Bitlis Eren Üniversitesi Fen Bilimleri Dergisi
BİTLİS EREN UNIVERSITY JOURNAL OF SCIENCE
ISSN: 2147-3129/e-ISSN: 2147-3188
VOLUME: 12 NO: 1 PAGE: 115-125 YEAR: 2023
DOI:10.17798/bitlisfen.1198854

Physical and mechanical properties of pozzolanic materials blended cement mortars before and after the freeze-thaw cycles

Berivan FİLİZ[1] Zehra Funda AKBULUT[2*], Soner GÜLER[3]

[1]*Department of Environmental Engineering, University of Van Yuzuncu Yıl, 65080, Van, Turkey*
[2*]Department of Mining Engineering, University of Van Yuzuncu Yıl, 65080, Van,Turkey
[3]*Department of Civil Engineering, University of Van Yuzuncu Yıl, 65080, Van, Turkey*
(ORCID: 0000-0001-8462-570X) (ORCID: 0000-0003-3055-3450) (ORCID: 0000-0002-9470-8557)

Keywords: Freeze thaw resistance, Industrial wastes, Mortars, Physical and mechanical properties

Abstract
Today, the production of Portland cement (PC) causes a significant release of carbon dioxide (CO2) gas into the atmosphere. The CO2 gases released into the atmosphere create environmental pollution worldwide and prevent current and future generations from living in a cleaner environment. To minimize the harmful effect of the PC on the environment, it is used in concrete mixtrues by displacing it in specific proportions with different industrial wastes. Using industrial wastes such as fly ash (FA), silica fume (SF), and marble powder (MP) in concrete mixtures by replacing cement in specific proportions is vital in terms of sustainability. The primary purpose of this study is to examine the effects of FA, SF, and MP comparatively replaced with cement at the rates of 10%, 20%, and 30% on the flowability, mass loss (ML), and residual compressive strength (RCS) of mortars before and after freeze-thaw (F-T) cycles. According to the results, the effects of FA, SF, and MP on mortars' fresh and hardened properties vary considerably. However, using FA, SF, and MP instead of cement significantly improves the matrix's weak cement/aggregate interface transition zones (ITZ) by showing the filler effect. They contribute considerably to reducing mass losses and increasing the RCS capacities of mortars. Compared to room conditions, the reduction in RCS capacities of the control mortar was 21.32% after 200 F-T cycles, while the decrease in RCS capacities of FA-, SD-, and MP-added mortars was between 7.86% and 19.85%. While the mass loss of the control sample after the 200 F-T cycle is 1.23%, the mass loss of mortars with FA, SF, and MP additives is lower and varies between 0.44% and 1.02%.

1. Introduction

The use of ready-mixed concrete in developing countries such as China, India, and Turkey is increasing daily due to the rapid augmentation in industrialization and infrastructure investments. In addition, the construction of new residences to meet the housing needs of the rapidly growing population in these countries quickly raises the demand for ready-mixed concrete. This situation leads to a significant increase in the use of Portland cement (PC), one of the main components of ready-mixed concrete [1]-[4]. The clinker production stage causes considerable carbon dioxide (CO_2) emissions in PC production processes. During the production of 1 ton of PC, approximately 0.85-1 ton of CO2 is released into the atmosphere [5]. As it is known, CO_2 is called greenhouse gas, and these gases cause significant air pollution and global warming. Therefore, in recent years, studies on using some industrial wastes in concrete mixes instead of PC have gained momentum in terms of sustainability. [6], [7]. Some of the most commonly used industrial waste supplementary cementitious materials in concrete mixtures that can be replaced with PC are fly ash (FA), silica fume (SF), and marble powder (MP). FA is a waste product resulting from the combustion of ground coal at 1100-1600 ^oC in thermal power plants. Approximately 50

*Corresponding author: fundaakbulut@yyu.edu.tr
Received: 03.11.2022, Accepted: 28.02.2023

million tons of lignite coal are used annually in thermal power plants in Turkey. As a result, each year, 12 million tons of waste FA are released into the environment [8]. SF is a gray-colored waste powder obtained from the reduction of high-purity quartzite with silicon or ferrosilicon alloy with coke in electric arc furnaces. As a result of production at Antalya Ferrochrome facilities in Turkey, around 500-1000 tons of waste SF material are produced annually [9]. MP is formed either during the production of block marble in quarries or during the processing of blocks by cutting them in the factory so that the marble can be used, or during the production of slab marble. Turkey is among the wealthiest countries in the world in terms of marble, with a total of 5.2 billion m^3 (13.9 billion tons) of marble reserves, which has approximately 40% of the world's resources [10]. In Turkey, 40-60% of the 7 million tons of marble produced annually, which can be expressed as a million tons, is dumped on the roadsides or around the production facilities [11]. Concrete is exposed to many physical and chemical effects depending on the time during its service life, and therefore its strength and durability properties are significantly reduced. One of the most important physical effects that negatively affect the strength and durability properties of concrete is the freeze-thaw (F-T) effect. As is known, when water freezes, it expands in volume. When the water in the pores and capillary spaces freezes, it expands and exerts stresses on the walls of these gaps and pores, resulting in micro cracks. As a result of the volumetric expansion and freezing of water after repeated F-T effects, deterioration, fragmentation, and ruptures occur in the concrete microstructure. As a result, the service life of concrete is shortened, maintenance costs increase, and it becomes uneconomical [12], [13]. One of the most effective ways to improve the strength and durability properties of concrete exposed to F-T cycles is to use FA, SF, and MP in concrete mixtures by replacing PC in specific proportions. The benefits of FA, SF, and MP such as strengthening the bonding at the aggregate interface of the cement matrix (paste), creating a denser structure with a higher composition by filling the voids in the concrete microstructure, preventing the formation of sweating and plastic shrinkage, and strengthening the concrete internal system against harmful chemicals by reducing permeability. Furthermore, due to the higher specific surface area of SF compared to cement, it accelerates the setting of concrete, creates an additional reaction surface for hydration, creates additional calcium-silicate-hydrate (C-S-H) gels due to its high pozzolanic activity, and fills gel voids and capillary voids. Thus, some researchers have strongly recommended using FA, SF, and MP to improve the strength and durability properties of concrete against F-T cycles [14]-[25].

2. Material and Method

CEM I 42.5 R type ordinary Portland cement (OPC) [26], CEN standard sand as fine aggregate [27], F-class FA, SF, MP, and superplasticizer were used in cement mortar mixtures. F-class FA was obtained from İskenderun Sugözü Thermal Power Plant. SF was supplied from Etibank Ferrochrome facilities in Antalya province. Marble slurry was obtained from the waste storage area of a marble factory in Van province and was used in cement mortar after drying in an oven for 24 hours at 105 oC. The chemical properties of OPC, FA, SF, and MP are given in Table 1. The physical properties of OPC, FA, SF, and MP are given in Table 2.

Table 1. Chemical properties of OPC, FA, SF, and MP materials

Composition (%)	OPC	FA	SF	MP
SiO_2	18.83	61.10	92.18	0.50
Al_2O_3	5.17	24.20	0.73	0.25
Fe_2O_3	3.47	8.22	0.66	0.21
CaO	64.4	1.97	0.40	55.30
MgO	3.76	2.42	0.27	0.12
TiO_2	0.04	1.27	0.11	0.04
K_2O	0.71	0.42	0.82	0.01
Na_2O	0.46	0.11	0.17	0.24
Loss on ignition	1.25	2.48	2.26	43.26

Table 2. The physical properties of OPC, FA, SF, and MP

Physical properties	OPC	FA	SF	MP
Specific gravity	3.17	2.24	2.20	2.70
Blaine fineness (cm^2/g)	3984	2872	200000	3412

Polycarboxylate ether-based Master Glenium SKY 3675 superplasticizer (SP) with a density of 1.03-1.07 kg/m3 and a pH value of 5-7 at 20 ℃ was used in the experiments. An automatic programmable cement mixer produced FA-, SF-, and MP-blended cement mortars. In the experimental study, 10 mortar mixtures were prepared, including 1 control and 9 SF, FA, and MP-added mixtures. Amounts of 500 g cement, 225 g water, and 1350 g standard sand were used in the control mortar [28]. The samples' ML, RCS, and RFS values were obtained by averaging the measurements made at room temperature and at the ends of 50, 100, and 150 F-T cycles. The samples' ML, RCS, and RFS values were obtained using 150 samples, 3 samples from each mixture. Samples of the same ages were tested and compared to determine the samples' reductions in RCS capacity. RFS values of all samples were carried out under 3-point loading on prismatic samples with dimensions of 40 x 40 x 160 mm. The residual compressive strength (RCS) test was carried out at 28 days on the unbroken side surfaces of the specimens with dimensions of 40 x 40 x 160 mm, divided into two parts. Therefore, six compressive strength values were obtained for the three specimens [27]. FA, SF, and MP were used by replacing 10, 20, and 30% of the cement weight, respectively. The amount of SP was used in different proportions for all mixtures to obtain appropriate workability values by considering the material properties of FA, SF, and MP. In the mixing phase of FA-, SF-, and MP-blended cement mortars, cement, waste materials, and distilled water were first added into the cement mixer and mixed for approximately 30 s at low speed. Throughout the next 30 s, standard sand was automatically poured into the mixer, and the mixer continued to mix at high speed for another 30 s. The mixer was stopped after 1 minute and 30 s, and the mortars adhering to the wall of the container were stripped. After fresh cement mortar experiments, control and FA-, SF-, and MP-blended cement mortars were filled into prismatic molds with 40 mm x 40 mm x 160 mm dimensions for hardened cement mortar tests. A shaking table set the cement mortar well in the molds. First, the mortar was filled to half the mold and compacted using 60 shaking processes. Afterward, 60 shakes were used to fill the entire mortar mold. The samples filled in the molds were kept under ambient conditions for 24 hours. After that, the molds were removed, and all the specimens were water-cured at 20 ºC for 28 days. The samples removed from the curing pool were kept in an oven at 105 °C for 48 hours until they reached a constant weight. First, the weights and the compressive and flexural strengths of the samples taken out of the oven (3 samples from each mixture) were determined at ambient conditions. The other samples were left in a plastic bucket filled with water and exposed to 50, 100, and 200 freeze-thaw cycles in the freeze-thaw test cabinet. After each freeze-thaw cycle, the samples were dried in an oven for 48 hours, and their weights, compressive and flexural strengths were measured again. The mix-design of the control and blended cement mortars are given in Table 3.

Table 3. Mix designs of control and blended cement mortars

Mix cod	Mixes	C (g)	S (g)	Su (%)	W (g)	SP (%)	w/b
P0	Control	500	1350	0	225	0.6	0.45
P1	FA-10	450	1350	10	225	0.6	0.45
P2	FA-20	400	1350	20	225	0.5	0.45
P3	FA-30	350	1350	30	225	0.4	0.45
P4	SF-10	450	1350	10	225	0.8	0.45
P5	SF-20	400	1350	20	225	0.9	0.45
P6	SF-30	350	1350	30	225	1.0	0.45
P7	MP-10	450	1350	10	225	0.7	0.45
P8	MP-20	400	1350	20	225	0.8	0.45
P9	MP-30	350	1350	30	225	0.9	0.45

Notation: C: Cement, Su: Supplement, S: Sand, W: Water

Firstly, the fresh properties of control and blended cement mortars were determined [29]. For this purpose, a flow table test was used to determine the fresh cement mortars' spreading diameters. The spreading diameters of the mortars were obtained by averaging two measurements perpendicular to each other on the spreading table. After the properties of fresh mortar were determined, the specimens with dimensions of 40 x 40 x 160 mm after 28 days of curing were subjected to 50, 100, and 200 F-T cycles. A freely programmable freeze-thaw test cabin was used in freeze-thaw tests of cement mortars. Specimens in the freeze-thaw test cabin were subjected to freezing for 12 hours at -20 ℃ and thawing for 12 hours at 20 ℃ in accordance with the ASTM C 666 standard [30]. Two procedures are given to evaluate the resistance of concrete against the freeze-thaw effect in accordance with the ASTM C666 standard. Both of these methods employ rapid freeze-thaw cycles. In procedure A, freezing and thawing are carried out in the water. In procedure B, freezing is carried out in the air, and thawing is carried out in the water. Procedure A was followed in this study. The mass loss and compressive strength of the control and cement blended mortars were carried out at room temperature and after 50, 100, and 200 F-T cycles. Three specimens were tested, and their average values were used to determine the physical and mechanical properties of control and FA-, SF-, and MP-blended cement mortars under room conditions and after each round of F-T cycles. The loading rates of flexural strength and compressive strength tests were 50 N/s and 2400 N/s, respectively. The Field Emission Scanning Electron Microscope (FESEM) analyses were performed only for the control and FA-20, SF-20, and MP-20 samples to observe the changes in the microstructural properties of the selected cement mortars at room temperature and after 50, 100, and 200 F-T cycles. To capture images with FESEM analysis, microscopic samples were taken from the specimens and subjected to SEM analysis using the Zeiss Sigma 300 FESEM brand device. FESEM analyses were carried out on the pieces with 1 x 1000 magnification. The schematic view of the testing stages is given in Figure 1.

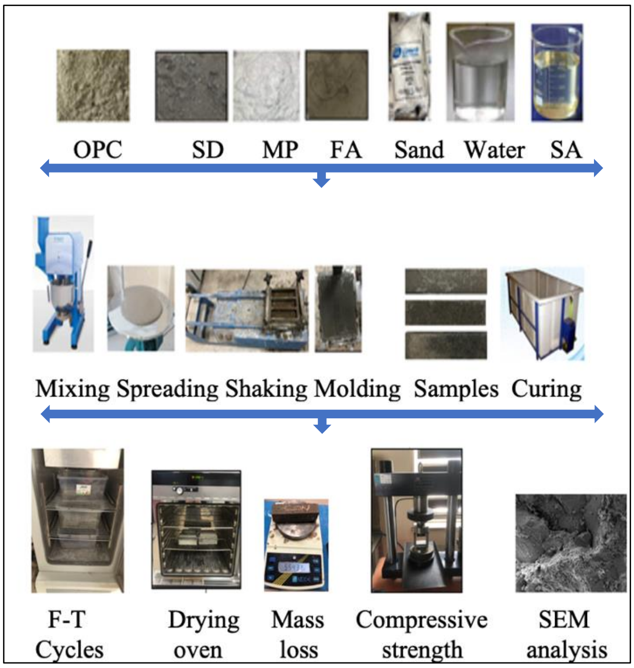

Figure 1.The schematic view of the testing stages

3. Results and Discussion

3.1. Flow Table

The flow table test was used to determine the workability of fresh cement mortars, and the spreading diameters of all the cement mortars were measured. The spreading diameters of fresh cement mortars are given in Table 4. Primarily, many trial mixes were prepared to ensure that the mixtures had similar workability properties. For the control and FA-, SF-, and MP-blended cement mortars to have nearly the same workability properties and thus have close spreading diameters, SP ratios were changed, provided that the water/cement (w/c) ratio was kept constant at 0.5. Therefore, while the SP was used as 0.6% of the control mortar's cement weight, it increased to 1.0% of the cement weight in the SF-30 mixture. Furthermore, the positive contribution of FA to the workability of cement mortar due to its spherical grain structure led to SP being used at a lower rate (0.4, 0.5, and 0.6%) in the FA-blended cement mortars. In addition, it was considered that the angular grain structure of MP would damage the workability of cement mortars, so the ratio of SP in MP-blended cement mortars (0.7, 0.8, and 0.9%) is a little higher than in control mortars. As seen from Table 2, while the spreading diameter of the control cement mortar was 175 mm, the spreading diameters of FA-, SF-, and MP-blended cement mortars vary between 180-191 mm, 168-174 mm, and 178-183 mm, respectively. It can also be said from these results that SF highly negatively affects the workability properties of concrete. The main reason is that fresh concrete has a higher viscosity with the addition of a large amount of SF, which decreases the workability of concrete. This situation has been stated by some researchers [31], [32]. However, the researchers could not agree on an optimum value for the cement-displaced SF ratio. Khedr and Abou-Zeid [33] stated that the workability properties of concrete decrease linearly when the SF content replacing cement increases from 0 to 25%. However, Zhang and Li [34] emphasized that the workability decreased significantly when the ratio of SF displaced by cement increased from 6% to 9%. Still, the decrease in workability was smaller when more than 9% SF was used.

Table 4. Spreading diameters of fresh cement mortars

Mix cod	Mixes	Diameter (mm)
P0	Control	175
P1	FA-10	180
P2	FA-20	187
P3	FA-30	191
P4	SF-10	174
P5	SF-20	170
P6	SF-30	168
P7	MP-10	178
P8	MP-20	183
P9	MP-30	182

3.2. Mass Loss

The mass loss (ML) results after F-T cycles are given in Table 5 and Figure 2. It was found that all samples had minimal mass losses after F-T cycles (lower than 1.5%). The pressure stresses applied by the water in the cavities of the concrete elements with a hollow structure due to the temperature drop below zero may cause the concrete to crumble, break up, and throw pieces. Such a result is due to the increased volume of water due to freezing. Some of the most effective ways to make concrete resistant to F-T cycles are reducing cement dosage, reducing shrinkage and heat of hydration, keeping the water/cement ratio low, and using air-entraining admixtures [35]-[37]. Since using air-entraining admixtures may cause a significant decrease in the mechanical properties of the concrete, it is recommended to be used under certain limits. In cases where no additives are used, one of the most effective ways to reduce cement dosage is to replace waste materials with a pozzolanic character, such as FA, SF, and MP replaced, with cement. Using these pozzolanic materials reduces the shrinkage and hydration rate, and as a result, it can limit the development of cracks on the outer and inner surfaces of the concrete. In this study, it is seen that the mass losses of all samples increase in direct proportion to the increase in the number of F-T cycles. The ML values of the P0 control sample after the 50, 100, and 200 F-T cycles were 0.48%, 0.88%, and 1.23%, respectively. When we look at the ML values of the P1 and P9 samples after 200 F-T cycles, it is seen that they vary between 0.44% and 1.02%.

Figure 1 shows that the P1-P9 samples have lower ML values than the P0 control sample after all F-T cycles, except for the P6 sample's mass loss after 50 F-T cycles. The most important reason is that FA, SF, and MP replaced with cement strengthen weak ITZ regions in the matrix and reduce the damage to the sample's internal structure after F-T cycles. This is to preserve the integrity of the samples better, minimizing the development of cracks in their internal structures. Furthermore, it can be said that P4-P6 specimens with SF-blended cement mortars have slightly lower ML values compared to FA- and MP-blended cement mortars. This is because SF showed higher pozzolanic activity, formed new C-S-H gels, and had a better filler effect in the microstructure of mortars [38-39]. As a result, the samples could maintain their integrity better. Ince et al. [25] showed that the mortar substituted with 20% SF instead of cement lost less mass than the control concrete after freezing and thawing. In addition, the increase in the number of F-T cycles caused the hydraulic pressure in the cement matrix and increased the formation of microcracks.

3.2. Residual Compressive Strength

The residual compressive strengths (RCS) of control and FA-, SF-, and MP-blended cement mortars are given in Table 6 and Figure 3. As seen in Figure 3, as the number of F-T cycles increased, the RCS of the samples decreased more. Compared to room conditions, the decrease in compressive strength of P0 control cement mortar after 200 F-T cycles was 21.32%. At the same time, the reduction in compressive strength of FA-, SD-, and MP-blended P1-P9 cement mortars was measured between 7.86 and 19.35%. Furthermore, the decrease in compressive strength of all P1-P9 mortars after 200 F-T cycles is lower than that of the P0 control mortar. In addition, while the proportion of MP substituted for cement increased from 10% to 30%, the compressive strength decreased proportionally for P7-P9 MP-blended cement mortars. At the same time, a linear trend could not be obtained for FA- and SF-blended cement mortars. Also, SF-blended cement mortars have higher compressive strengths after 200 F-T cycles than FA- and MP-blended cement mortars. Due to its high fineness and pozzolanic properties, silica fume fills the voids in the mortar much better than MP and FA additives and supports final strength development. In addition, the fact that the pozzolanic activity of silica fume is much higher than that of MP and FA additives has played a very influential role in gaining the strength of the cement mortar. Accordingly, it shows higher pozzolanic activity and forms C-S-H bonds with greater strength.

Furthermore, SF fills the gaps in the cement mortar better than FA and MP due to having a fineness modulus approximately 100 times higher than ordinary Portland cement fineness. As a result, SF minimizes the F-T effects in the cement microstructure and causes the specimens to gain more RCS. The contribution of SF obtained as a result of this study to the RCS of control cement mortar is highly consistent with the results of some studies [4], [14], [15], [18]. In addition, due to the high fineness of SD, it increases the density of mortar and concrete, allowing the capillary spaces between cement and aggregate to be filled with more hydration products [40]. Although FA-blended cement mortars do not reach the values of SF-blended cement mortars, they have a higher RCS against FT cycles than MP samples, thanks to the pozzolanic activity that does not exist in MP. Undoubtedly, it should be remembered that the contribution of FA after 28 days is more limited as it is faster to chemically react with water and form new C-S-H bonds at later ages. If the measurements were made later, it is evident that the microstructures of the FA-blended cement mortars would be more robust, and their RCS would be higher. The contribution of MP to the compressive strength of mortar after F-T cycles is due only to the pore-filling features. However, this contribution is effective up to 10%, decreasing at 20% and 30% replacement rates. This is because using MP at higher speeds has a dilution effect, limiting the impact of cement in forming C-S-H bonds. Substitution with more than 10% MP was also emphasized to reduce the binder content and cause dilution [41]. Ince et al. [25] also noted that the compressive strength loss of the cement and 20% SF-replaced mortar samples was

19%, while the compressive strength loss of the control mortar was 26% after 336 F-T cycles.

Table 5. Mass loss of control and blended cement mortars

Mix code	Mixes	50 F-T (%)	SD	100 F-T (%)	SD	200 F-T (%)	SD
P0	Control	0.48	0.02	0.88	0.03	1.23	0.03
P1	FA-10	0.37	0.04	0.61	0.03	0.75	0.04
P2	FA-20	0.18	0.04	0.56	0.02	0.54	0.02
P3	FA-30	0.41	0.03	0.68	0.02	0.89	0.02
P4	SF-10	0.19	0.03	0.47	0.03	0.64	0.02
P5	SF-20	0.14	0.02	0.36	0.02	0.44	0.04
P6	SF-30	0.23	0.02	0.51	0.04	0.72	0.04
P7	MP-10	0.35	0.02	0.72	0.04	0.87	0.03
P8	MP-20	0.47	0.04	0.65	0.03	0.81	0.02
P9	MP-30	0.51	0.03	0.81	0.04	1.02	0.02

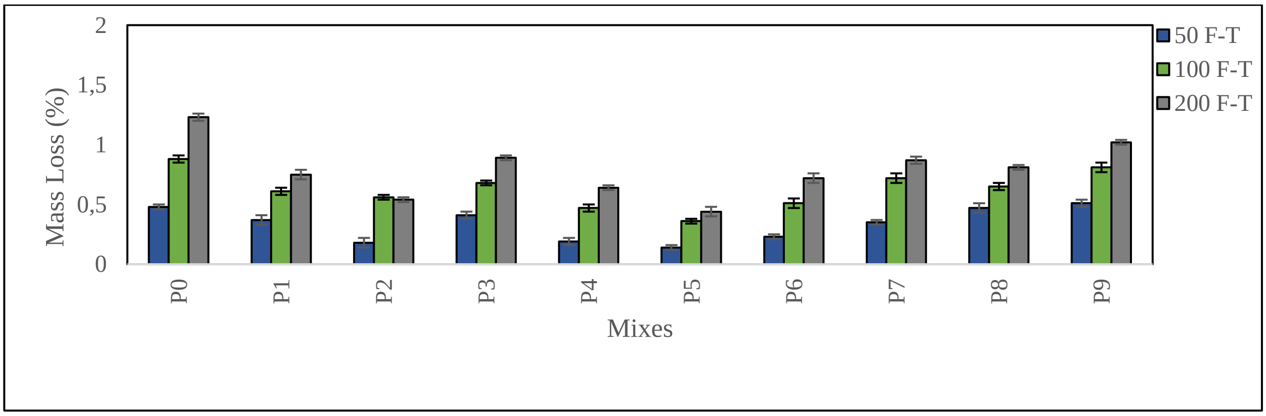

Figure 2. Variation of mass loss of control and FA, SF and MP blended cement mortar specimens

Table 6. Residual compressive strength (RCS) of control and blended cement mortars

Mix code		Residual Compressive Strength							
		0 F-T (MPa)	SD	50 F-T (MPa)	SD	100 F-T (MPa)	SD	200 F-T (MPa)	SD
P0	Control	49.15	1.63	46.27	1.66	42.49	2.41	38.67	2.54
P1	FA-10	50.65	2.23	50.32	2.00	47.08	2.05	43.76	2.16
P2	FA-20	48.82	2.15	48.21	1.41	45.69	0.63	42.89	1.70
P3	FA-30	50.28	2.03	48.91	0.68	44.12	1.53	42.11	2.69
P4	SF-10	63.26	2.34	62.87	1.69	59.63	2.18	56.38	2.87
P5	SF-20	68.14	1.82	67.76	2.55	64.98	1.57	62.36	2.51
P6	SF-30	75.91	2.33	73.95	0.98	72.11	3.31	69.94	1.61
P7	MP-10	49.51	2.62	48.79	2.42	45.87	1.90	42.34	2.94
P8	MP-20	45.43	1.90	44.17	2.19	41.21	2.88	37.76	2.42
P9	MP-30	48.52	1.66	46.52	2.76	41.87	2.67	39.13	1.66

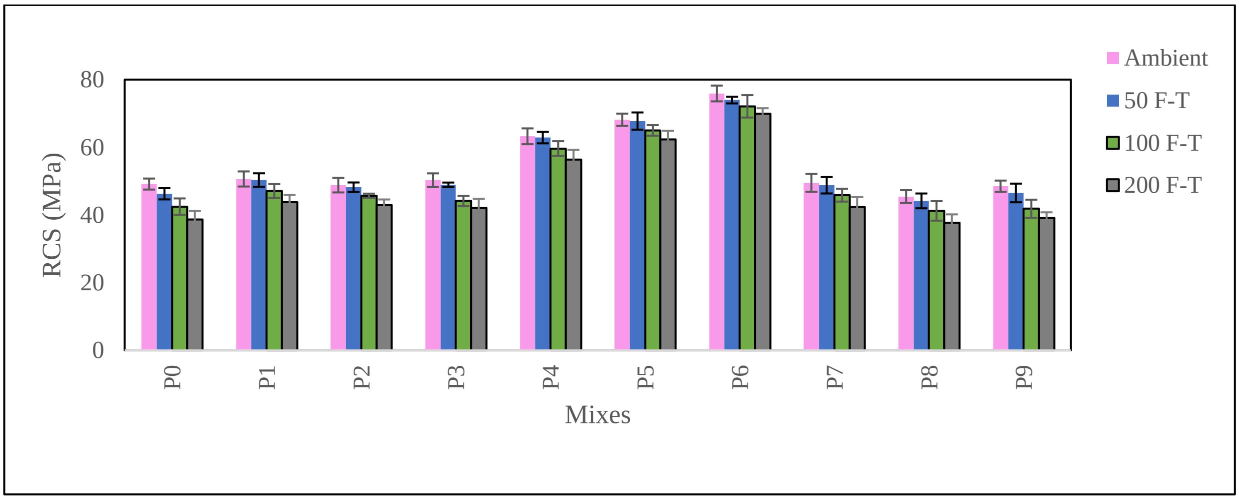

Figure 3. Variation of residual compressive strength of control and FA, SF and MP blended cement mortar specimens

3.2. FESEM Analysis

The FESEM images of control and FA-20, SF-20, and MP-20 blended cement mortars under room conditions, and after 50, 100, and 200 F-T cycles are shown in Figure 4. As seen in Figure 4, however, while control, FA-, SF-, and MP-blended cement mortars have a more robust microstructure under room conditions, it is seen that micro-cracks turn into larger macro cracks as a result of integrity deterioration in the microstructures after increasing F-T cycles. It is also seen that these cracks become more pronounced after 100 and 200 F-T cycles compared to 50 F-T processes. In addition, the control mortar appears to have larger and deeper cracks after 100 and 200 F-T cycles compared to FA, SF, and MP mortars. Although FA mortars have a more robust microstructure due to their high pozzolanic activity, it is not as much as SF mortars compared to MP mortars. In addition, it is observed that SF-blended cement mortars retain their integrity better than FA- and MP-blended cement mortars and have thinner and more minor cracks.

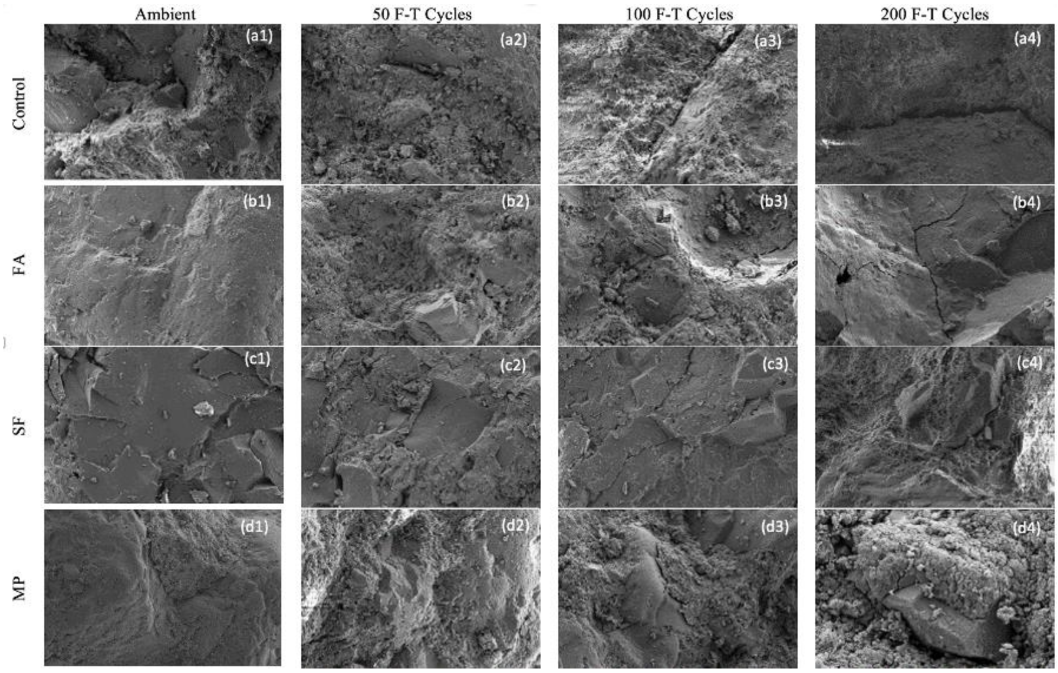

Figure 4. FESEM images of control and FA, SF and MP blended cement mortar specimens

One of the most important reasons is that SF has a high fineness modulus, so it fills the gaps between cement grains better and ensures that the mixtures have a better interfacial transition zone (ITZ) and impermeable microstructure [25], [42], [43], [44]. Another reason is that SF has high pozzolanic activity due to its high percentage of Si particles. Ca(OH)2 hydrated products are known to form as a result of the reaction of cement with water. Ca(OH)2 is a water-soluble phase that does not contribute to the strength and is responsible for the hollow structure of concrete. Thus, a minimum of $Ca(OH)_2$ is preferred in cement mortar. $Ca(OH)_2$ chemically reacts with SF, which has a very high amount of silicon (Si) in its chemical composition. Therefore, the new C-S-H bonds formed due to the chemical reaction of Si particles with $Ca(OH)_2$ enabled SF mortars to better protect their microstructure resistance after increasing F-T cycles and maintain higher residual compressive strength [45], [46].

4. Conclusion and Suggestions

This study examines the effects of fly ash (FA), silica fume (SF), and marble powder (MP), which are used at 10%, 20%, and 30% substitution for cement, on cement mortar's fresh and hardened properties after freeze-thaw (F-T) cycles. In addition, FESEM analyses at ambient conditions and after F-T processes were performed to investigate changes in the microstructures of samples. As a result of this study, the following conclusions were obtained.

•As the number of F-T cycles increases, there is a considerable decrease in the physical and mechanical properties of the samples. This decrease was more evident, especially after 200 F-T cycles.

•Adding FA and MP to the comparative mixtures to the P0 control mixture increased the workability of the mixes and, therefore, the spreading diameters. In contrast, the spreading diameters of the SF-added combinations decreased. Therefore, to obtain good workability in SF additive mixtures, the SP additive ratio should be slightly higher than in other varieties.

•ML values of FA, SF, and MP added to P1-P9 samples after F-T cycles were higher than the P0 control sample. The most important reason for this is that with the strengthening of the ITZ regions of FA, SF, and MP, the integrity of the sample is less lost after F-T cycles, and the cracks formed in the internal structures due to this are limited.

•SF-blended cement mortars (P3-P6) are much more effective than FA- and MP-blended cement mortars in improving control mortars' physical and mechanical properties against F-T cycles due to their high fineness modulus and high pozzolanic activity. FA, SF, and MP significantly improve the microstructure of the cement matrix and contribute considerably to the strength and durability properties of the concrete against the F-T cycles.

Acknowledgment

This study is supported by the Department of Scientific Research Projects of the University of Van Yüzüncü Yıl, Van, Turkey, with the project ID 8936.

Contributions of the authors

The contributions of each author to the article should be indicated.

Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

References

[1] D. Hatungimana, C. Taşköprü, M. Ichedef, M. M. Saç, and Ş. Yazıcı, "Compressive strength, water absorption, water sorptivity and surface radon exhalation rate of silica fume and fly ash based mortar," *Journal of Building Engineering,* vol. 23, pp. 369-376, 2019.

[2] M.M. Hossain, M.R. Karim, M. Hasan, M.K. Hossain, and M.F.M. Zain, "Durability of mortar and concrete made up of pozzolans as a partial replacement of cement: A review," *Construction and Building Material,* vol. 116, pp. 128-140, 2016.

[3] Y. Liu, Y. Zhuge, W. Duan, G. Huang, and Y. Yao, "Modification of microstructure and physical properties of cement-based mortar made with limestone and alum sludge," *Journal of Building Engineering,* vol. 58, pp.105000, 2022.

[4] J. Wang, B. Ma, H. Tan, C. Du, Z. Chu, Z. Luo, and P., Wang, “Hydration and mechanical properties of cement-marble powder system incorporating triisopropanolamine”, *Construction and Building Materials*, vol. 266, pp.121068, 2021.

[5] M. Criado, I. Sobrados, I., J.M. Bastidas, and J. Sanz, “Corrosion behaviour of coated steel rebars in carbonated and chloride-contaminated alkali-activated fly ash mortar,” *Progress in Organic Coatings*, vol. 99, pp. 11-22, 2016.

[6] F.N. Stafford, A.C. Dias, L. Arroja, J.A. Labrincha, and D.Hotza, “Life cycle assessment of the production of Portland cement: a Southern Europe case study,” *Journal of Cleaner Production*, vol. 126, pp. 159-165, 2016.

[7] N. Singh, A.R. Varsha, A. R. Sai, and M. Sufyan-Ud-Din, “Strength, electrical resistivity and sulfate attack resistance of blended mortars produced with agriculture waste ashes,” *Case Studies in Construction Materials*, vol. 16, pp. e00944, 2022.

[8] İ. Demir, “Uçucu külün hafif yapı malzemesi üretiminde kullanılması,” Yapı Teknolojileri Elektronik Dergisi, vol 1(1), pp. 21-24, 2015.

[9] H. Eker, A. Bascetin, “Influence of silica fume on mechanical property of cemented paste backfill,” *Construction and Building Materials*, vol. 317, pp.126089, 2022.

[10] M. E. Altinay, “Bucak ilçesi mermer sektörünün mevcut durum analizi (Master's thesis, Sosyal Bilimler Enstitüsü)”. 2019.

[11] Q. Iqbal, M. A. Musarat, N. Ullah, W. S. Alaloul, M. B. A. Rabbani, W. Al Madhoun, and S. Iqbal “Marble Dust Effect on the Air Quality: An Environmental Assessment Approach,” *Sustainability*, vol.14(7), pp. 3831, 2022.

[12] R. Wang, Z. Hu, Y. Li, K. Wang, H. Zhang, “Review on the deterioration and approaches to enhance the durability of concrete in the freeze–thaw environment,” *Construction and Building Materials*, vol. 321, pp.126371, 2022.

[13] C. Chen, R. Zhang, L. Zhou, and Y. Wang, “Influence of Waste Tire Particles on Freeze–Thaw Resistance and Impermeability Performance of Waste Tires/Sand-Based Autoclaved Aerated,” *Concrete Composites. Buildings*, vol. 12(1), pp. 33, 2022.

[14] T. Gonen, S. Yazicioglu, and B. Demirel, “The influence of freezing-thawing cycles on the capillary water absorption and porosity of concrete with mineral admixture,” *KSCE Journal of Civil Engineering*, vol.19(3), pp. 667-671, 2015.

[15] Karakurt, C., and Bayazıt, Y. “Freeze-thaw resistance of normal and high strength concretes produced with fly ash and silica fume,” Advances in Materials Science and Engineering, vol. 2015, pp. 1-8, 2015.

[16] M. Gruszczyński, and M. Lenart, “Durability of mortars modified with the addition of amorphous aluminum silicate and silica fume,” *Theoretical and Applied Fracture Mechanics*, vol. 107, pp. 102526, 2020.

[17] A. Mardani-Aghabaglou, G.İ. Sezer, and K. Ramyar, “Comparison of fly ash, silica fume and metakaolin from mechanical properties and durability performance of mortar mixtures view point,” *Construction and Building Materials*, vol. 70, pp.17-25, 2020.

[18] C. S. Shon, A. Abdigaliyev, S. Bagitova, C.W. Chung, D. Kim, “Determination of air-void system and modified frost resistance number for freeze-thaw resistance evaluation of ternary blended concrete made of ordinary Portland cement/silica fume/class F fly ash,” *Cold Regions Science and Technology,* vol. 155, pp. 127-136, 2018.

[19] A.Benli, K. Turk, and C. Kina, C. “Influence of silica fume and class F fly ash on mechanical and rheological properties and freeze-thaw durability of self-compacting mortars,” *Journal of Cold Regions Engineering*, vol. 32(3), p. 04018009, 2018.

[20] P.A. Shirule, A. Rahman and R.D. Gupta, “Partial replacement of cement with marble dust powder,” Int J Adv Eng Res Stud. vol. 1, pp.175–177, 2012.

[21] O. Gencel, C. Ozel, F. Koksal, E. Erdogmus, G. Martínez-Barrera, and W .Brostow, “Properties of concrete paving blocks made with waste marble,” *Journal of Cleaner Production*, vol. 2(1), pp. 62-70, 2012.

[22] Y. Xi, E. Anastasiou, A. Karozou, and S. Silvestri, “Fresh and hardened properties of cement mortars using marble sludge fines and cement sludge fines,” *Construction and Building Materials*, vol. 220, pp.142-148, 2019.

[23] J. Wang, B. Ma, H.Tan, C. Du, Z. Chu, Z. Luo, and P. Wang,"Hydration and mechanical properties of cement-marble powder system incorporating triisopropanolamine," *Construction and Building Materials*, vol. 266, no. 121068, p. 121068, 2021.

[24] K. Vardhan, S. Goyal, R. Siddique, and M. Singh, "Mechanical properties and microstructural analysis of cement mortar incorporating marble powder as partial replacement of cement. *Construction and Building Materials*," vol. 96, pp. 615–621,2015.

[25] Ince, C., Hamza, A., Derogar, S and Ball, R.J. "Utilisation of waste marble dust for improved durability and cost efficiency of pozzolanic concrete," *Journal of Cleaner Production*, vol. 270, no. 122213, p. 122213, 2020.

[26] Turkish Standards Institute, "Cement-Part 1: General Cements- Composition, Properties, and Suitability Criteria, "TS EN 197-1," Turkey, 2012.

[27] Turkish Standards Institute, Cement Test Methods - Part 1: Determination of Strength, "TS EN 196-1," Turkey, 2016.

[28] Turkish Standards Institute, "Mixing for concrete-specifications for sampling, testing and assessing the suitability of water, including water recovered from processes in the concrete industry, as mixing water for concrete," TS EN 1008, Turkey, 2003.

[29] Turkish Standards Institute, "Methods of Test for Mortar for Masonory-Part 3: Determination of Consistence of Fresh Mortar (by flow table)," "TS EN 1015-3/A2", Turkey, 2007.

[30] ASTM C666/C666M-15, Standard test method for resistance of concrete two rapid freezing and thawing," ASTM International, West Conshohocken, PA, 2015.

[31] G. Adil, J T. Kevern, D. Mann, "Influence of silica fume on mechanical and durability of pervious concrete," *Construction and Building Materials*, vol. 247, pp. 11845, 2020.

[32] D. D. L. Chung, "Improving cement-based materials by using silica fume," Journal of Materials Science. vol. 37 no. 4, pp. 673-682, 2002.

[33] A. S. Kheder, and N.M. Abou- Zeid, "Characteristics of Silica-Fume Concrete," *Journal of Materials in Civil Engineering*, vol. 6(3), 1994.

[34] P. Zhang, and Q. Li, "Effect of silica fume on the durability of concrete composites containing fly ash,"*Science and Engineering of Composite Materials,* vol. 20(1), pp. 57–65, 2013.

[35] L. E. Tunstall, M. T. Ley, and G. W. Scherer, "Air entraining admixtures: Mechanisms, evaluations, and interactions," *Cement and Concrete Research*, vol. 150, pp.106557, 2021.

[36] C. Foy, M. Pigeon, and N. Banthia, "Freeze-thaw durability and deicer salt scaling resistance of a 0, 25 water-cement ratio concrete," *Cement and Concrete Research*, vol. 18(4), pp. 604-614, 1988.

[37] H. Yazıcı, "The effect of silica fume and high-volume Class C fly ash on mechanical properties, chloride penetration and freeze–thaw resistance of self-compacting concrete," *Construction and building Materials*, vol. 22(4), pp. 456-462, 2008.

[38] B. Zhang, H. Tan, W. Shen, G. Xu, B. Ma, and X. Ji, "Nano-silica and silica fume modified cement mortar used as Surface Protection Material to enhance the impermeability," *Cement and Concrete Composites*, vol. 92, pp. 7-17, 2018.

[39] A. Sadrmomtazi, A. Fasihi, F. Balalaei, and A. K. Haghi, "Investigation of mechanical and physical properties of mortars containing silica fume and nano-SiO2," *In Proceedings of the third international conference on concrete and development*, Tehran, Iran (pp. 27-29), 2009.

[40] M. A. Sanjuán, C. Argiz, J. C. Gálvez, and A. Moragues, "Effect of silica fume fineness on the improvement of Portland cement strength performance," *Construction and Building Materials*, vol. 96, pp. 55-64, 2015.

[41] K. Yamanel, U. Durak, S. İlkentapar, İ. İ. Atabey, O. Karahan, and C. Duran, "Influence of waste marble powder as a replacement on the properties of mortar," Journal of Construction, vol.18 (2), pp. 290-300, 2019.

[42] S. Aparna, D. Sathyan and K. B. Anand, "Microstructural and rate of water absorption study on fly-ash incorporated cement mortar," *Materials Today: Proceedings*, vol. 5(11), pp. 23692-23701, 2018.

[43] F. Collins, J.G. and Sanjayan, "Effects of ultra-fine materials on workability and strength of concrete containing alkali-activated slag as the binder," *Cement Concrete and Research*, vol. 29(3), pp. 459-462, 1999.

[44] R. Choudhary, R. Gupta, R. Nagar, “Impact on fresh, mechanical, and microstructural properties of high strength self-compacting concrete by marble cutting slurry waste, fly ash, and silica fume,” *Construction and Building Materials,* vol. 239, pp.117888, 2020.
[45] M. J. Munir, S.M.S., Kazmi, and Y. F. Wu, “Efficiency of waste marble powder in controlling alkali–silica reaction of concrete: A Sustainable Approach,” *Construction and. Building Materials,* vol.154, pp. 590-599, 2017.
[46] P. Duan, Z. Shui, W. Chen, and C. Shen, “Effects of metakaolin, silica fume and slag on pore structure, interfacial transition zone and compressive strength of concrete,” *Construction and Building Materials,* vol. 44, pp. 1–6, 2013.