

EFFECT OF DELAYING ADDITION TIME OF SMF SUPERPLASTICIZER ON HYDRATION CHARACTERISTICS OF BLENDED CEMENT PASTES

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This paper aims to study the effect of delaying addition time of sulphonate melamine formaldehyde superplasticizer (SMF) on the rheology and hydration characteristic of blended cement pastes up to 90 days. The optimum dosage of superplasticizer was chosen as 1.0 mass %, this dosage gives the higher compressive strength and bulk density as well as lower total porosity. The optimum delaying time of superplasticizer was 7.5 min which gives the highest fluidity, bulk density, compressive strength and chemically combined water contents of cement pastes, whereas, the delaying addition time of 15.0 min shows the lower values of bulk density, compressive strength and combined water contents. This is attributed to the reduction of hydration rate of cement, due to the formation of a polymer film around the cement particles and/or hydrated products. The superplasticizing effect improved with a delayed sulphonate melamine formaldehyde superplasticizer addition time.

INTRODUCTION

Modern concrete technology projections indicate a great expansion in low-cost and time efficient constructions. Chemical admixtures have been beneficial found in offsetting some of the undesirable characteristics of concrete in hot climates. Some advantages of the superplasticizers are reducing water content, extending periods of workability (retard the initial and final setting times) and enhancing strength.

Superplasticizers are widely used in the production of concrete with excellent workability giving high performance, high strength and improve the durability. The factors affecting the performance of superplasticizers were type, dosage of the superplasticizers, type of cement, temperature and mixing procedure, as well as the addition time of the superplasticizers, which cause different fluidizing effects for the resulting cementitious materials [1].

Superplasticizers such as polynaphthalene sulphonate and polymelamine sulphonate formaldehyde condensates are widely used in the concrete industry. These two polymers have essentially the same dispersing mechanism. The main polymers chains adsorb to the surface of cement particles; the cement particles become negatively charged via the negatively charged sulphonate groups of the side chains. Consequently, electrostatic repulsion occurs between the cement particles. The cement particle arrangement in a paste matrix offsets the inter-particle attractive forces.

The effects of delayed additions of sulphonated naphthalene (SNF) and melamine- formaldehyde (SMF) condensates upon workability of Portland cement pastes have been investigated. The maximum workability is obtained when the admixture is added at the beginning of the dormant period of the cement hydration without admixture. The existence of optimum addition time is explained by correlating the dispersing effect of the superplasticizers to their adsorption rate on the cement particles and to the hydration rate of the cement pastes with admixture [2].

The fluidity of Portland cement pastes is greatly affected by the time of addition of superplasticizers. When the addition of polymer is delayed the workability is always found to be higher, and the reduction in fluidity with increased mixing time is lower in comparison with immediate addition of admixture (simultaneous addition). The maximum increase in workability is obtained when the addition time of superplasticizer (optimum addition time) corresponds to the beginning of the dormant period of the hydration of cement without admixture.

The influence of delaying addition time of sulphonated melamine and melamine-urea formaldehyde-free on the properties of cement pastes after 30, 60, 90 and 120 minutes was investigated [3]. The superplasticizers were added by simultaneous and/or delayed addition time. The minislump and electrical conductivity of cement pastes admixed with superplasticizers were studied. The initial electrical conductivity decreases with delayed addition time of admixture. Delaying addition time from 10 to

15 minutes increases the height of electrical conductivity maximum due to the increase of fluidity of cement pastes than that of simultaneous addition. The enthalpy values corresponding to the decomposition of calcium silicate, calcium aluminate and sulphoaluminate hydrated phases as well as $\text{Ca}(\text{OH})_2$ decrease with superplasticized specimens in comparison with neat cement pastes. This is attributed to the retardation effect of the superplasticizer. The maximum retardation was shown at 20 minutes delaying addition time.

The effect of delaying addition time of SMF and SNF on the rheological properties of OPC and SRC cement pastes through the first 120 min of hydration was studied [4]. Shear stress and apparent viscosity were determined at different shear rates ($3\text{--}146\text{ s}^{-1}$) after 30-120 min. The increase in delaying addition time reduces the shear stress, yield stress, and plastic viscosity of cement pastes at early (15 min) and at later ages (120 min). The optimum delaying addition time is 10-15 min. As the delaying addition time increases, the saturated adsorption amount of SNF decreases sharply at the beginning and then more slowly [5]. On the other side, the concrete workability decreases slightly at the early age and then falls abruptly. Most importantly, the transition points in both cases appear to be the same, at about 10-15 min. This strongly suggests that a close relationship exists between the SNF adsorption behavior on cement particles and the workability of concrete. In addition, the optimum addition time of SNF to concrete should be in this period, which corresponds to the beginning of the dormant period of the cement hydration process.

The present investigation aimed to evaluate the influence of delaying addition time of laboratory prepared sulphonate melamine formaldehyde superplasticizer (SMF) on the rheology and hydration characteristic of cement pastes up to 90 days.

EXPERIMENTAL

Materials and methods

The materials used were sulphate resisting cement (SRC) provided from El-Masria Cement Company, and condensed silica fume (SF) from Ferrosilicon Alloys Company, (Edfo, Aswan, Egypt). The chemical composition of these materials is shown in Table 1.

Table 1. Chemical analysis of starting materials (mass %).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	I.L	Total
SRC	21.40	3.67	5.05	64.73	1.50	2.05	0.30	0.22	2.60	99.70
SF	94.81	0.16	0.84	0.89	0.49	0.08	0.20	0.05	2.43	99.95

Superplasticizer

Laboratory synthetic water soluble polymer, sulphonate melamine formaldehyde superplasticizer condensate was prepared [6]. The structure of this synthesized polymer was shown in Figure 1. The solid content of the product is 34.4 mass %. The chemical structure of sulphonate melamine formaldehyde superplasticizer is shown in Figure 1.

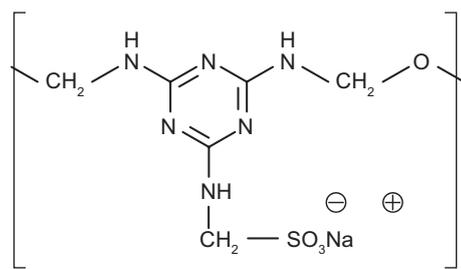


Figure 1. The chemical structure of sulphonated melamine formaldehyde superplasticizer.

Preparation of dry mixes

Sulphate resisting cement and 10 mass % silica fume were blended in a porcelain ball mill for two hours using a mechanical roller to insure complete homo-geneity.

Water of consistency and setting time

The required water of standard consistency gives a paste which permitted the settlement of the Vicat plunger (10 mm. in diameter) to a point 5-7 mm from the bottom of the Vicat mould (ASTM Designation: C187-92, 2008) [7]. The normal consistency was measured to get all specimens having the same workability. The setting times were determined according to ASTM specifications at 7.5 min delaying addition time [8]. The specimens were cured in a humidity chamber at $25 \pm 1^\circ\text{C}$ for 24 hours, then demolded and immersed in tap water until tested.

Fluidity of cement paste (minislump)

Minislump was carried out by a slump test. The cement pastes were prepared at constant W/C ratio of 0.55 with 0.0, 0.5, 1.0, 1.5 and 2.0 mass % of superplasticizer. The cement pastes were stirred by mechanical stirrer for three minutes, rest for one minute

and two minutes by the same speed, then poured into a stainless steel cone with the same geometry as Abram's cone for regular slump tests, but with reduced dimension [3]. The minicone is put on vibrated table, removed vertically and the diameter of the spread of cement paste is measured.

Bulk density measurement

The bulk density (dp) was determined from the following equation:

$$dp = \frac{W_1}{W_1 - W_2} \quad (1)$$

where W_1 is the saturated surface dry weight in air (g) and W_2 is the submerged weight in water (g) [8,9].

Total porosity measurement

The total porosity (ξ) was determined from the evaporable (W_e), total water (W_t) contents and bulk density (dp) of the hardened cement specimen using the following equation [8, 9]:

$$\xi = \left(\frac{0.99 W_e \times dp}{1 + W_t} \cdot 100 \right) \quad (2)$$

where 0.99 is the specific volume of free water.

Compressive strength measurement

A set of three cubes was used for the determination of compressive strength of cement paste (ASTM Designation: C-150, 2007). The compressive strength measurements were done on a compressive strength machine of SEIDNER, Riedinger, Germany, with maximum capacity of 600 KN force.

Determination of free lime

The free lime content of cement paste can be thermally determined. 0.5 g of the hardened cement was placed in a porcelain crucible and introduced into a cold muffle furnace. The temperature was increased up to 390 then to 550°C at heating rate of 3°C/min. The loss of weight occurred between 390 – 550°C with soaking time of 15 min is equal to the weight of water of calcium hydroxide. Therefore, the free lime can be calculated.

Stopping of the hydration

The non-evaporable water is retained in the sample after the free water is removed by drying the saturated cement pastes at 105°C for 24 hrs.

Determination of combined water

The combined water content (W_n) of samples predried at 105°C for 24 hours was determined as the

ignition loss at 1000°C for 1 hour. Combined water content = ignition loss of the hydrated sample – (ignition loss of anhydrous mix + water of free lime) [9].

Infrared spectroscopic analysis (IR)

The samples were prepared using alkali halide pressed disk technique as it gives a further reduction in scattering [10]. 2 mg of the sample was ground with 198 mg of KBr in an agate mortar to produce a homogeneous mixture. The mixture pressed under vacuum to give a transparent disc of 1 cm in diameter. The infrared spectral analysis was recorded from KBr-discs using Genesis -IR spectrometer in the range 400-4000 cm^{-1} .

RESULTS AND DISCUSSION

The required water of standard consistency, initial and final setting times

The use of superplasticizers is needed to eliminate defects from high percent of mixing water during the preparation of cement pastes, mortars or concrete. The high water/cement ratio produces a relatively amorphous material, which directly affects the mechanical properties as well as chemical performances. Superplasticizers are effective in breaking and dispersing the cement particle networks, preventing premature linkage as well as minimizing the amount of standard water of consistency required to suspend the particles and render the mix workable. The admixtures control the forces between particles and increase fluidity of the mix. As a result of this, less water is required to achieve a given consistency. These polymeric materials are adsorbed on cement particles, forming a good ionic repulsion raised from the negative charge of sulphonate groups, consequently increasing its fluidity [11]. The required water of standard consistency, initial and final setting times of OPC-SF cement pastes with superplasticizer content were given in Figure 2. The required water of standard consistency decreases with superplasticizer

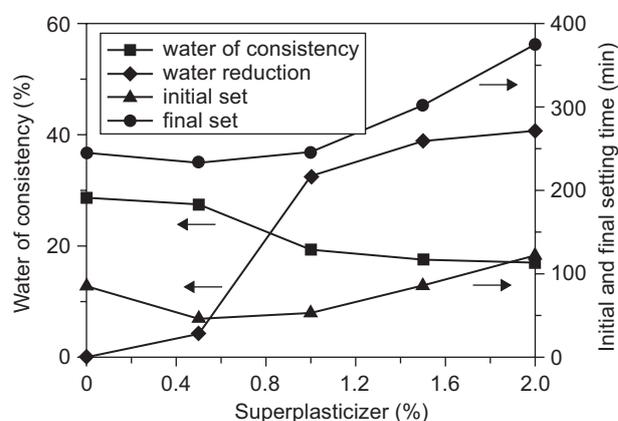


Figure 2. Water of standard consistency, water reduction, initial and final setting times.

content due to its coating effect. The water reduction increases with superplasticizer content [11].

Initial and final setting times of cement pastes with superplasticizer were determined at 7.5 min delaying addition time. Cement pastes with 0.5 % superplasticizer shows a relative decrease in the initial and final setting time than that of neat cement pastes; this may be attributed to water reduction effect of superplasticizer. Increase of the dosages of superplasticizer up to 2.0 mass %, the initial and final setting times were elongated due to the dispersion action on flocculated cement particles and reduction in the concentration of contact points between different grains. Also, SRC shows slow hydration rate, due to the lower content of C_3A , which causes rapid setting and hydration.

Fluidity of cement paste (minislump)

The relationships between the amount of SMF superplasticizer and the fluidity of cement pastes were shown in Figure 3. The fluidity of cement pastes is defined as the spread area of paste after removing the minislump cone. As the SMF superplasticizer was added to cement pastes, the rapid adsorption of the superplasticizer on cement particles causes a strong dispersion thus generating an initial high fluidity of the pastes (simultaneous addition). At the same time, the superplasticizer 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 [2, 3].

The effect of delaying addition on minislump of cement pastes is given in Figure 3. The optimum amount of superplasticizer was chosen as 1.0 mass %, this dosage gives the higher compressive strength and bulk density as well as the lower total porosity as shown later. The delaying addition time was taken up to 15.0

min. The spread area of minislump increases in the case of delaying addition than simultaneous addition. Simultaneous addition lowers the effectiveness of the admixture, due to a partial coating of early hydrated products [4]. When the superplasticizer was added in the case of simultaneous addition time, rapid adsorption of the polymer on cement particles occurred. But in the delaying addition, the superplasticizer was adsorbed to a lesser extent on C_3A /gypsum mixture already undergoing hydration of the ettringite formation and enough of the superplasticizer is left to promote the dispersion of the CSH and increase the system fluidity [2]. 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 [3]. Also, it is found that the optimum delaying addition time of superplasticizer was 7.5 min., which gives the highest fluidity of cement pastes. Increase the delaying addition time of superplasticizer (10 min) the fluidity of cement pastes decreased.

Bulk Density and total porosity

The bulk density and total porosity of cement pastes with 1.0 % of SMF mixed at delaying addition time up to 15.0 min for 90 days is given in Figure 4a. As the curing time proceeds, the bulk density increases, whereas total porosity decreases. More hydration products are formed, which fill up some available open pores [12]. As the delaying addition time increases for 7.5 min the bulk density increases, due to the decrease of the anhydrous C_3A content within the first 0.0 min to 7.5 min of hydration. The admixture adsorption takes place on anhydrous C_3S and C_3A phases, which is higher than that on the hydrated phases [4]. The superplasticizing effect is improved with a delayed addition because of the lower

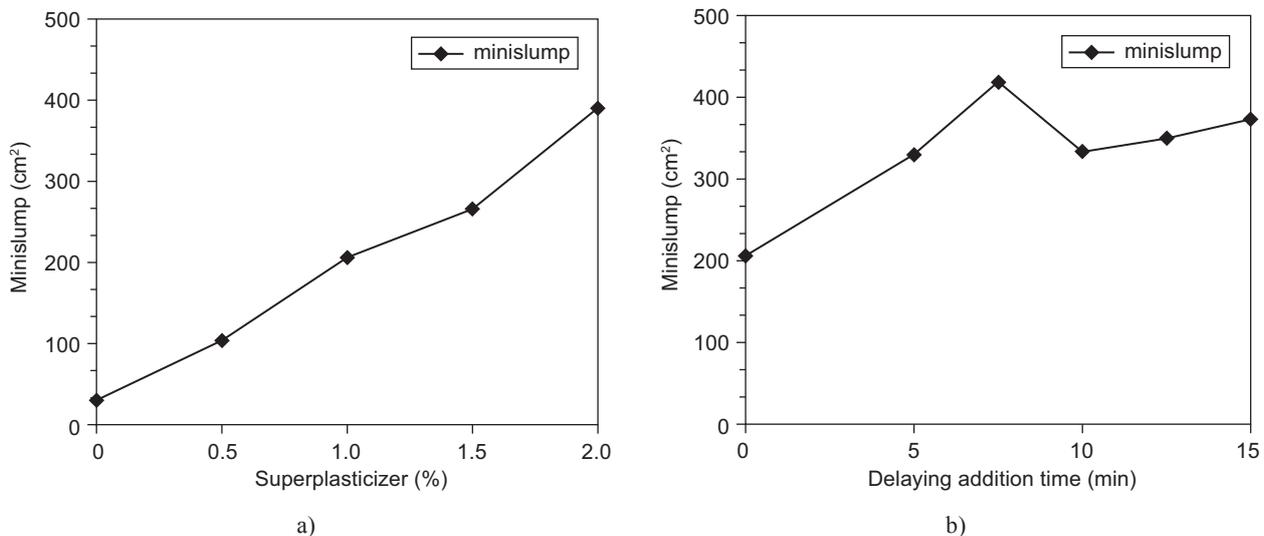


Figure 3. The minislump of cement pastes: a) effect of different dosages of superplasticizer; b) effect of delaying addition time of 1.0 % superplasticizer up to 15 min.

adsorption of the polymer on the C_3A hydrated products. Consequently, the adsorption of SMF molecules on the prehydrated cement surface is reduced and more superplasticizer molecules are left for dispersion of C_3S and β - C_2S that causes the subsequent dispersion action to be more effective than that recorded in the simultaneous addition procedure [13, 15].

In the delaying addition time, the amount of SMF superplasticizer adsorbed on the anhydrated cement particles is lower than that at simultaneous addition. At 15.0 min the bulk density decreases due to the retardation of the hydration. SMF polymer molecules can react with Ca^{2+} ions liberated from cement hydration forming complex with $Ca(OH)_2$ on the surface of CSH gel or $Ca(OH)_2$ crystals; the interweaved net structure consists of ion bonded large molecular system bridged by means of $Ca(OH)_2$ [16, 17]. A superplasticizer polymer molecule inhibits the growth of hydrates. The interaction between superplasticizer molecules and ettringite can be used to explain the important water consumption at early stages of mixing [3, 18].

The bulk density and total porosity of cement pastes admixed at delaying addition time of 7.5 min with SMF up to 2.0 % for 90 days is illustrated in Figure 4b. The data show that the total porosity decreases with curing time. This is mainly due to the increase of the amount of hydration products. The total porosity decreases with the SMF percent up to 1.0 mass %. This is attributed to the effect of superplasticizer polymer on the reduction of water of consistency, which is accompanied by increasing the bulk density as well as gel/space ratio values [11]. Increase the amount of superplasticizer up to 2.0 mass % the bulk density decreases due to the retardation effect of superplasticizer. Superplasticizer adsorbed preferentially onto C_3A and early formed hydration products prevents early hydration and modifies the microstructure of the hydrated phases. Fluidification of cement pastes depends

on the type and dosages of the admixture as well as particle size distribution and mineral composition of cement.

Compressive strength

Compressive strength of cement pastes containing 1.0 mass % SMF superplasticized mixed with delaying addition time up to 15.0 min cured for 90 days is given in Figure 5a. The compressive strength increases with curing time due to the increase of the amount of hydration products especially tobermorite-like gel (CSH) which is the main source of the strength. As the hydration proceeds, more hydration products are formed filling some of the open pores leading to increase the compressive strength of cement pastes. The hydration products possess a large specific volume than unhydrated cement components, therefore the accumulation of the hydrated products fill a part of originally filled spaces (pores). This leads to decrease the total porosity of cement paste as well as increase the bulk density and gel/space ratio values [11]. The increase of extent of hydration as measured by the combined water contents as shown later and the decrease of the total porosity with hydration time, which work together leading to increase the ultimate compressive strength. As the delaying addition time increases to 7.5 min the compressive strength increases due to the improvement of superplasticizing affect with the delayed addition of superplasticizer. Increase of delaying addition time for 15.0 min the compressive strength decreases due to the reduction of the hydration rate of the cement, as a result of the formation of a film around the cement particles and/or hydrated products [3].

The compressive strength of cement pastes admixed with different dosages of superplasticizer with delayed addition time at 7.5 min up to 90 days is represented Figure 5b. The compressive strength increases with

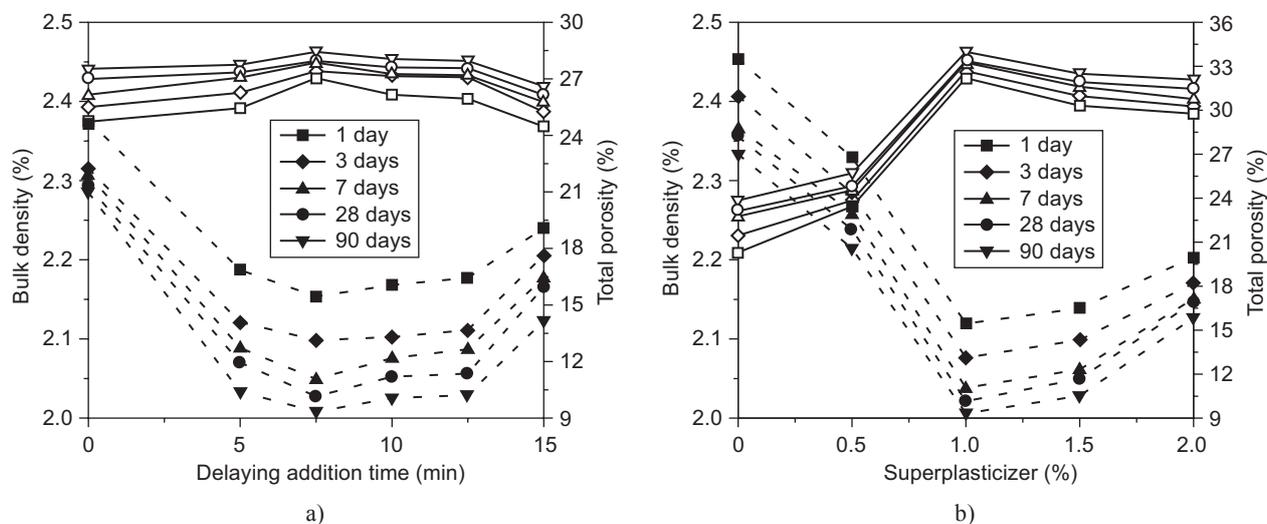


Figure 4. Bulk density and total porosity of cement pastes: a) effect of delayed addition time in the presence of 1.0 mass % superplasticizer for 15 min; b) effect of delaying addition time at 7.5 min in the presence of different dosages of superplasticizer.

curing time and superplasticizer content. As the amount of superplasticizer increases up to 1.0 mass %, the compressive strength increases due to the decrease of water of consistency as well as the total porosity and increase the bulk density. These two factors work together to increase the compressive strength of the cement pastes. Also, superplasticizer increases the pozzolanic activity of silica fume pozzolanic blended cement pastes (10 % silica fume) to form additional CSH, which is the main source of compressive strength. As the dosages of superplasticizer increases up to 2.0 %, the compressive strength decreases due to the retardation effect of superplasticizer [2].

Combined water content

The combined water contents of the cement pastes containing 1.0 % of superplasticizer mixed at simultaneous and delaying addition time of 15.0 min cured for 90 days are given in Figure 6a. The combined water content increases gradually with curing time for 28 days. This is due to the progress of the hydration and the pozzolanic reaction of SF with the liberated lime forming more hydration products. The combined water contents decrease at 90 days due to a release of water during polymerization of silicates [19]. Moreover, the decrease in combined water contents may be related

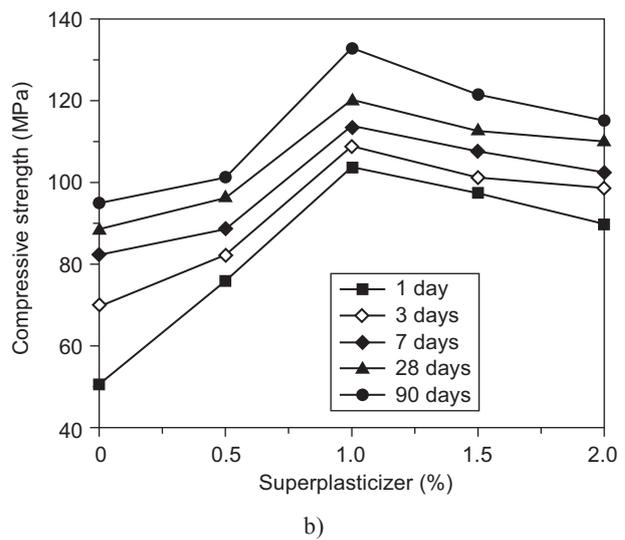
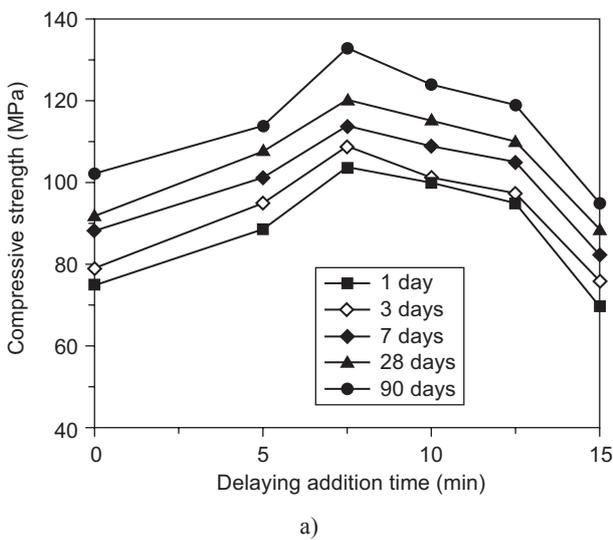


Figure 5. Compressive strength of cement pastes: a) effect of delaying addition time of superplasticized cement pastes with 1.0 % superplasticizer up to 15 min; b) effect of different dosages of superplasticizer delayed addition time at 7.5 min.

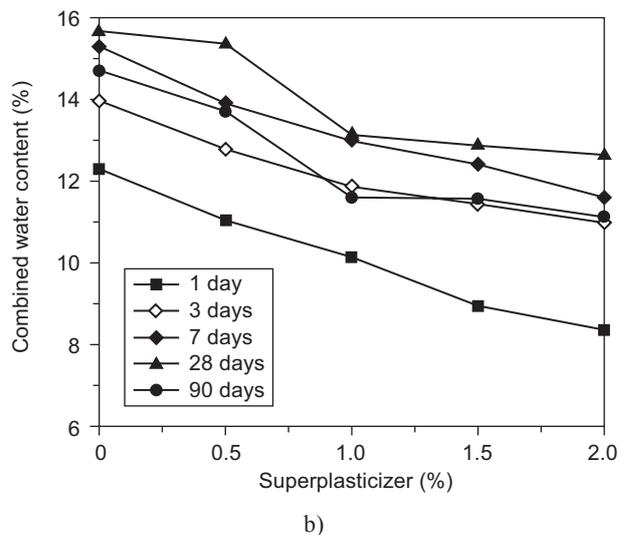
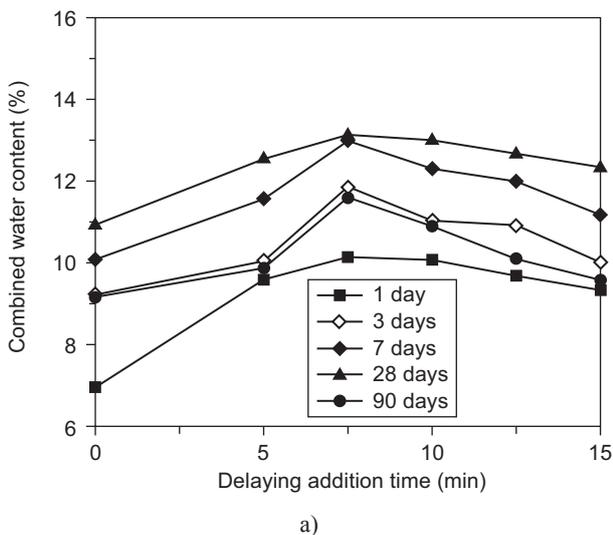


Figure 6. Combined water content of cement pastes: a) effect of delaying addition time of superplasticized cement pastes with 1.0 % superplasticizer up to 15 min; b) effect of different dosages of superplasticizer delaying addition time at 7.5 min.

to the transformation of the initially formed high lime CSH content, to low lime CSH content, which has lower combined water content [20]. As the delaying addition time increases up to 7.5 min the combined water content increases due to the superplasticizing effect which improved with the delaying addition time of superplasticizer because of the decrease in adsorption capacity of SMF on the C_3A hydration products. As the delaying addition time increases up to 15.0 min the combined water content decreases due to the retardation effect on the hydration process. At constant time of hydration and W/C ratio, the free admixture in the interstitial phase, the more retardation of cement paste occurred. This is due to the fact that delaying addition enhances the adsorption of dissolved sulphate ion on the positive sites of C_3A and C_4AF ; this decreases the adsorption of superplasticizer.

The combined water contents of cement pastes admixed with 0.0, 0.5, 1.0, 1.5 and 2.0 mass % superplasticizer at delayed addition time of 7.5 min up to 90 days are illustrated in Figure 6b. The combined water content increases gradually with curing time up to 28 days for all cement pastes due to the progress of hydration and pozzolanic reaction of silica fume with the liberated lime forming more hydration products. The combined water contents decrease at 90 days due to the release of water during polymerization of silicates [19] and the transformation of the initially formed high-lime CSH with higher combined water content, to low-lime CSH [20]. As the amount of superplasticizer increases up to 2.0 mass % the, the chemically combined water content decreases due to the reduction of water of consistency.

Free lime content

The free lime contents of cement pastes containing 1.0 % of superplasticizer mixed at simultaneous and

delaying addition time of 15.0 min for 90 days are given in Figure 7a. The free lime content increases gradually with curing time up to 7 days, then decreases for 90 days. The free lime increases at the early age up to 7 days, due to that the rate of liberation $Ca(OH)_2$ from the hydration of cement phases (C_3S and $\beta-C_2S$) is higher than the rate of consumption by pozzolanic reaction with SF to produce additional CSH. After 7 days up to 90 days the free lime decreases sharply due to the higher pozzolanic activity of SF or the rate of consumption is higher than that of liberation.

Figure 7b represents the free lime contents of cement pastes admixed with 0.0, 0.5, 1.0, 1.5 and 2.0 mass % superplasticizer respectively determined at 7.5 minutes up to 90 days. The free lime contents of all cement pastes increase up to 7 days, then decrease up to 90 days for all dosages. After 7-90 days the free lime decreases sharply due to the higher pozzolanic activity of SF. As the amount of superplasticizer increases to 2.0 mass % the free lime content decreases due to the reduction of mixing water which affects the hydration of cement paste as well as the pozzolanic activity of SF. The reduction of mixing water is due to the coating of the superplasticizer not only on cement grains but also on SF particles, forming double layer on SRC and SF grains; it influences the beginning and degree of pozzolanic reaction. The decrease of mixing water decreases the total porosity, which facilitates the approaching of SF from the lime released as a result of the hydration of cement phases (C_3S and $\beta-C_2S$) to form additional amounts of CSH. Therefore, the addition of SF in combination of superplasticizers tends to form low density, dispersed-hydrated products with low C/S and low $Ca(OH)_2$ content, leading to relatively homogeneous composites [21]. The presence of superplasticizer and SF the microstructure displayed a more dense arrangement of microcrystalline CSH as the main hydration products with minor or nil amounts of $Ca(OH)_2$.

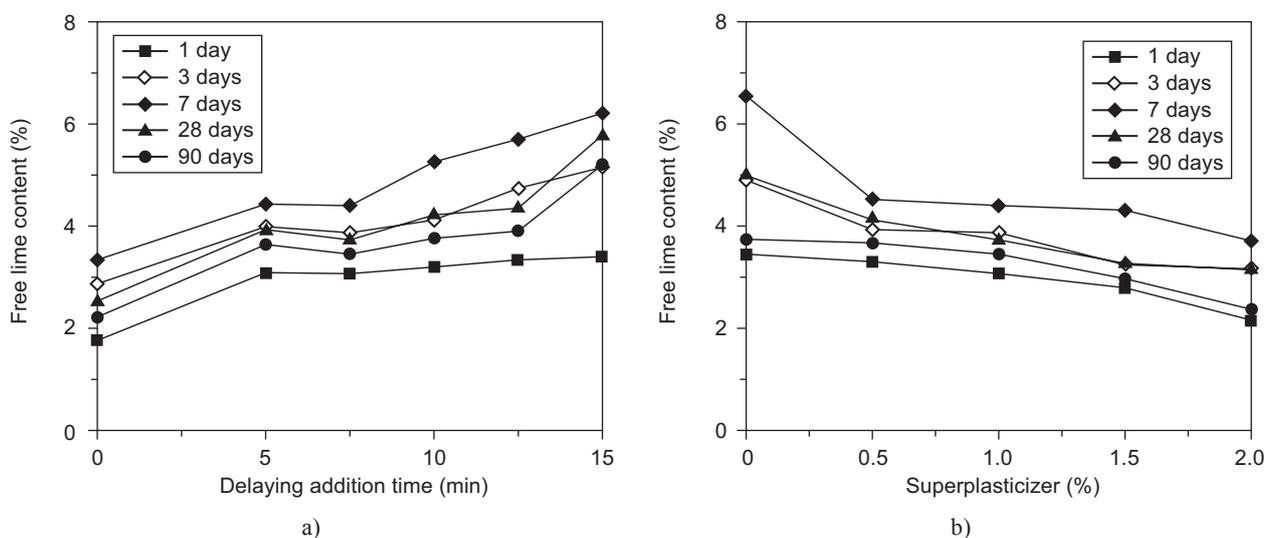


Figure 7. Free lime content of cement pastes: a) effect of delaying addition time of superplasticized cement pastes with 1.0 % superplasticizer up to 15 min; b) effect of different dosages of superplasticizer delayed addition time at 7.5 min.

FT-IR Spectroscopy

FT-IR Spectroscopy was carried out on some selected samples of hardened cement paste. FT-IR spectra of cement pastes admixed with 0.0, 1.0 and 2.0 mass % of superplasticizer with delaying addition time at 7.5 min for 7 days are illustrated in Figure 8. The broad band at 3400 cm^{-1} due to the symmetric stretching frequency of H_2O , that shows higher intensity at 1.0 mass % superplasticizer, but lower intensity at 2.0 mass % due to the retardation effect. This result is in a good agreement with the combined water contents. In addition, the band at 1650 cm^{-1} related to the bending frequency of H_2O indicates the formation of CSH. On the other side, the band appears at 965 cm^{-1} due to the symmetric stretching of bands O–Si–O and O–Al–O has higher intensity at 1.0 mass % superplasticizer due to the formation of hydration products, such as CSH and CAH. The band illustrated at 3799 cm^{-1} represents the free lime [22]. The adsorption band at 1490 cm^{-1} is due to the presence of CO_3^{2-} which increases in the absence of superplasticizer due to the higher content of free lime that can be easily carbonated. The carbonation decreases with the admixture content due to the lower content of CH.

Figure 9 shows the FT-IR spectra of cement pastes admixed with 1.0 and 2.0 mass % of superplasticizer at delayed addition time of 7.5 min for 90 days. The intensity of the band at 3740 cm^{-1} related to CH decreases with SMF due to the pozzolanic activity of SF towards $\text{Ca}(\text{OH})_2$. It is clear that the bands at 3432 and 1648 cm^{-1} as well as 975 cm^{-1} represent the hydration products which show high intensity at 1 % of SMF, but low at 2 % of SMF due to the retardation effect. The band at 1477 cm^{-1} is due to carbonate which decreases with the dose of the superplasticizer and the decrease of $\text{Ca}(\text{OH})_2$.

Figure 10 shows FT-IR spectra of cement pastes admixed with 2.0 mass % of SMF with delayed addition time of 7.5 min cured for 90 days. The very sharp band illustrated at 3799 cm^{-1} represented the free lime content, which decreases with curing time due to the consumption of lime by pozzolanic reaction of SF forming more hydration products. The broad band at 3400 cm^{-1} due to the stretching band of H_2O increases with curing time due to the progress of hydration. In addition, the band at 1650 cm^{-1} is related to the bending of H_2O and indicates the formation of CSH [23]. The absorption band at 1490 cm^{-1} is due to the presence of CO_3^{2-} . On the other hand, the band appears at 965 cm^{-1} due to the symmetric stretching of bands O–Si–O and O–Al–O increases with curing time related to the progress of hydration in the formation of hydration products, such as C–S–H and CAH [22].

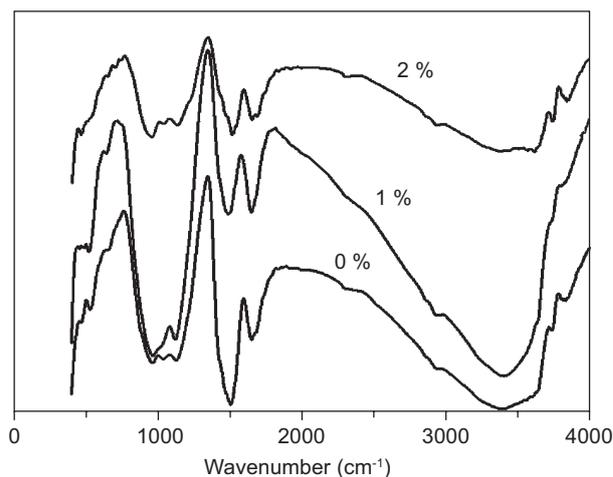


Figure 8. FT-IR spectra of cement pastes in presence of different dosages of superplasticizer determined at 7.5 min for 7 days.

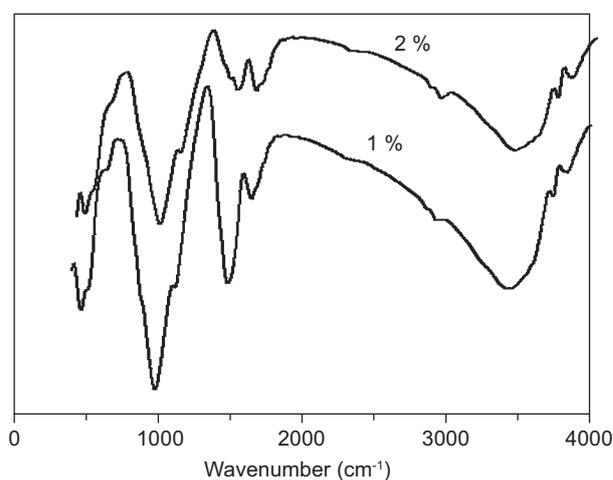


Figure 9. FT-IR spectra of cement pastes in presence of different dosages of superplasticizer determined at 7.5 min for 90 days.

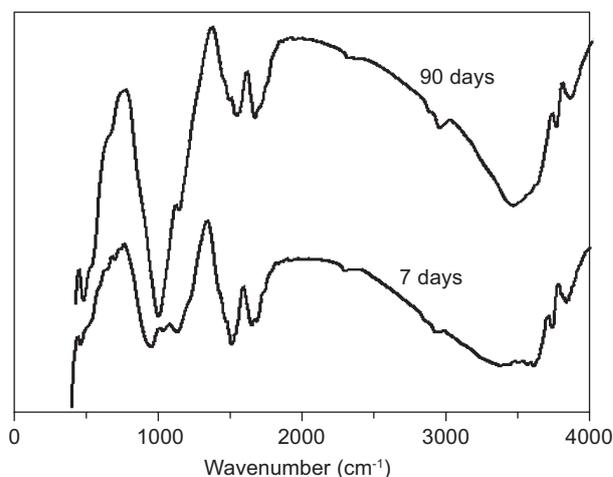


Figure 10. FT-IR spectra cement pastes admixed with 2.0 % superplasticizer determined at 7.5 min as a function of curing time up to 90 days.

CONCLUSIONS

From the above findings it can be concluded that:

- The optimum amount of SMF superplasticizer was chosen as 1.0 mass %.
- The spread area of minislump is larger with delaying addition time than simultaneous addition.
- The optimum delaying addition time of superplasticizer was 7.5 min, which gives the highest fluidity, bulk density, compressive strength and chemically combined water contents of cement pastes
- The delaying addition time of 15.0 min showed the lower values of bulk density, compressive strength and chemically combined water contents. This is attributed to a reduction in the hydration rate of the cement, due to the formation of a polymer film around the cement particles and/or hydrated products.
- The free lime contents of all cement pastes increase up to 7 days with the delaying addition time, then decrease for 90 days. As the amount of superplasticizer increases up to 2.0 mass % (7.5 min) the free lime content decreases.

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