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Research Article

Freeze-Thaw Resistance of Normal and High Strength Concretes Produced with Fly Ash and Silica Fume

Cenk Karakurt and Yıldırım Bayazıt

Department of Civil Engineering, Bilecik Seyh Edebali University, 11210 Bilecik, Turkey

Correspondence should be addressed to Cenk Karakurt; cenk.karakurt@bilecik.edu.tr

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This study is based on determination of the freeze-thaw resistance of air-entrained and non-air-entrained normal strength concrete (NC) and high strength concrete (HSC) produced with fly ash and silica fume according to surface scaling. The procedure allows us to measure the amount of scaling per unit surface area due to a number of well defined freezing and thawing cycles in the presence of deicing salt. The weight loss, surface scaling, moisture uptake, and internal damage were measured after 0 and after every 4th freeze-thaw cycle. The test results showed that the freeze-thaw resistance is influenced directly by the compressive strength property of the concrete. Silica fume significantly reduced the resistance of normal strength concrete against freeze-thaw effect without plasticizing agent. The surface scaling of silica fume concrete without admixture was 22% higher than reference normal concrete.

1. Introduction

Concrete is one of the most widely used construction materials for several structures such as buildings, homes, dams, roads, and bridges. Concrete performance is generally based on the mix design, material properties in the mixture, curing conditions, and environmental conditions during the service life of the structure. The most important durability problem of concrete under cold climate is freeze-thaw effect. In particular, dams, bridge deck surfaces, and concrete road pavements with wide open surfaces are under risk of frost at cold climates. This condition can cause the freezing of water inside the capillary pore structure of concrete with 9% of volume expansion. Cracking and spalling of concrete are the most common damage caused by expansion of the cement paste matrix under the effect of freeze-thaw cycles [1].

Several theories have been proposed to explain this type of damage, such as the hydraulic pressure [2], the osmotic pressure [3], and the micro-ice-lens model [4], which are the most important ones. The damage by frost is mainly studied in a laboratory by accelerated freeze-thaw cycles. The extent of damage caused by repeated cycles of freeze-thaw ranges from surface spalling to complete disintegration as layers of ice are formed, starting from the exposed surface of the concrete and extending inwards beneath the surface. Nonetheless, damage due to the frost attack can be reduced either by the reduction of the volume of capillary pores in the concrete by using a lower water to cement ratio or by applying a suitable admixture [5]. Jin et al. [6] concluded that the fractal dimension air voids size distribution had more significant influence on the freeze-thaw resistance of concrete than the air void spacing. The air voids in concrete can be reduced by using fine pozzolanic additives such as silica fume, fly ash, and ground granulated blast furnace slag. The smaller size of the capillary pores in concrete containing silica fume decreases the total amount of freezable water. However, the amount of carbon content in the silica fume and fly ash can cause air void stabilization problems for air-entrained concretes [7]. Researchers investigated the frost resistance of concretes containing different silica fume ratios by mass of cement. The findings of these studies showed that silica fume used concretes freeze-thaw resistances are better than traditional concrete mixtures. Also the water/cement ratio of the mixtures with 0.35 to 0.45 has beneficial effect on the surface scaling of specimens subjected to freeze-thaw cycles [8–10].

Fly ash is another widely used mineral additive for concrete. Nevertheless, this additive can cause some adverse effects on air-entrained hardened concretes when subjected to freeze-thaw effect [11–13] similar with silica fume. As main qualitative parameter of fly ash determining freeze-thaw resistance concrete with mineral addition, the content of the loss on ignition is indicated. Effects of the loss on ignition and fly ash content for reducing strength after freezing and thawing were studied by researchers. The obtained results clearly confirm the negative impact of high loss on ignition in the ashes on the frost resistance of concrete with their addition [14]. Some researchers also proved fly ash is not affected much for resistance to freezing and thawing action in concrete [15, 16]. In addition the cold weather conditions limit the percentage of fly ash that can be used in concrete due to potential retardation in setting and slow strength development when especially subjected to high levels of deicing salts [17, 18]. The objective of this study is to determine the effect of fly ash and silica fume on the freeze-thaw resistance of different strength and air content concretes. The capillary suction of deicing solution and Freeze-thaw (CDF) (test) technique is used to determine the surface scaling of the specimens [5].

2. Materials and Method

2.1. Materials. The raw materials of concrete mixtures were supplied from different sources. CEM I 42.5R type cement was obtained from Eskisehir CIMSA cement factory (Turkey) in accordance with TS EN 197-1 cement code [19]. Fly ash and silica fume were used as mineral for supplementary cementing material. Fly ash used in this study was supplied from Yatagan Thermal Power Plant in Mugla region. Using fly ash in concrete makes it less permeable than normal concrete. Another mineral additive silica fume was obtained from Antalya ETI Electrometallurgy plant. Silica fume is the industrial waste, which can be used as mineral additive for high performance concrete production. The average fineness of silica fume (~200.000 cm^2/g) is approximately 100 times higher than ordinary Portland cement fineness. This higher fineness helps to fill micro pores in the hardened concrete. It makes concrete impermeable but we know silica fume increases plastic shrinkage and water requirement of concrete. Plastic shrinkage causes micro cracking and it reduces durability [20]. The chemical compositions of these binders are given in Table 1.

Aggregate is granular material such as sand, gravel, crushed stone, blast furnace slag, and lightweight that usually occupy approximately 60 to 75% of the volume of concrete. In this study, crushed stone aggregate was supplied by Cimsa Concrete Plant in Eskisehir region, Turkey. Aggregate properties significantly affect the workability of plastic concrete and also the durability, strength, thermal properties, and density of hardened concrete. For this reason, three types of aggregates (0–5, 5–15, and 15–22 mm) were used for adequate gradation of concrete mixtures. The air-entraining agent and superplasticizer used in the concrete mixtures were obtained from SIKA Turkey named as Sika AER and Sikament RCM 310, respectively. Concrete mixtures were produced with Eskisehir tap water.

Table 1: Chemical composition of binders.

Main oxides (%)	CEM I 42.5R	Fly ash	Silica fume
SiO_2	19.96	51.07	92.20
Al_2O_3	5.03	22.65	0.65
Fe_2O_3	2.88	5.83	0.34
CaO	63.60	11.34	0.75
MgO	1.17	2.48	0.38
K_2O	0.80	2.40	0.70
Na_2O	0.27	0.80	0.31
SO_3	2.79	1.69	1.05
Cl^-	0.005	0.004	0.003
LOI	3.02	1.20	0.95

2.2. Method. The concrete specimens were produced as normal concrete (NC), high strength concrete (HSC), silica fume concrete (SFC), and fly ash concrete (FAC). In addition these concretes were produced with air-entraining agent in order to determine the effect of air entraining on freeze-thaw effect. Before the concrete mixed design the gradation and physical properties of the aggregates are determined by sieve analyses, specific gravity, and water absorption tests. Silica fume and fly ash were used with a replacement ratio of 15% by weight of cement in mineral concrete mixtures. Utilization of silica fume more than 15% may increase the water requirement of the concrete mixture. For this reason the optimum mineral additive ratio was chosen as 15%. The mix design of non-air-entrained concretes can be seen in Table 2. Superplasticizer is used only in HSC mixture for 1.5% by weight of cement. The air-entraining agent was used as 0.15% by weight of cement in air-entrained concrete specimens.

The freeze-thaw resistance of the concrete specimens determined in accordance with capillary suction, internal damage, and freeze-thaw (CIF) (test) method. The CIF test is based upon the CDF test, where precision data for scaling have been determined complements this test [21, 22]. In this method, high freezing rate is more pronounced on internal damage than scaling and in terms of scaling damage; slow freezing rate is more destructive compared to high freezing rate [23]. The test procedure consists of three steps: the dry storage, the presaturation by capillary suction, and the freeze-thaw cycles. The test procedure starts immediately after the curing period [5]. The test requires four 150 mm cubes. During the first day after casting the cubes are stored in the moulds and protected against drying by use of a polyethylene sheet. After 24 h, the cubes are removed from the moulds and placed in a water bath having a temperature of (20 ± 2)°C. After curing period the specimens must be sealed on their lateral surfaces. Sealing by aluminum foil with butyl rubber; butyl rubber is glued tightly on the lateral surfaces with an overlap of 20 mm. A durable interconnection must be ensured.

Following dry storage the specimens are placed in the test containers on the 5 or 10 mm high spacers with the test surface on the bottom. The freeze-thaw testing is a cyclic attack. The specimens are exposed to a freeze-thaw cycle in a temperature controlled chest (Figure 1).

Table 2: Concrete mix design for 1 m^3.

Concrete type	Cement (kg)	Water (kg)	w/c	Aggregate (kg)	Fly ash (kg)	Silica fume (kg)
NC	358	165	0.46	1897	—	—
HSC	407	122	0.30	1977	—	102
SFC	358	165	0.46	1897	—	53.7
FAC	358	165	0.46	1897	53.7	—

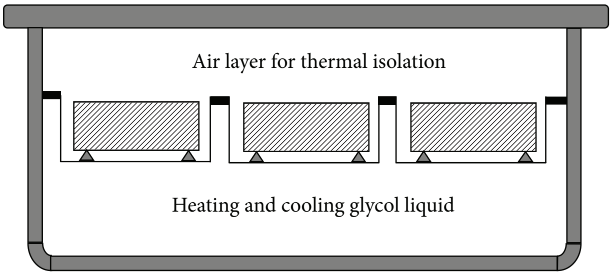


Figure 1: Temperature controlled chest.

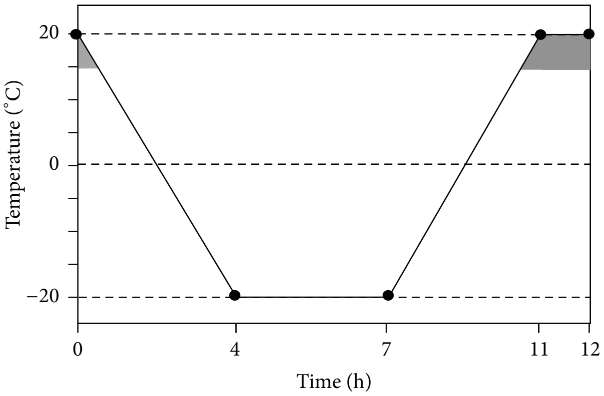


Figure 2: Temperature cycle.

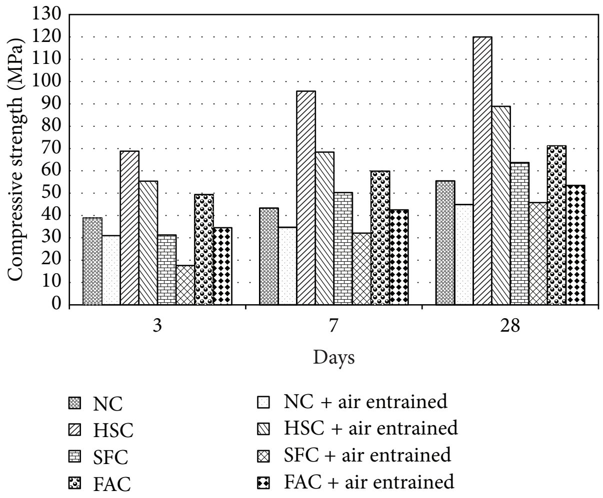


Figure 3: Compressive strength test results.

The temperature of the cooling and heating bath is controlled by an appropriate device. For this purpose automatic Schleibinger CDF/CIF freeze-thaw testing machine is used to apply adequate temperature cycles. The typical temperature variation of a 12 h freeze-thaw cycle can be seen in Figure 2. The temperature cycle is monitored at the reference point. A constant time shift between the test containers is acceptable. The damage parameters are measured while the temperature is above 15°C (shaded area in Figure 2). Machine applies freeze and thaw action during 14 days (28 cycles). An ultrasound water bath is used in order to obtain the sealed material from the surface of the concrete specimens which is subjected to freeze-thaw cycles.

The mechanical property of the concrete specimens is determined by using uniaxial compassion test apparatus on 150 mm cubic specimens. The surface hardness of concrete specimens is obtained with Schmidt Hammer test apparatus. The quality of the hardened concrete specimens also is controlled with ultrasound pulse velocity test machine. This test can give an idea about the stiffness, compactness, and internal damage of the material due to the transmission of the ultrasound waves inside a solid material.

3. Experimental Study

3.1. Compressive Strength Test. Compressive strength is the main important property for determination of the concrete quality. The strength ability of concrete basically depends on the properties of the mix ingredients, water/cement ratio, porosity, and curing conditions. Both produced air-entrained and non-air-entrained high strength and normal strength concrete mixtures were subjected to compressive strength test at ages 3, 7, and 28 days. The compressive strength test results are given in Figure 3.

The 3-day early age strength results showed that HSC specimen reached 69 MPa compressive strength value with the effect of lower water/cement ratio (0.3), higher cement, and silica fume dosage with plasticizer agent in the mixture. The 28-day strength value with air-entraining agent HSC reduced from 120 MPa to 88.90 MPa. The NC, SFC, and FAC concrete series strength values also are influenced by the air entraining inside concrete. The strength values of the silica fume used SFC specimens without plasticizing agent are lower than fly ash used FAC specimens. Despite this, high amount of silica fume used HSC mixture with lower water/cement ratio and plasticizer (Table 2) showed highest compressive strength. This difference is caused due to the absorption of fresh concrete mix water by finer silica fume grains in nonplasticizing agent used SFC mixture.

The strength reduction can be attributed to the reduction in the workability and improper compaction of the fresh SFC mixture with higher porosity. However, fly ash spherical particles increased the workability and compactness of the FAC specimens without any plasticizing agent.

3.2. Schmidt Hammer Test. The Schmidt hammer test involves hitting the in situ concrete with a spring-driven pin at a defined energy, and then the rebound is measured. The rebound depends on the surface hardness of the concrete and is measured by test equipment. By referring to some conversion tables, the rebound result of the test can be used to determine the compressive strength of the concrete. The concrete specimens Schmidt hammer test results are given in Figure 4.

According to test results the surface hardness of the concrete specimens increased with the aging of the specimens. Rebound numbers showed the same behavior when compared with the compressive strength test results. HSC specimen reached 47 rebounds at 28 days. However, a slight reduction occurred for each mixture when air-entraining agent used in concrete. The lowest values were obtained from SFC mixtures at early ages.

3.3. Ultrasonic Pulse Velocity Test. Ultrasonic methods are generally used for analyzing the porous structure and mechanical strength of the concrete and to detect internal defects (voids, cracks, delaminations, etc.) [24]. Mechanical behavior and determination of the internal damage after freeze-thaw test were determined with this test procedure. The test results of the concrete specimens before freeze-thaw test can be seen from Figure 5. The test results showed that SF is more effective on HSC mixture with lower w/c ratio and plasticizing agent. It is well established that silica fume starts to contribute to strength development as early as 3 days after mixing, whereas fly ash takes more than 14–150 days to make any significant contribution to strength development [25]. However, the SFC mixture contains no plasticizing agent. Thus, improper compaction and the entrapped air caused porosity increase with reduced ultrasound pulse velocity values for that type specimen. The air entraining in all concrete specimens is influenced as a reduction in ultrasound pulse velocities. This fact is depending on the increased air content in these mixtures which also caused porosity increase.

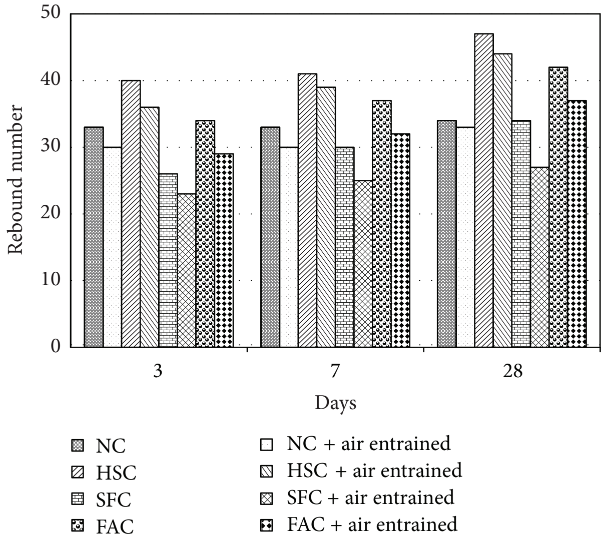


Figure 4: Schmidt hammer test results.

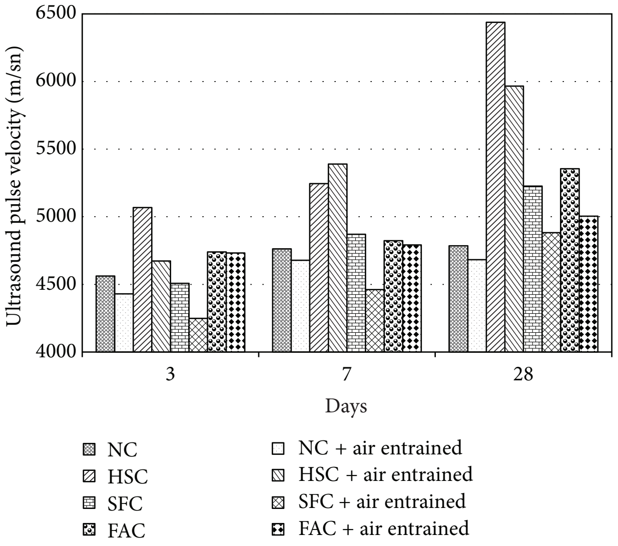


Figure 5: Ultrasound pulse velocity test results.

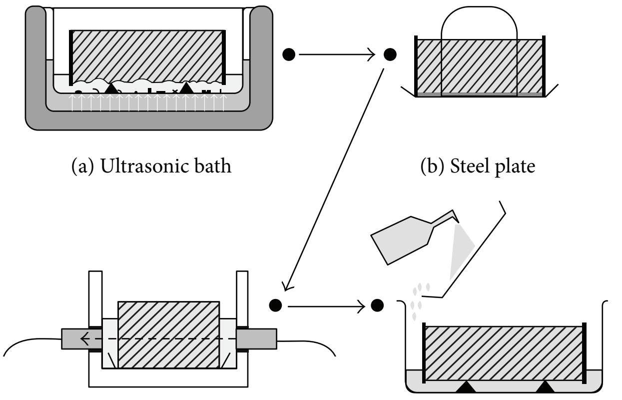
3.4. Freeze and Thaw Tests. Measurements are performed at the start of the freeze-thaw test (0 freeze-thaw cycles) and after every 4th or at least every 6th freeze-thaw cycle and in addition at the agreed criterion. The surface scaling, moisture uptake, and internal damage should be determined according to test procedure. Each 4 cycles the specimens are subjected to ultrasonic bath in order to remove the loosely adhering scaled material from the test surface. The bath solution also filtered by a filter paper to collect the scaled material. After determining surface scaling test specimen is laid on the steel plate to collect additional scaled material. The moisture uptake and internal damage properties are also considered in this method [22]. The flow of the test steps can be seen in Figure 6.

3.4.1. Surface Scaling Results. The solution containing the scaled material is filtered. The mass of the filter containing the dried scaled material μ_b is determined to 0.01 g precision. The mass of the empty filter μ_f is determined prior to filtering with the same precision. The mass of the scaled material μ_s is then determined as

$$\mu_s = \mu_b - \mu_f. \tag{1}$$

The total amount of scaled material related to the test surface after the nth cycle is to be calculated for each measuring interval and each specimen:

$$m_n = \frac{\sum \mu_s}{A}, \tag{2}$$


(a) Ultrasonic bath

(b) Steel plate

(c) Determination of transit time

(d) Collection of remaining scaled material

Figure 6: Specimen handling (reference method) [22].

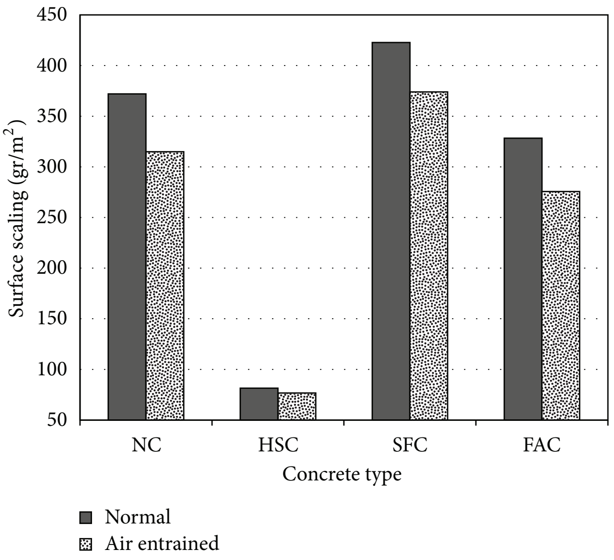


Figure 7: Surface scaling after 28 cycles.

where m_n is the total mass of scaled material related to the test surface after each measuring interval, in g/m^2. μ_s is the mass of scaled material at each measuring interval, in g with an accuracy of 0.01 g. A is the area of the test surface, in m^2. It is calculated on the basis of the linear dimensions.

The scaled material from the specimen surface after 28 freeze-thaw cycles in 3% NaCl solution for different type of concretes can be seen in Figure 7. According to CDF test results the lowest surface scaling was obtained from HSC specimen. This result can be attributed to the higher compressive strength, lower water/cement ratio, and silica fume content with plasticizing agent. It is well known that concrete contains various types of voids. The freeze-thaw damage occurs with the freezing of the water inside the capillary pores of concrete. The water inside the gel pores does not have any significant effect on that damage because water in the gel pores can freeze below −75°C. The capillary pores in HSC mixture filled with very fine silica fume particles therefore the diameter and the amount of the capillary pores reduced. Despite this, the SFC mixture with higher water/cement ratio and without plasticizer caused a decrease in the freeze-thaw resistance. This phenomenon can depend on the increased porosity of the specimens under the effect of reduced workability.

Fly ash used normal concrete specimen showed better performance than other normal concrete mixtures. The effect of fly ash on freeze-thaw resistance of concrete was studied by Michta. To achieve the freeze-deicing salt resistance of fly ash concrete, air entraining is not only necessary but also appropriate minimum value of water/binder = 0.38. However, the concretes with w/b = 0.45 showed a lack of resistance to frost with deicing [14]. According to Figure 7 the fly ash used (FAC) non-air-entrained and air-entrained concrete specimens surface scaling results are lower than normal concrete specimen as 12% and 12.5%, respectively. The water/binder ratio of prepared FAC mixture was 0.40 and showed similar scaling results with the mentioned study.

The empty entrained air voids generated by air-entraining admixture provide a reservoir for the water to escape when it freezes, thereby reducing the disruptive stresses [7]. The beneficial effect of air entraining in NC specimen can be clearly seen in Figure 8. The air-entraining agent reduced the surface scaling of NC, FAC, and SFC mixtures as 15, 16, and 11%, respectively.

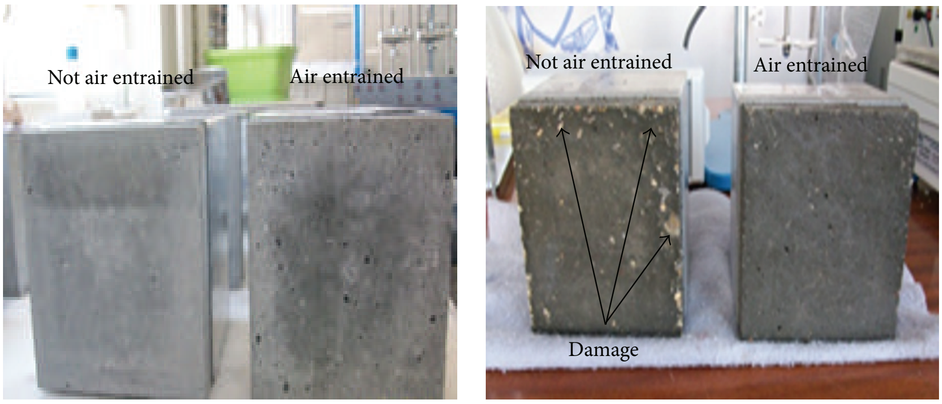
3.4.2. Moisture Uptake Results. After removing the scaled material from the test surface the specimens are placed vertically on an absorbent surface (laboratory towel) to permit the run-off of water from the test surface. The relative increase in mass of each specimen Δwn after the nth cycle is calculated by

$$\Delta w_n = \frac{w_n - w_1 + \sum \mu_s}{w_0} w * 100, \qquad (3)$$

where Δw_n is the moisture uptake in mass of each specimen after the nth cycle and μ_s is the mass of total scaled material at each measuring interval, in g with an accuracy of 0.01 g. w_0 is the reference mass of the each specimen without sealing mass after prestorage, in g. w_1 is the mass of each specimen including sealing mass before resaturation starts, in g. w_n is the mass of each specimen at each interval, in g.

Moisture uptake results are given in Figure 9. The test results showed a similar behavior as surface scaling test results. The increase in capillary pores caused an increase in the moisture uptake values for the SFC specimens. This effect can be attributed due to the lack of adequate compaction of the SFC mixtures without plasticizer. The reduced porosity of HSC with lower water/cement ratio and silica fume led to decrease the moisture uptake of these specimens.

3.4.3. Internal Damage. Internal damage is the deterioration of the internal structure of concrete which leads to a change in the concrete properties. The internal damage of concrete specimens was determined according to RILEM TC 176 method [22]. Dynamic modulus of elasticity was calculated according to ultrasonic transit time determination.



(a) Before (b) After

Figure 8: The effect of air entraining.

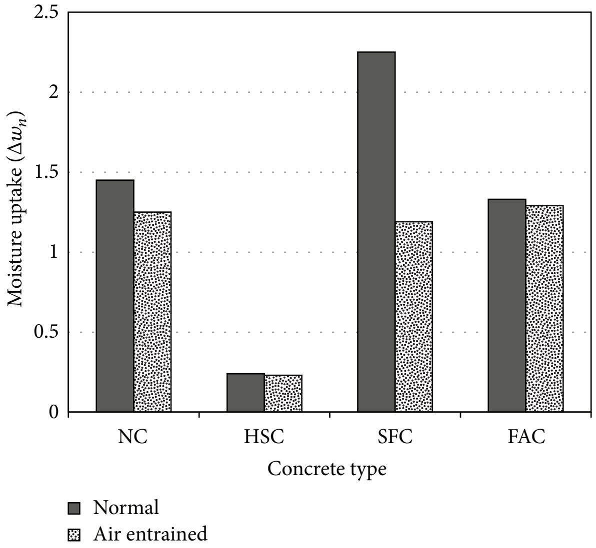


Figure 9: Moisture uptake test results.

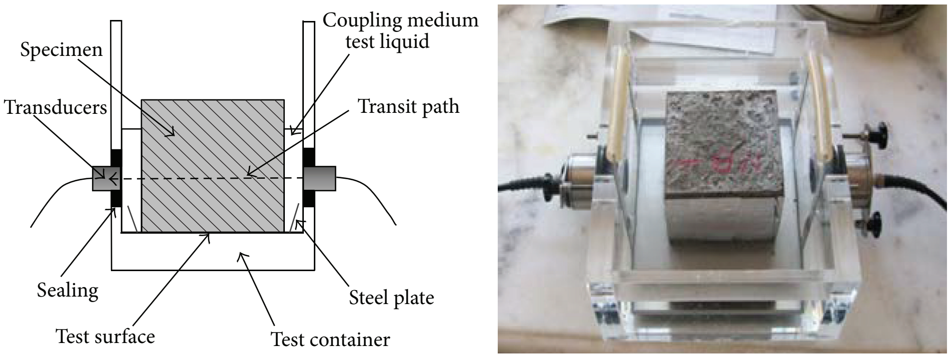


Figure 10: Ultrasound transit time determination.

As defined in CIF method the damage criterion is below the level 80%. The system of ultrasound transit time measurement on concrete specimen was shown in Figure 10.

The relative dynamic modulus of elasticity (E_{dyn}) results after 28 cycles was given in Figure 11. According to damage criterion all concrete types except for SFC are above the damage criterion. This behavior occurred with the improper compaction of SFC due to the increased water requirement. However, the highest E_{dyn} values are obtained from the HSC specimen. The air entraining in concrete increased

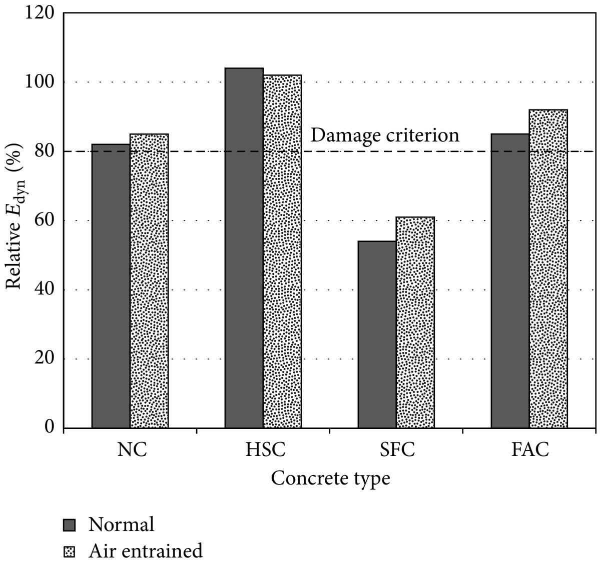


Figure 11: Relative dynamic modulus of elasticity.

the durability of concrete against freeze-thaw effect. Nevertheless, by reduction of the w/c ratio below 0.35 with decrease of the amount of freezable water, a higher frost resistance should be guaranteed, assuming that incompatibility problems between the cement and superplasticizer are prevented [26].

4. Conclusions

This study is carried out in order to determine the effect of water/cement ratio and air entraining on different strength concretes produced with fly ash and silica fume. According to test results, the following conclusions may be drawn:

(i) Air entraining to concrete decreased compressive strength on all concrete type. But it increased workability and freeze-thaw resistance.

(ii) High strength concrete surface is not destroyed in both concrete which are air-entrained and non-air-entrained. It was found that the surface scaling of HSC was 4.24 times lower than NC. This behavior can be attributed to the higher compressive strength with lower water/cement ratio (0.30) and proper compaction with plasticizer.

(iii) Silica fume concrete surface was much damaged than other types of concrete. This fact is dependent on the reduced workability and proper compaction of SFC specimen with increased capillary porosity.

(iv) Fly ash showed better performance than silica fume for concrete mixtures without plasticizing agent at 0.40 water/binder ratio.

(v) It is important to reduce the capillary pores in the composite in order to improve the freeze-thaw resistance of concretes.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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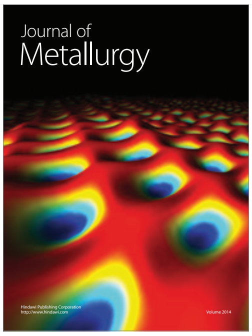
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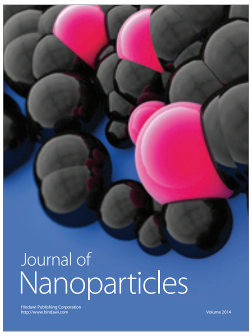
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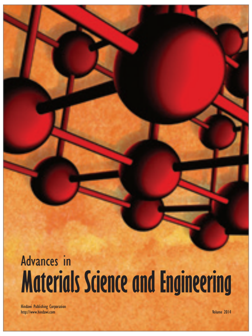
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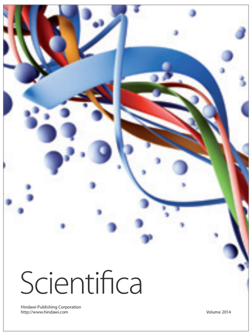
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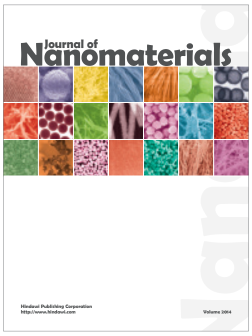
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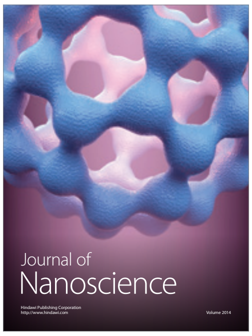
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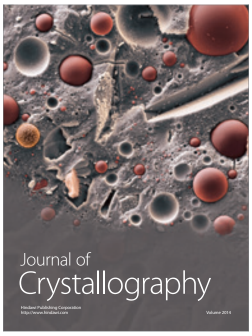
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