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A study on the effect of fly ash and silica fume substituted cement paste and mortars

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Nowadays, mineral additives due to the advantages such as evaluation of waste for ecological balance, improving the physical and mechanical properties of cement or concrete a wide field of use are found. Two of the waste materials are silica fume and fly ash. This study aims to determine mutual influence of fly ash and silica fume with Portland cement. For this purpose, physical, chemical, mineralogical and molecular were applied to these raw materials. The results indicated that fly ash and silica fume have shown different surface features compared to Portland cement. These variations effected on compressive strength of mortar samples. The ternary use of fly ash and silica fume provided the best performance, when the compressive strength properties of the cement mortars were taken into account.

Key words: Portland cement, fly ash, silica fume, FT-IR, XRD, setting time, water demand, compressive strength.

INTRODUCTION

Various types of pozzolanic materials that improve cement properties have been used in the cement industry for a long time (Drazan and Zelic, 2006). Pozzolanic materials are either natural (Trass, zeolite, volcanic tuff, metakaolinite, burned clay and so on) or artificial (silica fume, fly ash, blast furnace slag and so on) in origin (Yilmaz and Olgun, 2008). The use of additional cementitious materials due to economic, technical and environmental considerations has become very common in modern concrete construction (Fu et al., 2002; Worrell et al., 2000). A number of studies have been conducted on the durability and strength of concrete made with mineral admixtures (Ganjian and Pouya, 2009; Subaşı, 2009; Yildiz et al., 2007; Kaid et al., 2009).

Fly ash (FA) is waste materials from the thermal power plant; it is separated from the flue gas of the power station burning pulverized coal. According to ASTM C618, there are two basic types of FA: Class F (low-calcium FA) and Class C (high-calcium FA). Its physical and chemical properties depend exclusively on the quality of coal used and on technological conditions of burning (Drazan and Zelic, 2006). FA is added to Portland cement (PC) or directly to mortars or concretes (Aruntas, 2006). The use of FA as a replacement addition to cement in production is useful for a number of purposes. FA use partially dis-

places production of other concrete ingredients, resulting in significant energy savings, reductions in CO₂ emission and conserving resources (Perez et al., 2009; Ahmaruzzaman, 2009). FA makes substantial contributions to workability and chemical resistance. It is widely accepted that, they reduce the hydration heat, and block the alkali-silica reactions (Saraswathy et al., 2003). FA, when used as mineral admixtures in high performance concrete, can improve both the strength and durability properties of the concrete (Garces et al., 2009; Wang et al., 2008).

Silica fume (SF) is a by-product resulting from the reduction of high-purity quartz with coal in electric arc furnaces in the manufacture of ferrosilicon alloys and silicon metal (Neville, 2006). It has been shown that in some cases, the presence of such particles can reduce the positive effect of the SF on the microstructure and mechanical properties of the pastes (Sanchez and Ince, 2009; Mazloom, 2004). The use of SF decreases the permeability, thereby increasing the resistance of concrete against corrosion (Jo et al., 2007; Qing et al., 2007; Kelestemur and Yildiz, 2005), improving its strength and durability (Simsek et al., 2005; Song et al., 2010; Yildiz, 2008). The presence of silica fume agglomerates can result in matrix expansion due to the

Table 1. Chemical specifications of used materials.

Materials	PC (%)	FA (%)	SF (%)
Chemical composition: wt.%			
SiO ₂	21.82	53.39	78.50
Al ₂ O ₃	6.49	16.07	1.22
Fe ₂ O ₃	1.93	13.05	1.27
CaO	60.74	6.33	2.13
MgO	1.08	5.48	5.32
SO ₃	2.62	1.06	0.15
Na ₂ O	0.14	1.59	1.78
K ₂ O	0.65	1.71	4.11
Cl ⁻	0.012	0.005	0.036
Loss on ignition (LOI)	1.65	1.15	4.93
Free CaO	0.84	0.11	-
Reactive SiO ₂	-	45.18	76.2

alkali-silica reactions (Maas et al., 2007). The advantages of SF caused SF being the most well-known additive material for height strength concrete in recent years.

However, little information is presently known regarding to other properties of the FA-SF combination, such as its fresh properties (setting time, compressive strength and so on of the ternary cement), and the reaction mechanism of SF in the FA system. This work investigates ternary blends of FA, SF and PC using an extensive range of mixes. The properties of FA, SF and FA-SF blended cement mortars were examined in accordance with the standard cement tests. Mineralogical structures of raw materials with X-ray diffraction (XRD) were investigated. Moreover, molecular structures of raw materials with Fourier transform infrared spectroscopy (FT-IR) were investigated.

MATERIALS AND METHODS

Materials

The raw materials used in this study were PC, FA, SF standard aggregate and water. The PC was CEM I 42.5 R in accordance with TS EN 197-1, which was provided from Bursa Cement Plant; (Turkey). FA was participated in the production of this cement as minor additional components. FA was obtained from the Seyitömer Thermal power plant in Kütahya (Turkey). SF was obtained from Antalya Etibank (Turkey) elektro-ferrochrome business. CEN standard aggregate which was produced by SET Trakya Cement industry in accordance with TS EN 196-1 and Bursa-Kestel province tap water were used in the preparation in the cement mortar.

Methods

In the study, a total of eight different mixtures are obtained with PC

being the reference. The amount of PC is reduced by 10, 20 and 30% by weight being substituted by the same amount of FA and denoted by 10, 20 and 30FA, respectively. Similarly, the amount of SF substitution is 5 and 10% denoted by 5 and 10SF, respectively. Besides, in order to investigate the properties of ternary mixtures, the amount of PC is reduced by 10 and 20% by weight and substituted by equal amounts of FA and SF denoted by 5FA5SF and 10FA10SF, respectively. Chemical, physical, XRD and FT-IR analyses are conducted for the samples used in the experiments. Water/cement ratio is 0.5 in all cement pastes and mortars.

Chemical analyses of PC, FA and SF are performed on ARL 9900 X-ray workstation (XRF+XRD). Malvern Hydro 2000 G is used in the particle size analyses. Surface areas are determined as Blaine values by Toni Technik 6565 Blaine and specific weights are determined by Quantachrome MVP-3. The mineralogical properties are determined by Rigaku miniflex XRD device using Cu K_α (λ=1.54 Å) radiation. FT-IR analyses are conducted using Bruker Vertex 70. The samples are mixed with KBr in powder form and measured in the wave number range of 400 - 4000 cm⁻¹. Cement pastes and mortars are prepared according to TS EN 196-1. Water demand and setting time of cement paste samples are determined according to TS EN 196-3 by taking the average of 3 experiments. Water demand and setting time of each cement paste is determined using Atom Technik Vicat ring, probe and needle apparatus. The mentioned processes are carried out in a laboratory environment with 20°C temperature and 65% relative humidity.

In the preparation of mortar mixtures for compressive strength experiments, 450 g of cement, 1350 g of standard sand and 225 ml of water are used in each mortar mixture according to TS EN 196-1 and mixed in mortar mixer machine. Prepared mortars are poured into three-segmented rectangular prism moulds of size 40 x 40 x 160. Prepared samples are waited in the laboratory for 24 h. At the end of 24-h period, the samples are taken out of the moulds and waited in water pools to get cured and prepared for the compressive strength experiments. Compressive strength of each cement mortar is measured at the end of 2, 7, 28, 56 and 90 days using Atom Technik device and determined by taking the average of 6 samples.

EXPERIMENTAL RESULTS AND DISCUSSION

Chemical analysis

The results of the analyses demonstrating the chemical properties of the materials used are shown in Table 1. According to the chemical analysis results, PC consists of CaO with higher proportion and Al₂O₃, Fe₂O₃ and SO₃ compounds with lower proportion. The main component of FA is SiO₂.

Since the amount of S+A+F is above 70% according to ASTM C with a value of 82.51% and the amount of CaO is less than 10%, FA goes in Class F (low calcium). Besides, it goes in Class V (silica-like FA) according to TS EN 197-1 on the ground that the amount of reactive lime is less than 10 (5.58%). V Class FA satisfies all conditions since the amount of reactive silica is above 25 (45.18%). The main component of SF is SiO₂. The mineralogical structure of PC according to Bogue formula based on the chemical analysis is not calculated due to the use of FA as the additional minor component. The reason is that, the addition of FA alters the chemical composition of cement considerably with respect to clinker.

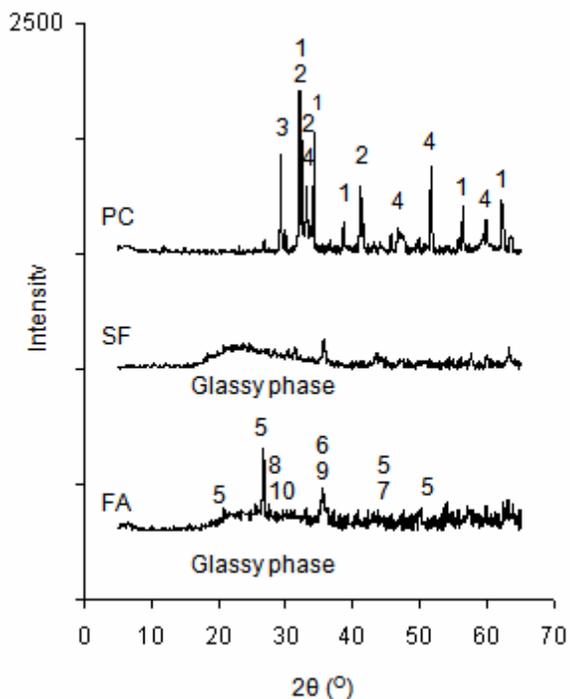


Figure 1. X-ray diffraction patterns of PC, FA and SF.
Note: 1: tricalcium silicate, 2: dicalcium silicate, 3: tricalcium aluminat, 4: brownmillerite, 5: quartz, 6: hematite, 7: mullite, 8: albite, 9: magnesioferrite, 10: sanidin.

XRD analysis

XRD analyses are conducted to determine the mineralogical structure of PC, FA and SF (Figure 1). According to the XRD analyses, the main components of PC are tricalcium silicate (alite), dicalcium silicate (belite), tricalcium aluminat and brownmillerite. It is observed from the XRD analysis of the mineralogical composition of FA that amorphous and crystal phases are formed. While the aluminum silicate present in FA forms mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), SiO_2 in the structure which is also present in quartz form. Iron, on the other hand, is present as hematite (Fe_2O_3) mineral. K^+ element in the structure comes from a feldspar mineral, sanidine. Also, it is seen that the amorphous phase reaches its maximum when 2θ is between $20 - 35^\circ$. Therefore, it is understood that the amorphous phase has silica-like characteristics since it is close to the maximum peak value of quartz crystal. According to the XRD analyses of SF, it can be stated that it has a denser amorphous structure when compared to FA (Figure 1). A general overview of XRD results yields that FA and SF have amorphous mineralogical structures whereas, PC has crystal mineralogical structure.

FT-IR analysis

FT-IR analyses can be used to define molecule groups in

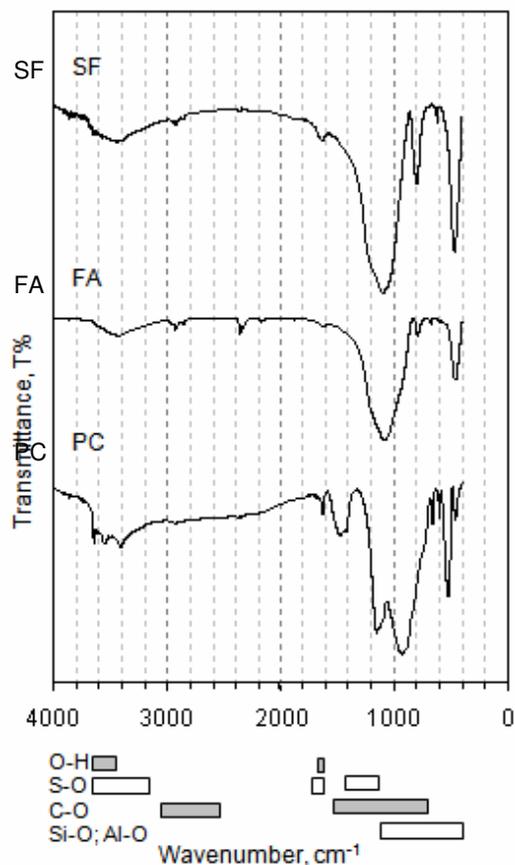


Figure 2. FT-IR spectrums of PC, FA and SF.

a particle. In the FT-IR study about cement and pozzolana conducted on this purpose, the infrared spectrum is considered in mainly 4 wide band regions. They are composed of peaks corresponding to the deviations in Si-Al, S, C and OH bonds (Varast et al., 2005; Puertas and Fernandez-Jimenez, 2003). Besides, the difference in the number of vibrations in this wave length can be evaluated locally. Surface structures of the molecules are determined from the FT-IR results obtained from the analyses and shown in Figure 2 schematically.

In FT-IR spectroscopy, vibration of the atoms forming solid cages and molecular vibrations are seen in $400 - 1600 \text{ cm}^{-1}$ and $1600 - 4000 \text{ cm}^{-1}$ region, respectively. Vibration peaks are observed at 418, 462, 523, 601, 661, 926, 1151, 1475, 1621, 3404, 3560 and 3643 cm^{-1} wave numbers from FT-IR analysis of PC (Figure 2). Al-O bonds present with Si-O give vibration peaks of 418, 462 and 523 cm^{-1} . Si-O bonds in cage structures are in the form of a vibration peak at 926 cm^{-1} wave number. Sulfur-oxygen (S-O) bonds which show the plaster in PC is seen at 601, 661, 1151 and 1621 cm^{-1} . CO_3^{2-} , on the other hand, is observed at 1475 cm^{-1} . Vibration peaks of water ions and molecules in the structure are at 3404, 3560 and 3643 cm^{-1} wave numbers (Gomes and Ferreira, 2005;

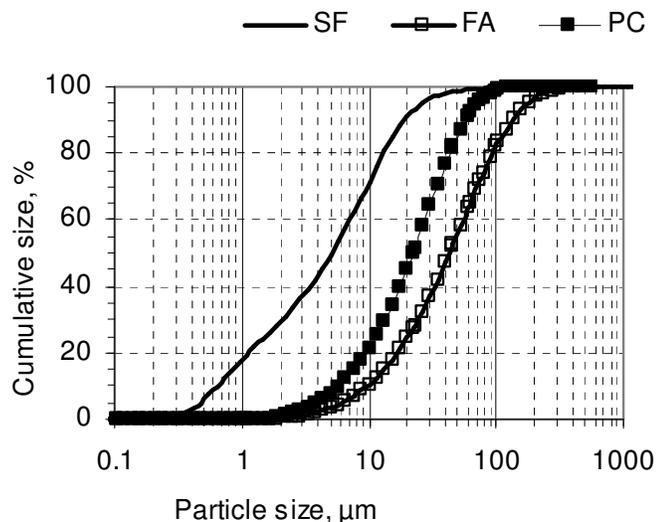


Figure 3. Particle size distributions of PC, FA and SF (under sieve).

Gomes et al., 2005; Govin et al., 2006).

FT-IR spectroscopy of FA indicates rare presence of absorbed water and alumina silicate. O-Si-O and O-Al-O bonds seen in the absorption region of 1083 cm^{-1} wave number are in the form of asymmetric vibrations. Largest area under the peak curve is in this region. The peak at 796 cm^{-1} wave number indicates the presence of quartz. Although, the mullite phases are generally observed at 1138 cm^{-1} , a strong peak occurs at 1083 cm^{-1} wave number. Hematite phases, on the other hand, occur at 463 cm^{-1} wave number. Small peaks at $500 - 650\text{ cm}^{-1}$ wave number indicate silicate and alumina silicate phases. Water bonded to the structure with hydrogen bridges is detected at the peaks in 3431 cm^{-1} region (Figure 2) (Drazan and Zelic, 2006; Yu-Fen et al., 2006).

Characteristic vibration peaks are observed at 477, 618, 804, 1109, 1645 and 3442 cm^{-1} wave numbers according to the FT-IR analysis results of SF (Figure 2). Si-O bonds at 477, 618 and 804 cm^{-1} wave numbers are in the form of asymmetric vibrations. Si-O-Si bonds observed in the absorption region at 1109 cm^{-1} wave number which is the largest area under the peak curve are in the form of asymmetric tension vibrations. Absorbed water in O-H-O molecules at 1645 cm^{-1} wave number is in the form of bending vibrations. Water bonded to the structure with hydrogen bridges is detected at the peaks in 3442 cm^{-1} region (Figure 2) (Hayakawa and Hench, 2000).

Physical analysis

Particle size distribution of PC, FA, SF and the physical properties of the materials used in the study are shown in Figure 3 and Table 2, respectively. Particle size, particle dimension distribution, Blaine value and specific weight are determined in the physical analyses. Particle size,

Table 2. Physical properties of cement mixture samples (wt.%).

Mixtures	Range dimension (over sieve, %)		Specific gravity (g/cm^3)	Blaine (cm^2/g)
	> 90 μm	> 45 μm		
PC	1.0	8.6	3.09	3830
FA	7.0	32	2.02	4890
SF	-	0.7	2.43	22310
10FA	1.6	10	2.88	3880
20FA	2.4	11.4	2.73	3900
30FA	3.4	14.8	2.59	4050
5SF	1.0	8.5	3.00	4700
10SF	0.9	8.2	2.98	5740
5FA5SF	1.5	10	2.93	4790
10FA10SF	1.4	12.6	2.85	5650

Blaine value and specific weight of the materials have different values. According to 90% screen underflow ratio, PC, FA and SF have particle sizes of 55, 150 and $20\text{ }\mu\text{m}$, respectively. It is determined that according to 45% screen underflow ratio, PC, FA and SF has particle sizes of 15, 45, and $5\text{ }\mu\text{m}$, respectively (Figure 3). In light of the results mentioned above, the smallest raw material is SF. According to Blaine values, SF is again the smallest raw material followed by FA in the second place (Table 2). FA having a greater Blaine value with respect to particle dimension distribution than PC shows that it has a porous structure.

As a result, cements having small particle physical structures are obtained by the substitution of FA and SF with smaller particles to PC (Table 2). FA and SF with small specific weight also decrease the specific weight of the cement obtained by the substitution of FA and SF to PC. While the specific weight of PC is 3.09 g/cm^3 , 10% SF substitution decreases the specific weight to 2.98 g/cm^3 and substitution of 30% FA reduces the specific weight up to 2.59 g/cm^3 (Table 2).

Water demand

As a result of the experiments conducted on cement paste samples in accordance with TS EN 196-3, water demand is determined by taking the average of 3 experiment results (Figure 4). Water demand of cements depends on parameters like chemical structure, specific surface and porosity (Termkhajornkit and Nawa, 2004). As the amount of FA in the cement is increased, Work ability and thus, water demand to obtain identical consistency increase relatively. This increase is 9, 15 and 27% in 10, 20 and 30FA coded cement pastes, respectively. Water demand of SF substitution decreases 1 and 4% in 5 and 10SF coded cement pastes, respectively.

This is thought to be due to the fact that fine, round silica fume particles go into larger cement particles and force the water out (Kelestemur and Yildiz, 2005). In

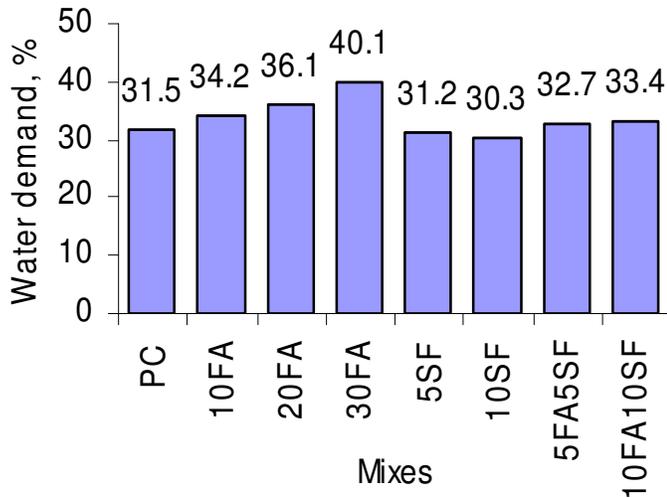


Figure 4. Water demand of cement pastes.

ternary mixtures, water demand of 10 and 20% substituted cements decrease with respect to FA of the same ratio and increase with respect to SF (Figure 4). According to the results, it can be stated that due to their fine particle structures, FA and SF have significant contribution in obtaining identical fresh concrete consistency without increasing water/cement ratio.

Setting time

Initial and final setting times obtained from the experiments carried out on cement paste samples according to TS EN 196-3 are shown in Figure 5 and 6, respectively. It was observed that, setting time of cement paste samples obtained by FA and SF substitution to PC increases linearly for all mixing ratios when compared to PC paste samples (Figure 5 and 6). In the FA substituted cement, 26, 49 and 88% increases occur in initial setting times directly proportional to substitution ratio for 10, 20 and 30FA coded cement pastes, respectively (Figure 5). There are 11, 67 and 93% increases in final setting times of 10, 20 and 30FA coded cement pastes, respectively (Figure 6).

In SF substituted cement, there are 54 and 134% increases in initial setting times of 5 and 10SF coded cement pastes, respectively (Figure 5). As for final setting times, there are 41 and 106% increases in final setting times of 5 and 10SF coded cement pastes, respectively. In ternary mixtures, setting times of 10 and 20% substituted cements increase with respect to FA of the same ratio and decrease with respect to SF (Figure 5 and 6).

In conclusion, setting time of cement paste increases linearly with FA and SF substitution as expected. Therefore, it can be stated that FA and SF slow down hydration speed, decrease hydration heat and

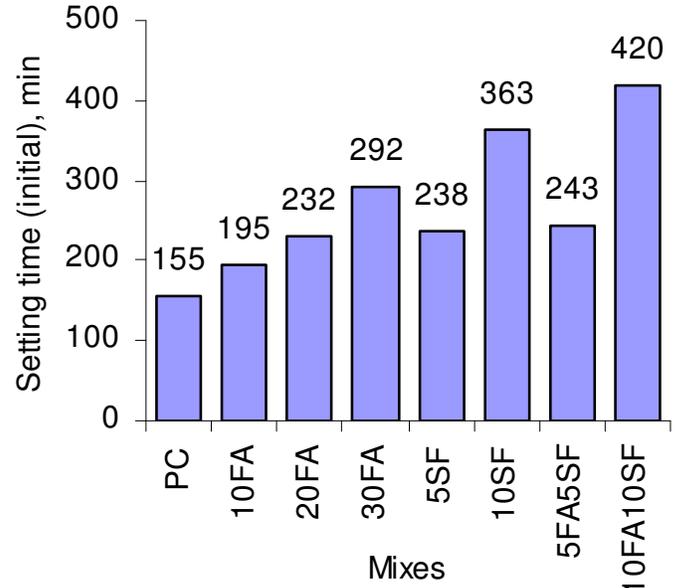


Figure 5. Initial setting time of cement pastes.

temperature, thus, are effective against contraction by increasing the setting time.

Compressive strength

Cements improve their strengths according to their hydrated parts. Pozzolans react with Ca(OH)_2 generated from the hydration of cement in the concrete, and therefore they have a diluting effect on PC during the period of Ca(OH)_2 accumulation after the addition of water to the concrete. However, Ca(OH)_2 accumulation causes the effects of pozzolans in favour of the system strength to arise. Therefore, it is expected that there will be a decrease in the early strength of concrete due to the use of pozzolana; however, the late strength is expected to reach the results of PC mortars and even exceed it (Erdogdu et al. 1999; Yetkin and Cavdar, 2005).

According to TS-EN 196-1, compressive strengths of cement mortar samples are shown in Table 3, compressive strengths of FA and SF substituted cement mortar samples relative to PC are shown in Figure 7. There are differences in strengths of FA and SF substituted cement mortars used in the study with respect to PC substituted cement mortars due to the differences in pozzolanic activity, particle size and Blaine values. Besides, in all substituted cement mortars, it is thought that the presence of pozzolanas in the environment causes a decrease in C_3S , C_3A and reduces CH formation, thus, the early strength is reduced since FA and SF cannot be completely hydrated (Yetkin and Cavdar, 2005; Kocak et al., 2010; Dorum et al., 2009). However, FA and SF having the pozzolanic property turn CH into new (pozzolanic) C-S-H gel by binding CH in time and especially fine SF particles block the pores by

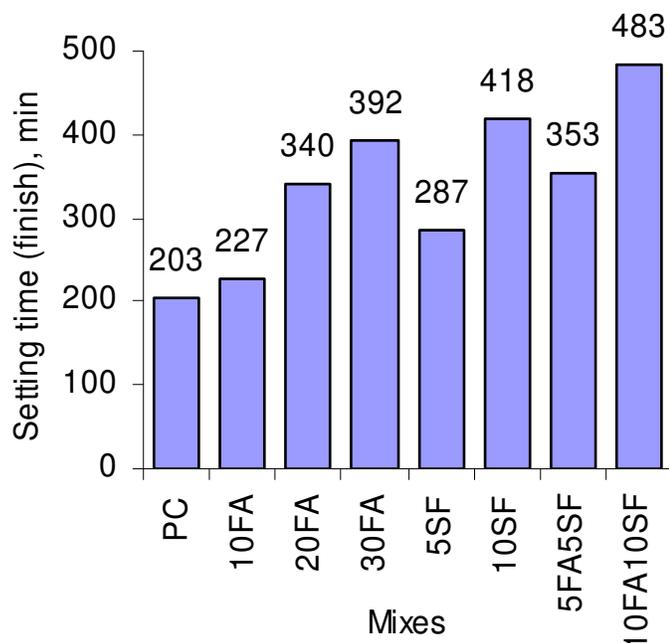


Figure 6. Final setting time of cement pastes.

tightening and strengthening aggregate-paste interface region and therefore, cause an increase in the late strength (Kelestemur and Yildiz, 2005; Kocak et al., 2010; Dorum et al., 2009).

According to the experimental findings, 10FA coded cement mortar samples exhibit 19% decrease in strength at the end of 2 days compared to the mortar samples produced by PC. However, this difference becomes 16, 12, 7 and 1% at the end of 7, 28, 56 and 90 days, respectively. When compared to mortar samples produced by PC, compressive strengths of 20FA coded cement mortar samples are decreased by 35, 30, 19, 20 and 17% at the end of 2, 7, 28, 56 and 90 days, respectively. Contrary to the previous findings, when compared to mortar samples produced by PC, compressive strengths of 30FA coded cement mortar samples are decreased by 61, 69, 47, 65 and 59% at the end of 2, 7, 28, 56 and 90 days, respectively (Table 3).

According to the mortar samples produced by PC, compressive strengths of 5SF coded cement mortar samples exhibit 11% strength decrease at the end of 2 days. However, this difference decreases by 9% at the end of 7 days, 1% at the end of 28 days and increases by 1% at the end of 56 days and reaches PC cement mortar strength at the end of 90 days. When compared to mortarsamples produced by PC, compressive strengths of 10SF coded cement mortar samples are decreased by 21, 20, 8 and 4% at the end of 2, 7, 28 and 56 days, respectively, reaching the strength of cement mortar sample produced by PC at the end of 90 days (Table 3). When the ternary mixtures are considered, compressive strengths of 5FA5SF coded cement mortar samples decrease by 12% at the end of 2 days, 14% at the end of

Table 3. Compressive strength of tested cement mortars.

Mortars	Compressive strength (N/mm ²)				
	2 days	7 days	28 days	56 days	90 days
PC	29.6	46.6	58.5	62.8	63.1
10FA	24.0	38.9	51.5	58.8	62.4
20FA	19.3	32.6	47.5	50.5	52.3
30FA	11.4	14.6	31.2	22.3	25.8
5SF	26.2	42.4	57.7	63.4	63.2
10SF	23.2	37.4	54.1	60.4	62.6
5FA5SF	26.1	39.9	53.1	57.9	63.9
10FA10SF	19.8	32.7	52.9	57.1	58.2

7 days, 9% at the end of 28 days and 8% at the end of 56 days and it increases by 1% at the end of 90 days. Compressive strengths of 10FA10SF coded cement mortar samples decrease by 33, 30, 10, 9 and 8% at the end of 2, 7, 28, 56, and 90 days, respectively (Table 3).

The results indicate that, compressive strength improvement speed of FA and SF substituted cement mortar samples is slower than PC; however, the difference can be overcome in time and even exceeded (Figure 7). Besides, the comparison of the minimum compressive strength value (42,5 N/mm²) of PC 42,5 R cement at the end of 28 days with the strength values obtained from the experiments are shown in Figure 8. According to the findings obtained in the study, compressive strengths at the end of 28 days of all cement mortar samples except for the 30FA coded sample are above the minimum compressive strength of 42.5 N/mm². While the compressive strength of 30FA is decreased by 27% with respect to the minimum strength at the end of 28 days, compressive strengths of 10 and 20FA, 5 and 10SF, 5FA5SF and 10FA10SF are increased by 21, 12, 36, 27, 25 and 24%, respectively (Figure 8). This indicates that, lower strength standard cement can be obtained from especially higher strength cement by the addition of relatively large amounts of FA. However, as seen from the results of 56 and 90 days later, it is thought that durability is ensured without any compromise on strength due to the properties of pozzolanas.

Conclusion

The findings obtained from the experiments and analyses can be summarized as follows:

- According to the chemical analysis results, FA is determined to be in Class F (low calcium FA) according to ASTM C 618 and Class V (silica-like FA) according to TS EN 197-1.
- According to XRD results, SF and FA have amorphous mineralogical structure whereas, PC has crystal mineralogical structure. However, FA, though in small amounts contains, crystal quartz, sanidine, albite and hematite.
- According to the particle size analysis, SF and PC

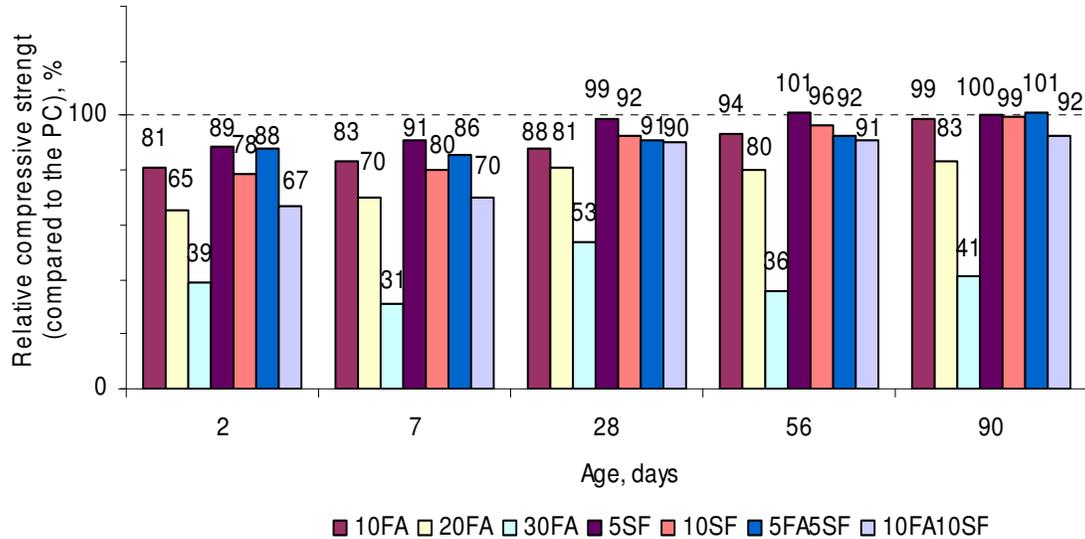


Figure 7. Relative strength values of tested cement mortars compared to the PC.

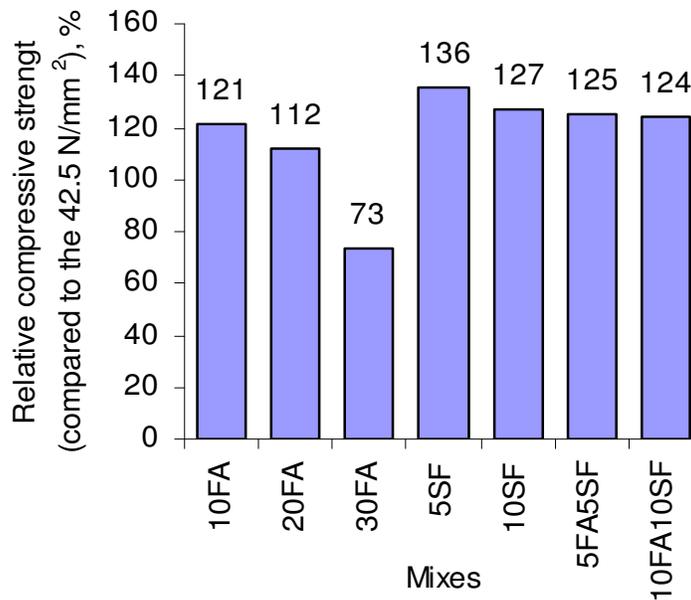


Figure 8. Relative strength values of tested cement mortars compared to the min. compressive strength (42.5 N/mm²) at 28 days.

have fine particles whereas, FA has coarse particles. Nevertheless, large specific surface area of FA shows that it has a porous structure.

(d) Low specific weights of SF and FA causes a reduction in the specific weight of FA and SF substituted cements.

(e) It can be stated that, as the FA and SF ratio is increased, workability is improved and the water demand to obtain identical consistency does not change considerably.

(f) Setting time of cement paste increases linearly with FA

and SF substitution as expected. Therefore, it can be stated that FA and SF slow down hydration speed, decrease hydration heat and temperature, thus, are effective against contraction by increasing the setting time.

(g) During hydration of cement mortars, CH formation is reduced due to FA and SF substitution, therefore, a lower compressive strength is obtained at the early ages when compared to PC. In the following hydration days, FA and SF having pozzolanic structure bind CH in time and

turn it into new (pozzolanic) C-S-H gel and cause the strength values to reach that of PC (except for 30FA coded cement).

(h) Compressive strength of 10 and 20FA, 5 and 10SF, 5FA5SF, 10FA10SF increase by 21, 12, 36, 27, 25 and 24%, respectively, with respect to 28 days minimum strength (42.5 N/mm²) of PC 42.5 R cement, while compressive strength of 30FA coded cement mortar is decreased by 27%.

(i) There is decrease in compressive strength in 30 FA coded cements despite an increase in hydration period; it shows that, this ratio is not appropriate to be used as the substitution material in this type of cement.

(j) In ternary mixtures (PC-FA-SF), especially due to the fine particle structure of SF and pozzolanic reaction have higher strength values which are obtained compared to FA substituted cement mortars of the same ratio.

As a general overview in light of the findings of the study, considering durability equally important as strength, durable cements yet not compromising the minimum strength requirements defined by the standards that can be produced by the substitution of appropriate amounts of FA and SF to high-strength cements (PC). It is thought that, durable cement and concrete mortars can be produced with these cements without any compromise on strength. Therefore, it will be beneficial to carry out research on other pozzolanas and their properties.

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