EFFECT OF POLYCARBOXYLATE SUPERPLASTICIZER
ON HYDRATION CHARACTERISTICS OF CEMENT PASTES
CONTAINING SILICA FUME

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This work aims to study the effect of the polycarboxylate superplalsticizer (PCS) on the rheology, electrical conductivity, physico-chemical and mechanical properties of ordinary Portland cement (OPC) pastes containing silica fume (SF). The blended cement pastes were prepared from OPC-SF (90 wt.% OPC + 10 wt.% SF). The results show that addition of PCS to the cement blends decreases their electrical conductivity during the early and later ages up to 28 days. In addition, PCS admixture considerably affects the amount of chemically combined water. Compressive strength of OPC pastes is higher than that of pastes containing SF. However, the addition of 0.75-1.5 wt.% PCS brings higher compressive strength values of OPC-SF pastes than those of OPC cement pastes.

INTRODUCTION

Modern concrete technology projections indicate a great expansion in low-cost and time efficient constructions. Concrete mix designs were likely more concerned on these chemical admixtures. Chemical admixtures have been found beneficial in offsetting some of the undesirable characteristics of concrete used in hot climates [1]. Some advantages of chemical admixtures (superplasticizers) are reducing water/cement ratio; increasing workability retention (retard the initial and final setting), increasing strength and decrease the drying shrinkage as well as the permeability.

Superplasticizers are widely used in the concrete industry; currently used are polynaphthalene sulphonate and polymelamine sulphonate formaldehyde condensates. These two polymers have the same dispersing mechanism. The main polymers chains (sulphonate groups $-SO_3^-$) adsorb onto the surface of cement particles; the cement particles become negatively charged. Consequently, electrostatic repulsion occurs between the cement particles. The cement particle arrangement in a paste matrix offsets the inter-particle attractive forces.

Polycarboxylate superplasticizer increase dispersion because of its superior performance in dispersing cement particles at smaller dosages and retaining concrete slump without prolonging setting times [2]. It is reported that electrostatic repulsion appears to play a minor role in the dispersing mechanism associated with the polycarboxylate superplasticizers, where it is believed that the dominant stabilizing mechanism through steric repulsion. Polycarboxylate polymer contains -COO- groups instead of $-SO_3^-$ groups, in sulphonated condensate.

During the last few decades, the use of both mineral admixtures (fly ash, silica fume, blast-furnace slag, sand) and chemical admixtures (retarders, accelerator, water reducers and superplasticizers) by the construction industry has been increased attention [3-6]. Mineral and chemical admixtures play an important role in changing the physical and chemical properties of concrete matrix.

Silica fume increases strength due to its pozzolanic activity, which leads to decrease the pore size as well as better bond at the aggregate-paste interface [7-10]. The pozzolanic reaction is characterized by many advantages, i.e. slow, low heat of hydration and lime consuming instead of lime producing, forming hydration products fill the capillary pores improving many properties [11].

Silica fume accelerates cement hydration, while superplasticizer retard early cement hydration, depending on the superplasticizer type, amount, and mode of use [12]. This work aimed to investigate the influence of PCS and silica fume on the electrical conductivity, rheology and hydration characteristic of cement pastes.

EXPERIMENTAL

Materials

The ordinary Portland cement (OPC) was provided from Helwan Portland Cement Company; the chemical composition was shown in table 1. The Blaine surface area was $\cong$ 3300 cm^2/g.

Table 1. Chemical composition of starting materials (wt.%).

Oxide	OPC	SF
SiO2	20.50	96.10
Al$_2$O$_3$	5.05	0.52
Fe$_2$O$_3$	2.99	0.70
CaO	62.00	0.21
MgO	2.07	0.48
SO$_3$	2.40	0.10
Na$_2$O	0.48	0.31
K$_2$O	0.09	0.49
L.O.I	3.10	1.14

Condensed silica fume (SF) was supplied from Ferro-Silicon Alloys Company (Edfo-Komombo), Aswan, Egypt. The chemical composition of SF is also shown in table 1, its specific surface area is about $\cong$ 20 m^2/g as measured by nitrogen adsorption with a relative density of 2.2.

The admixture used Complast SP600 provided by FOSROC chemical construction company, Egypt. Complast SP600 based on polycarboxylate/polyether. It was pale amber colored liquid, with a specific gravity of 1.1, chloride content < 0.1 wt.% and sulphate content < 3.0%.

Techniques

The silica fume-pozzolanic cement paste was prepared with partially substituted OPC with 10 wt.% SF. The blend was mixed in a porcelain ball mill with four balls for 6 h to assure complete homogeneity.

The superplasticizer was added to the mixing water. The solid mix of each paste was mixed with a sufficient amount of water to form a paste of standard consistency according to ASTM specifications [13]. The standard water/binder ratios are given in table 2. The pastes were molded into one-inch cubes and cured in a humidity chamber at 23±1°C for 24 hours, then demolded and cured under limewater up to 90 days.

A set of three samples of each mix at any required time were used for compressive strength of the blended cement pastes. The total porosity (ε) was determined by the determination of bulk density (d_p), the evaporable (W_e) and total water (W_t) contents of the hardened cement pastes according to the following equation [15]:

$$\varepsilon = \frac{0.99 W_e * d_p}{1 + W_t}$$

The total water W_t at any hydration period was calculated on the ignited mass basis by the ignition of two samples from the fresh paste at 1000°C for one hour soaking time.

The hydration reaction was stopped by pulverizing a represented sample 10 g at each hydration period, which placed into a beaker containing 1:1 methanol-acetone mixture, and then mechanically stirred for one hour [14]. The mixture was filtered through a gooch crucible, G_4, and washed with ether. The solid was then dried at 70°C for 30 minutes to complete evaporation of alcohol. The dried sample was kept into airtight bottles until the desired time of analysis.

The combined water content was calculated on the ignited weight basis. Approximately 2 g of the predried sample were gradually ignited up to 1000°C for 1hour soaking time. The results of combined water were corrected for the free lime present in each sample.

The determination of Ca(OH)$_2$ is based on extraction of Ca^{2+} by ethylene glycol method according to Egyptian Specification 1994.

Electrical conductivity measurements

In electrical conductivity measurements, the test cell was of the co-axial type, which included concentric inner and outer electrodes mounted on an insulated base plate [16-19]. The electrodes were polished before the experiments. The cement paste was placed in the space between the electrodes and the cell was kept in a dissecator at 100% relative humidity and 20 ±2°C during the test period up to 28 days. The electrodes were connected with RLC meter; model SR 720, operating at 1 kHz for resistance measurements.

Rheological measurements

Two mixes were made of OPC cement pastes and OPC containing 10 wt.% SF (blended cement) admixed with different dosages of the PCS at a constant W/C ratio of 0.30. The mixing was done at a speed of 120 rpm for 3 minutes continuously. Exactly 50 gm of each mix was transport to the Rheotest cell as described in an earlier publication [18], the ratio of radii of measuring tube and measuring cylinder (R/r) was 1.24. The test begins exactly after 6.5 min from contact of cement and water including the mixing time. The shear rate was measured were obtained using a Rheotest 2.1 in the range from 3 to 146 s^{-1}.

RESULTS AND DISCUSSION

Water reduction

Condensed silica fume is a finely powdered material, which increases the water required for a given degree of workability. The increase of water/cement ratio is due to the high surface area of SF (table 2). Addition of SF leads to an increase in water/cement ratio from 0.24 to 0.275. The use of PCS reduces the water of mixing necessary for required water of standard consistency as shown in table 2. The water reduction ranged between 12.50-31.25 % and 14.81-38.18 % for samples containing OPC and OPC+10 wt.% SF, respectively. The water reduction in samples containing silica fume is higher than that of the plain OPC paste. The reduction in required water arises from the dispersion mechanism of PCS not only on the cement grains but also on SF particles. This may be due to the formation of a double layer around the anhydrous cement grains or cement hydration products and, as a consequence, the particles become electrically charged and repel each other. This layer influences both the start and the degree of the pozzolanic reaction (20-23).

Table 2. Water/cement ratio and water reduction.

PCS (%)	Water/cement ratio		Water reduction (%)	
	OPC	90 wt.%OPC +10 wt.% SF	OPC	90 wt.% OPC +10 wt.% SF
0.00	0.240	0.275	----	----
0.50	0.210	0.230	12.5	14.81
0.75	0.190	0.200	20.8	27.27
1.00	0.175	0.180	27.08	34.55
1.50	0.165	0.170	31.25	38.18

Rheology

PCS is an anionic surfactant, when adsorbed on cement particles become negatively charged causing a repulsive effect with each other, consequently its fluidity increases; in addition, the side chains preserve water molecules in contact via hydrogen bonding creating osmotic pressure which increases the movement of these cement particles.

Figure 1 shows the shear stress-shear rate relationship of cement pastes made with and without 10 wt.% SF admixed with different dosages of PCS. It is clear that, addition of PCS decreases the shear stress values.

The yield values are the minimum shear stresses at zero shear rate, indicate the minimum forces, needed for pastes movements. The yield values were calculated from Bingham's model as shown in table 3. The yield values of superplasticized cement pastes decrease with PCS content. PCS increases the fluidity of cement pastes. The yield values range between 1.3-0.21 and 4.38-0.33 Pa for OPC and OPC+10 wt.% SF.

PCS improves the fluidity of cement pastes by the dispersion of cement particles. The adsorption of PCS superplasticizer molecules on the cement particles hinders their flocculation as a result of the electrostatic repulsion forces and/or through steric hindrance. Consequently, the particles are homogenously distributed in the aqueous solution, minimizing the amount of water needed for then to be dispersed, which leads to the higher fluidity and workability of cement pastes [24, 25]. The yield values decrease with the increase of PCS dosages. Silica fume-pozzolanic cement pastes have higher yield values than those of OPC pastes.

Table 3. Yield values.

Cement type	Dosage of admixture (%)	Yield value (Pa)
OPC	0.00%	1.30
	0.50%	0.36
	0.75%	0.35
	1.00%	0.30
	1.50%	0.21
90 wt.% OPC+10 wt.% SF	0.00%	4.38
	0.50%	0.71
	0.75%	0.56
	1.00%	0.52
	1.50%	0.33

Initial setting time

Figure 2 shows the effect of PCS on initial setting time of cement pastes. The initial setting times of cement pastes were measured from the electrical conductivity-time curves [16]. The partial replacement of OPC by SF prolonged the initial setting time by 13 % than control sample (OPC). Evidently, the addition of PCS to both OPC and OPC+10 wt.% SF pastes cured at 20 C extended the initial setting time.

PCS forms a complex with Ca^{2+} ions liberated on the surface of C–S–H gel or $Ca(OH)_2$ crystals; the interweaved net structure consists of ion bonded large molecular system bridged by means of $Ca(OH)_2$ [26,27]. In the presence of PCS, it appears that only a few number of Ca^{2+} ions go into the solution and do not becomes supersaturated with respect to $Ca(OH)_2$, i.e. PCS inhibits the growth of hydrates, leading to retard of the setting [28].

It is also clear that the initial setting time of OPC paste extended as the dosage of PCS increases; however, the initial setting time of silica fume pozzolanic cement pastes increases by 40 % as the PCS content increases up to 0.75 %. Setting times decrease by 5 % at higher dosages of PCS.
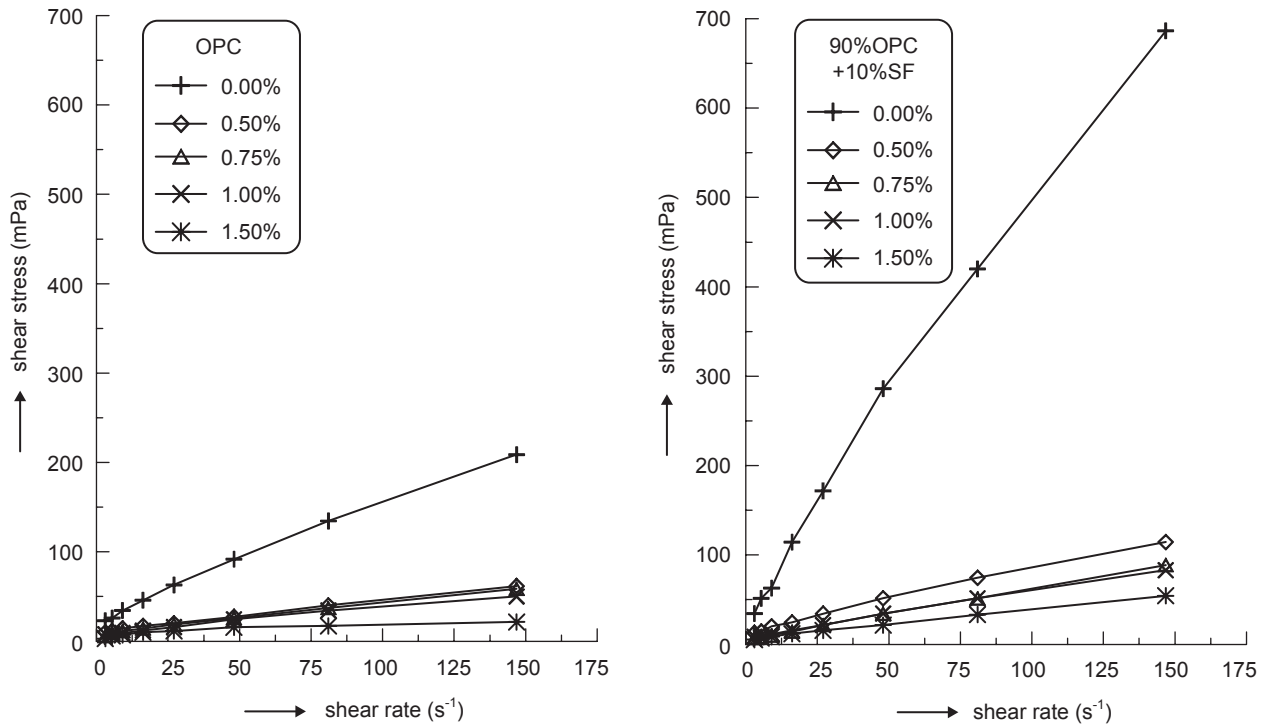
Figure 1. Shear stress as a function of shear rate of cement pastes made with and without SF (10 wt.%) in presence of different dosages of PCS after 15 minutes.
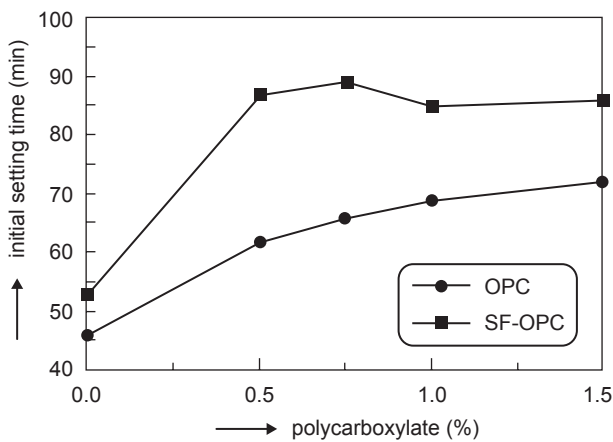

Figure 2. Effect of polycarboxylate superplasticizer on initial setting time.

Electrical conductivity

When cement is placed in contact with water, a rapid dissolution of ionic species into the liquid phase takes place. Alkali sulphates are dissolved completely contributing K^+, Na^+ and SO_4^{2-} ions. Calcium sulphate dissolves until saturation, thus contributing Ca^{2+} and SO_4^{2-} ions. Tricalcium silicate dissolves and a layer of a C–S–H precipitates at the surface of cement particle. Tricalcium aluminate dissolves and reacts with Ca^{2+} and SO_4^{2-} present in the liquid phase, producing ettringite.

Figure 3 shows the variations in the electrical conductivity of OPC and OPC + 10 wt.% SF with curing time. Initially the electrical conductivity increases as a result of the hydration of cement constituents; the charge carriers are K^+, Na^+, Ca^{2+}, OH^- and SO_4^{2-}. These ions are responsible for initial increase in electrical conductivity values. These ions are readily adsorbed by the formation of a thin layer of hydration products (C–S–H and ettringite). The hydration products form thin insulating layers around the hydrated cement grains. These layers consist of electrical double layers of adsorbed K^+, Na^+, Ca^{2+} ions lead to a decrease in both of the number and mobility of these ions, and consequently the electrical conductivity decreases [15,16]. As the curing time increases the electrical conductivity decreases due to the formation of an insulating electrical double layer around the cement particles.

Alternatively, one could hypothesize the formation of protective layers around ettringite grains, which when disturbed by osmotic pressure release sulphate and other ions into the environment in a transient way. On progressive hydration, the notable consumption of the number of ions (Ca^{2+}, Na^+ and K^+ ions), a result of the formation and later accumulation of hydration products, is responsible for the sharp decrease in the electrical conductivity values of the hardened cement pastes.

In the presence of PCS, the retardation action takes place; this leads to shifting and broadening of the peaks of electrical conductivity to longer hydration time. The

transformation of ettringite to monosulphate was delayed or even absent in the presence of SF containing cement pastes. This may be attributed to formation of a protective film around the ettringite particles. OPC + 10 wt.% SF suffers a sharp decrease in the electrical conductivity during the different hydration periods. The decrease in the electrical conductivity is mainly attributed to the decrease in the number of released ions during the hydration of OPC as its proportion decreases in the mixes, decrease of liberated free $Ca(OH)_2$ released during the hydration of OPC as a result of SF, and the interaction of SF with the $Ca(OH)_2$ released leading to the formation of C–S–H. The pozzolanic reaction not only decreases the quantity of $Ca(OH)_2$, but also decreases the volume of large pores and increases the fraction of narrow pores and then reduces the number of continuous pores in cement paste. SF consists of fine particles, which can fill the spaces between cement particles. As a result of these factors the electrical conductivity of blended cement pastes is lower than those of Portland cement pastes at early and especially at later ages.

McCarter *et. al.*, [17] studied the electrical response of cements during hydration to evaluate the microstructure of cement materials. The application of electrical resistance measurements has been widely studied by many authors as a useful method for monitoring the initial hydration of OPC [29]. Thus, proposed a method for evaluating the pozzolanic activity by measuring the electrical resistance of cement pastes containing pozzolana. In view of the increasing use of pozzolanic materials in hydraulic binders, there is great interest in designing a rapid and efficient method to evaluate their reactivity. Electrical measurements as a function of frequency are an experimental approach, which is used to characterize the response of ceramic materials, such as ionic conductors and semiconductors. One of the methods reported in the literature is based on pore structure and changes in ionic concentrations within the pore fluid [30].

Figure 4 illustrates the effect of curing ages and PCS dosages on electrical conductivity of cement pastes cured up to 28 days. It is clear that, the electrical conductivity of OPC and SF-cement pastes decreases with curing time and PCS dosage. The lower conductivity corresponding to a greater dosage of PCS may be attributed to the lower water/binder ratio. The electrical conductivity of OPC pastes at 1-day exhibited a higher value than that of OPC-SF cement pastes. SF-cement pastes undergo a sharp decrease in electrical conductivity during the period from 1 day up to 28 days. This is due to the pozzolanic reaction of SF with the liberated free $Ca(OH)_2$ leading to the formation of additional C–S–H. Evidently, as the PCS dosages increase, the electrical conductivity decreases. The results of electrical conductivity are confirmed with the results of compressive strength and total porosity as indicated later in this study.
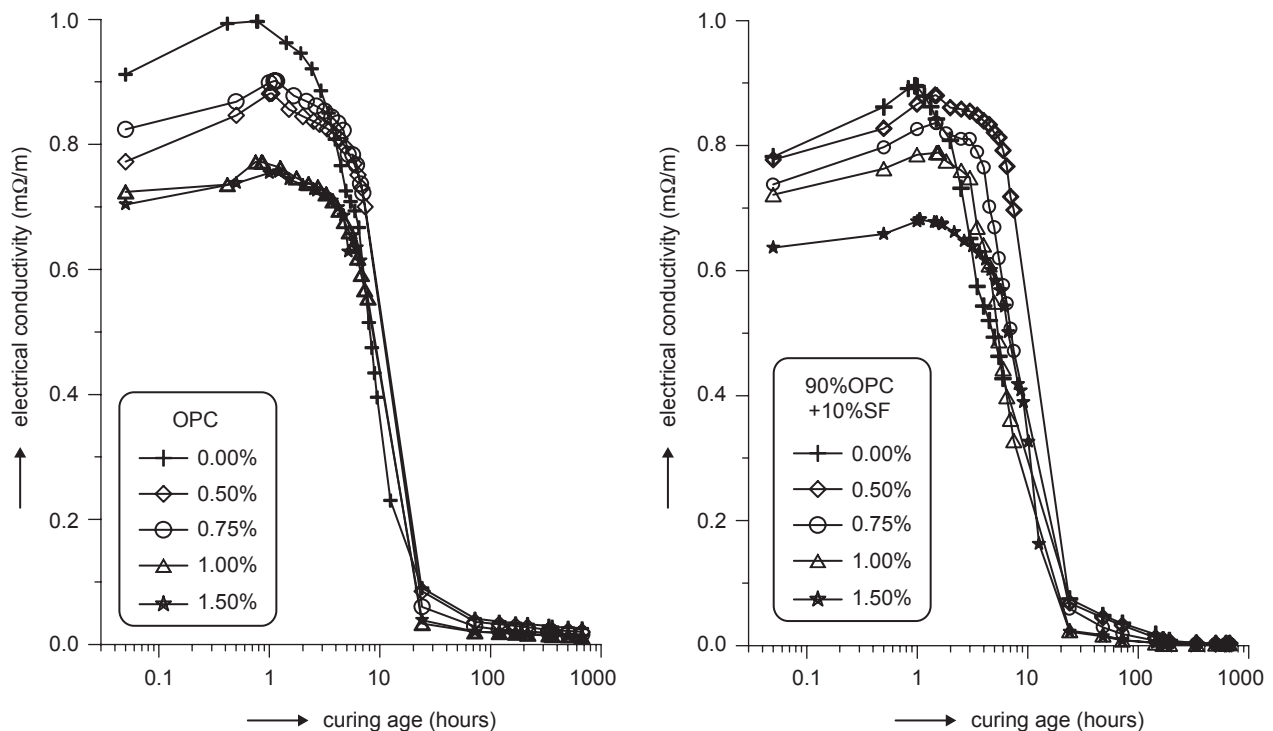

Figure 3. Electrical conductivity of OPC and 10 wt.% SF-OPC in presence of different dosages of PCS from 3 minutes after mixing up to 672 hours (28 days).
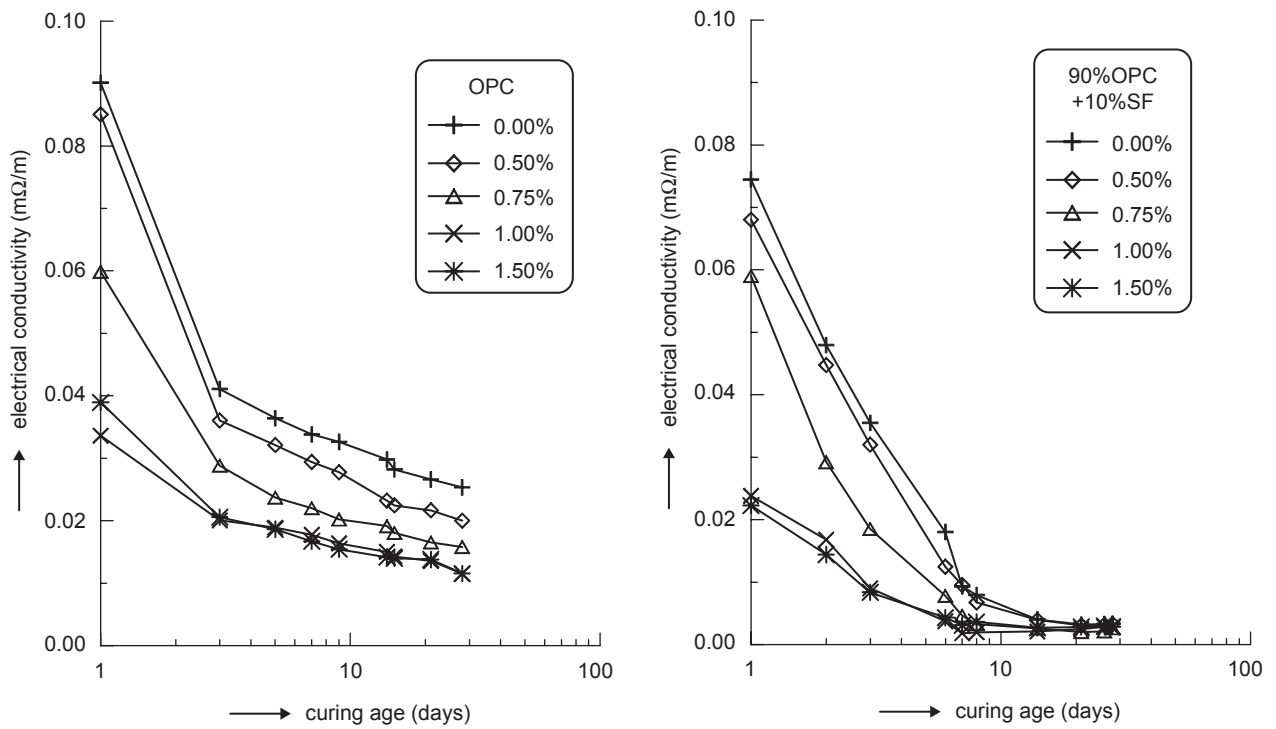
Figure 4. Electrical conductivity of OPC and 10 wt.% SF-OPC in presence of different dosages of PCS cured up to 28 days.
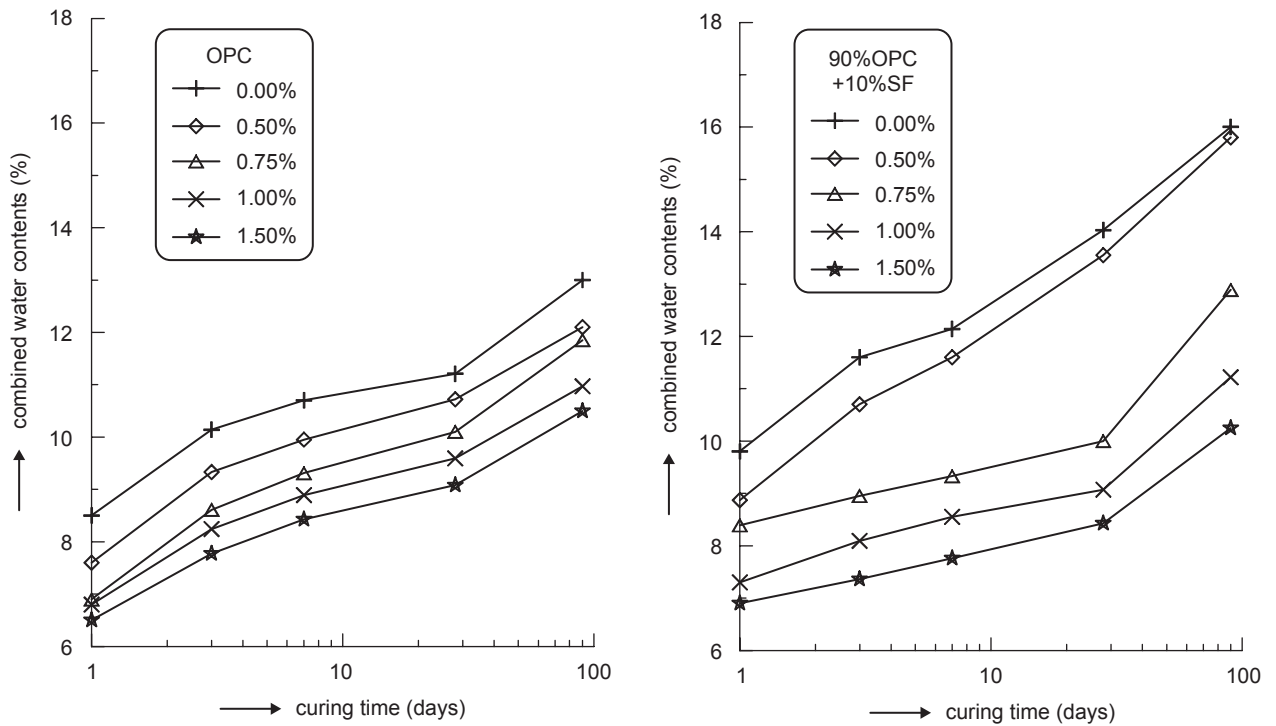

Figure 5. Combined water content of cement pastes mixed with and without SF as a function of curing time up to 90 days.

Combined water contents

The combined water contents of hydrated cement pastes containing OPC and OPC containing 10 wt.% SF are graphically plotted as a function of curing time in figure 5. The results show that the combined water content increases with the increase of curing time. Also, addition of SF increases the chemically combined water content, due to the increase in mixing water [31]. This leads to an increase in the formation of additional amounts of calcium silicate hydrates as a result of pozzolanic reaction. As the dosage of PCS increases, the chemically combined water content decreases. Increase of PCS dosages from 0.75-1.50 % results in a marked reduction in chemically combined water. Increase PCS dosage decreases the required water of standard consistency. SF increases the combined water content, this may be attributed to the polymerization of the silicates; it was claimed that the average chain length of poly-silicates in calcium silicate hydrate gel increases with SF content. This is also attributed to the reactivity of SF to react with lime forming additional C–S–H.

Free lime contents

The free lime contents of hydrated OPC and blended cement paste containing 10 wt.% SF in presence and absence of PCS are plotted in figure 6. The results indicate that free lime content of OPC pastes increases with curing time, whereas it decreases in presence of SF. The increase of free lime content of OPC pastes with curing time is mainly due to the continuous hydration of the main cement phases, namely, β-C_2S and C_3S with the liberation of $Ca(OH)_2$. The amount of free lime decreases with SF as well as PCS. This may be due to reduction of mixing water, which affects the hydration of cement paste. The decrease in mixing water leads to a decrease the initial porosity. The reduction of the initial porosity affects on the efficiency of pozzolanic reaction via approaching the grains of SF from grains liberated lime from OPC hydration forming additional amounts of calcium silicates hydrates. The deposition of additional hydrates in the available pores leads to the decrease the porosity.

Compressive strength

The compressive strength of OPC and OPC+ 10 wt.% SF cement pastes was illustrated as a function of dosages of PCS and curing age in figure 7. The results show that the compressive strength of all cement pastes increases with curing time. As the dosage of PCS increases, the compressive strength of all samples increases. This is mainly due to the decrease of mixing water, which leads to decrease of the total porosity that affects positively the compressive strength of the cement pastes.
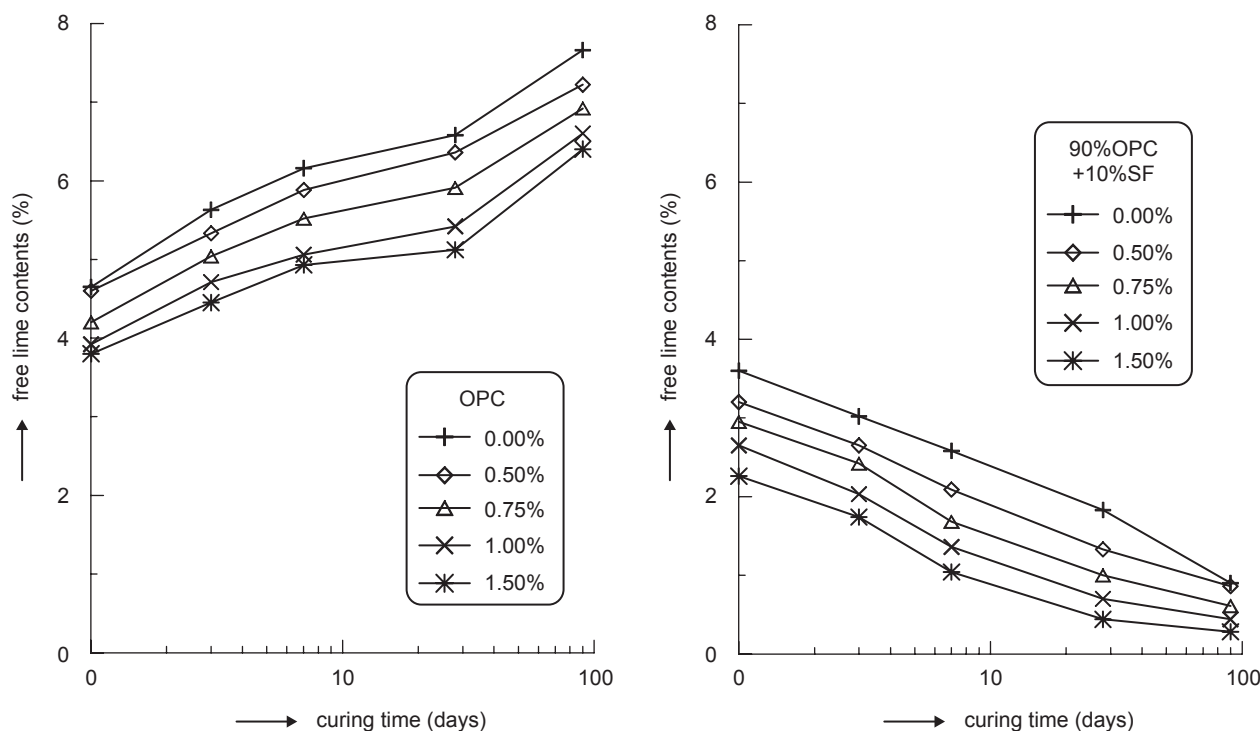

Figure 6. Free lime contents of OPC and 10 wt.% SF-OPC as a function of curing time up to 90 days.
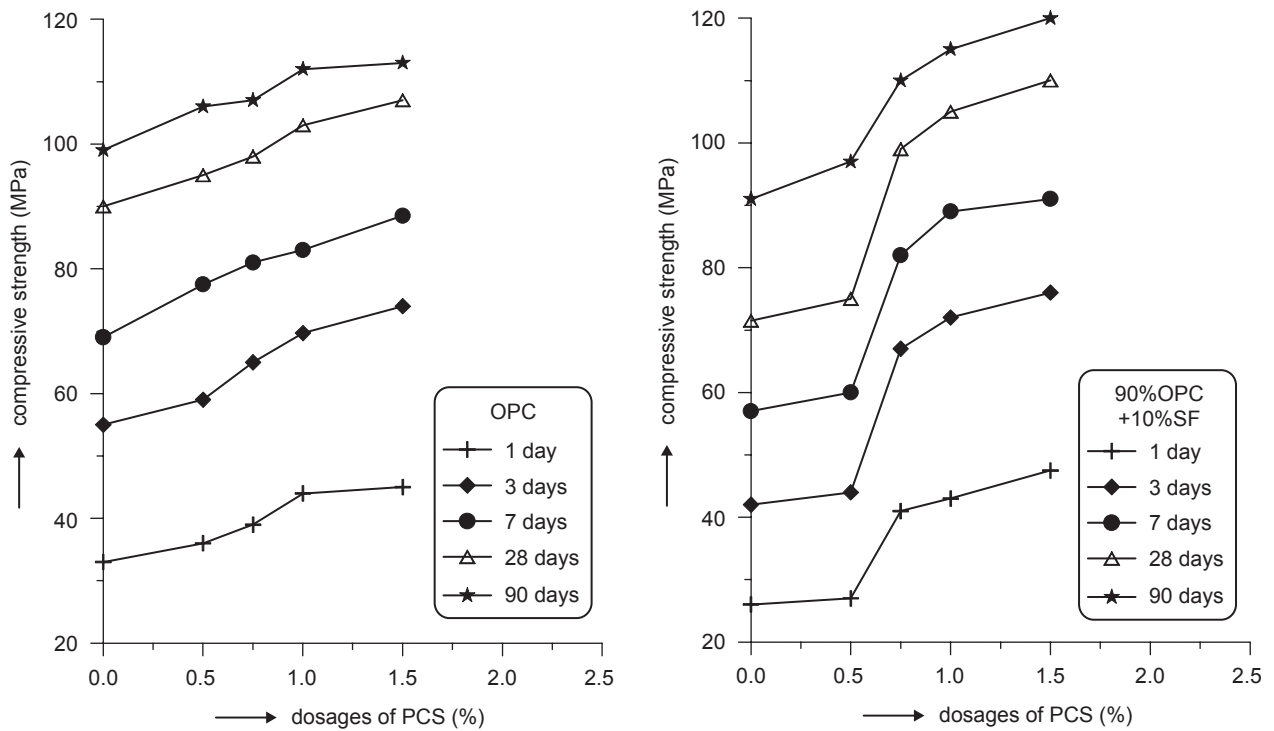
Figure 7. Compressive strength of cement pastes with and without SF (10 wt.%) in presence of different dosages of PCS cured up to 90 days.
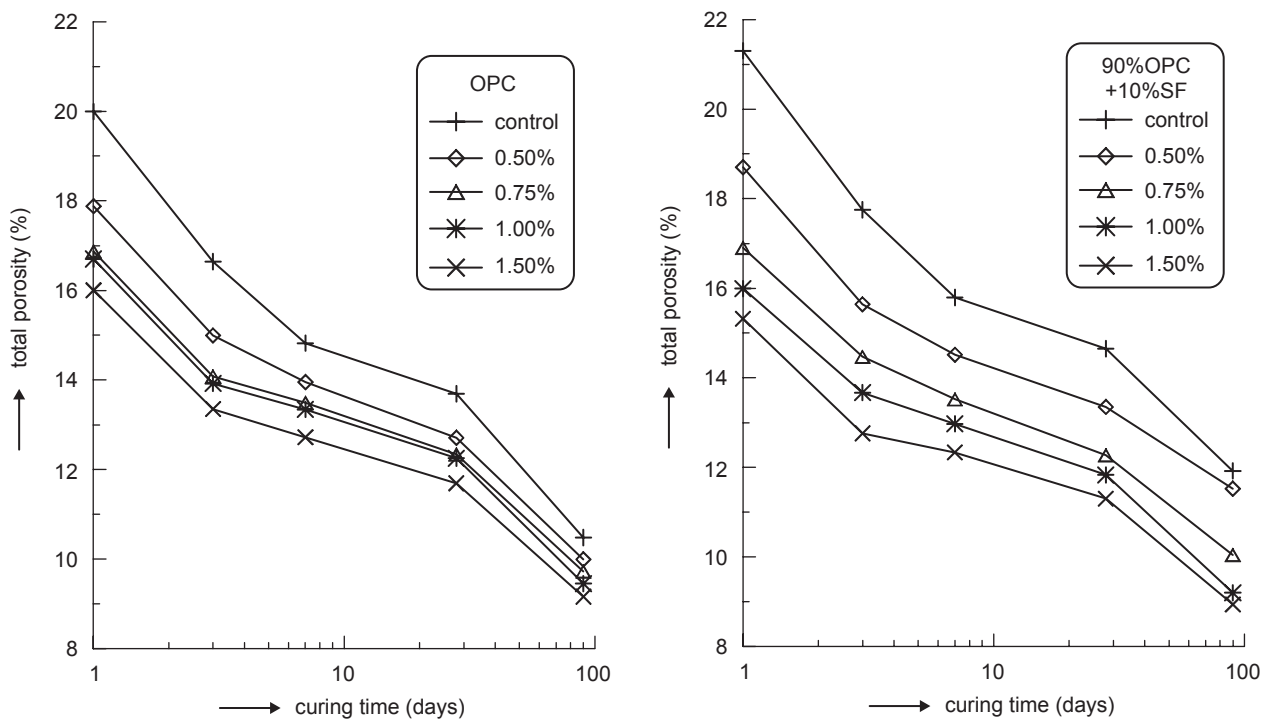

Figure 8. Variations of total porosity of cement pastes with and without silica fume (10 wt.%) as a function of PCS dosages cured up to 90 days.

Compressive strength values of plain OPC are higher than those of OPC+10 wt.% SF cement pastes free of PCS, whereas in the presence of 0.75-1.50 % of PCS, the compressive strength values of OPC+10 wt.% SF pastes are higher than those of OPC pastes. The results of compressive strength are in agreement with the results of electrical conductivity and porosity data previously shown [32].

Porosity

Figure 8 shows the variation in total porosity of cement pastes admixed with PCS. The porosity of cement pastes depended on cement type, water/cement ratio, degree of hydration and type of admixture. The total porosity decreases with curing time due to filling up a part of the available pores volume between the cement particles with the hydration products. Addition of PCS increases the workability and improves the degree of compaction [33]. The decrease of total porosity is attributed to the decreased water/cement ratio [34]. The increase of the dosages of PCS causes reduction of total porosity. As the porosity decreases, the compressive strength increase and electrical conductivity decreases.

PCS adsorbed preferentially onto C_3A and early formed hydration products prevents early hydration and modifies the microstructure of the hydration phases. Fluidification of cement pastes depends on the type and dosages of the admixture as well as the particle size distribution and mineral composition of cement. SF gradually disappear into the matrix gel and reacts with $Ca(OH)_2$ to form calcium silicate hydrate which is precipitated in the pores. This is strongly reflected by the decrease in the total porosity with the increase in total contents of binding centers of very high strength concrete undergoing progressive hydration and leading to an enhancement in the compressive strength [34,35].

CONCLUSION

The following conclusions can be drawn from the present study:
1. The partial replacement of OPC by 10 wt.% SF prolonged the initial setting time by 13% than OPC pastes. PCS extended the initial setting time of both OPC and OPC+10 wt.% SF pastes.
2. The electrical conductivity of OPC pastes at 1 day exhibited a higher value than OPC-SF cement pastes. Evidently, as the polymer dosages increase, the electrical conductivity decreases.
3. In the presence of SF, chemically combined water increases, whereas free lime content decreases.
4. Compressive strength values of OPC are higher than OPC + 10 wt.% SF cement pastes in absence of PCS, whereas in presence of 0.75-1.5 wt.% PCS the compressive strength values of OPC+10 wt.% SF are higher than those of OPC pastes.

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ÚČINEK POLYKARBOXYLÁTOVÉHO
SUPERPLASTIFIKÁTORU (PCS) NA HYDRATAČNÍ
CHARAKTERISTIKY CEMENTOVÝCH PAST
OBSAHUJÍCÍCH KŘEMIČITÝ ÚLET

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Tato práce studuje účinek polykarboxylátového superplasticizátoru (PCS) na reologii, elektrickou vodivost, fyzikálně chemické a mechanické vlastnosti past portlandského cementu (OPC) obsahujících křemenný úlet (SF). Směsné cementové pasty byly připraveny ze směsi 90 hmot.% OPC + 10 hmot.% SF. Výsledky ukazují, že přídavek PCS snižuje elektrickou vodivost cementové pasty v počátečních i pozdějších stádiiích hydratace až do 28 dní. PCS rovněž podstatně ovlivňuje množství chemicky vázané vody. Pevnosti v tlaku past obsahujících OPC jsou vyšší než u past s přídavkem SF. Přídavek 0,75-1,5 hmot.% PCS však přináší vyšší hodnoty pevnosti v tlaku než u samotných OPC cementových past.