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Compatibility of Slag-Blended Cement with Polycarboxylate Ether Superplasticisers: Rheological Properties Study

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ABSTRACT

The compatibility of slag blended-cement (SBC) with superplasticisers was investigated using rheological measurements. Accordingly, continuous flow tests and thixotropic behaviors were studied for different ages of hydration. In this work, the slag was used in the range of 0-40% in order to substitute the ordinary Portland cement (OPC). Two polycarboxylates ether superplasticisers (PCEs) were used, the acrylic copolymer (PA) and modified polycarboxylate (PC). The saturation point of each superplasticiser was determined on pastes by rheological tests, it is 2% on amount of cement, and the water/cement report is equal to 0.35. The investigation was carried out using a rotational rheometer AR2000 with coaxial cylinders' geometry. The results showed that the PC-admixture is more efficient than the PA-admixture in OPC; the consistence was consecutively 4.75 Pa.s and 10.45 Pa.s. In addition, the fluidizing effect of the admixtures on cement pastes is conditioned by the presence of slag. The use of PA-admixture in SBC improved rheological properties. However, the use of PC-admixture in SBC increased greatly the viscosity, which involves an incompatibility, the thixotropy increment was from 46898.9 Pa/s (F4) to 59690.1 Pa/s (F9).

1 Introduction

Blast furnace slag is used as supplementary cementing material for the production of slag blended-cement (SBC) and slag cement. The partial replacement is increasingly common practice due to the technological, economic, and environmental benefits [1]. Its use offers cost reduction due to lower prices compared with Portland cement. In other side, the production of slag cement is an environmentally friendly process. Finely, the use of slag in cement improves resistance to aggressive chemicals [2]. Due to the pozzolanic properties, Slag interacts with the $Ca(OH)_2$ to form an additional amount of a low-basic hydrated calcium silicate (CSH) in a hardened cement paste structure [3]. In other hand, the $Ca(OH)_2$ activates the slag hydration [4].

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The use of polycarboxylate ether superplasticiser (PCE) improves the rheological properties of cementitious pastes. Nonetheless, the use of PCEs in cement may pose problems of incompatibility, due in most cases to cement–admixture interaction. The incompatibility can increase greatly the viscosity and thixotropy and make the paste difficult to flow. The thixotropy is an isothermal, time-dependent and reversible process [5]. According to Barnes et al. [6], the thixotropy is a gradual decrease of the viscosity under shear stress followed by a gradual recovery of structure when the stress is removed. The hysteresis loops are often applied as preliminary attempts to "quantify" the thixotropy of the material [7, 8].

The term compatibility refers to the desired effect on performance when a specific combination of cement and chemical admixtures is used [9]. Several authors have reported that cement–admixture compatibility depends primarily on factors attributable to both admixtures and the cements. The factors associated with admixtures that determine their performance and fluidizing effect are their dosage, the manner and timing of inclusion in the mix and their chemical and structural composition [10]. The factors attributable to cements that affect compatibility, in turn, include their fineness [11], chemical and mineralogical composition, particularly their C3A content [12], and the amount and type of components such as calcium sulphate and alkaline sulphate. The presence of slag is another factor that may affect cement–admixture compatibility, since admixtures may interact not only with cement, but also with it. Thus, it is necessary to take account the compatibility of SBC and PCE admixtures to obtain the high flowability.

Alonso et al. [13] investigated the incompatibility of Blended cement and polycarboxylate ether superplasticisers in range of 0.7 to 1.2 %. Palacios et al. [4] concluded that PCEs induce greater flowability in slag-containing pastes than in unblended paste and this effect is enhanced with the rising percentage of slag in the pastes. These findings concur with the results reported by Hamada et al. [14], who found that the dosage of PCE admixtures required to attain a given flowability was much lower in slag-blended cement than in other type of cement.

The aim of the present study is to investigate the compatibility of SBCs/PCEs admixtures through rheological tests. It has also been studied the effect of the slag at 30% on the rheological and thixotropic behaviours of cement paste, at different times of hydration process (10, 40 and 70 min), in order to assess the effects of hydration process on the thixotropy of slag-blended cement.

2 Material and methods

2.1 Materials

The chemical composition of the CEM I 52.5 N cement (in wt%) is as follows: SiO_2 – 21.14, Al_2O_3 – 5.12, Fe_2O_3 – 4.01, CaO – 66.02. The cement mineral composition (Bogue calculation) was: C3S – 64.43 %, C2S – 12.05 %, C3A – 6.78 %, C4AF – 12.19 %, SO_3 – 3.47 %, LOI – 1.38 %. The specific surface is 1370 cm^2/g with particle density being 3.10 g/cm^3 and dry bulk density – 1.24 g/cm^3.

Table 1 – Superplasticisers characteristics

Characteristics	Acrylic copolymer (PA)	Modified polycarboxylate (PC)
commercial name	SIKA VISCOCRETE TEMPO 12	SIKA VISCOCRETE 3045
Appearance	A light brown liquid	brown/green liquid
density	1.19±0.01	1.11 ± 0.01
Ph	8-9	5 ± 1
Na_2O Eq. content	≤ 1 %	≤ 2.5 %
Dry extract	30.2 ± 1.3 %	36.4 ± 1.8 %
Cl-ions content	< 0.1 %	< 0.1 %
Notation	Tempo 12	3045

The granulated blast furnace slag is an amorphous material witch complies with European standard NF EN 15167-1. The slag has graphite-black color and dense structure. The chemical composition of the slag is SiO_2 = 36.7%, Al_2O_3 = 11.3%, Fe_2O_3 = 0.6%, CaO = 42.7%, MgO = 7.0%, MnO = 0.3%, TiO^2= 0.5%, SO_3= 0.3%, Cl^- = 0.01, S^{2-} = 1.0, Na_2O eq. = 0.7.

The specific gravity of the ground slag is 2.90 g/cm^3 and the dispersity determined by the specific surface according to Blaine is 4500 cm^2/g.

In this study, two types of polycarboxylates ether superplasticisers (PCEs) are used. The physical and chemical characteristics are listed in Table 1.

Paste rheology was studied by using a Haake Rheowin AR 2000 rheometer. The geometry used is à concentric cylinders also referred as coaxial cylinders or Couette cell. It consists of a static cylindrical cup and a concentric rotatable cylinder. The range of shear rate varies from 0 to 500 s^{-1} and the shearing time is 5 min. The volume sample needed for measuring is 14 ml. TRIOS software used to model the rheological curves and calculate the rheological parameters: the consistency, the behavior index the thixotropy and the apparent viscosity.

2.2 Methods

The optimum dosage for each superplasticiser is identified using rheological tests; they are constantly dosed as liquid at 2 wt% of cement plus slag. For the all studied samples is cased a constant water-to-cement ratio (W/C) of 0.35 by mass.

2.2.1 Rheological behavior tests

A series of samples are designed and their compositions are listed in Table 2. The measurements were made on paste specimens composed of blended cement containing 10, 20, 30 and 40wt% of slag and ordinary Portland cement (OPC). The pastes compatibility was tested according to the evolution of shear rate as shown in Fig. 3; the rheometer was configured to increase the shear rate by 0.33 Pa/s up to 500 s^{-1}. The cement pastes were prepared by mixing 100 g of cement with 35 ml of water for 4 min.

2.2.2 Thixotropic behavior tests

Hysteresis loop has been the most common method of measuring thixotropy of cementitious materials in last few decades (Fig 1). It was introduced by Green and Weltmann [15].

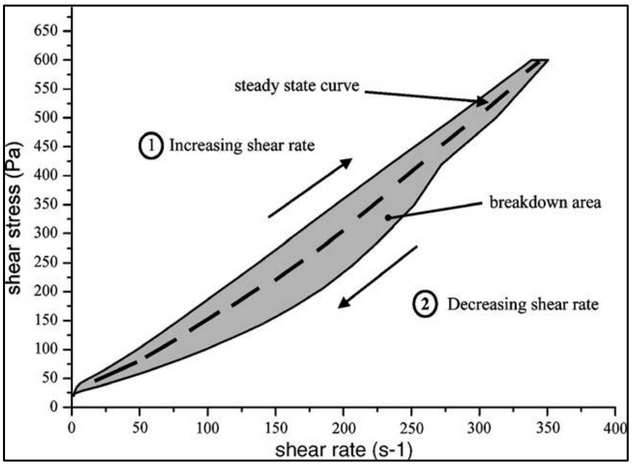


Fig. 1 – Example of thixotropic loop obtained with a cement paste submitted successively to increasing and decreasing shear rate ramps

In this study, the thixotropy was evaluated by hysteresis loop test (Fig. 1): the sample was subjected to increasing shear and then reducing shear, a hysteresis loop was formed by the up-curve and the down-curve. This method requires the use of rheometer and concentric cylinder geometry. The thixotropic test [16] was performed on paste specimen with 30% of slag at 10, 40 and 70 min. The test was conducted by using an AR2000 rheometer to apply a range of shear rates to the paste cement initially at rest. The shear rate in the rheometer was increased from 0 to 500 s^{-1} and then decreased back to zero. The time period over which the shear rate is increased (up-curve) is equal to the time period over which it is decreased (down-curve).

Table 2 - The composition of samples (wt %)

Samples	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
OPC	100	90	80	70	60	100	90	80	70	60
slag	0	10	20	30	40	0	10	20	30	40
PA	2	2	2	2	2	0	0	0	0	0
PC	0	0	0	0	0	2	2	2	2	2

3 Results and discusses

The compatibility study for the two different PCE admixtures and SBCs was based on the effect of the superplasticisers on pastes rheology (rheological behavior and thixotropy). We show the testing result of the rheological behavior of mixtures. Then, the rheological behavior and the thixotropic of the paste, with a focus on 30 wt% of slag, are tested over time (10, 40 and 70 min).

The structure of PCE admixture consists in a linear hydrocarbon backbone with carboxylate and ether group side chains. Their adsorption on cement particles (fig. 2), mediated by their carboxylate groups, disperses cement grains as a result of the repulsion generated by the long ether group chains. The steric repulsion was the prevalent mechanism than the electrostatic contribution to cement particles dispersion was negligible.

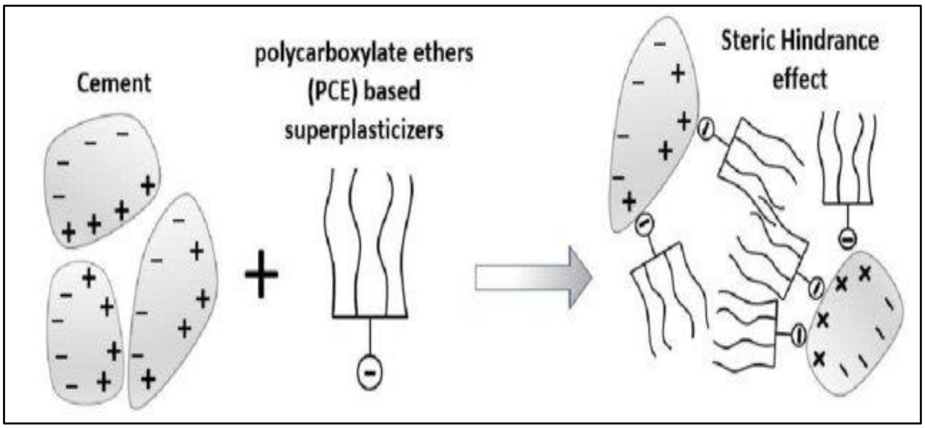


Fig. 2 – Mechanism of polycarboxylate ethers (PCE) superplasticiser, adapted from [17]

3.1 Effect of PCEs on rheological behavior

Rheological analysis of cement pastes was performed with a shear rate of 0 – 500 s^{-1}. The obtained measurement results enabled us to obtain shear-thinning curves of flow.

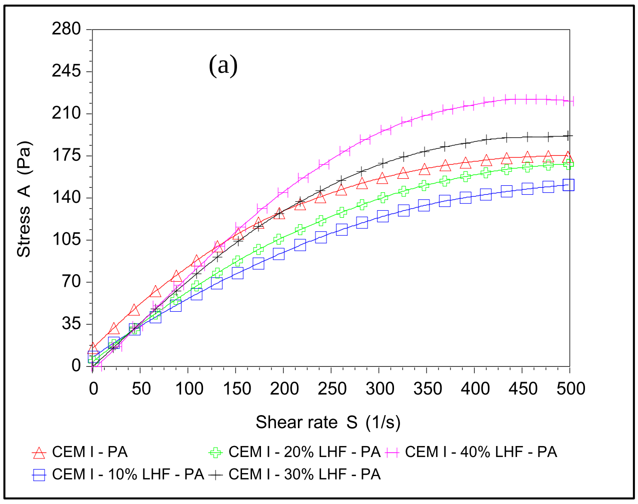
The effect of PA-admixture on the rheological behavior of OPC and SBCs paste was presented in the Fig. 3 (a) and Fig. 3(b). The results shown flow curves of stress over shear rate of PC-admixture effect on OPC and SBCs mixtures. In addition, the table 3 shows the variation in cement paste rate index (n), consistency (K) and correlation coefficient (R^2).

The stress curves were analyzed to determine the rheological parameters of the cement pastes. In all cases, these curves could be fitted by the Ostwald ‑ de Waele model (Eq.1), the correlation coefficient (R^2) is ⩾ 0.90.

$$\tau = K\dot{\gamma}^{n} \tag{1}$$

where K (Pa.s) is the consistency index and ‘ n ’ is the flow behavior index, τ and $\dot{\gamma}$ are the shear stress and applied shear rate respectively.

According to the results obtained in Fig. 3 (a) and Fig. 3 (b), the PC-admixture improved OPC rheological properties more efficiently than PA one. Also, the fluidizing effect induced by such admixtures depended on type of admixture and the percentage of slag in the cement [4]. The tests revealed that as slag using from 0 to 40, rheological behavior of Portland cement paste was improved in presence of PA-admixture : consistency index decreased from 10.45 Pa.s to 4.11 Pa.s and the behavior index increased from 0.46 to 0 .66. It is known that the hydration of particles cement in a blended cement is activated by the slag supplying $Ca(OH)_2$ in the fluid phase, which in turn is an activator for slag component [18]. However, the PC-admixture increased significantly the flow consistency index and decrease the flow behavior index of slag-blended cement when it was used with, the interaction between the slag particles and PC molecular was higher.



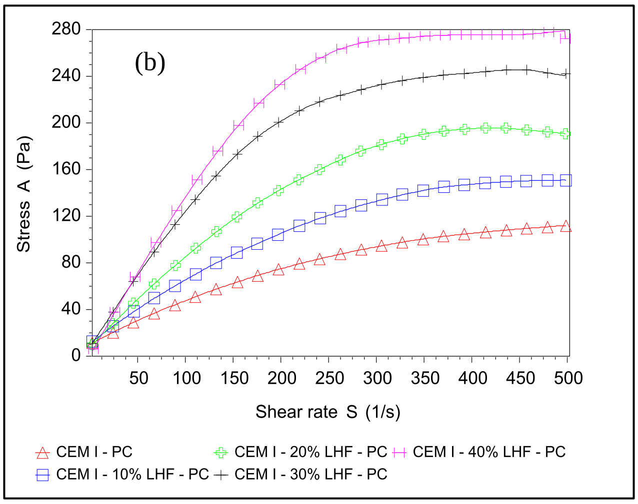


Fig. 3–Stress versus shear rate flow curves cement pastes investigated at various slag concentrations: with PA-admixture (a) and with PC-admixture (b).

Table 3 - The rheological properties data of the samples according to the Ostwald–de Waele model

Samples	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
flow consistency index (K)	10.45	3.89	4.12	4.91	4.11	4.75	6.75	8.75	15.96	16.88
flow behavior index (n)	0.46	0.60	0.61	0.64	0.66	0.51	0.52	0.51	0.45	0.47
Correlation coefficient (R^2)	0.97	0.98	0.98	0.96	0.95	0.98	0.97	0.94	0.93	0.90

3.2 Effect of PCEs on rheological behavior

The effect of PCE superplasticisers on the apparent viscosity of ordinary Portland cement and slag blended cements pastes with a slag content between 10 and 40% has been studied. The change of apparent viscosities versus shear rate, in presence of admixtures, was presented in Fig. 4 (a) and Fig. 4 (b).

According to the Fig. 4 (a) and Fig. 4 (b), the PC-admixture using in OPC presented more flowability than PA-admixture. It was a very significant difference in efficiency between the viscosities of superplasticisers. Also, it has been observed that the using of slag in the presence of PC-admixture gave an increase in apparent viscosity and consequently a decrease in flowability. The slag adsorbs superplasticiser and deteriorate the efficiency of water reducing agent.

The apparent viscosity is reduced in presence of PA-admixture at 10 to 40% in slag amount. These results are consistent with the consistence results presented in the table 3. According to Alonso et al. [1], CEM III/B pastes, with granulated blast furnace slag additions, show high rises in flowability. In addition, Alonso et al. [13] and Puertas et al. [19] reported that PC-admixtures had a greater dispersive effect on slag cement than on slag-free Portland cement pastes.

The fluidity is mainly attributed to a decrease in the amount of C3A available to adsorb and consume admixture to form an organo-mineral phase. However, the admixtures are absorbed onto the silicate phases of the clinker and onto the slag particles, inducing a repulsion and the concomitant reduction in apparent viscosity [4]. The more effective fluidization generated by PA-admixture in slag blended-cement, can be associated with the greater induced steric repulsion. This can be related to their higher molecular weight, which should induce an increase in the adsorbed layer thickness.

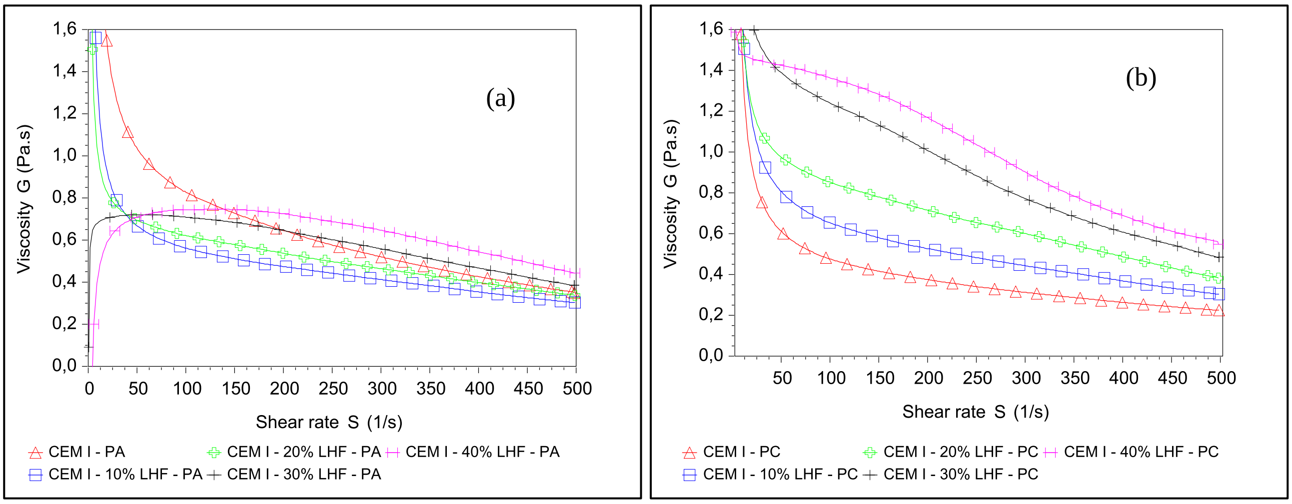


Fig. 4–Apparent viscosity versus shear rate of cement pastes investigated at various slag concentrations: with PA-admixture (a) and with PC-admixture (b).

3.3 Time effect on rheological properties

The thixotropic behavior of OPC and SBC (30 wt% of slag) was evaluated at different times of hydration. The substitution of cement with 30 wt% of slag is optimal both from the perspective of the impact on the technical characteristics and from the economic point of view [3]. Ogirigbo et al. [20] use the same slag account; two slags, of differing compositions, were combined with a CEM I 52.5 R at 30% replacement.

The area of the loop thus formed gave an idea of the extent of thixotropy present in the samples. The rheological behavior of the up-curves was best described by the power law model. The parameters model, consistency index (K) and behavior index (n), were calculated using the TRIOS software. The change of K and n are presented versus time (10, 40 and 70 min) for the sample series with and without slag (F1, F6) (F4, F9) respectively in the Fig. 5 (a) and Fig. 5 (b).

The variation of K value with time (10 – 70 min) of SBC is given in Fig. 5 for power law fitted model. At the beginning of hydration (10 min), the consistency index of OPC paste decrease with the use of slag, in the presence of PA-admixture. The consistency index varied from 13.1 to 8.2 Pa.s. However, in the contrast to the OPC paste, in presence of PC-admixture, the SBC paste present an increase in consistency index witch varied from 7.0 to 12.6 Pa.s.

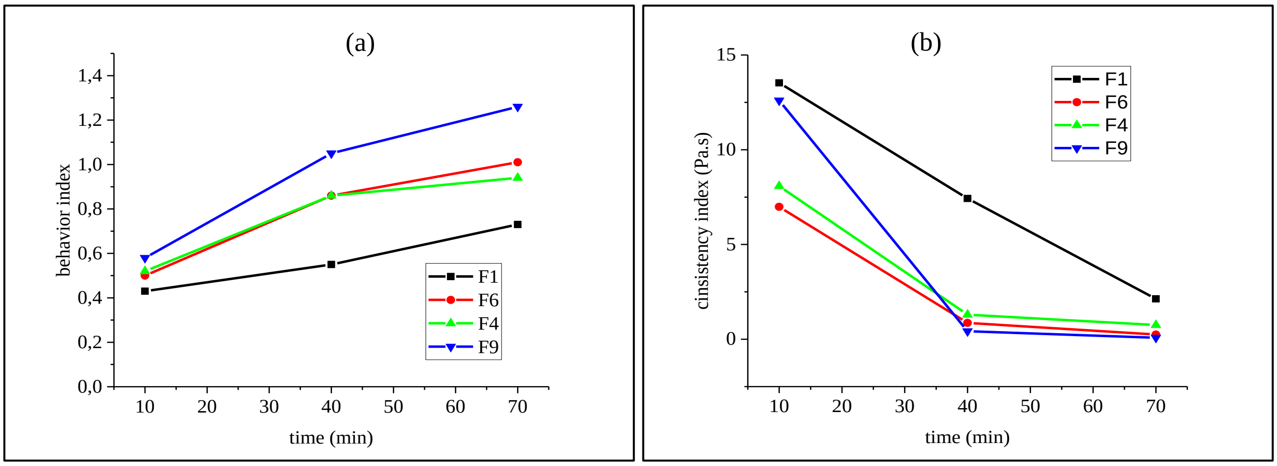


Fig. 5 –Time dependence of rheological properties of cement pastes: flow consistency index (a) and behavior index (b).

3.4 Time effect on thixotropic behavior

The entire area in between the up and down curves, and their evolution with time, is one way to assess thixotropy. The response thixotropy tests was recorded and plotted against the shear rate as shown in Fig. 6.

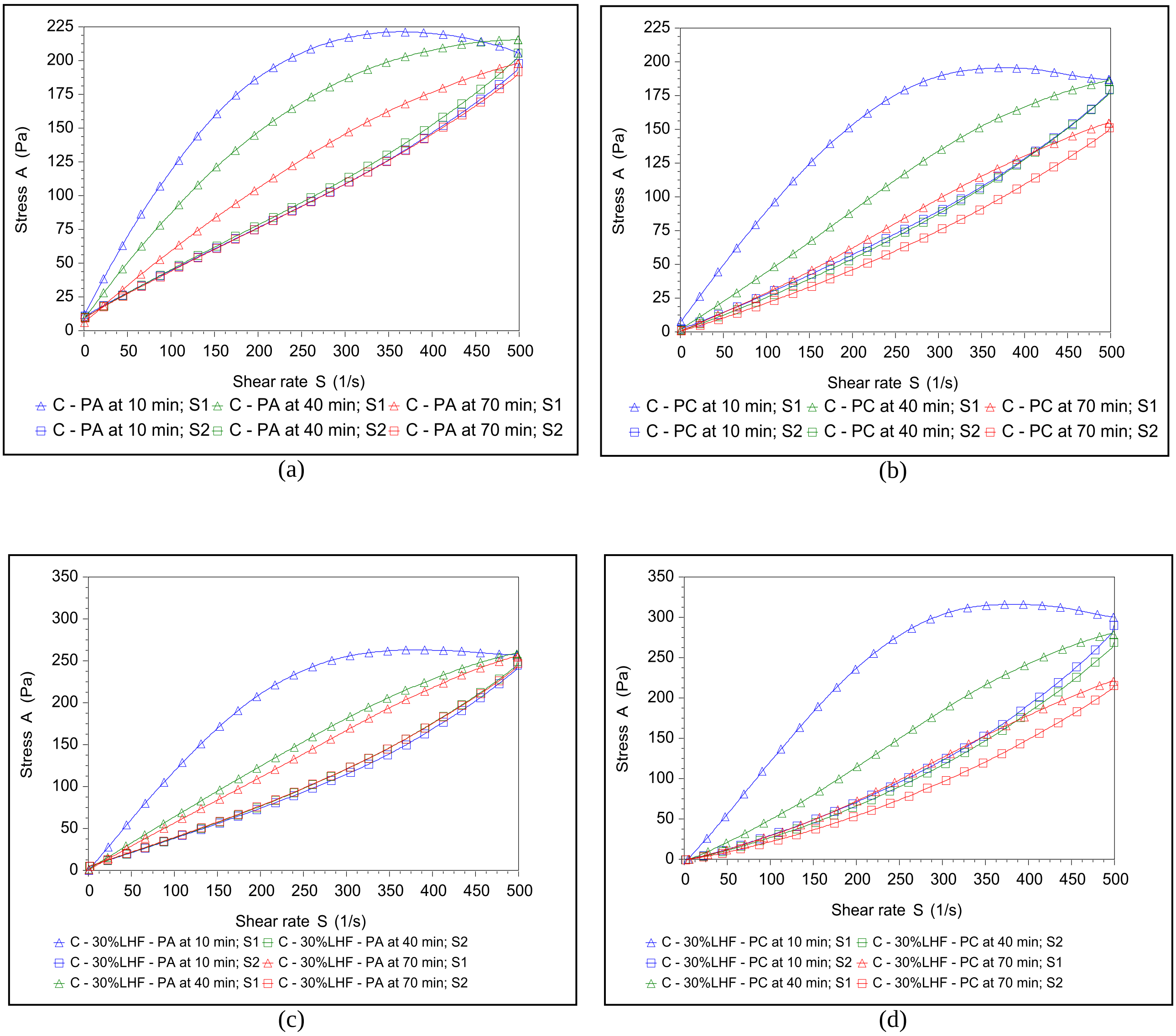


Fig. 6–Evaluation of thixotropic behavior over time by hysteresis loop measurement, the shear rate first increases (S1) and then decreases (S2): (a) OPC in presence of PA-admixture, (b) OPC in presence of PC-admixture, (c) SBC in presence of PA-admixture, (d) SBC in presence of PC-admixture.

The hysteresis area value is an indicator for the degree of system destructuring, higher values for thixotropic area indicating a higher thixotropy. The cement pastes are typically thixotropic materials with a yield stress and varying rheological properties over time [21]. However, the use of admixtures leads to a reduction in flocculation, which reduces the thixotropy.

According to the hysteresis cycles shown in Fig. 6, all the pastes were thixotropic; the stress was higher on increasing the shear rate than on the shear-rate-reduction part of the cycle. This is consistent with ordinary thixotropy; the steric hindrance witch is the inter-particle forces between solid particles result in formation of flocks. Portland cement paste thixotropy, in presence of admixtures, was largely influenced by slag and the hysteresis loop area for all the samples present decrement over times of hydration. The thixotropy results using TRIOS software are given in Table 4.

Table 4 - Thixotropy values of cement pastes with superplasticizers

Test	Formulations	Test times [min]	Thixotropy [Pa/s]	Normalized thixotropy [1/s]
1	F1	10	38257.4	1.15 e-3
2	F1	40	25526.2	7.92 e-4
3	F1	70	11851.9	4.00 e-4
4	F6	10	34020.2	1.16 e-3
5	F6	40	14977.2	5.38 e-4
6	F6	70	7261.2	3.13 e-4
1	F4	10	46898.9	1.19 e-3
2	F4	40	20413.6	5.26 e-4
3	F4	70	15504.6	4.03 e-4
4	F9	10	59690.1	1.26 e-3
5	F9	40	22132.3	5.28 e-4
6	F9	70	9867.0	2.97 e-4

The Fig. 7 showed that, at the beginning of hydration (10 min), the use of PC-admixture in OPC paste was more thixotropic than the use of PA-admixture. Also, the thixotropy of OPC paste decreased with the use of slag, in the presence of PA-admixture; the thixotropy varied from 38257.4 to 34020.2 Pa/s. However, in the contrast to the OPC paste, in presence of PC-admixture, the SBC paste thixotropy increased from 46898.9 to 59690.1 Pa/s.

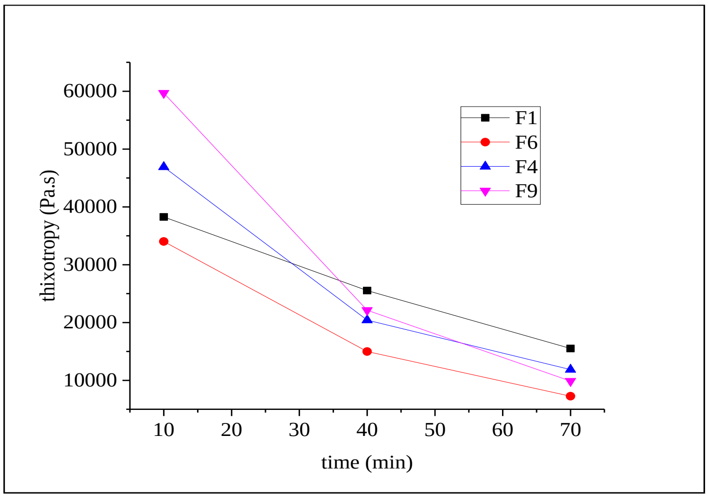


Fig. 7 – Thixotropy values over time of OPC and SBC in presence of superplasticisers.

The F9 mixture presented the highly thixotropic at 10 min Pa.s (table 4). The energy needed to break down the microstructure of the tested material will be important. This result implies that the PC-admixture is highly adsorbed by slag particles in beginning of hydration [6]. The result is consistent with the consistence index result presented in fig. 7. The increment of thixotropy is attributed to the incompatibility between PC-admixture and SBC. Petkova and Samichkov [22] suggested that the increase in thixotropy is attributable to the increased specific particle surface in the paste system. Salem [23] proposed that this effect is due to the pozzolanic reaction as well as the rapid transformation of ettringite to monosuflate.

After 70 min of hydration, the thixotropy of OPC with AP or PC-admixture (11851.9 Pa/s and 15504.6 Pa/s, respectively) were higher than those of SBC (7261.2 Pa/s and 9867.0 Pa/s) indicating stronger thixotropy for OPC in comparison to SBC. The F6 mixture present the slightly thixotropic at 10, 40 and 70 min, decreasing from 59690.1 Pa/s to 9867 Pa/s.

4 Conclusion

In this paper, a study of the compatibility of slag-blended cement/ether polycarboxylates over the time was investigate. The adequate Ostwald–de Waele model was applied for the approximation of curves of flow. It has been conclude that:

The PC-admixture was more efficient in OPC than the PA-admixture. Also, the fluidizing effect of the admixtures on cement pastes was conditioned by the presence of slag.

The tests revealed that SIKA VISCOCRETE TEMPO 12 superplasticiser was compatible with Slag blended cement. As slag using from 10 to 40 in presence of PA-admixture, rheological behaviour of Portland cement paste was improved: consistency decreased from 10.45 Pa to 4.11 Pa.s and the index behaviour increased from 0.46 to 0.64.

The SIKA VISCOCRETE 3045 additions in slag-blended cement affected the rheology of cement paste. It proved that the using of PC-admixture in slag blended-cement increased highly the apparent viscosity and consequently was incompatible.

This study revealed that the PC-admixture increase both the consistence and thixotropy of slag blended-cement (F9) at the beginning of hydration (10 min). The PA-admixture, on the contrary, decreased the consistence and thixotropy.

After 70 min of hydration, the thixotropic areas of OPC with AP or PC-admixture (F1 and F4, respectively) were higher than those of SBCs (F6 and F9), indicating stronger thixotropy for OPC in comparison to SBC.

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