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Effect of Acidic Water on Physico-Mechanical Properties of Concrete Containing Micro-Silica

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Abstract

This work investigates the effect of adding micro-silica as a pozzolan and a replacement for part of concrete cement when placing concrete in an acidic environment. Two types of ordinary concrete and concrete-containing micro-silica are constructed. The specimens are subjected to 0, 1, 5, and 10 cycles for two hours inside two types of acidic water containing sulfuric and nitric acid with pH = 3 and normal water with pH = 7. Mechanical properties including Brazilian tensile strength and uniaxial compressive strength, and physical properties including effective porosity, water absorption, and the longitudinal wave velocity of specimens are determined after the mentioned number of cycles. Thr results show that by decreasing the pH and increasing the number of cycles, the effective porosity and water absorption increase, and the velocity of longitudinal waves, Brazilian tensile strength, and uniaxial compressive strength of concrete decrease. Replacing 10% of micro-silica as a part of concrete cement has boosted the durability of concrete in corrosive conditions containing sulfuric and nitric acid more than ordinary concrete.

1. Introduction

Each project must consider increasing concrete efficiency in various environments to stabilize a concrete structure. The increasing use, vogue, and ease of use of concrete have caused it to be used in projects under different conditions.

Various factors cause a concrete structure to become unstable and disrupt the process of a project and its operation. Many environmental factors as the weather conditions of the region (such as the occurrence of acid rain phenomena due to the high percentage of pollution, successive wetting, and drying caused by rain and freezing phenomenon in cold regions), the surrounding environment of the structure (such as the placement of concrete structures in marine environments with a high percentage of corrosiveness of salts, leakage of acid drainage into concrete foundations and leakage of underground water), and the usage of the structure (such as the effect of dissolution and corrosion of the structure due to the flow of corrosive fluids inside it such as water transfer tunnels and industrial wastewater and wells) threaten the stability of the concrete structure [1-3].

Environmental conditions are essential factors affecting the physical and mechanical properties of rock and cement-based materials such as concrete, shotcrete or mortar. One of the environments that affect concrete is environments with corrosive conditions [4]. According to the type of environment such as marine environments and salt solutions in it, industrial environments with the percentage of carbon dioxide pollution and toxic gases and acid rains, the corrosion rate and durability of concrete structures are different. Therefore, preventing the annihilation of concrete structures is always considered one of the concerns of each project [5].

Multiple types of research works have been conducted to prevent the destruction of concrete structures, the results of which have provided

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numerous achievements and suggestions. One of the most common proposed methods is strengthening concrete using different additives. Additives can be replaced as part of the cement such as pozzolan or added to the concrete as a percentage of the total volume of concrete produced such as glass fibers and polypropylene. One of the pozzolans used in concrete is micro-silica or silica fume. Due to its active silica, micro-silica can convert calcium hydroxide in concrete cement, considered a weakening factor of concrete, into calcium silicate. Correspondingly, its fine grain size can fill concrete voids. Therefore, permeability, one of the essential effective parameters in the durability of concrete structures, is reduced by adding micro-silica and prevents concrete degradation caused by corrosive fluid conditions [1].

Numerous researchers have accomplished different studies that showed the effectiveness of micro-silica on the properties of concrete. For example, Mazloom *et al.* [6], regarding research on micro-silica in concrete, showed that adding micro-silica increases concrete compressive and tensile strength at young ages of concrete.

Other additives can improve the physical and mechanical properties of concrete in addition to micro-silica such as metakaolin, zeolite powder, and glass fibers (Table 1).

Table 1. A review of studies on the effect of different additives on the properties of concrete.

References	Type of additives	Results
Deshmukh *et al.* [7]	Glass fibers	Increasing flexural, tensile, and compressive strength
Wankhede and Fulari [8]	Fly ash	Compressive strength: increase (in replacement case, it decreases by more than 20%)
Lekha and Vanreyk [9]	Zeolite powder	Compressive strength: increase (in old ages of concrete)
Usman *et al.* [10]	Recycled plastics	Increasing flexural, tensile, and compressive strength
Zhang *et al.* [11]	Metakaolin	Tensile strength: increase (in replacement case, it decreases by more than 15%) Compressive strength: increase (in replacement case, it decreases by more than 15%) Weight loss: decrease (in replacement case, it increases by more than 15%)
Pirmohammdi Alishah *et al.*, [12]	Pumice	In 28-day-old concrete self-compacting specimens, compressive strength decreased. Although increasing the amount of pumice by up to 10% increases the compressive strength of 90-day-old concrete samples by 11%, the strength decreases by adding more than 10% of pumice

In freezing conditions, utilizing micro-silica enhances the properties of concrete, reduces permeability, and makes the concrete specimens more enduring than ordinary concrete [13]. In high-temperature conditions, using micro-silica in the concrete mixing design as a part of concrete cement causes the concrete more durable [14].

Investigating the effect of acid environments as one of the corrosive conditions on concrete containing micro-silica in such environments is extremely important. The severity of corrosion and durability of concrete in an acidic environment depends on the type of acid and its pH (power of hydrogen) ratio. Sulfuric acid and nitric acid attacks are the most common corrosive environments.

Sulfuric and nitric acid are the most harmful acids for concrete structures. Sulfuric acid can react with calcium hydroxide in concrete to form calcium sulfate. Calcium sulfate with the C3A (a compound in Portland cement) can create the mineral "Ettringite" (or hydrous calcium aluminum sulfate), which increases the internal pressure in concrete due to the increase in volume and increases cracks inside of concrete specimens. Nitric acid is weaker than sulfuric acid but still affects concrete and can produce calcium nitrate in a chemical reaction with calcium hydroxide. Calcium nitrate as a salt weakens the cement paste of concrete. The effect of nitric acid is more significant in short-term exposure to concrete than sulfuric acid [15].

Barbhuiya and Kumala have investigated the effect of acidic corrosive environments on concrete and the effect of different pozzolans in concrete in sulfuric or nitric acid conditions [15]. In this laboratory study, the effect of adding Fly Ash (FA) and Ultra Fine Fly Ash (UFFA) including five different mixing designs (normal concrete without any FA and UFFA, 20% FA with 10% UFFA, 30% FA with 10% UFFA, 40% FA with 10% UFFA, and 50% FA with 10% UFFA) in the conditions of 1.5% nitric acid and 3% sulfuric acid after 28 and 90 days was investigated. The results showed that in nitric acid condition, uniaxial compressive strength of concrete containing 20% FA with 10% UFFA, 30% FA with 10% UFFA, and in sulfuric acid condition, 30% FA with 10% UFFA were less than other mixing designs.

Mahdikhani *et al.* [16] investigated the effect of sulfuric acid rain on concrete containing nano-silica. Their study showed that sulfuric acid rain at different pH ratios reduced the strength of concrete, and comparing two types of normal concrete and concrete containing nano-silica, specimens containing nano-silica have better compressive strength and durability than ordinary concrete specimens.

Hosseini *et al*. [17], in a study on the effect of sulfuric acid rain on the mechanical and physical properties of concrete, showed that replacing 5% limestone powder and 10% micro-silica as part of concrete cement enhanced the strength parameters and reduced water absorption and effective porosity of concrete. Concrete containing micro-silica and limestone powder performs better than ordinary concrete specimens in all no-rain, normal rain with pH = 7, and sulfuric acid rain with pH = 2 conditions.

The corrosive environments of concrete are not limited to acids. Several studies have been conducted, the results of which have led to suggestions for improving the mechanical properties of concrete in non-acidic corrosive conditions. For example, Ramezanianpour *et al*. [1] showed that the increase in the replacement rate of limestone in concrete in the corrosive environment of sodium sulfate and magnesium sulfate decreases the compressive strength of concrete. Hosseini *et al*. [18] showed that using 10% micro-silica (in concrete containing micro-silica) and 10% zeolite powder (in concrete containing zeolite powder) replacement as a part of cement in concrete increases the durability of concrete compared to normal concrete in an environment containing corrosive water containing 14% chlorine ions and sodium sulfate.

Although sulfuric acid is more potent than nitric acid, most concrete structures are exposed to nitric acid pollution, so it is essential to investigate the performance of concrete in these conditions compared to sulfuric acid. In the past research works (such as the researches of Mahdikhani *et al*. and Hosseini *et al*.), the effect of acidic water containing sulfuric acid on the properties of concrete has been investigated without considering the effect of wetting-drying cycles. However, in this study, the effect of acidic water containing nitric acid on the properties of concrete has been investigated considering the effect of wetting-drying cycles. Furthermore, in this study, the factor of the chemical reaction of acid and concrete has been investigated. The mentioned items are among the innovations of this research work.

2. Materials

This study used Portland cement type 2 with grade 350, sand, gravel, superplasticizer, and micro-silica to make concrete specimens. The coefficient of the softness of the sand is 3.11. Consumed gravel is passed through sieve 3/8 in, and sand is passed through sieve No. 4. A gradation test was conducted according to Iranian National Standard No. 302 [19]. The granulation diagrams of gravel and sand used are given in (Figures 1 and 2).

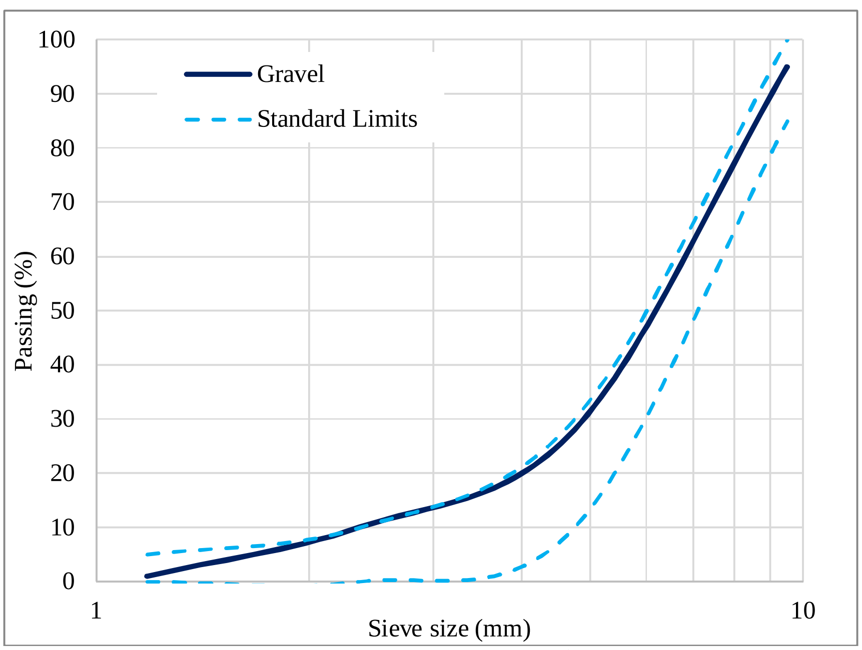

Figure 1. Granulation diagram of gravel.

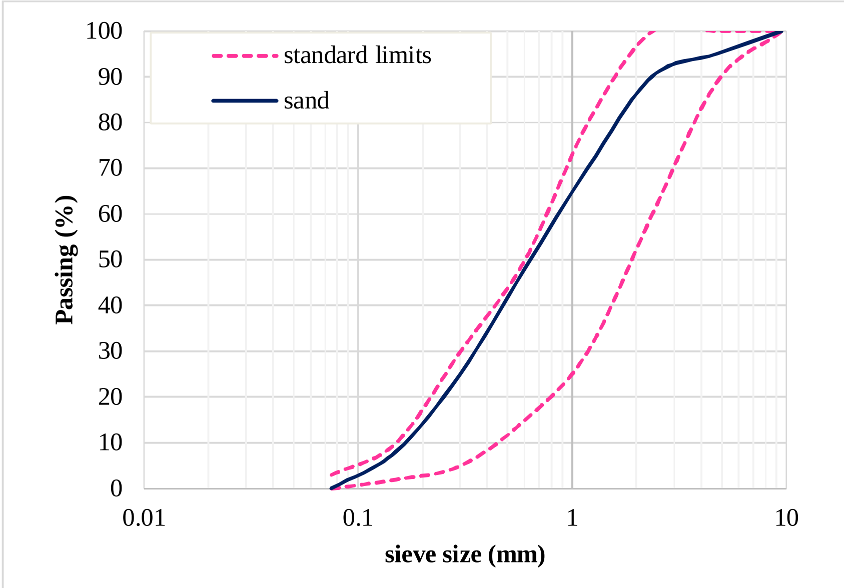

Figure 2. Granulation diagram of sand.

In the mixing design, the percentage of micro-silica is selected based on the studies of Mazloom *et al.* [6], Zahiri *et al.* [20], and Zareei *et al.* [21]. Subsequently, the results of XRF (X-ray fluorescence) of sand, gravel, and micro-silica used in this research work are given in Table 2. Furthermore, the mixing designs used in this experimental study are given according to (Tables 3 and 4) for constructing two types of concrete including normal concrete and concrete containing micro-silica to produce one cubic meter of concrete.

Table 2. XRF analysis of the sand, gravel, and micro-silica samples.

Combinations	Sand (%)	Gravel (%)	Micro-silica (%)
SiO_2	26.6	18.3	99.5
Al_2O_3	15.2	1.4	0.008
Fe_2O_3	11.6	1	0.019
Na_2O	3.9	0.4	0.03
K_2O	2.3	0.5	0.001
CaO	16.1	52.1	0.23
MgO	2.1	0.88	0.01
SO_3	<0.1	<0.1	0.03
TiO_2	2.1	<0.1	0.002
MnO	0.2	<0.1	-
P_2O_5	0.2	<0.1	-
L.O.I	19.3	24.31	0.008

Table 3. Mixing design for ordinary concrete (for one cubic meter).

Components	Measure
Portland cement (Kg)	350
Sand (Kg)	1150
Gravel (Kg)	700
Water (Kg)	140
Superplasticizer (Kg)	3.15

Table 4. Mixing design for concrete containing micro-silica (for one cubic meter).

Components	Measure
Portland cement (Kg)	315
Sand (Kg)	1150
Gravel (Kg)	700
Micro-silica (Kg)	35
Water (Kg)	140
Superplasticizer (Kg)	3.15

For constructing concrete specimens containing micro-silica, micro-silica pozzolan is first added to water, then water containing micro-silica to the materials inside the mixer including sand, gravel, and cement. According to the ISRM standard and the mixing design mentioned in Tables 3 and 4, concrete cylindrical specimens with a height of 120 mm and a diameter of 60 mm were constructed [22]. After 24 hours, the specimens are removed from the mold and placed inside the water containing calcium carbonate ($CaCO_3$) for 28 days until the samples are cured [23]. After this period, the samples of the Brazilian test are prepared in the form of disks with a thickness-to-diameter ratio of approximately 0.5 [24].

3. Method

The prepared specimens should be placed under wetting–drying cycles. Waragai [25] proposed the experimental method for this purpose.

In this research work, three tanks including normal water (with pH = 7), water containing nitric acid (with pH = 3), and water containing sulfuric acid (with pH = 3) are prepared. In each cycle, the specimens are placed in the tank for two hours and then in a dry environment for one hour. The pH value was selected based on the study of Dyre [26] and Hosseini and Fakhri [27], Laboratory tests were conducted according to the International Society of Rock Mechanics (ISRM) standards.

Throughout this work, OCN represents ordinary concrete in normal water, OCSW is ordinary concrete in water containing sulfuric acid with pH = 3, OCNW is ordinary concrete in water containing nitric acid with pH = 3, CMN represents concrete containing micro-silica in normal water, CMSW is concrete containing micro-silica in water containing sulfuric acid with pH = 3, and CMNW is concrete containing micro-silica in water containing nitric acid with pH = 3.

4. Effect of Sulfuric and Nitric Acid on Physico-mechanical Behaviour

4.1. Effective porosity

The dimensions of specimens were precisely measured by callipers to calculate their volumes in order to determine the effective porosity. After that, the specimens were saturated in water for 1 h under a vacuum pressure of less than 800 Pa, and the saturated mass was calculated. The specimens were then placed in a 105 °C oven for 6-7 h before being measured.

For each case, five specimens were tested to determine the effective porosity. The mean results for the concrete specimens are shown in Table 5.

Table 5. Mean effective porosity of concrete specimens.

Type of concrete	No. of cycles	Effective porosity (%)
OCN	0	5.67
	1	5.67
	5	5.84
	10	6.10
OCSW	0	4.21
	1	4.37
	5	4.88
	10	5.24
OCNW	0	4.45
	1	4.74
	5	4.89
	10	5.07
CMN	0	2.88
	1	2.89
	5	3.02
	10	3.11
CMSW	0	2.53
	1	2.57
	5	3.12
	10	3.31
CMNW	0	2.80
	1	2.94
	5	3.12
	10	3.19

4.2. Longitudinal wave velocity

In order to measure the longitudinal wave velocity, transducers were placed on the designated locations, and the transmitter was pushed on the specimen with a pressure equivalent to 10 N/cm^2, and then the wave passage time was recorded. The wave motion distance, the centre-to-centre distance of transducers was also measured. In order to obtain the lost time between the specimen and the transducers, transducers were placed on each other, and after that the displayed time was deducted from the recorded times.

For each case, five specimens were tested to determine the longitudinal wave velocity. Table 6 shows the mean results for the concrete specimens.

Table 6. Mean longitudinal wave velocity in concrete specimens.

Type of concrete	No. of cycles	longitudinal wave velocity (m/s)
OCN	0	4014.3
	1	4011.8
	5	4008.9
	10	3998.2
OCSW	0	4099.5
	1	4061.8
	5	4000.5
	10	3982.7
OCNW	0	4086.4
	1	4027.1
	5	4018.2
	10	4010.1
CMN	0	4258.2
	1	4255.9
	5	4236.1
	10	4228.9
CMSW	0	4302.5
	1	4289.1
	5	4265.8
	10	4186.3
CMNW	0	4139.2
	1	4116.7
	5	4100.0
	10	3981.2

4.3. Water absorption

Water absorption was calculated using Equation 1, respectively [28].

$$WA = \frac{B-A}{A} x100 \tag{1}$$

where B is the mass of specimen immersed in water after 48 h, A is the dry mass, and WA shows water absorption. Tables 7 represent the mean results for the concrete specimens.

Table 7. Mean water absorption of concrete specimens.

Type of concrete	No. of cycles	Water absorption (%)
OCN	0	1.51
	1	1.50
	5	2.05
	10	2.56
OCSW	0	1.62
	1	1.96
	5	2.76
	10	3.14
OCNW	0	1.80
	1	1.92
	5	2.56
	10	2.62
CMN	0	1.03
	1	1.07
	5	1.13
	10	1.17
CMSW	0	0.98
	1	1.11
	5	1.21
	10	1.46
CMNW	0	1.09
	1	1.19
	5	1.32
	10	1.37

4.4. Tensile strength

Brazilian test was the rehearsal for the first time between 1930 and 1960 at the National Institute of Brazil (INT) in Rio de Janeiro. In 1940, when Brazil joined World War II, this test contributed significantly to the determination of the tensile strength of concrete [24].

Table 8. Mean Brazilian tensile strength of concrete specimens.

Type of concrete	No. of cycles	BTS (MPa)
OCN	0	4.55
	1	4.48
	5	4.21
	10	4.13
OCSW	0	4.55
	1	4.28
	5	3.86
	10	3.39
OCNW	0	4.55
	1	4.40
	5	4.12
	10	3.78
CMN	0	5.62
	1	5.58
	5	5.37
	10	5.02
CMSW	0	5.58
	1	5.39
	5	5.08
	10	4.78
CMNW	0	5.58
	1	5.05
	5	4.76
	10	4.61

4.5. Uniaxial compressive strength (UCS)

Uniaxial compressive strength is considered one of the most critical mechanical parameters of concrete. To determine the uniaxial compressive strength, samples with a ratio of length to diameter equal to two have been prepared. Having the diameter and calculating the initial cross-sectional area of the samples after applying the axial load by the loading device by recording the axial load at the moment of sample's failure.

The mean results of UCS for the specimens of ordinary concrete, and concrete containing micro-silica are given in Table 9.

Table 9. Mean uniaxial compressive strength of concrete specimens.

Type of concrete	No. of cycles	USC (MPa)
OCN	0	36.1
	1	35.7
	5	35.2
	10	34.8
OCSW	0	36.1
	1	34.2
	5	33.7
	10	31.9
OCNW	0	36.1
	1	33.3
	5	32.4
	10	31.6
CMN	0	49.8
	1	49.9
	5	48.9
	10	48.2
CMSW	0	49.8
	1	47.9
	5	45.1
	10	42.7
CMNW	0	49.8
	1	48.4
	5	46.1
	10	44.3

5. Discussion

Patil *et al*. [30], by studying on microscopic images of concrete containing micro-silica and nano-silica showed that micro-silica and nano-silica in the form of small and almost spherical crystals increase the contact of concrete particles with each other, and reduce the pores in concrete. Reducing pores cause reducing water absorption, and effective porosity.

$$SiO_2 + Ca(OH)_2 \rightarrow CaSiO_3 + H_2O \qquad (2)$$

According to Equation 6, activated silica is able to convert calcium hydroxide ($Ca(OH)_2$) in concrete cement, which is considered a weakening factor of concrete, into calcium silicate ($CaSiO_3$) [1].

$CaSiO_3$ is more resistant to nitric acid, and sulfuric acid than $Ca(OH)_2$, so the replacement of micro-silica as part of concrete cement reduces the amount of $Ca(OH)_2$, and has more durability in acidic environment [31]. Therefore, the tensile and compressive strength of concrete containing micro-silica is more than normal concrete. By increasing the number of wetting-drying cycles of concrete in an acidic environment, the porosity and water absorption increased, and the longitudinal waves velocity, tensile and uniaxial compressive strength decreased (Figures 3 to 7).

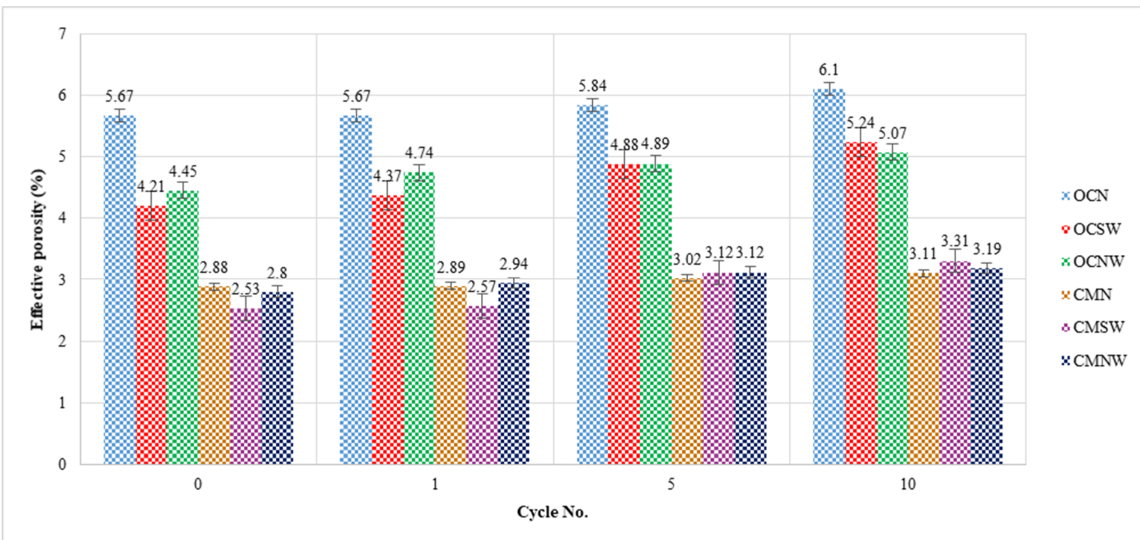

Figure 3. Effect of number of wetting-drying cycles on effective porosity.

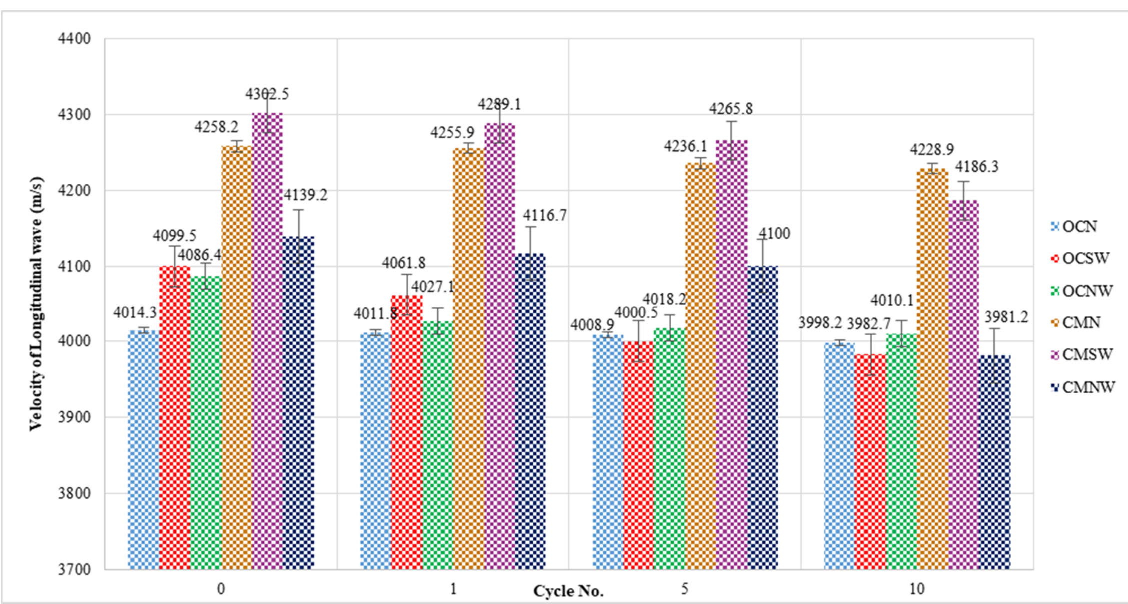

Figure 4. Effect of number of wetting-drying cycles on longitudinal waves velocity.

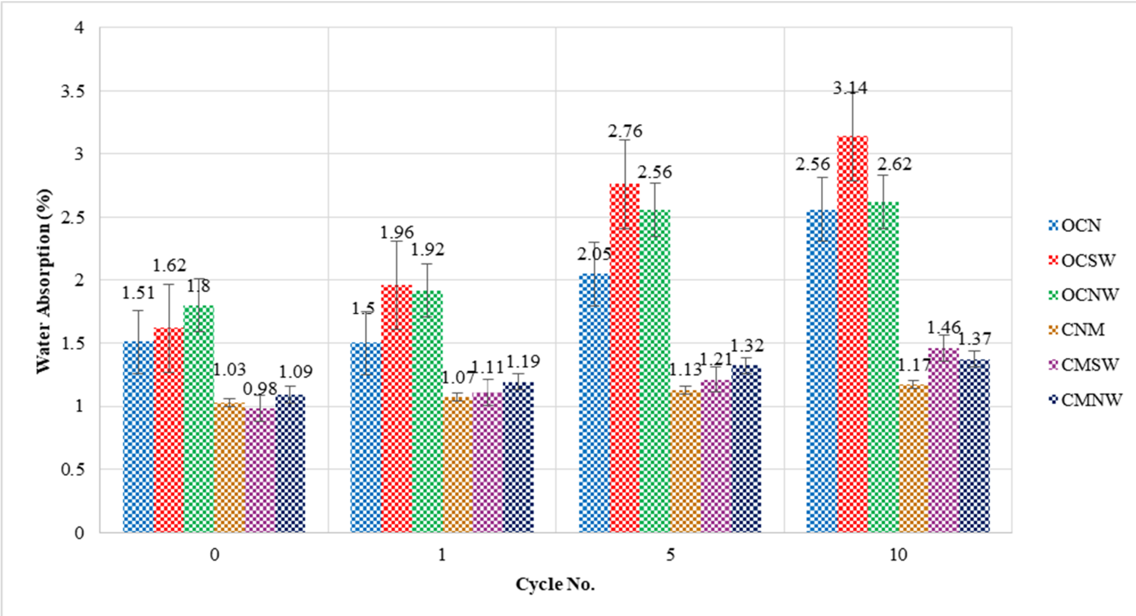

Figure 5. Effect of number of wetting-drying cycles on water absorption.

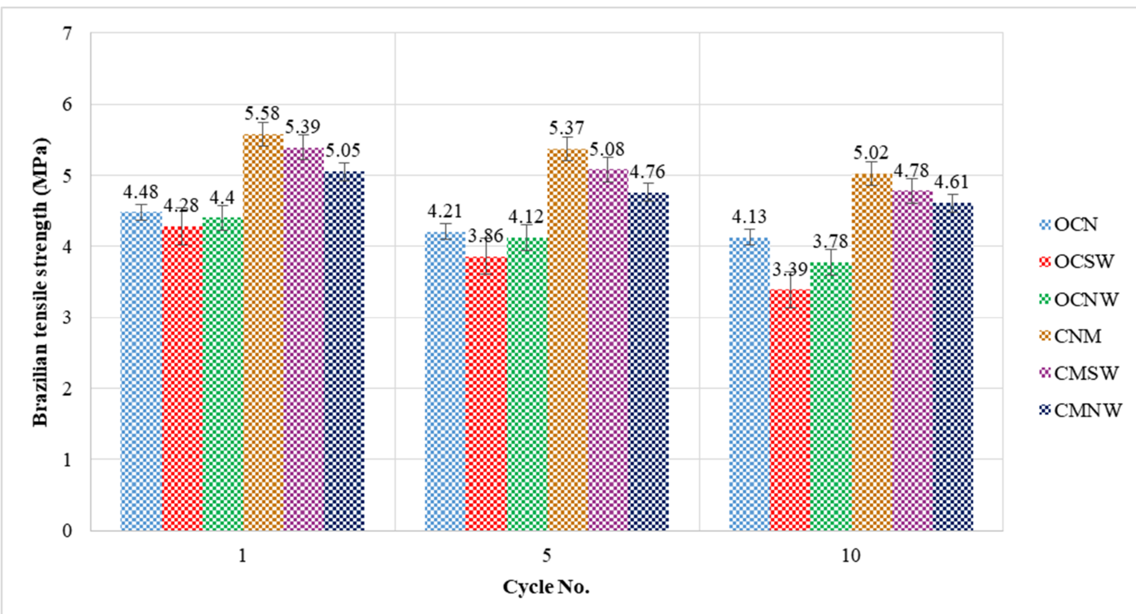

Figure 6. Effect of number of wetting-drying cycles on tensile strength.

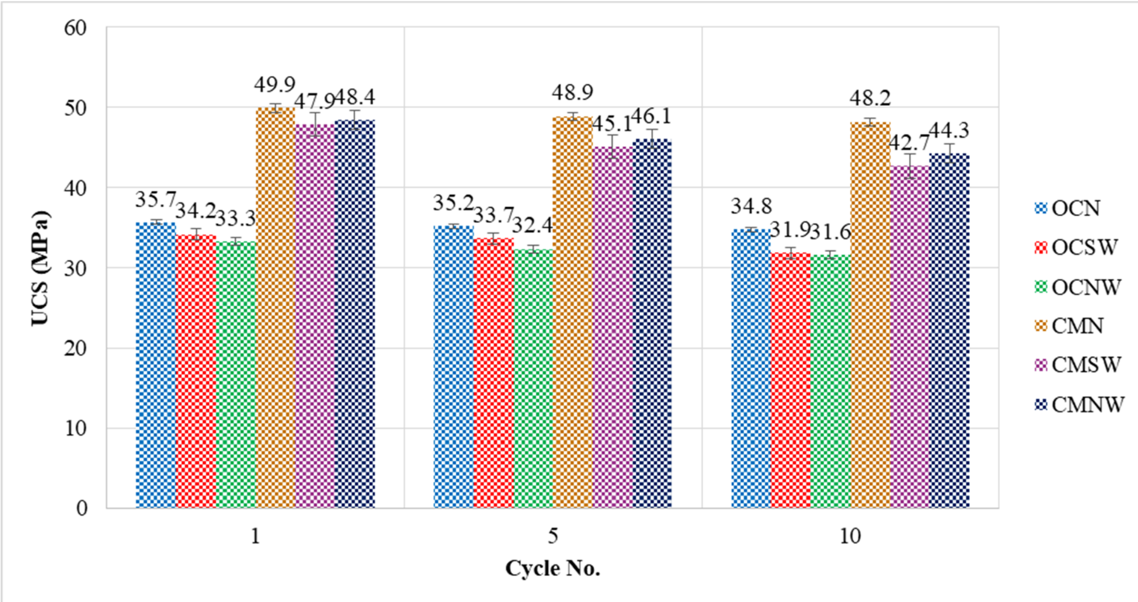

Figure 7. Effect of number of wetting-drying cycles on uniaxial compressive strength.

According to Figure 3, the variation of effective porosity depends on the pH of the environment. The effective porosity in the environment with pH = 3 is more than in the environment with pH = 7. Effective porosity of ordinary concrete samples and concrete containing micro-silica with increasing the number of wetting–drying cycles compared to the initial state increases (the state where the specimens have not experienced any wetting-drying cycles (cycle No. 0)). By increasing the number of wetting-drying cycles, effective porosity increases because acid and concrete have more time to react. According to Figure 4, the longitudinal wave velocity decreases with increasing pH and the number of cycles. The decrease in the velocity of the longitudinal wave is due to the increase in porosity and the creation of pores in the specimens. By increasing the number of pores in the sample, the P-wave in the air (pores) becomes more damped and reaches the receiver later. According to Figure 5, it is evident that the amount of water absorption increases with the increase in pH and the number of wetting and drying cycles. This parameter is considered an essential parameter for concrete structures and their stabilities. Based on the studies of Behnood *et al.*, the increase of water absorption content parameter in concrete and its sudden changes can be due to changes in the acidity of fluids around the structure. A primary solution to ensure concrete safety is boosting materials such as nano-silica as part of concrete cement [32]. The uniaxial compressive strength and tensile strength of concrete decrease when exposed to acid and the number of wetting and drying cycles. The main reason for the reduction of strength can be due to the creation of soft compounds during the chemical reaction of the concrete matrix with nitric acid or sulfuric acid. Micro-silica use has improved the mechanical and physical properties of concrete when exposed to sulfuric acid and nitric acid. The results from the effective porosity, velocity of P-waves, water absorption, BTS, and UCS tests show that the corrosion rate of ordinary concrete and concrete containing micro-silica is higher in the environment containing sulfuric than in the environment containing nitric acid. By increasing the number of cycles, the corrosion rate of all concrete specimens in the environment containing sulfuric acid increases, while the rate of concrete corrosion in the environment containing nitric acid after five cycles of wetting-drying does not change (Figure 8). This result shows that the effect of nitric acid is more significant in short-term exposure to concrete, as Barbhuiya and Kumala [15] found out in their research work.

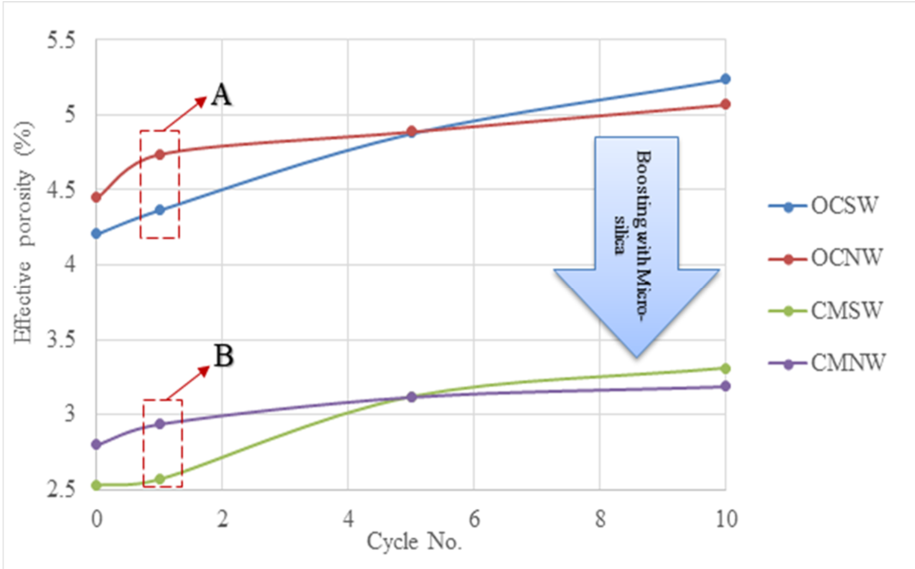

Figure 8. Comparison of the effect of the number of wetting-drying cycles on the effective porosity of the all-concrete specimens in the environment containing nitric acid and sulfuric acid.

In Figure 8, part A corresponds to standard concrete, and B corresponds to micro-silica concretes. After going through one wetting-drying cycle of ordinary concrete in the environment containing sulfuric acid and nitric acid, the effective porosity has increased by 3.8% and 6.52%, respectively, compared to the initial state (zero wetting-drying cycles in part A). After going through one wetting-drying cycle of concrete containing micro-silica in the environment containing sulfuric acid and nitric acid, the effective porosity has increased by 14% and 5%, respectively, compared to the initial state (zero wetting-drying cycles in part B). In acidic conditions, the tendency of the Chemical reaction of nitric acid with calcium hydroxide is more significant than with sulfuric acid, and with time, its reactivity decreases. For this reason, nitric acid is more effective than sulfuric acid in the first wetting-drying. When the amount of active silica in concrete increases, calcium hydroxide turns into calcium silicate, less reactive than nitric acid [33, 34].

6. Conclusions

This laboratory study shows that:

- Replacing 10% of micro-silica as a part of concrete cement increases the physical and mechanical properties of concrete compared to concrete without micro-silica. By replacing 10% of micro-silica as part of concrete cement in CMN concrete compared to OCN concrete (for number of wetting-drying cycle of zero), the effective porosity and water absorption decreased by 49.21% and 31.79%, respectively. The uniaxial compressive strength, Brazilian tensile strength, and the longitudinal waves velocity increased by 27.51%, 18.45%, and 1.6%, respectively.
- Sulfuric acid is more effective to make concrete destroying than nitric acid.
- By increasing number of wetting-drying cycles in environment of nitric acid, the corrosion rate of concrete decreases over time, which is due to the decrease in the speed of the chemical reaction of nitric acid with the concrete matrix.

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اثر آب اسیدی بر روی خواص فیزیکی- مکانیکی بتن حاوی میکروسیلیس

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چکیده:

این مطالعه به بررسی اثر افزودن میکروسیلیس به عنوان پوزولان و جایگزینی بخشی از سیمان بتن در هنگام قرار گرفتن بتن در محیط اسیدی می‌پردازد. دو نوع بتن معمولی و بتن حاوی میکروسیلیس ساخته شده‌است. نمونه‌ها تحت تعداد سیکل‌های صفر، 1، 5 و 10 به مدت دو ساعت در دو نوع آب اسیدی حاوی اسید سولفوریک و نیتریک با pH=3 و آب معمولی با pH=7 قرار گرفتند. خواص مکانیکی شامل مقاومت کششی برزیلی، مقاومت فشاری تک محوری و خواص فیزیکی شامل تخلخل موثر، میزان جذب آب و سرعت امواج طولی نمونه‌ها پس از تکمیل تعداد سیکل ذکر شده تعیین شده است. نتایج نشان می‌دهد که با کاهش pH و افزایش تعداد سیکل‌ها، تخلخل موثر و میزان جذب آب افزایش یافته و همچنین سرعت امواج طولی، مقاومت کششی برزیلی و مقاومت فشاری تک محوری بتن کاهش یافته‌است. جایگزینی 10 درصد میکروسیلیس به عنوان بخشی از سیمان بتن، دوام بتن را در شرایط خورنده حاوی اسید سولفوریک و نیتریک بیشتر از بتن معمولی افزایش داده است.

کلمات کلیدی: نیتریک و سولفوریک اسید، شرایط خورنده، خصوصیات فیزیکی- مکانیکی، بتن، میکروسیلیس.