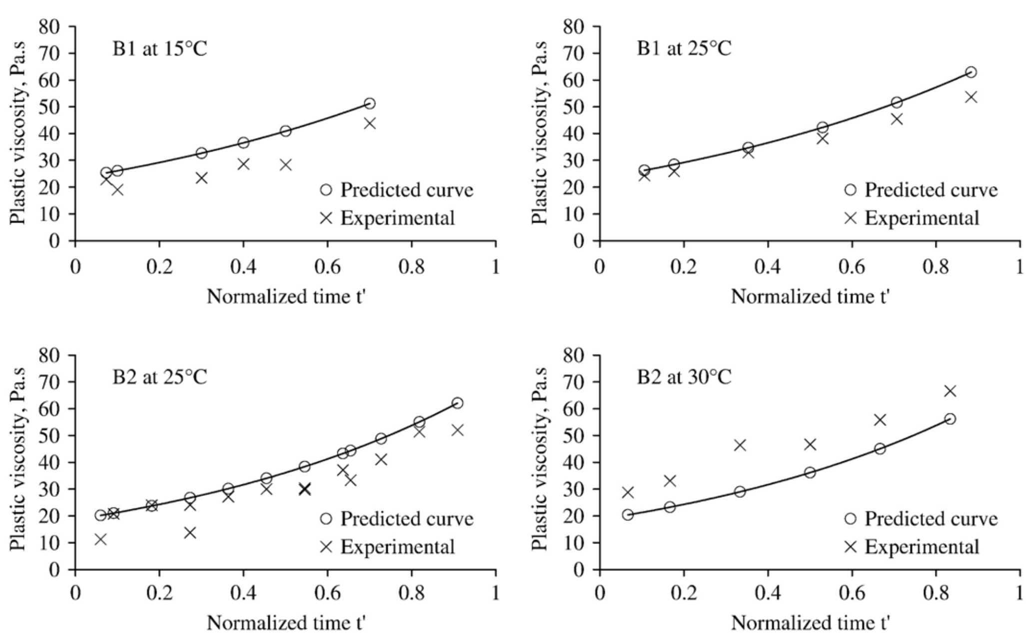

Fig. 11. Comparison of theoretical plastic viscosity curves with experimental results for B1 and B2.

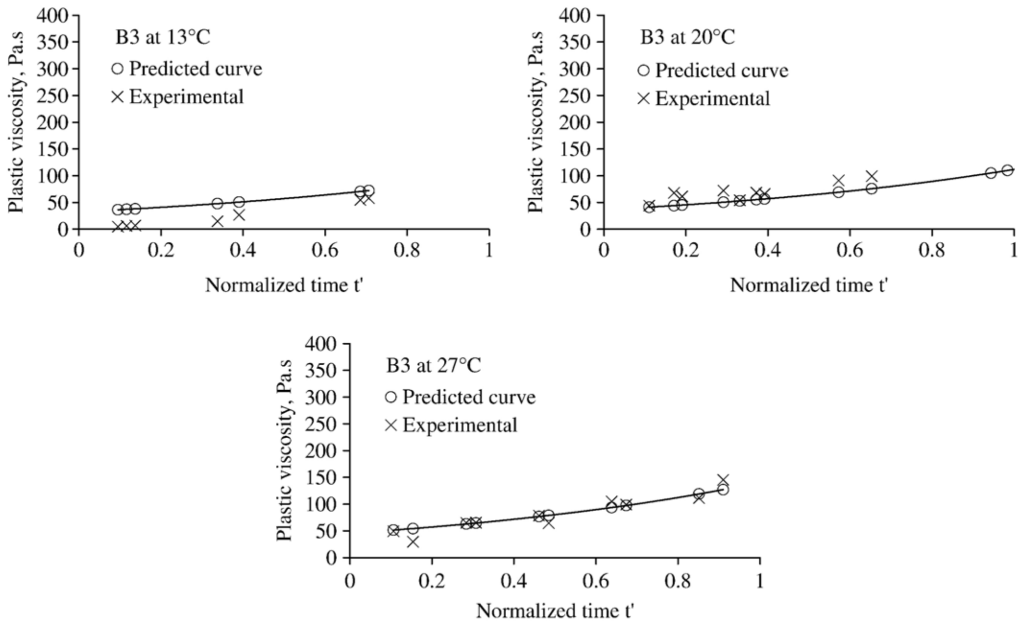

Fig. 12. Comparison of theoretical plastic viscosity curves with experimental results for B3.

calculated, using the average size of every considered granular classes d_i, as (Eq. (17)):

$$a_i = 0.736 - 0.216\log(d_i). \quad (17)$$

Introducing p as the maximum indicia of the granular classes for mortars and n for concrete, and using Eq. (16), Initial yield stress and plastic viscosity can be written as (Eqs. (18) and (19)):

○ For mortar mixtures

$$\tau_{\mathrm{o,m}}(0) = \exp\left(2.537 + \sum_{i=1}^{p} a_i.K_i^{'}\right) \quad (18)$$

○ For concrete mixtures:

$$\tau_{\mathrm{o,c}}(0) = \exp\left(2.537 + \sum_{i=1}^{n} a_i.K_i^{'}\right). \quad (19)$$

Consequently, the relative yield stress (τ_{re}) can be calculated as the ratio between Eqs. (19) and (18). The following relationship is obtained (Eq. (20)):

$$\tau_{\mathrm{re}} = K = \frac{\tau_{\mathrm{o,c}}(0,T)}{\tau_{\mathrm{o,m}}(0,T)} = \exp\left[\sum_{i=p}^{n} a_i \cdot K_i^{'}\right]. \quad (20)$$

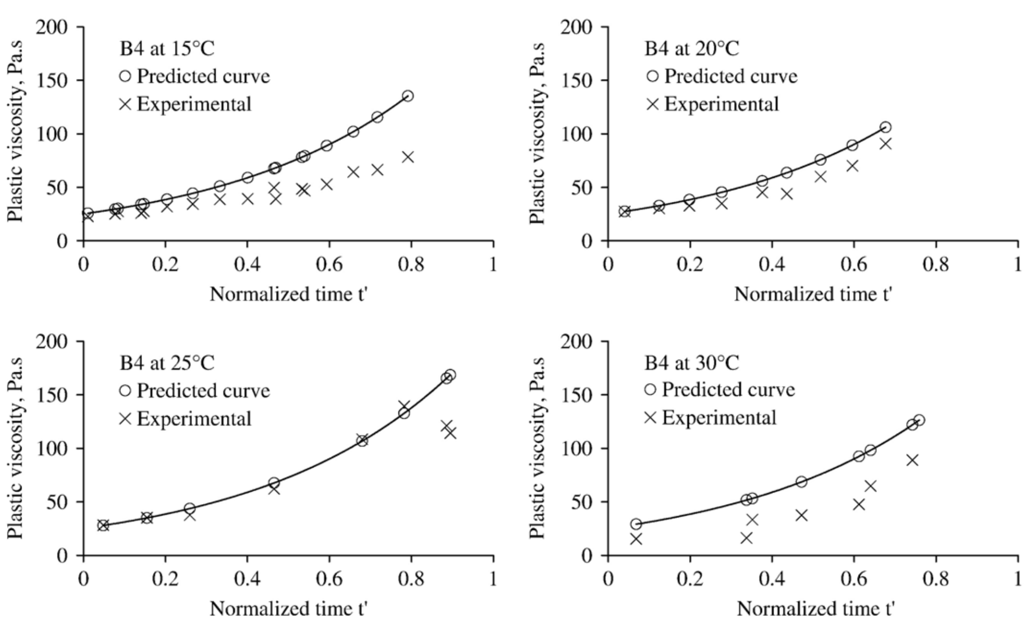

Fig. 13. Comparison of theoretical plastic viscosity curves with experimental results for B4.

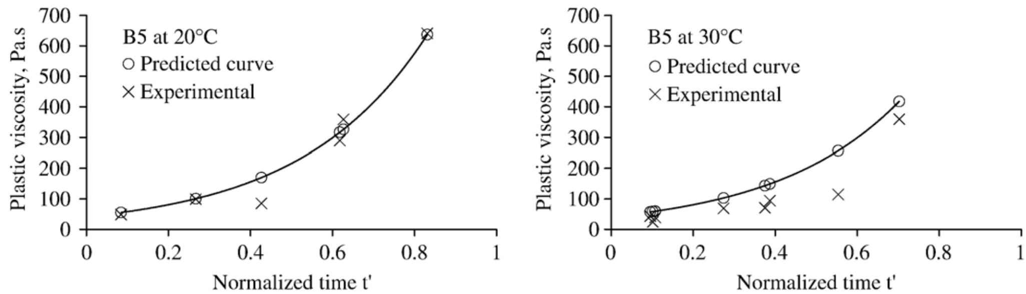

Fig. 14. Comparison of theoretical plastic viscosity curves with experimental results for B5.

As a consequence, the variation of the yield stress on SCC with normalized time can be rewritten as (Eq. (21)):

$$\tau_{o,c}(t') = \tau_{o,m}(0,T).\exp\left[\sum_{i=p}^{n} a_i \cdot K_i'\right].\exp[\alpha.t'] \quad (21)$$

It is to notice that the effect of the HRWRA, which was described as supplementary terms in Eqs. (18) and (19) by Ref [3], is included in the proposed model through the value of the measured initial yield stress on mortar $\tau_{o,m}$ (0).

A logarithmic correlation was found between the α value (Eq. (5)) obtained on SCC and $\Delta\tau_{eq}$ (Eq. (2)) obtained on the corresponding mortar (Eq. (22)):

$$\alpha = 0.94\ln(\Delta\tau_{eq}) + 0.06 \quad (22)$$

Thus, the yield stress with normalized time on SCC could be described by an equation from which all experimental constants depend on the corresponding mortar (Eq. (23)):

$$\tau_{o,c}(t') = \tau_{o,m}(0,T).\exp\left[\sum_{i=p}^{n} a_{i.} \cdot K_i'\right].\exp[(\sigma.\ln(\Delta\tau_{eq}) + \theta)t'] \quad (23)$$

where σ and θ are experimental constant found to be respectively 0.94 and 0.06, and the correlation coefficient R^2 was calculated as 0.94. These constants should only depend on aggregates and the *W*/*B* ratio; nevertheless, more investigations are needed to establish it and the results will be presented in a future paper.

6. Experimental validation of the proposed models

6.1. Plastic viscosity equations

Correlation coefficients (R^2) calculated using experimental data vs. predicted values are ranging from 0.93 to 0.99 on SCC proportioned with PNS-HRWRA, and from 0.91 to 0.99 on mixtures made with PCP-HRWRA (Table 6). It is remarkable that the correlation obtained with all data is quite good (Figs. 11–14). The obtained data are matching the exponential trend calculated using the model. Nevertheless, a constant discrepancy, due to the $\mu_m(0, T)$ value experimentally measured on mortars, is seen on most curves. It seems that the proposed model is very sensitive to the quality of the rheological results obtained on the thinnest part of the SCC since a 6% standard deviation on the plastic viscosity measured on mortar mixtures can lead to a difference of 10 Pa s between predicted an experimental values of μ on SCC. Even so, the proposed model

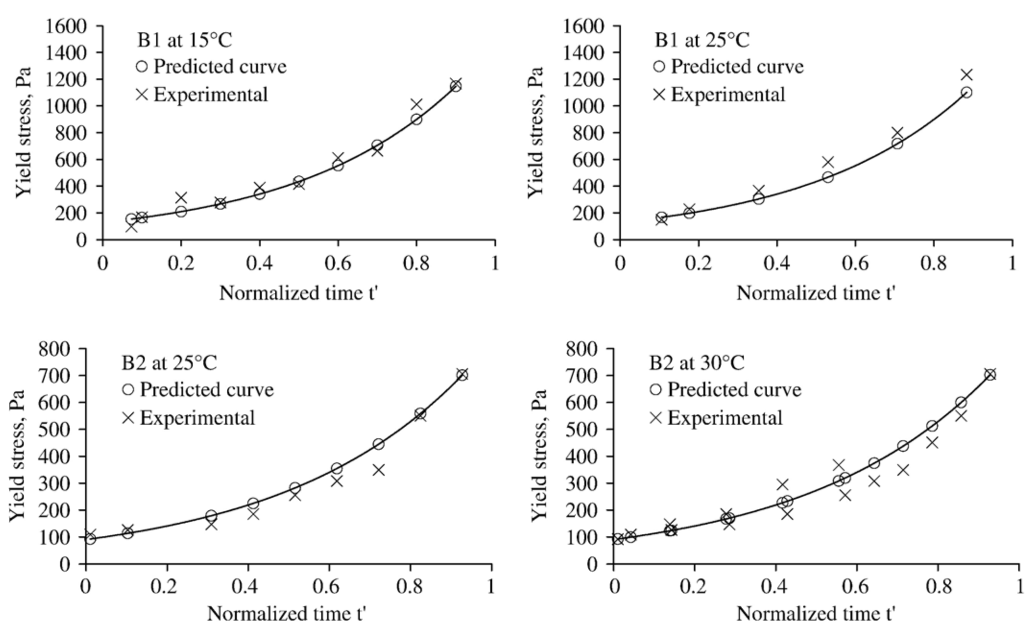

Fig. 15. Comparison of theoretical yield stress curves with experimental results for B1 and B2.

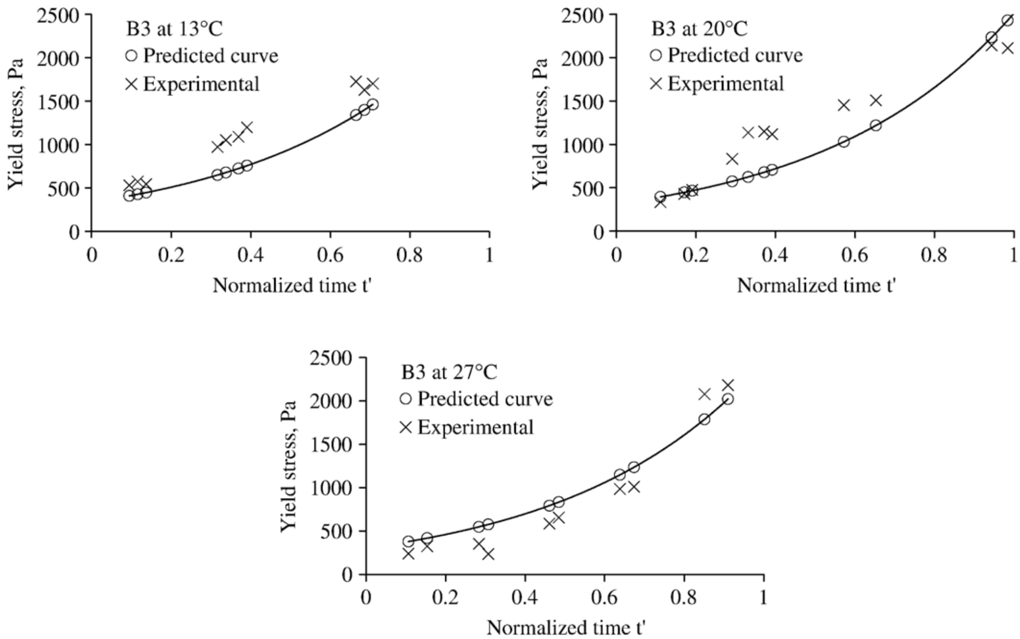

Fig. 16. Comparison of theoretical yield stress curves with experimental results for B3.

is reliable and can be employed to accurately estimate the variations of plastic viscosity with normalized time on concrete mixtures.

6.2. Yield stress equations

As can be observed in Figs. 15–18, the proposed model is effective to estimate the variation of yield stress on SCC with normalized time knowing the initial yield stress on the corresponding mortar at a given temperature. For mixtures proportioned with PNS-HRWRA, the fitness of the model is significant, with R^2 coefficients ranging from 0.83 to 0.99 (Table 6). Nonetheless, the general equation proposed in this study cannot fit the behaviour observed on SCC made with PCP-HRWRA and made at low temperature. For B4 concrete tested at 15 °C, it is first observed a retention of the yield stress until a t' value of 0.6 from which an increase of τ with normalized time is seen (Fig. 17). A similar behaviour is seen with B5 mixture tested at 20 °C (Fig. 18). Such behaviours had already been observed and described [8], but they cannot be integrated in a general equation since the thermodynamic of the reaction obeys more complex laws as for mixtures made with PCP-HRWRA

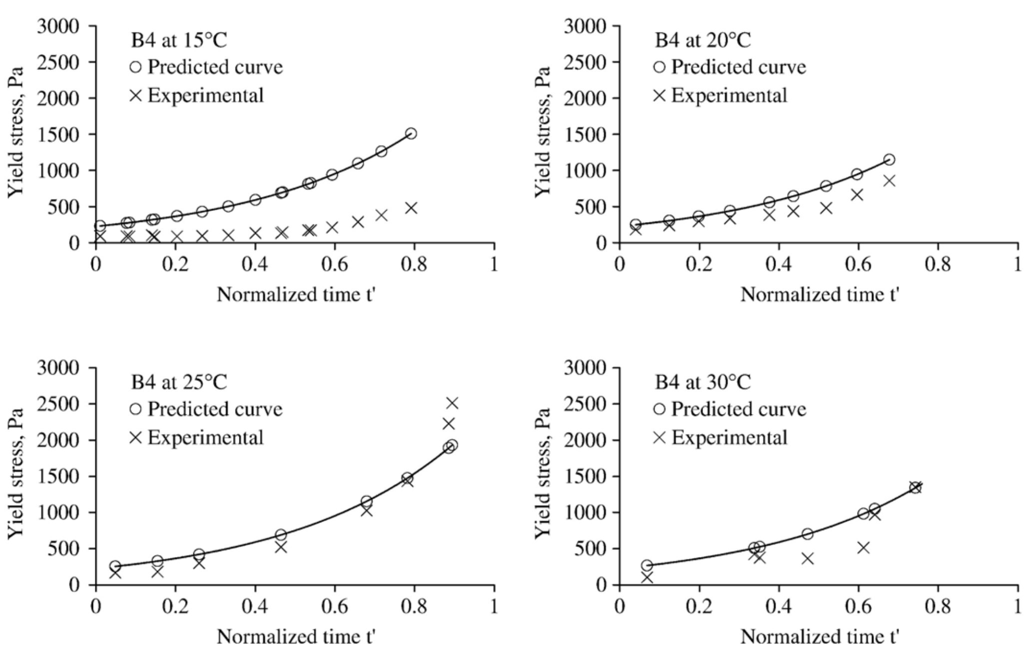

Fig. 17. Comparison of theoretical yield stress curves with experimental results for B4.

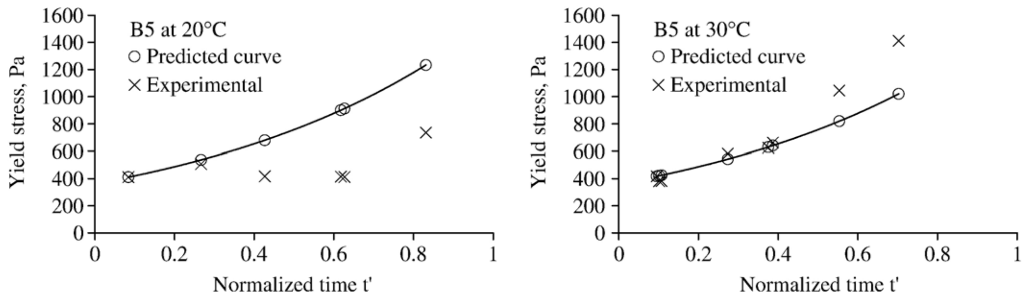

Fig. 18. Comparison of theoretical yield stress curves with experimental results for B5.

and tested at high temperature or prepared with PNS-HRWRA [8,20]. Ref. [20] proposed that, for mixtures made with PCP HRWRA, a critical temperature leads the behavior of the SCC. When the mixture temperature exceeds this threshold value, τ_0 is shown to vary exponentially with either the elapsed time or the normalized time. For mixtures prepared below the critical temperature, the variation of the yield value can be separated into three stages. At first, the yield value is slowly increasing or remains constant before undergoing a decrease with time. After reaching a minimum value, defined by Ref [20] as the "end of steric effect", a sharp increase in the yield value will then take place up to the end of the dormant period.

Some deviation is observed between predicted and experimental curves on B2, B4 and B5 SCC at 30 °C, pointing out a weakness of the proposed model at high temperatures. Even so, the proposed mathematical model is reliable in most studied cases and can be employed to accurately estimate the variations of the yield stress with normalized time on concrete mixtures.

6.3. Perspective

Further investigations are under way to improve the proposed models, taking into account the dosage and nature of the additions and the aggregates, their water demand, the effect of *W*/*B*, and the nature of the binder. They aim at evaluating the empirical data, such as σ and θ for the yield stress equation (Eq. (23)) and γ and ε for the plastic viscosity equation (Eq. (13)). Results will be presented in a future paper.

7. Conclusions

The influence of temperature and time on the evolution of the rheological properties and heat flux of mortars were investigated. Based on the results presented in this paper, the following equations to describe the variations of the yield stress and plastic viscosity are proposed:

1. The variations of the yield stress with normalized time t′ on SCC can be deduced from the knowledge of the data obtained on the corresponding mortar which are the initial yield stress at the test temperature, $\tau_{o,m}(0, T)$, the increase in yield stress with normalized time ($\Delta\tau_{eq}$), experimental constants (σ and θ), and measurement data necessary for the use of De Larrard granular compaction model which are the maximal granular classes of the two materials (*p* and *n*), the contribution of all considered fraction (*i*) to the mixture compaction indicia (K'_i), the average size of every considered granular classes (d_i). The following equation is suitable to describe these variations:

$$\tau_{o,c}(t') = \tau_{o,m}(0,T).\exp\left[\sum_{i=p}^{n} a_i \cdot K_i^{'}\right].\exp[(\sigma.\ln(\Delta\tau_{eq}) + \theta)t']$$

2. The knowledge of initial plastic viscosity at the test temperature $\mu_m(0, T)$, the rate of increase in plastic viscosity with normalized time ($\Delta\tau_{eq}$), experimental constants (γ and ε) on the corresponding mortar, and measurement data necessary for the use of Murata and Kukokawa model which are the volumetric concentration of differential aggregates (*V*), the solid volume ratio of differential aggregates (*C*), the aggregates fineness modulus of differential aggregates *f*, and three experimental constants (*a*, *b* and *β*) allows describing the variations of plastic viscosity on SCC with the normalized time using the equation:

$$\mu_c(t') = \mu_m(0,T). \quad (1-V/C)^{-a.f+b}.\exp[[\gamma\ln(\Delta\mu_{eq}) + \varepsilon].t']$$

3. A database can be created with experimental data obtained on either the aggregates or the mortar at different temperatures to match the average temperatures encountered at the batching plant each season. From the knowledge of these data and the use of the equations proposed in this investigation, the variation of the yield stress and plastic viscosity of SCC can be predicted, and even computerized, in order to enhance concrete robotization at the batching plant and adapt the concrete proportioning to the ambient casting temperature. Since the data necessary to forecast the SCC behaviour are obtained on mortars or aggregates (to obtain *n*, *p*, *V*, *C*), the proposed methodology will probably save money avoiding costly tests on concrete.

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