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EVALUATION OF CURING TIME FOR MICRO CONCRETE MIXES CONTAINING SILICA FUME, NANO-SILICA AND FLY ASH

SİLİKA DUMANI, NANO-SİLİKA VE UÇUCU KÜL İÇEREN MİKRO BETON KARIŞIMLARI İÇİN KÜR SÜRESİNİN DEĞERLENDİRİLMESİ

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Abstract

Within the scope of the study, research on the use of silica fume (SF), nano silica (NS) and fly ash (FA) together or separately in the production of micro concrete is presented. It is aimed to examine the changes in mechanical properties because of water and air curing in mixtures produced using SF, FA, and NS. While cement dosage and water/binder ratio in the mixtures were chosen as 670 kg/m^3 and 0.53 respectively, the amount of SF, FA and NS was limited to 150 kg/m^3 in total. In the study, samples were produced using 40x40x160 mm prism molds. All samples were divided into two different groups after 7 days of water curing and water (1st group) and air (2nd group) were applied up to 56 days. Flexural and compressive strength tests were performed on the water and air cured specimens for 7-56 days and 28-56 days, respectively. In addition, the porosity and unit volume weight values of the samples were examined. The results show that both flexural and compressive strengths of micro concretes increased after 28 days thanks to water curing.

Keywords: Fly ash, micro-concrete, nano silica, silica fume**.**

Öz

Çalışma kapsamında mikro beton üretiminde silis dumanı (SD), nano silika (NS) ve uçucu külün (UK) birlikte veya ayrı ayrı kullanımına yönelik araştırmalar sunulmaktadır. SD, UK ve NS kullanılarak üretilen karışımlarda su ve hava kürlenmesi nedeniyle mekanik özelliklerde meydana gelen değişikliklerin incelenmesi amaçlanmaktadır. Karışımlarda çimento dozajı ve su/bağlayıcı oranı sırasıyla 670 kg/m^3 ve 0.53 olarak seçilirken SD, UK ve NS miktarı toplamda 150 kg/m^3 ile sınırlandırılmıştır. Çalışmada 40x40x160 mm prizma kalıpları kullanılarak numuneler üretilmiştir. Tüm numuneler 7 gün su küründen sonra iki farklı gruba ayrıldı ve 56 güne kadar su (1. grup) ve hava (2. grup) uygulandı. Su ve hava ile kürlenen numunelere sırasıyla 7-56 gün ve 28-56 gün boyunca eğilme ve basınç dayanımı testleri yapılmıştır. Ayrıca numunelerin gözeneklilik ve birim hacim ağırlık değerleri incelenmiştir. Sonuçlar, su kürü sayesinde mikro betonların hem eğilme hem de basınç dayanımlarının 28 gün sonra arttığını göstermektedir.

Anahtar Kelimeler: Micro beton, nano silika, silis dumanı, uçucu kül.

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1. INTRODUCTION

Micro-concrete is a type of cement-based material that is produced with aggregates reduced to the level of cement fineness when choosing the maximum aggregate particle size and can offer many usage options. Considering these usage purposes, it may be possible to use it selectively, especially during the repair or strengthening of reinforced concrete structures, if the micro-concrete mixture and its components are selected and used in appropriate proportions. On the other hand, it has a production potential as an element that can be used as a plate in prefabricated buildings (Felekoğlu, 2009). The term micro-concrete was first used in the 1960s-70s to describe mixtures without coarse aggregates prepared for the purpose of modeling the structures to be produced in small scales with reinforced concrete at scale suitable for research (Aldridge & Breen, 1970; Litle & Paparoni, 1966). An example is the use of different types of micro-concrete designs for repair purposes for a different use (Dhir & Roderick Jones, 1996; Jumaat et al., 2006; Nounu & Chaudhary, 1999). Another feature is that they can be produced as panel elements thanks to various molding properties and methods, and the micro-concrete products that researchers have patented and produced are also included in the literature (Felekoğlu, 2009).

With the increasing population rate, a global increase in housing and building stock is formed. It creates an increasing trend in the interest in concrete/reinforced concrete, which is a fast and traditional production technique. This situation is increasing in parallel with the new construction and repair works and the cement industry. Consumption that occurs in parallel with this increased capacity is considered as the cement industry being among the industries that consume the most energy. Moreover, approximately 6-7% of annual global human-induced CO2 emissions are produced by this sector (Biricik & Sarier, 2014; Pacheco-Torgal et al., 2013; Palomo et al., 2011; Sanchez & Sobolev, 2010). There are many studies by researchers to minimize the dosage of cement used in the reinforced concrete and concrete-based construction industry (Pacheco-Torgal et al., 2013). Compounds or mixtures with different properties have been experimentally investigated instead of ordinary Portland cement, which is required to reduce cement usage, instead of cement-specific materials (Said et al., 2012; Zhang & Islam, 2012) with pozzolanic or hydraulic binding properties. Among the cement composites, fly ash (FA) and silica fume (SF) (Hou et al., 2013; Jo et al., 2007) are most common, which are deposited as by-products or waste products by the coal industry and the silicon-ferrosilicon industry, respectively. When FA and SF are used, they improve durability properties as well as long-term strength gains like cement composites, mainly due to pozzolanic reactions with calcium hydroxide that occur during the hydration of calcium silicates (Gaitero et al., 2010; Garboczi, 2009; Hou et al., 2013; Jo et al., 2007; Mazloom et al., 2004). On the other hand, there are concerns about some property improvements in the production process of blends formed with FA or SF. For example, strength development in concrete produced with F class FA is slower than normal concrete. For this reason, it is not preferred in many applications that require early strength, such as the need for repair or rapid construction (Said et al., 2012; Zhang & Islam, 2012). On the other hand, although SF has a large specific surface area, it exhibits a more sensitive behavior in terms of plastic shrinkage in concretes where it is used compared to other concrete types (Al-Amoudi et al., 2004). Recently, the substitution of nanoparticles into cement paste, mortar and concrete produced with ordinary Portland cement (OPC) and its ability to produce superior mechanical and physical properties in OPC-produced concrete-derived formations has created an interesting field. This has been demonstrated by studies that nanotechnology can be directly applied in the construction industry (Lee et al., 2010). In this respect, studies in the literature show that the inclusion of products containing nanoparticles in the concrete content improves the fresh and hardened state properties compared to the mineral additives traditionally used in concrete mixes (Lee et al., 2010; Li et al., 2004; Mukhopadhyay, 2011). In this context, it shows that the nanoparticle SiO_2 (nano silica, NS) component may become an increasingly important component for special concretes and other advanced cement-based productions. Thanks to the particle size that can be produced at nanoscale,

a high surface-area/volume ratio is achieved, while at the same time, it has been shown that advanced chemical reactivity potential can occur in concrete thanks to its amorphous silicon dioxide structure (Biricik & Sarier, 2014; Fahmy et al., 2020; Jalal et al., 2015).

Within the scope of the study, micro concrete mixtures were produced using SF, FA and NS. In the mixtures produced, in addition to the mixtures containing only SF, only FA and SF-FA, a total of 4 mixtures containing SF-FA-NS were produced. In addition to the effects of these different combinations used on microconcrete, it is aimed to examine the effects on the mechanical properties of these mixtures as a result of water curing and air curing. For this purpose, a preliminary evaluation study on the combined use of SF, FA and NS contents in micro concrete is presented. In addition, 2 groups of sample orders were created in the study. The first group consists of samples that are cured with water curing for up to 7 days and await the next test age. In the second group of samples, there are samples that are tested after water curing up to 28 days of age. Both sets of samples were subjected to compression tests after the flexural tensile test.

2. MATERIALS AND EXPERIMENTAL CAMPAIGN

Within the scope of the study, the details of the materials used in the production of the mixtures and the methodological procedures showing the production and post-production test stages are included in the sub-headings. A brief flowchart of the study methodology is given in Figure 1.

Figure 1. Experimental Workflow

2.1. Materials

CEM-I 42.5R Portland cement, SF, FA, and NS were used as binders in the mixtures produced within the scope of the study. The micro-aggregate river sand in the mixtures was sieved through a 1 mm perforated sieve. Chemical properties of SF, FA, NS, and cement are given in Table 1. Sieve analyzes of microaggregates obtained by sieving are given in Figure 2.

Table 1. Chemical Composition of Binder

Chemical composition (%)	Portland Cement	Silica Fume	Fly Ash	Nano Silica
SiO_2	19.79	90.75	60.94	99.8
AlO_3	3.96	-----	-----	-----
Al_2O_3	3.85	0.72	20.66	-----
Fe_2O_3	4.15	2.29	7.95	-----
CaO	61.84	0.56	2.32	-----
MgO	3.22	-----	-----	-----
K_2O	-----	1.51	-----	-----
Na_2O	-----	0.55	1.56	-----
SO_3	2.32	0.51	0.11	-----
P_2O_5	-----	-----	-----	-----
TiO	-----	-----	-----	-----
Cr_2O_3	-----	-----	-----	-----
Mn_2O_3	-----	-----	-----	-----
MgO	-----	-----	-----	-----
Loss of ignition	0.87	3.11	1.92	-----
Blaine (cm^2/g)	3260	2108	3790	150000

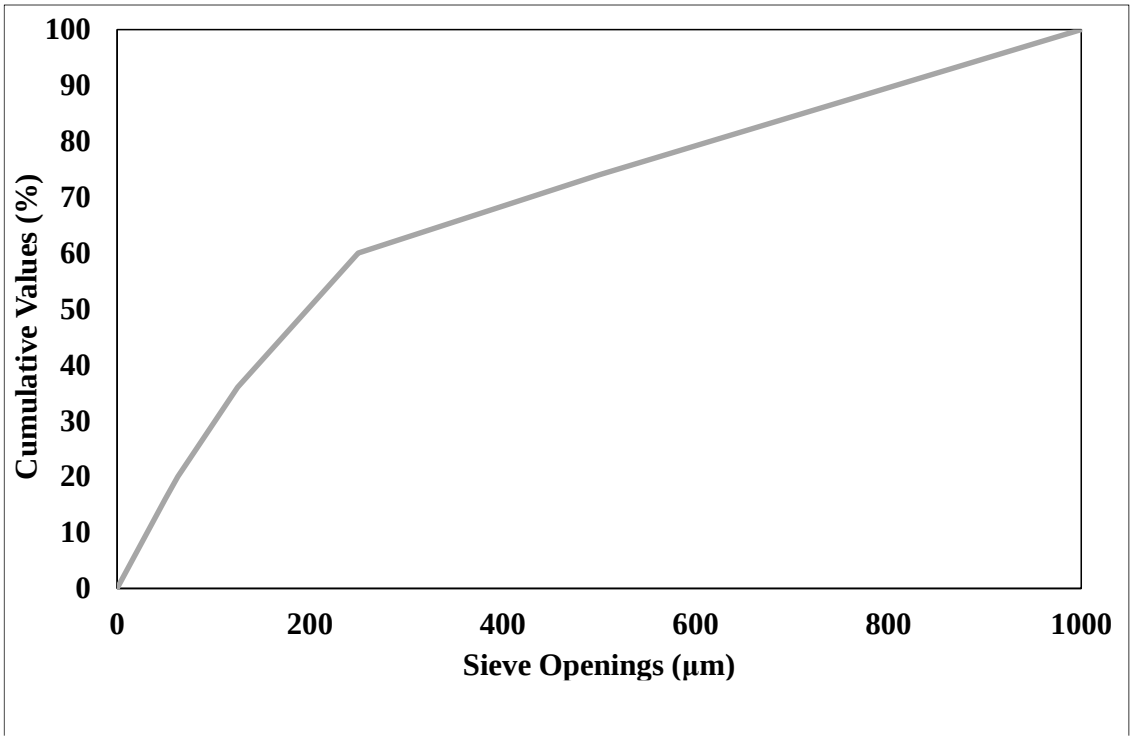

Figure 2. The Sieve Analysis of Micro Aggregate

In addition, water, and superplasticizer, namely polycarboxylate ether-based water-reducing chemical additive (HRWR) was used to increase the workability capacity of the blends. In this way, it is aimed to maintain the homogeneity of the mixtures produced, as well as to have high fluidity capacities. The specific gravity values used for cement, SF, FA, NS and HRWR, which are the materials used in the mixtures produced as micro-concrete, were calculated as 3.1, 2.2, 2.00, 2.2 and 1.055, respectively.

(a) (b) (c) (d) (e)

Figure 3. Mix materials a) Cement, b) SF, c)NS, d)FA and e)micro aggregate

2.2. Mix Design

The mixtures produced were produced in 4 groups. Each mixture group has a total binder content of 820 kg/m^3. The amounts of SF, FA and NS in the mixture were used as 150 kg/m^3. Therefore, the amount of Portland Cement in each mixture is 670 kg/m^3. In the mixtures produced, natural river sand was sieved and used instead of microaggregate (0-1 mm). A total of 4 mixtures were produced. In the group mixtures produced, 40x40x160 mm^3 prism samples were produced for mechanical testing for each mixture. For each mix group, 15 samples were produced.

During the preparation of the produced micro-concrete components by mixing, it was produced homogeneously with the help of a standard mortar mixer specified by the existing standards (ASTM, 2009) until the total homogeneity of the mixture was observed. As summarized in Table 2, the water/binder (w/b) ratio of 0.53 was used in the mixtures. The amount of water used was calculated as 434 kg/m3. The mixtures were basically produced in 2 groups and the contents of SF, FA and NS were changed in these groups, provided that the dosage of the binder remained constant. When the mixtures were examined, the binder content was 18% SF in the first mixture, 9% SF and 9% FA in the second mixture, 6% SF, 10% FA and 2% NS in the third mixture. The mixtures were completed by using 18% FA of the binder dosage in the final mixture (Table 2).

Table 2. Mix Design

	Cement	Silica Fume	Fly Ash	Nano Silica	Water	HRWR	Micro-aggregate
MIX-ID	kg/m^3						
MC1	670	150	0	0	434	13	1130
MC2	670	75	75	0	434	13	1130
MC3	670	50	83	17	434	13	1130
MC4	670	0	150	0	434	13	1130

2.3. Sample Facture and Mixing Procedure

During the production of micro concrete, a standard mixer given in Figure 4a was used. In addition, the flowability of the produced micro-concrete mixtures was checked using mini-slump and their

workability was tested. In the first stage of mixing, the dry mix of micro-concrete cement, SF, FA, NS and micro-aggregate was mixed for one minute. Then, 1/3 of the mixing water was added to the dry mixture and mixed for 1 more minute. Afterwards, 2/3 mixing water and HRWR were added to the mixture by mixing and the mixture was continued for 1 more minute. Finally, the mixture, which was rested for 30 seconds, was mixed for 1 more minute and placed in the molds after being visually and manually examined. The images obtained during the mini slump flow test are given in Figure 4b. The fresh mixtures produced were filled into 40x40x160 mm prism molds (Figure 4c). The samples were removed from these molds after 24 hours (Figure 4d) and allowed to water cure at 20±2°C (Figure 4e). While the samples were curing, one batch was removed from the pool after the 7-day curing period, and the remainder continued to cure for 28-days. After the completion of the curing periods in water, the remaining samples for the 7-, 28- and 56-days tests were taken from the curing tank.

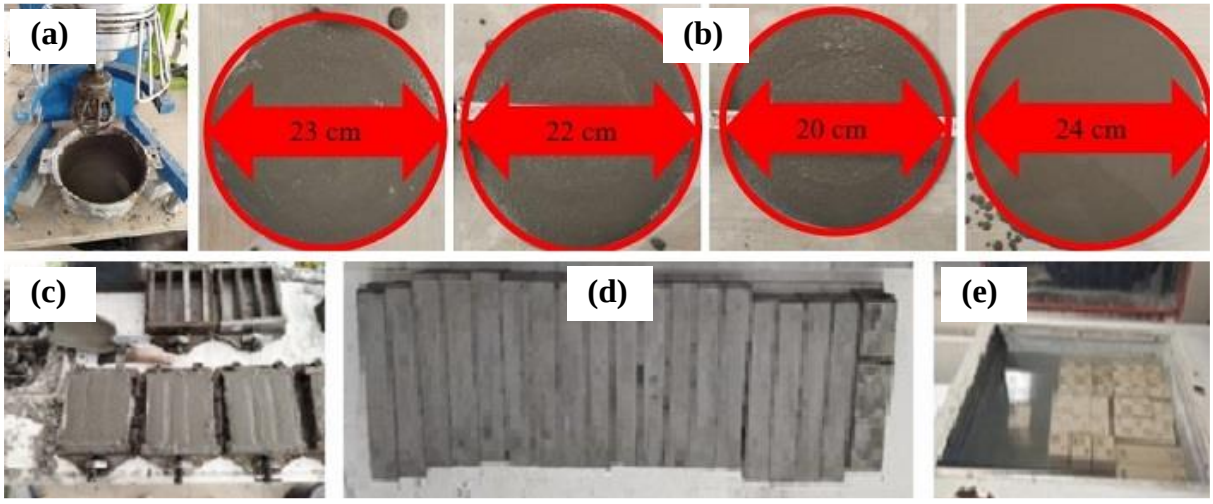

Figure 4. a) Mixer, b) Slump Flow c) Molding, d) Demolding and e) Water Curing

2.4. Hardened State Testing Procedure

The mixtures produced were filled into metal molds while they were fresh and removed from the molds after 24 hours. Afterwards, the samples were left to cure in lime-saturated water at 20±2°C. Within the scope of the study, mechanical tests were applied to 7-, 28- and 56-days old samples. Flexural and compression tests were performed in accordance with ASTM C348-14 (ASTM C348-19, 2018) and ASTM C349 (ASTM C349-08, 2014). 40x40x160 mm prism molds were used to determine the effects of 7-, 28-, and 56-day age water curing times on flexural and compressive strength. Three prismatic samples were used for testing from each mixture and age group. Firstly, the broken prisms obtained from the flexural tensile test were used for the determination of compressive strengths after testing for the prism specimens subjected to the flexural tensile test. The compressive strength of the fractured prisms was also determined at 7-, 28-, and 56-days ages. The load value obtained because of the flexural strength test according to the ASTM C348-14 (ASTM C348-19, 2018) code was converted to the flexural strength value by using the S_f=0.0028×P equation. In this equation, S_f and P are defined in the relevant standard as flexural strength and maximum load, respectively. The units of Sf and P used in the equation are MPa and N, respectively. In addition, for the compressive strength of the broken prisms after the flexural test, the test in accordance with the ASTM C349 (ASTM C349-08, 2014) standard was applied and the compressive strength was obtained using the S_c=0.00062×P equation. In this equation, S_c and P are defined in the relevant standard as compressive strength and maximum load, respectively. The units of S_c and P used in the equation are MPa and N, respectively.

Axial compressive strength tests were applied to the prism samples produced within the scope of the study, to the prism parts that were divided into two after the flexural tensile strengths. During the application, a loading speed of 0.9 kN/s was realized with a universal test device. Since the average of 3 prisms was taken in flexural tensile strength, it was applied to 6 broken prism pieces consisting of these prisms in the compressive strength tests. Flexural tensile strengths tests were applied to the prism samples produced within the scope of the study. During the application, a load of 0.5 kN/s was made with a universal test device. When presenting the results obtained, the mix ID is coded for water-cured samples by adding the term "-W". Similar to the air-cured portions, the mixture is coded in the notation by adding the term "-A" next to the mix ID.

In the study, 2 groups were formed, the first group was taken to the laboratory environment after curing in water for 7 days, and wet tests were performed for 28 and 56 days. In the second group, after completing the 28-day age with cure, mechanical testing was performed at 28 and 56 days of age. Three 40x40x160 mm prisms containing air and water cure were randomly selected for each design set for flexural strength testing according to ASTM C348 (ASTM C348-19, 2018). Then, the axial compressive strength test according to ASTM C349 (ASTM C349-08, 2014)was performed on the parts consisting of broken prisms (Figure 5b). Hardened prisms and instant test views are shown in Figure 5a.

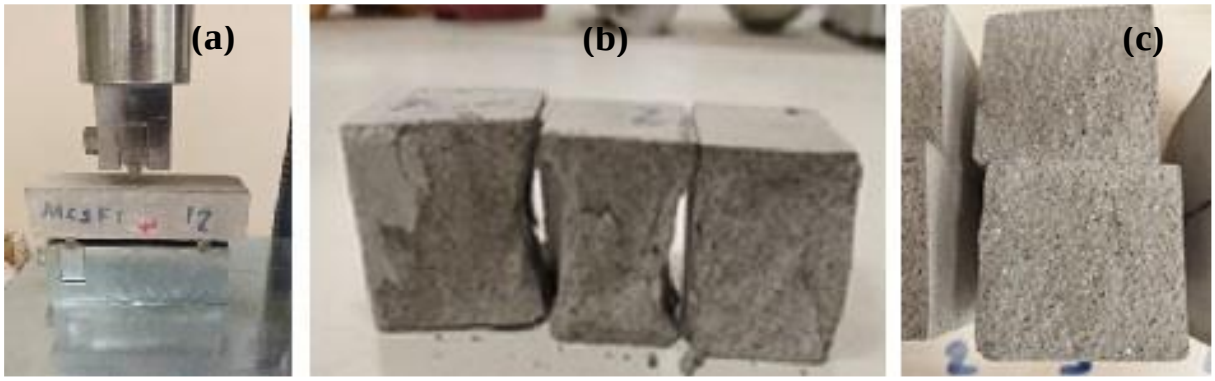

Figure 5. a) Prisms for Flexural Strength, b) Broken Prisms After Test and c) Fracture Surface in Flexure

2.5. Porosity and Bulk Density Tests

It is stated in the literature that it provides information about cohesion, discontinuity and porosity thanks to porosity and bulk density tests (Torres & García-Ruiz, 2009). For this reason, porosity and bulk density tests were carried out in accordance with ASTM C-642 (ASTM C642-13, 2014) standard recommendations, as soon as 28-day-old prism samples were cured. Within the scope of porosity and bulk density tests, the weights of prism samples in water and surface dry saturated weights were measured. Weight measurements using the ASTM C-642 standard have been made within the scope of other studies in the literature from past to present (ASTM C642-13, 2014). After the water contact weights were taken, the samples were kept in an oven at 100 ± 2 ºC for 24 hours until their weights remained constant. The porosity and bulk density of the samples were calculated according to ASTM C-642 (ASTM C642-13, 2014). The $Porosity\,(\%) = (w_3 - w_1)/(w_3 - w_2)$ and $Bulk\ Density = w_1/(w_1 - w_2)$ equations are used to make these calculations. In the equation, w_1 is the final weight that the samples could reach at a constant temperature, w_2 is the measured weight of the fully saturated sample, and w_3 is the weight of the surface-dried saturated.

3. RESULTS

3.1. Flexural Strength Test Results

Obtained results are presented in the Figure 6. Within the scope of the study, the flexural tensile strengths of MC1-W, MC2-W, MC3-W and MC4-W mixture samples at 7 days of age were obtained as 4.303, 4.321, 3.845 and 6.291 MPa, respectively. The flexural tensile strength of the mixtures obtained after 28 days of water curing increased by 14%, 37%, 77% and 11%, respectively, compared to the results obtained after 7 days. When the results obtained at the age of 56 days were examined, it was observed that there was an increase of 86%, 69%, 150% and 16% compared to the flexural tensile strengths obtained at the age of 7 days (Figure 6).

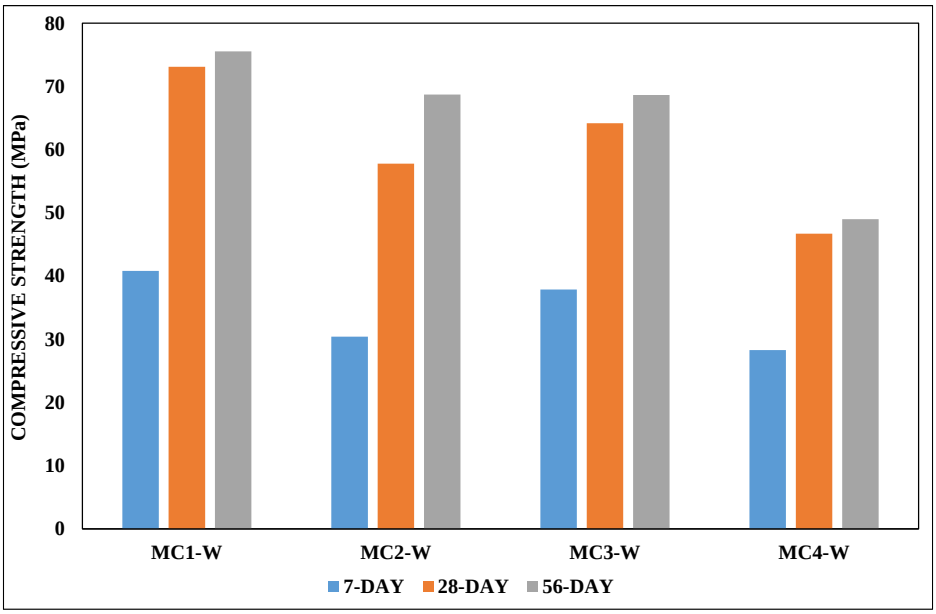

Figure 6. Flexural Strength Results of Water Cured Samples

The flexural tensile strengths of the second group samples, MC1-A, MC2-A, MC3-A and MC4-A mixture samples at 28 days of age, were obtained as 4.303, 4.321, 3.845 and 6.783 MPa, respectively (Figure 7). The flexural tensile strength results of MC1-A, MC2-A, MC3-A and MC4-A blends obtained over 28 days were 7%, 20%, 59% and 7% higher, respectively, than the 7-day samples. The flexural tensile strength obtained because of water curing at 28 days of age was calculated as 6%, 14%, 11% and 8% higher, respectively, than the flexural tensile strength of the samples stored in the laboratory. A difference of 18%, 9%, 49% and 2% was observed between the samples cured with water for 56 days and the samples kept under laboratory conditions. The flexural tensile strength obtained from water-cured samples is higher.

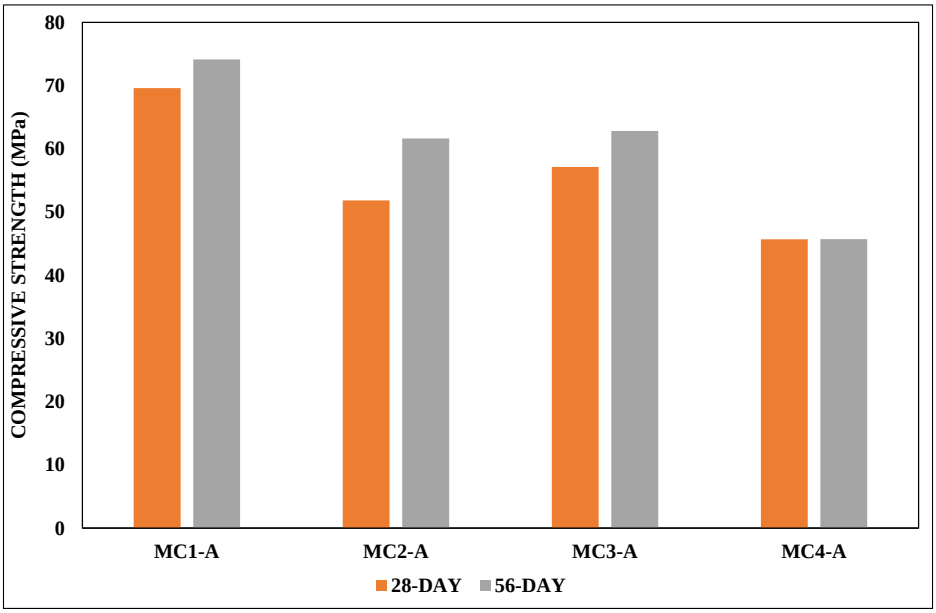

Figure 7. Flexural Strength Results of Air Cured Samples

3.2. Compressive Strength Test Results

Compressive strengths were determined by taking the average of these 6 samples compressive strength test results. Results for the 7-day-old MC1-W, MC2-W, MC3-W and MC4-W samples were 40.8, 30.4, 37.9 and 28.29 MPa, respectively (Figure 8). This mixture given in other samples was evaluated respectively. It was calculated that the samples cured with water for 28 days were 79%, 90%, 69% and 65% higher, respectively, compared to the 7-day age. It was calculated that samples cured with water for 56 days were 85%, 126%, 81% and 73% higher, respectively, compared to 7 days (Figure 8).

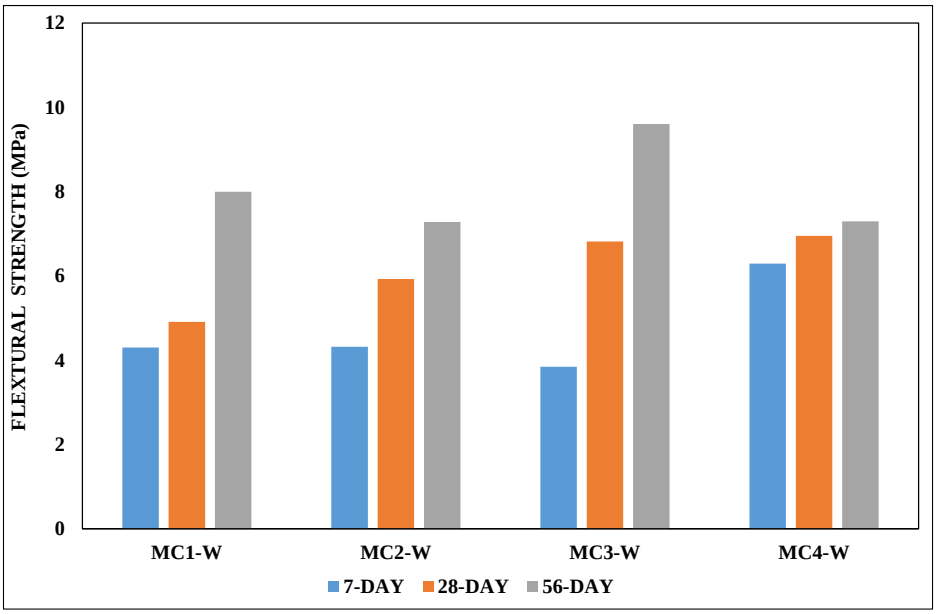

Figure 8. Compressive Strength Results of Water Cured Samples

Results for the 28-day MC1-A, MC2-A, MC3-A, and MC4-A samples were 70%, 70%, 51%, and 61% higher, respectively, than the 7-day test samples (Figure 9). The 28-day water-cured samples were 5%, 11%, 12%, and 2% higher than the non-water-cured samples. At 56 days of age, water-cured samples were 2%, 11%, 9%, and 7% higher than non-water-cured samples (Figure 9).

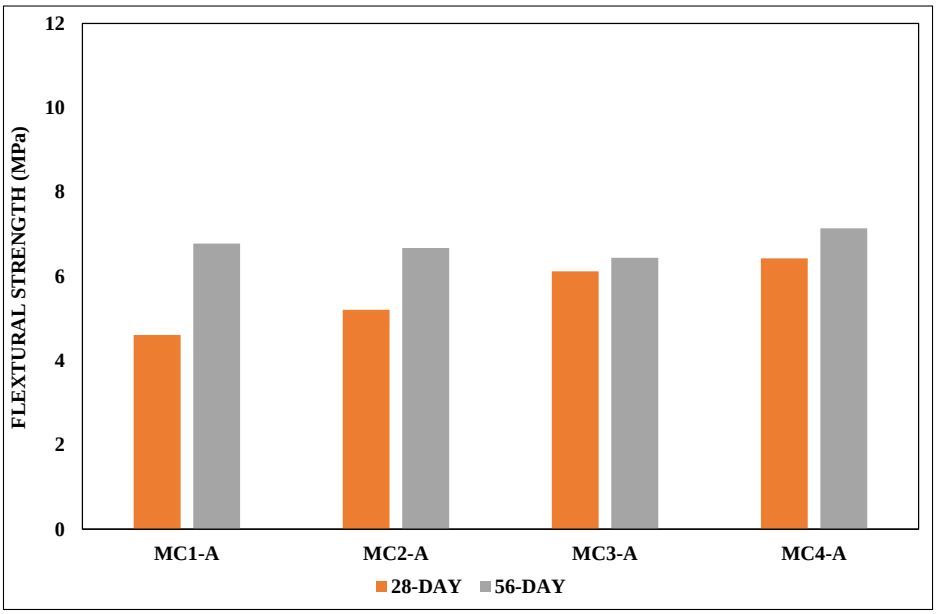

Figure 9. Compressive Strength Results of Air Cured Samples

3.3. Physical Properties

Within the scope of the study, necessary measurements were made for the porosity and bulk density of the samples removed from the curing tank at the end of the 28-day curing period. Measurements were made on all prismatic samples removed from the curing tank. Obtained porosity results were calculated as 26.53%, 28.25%, 29.43% and 29.05% for MC1, MC2, MC3 and MC4, respectively (Figure 10).

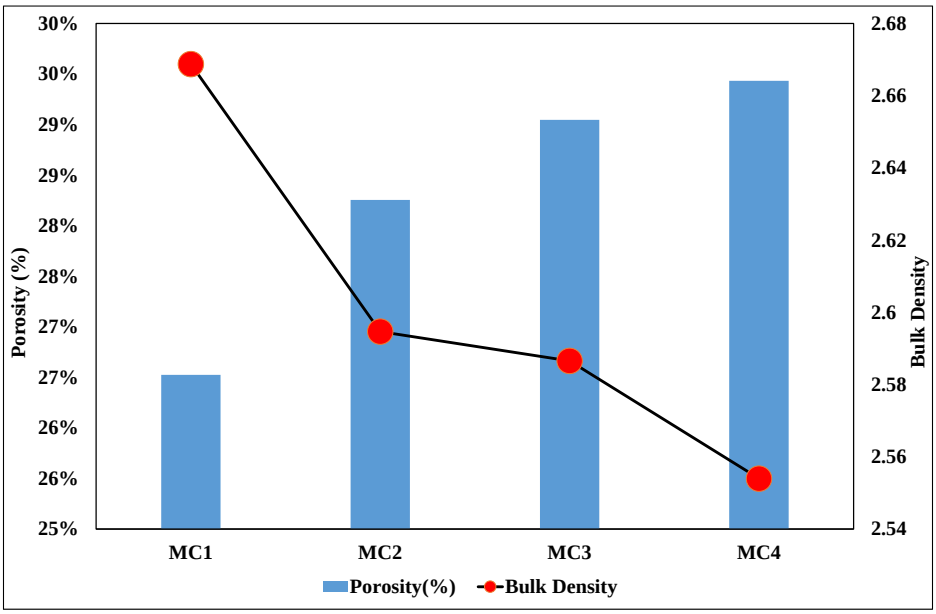

Figure 10. Porosity and Bulk Density Results

For the lowest porosity values, the compressive strength of the MC1-W mixture reaches the highest value of 73 MPa in 28-day age tests. The highest porosity value was achieved in the MC4-W mixture, with a flexural tensile strength of 6.9 MPa. The bulk density value varies between 2.55 and 2.67. For 28 days of age, the lowest bulk density value was 2.55 and the lowest compressive strength was obtained in 46.69 MPa MC4-W mixture. The highest bulk density value at 2.67 was obtained from the MC4-W mix with the highest flexural strength of 6.9 MPa for 28-days age (Figure 10).

4. CONCLUSIONS

Within the scope of the study, it was evaluated how the micro-concrete mixtures with SF, FA and NS contents changed in terms of their mechanical properties at the end of different water curing periods. The results obtained are summarized below.

- Only micro-concrete mixtures with SF content show the best compressive strength.
- When SF and FA are used together, significant reductions in micro-concrete compressive strength can occur for 28-days age.
- When NS is added in addition to SF and FA, there is a significant increase in the compressive strength caused by FA.
- In flexural tensile denier, it is seen that FA content produces better results in contrast to SF and NS.
- While SF reaches the lowest porosity value, the substitution of FA and the use of FA only increases the porosity.
- As a result of the prolongation of the water curing period, remarkable strength gains stand out for the 28-day age, especially in the mixtures containing SF-FA and SF-FA-NS.

Statement of Research and Publication Ethics
Research and publication ethics were observed in the study.

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