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Effects of silica fume, superplasticizer dosage and type of superplasticizer on the properties of normal and self-compacting concrete

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Abstract. In the present study, a special attention has been paid to the effects regarding the use of different superplasticizers in different dosages. To do so, 36 mixes of normal and self-compacting concrete with two water/binder ratios of 0.35 and 0.45, four different types of superplasticizer including melamine-formaldehyde, naphthalene-formaldehyde, carboxylic-ether and poly-carboxylate, four different superplasticizer/cement ratios of 0.4%, 0.8%, 1.2% and 1.6% and two silica fume/cement ratios of 0% and 10% have been cast. Moreover, the initial and final setting time of the pastes have been tested. For self-compacting mixes, flow time, slump flow, V-funnel, J-ring and L-box tests have been carried out as well as testing the compressive strength and rupture modulus. For normal concrete mixes, slump test has been conducted to assess the workability of the mix and then for each mix, the compressive strength and rupture modulus have been determined. The results indicate that in addition to the important role of superplasticizer type and dosage on fresh state properties of concrete, these parameters as well as the use of silica fume could affect the hardened state properties of the mixes. For instance, the mixes whose superplasticizer were poly-carboxylic-ether based showed better compressive and tensile strength than other mixes. Besides, the air contents showed robust dependency to the type of the superplasticizer. However, the use of silica fume decreased the air contents of the mixes.

Keywords: superplasticizer type; superplasticizer dosage; silica fume; self-compacting concrete, normal concrete

1. Introduction

In recent decades, the use of admixtures and additives has become an inseparable part in mix design of different types of concrete. The reason behind this situation could be originated from the complexity of new structures such as congested reinforcing. Among the admixtures, silica fume (SF) and superplasticizer (SP) are of great importance due to their high amount of usage. Comprehensive studies have been conducted by researchers for both the SF and SP (Akhlaghi *et al*. 2017, Bani Ardalan *et al*. 2017, Mazloom, Allahabadi *et al*. 2017, Mazloom and Miri 2017).

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For instance, researchers introduced a new approach for application of silica fume in concrete (Motahari Karein *et al*. 2017). They produced silica fume granules mixed with a solid superplasticizer and tested the durability and mechanical properties of mixed concrete. However, they observed similar properties for granular SF to slurry SF. Mazloom and his co-workers conducted an experimental survey to observe how the interaction of magnetic water, SF and SP could lead to a concrete with better fresh and hardened results (Mazloom and Miri 2017). In another research, effect of silica fume and polyepoxide-based polymer were investigated on mechanical and durability properties of self-compacting lightweight concrete (Mazloom, Allahabadi *et al*. 2017). They showed that the use of polymeric contents and SF both could enhance the mechanical and durability properties of the mentioned type of concrete. However, the optimum contribution for quaternary mixes was observed to be 10% for SF and polymeric contents. Some researchers looked deeper inside the microstructure of pastes to see how SF changes the composition of C-S-H in pastes. In this regard, in a study conducted by (Rossen *et al*. 2015), new results have been reported showing that the addition of SF changes the development of the microstructure of pastes. For example, portlandite (CH) participates as platelets and even around clinker grains as 'CH rims' and the SF consumed it by pozzolanic reaction. Some researchers put their emphasis on the effects regarding the use of SF on fresh state behavior including yield stress and plastic viscosity of silica fume modified concrete (Lu *et al*. 2015). In fact, they used Bingham's model to describe the rheology of the mix and reported that the SF strongly affected the plastic viscosity and yield stress. They further developed a correlation between slump flow and rheological properties of self-compacting concrete (SCC) with SF. In another study conducted by (Baldino *et al*. 2014), Influences regarding the use of zeolite, limestone and SF were investigated to see how they affect the rheological behavior of fresh cement pastes. To do so, they adopted small amplitude oscillations and conducted time sweep and frequency sweep tests to find out the structure development differences among the mentioned additives. They reported that the synthetic zeolite is better in enhancing the mechanical strength and fluidity of the cement paste than the mentioned additives (Baldino *et al*. 2014).

Superplasticizers are the other mostly used admixtures in the concrete industry. Numerous studies have pointed out the use of this important kind of admixture in the literature (Brooks, Megat Johari *et al*. 2000, Mazloom, Ramezanianpour *et al*. 2004, Mazloom 2008, Huang *et al*. 2016, Kanema *et al*. 2016, Karamloo, Mazloom *et al*. 2016a, Karamloo, Mazloom *et al*. 2016b, Akhlaghi *et al*. 2017, Karamloo, Mazloom *et al*. 2017, Mazloom, Allahabadi *et al*. 2017, Mazloom and Mahboubi 2017, Mazloom and Miri 2017, Msinjili *et al*. 2017, Roudak, Shayanfar *et al*. 2017a, Roudak, Shayanfar *et al*. 2017b, Zou *et al*. 2017, Feng *et al*. 2018, Kim *et al*. 2018, Ma *et al*. 2018, Mangane *et al*. 2018). For instance, Yousuf and his co-workers benefited from using the electrical resistivity measurement method and evaluated the influence of a superplasticizer on the hydration of varying composition cements (Yousuf *et al*. 2017). Their results introduced the electrical resistivity measurement by a non-contact method as a sensitive monitoring technique for cement hydration and microstructure development. They reported that superplasticizer has lowered the rates of electrical resistivity development. This finding was attributed to the retardation caused by chemical. The effect of poly-carboxylic ether based superplasticizer on zeta potential of particles, spread flow, kinetics of hydration, autogenous and chemical shrinkage and setting time of ultra-high-performance concrete has been investigated in a comprehensive research conducted by (Li *et al*. 2017). They reported that the dispersing ability of the SP is related to its chemical structure. In the poly-carboxylic-ether type of SP, an exponential correlation between the SP dosage and flowability of the paste has been observed (Li *et al*. 2017). They further reported that both the absorbed SP and remained SP contributed to retardation of hydration. Based on the mentioned research, the SP dosage had a prominent effect on autogenous shrinkage of samples. Recently, some researchers focused on modifying the chemical structure of SPs. In this regard, Ma *et al*. conducted an experimental survey in which the effect of hydroxypropyl-methyl cellulose ether on the rheology of cement paste plasticized by poly-carboxylate-based superplasticizer has been investigated (Ma *et al*. 2018). They claimed that the hydroxypropyl-methyl cellulose ether could enhance the bleeding and segregation, however, it could lead to some negative effects on dispersion of poly-carboxylate-based SP. By using the total organic carbon analyzer, X-ray photoelectron spectrometer and dynamic light scattering, they showed that the addition of the mentioned chemical admixture could obviously increase the plastic viscosity and yield stress of the plasticized cement paste. Apart from the mentioned in-depth studies conducted investigating the effects of SP and SF, there is still a simple question to be answered. How the type and dosage of superplasticizer affect the fresh and hardened state properties of concrete either in the presence of SF or in its absence. As stated in this section, many studies have been conducted around the world in order to investigate the effect of superplasticizer and silica fume on the properties of concrete. However, to the authors' knowledge, studies regarding the interactive effects of superplasticizer type, dosage and silica fume in both type of SCC and NC, are lacking. Hence, In the present study, two series of experiments have been conducted, the first was allocated to consider the effect of the dosage of SP on fresh and hardened properties of SCC and the second was allocated to investigate the effects regarding the type of SP in the context of normal concrete (NC). In the former, sixteen mixes have been designed in which four different amount of SP (0.4%, 0.8%, 1.2% and 1.6%) and two amounts of SF (0% and 10%) were tested. In addition, two water/binder ratios of 0.35 and 0.45 have been considered. In the latter, sixteen mixes have been cast in which four different types of SP (Naphthalene-formaldehyde-based, Melamine-formaldehyde-based, Carboxylic-ether-based and poly-carboxylate-based) have been considered.

2. Materials

As mentioned in the previous section in the present study, two different series of experiments have been conducted separately to investigate the effects of SP dosage as well as its type. Series A is allocated to SCC mix compositions. In series A, the effects of SP dosage on air content, setting time, slump flow, J-ring, V-funnel, L-box, U-box, 7-day compressive strength, 14-day compressive strength, 28-day compressive strength,90-day compressive strength, 7-day rupture modulus, 14-day rupture modulus, 28-day rupture modulus and 90-day rupture modulus have been considered. In series B, NC mixes, made of four different types of SP with two different water/binder ratios have been tested. The variation of air contents, setting time, slump, 7-day compressive strength, 14-day compressive strength, 28-day compressive strength, 90-day compressive strength, 7-day rupture modulus, 14-day rupture modulus, 28-day rupture modulus and 90-day rupture modulus has been considered in accordance with the type of SP. In the following sections, detailed explanations have been presented for each series.

2.1 Mixes and constituents of series A

2.1.1 Constituents

Aggregates are of the most consumed constituents in mix composition of normal weight

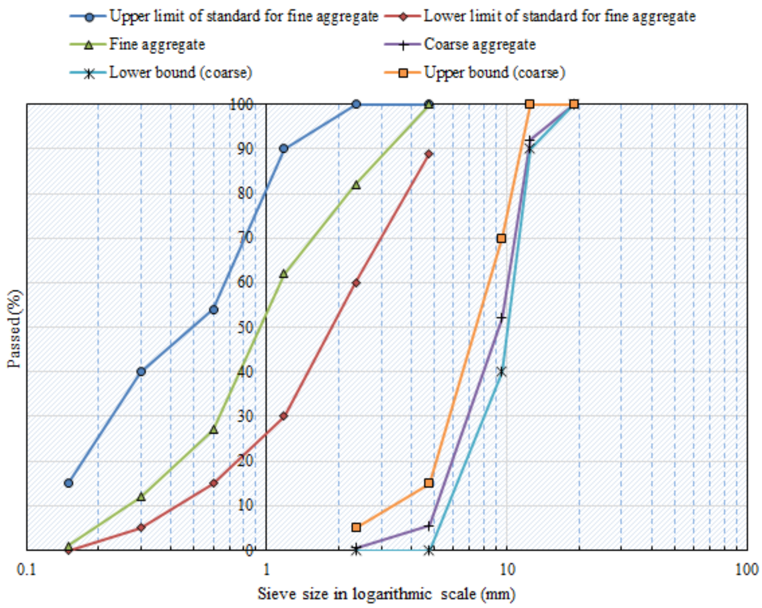

Fig. 1 Grading of coarse and fine aggregates of series A

Table 1 Properties of cement and silica fume

Chemical analysis (%)	Cement	Silica fume	BOGUE (%)	Cement
SiO_2	21.15	92.1	C_3S	52.2
Al_2O_3	4.71	0.89	C_2S	21.3
Fe_2O_3	3.48	0.85	C_3A	6.9
CaO	63.02	1.52	C_4A	10.4
MgO	3.16	1.82	$CaSO_4$	3.2
SO_3	1.92	0.74	CaOfree	0.8
Na_2O	0.21	-	Physical Tests	
K_2O	0.58	-	Fineness: Blaine (cm^2/g)	3300
Cl	0.04	0.08	Autoclave expansion (%)	0.14
LOI	1.2	2	Compressive Strength (MPa)	
Insoluble Residue	0.53	-	3-day	17.00
Total	100	100	28-day	40.00

concrete. Subsequently, minor differences within their properties could lead to a major change in the fresh and hardened state properties of concrete. For instance, Karamloo and his co-workers investigated the effects of maximum nominal size of coarse aggregate on fracture behavior of self-compacting lightweight concrete (Karamloo, Mazloom *et al*. 2016a). Ghasemi *et al*. considered the mentioned effect in the context of fiber reinforced concrete (Ghasemi, Ghasemi *et al*. 2018).

Natural river sand with the specific gravity of 2536 kg/m^3 and water absorption of 2.512% was provided from Zarnan industry (Karaj, Iran). The coarse aggregate, with the maximum nominal size of 16mm, water absorption of 1.523% and specific gravity of 2563 kg/m^3, was also provided from the mentioned industry. The grading results for coarse and fine aggregate are presented in

Table 2* Mix proportions of series A

Mix ID	W/B	kg/m^3					Admixtures (%)	
		Water	LP	Coarse	Fine	Cement	SF/C	SP/C
S1W35SF0	0.35	175	155	867	668	500	0	0.4
S2W35SF10						450	10	0.4
S3W35SF0						500	0	0.8
S4W35SF10						450	10	0.8
S5W35SF0						500	0	1.2
S6W35SF10						450	10	1.2
S7W35SF0						500	0	1.6
S8W35SF10						450	10	1.6
S9W45SF0	0.45	175	150	833	722	400	0	0.4
S10W45SF10						360	10	0.4
S11W45SF0						400	0	0.8
S12W45SF10						360	10	0.8
S13W45SF0						400	0	1.2
S14W45SF10						360	10	1.2
S15W45SF0						400	0	1.6
S16W45SF10						360	10	1.6

*W/B=Water/Binder, LP=Limestone Powder, C=Cement

Fig. 1. As a neutral filler, limestone powder, whose maximum nominal size was 0.125 mm, was provided to enhance the viscosity of the mix. Ordinary Portland cement (CEM I 42.5 N), whose chemical and physical properties are reflected in Table 1, was provided from Tehran cement factory (Tehran, Iran). Silica fume, whose specific gravity was 2.14 $\frac{gr}{cm^3}$, was used as a pozzolanic material. Table 1 shows the chemical properties of the used SF. In order to enhance the fluidity of the mixes, a poly-carboxylic-ether based superplasticizer has been provided, whose *cl* was zero and was of type B, D, G based on (ASTM C494 2001).

2.1.2 Mix compositions

Totally, sixteen mixes have been designed based on the recommendation of SP producer to see how the dosage could affect the properties of SCC. Two groups of water/binder ratios of 0.35 and 0.45 were considered in each of which the portion of aggregate were kept constant. This could lead to a better understanding of the question that how the SP dosage could change the fresh and hardened properties of SCC. In each group of water/binder ratio, two amounts of SF (0% and 10%) were considered. Table 2 shows the constituents of series A mixes.

Beside the sixteen mentioned mixes, four control mixes (normal concrete) have been cast to make comparison of SCC and NC possible. In designing the control mixes, for each water/binder ratio, two portions of SP (0 and 0.4), were considered. In the mixes without SP, no limestone powder was added, while in the mixes with 0.4% SP, equal amount of limestone powder in comparison with corresponding SCC mix was added. The constituents of the control mixes of series A are tabulated in Table 3.

Table 3 Control mixes for series A

Mix ID	W/B	kg/m³					Admixtures (%)		Slump (mm)
		Water	LP	Coarse	Fine	Cement	SF/C	SP/C	
C1SW35	0.35	175	0	867	823	500	0	0	16
C2SW35			155		668		0	0.4	185
C3SW45	0.45	180	0	833	872	400	0	0	28
C4SW45			150		722		0	0.4	197

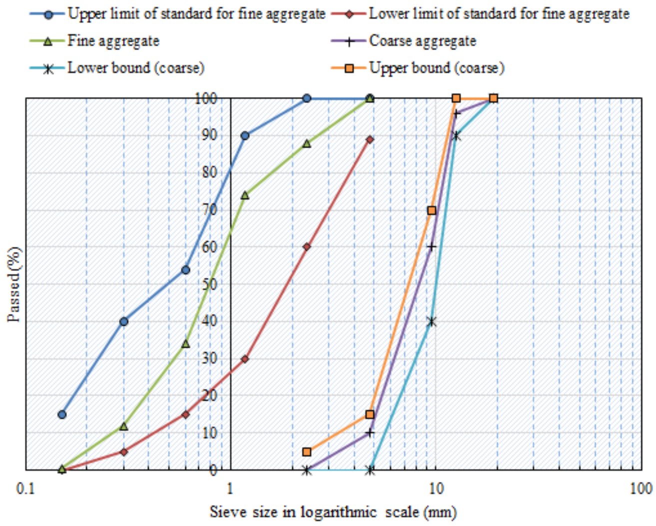

Fig. 2 Grading of coarse and fine aggregates of series B

2.2 Mixes and constituents of series B

2.2.1 Constituents

For series B, which consisted of NC mixes, the fine and coarse aggregates were crushed granite provided from Zanjan, Iran. Maximum nominal size of the coarse aggregate was considered 10 mm. Fig. 2 shows the grading of the aggregates. Ordinary Portland cement (CEM I 42.5 N), whose chemical and physical properties are reflected in Table 1, was provided from Tehran cement factory (Tehran, Iran). The used silica fume in series B was identical to that used for casting the mixes of series A. Therefore, its properties are reflected in Table 1. Four types of superplasticizer (Naphthalene-formaldehyde-based, Melamine-formaldehyde-based, Carboxylic-ether-based and poly-carboxylate-based) have been used to investigate the effects of superplasticizer type on fresh and hardened state of NC.

2.2.2 Mix proportions

Four categories of NC mixes have been designed in which two water/binder ratios of 0.35 and 0.45 have been considered. Besides, two SF/C ratios of 0 and 10% have been considered to investigate the interactive effect of SF and SP type. For each mix category, four mentioned types of SP have been used separately in order to find out how this variable could affect the results. These categories of mix proportions are reflected in Table 4.

Table 4 Mix proportions of series B

Mix ID	W/B	Constituents (kg/m^3)						
		Water	LP	Coarse	Fine	Cement	SF	SP
NC35SF0	0.35	175	200	819	756	500	-	5
NC35SF10	0.35	175	200	819	756	450	50	5
NC45SF0	0.45	180	200	825	825	400	-	4
NC45SF10	0.45	180	200	825	825	360	40	4

Table 5 Effect of SF/C and SP/C ratios on initial and final setting time of pastes

Sample ID	W/B	SF/C (%)	SP/C (%)	Initial setting (hr)	Final setting (hr)
1	0.35	0	0.4	2.4	4.65
2	0.35	10	0.4	2.25	4.5
3	0.35	0	0.8	2.65	4.85
4	0.35	10	0.8	2.43	4.63
5	0.35	0	1.2	3.05	5.15
6	0.35	10	1.2	2.77	4.85
7	0.35	0	1.6	3.55	5.72
8	0.35	10	1.6	3.15	5.25
9	0.45	0	0.4	2.85	5.35
10	0.45	10	0.4	2.6	4.85
11	0.45	0	0.8	3.23	6.6
12	0.45	10	0.8	2.95	5.47
13	0.45	0	1.2	3.85	5.75
14	0.45	10	1.2	3.38	6.15
15	0.45	0	1.6	4.62	8.6
16	0.45	10	1.6	3.87	7.42

3. Test procedures

For both series, all specimens have been cast and cured according to ASTM C31. For the hardened state properties, rupture modulus and cubic compressive strength have been tested in accordance with ASTM C78 and (BS EN 12390-4:2000 2000), respectively. In addition, to assess the interactive effect of aging, SP and SF, all specimens were tested after 7, 14, 28 and 90 days. However, the fresh state properties of NC and SCC should be tested separately. In order to evaluate the effect of SP dosage on fresh state properties of SCC (series A), the slump flow, L-box, J-ring, U-box and V-funnel test have been conducted in accordance with (EFNARC 2002). On the other hand, for NC mixes (series B), slump test has been carried out to evaluate the effects of SP type on the fresh state properties of NC. Apart from the mentioned tests, for all pastes made of different types and dosages of SP and SF, the setting time have been measured based on ASTM C 807. Moreover, since measuring the air contents could lead to a better understanding about the mechanical behavior and durability of the concrete, the interactive effects of dosage and type of SP on air contents of concrete have been considered.

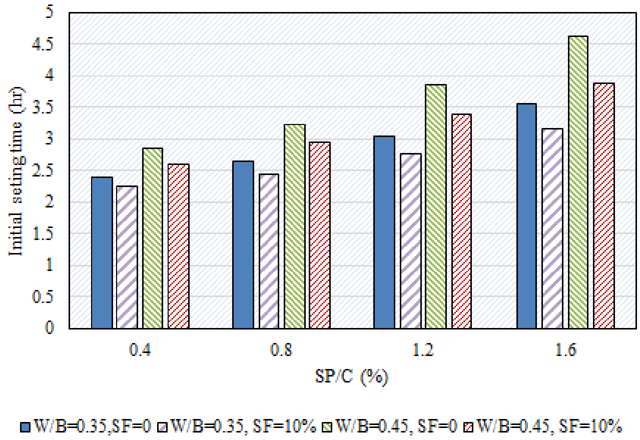

Fig. 3 Effect of superplasticizer dosage on initial setting time of pastes

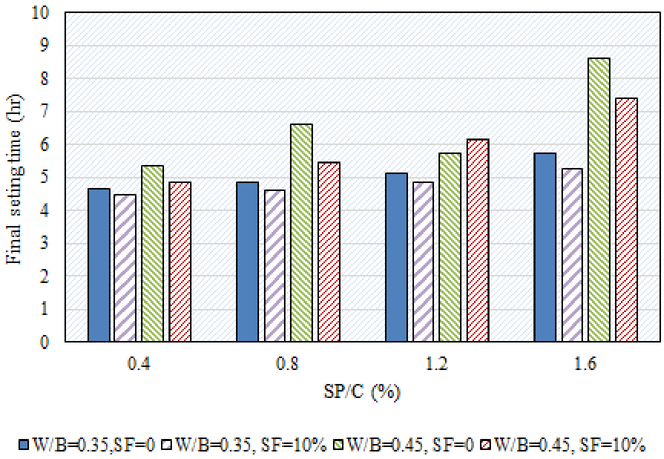

Fig. 4 Effect of superplasticizer dosage on final setting time of pastes

4. Results and discussion

4.1 series A

4.1.1 Fresh state properties

SCC is a new generation of concrete whose properties are in line with the demarche of sustainable development (Zarghami *et al*. 2017, Zarghami *et al*. 2018). This convergence with the sustainability stems from the fact that in this type of concrete, high amounts of ultra-fine particles, such as limestone powder, is using which is a byproduct of other industries. In other words, this type of concrete could help the goal of waste management (Karamloo, Mazloom *et al*. 2017, Karamloo and Mazloom 2018, Mazloom and Karamloo 2019). This generation of concrete could flow in congested reinforcing and complex formworks without bleeding or segregation (Beygi *et al*. 2014). These properties are directly related to fresh state properties of this kind of concrete

Table 6 The amounts of air contents in mixes

W/B	SF/C (%)	SP/C (%)	Air contents (%)
0.35	0	0.4	0.42
		0.8	0.5
		1.2	0.52
		1.6	0.57
	10	0.4	0.38
		0.8	0.46
		1.2	0.5
		1.6	0.54
0.45	0	0.4	0.63
		0.8	0.67
		1.2	0.68
		1.6	0.71
	10	0.4	0.6
		0.8	0.65
		1.2	0.67
		1.6	0.7

and obviously the amount of superplasticizer. Setting time of concrete is related to the cement type; size distribution of cement particles, water/cement ratio, concrete temperature and properties of admixes. In mixes of series A, all variables have been kept constant except the amount of SP to see how this variable could affect the results. As it can be seen from Table 5, the use of polycarboxylic-ether based superplasticizer, lead to a general increase in setting time of the pastes. This finding can be attributed to the existence of hydroxy-carboxylic acids, which causes a retardation in concrete setting (Plank *et al*. 2015). The variation of initial and final setting times for pastes are shown in Figs. 3 and 4, respectively. As it can be seen, the addition of 6 kg and 4.8 kg of carboxylic-ether based SP, when water to binder ratio equals to 0.35 and 0.45, respectively, increased the initial setting time of the cement paste in the absence of SF by 35.4% and 62.1%, respectively. However, the replacement of 50 kg and 40 kg SF by cement increased the retardation process and one can observe that the setting time of pastes with W/B ratios of 0.35 and 0.45, respectively, increased by 40 % and 48.84% respectively. Of course, it is apparent that at the same SP dosage, the addition of SF has led to a decrease in setting time. Besides, in water/binder ratios of 0.35 and 0.45, it is observed that the final setting time is affected by both silica fume addition and SP dosage.

Measurement of the air contents of the mixes is of great importance, especially, when the admixtures such as superplasticizer were used. Hence, the air contents of the mixes have been determined according to ASTM C 231. These measurements are tabulated in Table 6.

It is clear that the use of poly-carboxylic-ether based SP increases the air contents of the concrete. In other words, in water to binder ratios of 0.35 and 0.45 and in the absence of silica fume, the increase of SP dosage from 0.4% to 1.6% has led to an increase of air contents by 35.7% and 12.7%, respectively. These numbers, however, in the mixes with silica fume, have changed to 42.1% and 16.67%, respectively. In the same amount of superplasticizer, the experiments show a

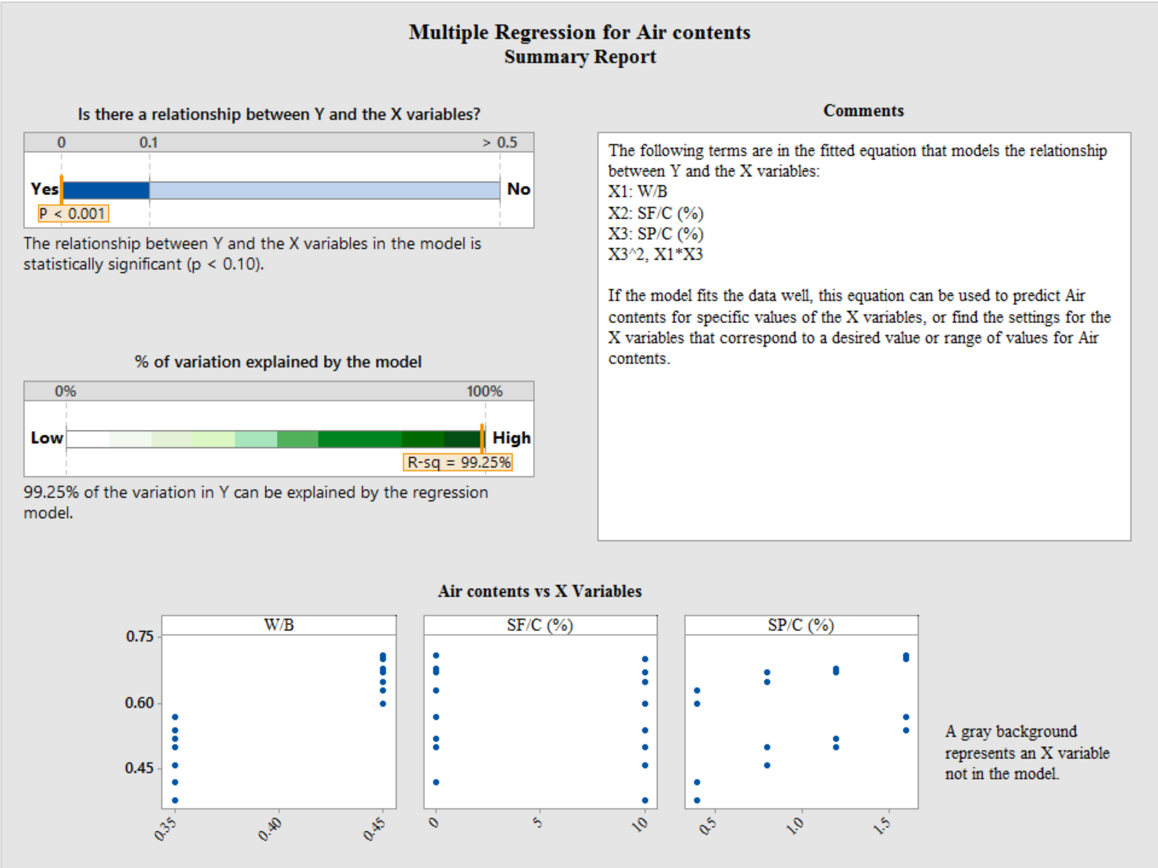

Fig. 5 The statistics of regression analysis

decrease in air content when the silica fume was used. This observation is in line with durability enhancing properties of silica fume, which is caused by its pozzolanic activity. Since the air contents have prevalent effects on mechanical properties of concrete, predicting its amount based on mix design parameters could lead to a better insight into the behavior of concrete. In this regard, a multiple regression analysis has been conducted, whose results are shown in Fig. 5 and Eq. (1).

$$Air(\%) = -0.4612 + 2.3\left(\frac{W}{B}\right) - 0.0025\left(\frac{SF}{C}\right) + 0.3856\left(\frac{SP}{C}\right)^2 - 0.525\left(\frac{W}{B}\right)\left(\frac{SP}{C}\right) \quad (1)$$

As it can be seen, the p-value of the model, which corresponds to the significance of the regression model, shows that the relation between the mix design parameters and air contents are statically significant (as shown in Fig. 5). Besides, the R^2 value shows that the proposed model can explain the amount of air content. Another question, which could be raised, is which mix design parameter is more statically important in the regression model? The answer could be explained by Fig. 6 in which the effect of variables on R^2 value has been assessed.

As it can be observed in Fig. 6, water to binder ratio had the most important effect on the validity of the model and alone could affect the model by about 75%. In contrast, the SF/C ratio

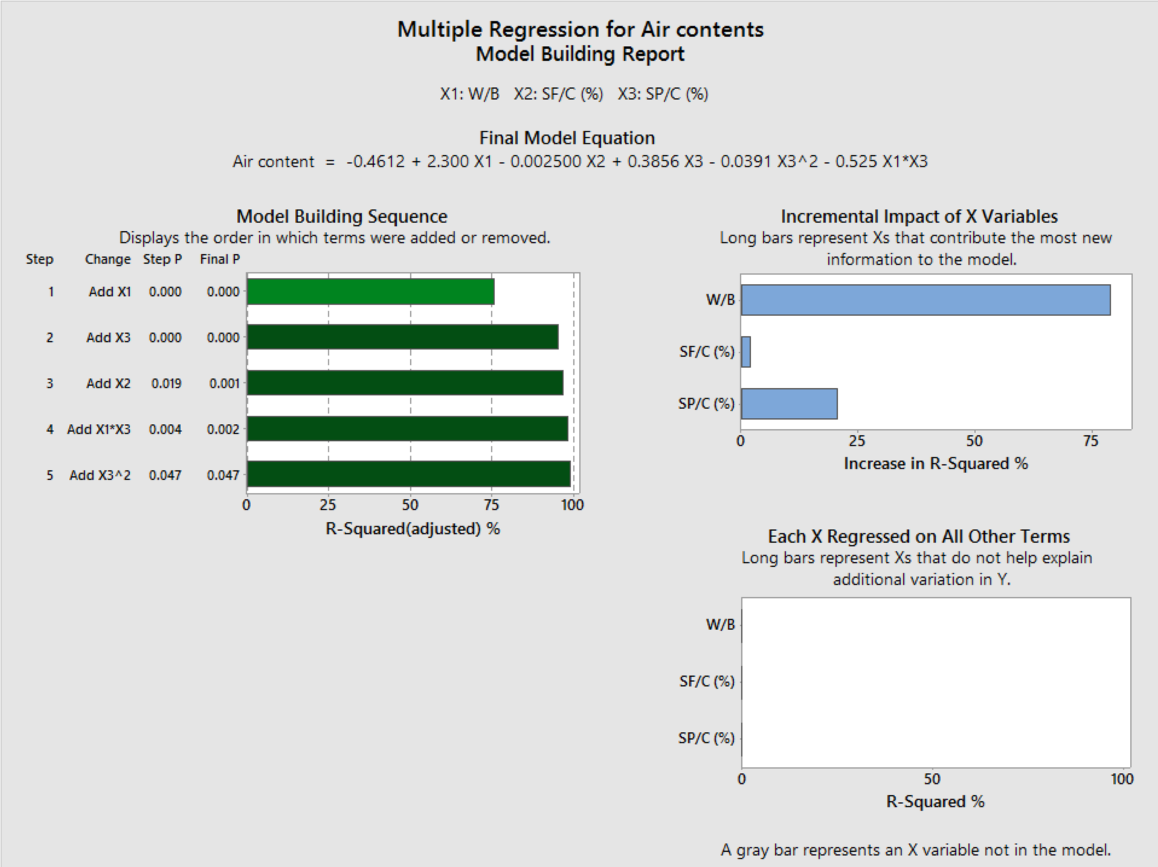

Fig. 6 Assessing the effects of variables

had the least effect on the results. Of course, it should be mentioned that 16 mixes are not a sufficient sample size to judge the effects of mixing parameters and this context needs more comprehensive data.

For all mixes, slump flow, J-ring, U-box, L-box and V-funnel tests have been carried out to find out the effect of SP dosage on flowability, segregation resistance, passing ability, bleeding and blockage. The results of these tests are tabulated in Table 7. It should be noted that some mixes did not pass the fresh state conditions of SCC. Therefore, the ordinary slump test, which is for NC, has been conducted for them and the results are reported as well. It should be noted that the effect of a superplasticizer on workability of a SCC is completely related to the microstructure of SP. Effects of chemical structure of poly-carboxylate based SPs have been studied by (Felekoğlu and Sarıkahya 2008). They studied three synthetic poly-carboxylate based SPs which were synthetized by radical polymerization technique. It is reported that the manipulation of the bond structure between main backbone and side-chain of copolymer could lead to major changes in workability retention, setting time and compressive strength of concrete.

As mentioned in previous sections, four control mixes of normal concrete have been cast, whose details and slump are reported in Table 3. As it can be seen, limestone powder and superplasticizer enhanced the slump of NC.

Table 7 Workability of SCC mixes of series A

W/B	SF/C (%)	SP/C (%)	Flow time (Sec)	Flow dia. (mm)	V-funnel time (Sec)	L-box ratio	Flow dia-J-ring dia. (mm)	U-box height difference (mm)	Normal slump (mm)	Observations and grade of workability based on EFNARC
0.35	0	0.4	-	-	-	-	-	-	238	The behavior was similar to NC
		0.8	4.8	730	6.5	0.86	12	12	-	High viscosity, SF2, Low passing ability, VS1/VF1, no bleeding, PA2
		1.2	2.3	785	5.4	0.9	6.3	7	-	SF3, good passing ability, no bleeding, VS1/VF1, PA2
		1.6	2	825	4.8	0.95	4	3	-	Minor segregation, SF3, good passing ability, VS1/VF1, a little bleeding tendency, PA2
	10	0.4	-	-	-	-	-	-	215	The behavior was similar to NC
		0.8	8	550	8	0.62	14.5	36	-	High viscosity, SF1, low passing ability, VS2/VF2, no bleeding, PA1
		1.2	4.5	670	6.2	0.82	8	18	-	SF2,good passing ability, VS1/VF1, no bleeding, PA2
		1.6	3	780	5.3	0.9	5.5	5	-	SF3, good passing ability, VS1/VF1, no bleeding, PA2
0.45	0	0.4	-	-	-	-	-	-	216	The behavior was similar to NC
		0.8	4.5	730	4	0.88	14	8	-	SF2, good passing ability, VS1/VF1, no bleeding, PA2
		1.2	2.8	810	3.6	0.96	6.5	4	-	SF3, good passing ability, VS1/VF1, bleeding tendency, PA2
		1.6	2.6	830	3.3	0.98	4.2	1	-	Segregation, SF3, poor passing ability, VS1/VF1, bleeding tendency, PA2
	10	0.4	-	-	-	-	-	-	185	The behavior was similar to NC
		0.8	6.5	530	4.8	0.57	17	24	-	High viscosity, SF1, low passing ability, VS1/VF1, no bleeding, PA1
		1.2	3.2	760	4.2	0.86	12	13	-	SF3, good passing ability, VS1/VF1, no bleeding, PA2
		1.6	2.8	770	3.8	0.9	11	6	-	SF3, good passing ability, VS1/VF1, bleeding tendency, PA2

4.1.2 Hardened state properties

Compressive strength of concrete is an essential mechanical property to be known for design purposes. Since concrete properties are time dependent, the effect of aging is of great importance. In addition, the constituents have prominent effects on the properties of concrete materials such as concrete. Therefore, in the present study, effects of superplasticizer dosage on compressive strength of SCC have been considered as well as the aging effect. This makes it possible to see how the interaction of aging and the use of superplasticizer could affect the compressive strength of SCC. Moreover, these tests have been carried out for NC control mixes, which this makes it possible to compare the behavior of NC and SCC. The results are reflected in Table 8.

As stated previously, the air content of concrete is closely related to the mechanical properties of concrete. In this regard, in Fig. 7, a linear correlation between the air content and 28-day compressive strength has been proposed. As it can be seen, the results show a robust relation between the air contents and the 28-day compressive strength. In addition, by considering Table 8, it is clear that in almost all of the mixes, by increasing the dosage of superplasticizer, the compressive strength of SCC mixes decreased (as an example see Fig. 8). However, the addition of silica fume enhanced the compressive strength of SCC mixes. These trends were in agreement with those reported in the study of Antoni *et al*. (Antoni *et al*. 2017).

The other important mechanical property is tensile strength, which could be measured by using

Table 8 Compressive strength of samples of series A in different ages

Mix ID	Type	Compressive strength (MPa)			
		7-day	14-day	28-day	90-day
S1W35SF0	SCC	44	58.3	61	65
S3W35SF0		40	58.3	58	63.2
S5W35SF0		39	35.5	58	62
S7W35SF0		35	48.5	56	60.8
S2W35SF10		46	60	69	72
S4W35SF10		42	56	62	67
S6W35SF10		40	54.2	60	65.7
S8W35SF10		38	52.3	58	63
S9W45SF0		32	41.3	47	52
S11W45SF0		30	37.2	42	46.5
S13W45SF0		29	37	40	44
S15W45SF0		27	34.8	37	41.2
S10W45SF10		34	43.3	48	53
S12W45SF10		31	37.8	45	49
S14W45SF10		30	38.5	46	49.5
S16W45SF10		28	36.2	41	45.2
C1SW35	NC	15	17.3	19	21.3
C2SW35		20.5	25	27	31
C3SW45		12	15.7	17	19.2
C4SW45		18	21.2	24	27

three different methods i.e., direct tensile test, indirect tensile test and modulus of rupture. Generally, modulus of rupture test results in an overestimation in determination of tensile strength of concrete. This was attributed to two reasons. First, the volume of concrete exposed to tensile stress is less than that of other methods so that there is an inferior chance of a weak component (Bažant and Li 1995). Second, the rupture and splitting test methods include non-uniform stress scatterings, which obstruct the spread of a crack; hence, delay the final fracture (Mazloom, Saffari *et al*. 2015). However, this method is very simple and is one of the most popular methods of testing of the tensile strength. In the present study, effects of SP dosage on the rupture modulus of the samples have been studied. Table 9 shows the rupture modulus of each mix.

As it can be seen, SCC mixes had more tensile strength than NC control mixes which is due to consolidation of the SCC mixes. In addition, in almost all of SCC mixes, by increasing the amount of superplasticizer, the rupture modulus decreased. By observing the growth rate of rupture modulus, it is obvious that in the mixes with silica fume, the growth of rupture modulus after 28 days was negligible in comparison to the trend observed in early age.

In many practical cases, the compressive strength of concrete is available, while the modulus of rupture is also needed for design purposes. Consequently, a correlation between these two mechanical properties is essential for preliminary design purposes. Almost all relations proposed by national codes are of the form $f_r = \alpha\sqrt{f_c}$, in which f_r denotes the rupture modulus and f_c is the

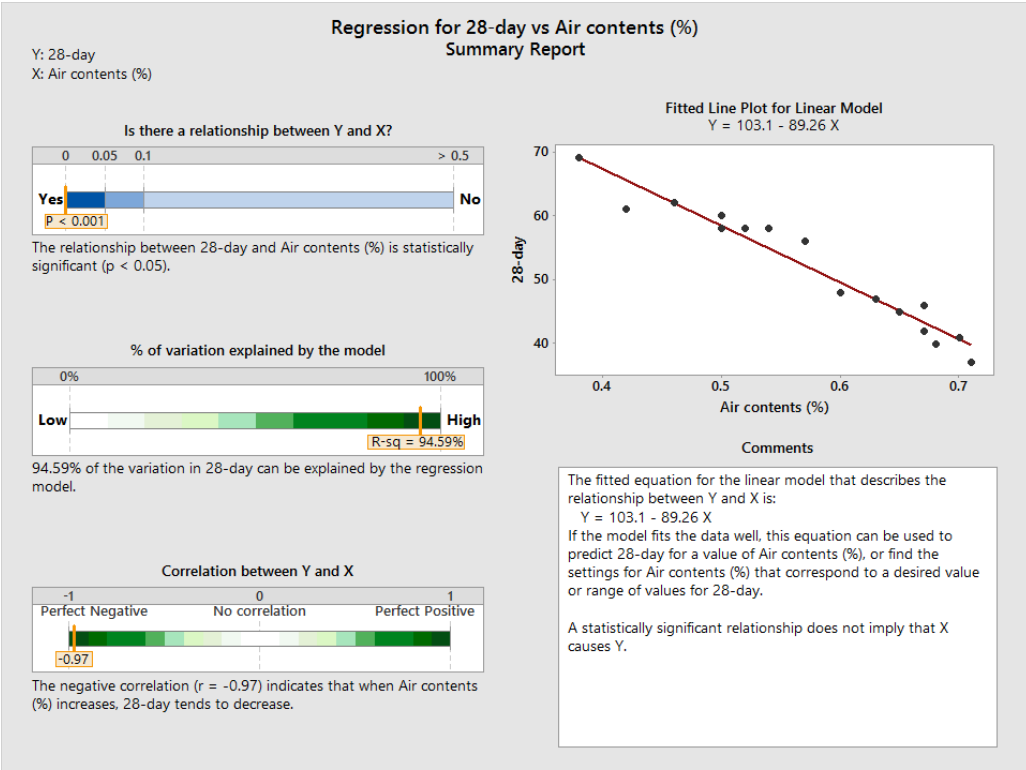

Fig. 7 The correlation between the air contents of the samples of series A and their 28-day compressive strength

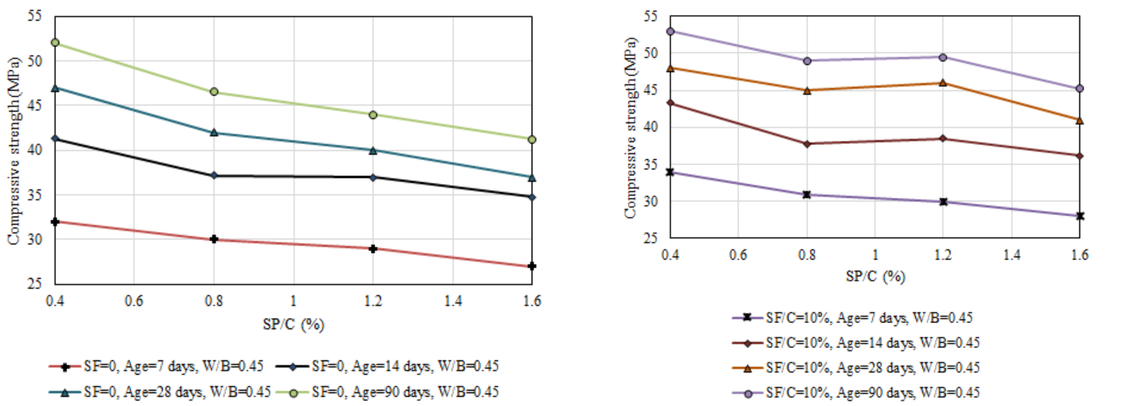

Fig. 8 The effect of SP dosage on compressive strength of mixes with W/B=0.45, SF=0 (left) and SF/C=10% (Right)

compressive strength of concrete. In the present study, the abovementioned is used and the results are reflected in Figs. 9 and 10. As it can be seen, α equals to 0.64, which differs a little with the α = 0.75 recommended by ACI318 2005 for normal concrete. This difference could stem from either the fact that the constituents have prominent effect on mechanical properties of concrete, or the sample size of this study, which is too small to lead to a comprehensive result.

Table 9 Modulus of rupture for mixes of series A and control NC mixes

Mix ID	7-day	14-day	28-day	90-day
S1W35SF0	4.19	5.03	5.14	5.27
S3W35SF0	3.87	4.77	5.05	5.25
S5W35SF0	3.79	4.75	5.00	5.22
S7W35SF0	3.47	4.43	4.87	5.15
S2W35SF10	4.3	5.14	5.47	5.62
S4W35SF10	4.02	4.89	5.18	5.36
S6W35SF10	3.87	4.79	5.1	5.31
S8W35SF10	3.74	4.72	5.05	5.23
S9W45SF0	3.3	3.93	4.36	4.72
S11W45SF0	3.24	3.69	3.96	4.35
S13W45SF0	3.26	3.68	3.86	4.18
S15W45SF0	3.19	3.46	3.67	3.92
S10W45SF10	3.4	4.26	4.42	4.74
S12W45SF10	3.27	3.73	4.22	4.49
S14W45SF10	3.24	3.76	4.27	4.53
S16W45SF10	3.19	3.58	3.91	4.26
C1SW35	1.27	1.43	1.54	1.82
C2SW35	2.47	3.02	3.19	3.27
C3SW45	1.03	1.32	1.4	1.56
C4SW45	2.19	2.56	2.86	3.19

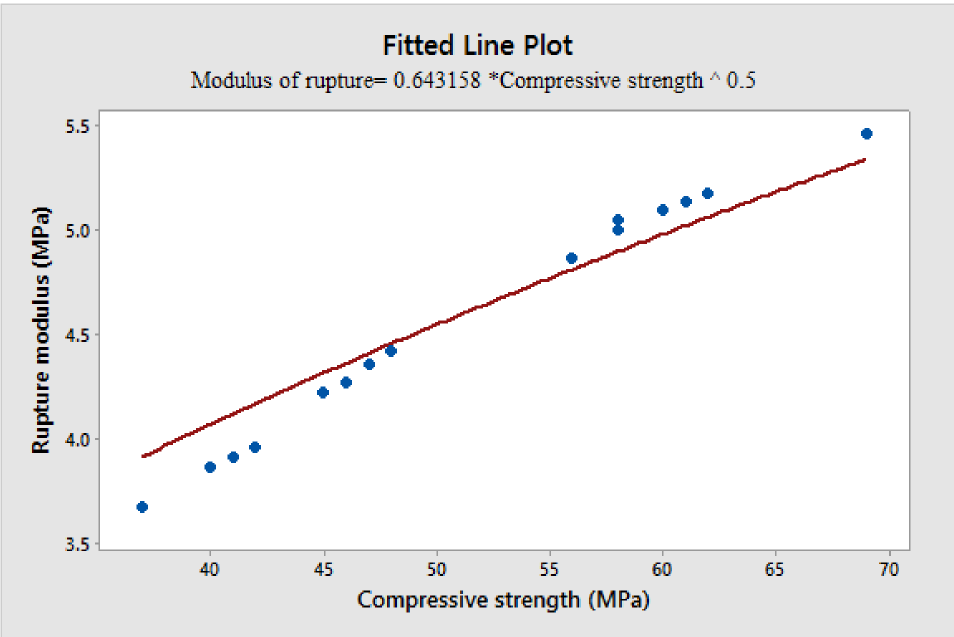

Fig. 9 28-day rupture modulus versus 28-day compressive strength

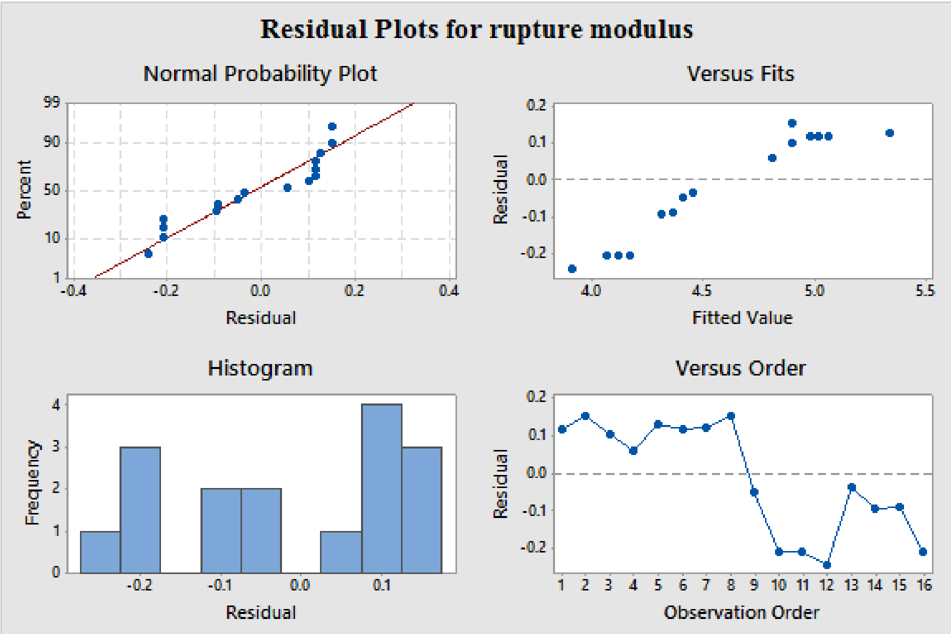

Fig. 10 The residual plots for regression analysis between compressive and tensile strength

Table 10 Setting time for pastes of series B

SF/C (%)	SP type	Initial setting time (hr.)	Final setting time (hr)
0	Melamine-formaldehyde	1.7	2.67
	Naphthalene-formaldehyde	2.37	3.25
	Carboxylic-ether	3.20	4.25
	poly-carboxylate (Super TM-ON-S 2000)	1.95	3.00
10	Melamine-formaldehyde	1.33	2.67
	Naphthalene-formaldehyde	1.37	2.67
	Carboxylic-ether	5.36	7.70
	poly-carboxylate (Super TM-ON-S 2000)	1.76	3.08

4.2 series B

4.2.1 Fresh state properties

In order to consider the effect of SP type on fresh properties of concrete, three categories of tests have been carried out, including the assessment of initial and final setting time, determination of air contents and evaluation of the workability of the mixes. To assess the effect of SP type on setting time of the pastes containing different type of SP, the ASTM C191 test method has been used. To do so, in this series of study, the normal consistency test has been conducted to find the amount of water needed to produce a consistent paste. This experiment, which was carried out according to ASTM C 187, resulted in water/cement ratio of 22%. Afterwards, eight pastes made of four mentioned types of SP and two amounts of SF have been cast. The results are reflected in Table 10 and Figs. 11 and 12.

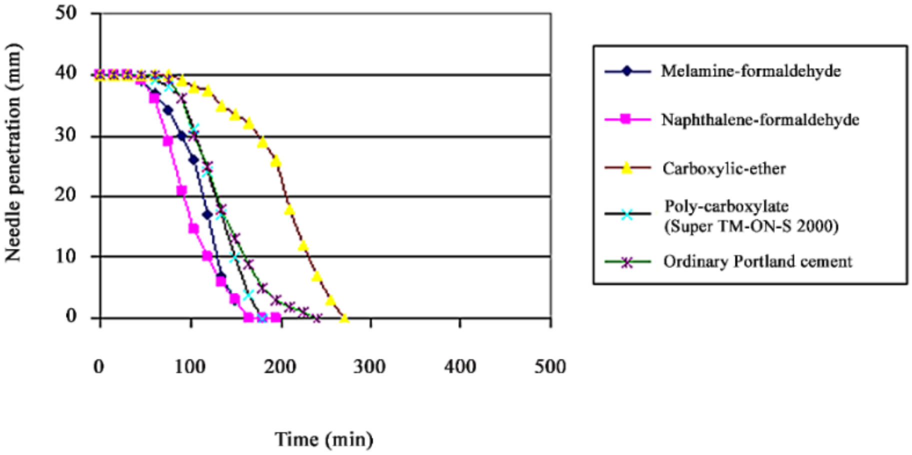

Fig. 11 Setting time of paste with SF=0

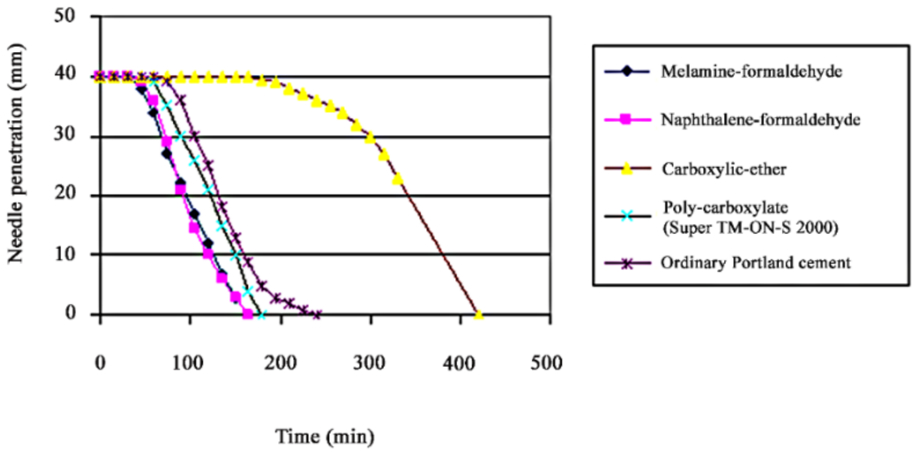

Fig. 12 Setting time of paste with SF=10%

As it can be seen in Table 10, Fig. 11 and Fig. 12, superplasticizers and silica fume affected the initial and final setting time of pastes. In other words, the retarding effect of the carboxylic ether based SP was more prominent than melamine-formaldehyde, naphthalene-formaldehyde and even poly-carboxylate. This feature was even stronger in the presence of silica fume. The retardation of hydration process infers that practitioners should be more cautious about using this admixture in cold regions. The effects of SP on initial setting time and workability retention of cement pastes was also studied by Zhang *et al*. (Zhang *et al*. 2010). They used three SP types including a newly modified lignosulphonate SP, poly-carboxylate and poly-naphthalene SPs. In order to monitor the workability loss, they considered yield stress and effective viscosity of the pastes. Besides, they monitored the initial setting time, the heat development, change of rheological parameters and the penetration depth. The results indicated that the pastes with lignosulphonate showed longer initial setting time compared with those made with poly-carboxylate and poly-naphthalene (Zhang *et al*. 2010). Besides, the replacement of silica fume by 10% cement led to a decrease of initial setting times in almost all cases except those pastes made from poly-carboxylic ether based SPs.

However, the effect of silica fume on final setting time of almost all cases except the case made of poly-carboxylic ether SP was less than initial setting times. These differences in the behavior of paste with SF and those without SF could be originated from the fact that the behavior of superplasticizer depends on the microstructure of cementitious materials. For example, the interaction of poly-carboxylate based SP with the amount of C_3A in cement has been studied by (Zingg *et al*. 2009). This parametric study correlated the molecular structure of a carboxylate with C_3A contents in cement and claimed that the workability and retardation of cement paste is affected by this interaction.

The series B consisted of normal concrete mixes, therefore, the slump test has been carried out to assess the effect of SP type on workability of concrete. The results are shown in Table 11. It is clear that the type of SP has a prominent effect on workability of NC samples. It is also apparent that the poly-carboxylate based SP and the poly-carboxylic ether based SP showed far better results in slump results. Besides, increasing the SF content in NC samples has led to a decrease of workability. Based on the researches in the literature, the type of SP also could influence the packing density, water film thickness and flowability o cementitious pastes. Lie *et al*. who studied the mentioned effect, reported that both the naphthalene-based SP and poly-carboxylate based SP significantly increased the packing density and water film thickness (Li and Kwan 2015). However the latter type was more effective than the former. Mardani-Aghabaglou and his co-workers investigated the effect of SP type on fresh, rheological and strength properties of SCC (Mardani-Aghabaglou *et al*. 2013). They used SPs whose main chain and polymer structures was the same. However, the molecular weight and side chain density of them were different. Their study showed that the V-funnel flow time, plastic viscosity and slump retention of SCC mixes are considerably related to side chain density of polymers. In addition, they reported that the compressive strength of SCC could be influenced, especially in early age, by the structure of superplasticizers.

Table 11 Effect of SP type on slump of the mixes in series B

W/B	SF/C	SP type	Slump (mm)
0.35	0	Melamine-formaldehyde	25
		Naphthalene-formaldehyde	50
		Carboxylic-ether	120
		poly-carboxylate (Super TM-ON-S 2000)	150
	10	Melamine-formaldehyde	15
		Naphthalene-formaldehyde	30
		Carboxylic-ether	100
		poly-carboxylate (Super TM-ON-S 2000)	85
0.45	0	Melamine-formaldehyde	30
		Naphthalene-formaldehyde	55
		Carboxylic-ether	150
		poly-carboxylate (Super TM-ON-S 2000)	170
	10	Melamine-formaldehyde	20
		Naphthalene-formaldehyde	40
		Carboxylic-ether	120
		poly-carboxylate (Super TM-ON-S 2000)	100

Table 12 Air contents of the mixes of series B

W/B	SF/C	SP type	Air contents (%)
0.35	0	Melamine-formaldehyde	1.15
		Naphthalene-formaldehyde	1
		Carboxylic-ether	0.5
		poly-carboxylate (Super TM-ON-S 2000)	0.3
	10	Melamine-formaldehyde	1.1
		Naphthalene-formaldehyde	0.6
		Carboxylic-ether	0.45
		poly-carboxylate (Super TM-ON-S 2000)	0.25
0.45	0	Melamine-formaldehyde	1.25
		Naphthalene-formaldehyde	1.1
		Carboxylic-ether	0.65
		poly-carboxylate (Super TM-ON-S 2000)	0.5
	10	Melamine-formaldehyde	1.2
		Naphthalene-formaldehyde	0.8
		Carboxylic-ether	0.5
		poly-carboxylate (Super TM-ON-S 2000)	0.35

Table 13 Compressive strength of the mixes of series B in different ages

W/B	SF/C	SP type	Compressive strength (MPa)			
			7-day	14-day	28-day	90-day
0.35	0	Melamine-formaldehyde	45	47.5	54	57
		Naphthalene-formaldehyde	50.7	52.8	57.4	62
		Carboxylic-ether	50.4	56.8	59.8	64.3
		poly-carboxylate (Super TM-ON-S 2000)	50	56.3	64	68
	10	Melamine-formaldehyde	43.9	47	53	55
		Naphthalene-formaldehyde	51.4	54	62.1	63.5
		Carboxylic-ether	53	63.8	69	72
		poly-carboxylate (Super TM-ON-S 2000)	53.6	63.7	70	74
0.45	0	Melamine-formaldehyde	32.1	38.7	40.3	44
		Naphthalene-formaldehyde	30.5	41.5	42.4	48
		Carboxylic-ether	40.6	45.4	45.9	50.8
		poly-carboxylate (Super TM-ON-S 2000)	34.8	41.6	47.8	52
	10	Melamine-formaldehyde	30	35	39.7	44
		Naphthalene-formaldehyde	33.5	38.8	43.1	48
		Carboxylic-ether	35	43	48	56
		poly-carboxylate (Super TM-ON-S 2000)	39.3	41	48.5	57

Another main property of concrete, which plays an important role in mechanical behavior of concrete, is the amount of air contents. In other words, an increase of the air contents could lead to

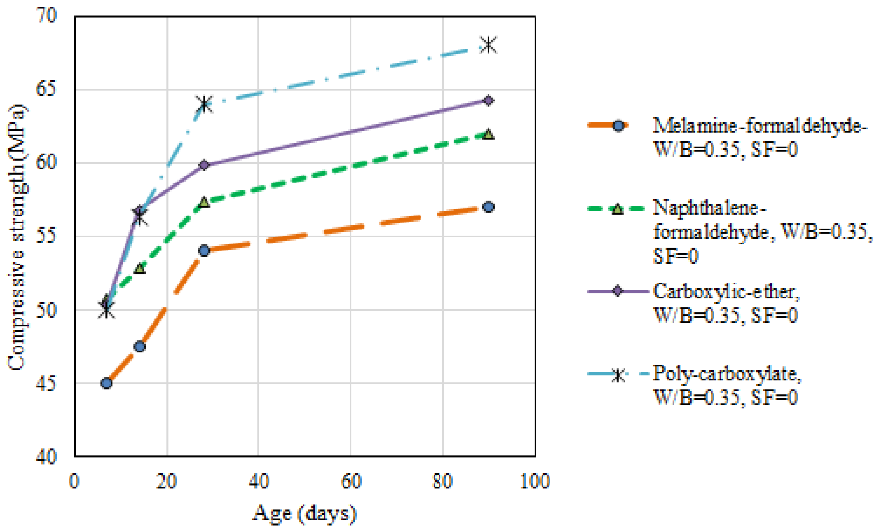

Fig. 13 Variation of compressive strength with time in mixes with W/B=0.35 and SF=0

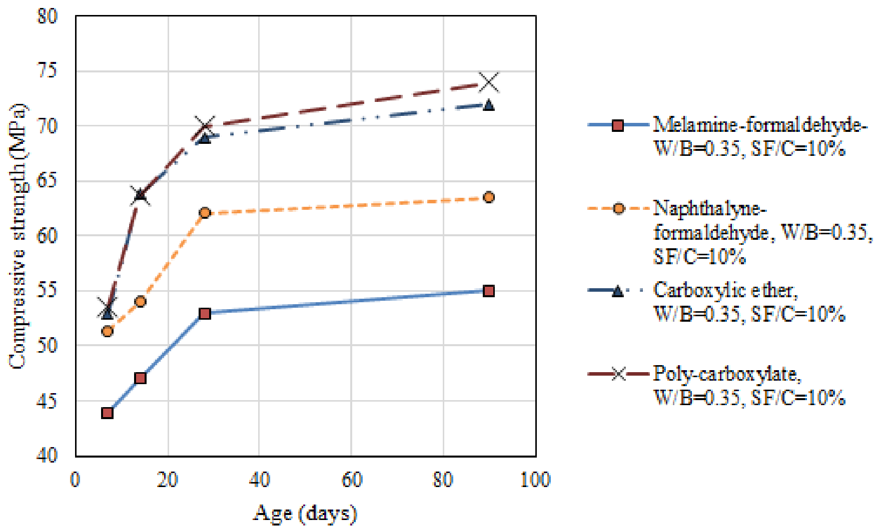

Fig. 14 Variation of compressive strength with time in mixes with W/B=0.35 and SF/C=10%

a decrease of compressive strength. Therefore, a study in this context could lead to a better understanding about the mechanical properties of the concrete. In addition, an increase of this parameter could be harmful for durability of the concrete. In this regard, the effect of SP type on the amount of produced air in the mix has been investigated and the results have been reflected in Table 12. It is apparent that the mixes made by poly-carboxylate based SP had the least and melamine-formaldehyde based SP had the most air contents within the mixes. In addition, it is clear in Table 12 that silica fume led to a decrease of air contents. This can be integrated with the durability-beneficial properties of silica fume.

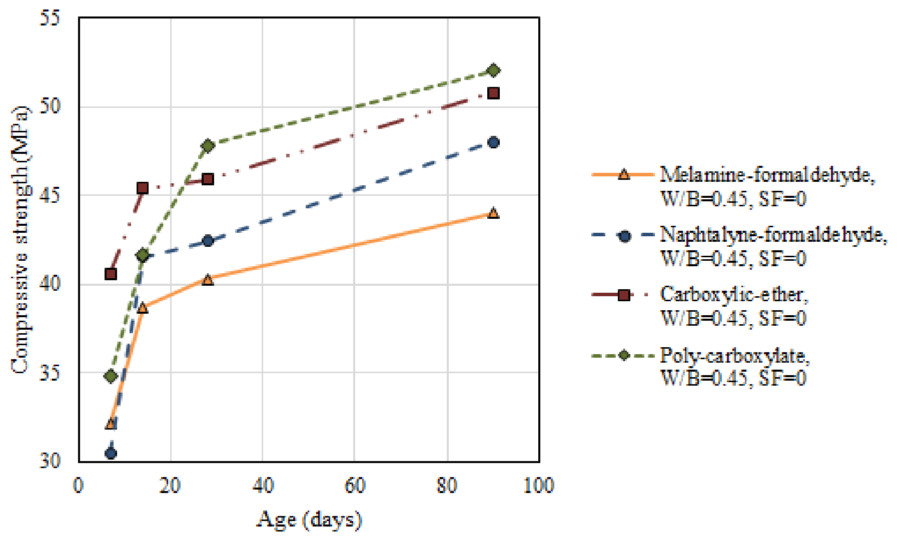

Fig. 15 Variation of compressive strength with time in mixes with W/B=0.45 and SF/C=0%

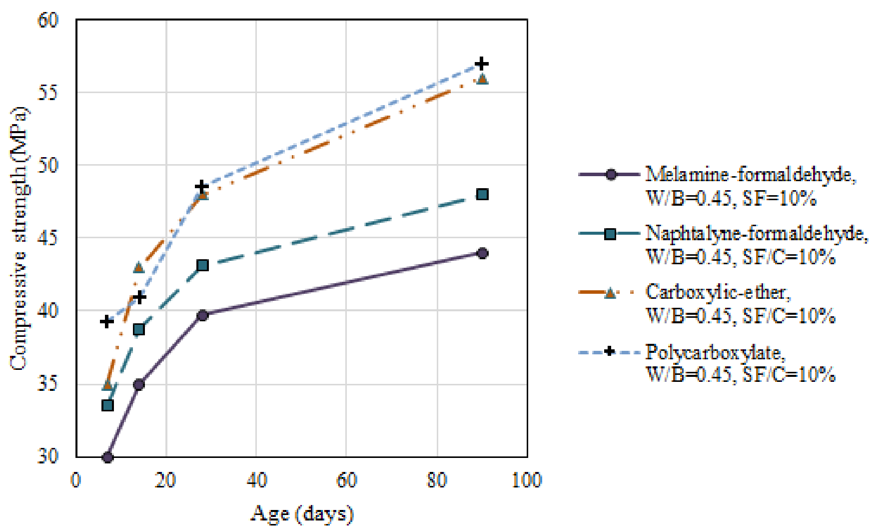

Fig. 16 Variation of compressive strength with time in mixes with W/B=0.45 and SF/C=10%

4.2.2 Hardened state properties

Superplasticizers can affect the microstructure of concrete (Huang *et al*. 2016) upon which the global mechanical behavior of concrete is established. On the other hand, different SP types have their own special microstructure and property. Therefore, studying the effect of SP type on mechanical properties of concrete is of great importance. In this regard, in the present study, for two W/B ratios of 0.35 and 0.45 and also for two different amounts of SF/C ratios, the effects of utilizing four different SP type on Compressive strength and rupture modulus of NC have been

Table 14 Rupture modulus of the mixes of series B in different ages

W/B	SF/C	SP type	Rupture modulus (MPa)			
			7-day	14-day	28-day	90-day
0.35	0	Melamine-formaldehyde	4.25	4.4	4.72	4.86
		Naphthalene-formaldehyde	4.57	4.6	4.73	4.88
		Carboxylic-ether	4.5	4.66	4.78	5.01
		poly-carboxylate (Super TM-ON-S 2000)	4.5	4.67	4.94	5.09
	10	Melamine-formaldehyde	4.2	4.38	4.59	4.70
		Naphthalene-formaldehyde	4.6	4.68	4.87	5.0
		Carboxylic-ether	4.65	4.98	5.11	5.21
		poly-carboxylate (Super TM-ON-S 2000)	4.38	4.98	5.14	5.23
0.45	0	Melamine-formaldehyde	3.25	3.53	3.79	3.95
		Naphthalene-formaldehyde	3.3	3.54	3.81	4.01
		Carboxylic-ether	3.8	4.02	4.23	4.33
		poly-carboxylate (Super TM-ON-S 2000)	3.38	3.96	4.22	4.33
	10	Melamine-formaldehyde	3.07	3.40	3.8	3.97
		Naphthalene-formaldehyde	3.3	3.70	3.9	4.02
		Carboxylic-ether	3.5	4.05	4.4	4.52
		poly-carboxylate (Super TM-ON-S 2000)	3.64	3.76	4.53	4.57

investigated. Besides, in order to consider the effect of time on compressive strength and rupture modulus of specimens, all tests carried out after 7 days, have been repeated at ages 14, 28 and 90 days. The results of compressive tests and rupture modulus experiments are reflected in Tables 13 and 14, respectively. The durability behavior of concrete is related to its time dependent properties and is of great importance especially in design of infrastructures such as bridges, dams etc. The superplasticizers can affect the durability of concrete and in a simple manner the mechanical behavior of concrete during a period. In Figs. 13-16 the interactive effects of SP type, SF and the age on compressive strength of concrete have been depicted. It is apparent that melamine-formaldehyde based SP showed the least initial and final compressive strength among the mixes made of different SP types. This could be explained by Table 12 in which the amounts of air contents of the mixes have been reflected. In other words, the more the air contents exist the less the compressive strength could be reached. In addition, considering the slop of lines connecting 28-day compressive strength to 90-day compressive strength in Figs 13-16, it could be observed that the rate of compressive strength development in mixes made of melamine-formaldehyde were less than those made by other types of SP. In contrast, mixes consisted of poly-carboxylate based SP not only showed the best 28-day compressive strength, but also benefited the better strength development growth rate than the other types. In almost all cases, it also showed the best early age strength, which turn this superplasticizer into the best volunteer to be used in cases where the strength is the most important matter. However, the hydration heat and the microstructural properties of superplasticizers should be considered carefully in construction of massive projects. In the cases in which the interaction of silica fume has been studied, it could be seen than poly-carboxylate based SPs performed similar to poly-carboxylic ether based SPs. This finding could be originated from the interaction between the microstructures of SF and SP.

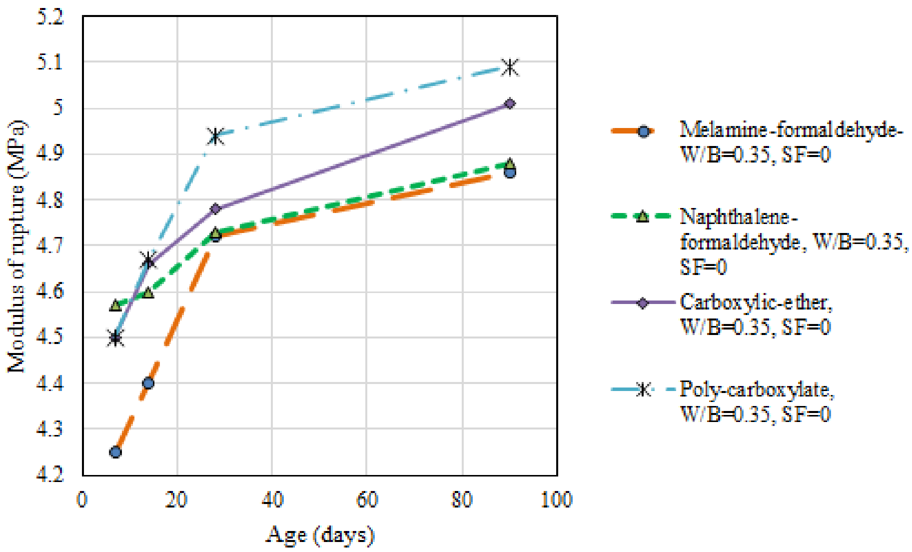

Fig. 17 Variation of rupture modulus with time in mixes with W/B=0.35 and SF/C=0%

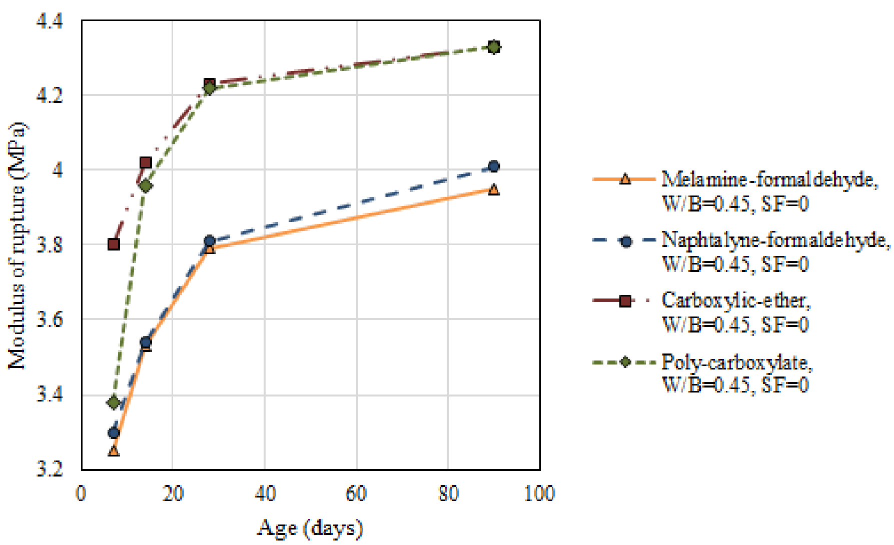

Fig. 18 Variation of rupture modulus with time in mixes with W/B=0.45 and SF/C=0%

The other important mechanical parameter is modulus of rupture. In this section of study, the effects of SP type on rupture modulus of concrete have been considered as well as aging. The results are reflected in Table 14. In addition, the interactive effects regarding the use of SP and SF along with the effect of aging are shown in Figs. 17-20. It is clear that the poly-carboxylate-based SP had the best performance within the tested SPs. However, the poly-carboxylic-ether based SP in high water/cement ratios could lead to a better early age rupture modulus. The melamine-formaldehyde based SP in both early age and 90-day age had the least modulus of rupture. Therefore, the practitioners should consider these properties of this kind of SP in either design

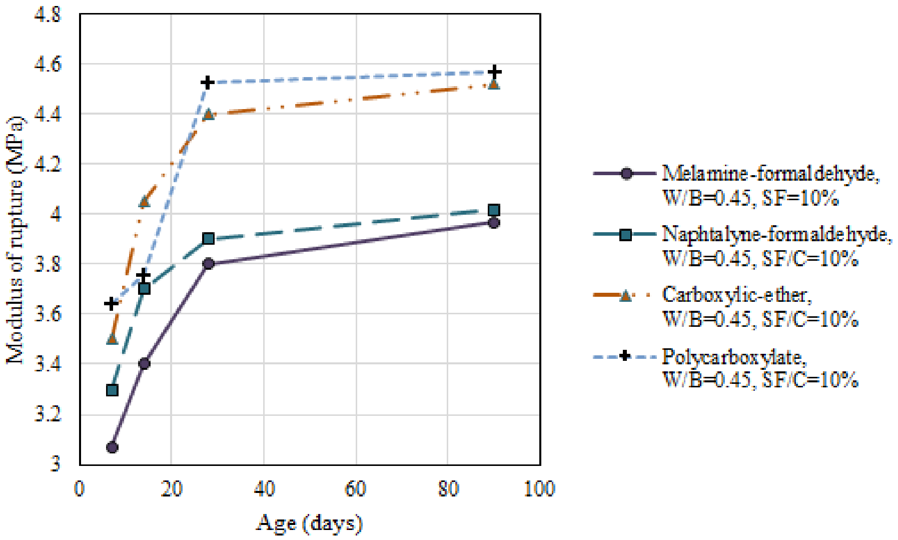

Fig. 19 Variation of rupture modulus with time in mixes with W/B=0.45 and SF/C=10%

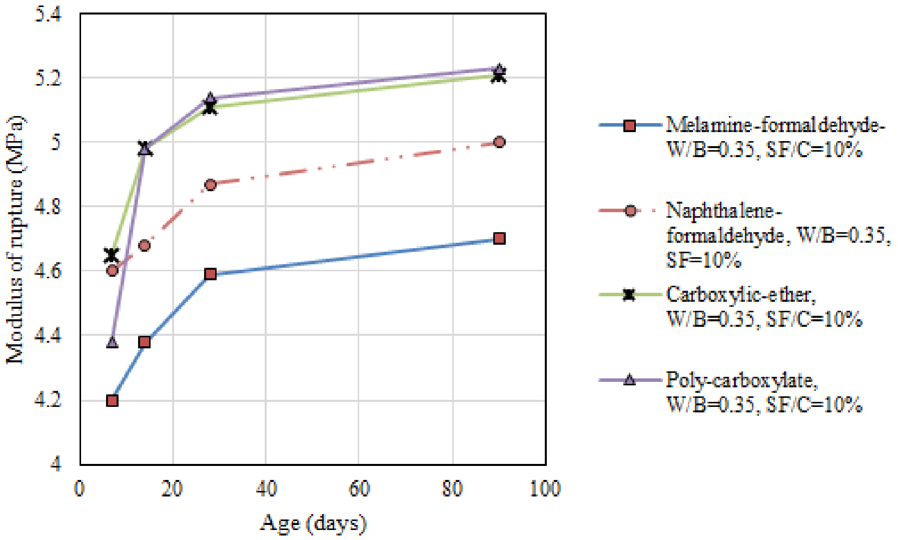

Fig. 20 Variation of rupture modulus with time in mixes with W/B=0.35 and SF/C=10%

procedure or construction stage. The application of silica fume did not strongly affect the rupture modulus of concrete and the effects were too negligible. The other important point which should be noted is the strength development of specimens. As it can be observed, in almost all specimens, the rate of strength development until the 28th day was more than 28th to 90th day.

In many of the design codes, a relation between the compressive and tensile strength have been proposed in order to be used in preliminary design purposes. In this regard, the well-known form $f_r = \alpha\sqrt{f_c}$ has been used and the results are reflected in Figs. 21 and 22.

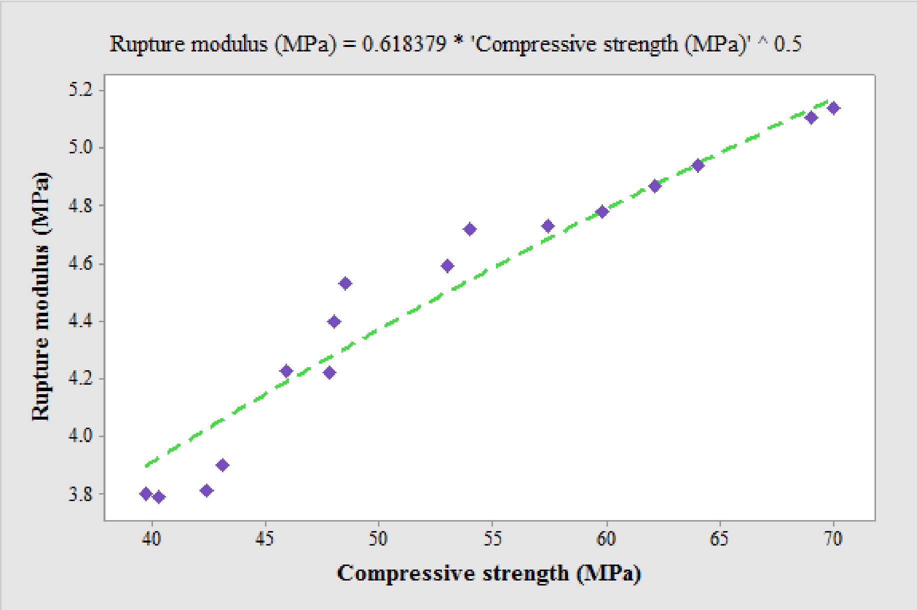

Fig. 21 Correlation between modulus of rupture and compressive strength

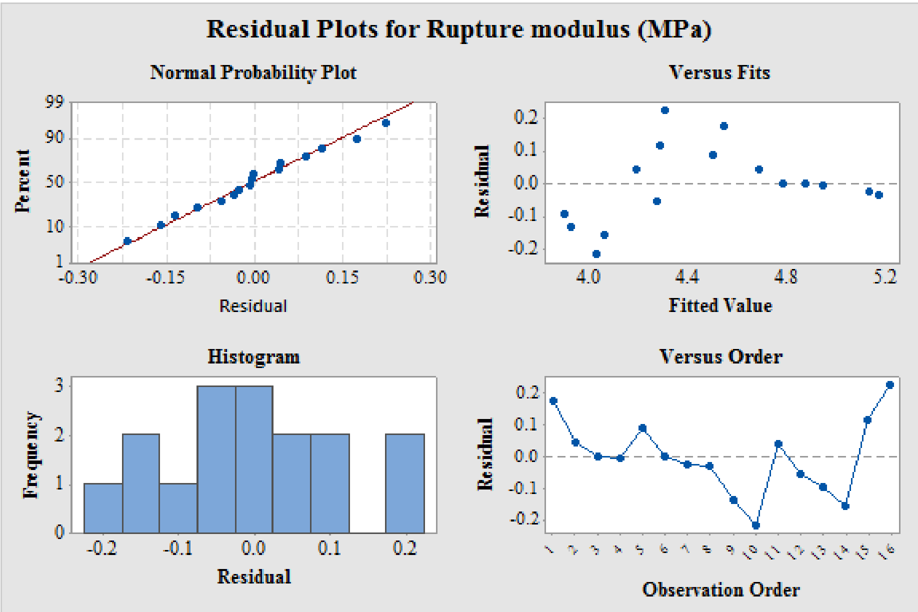

Fig. 22 Residual plots for the fitted curve of rupture modulus

5. Conclusions

From the results of this study the following conclusions could be drawn:

• The mixes made of poly-carboxylate-based superplasticizer showed the most compressive and tensile strength within the mixes made with four studied superplasticizer types.

- The early stage compressive and tensile strength of mixes with poly-carboxylic-ether based superplasticizer were better than those observed for the other mixes.
- Setting time is a key parameter especially in massive concrete structures. It could be observed that the final and initial setting time of pastes were affected by both the type and dosage of superplasticizers.
- As the superplasticizer contents increased, the air contents increased. The increase of air content could be harmful for mechanical behavior of concrete.
- Mixes made of melamine-formaldehyde based superplasticizers showed the most air contents and those mixes made from poly-carboxylate based superplasticizers showed the least amount of air. This finding should be integrated with both durability behavior and freeze-thaw resistance of concrete structures.
- The mixes made by poly-carboxylate had far better tensile and compressive strength than those mixes with melamine-formaldehyde.
- Utilization of silica fume decreased the air contents, enhanced the mechanical behavior and increased the bleeding and segregation resistance of the mixes.
- The mixes made of carboxylic-ether based superplasticizers showed better early stage mechanical properties. This feature turns these mixes into a volunteer to be used in cases in which there is a need to early strength.
- As the dosage of superplasticizer increased, the compressive strength and the rupture modulus decreased. Of course, it should be noted that the water/binder ratio could play an important role in this finding. In other words, in water/binder ratios lower than those studied in this paper, different behavior could be seen.

The effect of silica fume replacement on rupture modulus of concrete was negligible.

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