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Effect of utilization of different type of mineral admixture on fresh and hardened properties of cementitious systems

Ali Mardani-Aghabaglou*[1], Ece Geven[1], Ali Nematzadeh[1]

Abstract

In this study, the effect of mineral admixture utilization on fresh and some hardened state properties of cement paste and mortar mixtures were investigated. For this purpose, in addition to the control mixture containing no mineral admixture, three more series of mixtures were prepared. In the first and second mixtures 30% and 10 w.t% of cement was replaced with silica fume and fly ash respectively. In the third series, cement was replaced with both of silica fume and fly ash by 30% and 10%, respectively. According to test results, fresh state properties of paste mixtures were affected by mineral admixture use positively in general. However, a reverse result was observed for the fresh state properties of mortar mixtures. Compressive strength values of the mixtures including fly ash as well as the ones including silica fume and fly ash was found to be less than that of control mixture. Nevertheless, the difference between those values reduced at the end of 90 days. Mixture including silica fume showed the highest strength value from the beginning. 90-day water absorption values of the mortar mixtures were observed to be less compared to that of control mixtures with the use of mineral admixture. The mixture having ternary binder system (including cement, silica fume and fly ash) showed better performance in terms of hardened properties. The water absorption capacity value of mentioned mixture was observed as 42% less than that of control mixture.

Keywords: Mineral admixture, Marsh funnel, mini-slump, flow, compressive strength, water absorption capacity

1. INTRODUCTION

Chemical and mineral admixture use in concrete production became widespread with the development in concrete technology recently. It is almost impossible to produce concretes without admixture. Fly ash (FA), silica fume (SF), metakaolin (MK) and blast-furnace slag (BFS) are among the primary mineral admixtures with pozzolanic nature used for concrete production.

It is a known fact that mineral admixture uses in cemented systems increase the strength and durability of the system due to the physico-chemical effects. Since mineral admixtures are quite fine materials physically, they provide a more void-free structure by plugging of the voids. On the other hand, chemically, they convert calcium hydroxides (CH) into calcium silicate hydrates (CSH) which belong a stronger structure and acquire the property of being binder to the cemented systems resulted from pozzolanic reaction. Fineness of mineral admixtures, level of amorphism and chemical composition effect the pozzolanic reaction considerably. Thus, the age for pozzolanic reaction creation becomes earlier in case the mineral admixtures are fine and included reactive silica are high [1]. Some related studies are summarized below.

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Topçu et. al. investigated the effect of replacing different amounts of silica fume with CEM II/A-M 32.5 cement on mechanical behaviors of concrete mixtures [2]. Within this aim, cements weighing 5%, 10% and 15% replaced with silica furnace. According to the results, with the increase of replacement amount for silica furnace, mechanical properties of concrete mixtures were affected positively. However, that increased admixture requirement in order to provide a certain workability.

The fact that the mechanical properties were affected positively with the use of silica furnace was explained by the authors as the calcium hydroxides (CH) turning into calcium silica hydrates (CSH), lowering the voids and becoming a more void-free structure and creation of a stronger aggregate paste interface.

According to a study conducted by Aïtcin and Laplante [3], addition of silica fume as 5% and less showed no effect on water requirements of the concrete mixtures. However, the water requirement of concrete mixtures increased with the use of silica fume higher than that amount.

On the other hand, in another study, as a result of the experiments conducted with porosimeter device, it was determined that silica fume decreased the number of coarser pores as well as a large number of homogeneously spread finer pores occurred as a result of it. Total porousness of the microstructure was not affected by that situation. It was stated that the water was evaporated in a slower and more controlled way through the homogeneously spread finer pores. Thus, permeability of the mixtures with silica fume was less than that of the control mixture without silica fume [4].

Effect of mineral admixture use on performance of high-strength concrete mixtures was conducted by Hassan et. al. [5] in a similar study. Within this aim, three different mixtures were prepared with replacing 10% and 30% by weight silica fume and fly ash instead of portland cement. Early and advanced age compressive strength, void structure, oxygen permeability and chlorine-ion permeability of the produced concrete mixtures were investigated. According to the test results, mineral admixture use improved aforementioned properties of the high strength concrete mixtures in general. However, silica fume showed better performance compared to fly ash in terms of mineral admixture use. Effect of fly ash appeared in the advanced ages while silica fume improved both the early and advanced age properties.

In another study, the effect of using fly ash instead of various amounts of cement on mechanical properties of concrete mixtures was investigated. For this purpose, five different amounts of fly ash as 0% and 40% of cement weight were replaced. According to test results, the use of fly ash instead of 30% cement affected mechanical properties of concrete mixtures positively. The most successful result was observed to be provided by 20% fly ash replacement [6].

Güçlüer and Ünal [7], in another study, investigated the effect of using fly ash instead of 10%, 20%, 30% cement on 7, 28 and 56-day compressive strength, ultrasonic pulse velocity and 60-day permeability properties of concrete mixtures. Water/cement (w/c) ratios and cement contents of all mixtures were stabilized as 0.5 and 300 kg/m^3. According to test results, with the use of fly ash up to 20% distinct improvements were observed in the mentioned properties of concrete mixtures. Compressive strengths reduced while ultrasonic pulse velocity and permeability properties were affected positively by replacing fly ash above that amount. Those reductions in compressive strength were also observed in 56-day specimens. Similar results were obtained by Delikurt and Sevim [8].

The effect of replacing cement with fly ash, silica fume and metakaolin on compressive strength, dynamic elasticity module, chlorine-ion penetration, water absorption, capillary absorption, freeze-thaw and sulfate resistance of mixtures were investigated comparatively. Moreover, microstructure analysis was realized on some selected mixtures. In addition to this, regression analysis was conducted on sulfate resistance test results. Authors stated that existence of different mineral admixture types in mortar mixtures changed the ettringite morphology. It was observed that ball type and a special type ettringite occurred in the mixtures with silica fume and metakaolin. Needle-like and ball type ettringites occurred in the mixtures containing fly ash. In control mixtures, on the other hand, needle-like, ball type and massive type ettringites were determined to be occurring. Test results showed that the range of mixtures in terms of their performance is as follows: the ones with silica fume, metakaolin, fly ash and control mixture, respectively [9].

Pandey and Sharma [10] revealed that 7 and 28-day pore distributions of the fly ash replaced mortar mixtures were more than that of the control mixture without fly ash. However, they also stated that the pores decreased with the increase of pozzolanic reactions during 90 days and more.

Reza Saleh et. al. [11] conducted a study related to the change of time-dependent rheological properties of the self-consolidating concrete (SCC) mixtures containing different mineral admixtures. For this purpose, 17 SCC mixtures were prepared in total by using different amounts of SF, MK and F type FA and C type FA and BFS as binary and ternary cementitious systems. Slump-flow values in all SCC mixtures were stabilized. T_{50} flow time, plastic viscosity, visible critical shear stress and thixotropic properties of the produced SCC mixtures were investigated. According to test results, compared to the other mixtures, those containing F type and C type FA and BFS showed more tendency to segregation and bleeding. Critical shear stress and thixotropy of the mixtures increased significantly by time. Nevertheless, time dependent plastic viscosity values showed no significant change. MK and C type FA affected the shear stress and time dependent change of plastic viscosity more than that of other mineral admixtures.

In another study, the effect of magnesium sulfate concentration on microstructure and strength properties of light weight mortars including fly ash and silica fume was investigated. Within this aim, in addition to control mixture without mineral admixture, mixtures including 15% FA and 10% SF of total concrete weight was prepared. After 28-day of curing period, the specimens were exposed to different sulfate concentrations for a whole year. As a result of the conducted microstructure analyses, it was found out that degradation mechanism of the exposed light mortars changed according to magnesium sulfate concentration percentage and mineral admixture type. Moreover, compressive strength of the mixtures with SF were observed to be more than those of control and FA mixtures [12].

As it can be understood from the literature, a large number of various studies were conducted regarding the effect of utilization of mineral admixtures on the properties of cementitious systems. In the mentioned studies, the effect of the use of mineral admixtures was generally examined in the mixtures having plain and binary binder systems. However, in this study, in order to understand the influence of the matrix type, in addition to the use of fly ash and silica fume as binary binder systems, the effect of their usage as ternary binder systems on mortar mixture properties was investigated as well. Besides, it can be understod from the literatur survey, the effect of mineral admixture use was generally examined in mortar and concrete mixtures. There are not many studies on the effect of the mentioned admixtures on the fresh state properties of the paste mixtures. However, in this study, in addition to investigating the effects of the use of these additives on the properties of mortar mixtures, the effect of the use of different mineral admixtures on the Marsh-funnel flow time and mini-slump values of the paste mixtures was investigated. Within this aim, mixtures containing mineral admixtures were prepared within different combinations and their performances were observed comparatively with the control mixture without mineral admixture. It was aimed to determine the mixtures showing the required behavior in terms of both fresh and hardened state properties. In order to provide the required flow value within all of the mortar mixtures a polycarboxylate-ether based high range water reducing admixture was used. The effect of mineral admixtures on the compatibility between cement and water reducing admixture was investigated. Besides, compressive strength and water absorption capacity results of the mortar mixtures was compared in terms of the mineral admixture type.

2. EXPERIMENTAL STUDY

2.1. Materials

In this study, CEM I 42.5 R type portland cement conforming to TS EN 197-1 standard [13] and different amounts of fly ash (FA) and silica fume (SF) were used as binder and mineral admixture, respectively. CEN Standard sand conforming to TS EN 196-1 [14] was used as aggregate. Chemical composition and physical properties of the cement, fly ash and silica fume provided from their manufacturers are shown in Table 1 and 2, respectively. Grain size distribution of the standard sand is revealed in Table 3. Specific gravity and water absorption capacity of the aggregate were determined as 2.72 and 0,7% by mass, respectively in accordance with TS EN 1097-6 standard [15]. All the flow values of the mortar mixtures were stabilized. In order to provide the required flow value, a polycarboxylate-ether based high range of water reducing admixture properties of which were shown in Table 4 were used.

Table 1. Chemical composition, physical and mechanical properties of cement.

Chemical properties	
Oxide	**(%)**
SiO_2	18.86
Al_2O_3	5.71
Fe_2O_3	3.09
CaO	62.70
MgO	1.16
Free CaO	1.26
Na_2O+0.658 K_2O	0.92
Cl^-	0.01
SO_3	2.39
LOI	3.20

Mechanical and physical properties		
Properties		**Cement**
Compressive strength (MPa)	1-Day	14.7
	2-Day	26.80
	7-Day	49.80
	28-Day	58.5
Fineness	Blaine Specific Surface (cm^2/g)	3530
	Residual of 0.090 mm sieve (%)	1.2
	Residual of 0.045 mm sieve (%)	7.6

2.2. Mix proportion

In this section, properties of the produced cement paste and mortar mixtures were explained in detail. In this study, the effect of using silica fume and fly ash instead of different amounts of cement on cement paste and mortar mixtures was examined. Within this aim, in addition to the control mixtures without mineral admixture, three different paste and mortar mixtures with binary and ternary binding systems were prepared. In the first and second series of binary binder systems, 30% and 10% fly ash and silica fume by weight replaced with cement. In the paste and mortar mixtures with ternary binder systems, on the other hand, fly ash and silica fume as 30% and 10% by cement weight were used, respectively. In the control mixtures with single binder, portland cement was used as binder. Denomination of the mixtures were realized in terms of included mineral admixture type. Control mixture with single binder system and excluding mineral admixture, for instance, was shown with (C). The mixture with ternary binder system including both fly ash and silica fume was called as C-FA-SF.

Table 2. Chemical composition and physical properties of fly ash (FA) and silica fume (SF).

	FA	**SF**
Physical properties		
Specific gravity	2.31	2.41
Blaine Specific surface (cm^2/g)	4300	3520
Puzolanic Activity Index (%)		
7-day	70.9	96.08
28-day	77.7	102.73
90-day	91.2	103.70
Chemical properties	**FA**	**SF**
Item	**(%)**	
SiO_2	59.22	76.70
Al_2O_3	22.86	2.22
Fe_2O_3	6.31	0.84
$SiO_2 + Al_2O_3 + Fe_2O_3$	88.39	79.76
MgO	1.31	12.37
Na_2O	0.41	1.79
K_2O	1.51	1.55
SO_3	0.17	0.20
CaO	3.09	0.55
Free CaO	0.00	0.14

Table 3. Particle size distribution of CEN reference sand.

Suare mesh size (mm)	Residual of sieve (%)	Cumulative sieve residue (%)
2.00	0	0
1.60	4.32	7 ± 5
1.00	33.98	33 ± 5
0.50	67.11	67 ± 5
0.16	86.85	87 ± 5
0.08	99.83	99 ± 5

Table 4. Properies of polycarboxylate-ether based high range of water reducing admixture properties.

Properties	Analysis Results
Color	Brown
Relative density (g/cm^3)	1.097
Amount of solid matter (%)	36.35
Ph	3.82
Chloride content	<0.1
Alkali amount (Na_2O)	<10

Preparation of Cement Paste Mixture

W/C ratio of the paste mixtures were selected as 0.35 for the Marsh funnel and mini slump tests considering the previous studies [16]. Within this aim, 7 different paste mixtures were prepared by using different amounts of high-range water reducing admixture (HRWR) as between 0.5% and 2% by cement weight for each series.

Preparation of Mortar Mixture

Mortar mixtures were prepared in accordance with ASTM C109 [17] Standard. Water/binder (w/b), sand/binder (s/b) ratios and flow values in all mixtures were fixed as 0.485, 2.75 and 270 ± 20 mm, respectively. As it was stated previously, a polycarboxylate-ether based high range water reducing admixture was used to provide the required flow value. Mixtures were prepared in Hobart mixer homogeneously. Mix proportion of mortar mixtures are shown in Table 5.

Table 5. Mix proportion of mortar mixtures (g) and spreading values.

	Series name			
	K	**UK**	**SD**	**K-UK-SD**
Cement	500	350	450	300
FA	0	150	0	150
SF	0	0	50	50
Water	242.5	242.5	242.5	242.5
StandardSand	1375	1375	1375	1375
HRWRA*	2.5	2.1	2.8	2.9
Flow value, mm	265	265	270	270

*High range water reducing admixture.

2.3. Test Procedures

Paste Mixtures

Marsh funnel flow time and mini slump flow value of the paste mixtures were carried out in accordance with the method proposed by Aïtcin [16] and Kantro [18]. The Marsh-funnel flow time and mini-slump value measurement of paste mixtures are shown in the Figures 1 and 2, respectively. Prepared paste mixtures were poured into a standard Marsh funnel. Time of the flow for of 700 mm paste mixture from the funnel was measured. The measured time was recorded as Marsh funnel flow time. The prepared paste mixture for Marsh funnel flow time was also used for mini-slump test. In this testing method, paste mixture was filled in a frusto-conical mold placed in the center of a smooth surface with bottom inner diameter as 38.1 mm, top inner diameter as 19 mm and height as 57.2 mm [18]. Slump mold is removed vertically and slowly and it is waited for a while (15-20 sec) in order for the completion of flow. Afterwards, two perpendicular flow diameters were measured and the mean of them was calculated and recorded.

Figure 1. Marsh-funnel flow time measurmnet of cement paste mixtures

Figure 2. Measurement of mini-slump values of cement paste mixtures

Mortar Mixtures

Flow values of mortar mixtures were measured in accordance with ASTM C1437 standard [19] in every 20 minutes during an hour. Moreover, the 3, 7, 28 and 90-day compressive strengths of mentioned mixtures were determined on 50 mm cubic specimens in accordance with ASTM C109 standard [17]. Besides, 90-day water absorption capacity of mortar mixtures were obtained in accordance with ASTM C642-13 standard [20] over 50 mm cubic specimens. In this experiment, the specimens reserved in water for 90 days were moved out of water, the surface was toweled and the saturated surface dry weight (b) was calculated. Thereafter, they were dried up to a stable weight in a drying oven with 105°C temperature and their weight (a) were measured. Water absorption of mixtures (m) was calculated according to Equations 1.

$$m = \left[(b-a)/a \right] \times 100 \qquad \text{[Eq. 1]}$$

The measurement of flow value, compressive strength and water absorption capacity of mortar mixtures were shown in Figure 4-6, respectively.

Figure 3. Measurement of flow values of mortar mixtures

Figure 4. Measurement of compressive strength of mortar mixtures

Figure 5. Drying process of mortar mixtures in the oven for water absorption measurement

3. RESULTS

3.1. Fresh State Properties

Test Results of Paste Mixtures

Results of Marsh funnel and mini-slump tests conducted on paste mixtures are shown in Table 6 and Figure 6. As it can be understood from the results, Marsh funnel flow times of the mixtures decreased as expected with the increase of water reducing admixture independently from the mineral admixture use. However, when the amount of water reducing admixture increased above a certain amount, no significant change was observed in terms of flow times of the cement pastes. Aforementioned water reducing admixture dosage is defined as saturation point for cement-admixture pair [21]. The saturation points of water reducing admixture in all mixtures was realized when it is 0.75% of water reducing admixture/cement ratio. As can be understood from the results, mineral admixture use showed no effect on saturation point of the admixture. Nevertheless, considering the Marsh funnel flow time at the saturation point, flow times of the mixtures with fly ash was observed to be 12% higher than that of the control mixture. A reverse situation was concluded from the mixtures including silica fume. Since the silica fume is finer than that of cement, the workability of the mixture was affected negatively. However, flow times of the mixtures with silica fume are measured as 40% less than that of control mixture. Total fine material amount in the mixture increases with the use of silica fume. It is considered that the decrease of Marsh funnel flow time with the increase of total fine material amount in the mixtures is resulted from the fact that water reducing admixture is possibly to be absorbed on fine grains more easily. Similar results were also stated by Ali Mardani-Aghabaglou et. al. [21]. Researchers used a portland cement within the mentioned study. It was declared by the authors that Marsh funnel flow times reduced with the increase of cement fineness. That effect is expressed by the authors

as the admixture is to be able to be absorbed more easily on finer grains when the water reducing admixture exists in a sufficient amount within the cemented system. However, it was found out by the authors that the workability of the mixtures decreased with the increase in cement fineness including less water reducing admixture. A more fluent and sufficient amount of mixture is required to be obtained through the Marsh funnel test considering the testing method.

Table 6. Results of Marsh-funnel and mini-slump tests conducted on paste mixtures

Admixture/Cement ratio (%)		**0.50**	**0.75**	**1.00**	**1.25**	**1.50**	**1.75**	**2.00**
Flow time, Sec.	**C**	84.7	68.5	64.2	63.6	63.9	65.8	67.3
	FA	93.9	77.6	76.8	77.1	80.8	86.5	90.4
	SF	61.2	40.9	36.5	37.8	38.1	38.6	38.5
	C-FA-SF	60.4	43.2	43.1	43.2	44	45.5	47.1
Mini-slump, mm	**C**	175	200	195	185	190	200	200
	FA	170	180	190	180	185	175	185
	SF	150	190	200	205	200	210	195
	C-FA-SF	160	170	180	185	185	185	195
Temperature, °C	**C**	29.3	28.8	27.6	27.3	27.5	27.4	27.9
	FA	32.3	31.6	31.4	29.5	29.1	28.4	27.6
	SF	32.3	31.5	30.7	30.2	29	29	27.9
	C-FA-SF	34.7	32.5	31.7	30.4	30.2	29.4	29.2

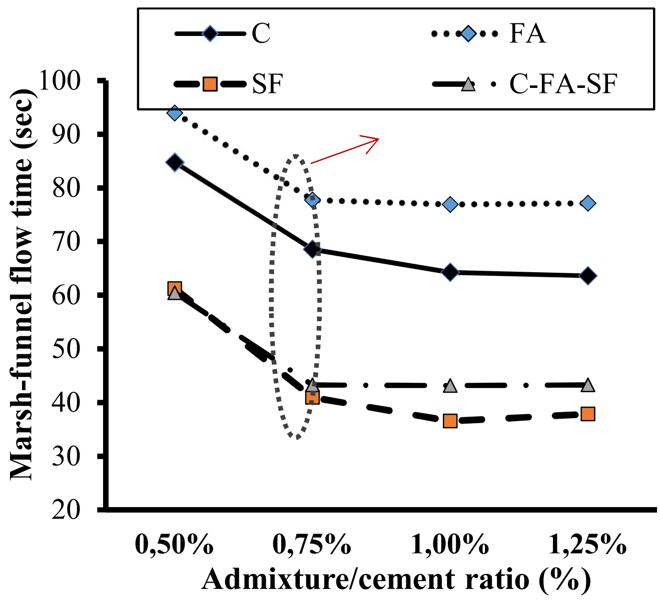

Figure 6. Marsh-funnel flow time values of the paste mixtures varying amounts of admixture.

Considering mini-slump values of paste mixtures, mini slump values of the mixtures reduced with the use of water reducing admixture independently from mineral admixture use. That is resulted from the decrease of critical shear stress values of the mixtures with the use of water reducing admixture [22]. Nonetheless, flow values of the mixtures performed no change over a certain amount of admixture use. Similar results with Marsh funnel were observed when the mini flow values at the saturation points of paste mixtures were compared. Regarding the temperature change in the mixtures, with the use of mineral admixture and increase of admixture amount, no significant changes were observed in terms of temperature.

Test Results of Mortar Mixtures

The time-dependent flow values of the mortar mixtures are demonstrated in Table 7 and Figure 7. As expected, independently from mineral admixture use, flow ratios of the mortar mixtures decreased by time. As also emphasized before, flow values of all mortar mixtures were stabilized as 270 ± 20 mm. It can be seen in Table 5 that in order to provide the mentioned flow values, admixture requirement increased in SF mixtures including silica fume as 10% compared with the control mixture. However, within the FA mixture including 30% fly ash, the required flow value was provided by using 15% less admixture than that of the control mixture. As it can be understood from the results, in comparison with the control mixture, even though the admixture requirement decreased as 15% in order to provide the required flow value in the FA mixture including 30% fly ash, 7% more flow loss was observed at the end of 60 minutes. Including both fly ash and silica fume, C-FA-SF mixture with ternary binder system belonged 15% more flow loss compared to that of the control mixture at the end of 60 minutes. It can be observed from the results that the mineral admixture uses in general affected the flow value of the mixtures negatively. That negative effect became more obvious by time. The effect of mineral admixture use on flow values of mortar mixtures is contradictory with that of Marsh funnel results of paste mixtures. As also stated in the section related to results of paste mixtures, when the water reducing admixture exists sufficiently, the negative effect of increase in fine material amount on fresh properties was not considerably dominating.

As predicted, since the amount of fine material increased with the mineral admixture use, workability of the mixture was affected negatively.

Table 7. Time-dependent flow values of the mortar mixtures (mm).

No	0 min	20 min	40 min	60 min
C	257.5	250	240	225
FA	267.5	257.5	240	240
SF	277.5	267.5	260	250
C-FA-SF	270	270	245	220

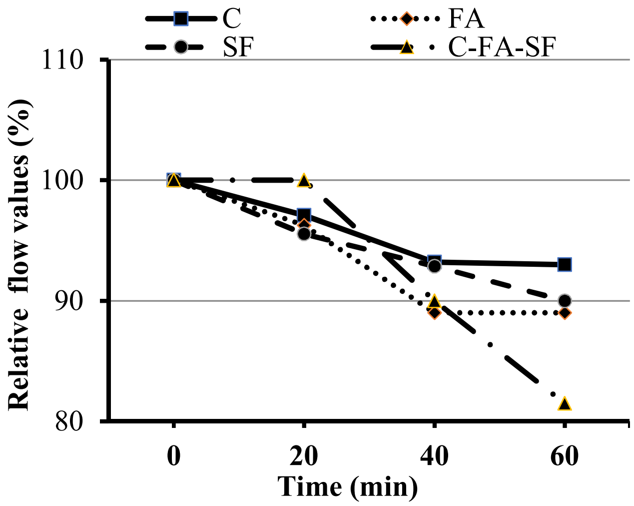

Figure 7. Time-dependent flow values of the mortar mixtures

3.2. Hardened State Properties

3.2.1. Compressive Strength

The 3, 7, 28 and 90-day compressive strength results of mortar mixtures are shown in Figure 8. Each value was obtained as the average of three measurements. As it can be understood from Figure 4, compressive strength of FA mixture including 30% fly ash was less since the beginning compared to the control mixture. A similar negative effect was also observed in 90-day specimens. However, the difference between strengths is observed to be decreasing by time. SF mixture including silica fume showed the highest compressive strength from the beginning. Nevertheless, even though it showed less strength than that of control mixture from the beginning, including both fly ash and silica fume, C-FA-SF mixture showed similar results with control mixture at the end of 90 days.

Since the silica fume carries high pozzolanic activity index value, it affected compressive strength positively. However, its performance on strength was lower than that of silica fume as pozzolanic activity index of fly ash used in this study is less than fly ash.

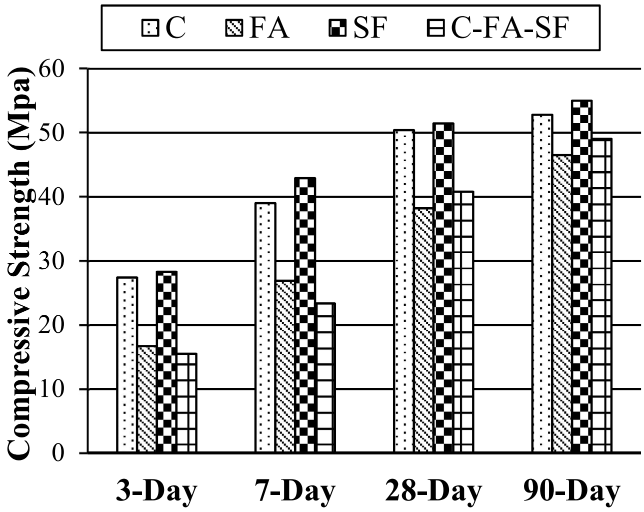

Figure 8. Compressive strength of mortar mixtures (Mpa)

3.2.2. Water Absorption

The 90-day water absorption ratio of mortar mixtures is shown in Figure 9. Each result is calculated as average of three measurements. As it can be seen from the results, 90-day water absorption capacity of the mortar mixtures including mineral admixture are less than that of control mixture. Including both fly ash and silica fume C-FA-SF mixture is determined as the most successful admixture in terms of water absorption performance. Aforementioned mixture is 42% less permeable compared to that of control mixture. It was previously emphasized that the positive effect of mineral admixture on water absorption ratios resulted from physico-chemical effect of mineral admixture.

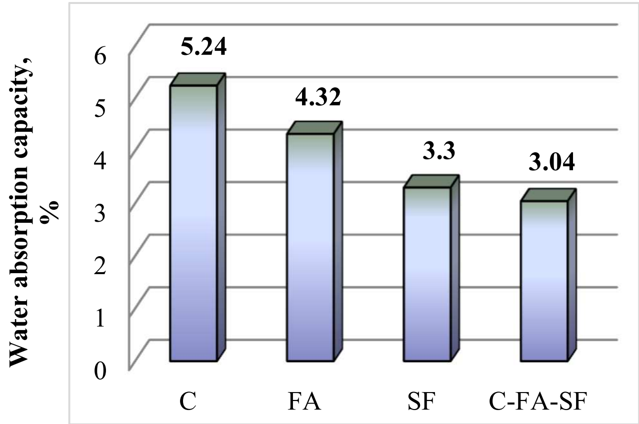

Figure 9. The 90-day water absorption capacity of mortar mixtures.

4. CONCLUSION

As a result of the conducted experimental study, following findings are concluded.

- ✓ It was revealed that Marsh funnel flow time of cement paste was affected positively in case sufficient amount of water reducing and very fine mineral admixture use was provided. This effect is thought to be resulted from the increasing number of finer materials for the mixtures with silica fume as a result of the fact that water reducing admixture could be more easily absorbed on grains. Mini-flow results of the mixtures were similar with the Marsh funnel results.

- ✓ Time-dependent flow values of the mortar mixtures was affected negatively as a result of mineral admixture use. Workability of the mixtures decreased with the increase of fine material amount.

- ✓ Compressive strength values of the mixture including fly ash at all ages were less than the control mixture values. That behavior was also observed even in the 90-day specimens and the difference between values reduced by time. The mixture including 10% silica fume showed the highest strength since the beginning compared to control mixture. While a lower strength was observed in the mixture with both fly ash and silica fume at early ages than control mixture, similar results with the control mixture was observed at the end of 90 days. Irrespective from the mineral admixture type, 90-day water absorption values were less than that of the control mixture. Within this frame, the most successful result was observed in the mixture with ternary binder including both fly ash and silica fume. That mixture showed 42% less water absorption performance than that of control mixture.

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