

# INFLUENCE OF DELAYING ADDITION TIME OF SUPERPLASTICIZERS ON CHEMICAL PROCESS AND PROPERTIES OF CEMENT PASTES

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Submitted November 1, 2007; accepted December 15, 2007

**Keywords:** Superplasticizers, Delayed addition time, Minislump and electrical conductivity

*This paper presents the influence of delaying addition time of water-soluble polymer based on poly condensate products namely; sulphonate melamine (SMFF) and melamine-urea formaldehyde-free (SMUFF) on physico-chemical properties of cement pastes. The superplasticizers were added by two different methods, a) either together with mixing water (simultaneous addition time) or b) later addition, after specified time (delayed addition time). The minislump was determined at different time intervals namely 30, 60, 90, and 120 minutes. The electrical conductivity of cement pastes admixed with and without superplasticizers was also studied. The results show that the initial electrical conductivity decreases with the delayed time of admixture addition. Delaying addition time from 10 to 15 minutes resulted in an increase in the height of electrical conductivity maximum. Delaying addition time enhances the fluidity of cement pastes than simultaneous addition time. From the results it has been shown that the enthalpy values corresponding to the decomposition of the calcium silicate, calcium aluminate and sulphoaluminate hydrated phases as well as  $\text{Ca}(\text{OH})_2$  decrease in superplasticized specimens when compared to neat cement paste. This is attributed to the retardation effect of the superplasticizer. The maximum retardation was shown at 20 minutes delaying addition time.*

## INTRODUCTION

Some organic admixtures are polymers with hydrophobic groups and ionizable and/or polar functional groups. The nature of hydrophilic and hydrophobic groups should insure minimum surfactancy to avoid foaming and air entrainment by the admixtures. The commercial superplasticizers are sulphonate lignin, sulphonate naphthalene formaldehyde (SNF), sulphonate melamine formaldehyde (SMF), and polycarboxylates [1,2].

Superplasticizers are widely used in the production of concrete with excellent workability giving high performance, high strength and improve the durability. Superplasticizers improve the dispersion of dispersing cement particles in paste [3]. Sodium salt of formaldehyde condenses disperse cement particles by electrostatic repulsion through the adsorption on cement particle surfaces. The resulting concrete can be used with little or no compacting, bleeding and segregation.

There are too many factors affecting the performance of superplasticizers namely, type, dosage of the superplasticizers, type of cement, temperature and mixing procedure, as well as the addition times of the superplasticizers, which cause different fluidizing effects for the resulting cementitious materials [4].

The influence of the addition time on the rheological properties of ordinary Portland and sulphate-resisting cement pastes was investigated [5,6]. Uchikawa et al. [7] confirm that SNF polymer is adsorbed more particularly on the  $\text{C}_3\text{A}$  hydration products, when the immediate addition procedure is adopted. The superplasticizing effect improved with the delayed addition time of SNF because of the decrease adsorption capacity of the polymer on the  $\text{C}_3\text{A}$  hydration products.

Flatt and Houst [8] described the interactions and state of the superplasticizers with the cement suspensions into three parts. The first part is consumed by chemical reactions, in particular during the formation of AFt and C-S-H. The second part is adsorbed onto the surface of cement particles and help to disperse cement agglomerates. The third part is the superplasticizer, which remained in the aqueous phase once enough polymers have been added to satisfy the polymer consumption, the system can be said to be saturated with polymer.

The electrical conductivity is an important parameter to study the hydration process of cement pastes at early stages during the setting and hardening [9,10].

The aim of the present investigation is to evaluate the influence of new formaldehyde-free superplasticizers on the electrical conductivity, minislump and mor-

phology of cement pastes prepared by two addition methods as well as compressive strength of the hardened cement pastes. The phase composition was confirmed by the differential scanning calorimetry.

## EXPERIMENTAL

### Cement

A freshly produced sample of ordinary Portland cement (OPC) was supplied by Helwan Portland Cement Company. Table 1 shows the chemical composition of the OPC, Blaine surface area and the cement phase composition.

Table 1. Oxide composition, Blaine surface area and phase composition of OPC.

Oxide (wt.%)	OPC
SiO <sub>2</sub>	21.05
Al <sub>2</sub> O <sub>3</sub>	5.45
Fe <sub>2</sub> O <sub>3</sub>	3.42
CaO	63.41
Na <sub>2</sub> O	0.48
K <sub>2</sub> O	0.09
MgO	2.09
SO <sub>3</sub>	2.29
L.O.I	1.00
C <sub>3</sub> S	50
C <sub>2</sub> S	23
C <sub>3</sub> A	8.6
C <sub>4</sub> AF	10.4
C $\bar{S}$ H <sub>2</sub>	4.92
Free CaO	0.30
Blaine surface area, cm <sup>2</sup> /g	3154

### Preparation of superplasticizers

Sulphonated melamine formaldehyde free (SMFF) and sulphonate melamine urea formaldehyde free (SMUFF) were prepared through polycondensation of melamine or melamine/urea of (0.5) with glyoxylic acid at *pH* 3-4 and temperature of 50-60°C; which undergo farther condensation with sulphanilic acid at *pH* 5 and temperature of 50°C until final viscosity of 5.5 and 2.5 cSt; (20 wt% solution at 20°C), for SMFF and SMUFF respectively. The reaction mixture was adjusted to *pH* 10 and cooled to 25°C, the solid contents were 40.5 % and 38.5 % for SMFF and SMUFF respectively.

The chemical structure and properties of SMFF and SMUFF compared to other melamine based SMF are given in the previous publication [11].

### Fluidity of cement paste (minislump)

As indicated by its name, this method consists of a modified slump test designed to carry out on small amounts of cement paste. The cement paste is poured into a stainless steel a truncated cone with the same geometry as Abram's cone for regular slump tests, but with reduced dimensions (height of 40 mm, top diameter of 20 mm and bottom diameter of 40 mm) open at both ends (Figure 1). The polymer dosage of the admixture was chosen as 0.5 wt.%. The cement pastes were prepared at constant W/C ratio 0.4 and 0.50. Only 75 % of the mixing water was initially added and mixed for 3 minutes, then the rest of mixing water containing 0.5% of SMFF or SMUFF by wt.% of cement, added after delaying times of 0, 5, 10, 15 and 20 minutes. The mini-cone is removed and the spread area of the cement paste is measured. The measurements were made every 30 min up to 120 min.

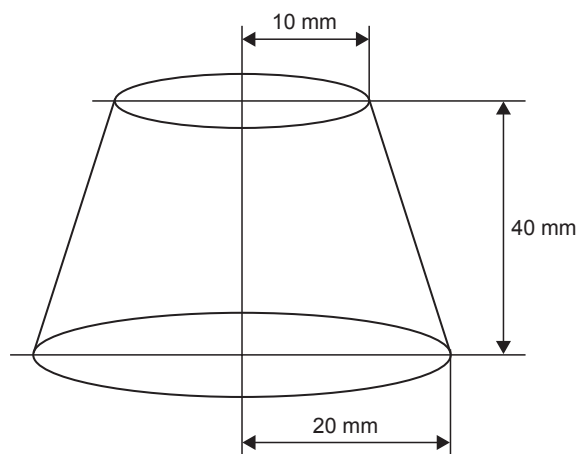


Figure 1. Mini-cone geometry.

### Electrical conductivity measurements

In electrical conductivity measurements, the test cell was of the co-axial type, which included concentric inner and outer electrode mounted on an insulated base plate [12,13]. The electrodes were polished before the experiments. The cement pastes with W/C ratio = 0.3 were placed in the space between the electrodes and kept at 100% relative humidity at 30 ± 1°C during the test for 24 hours. The electrodes were connected with RLC meter; model SR 720, operating at 1 kHz for resistance measurements. Only 75 % of the mixing water was initially added and mixed for 3 minutes, then the rest of mixing water containing 0.5 wt.% of SMFF or SMUFF were added after delaying times of 0, 5, 10, 15 and 20 minutes. The measurement of the electrical

conductivity began exactly after 25 minutes (zero time was measured after the final delayed time 25 minutes) from the initial mixing with water (zero time), during the test period up to 24 hours.

#### Differential scanning calorimetry

Differential scanning calorimetry runs were conducted on hydrated cement pastes cured at 3 days age, using a Shimadzu DSC-50 thermal analyzer at a heating rate of 10°C/min. The sample chamber was purged with nitrogen at a flow of 20 ml/min.

#### Scanning electron microscopy

The morphology of the hydration products was investigated through ESE-MEDX after specified hydration times. The accelerated voltage used was 25 kV. The specimens of crushed pastes were immersed in 1:1 acetone methanol overnight and dried at 70°C to remove the evaporable water content then coated with gold before examination under the electron microscopy.

## RESULTS AND DISCUSSION

### Fluidity of cement paste (minislump)

The variations of minislump of cement pastes with delaying addition time are given in Figure 2. The increase in fluidity of cement paste was caused by increase of electric repulsive force between the particles. Simultaneous addition time lowered the effectiveness of the admixture; when added with mixing water, due to a partial coating of the admixture by the early hydrated products. As the superplasticizer was added to cement pastes in simultaneous addition time, the rapid adsorption of the polymer on cement particles causes a

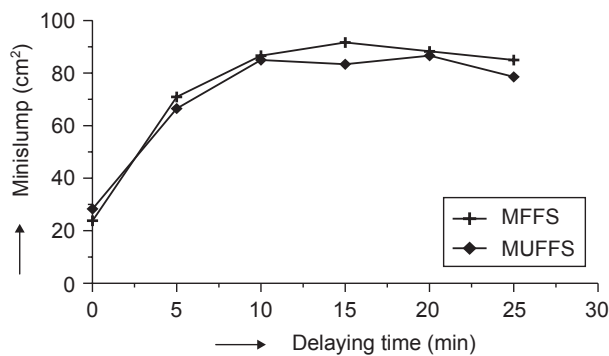


Figure 2. Effect of delayed addition time on minislump determined at 30 minutes in the presence of 0.5 % SMFF or SMUFF (W/C = 0.4).

strong dispersion thus generating an initial, high fluidity of the pastes. At the same time, the polymer causes a reduction in the hydration rate of the cement, due to the formation of a film around the cement particles and/or hydrated products [14].

The fluidity of cement pastes prepared in the presence of 0.50 % SMFF or SMUFF shows a higher value in the case of delaying addition time. Probably the superplasticizer is not incorporated within the hydrated products and, therefore remains almost completely, thus a larger fraction is available for dispersion of the cement particles. Consequently, the fluidity of the pastes is higher than that found for simultaneous addition time. In the delaying addition time, the amount of polymer adsorbed on the unhydrated cement particles is much lower than that at simultaneous addition time.

Delaying addition time enhances the fluidity of cement pastes, due to the decrease of the anhydrous content of C<sub>3</sub>A content within the first 10-15 minutes of the hydration and the admixture adsorption on anhydrous C<sub>3</sub>S and C<sub>3</sub>A phases is higher than that on hydrated phases [5]. The positive sites of aluminate phases are responsible for consuming the amounts the superplasticizers, which reduced within delayed addition time.

Figure 3 shows the effect of delayed addition (15 min) of the superplasticizers on the minislump of cement pastes with time. The spread area of minislump is large in the case of delaying addition time than simultaneous addition time. In the delayed addition, the superplasticizer was adsorbed to a lesser extent on C<sub>3</sub>A/gypsum mixture already undergoing hydration of the ettringite formation and enough of the superplasticizer is left to promote the dispersion of the C-S-H phases and increases the system fluidity [14]. The adsorption of superplasticizer is virtually terminated after 1 to 2 minutes of mixing and as such a great part of superplasticizer is adsorbed on the hydrated products so that the dispersing effect of the admixture is greatly reduced.

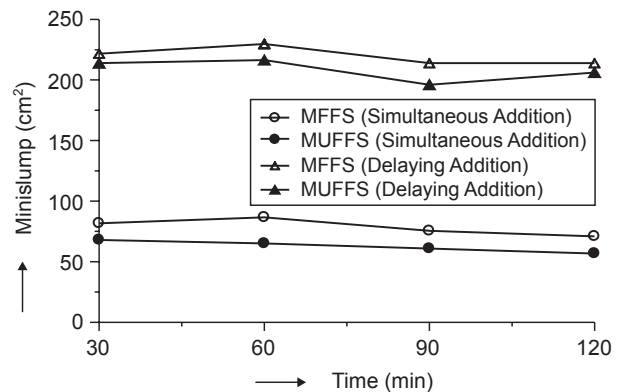


Figure 3. Effect of delayed addition time (15 min) on minislump in the presence of 0.5 % SMUFF or SMFF (W/C = 0.5).

## Electrical conductivity

Figures 4 and 5 show the effect of delaying time on the electrical conductivity. As the delaying time increases the initial electrical conductivity decreases. Also, the delaying addition time for SMFF and SMUFF respectively shows an increase in the height of electrical conductivity maximum within ten up to fifteen minutes. This is due to the changes in the rheological properties of cement pastes, which are the result of changes in the disperse structure of the cement particles. It is well known that the adsorption of superplasticizers on the surfaces of the cement particles and its hydrates generate high negative zeta potentials, thus causing a strong dispersion of the cement particles. Steric stabilization is also a factor contributing to the dispersing effect of the superplasticizers. The overall result is an increase in fluidity of the cement pastes.

The polymer gets in the solution and adsorbs over the surface of cement grains blocking the hydration. Since, polymer has a depressive effect; it increases the

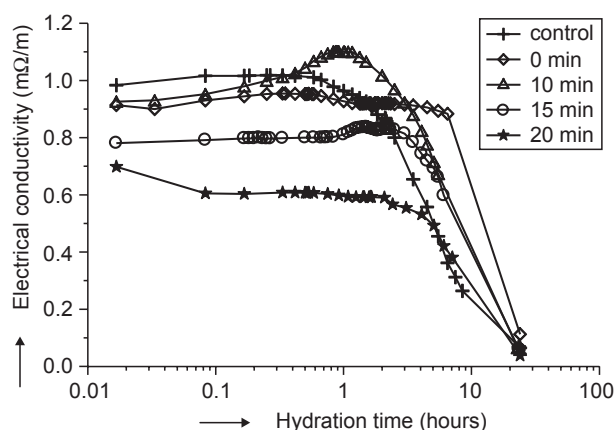


Figure 4. Electrical conductivity of OPC in presence of SMFF, simultaneous and delaying addition time.

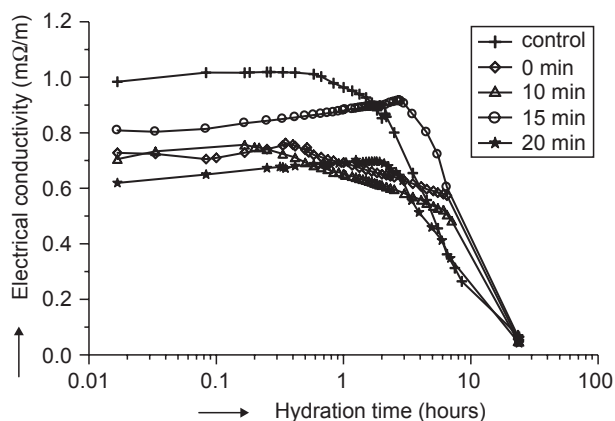


Figure 5. Electrical conductivity of OPC in presence of SMUFF, simultaneous and delaying addition time.

workability of the paste. In the presence of polymer, it appears that only a few  $\text{Ca}^{2+}$  ions goes into the solution and supersaturation with respect to  $\text{Ca}(\text{OH})_2$  is not reached. The dissolution of  $\text{Ca}^{2+}$  ions increases with time resulting in an increase of the conductivity. Conductivity continues to increase until the  $\text{Ca}^{2+}$  ions becomes saturated with respect to  $\text{Ca}(\text{OH})_2$ . As the amount of admixture added, the conductivity maximum becomes broader with a lower intensity and shifted to a longer time [9].

Superplasticizer forms a complex with  $\text{Ca}^{2+}$  ions liberated on the surface of C-S-H gel or  $\text{Ca}(\text{OH})_2$  crystals; the interweaved net structure consists of ion bonded large molecular system bridged by means of  $\text{Ca}(\text{OH})_2$  [5, 15, 16]. Superplasticizer inhibits the growth of hydrates as indicated by shifting and broadening of the electrical conductivity peak (Figures 4, 5).

The conductivity maximum becomes broader and shifted upon giving longer hydration time as shown in simultaneous addition time. This period begins with a slow decrease in the electrical conductivity. After the short initial period of fast hydration, the overall hydration reaction rate slows down significantly for a period of a few hours (dormant period) [17]. The production of ettringite (AFt) is depressed in the cement [16].

The electrical conductivity peak is mainly due to two factors. The first one is due to the partial transformation of ettringite to monosulphate; the formation of monosulphate produces of  $2\text{Ca}^{2+}$  and  $2\text{SO}_4^{2-}$  gm ions per one mole of ettringite, leading thus to an increase in the number of ions [18-20]. The second factor is osmotic pressure development around the cement grains, which leads to an increase in the ionic mobility.

## Differential scanning calorimetry

Figures 6-8 show DSC thermograms of neat OPC (Blank sample) and OPC pastes containing 0.5 wt% of SMUFF or SMFF superplasticizers mixed with simultaneous addition (0 min.) and delaying addition time of 0, 5, 10, 15 and 20 minutes, then the specimens cured at 3 days. The thermograms show five endothermic peaks located at 88-100, 120-135, 200-225, 340-380 and 420-450°C. The first endothermic peak is due to decomposition the calcium silicate hydrates (C-S-H). The second endothermic peak located at 120-135°C is due to decomposition of ettringite phase ( $\text{C}_3\text{A}\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$ ). The third endothermic peak between at 200-225°C is due to dehydrated of monosulphate phase ( $\text{C}_3\text{A}\cdot \text{CaSO}_4\cdot 12\text{H}_2\text{O}$ ). The peak between 420-450°C is attributed to the dehydroxylation of  $\text{Ca}(\text{OH})_2$ .

Figure 6 shows the DSC thermograms for neat OPC and superplasticized pastes obtained by simultaneous addition time. It is clear that the enthalpy values of

peaks corresponding to the decomposition of the calcium silicate and aluminate hydrate phases as well as  $\text{Ca}(\text{OH})_2$  of superplasticized specimens are less than that of neat cement paste. This is attributed to the retardation effect of superplasticizers.

Figure 7 shows DSC thermograms of superplasticized pastes (SMFF) mixed after delaying times of 0, 5, 10, 15 and 20 minutes. The enthalpy values of pastes

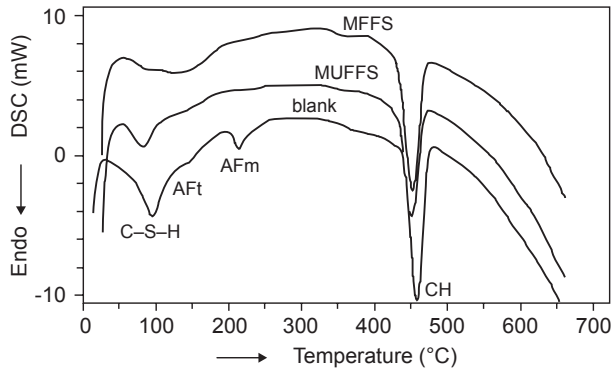


Figure 6. DSC thermograms of OPC containing SMUFF or SMFF superplasticizers mixed by simultaneous addition time.

containing SMFF corresponding to decomposition of C-S-H and  $\text{Ca}(\text{OH})_2$  decrease with delaying addition time. The superplasticizer retard the conversion of  $\text{C}_3\text{A}\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$  to  $\text{C}_3\text{A}\cdot \text{CaSO}_4\cdot 12\text{H}_2\text{O}$  as shown in the Figure 7, the maximum retardation of this conversion reaction was shown at 20 minutes delaying addition.

Figure 8 shows DSC thermograms of superplasticized pastes with SMUFF. The enthalpy values of pastes containing SMFF corresponding to  $\text{Ca}(\text{OH})_2$  and C-S-H somewhat affected by the delaying addition time up to 15 minutes. At delaying addition time of 20 minutes, show the same retardation effect as shown in Figure 7.

At delaying addition time 20 minutes, superplasticizer molecules are not only adsorbed on unhydrated cement phases but also adsorbed on the hydrated products. This adsorption on the hydrates slows down drastically or even stops the growth of ettringite germs. However, when all the initial superplasticizer molecules have been consumed, normal growth of ettringite resumes. This interaction between superplasticizer molecules and ettringite can be used to explain the important water consumption in the early stages of mixing [21].

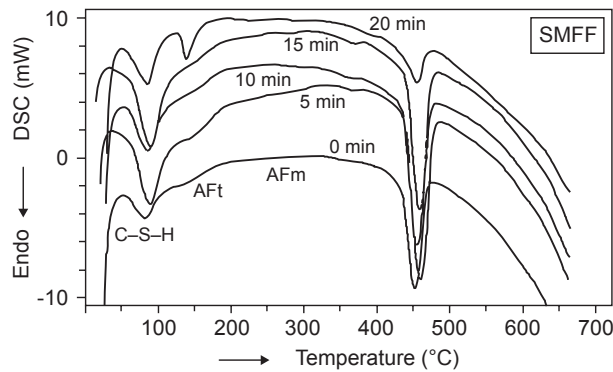


Figure 7. DSC thermograms of OPC and OPC containing SMFF superplasticizers with delayed addition time.

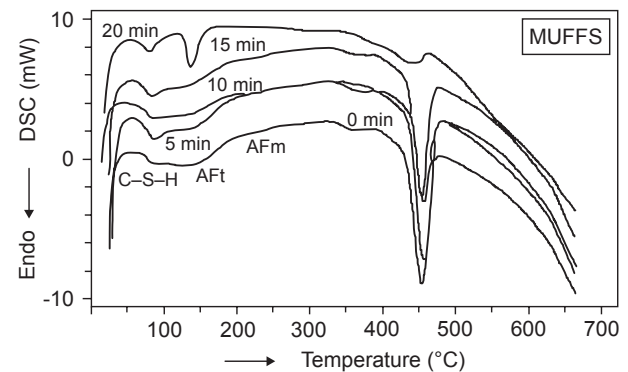


Figure 8. DSC thermograms of OPC containing SMUFF superplasticizer with delayed addition time.

Table 2. The enthalpy values of the characteristic endothermic peaks with temperatures (J/g).

Delayed Time		88-100°C	120-135°C	200-225°C	340-380°C	420-450°C
Blank	0	36.89	0.694	7.17	0.981	47.05
SMFF	SMFF 0	0.189	8.35	0.189	1.37	33.38
	SMFF 5	3.35	4.78	0.026	1.03	39.29
	SMFF 10	3.71	3.13	0.388	1.04	33.25
	SMFF 15	2.62	0.665	0.314	-	19.85
	SMFF 20	5.09	8.64	0.418	0.622	5.34
SMUFF	SMUFF 0	16.81	1.12	0.573	0.513	43.07
	SMUFF 5	29.92	1.01	0.212	0.522	43.79
	SMUFF 10	26.36	1.10	0.233	0.693	47.88
	SMUFF 15	39.63	0.272	0.156	1.026	40.62
	SMUFF 20	18.50	6.46	0.614	0.331	10.47

The enthalpy values of the characteristic endothermic peaks of decomposed hydrates containing SMFF and SMUFF superplasticizers mixed delayed addition time up to 20 minutes for 3 days are given in Table 2. It was shown that SMFF retard the formation of C–S–H. The SMUFF retards the conversion of  $C_3A \cdot 3CaSO_4 \cdot 32H_2O$  to  $C_3A \cdot CaSO_4 \cdot 12H_2O$ ; the maximum retardation of this conversion reaction was shown at 20 minutes delaying addition.

### Scanning Electron Microscopy

Figure 9 (A) shows the microstructure of the OPC paste cured at 1 day. The micrograph detected the formation of gel around the cement particles and the honeycomb structure of the C–S–H (II). Secondary growth of ettringite was also seen in micrograph, where rods of approximately 1-1.5  $\mu m$ .

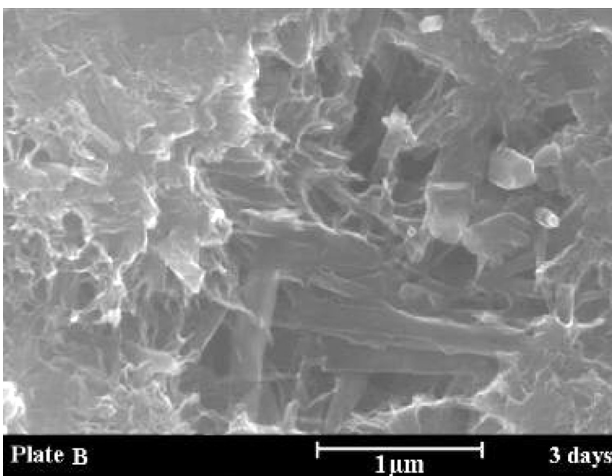
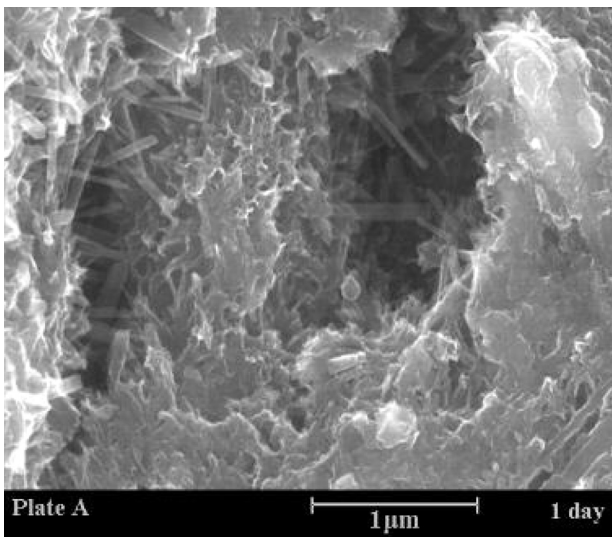


Figure 9. SEM micrograph of OPC pastes cured at 1 and 3 days.

After 3 days, OPC paste has a homogeneous microstructure Figure 9 (Plate B), with few ettringite rods. The apparent decrease in the amount of ettringite can be due to the formation of AFm. Also, AFm usually grows inside the shell of the cement grains due to the drop in the concentration of sulphate, which makes their observation through SEM difficult.

Superplasticized pastes have homogeneous microstructures (as in Figures 10, 11 Plates (C-F) similar to those observed at 1 day. The morphology of the C–S–H in the superplasticized pastes appears to be slightly different, being less dense than that of the OPC paste. The secondary growth of ettringite is evident in Figure 10 (Plate C), with the number and size of the rods being smaller than in the OPC paste, where some clusters of rods with radial growth have also been observed Figure 11 (Plate E). Few rods of ettringite have been found in the D and F pastes.

Chandra et al. [16] suggested that the retardation effects of superplasticisers in cement/cement admixtures is due to the interaction of  $Ca^{2+}$  ions with superplasticisers, that holds their action and causes the acceleration of early ettringite gel formation or inhibition of the conversion of ettringite to monosulphate.

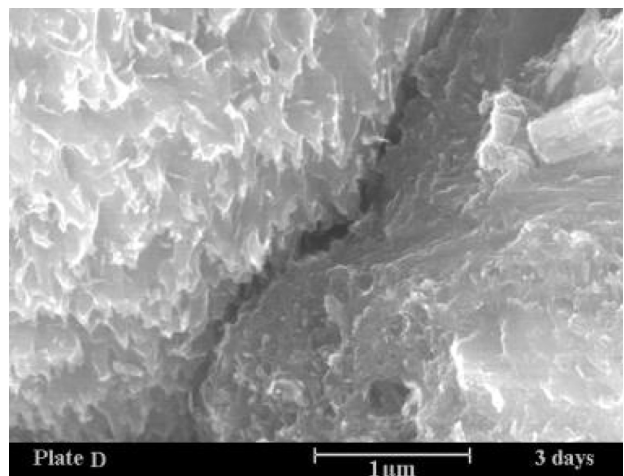
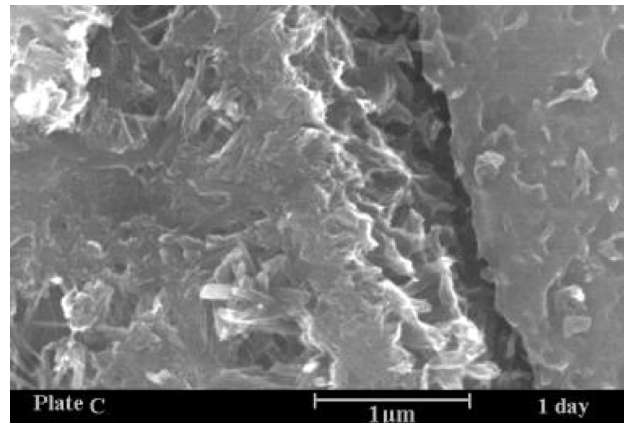


Figure 10. SEM micrograph of OPC pastes cured at 1 and 3 days in presence of SMFF.

Presence of superplasticized pastes, well-formed hexagonal crystals of CH was observed as well as the massive clusters of parallel crystals. These crystals are fewer in the SMFF paste than in the others superplasticized pastes (SMUFF). The absence of crystals of CH seems to complete the DSC thermograms in a way that microcrystalline CH with a high range of nucleation predominates in these cement pastes at 3 days.

The SEM micrograph observations of the OPC paste seem to confirm this high degree of supersaturation, since large crystals of CH have not been observed. Interestingly, admixture can be seen in Figures 10, 11, Plates C-F as thin curved plates.

### Compressive strength

Figure 12 shows the variation of compressive strength as result of delayed addition time on cement pastes with 0.5 % SMFF (W/C = 0.3). The compressive strength values enhance with the addition of the admixture at early and later ages (28 days simultaneous addition time). The compressive strength values increase with delaying addition time than simultaneous addition.

### CONCLUSIONS

On the basis of the studies carried out it can be concluded:

1. The initial electrical conductivity decreases with the delayed addition time of superplasticizer.
2. The delayed addition time enhancing the fluidity of cement pastes, due to  $C_3A$  content that is minimized by hydration until 10-15 minutes. The spread area of minislump increases in the case of the delaying addition time than that of simultaneous addition time.
3. The DSC thermograms of peaks corresponding to the decomposition of the calcium silicate and aluminate hydrated phases as well as  $Ca(OH)_2$  decreases in superplasticized specimens than neat cement paste. This is attributed to the retardation effect of superplasticizer; the maximum retardation was shown at 20 minutes delaying addition time.

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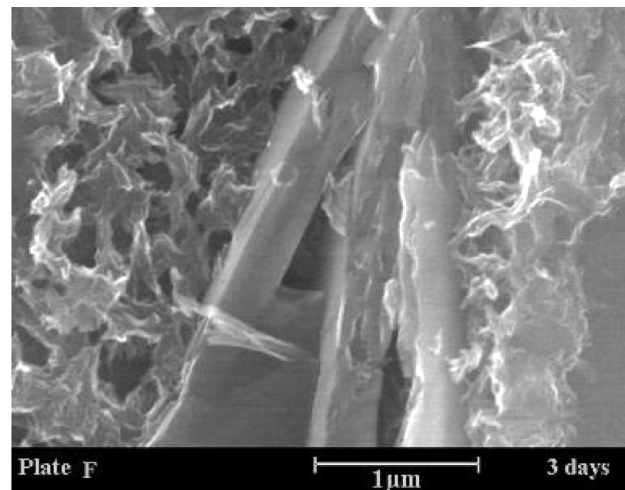
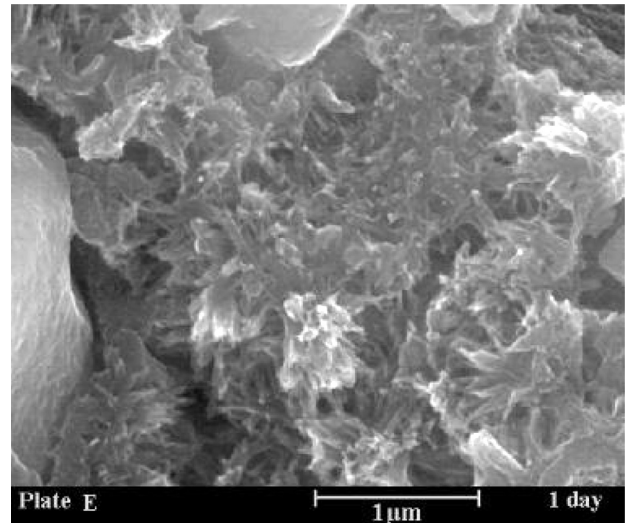


Figure 11. SEM micrograph of OPC pastes cured at 1 and 3 days in presence of SMUFF.

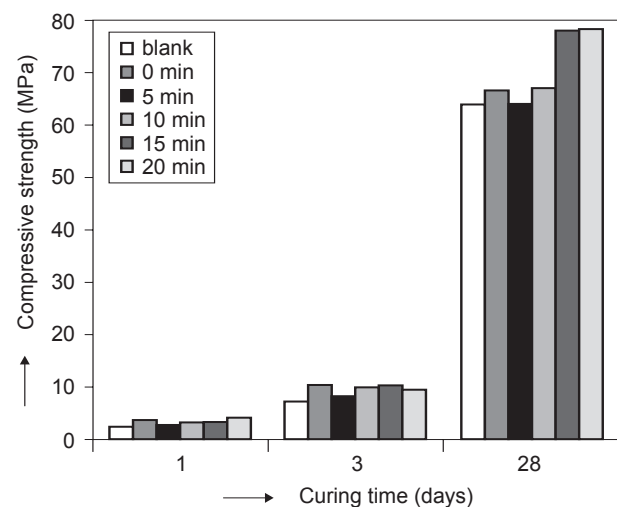


Figure 12. Compressive strength of cement pastes mixed with delayed addition time in the presence of 0.5 % SMFF (W/C = 0.3).

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VLIV ODDÁLENÍ DOBY PŘÍDAVKU  
SUPERPLASTIFIKÁTORŮ NA CHEMICKÝ PROCES  
A VLASTNOSTI CEMENTOVÝCH PAST

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Tato práce prezentuje vliv oddálení doby přidavku vodorozpustných polymerů na bázi polykondenzátů, konkrétně melaminsulfonátu (SMFF) a kondenzačních produktů melaminu-močoviny bez obsahu formaldehydu (SMUFF), na fyzikálně chemické vlastnosti cementových past. Superplastifikátory se přidávaly dvěma různými metodami: a) buď společně se záměsovou vodou (současné dávkování); nebo b) později, po určité době (oddálený čas dávkování). Byla provedena malá zkouška rozlitím kužele (minislump) v různých časových intervalech: 30, 60, 90 a 120 minut. Dále byla studována elektrická vodivost cementových past připravených s nebo bez přidavku superplastifikátorů. Výsledky ukazují, že původní vodivost se s rostoucím oddálením přidavku přísady snižuje. Doba oddáleného dávkování od 10 do 15 minut vedla k nárůstu výšky maxima elektrické vodivosti. Doba oddáleného dávkování zvyšuje tekutost cementových past ve srovnání s dávkováním současně se záměsovou vodou. Výsledky dále ukazují, že hodnoty entalpie, které odpovídají rozkladu křemičitanu vápenatého, hlinitanu vápenatého a hydratovaných fází sulfohlinitanu a také  $\text{Ca}(\text{OH})_2$ , ve vzorcích s přidavkem superplastifikátorů v porovnání s cementovou pastou bez superplastifikátorů klesají. To se přičítá retardačnímu efektu superplastifikátoru. Maximální retardace byla zjištěna při době oddáleného dávkování 20 minut.