

# Assessing Rheological Properties of Cement Paste as a First Step in Predicting Robustness of Self-compacting Concrete

MARIUS DUMITRESCU<sup>1</sup>, ALINA BADANOIU<sup>1\*</sup>, CONSTANTIN DORINEL VOINITCH<sup>2</sup>, GEORGETA VOICU<sup>1</sup>

<sup>1</sup> University Politehnica Bucharest, 1-7 Polizu Str., 011061, Bucharest, Romania

<sup>2</sup> Technical University of Civil Engineering, 124 Lacul Tei Blvd., 020396, Bucharest, Romania

*This paper proposes a methodology to assess the rheological behavior of cement paste as a first step to linking this behavior to the robustness of the Self Compacting Mortar (SCM) and further extending this to Self-Compacting Concrete (SCC). Cement paste's rheological behavior was assessed in terms of spread (using a mini-cone) and time of flow (using the Marsh cone). The results show that the type of superplasticizer (SP) admixture has a great influence on the rheological behavior of cement paste and for each combination of binder and superplasticizer admixture there is a specific range of water to binder ratio in which the rheology of paste seems to be appropriate for obtaining a robust SCC mix. The influence of SP and limestone filler addition on the kinetic of cement hydration process was assessed by X Ray Diffraction and thermal analysis (TG-DTA). Based on these results, i.e. an important delaying effect exerted by superplasticizer additions on cement hydration process at early ages (1 day), it can be concluded that when designing SCC for the precast industry - where the early strength of concrete is of high importance, analyzing the early strength of binder paste together with the robustness properties is very important. The correlation between the two aspects - rheology and early strength - is very important in this case in order to obtain applicable results in practice.*

*Keywords: Robustness, Self-compacting concrete, Rheology, Cement paste*

Self-compacting concrete (SCC) is an innovative type of concrete which was initially developed in Japan [1]. Its main characteristics, as opposed to the conventional concrete, are to completely fill the forms, pass through narrow gaps and consolidate without the need of vibration. The growing interest on SCC technology which took place in the past years in the construction industry is determined mainly by its ability to be rapidly cast in heavily reinforced structures. Moreover, in SCC production can be valorized high quantities of mineral residues (wastes) such as fly ash and blast furnace slag.

The properties of SCC depend on the mix design, properties of constituent materials and mixing procedure. For this reason, the constituents of SCC should be carefully selected and strict limits should be adopted regarding their properties and dosage [2-6]. The mixing process of an SCC can be very complex and numerous parameters should be monitored in order to obtain a concrete mix with a self compacting ability [3-5].

Because of its characteristics in fresh state, and its mix design, the SCC is susceptible to high variations of the properties in fresh state due to changes in the constituent materials. This is why one important step of the mix-design process of SCC in *The European Guideline for Self-Compacting Concrete* [7] is checking the robustness of the mix. Robustness can be defined as the ability of concrete mix to tolerate modifications of component's dosage without affecting its main properties in fresh state (workability) and hardened state (mechanical strengths).

Not having a robust mix to the variations of its components (dosage or properties) or other external parameters (i.e. temperature), a SCC mix design which shows good results in laboratory may cause many problems in practice. Many studies were conducted on this topic, aiming either to determine deciding factors which influence the robustness of a SCC mix design either to develop methods to assess the robustness of a mix or methods to increase the robustness of the mix [8-12].

Only few studies have been conducted so far on the robustness of cement paste or self-compacting mortar (SCM) and its relation with the robustness self-compacting concrete (SCC) associated to it. Libre *et al.* [13] proposed a method to evaluate the stability of SCM starting from water to different binder pastes, with various admixtures and water to cement ratios and then extended the results to the SCC's stability. Asghari *et al.* [14] studied the influence of mixing procedure and addition of viscosity-modifying agents (VMA) on the robustness of cement-pastes and concluded that the modification of water dosage in order to assess the robustness can represent an effective method.

In this respect, this paper aims to determine the rheological behavior of binder paste, connecting its robustness to water to binder ratio (w/b), as a first step in the designing process of a SCC. In the cement paste, the most important factors which influence its rheology and robustness in fresh state are: w/b ratio, the cement particle size distribution, interaction between the cement and superplasticizer admixture as well as the temperature [9,14].

Knowing if a cement-superplasticizer combination can lead to a robust SCC mix only by testing the cement paste would be beneficial and would save a lot of work in the mix design process. The advantage of working with cement paste instead of concrete mixtures, is that testing is much faster and reduces the work required to establish the best binder-admixture combination. It also permits a more rigorous control of concrete mix design.

This paper presents also the influence of the selected superplasticizer addition on the kinetic of cement hydration and hardening processes. This is an important aspect when the designed SCC mixture should have early strengths in correlation with its area of use.

## Experimental part

### *Materials and methods*

The cement used in this study was Type I ordinary Portland cement (CEM I 42.5 R) and the limestone filler

\* email: [alina.badanoiu@upb.ro](mailto:alina.badanoiu@upb.ro)

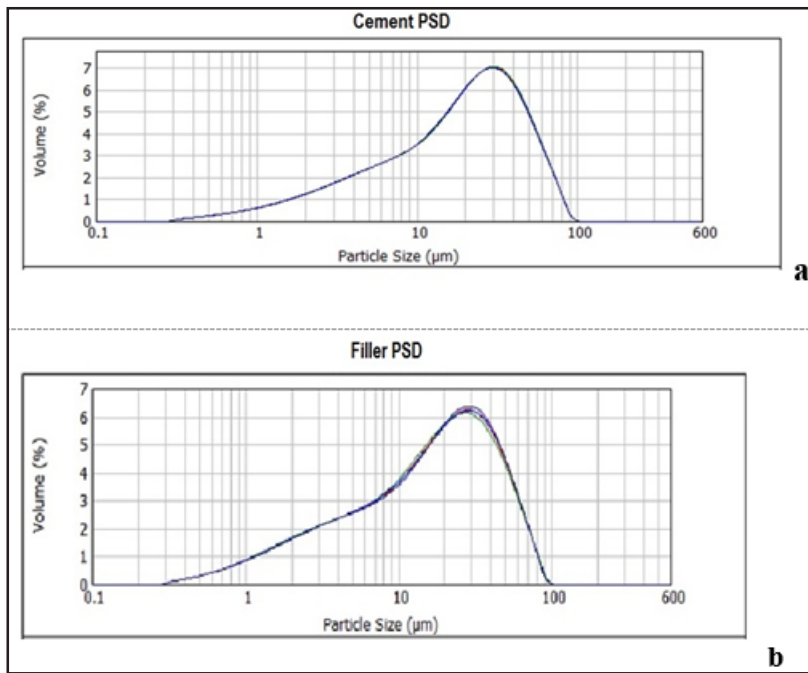


Fig.1. Particle size distribution (PSD) of portland cement (a) and limestone filler (b)

**Table 1**  
MAIN PROPERTIES OF THE SUPERPLASTICIZERS

Type of admixture	Appearance	Specific gravity at 20°C (g/cm <sup>3</sup> )	pH-value:	Alkali content (%):	Chloride content (%):
SP1	Brown liquid	1.05 ± 0.02	7.0 ± 1	≤ 1.00 by mass	≤ 0.10 by mass
SP2	Brown liquid	1.04 ± 0.02	6.5 ± 1	≤ 1.00 by mass	≤ 0.10 by mass

with a CaCO<sub>3</sub> content over 90% comes from the same cement producer.

The particle size distributions of cement and limestone filler, assessed by laser granulometry, are shown in figure 1. It can be noticed that both materials have similar particles sizes distributions which is beneficial when limestone filler is used to partially substitute the cement.

Two types of polycarboxylic superplasticizers admixtures were used in this study i.e. one which is commercially available for the ready mix concrete market (SP1) and the other one for the precast concrete market (SP2) with higher water reduction capacity. The main characteristics of these superplasticizers (according to the producer data sheets) are presented in table 1.

#### Assessment of rheological properties of the cement paste

The rheological properties of cement pastes (with/without superplasticizers or/and limestone filler additions) were assessed in three steps:

- I.determination of the zero-flow w/b ratio;
- II.determination of the optimum superplasticizer ratio (dosage);
- III.determination of the cement paste robustness vs. w/b ratio

I. Determination of the *zero-flow volumetric w/b ratio* is described in [15] and consists in testing several cement pastes, prepared with various amount of water in order to

determine the water to binder ratio which corresponds to the zero relative slump flow.

The experimental procedure is:

- water amount, corresponding to various values of w/b ratios, is mixed with cement using a mixing machine as that described in the SR EN 196-1; a detailed description of mixing procedure is presented in figure 2;
- the obtained paste is then poured in the mini-cone and is left to settle for 10 s;
- the mini-cone is lifted slowly (in approximately 2-3s) and the slump-flow diameters are measured and recorded;
- the relative slump-flow ( $\Gamma_{p/m}$ ) is determined according to the following formula:

$$\Gamma_{p/m} = (d/d_0)^2 - 1 \quad (1)$$

where:

$d_0$  = mini-cone diameter (100 mm)

$d = 0.5 \cdot (d_1 + d_2)$ ;

$d_1$  and  $d_2$  = two diameters of slump-flow measured at a 90 degrees angle.

The water to binder ratio vs. relative slump-flow values is graphically represented in order to determine by linear regression the  $\beta_p$  value;  $\beta_p$  is the value of w/b ratio for which the relative slump-flow ( $\Gamma_{p/m}$ ) is zero.

Knowing the  $\beta_p$  value, the zero-flow volumetric w/b ratio is chosen in the range [0.8...0.9] of  $\beta_p$  value. This method is used in current practice to measure the water demand of a binder and it is specific to each type of binder.

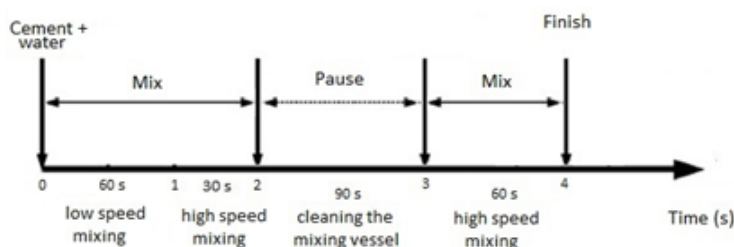
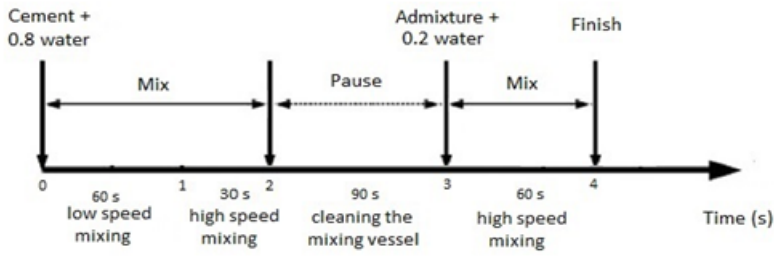
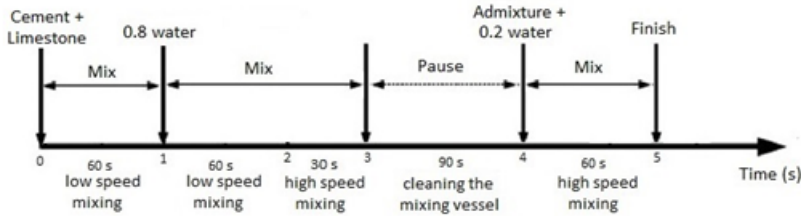


Fig. 2. Mixing procedure of cement pastes



a

Fig. 3. Mixing procedure for cement pastes with superplasticizers additions: a) without limestone filler; b) with limestone filler



b

Table 2  
COMPOSITION OF CEMENT PASTES

Component	Cement (CEM) (%)	Limestone filler (LF) (%)	Superplasticizer admixture SP1 or SP2* (%)	Water to binder ratio /b)
E	100	-	-	0.30
CEM+SP1	100	-	1.2	0.30
CEM+SP2	100	-	0.8	0.30
CEM+30%LF+SP2	70	30	0.7	0.30

\*) Superplasticizers dosages were calculated with reference to binder content

II. The assessment of the *optimum superplasticizer ratio (dosage)* was performed on cement pastes prepared with water dosage corresponding to the zero-flow w/b ratio (step I) with various amounts of superplasticizer additions.

This method, proposed by [2], considers that the optimum w/b ratio for each binder-superplasticizer combination corresponds to the value beyond which an increase in the superplasticizer dosage does not determine a notable increase in the slump-flow value.

The mixing procedure for the cement paste preparation is described in figure 3a. When limestone filler is added in the cement paste, the mixing procedure has additional extra 30s of dry-mixing at the beginning of the mixing procedure (fig. 3b).

III. Determination of the *pastes' robustness vs. w/b ratio* – for each cement-superplasticizer combination, considering the superplasticizer dosage corresponding to optimum superplasticizer ratio; the w/b ratio of the paste is then chosen between reasonable values according to the water-reduction capacity of the superplasticizer - for example, for SP1 the w/b interval is between 0.32 and 0.48 and for SP2 the w/b interval is between 0.24 and 0.40. The rheological properties of the cement paste were measured by means of flow-time measured on the Marsh Cone, as described in EN 445.

The flow-time measured on the Marsh cone consists in pouring 1L of cement paste and measuring the time it takes for 0.5 L of paste to flow through the cone.

#### Assessment of the influence of limestone filler and superplasticizer additions on the kinetic of cement hydration and hardening processes

In order to assess the influence of superplasticizer (and limestone filler) additions on the kinetic of cement paste hydration and hardening process, two methods were used: X ray diffraction analysis and thermal analysis (thermogravimetry and differential thermal analysis).

These analyses were performed on cement pastes (table 2) hardened for 2, 7 and 28 days.

Cement pastes were prepared following this procedure: i) cement and limestone filler were mixed manually in a plastic recipient - 30 s dry homogenization; ii) addition of 50% of the total volume of water - 30 s mixing; iii) addition of the rest of the water - 60 s mixing ; iv) curing of the pastes in sealed plastic recipients for 1, 28, 90 days; v) stopping of the cement hydration by grinding and ethanol washing, followed by drying at 60°C .

The XRD patterns were obtained on a Shimadzu XRD 6000 diffractometer, with monochromatic CuK $\alpha$  radiation ( $\lambda = 1.5406\text{\AA}$ ); scanning was performed in the range  $2\theta = 5 - 60$  degrees.

Thermal analysis (TG and DTA) were performed with a Shimadzu DTA-TG-50H instrument. The heating was performed with a rate of 10°C/min, in the temperature range: 20-1000°C.

## Results and discussions

### Rheological properties of cement pastes

The zero-flow w/b ratios of the cement pastes (with/without limestone filler addition) were determined on the graph presented in figure 4.

As it can be seen from figure 4 the  $\beta_p$  value is 1.05 for cement paste (CEM) and 0.97 for cement paste with 30% limestone filler (CEM+30%LF); these data confirm the positive effect of limestone filler addition on the workability of fresh cement paste [16-18].

Based on the determined  $\beta_p$  value for CEM paste i.e. 1.05 one can choose the value of zero - flow volumetric w/b ratio - 0.945 (0.9 of  $\beta_p$  value). Using the same rationing the of zero-flow volumetric w/b ratio for CEM+30% LF is 0.945 (0.97 of  $\beta_p$  value i.e. 0.974). These values can be converted in zero-flow mass w/b ratio by division to the density of cement and the suitable water to binder ratios, for the determination of optimum superplasticizer dosage, are 0.30 for CEM paste and 0.24 for CEM+30%LF.

In order to obtain a cement paste (CEM) with a viscosity suitable for SCC, the water to cement ratio was fixed to 0.32 and the superplasticizers (SP1 and SP2) dosage varied



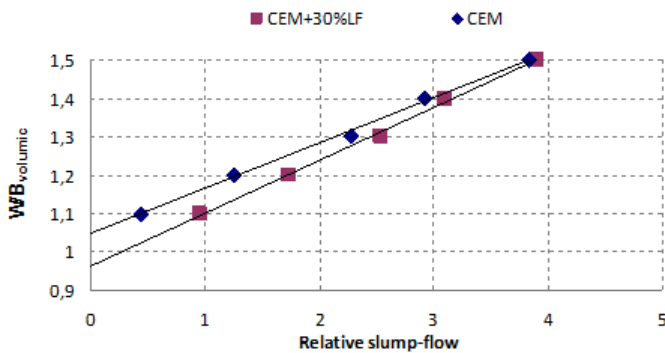


Fig. 4. Determination of zero flow w/b ratio ( $\beta_p$ )

between 0.5% up to 2%. The slump-flow diameter of pastes vs. superplasticizers dosages is presented in figure 5.

Based on results presented in figure 5, the optimum superplasticizer dosage seems to be 0.8% for SP2 and 1.6% for SP1. The difference between the two admixtures is

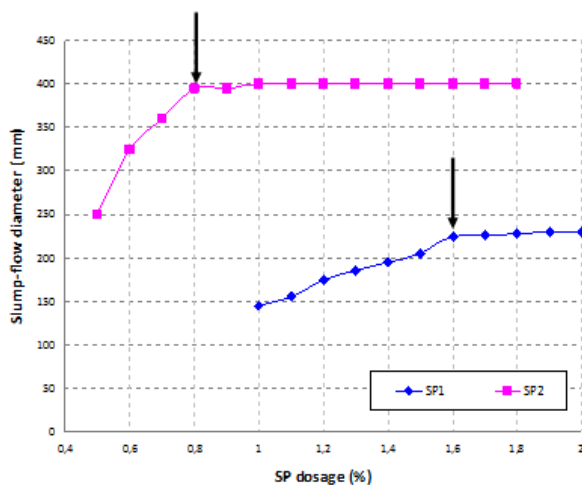


Fig. 5. Slump-flow diameter for cement pastes (CEM) vs. superplasticizer (SP) dosage (water to binder ratio=0.32)

mostly due to the design of the polymer used which has an important impact on the rheology of cement paste. As the provider of this chemical admixtures states, the SP1 admixture is designed for the ready-mix concrete industry (where workability retention is of high importance) and the SP2 is designed for the precast industry (where the most important factor is the water reducing capability and development of high early strengths).

Considering the higher efficiency (water reduction) of SP2, the optimum superplasticizer ratio (dosage) for

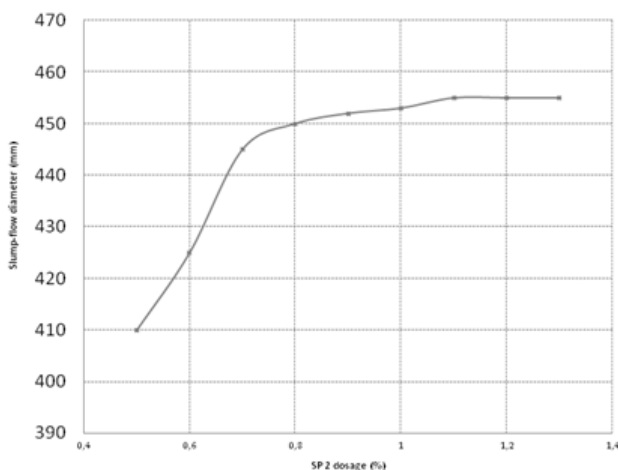


Fig. 6. Slump-flow diameter for cement pastes (CEM+30%LF) vs. superplasticizer (SP2) dosage (water to binder ratio=0.24)

cement paste with limestone filler (CEM+30%LF) was assessed only for SP2 (fig. 6).

For CEM+30% LF combination, the optimum dosage of superplasticizer is 0.7%, considering that the 5 mm increase of slump-flow diameter for a 0.8% SP2 dosage is not economically justified. It can be also noticed that the addition of limestone filler may contribute also to the superplasticizer dosage optimization.

It can be observed, from figures 5 and 6, that for each of the binder-superplasticizer combination there is a superplasticizer dosage beyond which the slump-flow diameter of the paste do not increase anymore; this behavior was also reported by [2]. This means that so far the rheology of paste is concerned, at that particular w/b ratio corresponding to the zero-flow paste, adding more than the optimum SP dosage is a waste of superplasticizer, at least from a rheological point of view.

This method used for the establishment of the optimum SP dosage in cement paste is very simple, requires a small amount of work in the laboratory and provides reliable information. In future studies will be assessed also how this value of optimum SP dosage is translated into mortar and concrete mixtures.

*Robustness of pastes with various binder-superplasticizers combinations* was assessed by slump flow diameter and Marsh cone flow time determination on pastes with various w/b ratios (figs. 7 and 8).

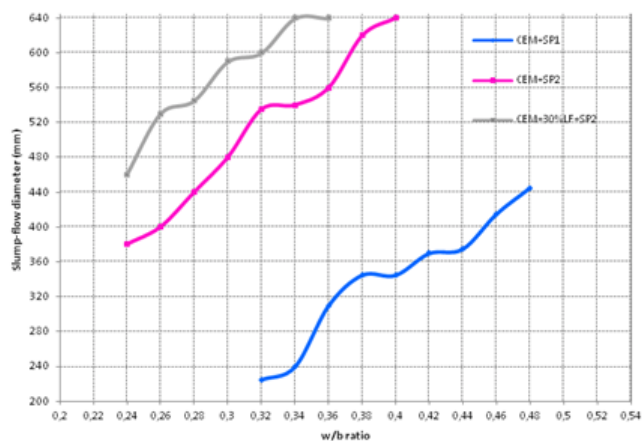


Fig. 7. Slump-flow diameters of cement pastes with SP1 and SP2 and cement paste with limestone filler and SP2 additions vs. water to binder ratio

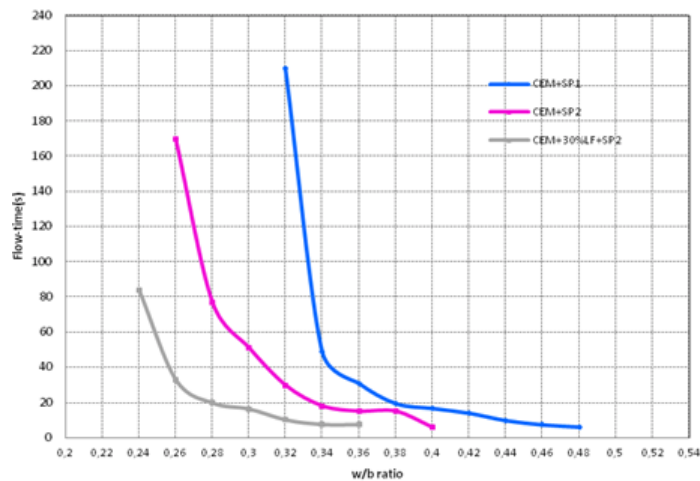


Fig. 8. Marsh cone flow time vs. of cement pastes with SP1 and SP2 and cement paste with limestone filler and SP2 additions vs. water to binder ratio

For each combination of binder and superplasticizer, for some particular intervals of w/b ratio the slump-flow diameter does not increase anymore (robust behavior); for these intervals it might be also expected that corresponding SCM or SCC to have a robust rheological behaviour. The results presented in figure 7 and 8 show that the robust behavior for the combination CEM+SP1 corresponds to w/b=0.38-0.42 domain and in the case of CEM + SP2 the paste is robust for w/b= 0.32-0.36 domain.

The substitution of cement with 30% limestone filler led also to a decrease in the water demand of the binder and to a decrease in the superplasticizer dosage when reported to the binder content. Using limestone filler led to better rheological properties (figs. 7 and 8), as for the same w/b ratio the cement paste with limestone filler (CEM+30%LF+SP2) had a considerably bigger slump-flow diameter as compared with the one without LF (CEM+SP2).

The results obtained for cement (binder) pastes represent a good indicator for the rheology of corresponding concrete mixtures. Yet, it must be taken into account that the w/b ratio in the paste alone translate into a higher w/b ratio in mortar or concrete; this is mainly due to the partial absorption of mixing water by the aggregates. Thus, if we aim to correlate the results obtained for cement paste with those of corresponding mortar and concrete, this aspect must be taken into consideration. For example, a 0.01 variation in the w/b ratio of the cement paste is the equivalent of a 6L variation in the water dosage of a self-compacting concrete with 600 kg binder/m<sup>3</sup>. This kind of water content variation in the usual production process of concretes happens quite often due to the variation of aggregate moisture.

### *Influence of limestone filler and superplasticizer additions on the cement hydration and hardening processes*

In order to assess the influence of superplasticizers and limestone filler addition on the kinetic and hydrates formed in cement hydration process, the cement pastes hardened for 1, 28 and 90 days, were analyzed by XRD and complex thermal analysis (TG-DTA).

The X-Ray Diffraction (XRD) patterns for all combination of binder - superplasticizer (SP1 or SP2) with/without limestone filler are presented in figure 9.

As it can be seen from figure 9, the main crystalline phases assessed by this method are:

- anhydrous compounds from portland cement i.e. calcium silicates -  $3\text{CaO}\cdot\text{SiO}_2$  ( $\text{C}_3\text{S}$ ) and  $2\text{CaO}\cdot\text{SiO}_2$  ( $\text{C}_2\text{S}$ ), as well as calcium carbonate -  $\text{CaCO}_3$  for limestone filler (fig. 9d);

- hydrates formed during portland cement hydration i.e. portlandite -  $\text{Ca}(\text{OH})_2$  and ettringite ( $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 31\text{H}_2\text{O}$ ).

The presence of superplasticizer additions, especially SP2, determines a delay of cement hydration process, easier to notice at early ages; the lower intensity of portlandite peaks in the cement pastes with SP2 at early ages (1 day) as compared with reference (fig. 9c and d as compared with 9a), is a clear indication of this phenomenon.

The stronger retarding effect of SP2, as compared with SP1, can be correlated with its stronger water reducing effect i.e. SP2 is absorbed in a higher quantity at the surface of anhydrous cement grains forming a protective layer which inhibits cement hydration process; this effect is more important at early hydration ages (1 day).

Thermogravimetry (TG) and differential thermal analysis (DTA) of cement pastes, hardened for different periods of time, provided supplementary quantitative information

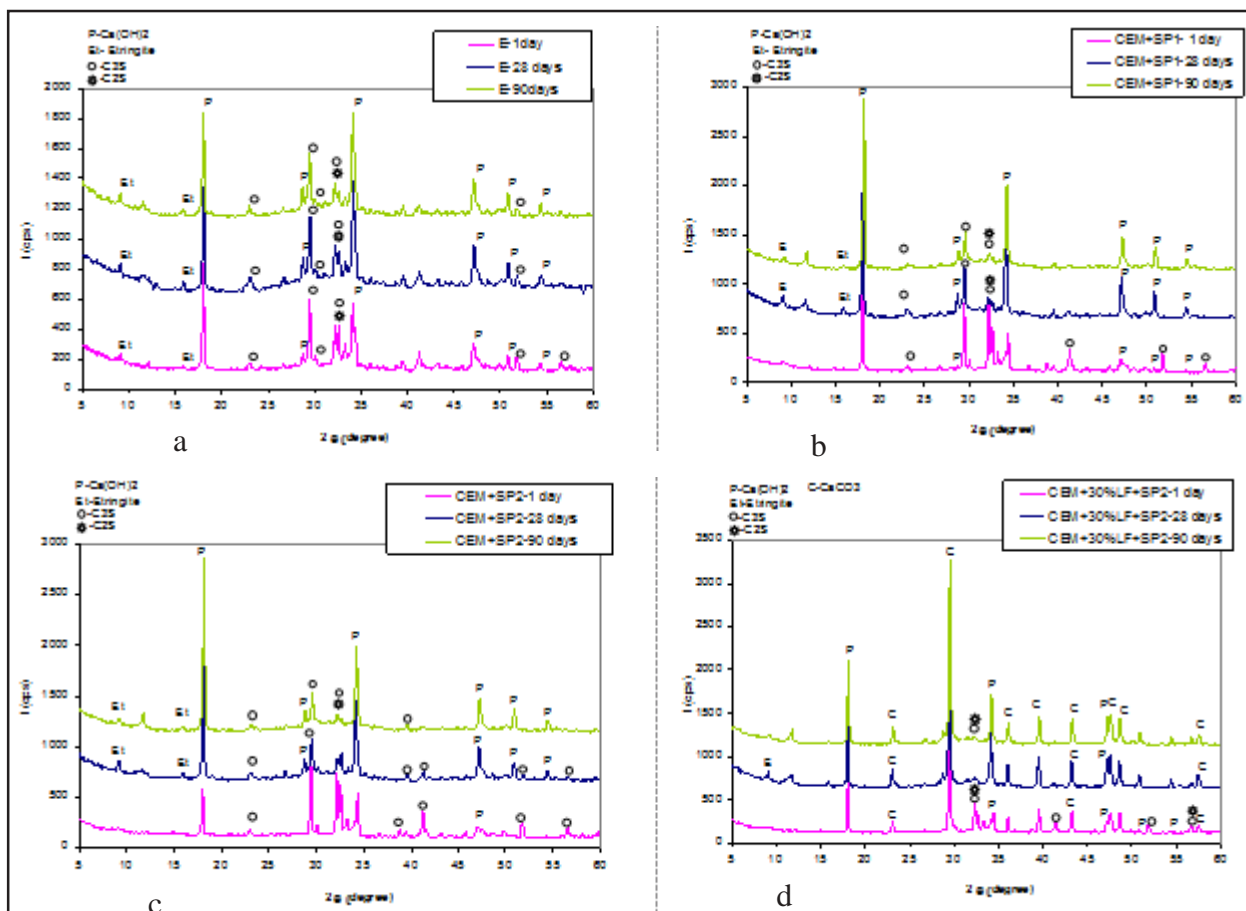


Fig.9. XRD patterns of pastes hardened for 1, 28 and 90 days

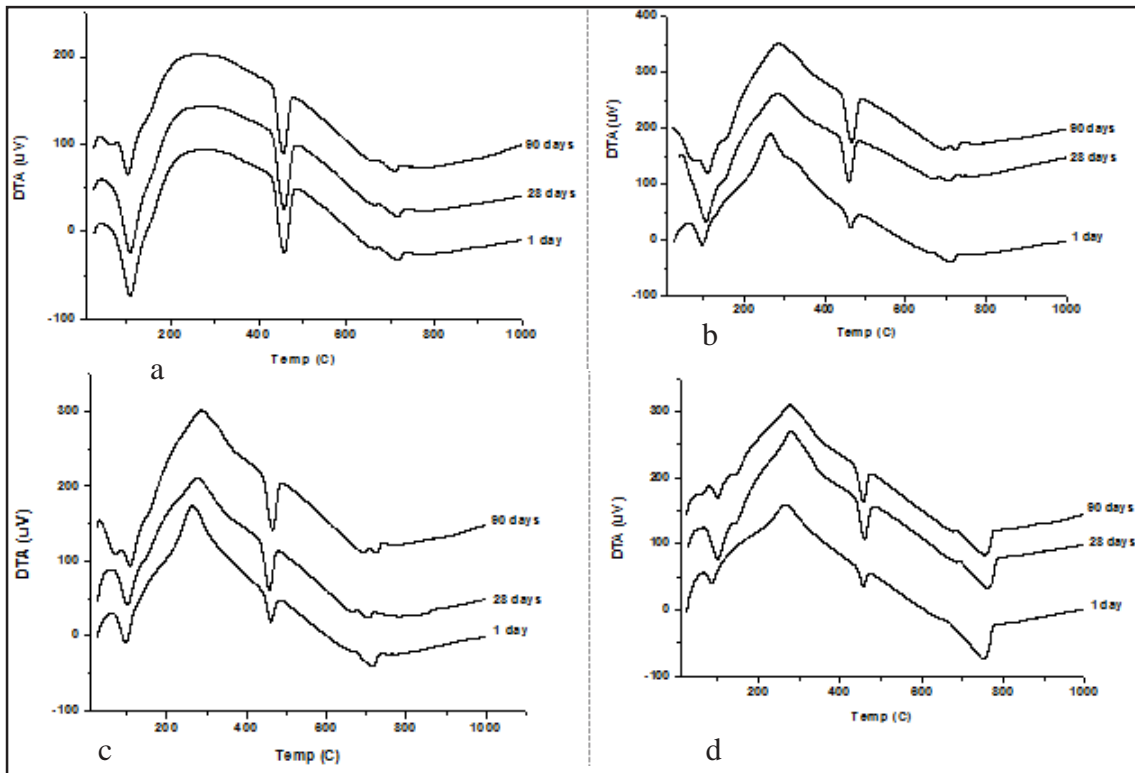


Fig. 10. DTA curves of pastes hardened for 1, 28 and 90 days: a) E; b) CEM+SP1; c) CEM+SP2; d) CEM+30%LF+SP2.

regarding crystalline hydrates ( $\text{Ca}(\text{OH})_2$ , ettringite and monosulphate phase) as well as calcium silicates hydrates with a lower structuration degree [19,20].

The DTA curves recorded on cement pastes, hardened for 1, 28 and 90 days, are presented in figure 10.

The endo-effects present on DTA curves of cement pastes hydrated 1, 28 and 90 days, can be assigned to the following processes [19, 20]:

- the large endothermic effect recorded from 20°C to 300°C, is due to the superposition of three endo-effects i.e. the first one with maximum at 60-70°C is determined by loss of moisture, the second effect from 86°C to 110°C is assigned to loss of water bound in calcium silicates hydrates with a low structuration degree) and the endo-effect from 133-157°C can be attributed to water loss from calcium sulfate aluminates hydrates (AFt and AFm);
- the endothermic effect from 455 to 467°C is attributed to the loss of water bound in calcium hydroxide;
- the endo-effects from 652-683°C and 710-760°C can be assigned to calcium carbonate decomposition (used as addition in the composition CEM+30%LF+SP2 and also formed by the  $\text{Ca}(\text{OH})_2$  carbonation process during preparation and curing of pastes).

The values of total weight loss (TWL) recorded on TG curves corresponding to 20-1000°C temperature range and the amount of portlandite (calculated considering the weight loss recorded between 440-490°C) are presented in figure 11.

Analyzing these results, it can clearly be concluded that at early ages (1 day) the amount of calcium hydroxide resulted in the cement hydration process is smaller in the compositions with both types of superplasticizer additions, as compared with reference (E); the higher retarding effect of SP2 is confirmed by the lower values of portlandite amount as compared with reference (E) and CEM+SP1 paste.

The lower value of portlandite content recorded in CEM+30%LF+SP2 paste, as compared with CEM+SP2, can be explained by reduction of cement amount due to the partial substitution of cement with limestone filler. On the other hand, the important increase of TWL in the cement paste with limestone filler addition is due to the important weight loss determined by the  $\text{CaCO}_3$  decarbonation.

At later ages (28, 90 days) the cement hydration process seems to have the same progress when SP1 and SP2 admixture are used, suggesting that the important delaying effect exerted by SP2 at early ages is no longer present.

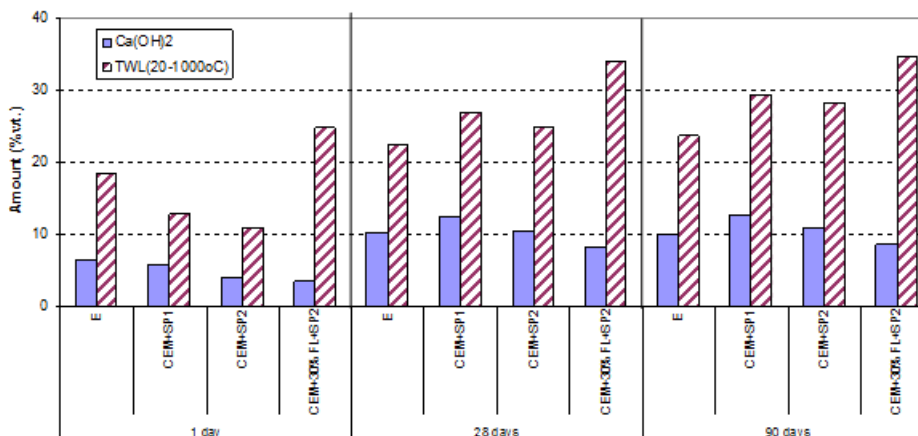


Fig. 11. The amount of portlandite and total weight loss (TWL) recorded between 20-1000°C on binder pastes hardened 1, 28 and 90 days

In current practice the retarding effect of these superplasticizer admixtures is not easily visible because often concrete mixtures are tested at similar workability values. The water reducing effect of the SP admixture allows concrete to develop high compressive strength. This strength gain overrides the retarding effect noticed for the superplasticizer additions at short terms (1 day).

## Conclusions

The method used in this study, to set the optimum dosage of superplasticizer additions and to assess the interaction between binder and superplasticizer addition from a rheological point of view, is simple and effective. The water reducing ability of the superplasticizer (SP) addition and the interaction between binder and SP are quickly assessed by this method, with minimal amount of work in the laboratory.

Each studied binder-superplasticizer combination had specific water to binder ratio domains for which the paste had a robust behavior to water dosage variation.

Working with cement paste is a good way to isolate the interaction between binder and admixtures (SP or limestone filler) and eliminate the effect of the aggregate over the rheology of SCC. Working in cement paste as a first step in designing SCC mixtures can help understanding the impact of raw materials (constituents) over the rheological properties of SCM and SCC. Further studies will be carried in order to link the results obtained in this study with the rheology of SCM and SCC.

Based on the XRD and TG-DTA results, i.e. an important delaying effect exerted by superplasticizer additions on cement hydration process at early ages (1 day), it can be concluded that when designing SCC mixtures for the precast industry (where the early strength of concrete is of high importance) analyzing the early strength of binder paste together with the robustness properties is very important step. The correlation between the two aspects - rheology and early strength - is very important in this case in order to obtain applicable results in practice.

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