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Coupled effect of time and temperature on variations of plastic viscosity of highly flowable mortar

Jean-Yves Petit [a],*, Kamal H. Khayat [b], Eric Wirquin [a]

[a] Université d'Artois, LAMTI, Technoparc Futura, F62400 Béthune, France
[b] Université de Sherbrooke, 2500 Bd Université, Sherbrooke, Qc, Canada J1K 2R1

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ABSTRACT

Self-consolidating concrete (SCC) is being increasingly used as construction material for its workability. However, the rheological properties of such concrete, which is made with significant concentration of high-range water-reducing admixture (HRWRA), depend in most cases on the casting temperature of the material. The study presented herein aimed at evaluating the coupled influence of time and temperature on the variations of plastic viscosity (μ) of micro mortar made with polymelamine (PMS), polynaphtalene (PNS) and polycarboxylate (PCP) polymer. In total, seven micro mortar mixtures proportioned with various binder compositions and water-to-binder ratios of 0.42 and 0.53 were prepared at 10 to 33 °C. Test results show that the plastic viscosity varies linearly with the coupled effect of time and temperature for mixtures made with PNS or PMS HRWRA. However, for mixtures made with PCP-HRWRA, both temperature and mixture proportioning have influence on the variation of viscosity with time.

1. Introduction

In order to improve workability of concrete, high-range water-reducing admixtures (HRWRAs) are incorporated into the cement mixture. The most commonly used HRWRAs are polynaphtalene sulfonate (PNS) and polymelamine sulfonate (PMS). New classes of long-chained polymers with significant molecular weights have been recently developed to further enhance water reduction and its retention with time through a more effective steric dispersing mechanism. Polycarboxylate polymers (PCPs) constitute a new family of these newly-used admixtures. The dispersion properties of PCPs on cement particles is especially noted in mixtures made with low water-to-binder ratios (W/B), compared to PNS- or PMS-based HRWRA that can exhibit sharper drops in fluidity with time and, in some cases, delay in setting [1].

The mixture composition and mixing procedure have some impact on the efficiency of PCP-based HRWRA in improving fluidity of cement paste [2,3]. A relatively high concentration of sulfate-ion in the interstitial solution can lead to some incompatibility between the binder and HRWRA, thus affecting the adsorption behavior of the PCP onto cement particles, resulting in a loss of workability [4–8]. The cement hydration process was also shown to reduce dispersion efficiency of the HRWRA due to some intercalation of the HRWRA into the hydration products [7–9]. Interaction between cement, viscosity-enhancing agents (VEA) and HRWRA can also lead to loss in fluidity or delay in set time; this depends on the concentration and type of the admixtures [10–12]. Leeman and Winnefeld [12] reported that a reduction of W/B from 0.48 to 0.38 can lead to an increase in plastic viscosity from 5 to 22 Pa s in mortar systems made with PCP-HRWRA. Such observation was also reported for mortars containing natural polysaccharide VMA at dosages of 0.2%, 0.4%, and 0.8% by mass of binder, for mortar mixtures with 0.38 to 0.48 W/B. In some cases, the reduction of W/B can cause sharper increase of plastic viscosity than an increase in VMA at an equivalent yield stress [12].

Temperature also affects the level of incompatibility between the cementitious materials and various admixtures [13–18]. Golaszewki and Szwabowski [15] showed that the rheological properties of mortar mixtures made with PNS and PCP HRWRAs are strongly influenced by mixture temperature. An increase in mixture temperature can lead to an increase in yield value and decrease in initial plastic viscosity. For mortars prepared at a W/B of 0.40 to 0.50 and 1% to 2.25% HRWRA dosage, by mass of binder, the increase in temperature from 10 to 30 °C was found to decrease the plastic viscosity (μ with time; this behavior was found to depend on the binder type. Similar results were found on micro mortars prepared with W/B of 0.42 to 0.53 and either PNS or PMS HRWRA at dosage rates ranging between 0.28% and 0.8%, by mass of binder [16]. The increase in mixture temperature from 10 to 30 °C resulted in a linear increase in plastic viscosity with time, but a decrease in the initial μ value. A decrease in temperature of the micro mortar resulted in reducing the rate of cement hydration and HRWRA adsorption, thus prolonging the dormant period [16,17]. For example, Jolicoeur et al. [17] showed that the concentration of adsorbed and residual PNS-HRWRA can vary with paste temperature, resulting in direct implication on rheological properties of the paste.

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* Corresponding author. Fax: +33 3 21 63 71 23.
E-mail address: jyves.petit@univ-artois.fr (J.-Y. Petit).

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Changes in rheological properties with time can also be due to the restructuring of the microstructure, cement hydration, variation in free water content with temperature, mixture composition, and interaction between binder and HRWRA. For micro mortars prepared with a W/B of 0.53 and either PNS or PCP-HRWRA at dosage rates of 0.28% and 0.27%, by mass of binder, the plastic viscosity was reported to increase by 50% over the duration of the dormant period, from 0.25 to 0.35 Pa s for mixtures prepared with PNS-HRWRA, and from 0.20 to 0.30 Pa s for mortars made with PCP-based HRWRA [19]. Similar results were found for cement pastes prepared with PNS-HRWRA at dosage ranging from 0.4% to 1%, by mass of binder, and W/B of 0.35 for which fluidity measurements ($1/\mu$) decreased by 40% to 60% within 2 h, depending on the initial HRWRA concentration [20].

The fineness of binder and mineral additions is another factor influencing the rheology. The addition of fly ash as partial cement replacement improves workability [21] and leads to a decrease in the HRWRA demand at a given viscosity [22]. The workability enhancement is explained by the spherical shape of fly ash which permits particles to roll over one other, thus decreasing interparticle friction [23]. In addition, the spherical shape also minimizes the surface to volume ratio of particle, resulting in a higher packing density and lower water demand for a specific workability [24]. However, it was shown that fluidity, defined as the inverse of viscosity, is strongly influenced by the average particle size with a pessimum value [24]. Ferraris et al. [22] also showed that, on cement paste tested at 0.35 W/C and 0.45% HRWRA by active mass of cement, the lowest yield stress value and best rheological properties were achieved for a 12% replacement of cement by ultra-fine fly ash (UFFA) by mass. It was explained that the maximum packing density, at an optimal particle size, helped to achieve maximum fluidity [22,24].

Although the influence of temperature on hydration kinetics and setting of cement is well documented [4,16,19,20], limited data are available on the coupled effect of HRWRA type and temperature on the variation of fluidity of cement-based materials over the dormant period. The study reported herein aims at evaluating the coupled effect of temperature, time and HRWRA type on plastic viscosity. Such data are necessary to highlight some of the mechanisms affecting workability retention of SCC over transportation time, which affect concrete processing. As the study focuses on variations in rheology due to the non-reversible evolution of the microstructure due to hydration, it is note worthy that no evaluation of thixotropy was carried out during this investigation, and that all samples were presheared before each rheological measurement.

2. Materials and mixture proportioning

As summarized in Table 1, rheological parameters and heat flux were determined to monitor either the variation of plastic viscosity and hydration kinetic during the dormant period. The testing program was carried out using micro mortars made with three types of HRWRA (PNS, PMS, and PCP), three binder compositions, and two W/B of 0.42 and 0.53. Temperatures chosen for the tests ranged from 10 to 33 °C. Both the yield stress and plastic viscosity were monitored over time; however, only the results of the variations of plastic viscosity with time and temperature are presented in this paper since the coupled influence of time and the HRWRA on yield stress has already been reported in ref 6 and 19.

Table 1
Summary of experimental program

Micro mortar	M1	M2	M3	M4	M5	M6	M7
10 or 12 °C	X	X		X		X	X
14, 15 or 18 °C	X	X	X	X	X	X	X
20 or 23 °C	X	X	X	X	X	X	X
25 or 27 °C	X	X	X	X		X	
30° or 32 °C		X					
Calorimetry	X	X	X	X	X	X	X
Rheology with co-axial rheometer	X	X	X	X	X	X	X

X refers to tested combination.

Table 2
Micro mortar mixture proportioning

Materials	Micro mortars						
	M1	M2	M3	M4	M5	M6	M7
CEM II B 32.5 R cement, kg/m^3	837	837	732				
CEM I 52.5 cement, kg/m^3				837			
10E SF-FA Ternary cement, kg/m^3	–	–	–		979	979	979
Fly ash, kg/m^3	–	–	76		–	–	–
Sieved sand (≤315 µm), kg/m^3	728	728	728	728	686	686	686
PCP-HRWRA, % of cement mass	–	0.27	0.27	0.27	–	–	0.80
PNS-HRWRA, % of cement mass	0.28	–	–	–	0.50	–	–
PMS-HRWRA, % of cement mass	–	–	–	–	–	0.88	–
W/B	0.53	0.53	0.53	0.53	0.42	0.42	0.42

The micro mortar mixtures are based on SCC mixtures whereby aggregate particles coarser than 315 µm are excluded from the mixture by sieving. 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 [25] using a co-axial rheometer for which the difference between the inner and the outer radius was only of 1.17 mm. It was assumed that no slippage can occur between the cylinders of the viscometer and cementitious material due to the deformability of the tested sample. Thus, the surfaces of the inner and outer cylinder of the viscometer were not roughened since the materials tested in this investigation were highly flowable. Table 2 summarizes the mixture proportioning for the tested micro mortars.

The dosage rates of the HRWRA were determined using a modified Marsh cone to obtain the same initial flow time [26]. The targeted afflux time through 8-mm opening of a sample of 1200 mL was 78 ± 1 s for a flow of 1000 mL. While the HRWRA dosage was adjusted on micro mortar mixtures with fixed flow time, the corresponding SCC had an approximate slump flow of 600 to 650 mm.

The first micro mortar (mixture M1) was based on cast-in-place SCC used in Northern France. This SCC was cast at ambient temperatures varying between 5 and 30 °C. The placement was done up to 90 min after batching, depending on traffic conditions. No special provisions to adjust the mixture composition to the demanded self-consolidating characteristics were made on site. Thus, a stiffening of the concrete was observed depending on the placement conditions. This non-air entrained SCC was proportioned in compliance with European EN 206-1 Standard, with a relatively high water-to-binder ratio (W/B) of 0.53. The M1 mixture was proportioned using crushed limestone sand with a fineness modulus of 2.60, limestone filler, and PNS-HRWRA incorporated at 0.28%, by mass of cement.

The M2 mixture was prepared with the same materials and mixture proportioning as that of the M1 mixture, except for the use of PCP-HRWRA, targeting the same Marsh cone time. The HRWRA dosage in the M2 mixture was 0.27%, which is similar to those obtained with the M1 and M3 mixtures. In the case of the M3 mixture, 12.5% of the cement volume, or 50 kg by mass, was replaced by the same volume of Class F fly ash, without altering the W/B, and hence the paste fraction. This choice was made to keep the paste-to-aggregate volume ratio constant, thus enabling comparison between the two mixtures in terms of rheology. Otherwise, the M3 mixture had the same mixture proportioning and materials as that of the M2 micro mortar.

The M1 to M3 mixtures were prepared with CEM II B 32.5 R cement, complying with the French NF P 15-301 Standard. This cement contains approximately 21% of granulated blast furnace slag and 6% of limestone filler.

The M4 mixture was designed as a variant of the M2 micro mortar changing the CEM II B cement to a CEM I which contains about 97% of

Table 3
Characteristics of binders

Chemical composition (%)				Physical characteristics			
	CEM II B cement	CEM I cement	10E SF-FA ternary cement		CEM II B cement	CEM I cement	10SF-FA ternary cement
SiO_2	22.4	20.4	29.3	Blaine fineness, m^2/kg:	320	410	460
Al_2O_3	6.2	5.0	8.6	Specific gravity:	3.05	3.10	2.93
Fe_2O_3	2.7	3.2	6.0	Setting time (Vicat), min:			
CaO	58.8	63.8	46.8	Initial	180	150	185
MgO	2.2	0.9	1.75	Final:		220	280
NaO_2 Eq.	0.73	0.76	0.62	Compressive strength, MPa			
Clinker	73	97	70	2 days:	21	35	20
Limestone filler	6			7 days:	–	–	26
Blast furnace slag	21			28 days:	48	62	–
Fly ash			25				
Silica fume			5				
Others		3					

clinker. Otherwise, the M4 mixture had the same mixture proportioning as that of the M2 micro mortar.

In the case of the M5 mixtures, the SCC from which the micro mortar was designed had a slump flow of 650 mm. A ternary Canadian binder containing approximately 5% silica fume and 25% Class F fly ash replacements, by mass of binder, was used. A natural siliceous sand of fineness modulus of 2.35 was employed. A PNS-based HRWRA was used for the M5 mixture at 0.50%, by active mass of the binder.

The mixture proportioning of the M6 and M7 mixtures was done as a variant of the M5 mixture: a PMS-based HRWRA was incorporated instead of the PNS-HRWRA for the M6 mortar, and a PCP-HRWRA for the M7 mixture, targeting the same Marsh cone time of 78 s.

The chemical and physical characteristics of the binders are summarized in Table 3.

3. Test methods

The mortars were prepared in batches of 6 L using a mixer with paddles rotating helicoidally at successive speeds ranging between 140 and 285 rpm. The mixing procedure was in compliance with ASTM C 105. The temperature of the raw materials was adjusted to the targeted test temperature. In order to avoid heat loss or gain during mixing, all apparatuses were maintained at the targeted temperature. At the end of mixing, samples were taken to monitor heat generation during the dormant period.

Isothermal calorimetry was adopted for the M1 to M4 mixtures [27]. The SCC mixtures, from which the micro mortars are extracted were used in the construction of wall elements. Thus, the use of isothermal calorimetry, whereby the temperature of the test sample is maintained at the curing temperature, can correspond to actual temperature conditions in relatively narrow formwork. The use of isothermal calorimetry was more appropriate than that of semi-adiabatic calorimetry to assess heat flux in relatively thin sections. The test consisted of maintaining an 90 × 90 × 160 mm prismatic samples at the curing temperature throughout the testing period. A maximal variation of temperature between the core and surface of the sample of 0.5 °C is obtained for a test temperature of 30 °C [19,27].

In the case of the M5, M6 and M7 micro mortars, a semi adiabatic calorimeter was employed to evaluate heat flux [28]. In this method, the sample is introduced into an isolated cell for which the heat loss coefficient α is known. The increase in temperature of the mixture due to cement hydration is then measured. The heat Q, expressed in Joule per gram of binder (J/g), is obtained by adding the heat produced by the binder, knowing the mixture design, the thermal capacity of the various materials, and heat lost which is obtained by integrating the lost heat flux with time.

The monitoring of rheological parameters was terminated when an increase of heat flux (ϕ was obtained from the calorimetric testing, as illustrated in Fig. 1. Instead of considering the end of the dormant period of cement hydration to correspond to the intersection of the slope of the heat of hydration (Q) during the dormant period and the slope of Q during the accelerated period of cement hydration, the end of the dormant period is defined from the heat flux (Φ) curve, as defined in Fig. 1 with the t_f parameter. The t_f value corresponds to the first loss of linearity of the Φ *vs* time chart. This reflects the beginning of the accelerating period where an evolutive flux due to acceleration of the rate of cement hydration takes place, resulting in an increase of heat flux [27,29]. Rheological measurements were determined at set intervals on a micro mortar remixed at low speed of 140 rpm during 60 s. The pre-shearing of the samples before rheological evaluation was made in order to erase any restructuration at rest happening between tests and, thus, to focus on non-reversible evolution of plastic viscosity with time and temperature due to hydration and HRWRA. In order to avoid evaporation between the various periods of rheological assessments, the mortar was kept in the mixing bowl covered with plastic sheet at isothermal temperature matching the targeted test temperature. A 350-mL sample was used for the rheological measurement using a coaxial cylinder viscometer. The shear stress (τ) was determined after a stabilization time of 20 s at a given shear rate ($\mathring{\gamma}$ at five rotation speeds varying between 3 and 300 rpm, which correspond to shear rates of 5.1 to 510 s^{-1}. The yield stress (τ 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. The τ and μ values are derived by regression analysis using the shear stress–shear rate data, assuming a polynomial response (modified Bingham model [30]) where the second order value can be considered as insignificant and is suppressed, the response can be expressed as $\tau = \tau_0 \ \mu p \ \mathring{\gamma}$ representing the behavior of a Bingham fluid. Two replicates were made and tested at 15 and 20 °C for each mixture. Repeatability experiments showed little dispersion

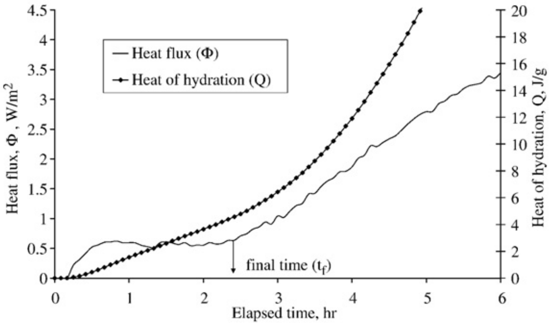

Fig. 1. Variation of heat of hydration (Q) and heat flux (Φ) with time for the M1 micro mortar tested at 27 °C using isothermal calorimetry.

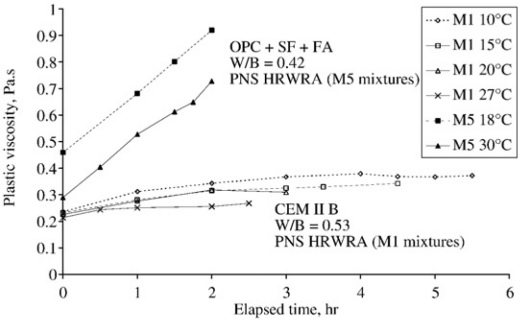

Fig. 2. Variation of plastic viscosity with time for the M1 and M5 mixtures made with PNS-HRWRA tested at 10 to 30 °C.

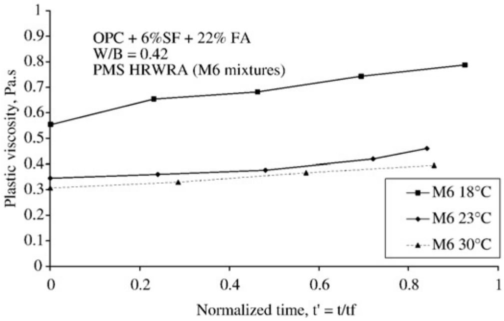

Fig. 4. Variation of plastic viscosity with normalized time for the M6 mixtures made with PMS-HRWRA tested at 18 to 30 °C.

of the results since the maximal relative deviation was 6% for the τ and μ parameters. After each rheological test, the sample which has been already sheared was discarded to ensure the same shear history between replicate test samples.

4. Results and discussion

4.1. Coupled effect of temperature and time on changes in plastic viscosity of micro mortar made with PNS- or PMS-HRWRA

As shown in Fig. 2, the μ values of the M1 and M5 micro mortars prepared with PNS-HRWRA at 10 to 27 °C increase with time. However, the initial plastic viscosity decreases with temperature, so as the rate of increase of μ with time. As a first approach, these variations can be fitted in linear regressions with R^2 values greater than 0.90.

The variations in plastic viscosity of mixtures prepared at different temperatures with either PNS- or PMS-HRWRA are expressed using the normalized t' time in Fig. 3 for M1 and M5 micro mortars (PNS-HRWRA) and in Fig. 4 for M6 mixtures (PMS-HRWRA). This can enable the elimination of the effect of temperature on the hydration kinetics affecting the duration of the dormant period [16,19]. As shown on Figs. 3 and 4, μ varies in a linear trend with the normalized time t'. Moreover, this variation is independent of temperature. The only effect of temperature is to accelerate or slow down the hydration of cement. Thus, comparing the evolution of rheological parameters using the same scale of relative time shows a unique variation for each mixture composition, independently of material temperature. Regardless of the temperature and initial adsorption of the PNS-HRWRA for the M1 and M5 micro mortars, the rate of formation of hydrates, which is related to the slope of the variation of μ with t', can be considered to be constant (Figs. 3 and 4). Nevertheless, this rate is greater for mixtures made with 0.42 W/B (M5 mortars) than those with 0.53 W/B (M1 mortars) as observed on Figs. 2–4. Jolicoeur et al. [17] found that the fluidity ($1/\mu$) of a cement grout prepared with W/B of 0.35 and tested at temperatures of 0 to 40 °C decreases with time in a way inversely to the dosage rate of the PNS-HRWRA; the fluidity is indeed proportional to the residual concentration of HRWRA in the pore solution. Thus, with the normalized rate of hydration shown in Fig. 3 for the M1 and M5 mixtures, it is expected to find a unique trend that is independent of the test temperature. The variation of μ is only dependant on the increase of residual PNS in the solution due to the decrease of adsorbed HRWRA with the hydration progress [17]. From the data presented in Figs. 3 and 4, it can be assumed that these phenomena vary quite linearly with time. Petit et al. [6,18] showed that the evolution of yield stress of micro mortar made with PNS-based HRWRA is dependent mainly on mixture temperature and varies linearly with either the absolute time or normalized time. Such conclusion can also be extended to the variation of plastic viscosity for mixtures made with PNS- or PMS-HRWRA.

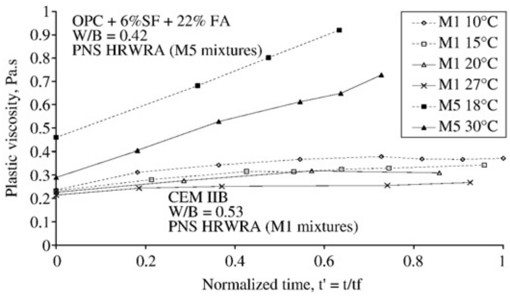

Fig. 3. Variation of plastic viscosity with normalized time for the M1 and M5 mixtures made with PNS-HRWRA tested at 10 to 30 °C.

While the linear evolution of μ with either time or t' is similar to linear restructuration at rest due to thixotropy [31–33], it is important to note that only the evolution plastic viscosity due to the structuration of the cementitious matrix caused by hydration is monitored in this study. Reversible restructuration is effaced using the measurement protocol previously described.

4.2. Coupled effect of temperature and time on changes in plastic viscosity of micro mortar made with PCP-HRWRA

The evolution of plastic viscosity for the M2, M4 and M7 micro mortars with time and normalized time is presented in Figs. 5–7, respectively. As observed by Leeman and Winnefeld [12], the reduction of W/B from 0.53 to 0.42 leads to an increase in initial plastic viscosity on mixtures made with PCP-HRWRA (Fig. 5). The efficiency of the PCP-HRWRA on cement particles dispersal is shown to depend on a critical temperature, as demonstrated by Petit et al. for the variation of the yield stress on mixtures made with PCP-HRWRA [6,16]. Such temperature can have noticeable effect on the rate of adsorption of the PCP-HRWRA onto cement grains, which is in turn

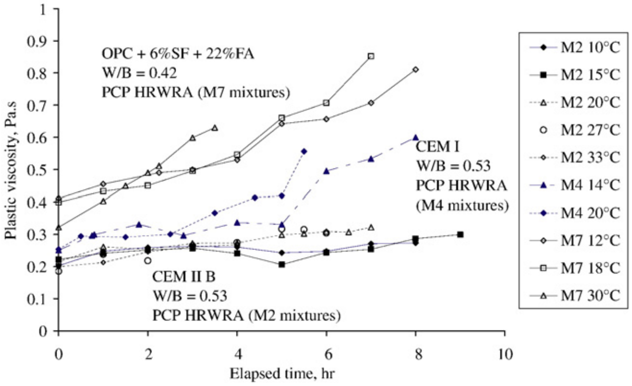

Fig. 5. Variation of plastic viscosity with time for the mixtures made with PCP-HRWRA tested at 10 to 33 °C.

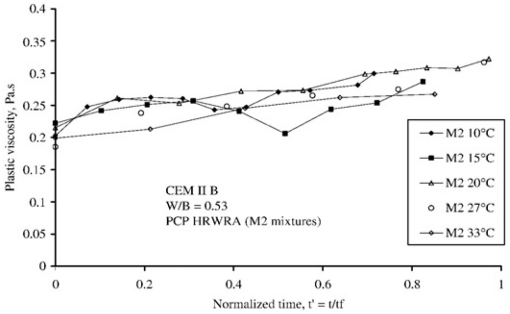

Fig. 6. Variation of plastic viscosity with normalized time for the M2 mixture made with PCP-HRWRA tested at 10 to 30 °C.

affected by the rate of cement hydration and the concentration of sulfate ions in the aqueous solution [4,16]. At a threshold temperature of 15 to 20 °C for M2 mixtures, the plastic viscosity varies in a quite linear trend; however, such behavior is quite different for mixtures tested at 10 and 15 °C. For the M2 micro mortars tested at 10 and 15 °C, an increase of plastic viscosity with time is observed initially before some decrease in μ is seen for t' from 0.30 to 0.50, before undergoing subsequent gain in viscosity (Fig. 6). Similar phenomenon was observed for the M4 micro mortars tested at 14 and 20 °C (Fig. 7). A constancy or slow increase of μ is first observed until a t' value of 0.65 from which the rate of increase of plastic viscosity is amplified. The M7 micro mortar behaves as the M2 mixture with a threshold temperature between 18 and 23 °C (Fig. 7). A gain in the rate of increase of viscosity is observed at t' of 0.5 to 0.6.

Yamada et al. [4] suggested that the efficiency of dispersion of cement particles is related to the concentration of sulfate ions in the cement paste solution. The adsorption of the PCP-HRWRA decreases when the sulfate-ion concentration is high in the aqueous phase, thus affecting the degree of retention of fluidity with time. Yamada et al. [34] also noted that high sulfate-ion concentration in the aqueous phase of cement paste can initially prevent the adsorption of the PCP polymer onto cement grains for mixtures proportioned with 0.3 W/B and 1% PCP-HRWRA, by mass of cement. After a given period of time, the sulfate-ion concentration decreases, allowing more adsorption of PCP-HRWRA onto cement particles. During the first 30 min following the contact of cement with water at 20 °C, the PCP-HRWRWA adsorption was shown to increase with the decrease in sulfate ion concentration in cement paste tested at 5 °C. After 30 min, a decrease in polymer adsorption was measured in spite of the continuous decrease in sulfate-ion concentration. At the lower temperature of 5 °C, a smaller increase in cement hydration and larger decrease in sulfate-ion concentration can lead to an increase in the adsorption of PCP onto cement grains. It is reasonable to think that this competition between the polymer adsorption and sulfate ion concentration can explain the gain in fluidity observed on the tested mixtures made with PCP-HRWRA at low temperatures. At higher temperature, the greater increase in hydration and smaller decrease in sulfate-ion concentration in the solution can decrease the degree of PCP adsorption, thus causing fluidity loss [34]. Moreover, a competition between the adsorption of the PCP-HRWRA onto cement particles and concentration of sulfate ions in the solution takes place, depending on the mixture temperature [16]. The former can promote dispersion of cement particles and enhance fluidity retention, while the latter can contribute to the shrinking of the steric size of the polymer [4], thus resulting in a sharp increase of plastic viscosity. As shown in Figs. 5 and 6, the former phenomenon is observed for the M2 mixtures made at 10 and 15 °C that lie below the critical temperature. With the progress of cement hydration of the M2 mixture at 10 and 15 °C, increase in sulfate-ion concentration at an approximate t' value of 0.50 can lead to the reduction in steric size of the PCP polymer, and hence the retention of fluidity of the mixture. Beyond this limit, μ is shown to increase until the end of the dormant period. Such conclusion can be extended to the M4 and M7 micro mortars with t' values of 0.65 and 0.55 respectively (Fig. 7).

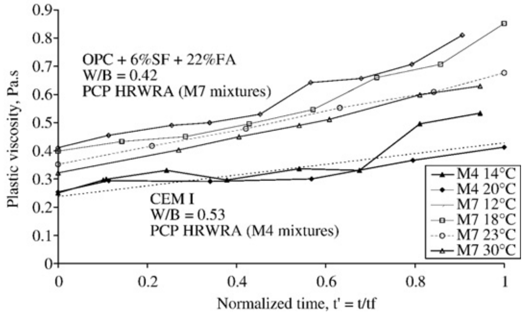

Fig. 7. Variation of plastic viscosity with normalized time for the M4 and M7 mixtures made with PCP-HRWRA tested at various temperatures.

4.3. Effect of fly ash on plastic viscosity of micro mortar made with PCP-HRWRA

Test results from Fig. 8 show that a 12.5% volumetric replacement of part of the cement with Class F fly ash (M3 mixtures) resulted in greater initial viscosity values compared to M2 micro mortars made with 100% CEM II B cement; these mixtures had viscosity values of 0.22 Pa s to 0.27 Pa s for mixtures tested at 10 or 12 °C. Consequently, the M3 mixtures exhibited 20% greater values of plastic viscosity than those monitored on the M2 micro mortars, independently of mixture temperature. It was shown that a replacement of cement by fly ash increases viscosity [35,36]. Sonebi showed that the incorporation of 5% pulverized fly ash in 0.40 W/B grouts containing 0.04% diutan gum as VMA and 1% HRWRA resulted in an increase of plastic viscosity from 0.60 to 0.77 Pa s. Such increase was from 0.45 Pa s to 0.60 Pa s for grouts made with 0.04% welan gum [37]. Uncombusted carbons in the fly ash is known to hinder workability of cementitious materials due to a higher rate of adsorption of HRWRA [38].

Although an initial sharp increase of μ during the first hour is seen for M3 mixtures containing 12.5% FA in volume, a regular increase in plastic viscosity with time is then observed. As opposed to micro mortars made with blended cement (M2 mixtures), no dependency on a critical temperature is observed for mixtures containing fly ash. It can be assumed that, on M3 mortars made with 12.5% FA by volume, the critical concentration of sulfate ions in the solution is reached within the first hour, thus contributing to the shrinking of the steric size of the polymer [4].This can lead to an increase of plastic viscosity

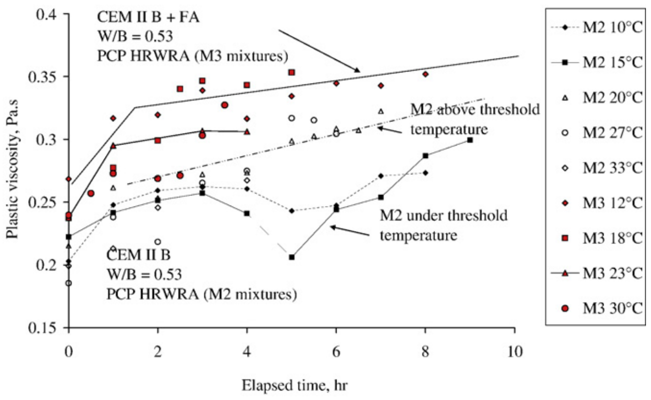

Fig. 8. Variation of plastic viscosity with time for the M2 and M3 made with PCP-HRWRA and mixtures tested at various temperatures: effect of partial replacement of cement with fly ash.

mainly due to the hydration reaction in the case of micro mortars made with PNS-HRWRA.

5. Conclusions

The influence of the coupled effect of temperature and time on the evolution of the rheological properties of micro mortars proportioned with PNS-, PMS-, and PCP-based HRWRAs was investigated. Based on the results presented in this paper, the following conclusions appear to be warranted:

1. The evolution of plastic viscosity of micro mortar made with PNS- or PMS-HRWRA with time and temperature can vary in a linear fashion up to the end of the dormant period (t_f). The rate of increases of μ is dependant on temperature given the effect of temperature on the hydration kinetics of the cement.
2. The variation of viscosity with temperature is more accentuated in the case of mixtures proportioned with W/B of 0.42 compared to 0.53.
3. In the case of the micro mortars prepared with PCP-HRWRA, changes in plastic viscosity with time are shown to vary in different ways depending on mixture temperature. Above a threshold temperature, the micro mortar made with PCP-HRWRA can behave in a similar manner as mixtures made with PNS-HRWRA where μ increases linearly with time or normalized time. Below this threshold temperature, a gap in the continuous increase of plastic viscosity with time is observed between 30% and 70% of the dormant period, with a minimum value reached at t' of 50% to 60%. Sharp increase in plastic viscosity is observed beyond this minimum.
4. No threshold temperature is observed for mixtures made with PCP-HRWRA and fly ash used at 12.5% volumetric replacement of cement. The M3 micro mortars seems to behave in a similar manner as the mixtures made with PNS-HRWRA where μ increases in a linear way with elapsed time, except for the first hour where a sharp increase of plastic viscosity is observed.

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