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Fresh Properties and Corrosion Resistance of SCC Containing Nano-SiO_2

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Abstract- The Self-Compacting Concrete (SCC), as a new kind of high-performance concrete with growing demand in the construction industry, is vulnerable to corrosion like other concretes. So, the main focus of this study is the role of Nano-Silica (NS) on durability against the chloride electrochemical and sulfuric acid chemical attacks. To do so, an experimental program was conducted to investigate the effect of colloidal NS on the chloride and sulfuric acid corrosion beside the rheological properties of the fresh SCC specimens.

The SCC workability demand was fulfilled by Superplasticizer and Air-Entraining Agent (AEA) in order of 2% and 0.2% by weight of cement. The cement and water-to-cementitious materials ratio were respectively taken as 450 Kg/m^3 and 0.5. The NS replacement was: 0%, 2.5%, 5% and 7.5% by weight.

The results indicated that the highest and lowest LS occurred, respectively for 0% and 2.5% Ns after 6 weeks in sulfuric acid solution. In NaCl environment, no steel corrosion found after 14 and 28 days. But at 90 days, the probability of corrosion by half-cell test in the mixture with 7.5% NS was the least.

Keywords- *Fresh Properties, SCC, Corrosion, Nano-Silica, Compressive Strength*

I. INTRODUCTION

In a general definition, corrosion is defined as deterioration and degradation or change in characteristics and properties of materials due to their reaction with the surrounding environment [1]. Corrosion costs are more than 3% of Gross Domestic Product in the United States [2], also in the north-western of Europe (Belgium), biogenic sulfuric acid corrosion allocated approximately 10% of total expenditures of the sewerage treatment systems to itself [3]. The destructive effects of corrosion can lead to repair, rehabilitation, and early deterioration of the concrete structures.

Sulfuric acid is the sole inorganic acid that can be produced naturally by the oxidative weathering of pyrite and marcasite. If adequate amounts of oxygen and moisture become available, pyrite can be oxidized to ferrous sulfate and sulfuric acid by the following reactions [4]:

$$2FeS2 + 7O2 + 2H2O \rightarrow 2FeSO4 + 2H2SO4 \tag{1}$$

$$2FeSO4 + O2 + 2H2SO4 \rightarrow 2Fe2\neg\ (SO4)3 + 2H2O \tag{2}$$

$$7Fe2\ (SO4)3 + FeS2 + 8H2O \rightarrow 15FeSO4 + 8H2SO4 \tag{3}$$

In the wake of sulfuric acid presence and the formation of gypsum through the reaction process, surface cracking and spalling of concrete are expected [5]. If the gypsum is accumulated on the surface, it may fill the pores in concrete and subsequently may retard the sulfuric acid corrosion process [6]. This corrosion products and its reactions with aluminate components of cement constitute the ettringite as following [7]:

$$Ca\ (OH)2 + H2SO4 \rightarrow CaSO4.2H2O \tag{4}$$

$$3CaO.Al2O3.12H2O + 3(CaSO4.2H2O) + 14H2O \rightarrow 3CaO.Al2O3.3CaSO4.32H2O \tag{5}$$

$$CaSiO2.2H2O + H2SO4 \rightarrow CaSO4 + Si\ (OH)4 + H2O \tag{6}$$

On the other hand, corrosion of steel reinforcement in concrete imposes heavy costs on the societies all around the world that its main destructive cause is due to the 6 to 10 times volume increase of the corrosion products [8]. This matter highlights the importance of this research. Thus, the colloidal NS is utilized to study for overcoming the possible corrosion damages of the concretes.

SCC has the considerable plasticity, passing ability, flowability, and segregation resistance, and flow into congested reinforcements and complicated formworks [9]. In recent years there has been growing interest in the use of nanoparticles in concrete [10-16]. Despite this interest, no one to my knowledge has investigated the effects of colloidal NS on the corrosion of SCC exposed to sulfuric acid and chloride environments.

Tamimi [17] subjected high-performance concrete mixtures with fly ash replacement of 20, 40, 60 and 80%, to sulfuric acid and HCL. The author was aimed to evaluate the optimum content of supplementary materials for resistance to acidic chemical attack. The superior resistance to acid aggression was obtained from a ternary mixture containing 10% silica fume and 60% fly ash.

In another study [18], the influence of different chloride-inhibiting systems was examined on a 5-year-old concrete bridge. It was concluded that the best performance was related to inorganic admixture.

Moon and Shin [19] performed half-cell potential test on the ternary anti-washout underwater concrete after 14 cycles (98 days) of freezing and thawing. The test results revealed that the corrosion probability was less than the ASTM C876 critical limit.

There are different cases where concrete structures are affected by sulfuric acid exposure, e.g., structural elements in a chemical plant and concrete pipes in sewerage systems. There are three approaches for determining the behavior of concrete subjected to sulfuric acid attack: chemical, microbiological and in-situ [7]. In this paper, we implemented an immersion test method for assessing the durability of concrete in 3% sulfuric acid solution.

On the other hand, it is known that the corrosion of steel reinforcement is the most important type of corrosion in the concrete structures [20]. A number of researches have been conducted investigating the corrosion costs of interstate [8] and all U.S. highway bridges [2], the maintenance and repair costs of bridges [21], and saline corrosion of concrete bridges and highways in the UK [22].

II. MATERIALS AND EXPERIMENTS

Type I normal Portland cement (ASTM C150) and colloidal NS were used in this research. Their chemical compositions are also given in Table 1. A Poly Carboxylate Ether (PCE) type superplasticizer and an Air-Entraining Agent (AEA) were used in all the concrete mixtures. Gravel with a maximum nominal size of 19 mm was employed as the coarse aggregate, and local natural sand was employed as the fine aggregate. The coarse aggregate had an SSD specific gravity of 2.68 with water absorption of 0.5%. The fine aggregate had an SSD specific gravity of 2.37, and water absorption of 0.8%.

Four concrete mixtures were prepared in this study to evaluate the effect of NS replacement of cement on the durability and fresh properties of SCCs. The cement amount and water-to-cementitious materials ratio were taken 450 Kg/m3 and 0.5, respectively. The proportions of concrete mixtures are shown in Table 2. All tests were performed on three specimens, and the reported results are based on the average of the three readings.

Fresh SCC tests including the slump flow, J-ring, V-funnel, L-box, and U-box tests were carried out to assess the filling ability, passing ability, flowability and segregation resistance of SCC mixtures according to EFNARC [23] standards. For more details about the SCC tests, interested readers are referred to [23].

Compressive strength is done in compliance with ASTM C39 on cylindrical specimen (Φ 150 × 300 mm) after 14, 28 and 90 days of curing in water.

Loss of Strength (LS) and Loss of Weight (LW) were measured after 14 days of curing, and concrete samples were then immersed in 3% sulfuric acid solution for 2, 4 and 6 weeks. After each period of exposure, surfaces of samples were rinsed. The LS and the LW were calculated by following equations:

$$WL(\%)=\frac{W_1-W_2}{W_1}\times 100 \tag{7}$$

$$SL(\%)=\frac{f_{c1}-f_{c2}}{f_{c1}}\times 100 \tag{8}$$

Where W_1 and W_2 are the weight of samples (gr) before and after immersion in 3% sulfuric acid solution, respectively. Also, f_{c1} is the compressive strength of samples cured in tap water and f_{c2} is the compressive strength of similar samples exposed to 3% sulfuric acid solution.

TABLE I. CHEMICAL COMPOSITION OF CEMENT AND NANO-SILICA

Compounds	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O
Nano-Silica	97.6	0.06	-	-	-	-	-	-
Cement	20.1	5.04	3.08	64.3	1.13	2.09	0.3	0.38

TABLE II. PROPORTION OF MIXTURES

Mixture	Cement (Kg/m^3)	w/c	Water (Kg/m^3)	S^1 (Kg/m^3)	G^2 (Kg/m^3)	SP^3(%)	AEA^4(%)	NS^5(%)
0%NS	450	0.5	225	960	640	2	0.2	0%
2.5%NS	450	0.5	225	960	640	2	0.2	2.5
5%NS	450	0.5	225	960	640	2	0.2	5
7.5%NS	450	0.5	225	960	640	2	0.2	7.5

1. S: Sand 2. G: Gravel 3. SP: Superplasticizer 4. AEA: Air Entraining Agent 5. NS: Nano-Silica

In the case of the NaCl corrosion, it should be noted that compressive strength, electrical resistivity, and water absorption tests were performed on specimens when corrosion probability was more than 90%, according to half-cell potential test. Since most of the corrosion occurs in the tidal zone of marine environments [24], this condition simulated in the laboratory. Two-third of the height of specimens was immersed in 10% NaCl solution, and the rest of their height was subjected to ambient conditions. Moreover, circulation of the solution was provided by means of an aquarium pump. Meanwhile, before embedding in the SCC samples, the steel bars abraded with No. 600 grit emery paper and cleaned with acetone. Finally, control mixture (without NS replacement) was used for determination of the pozzolanic activity of colloidal NS.

Half-cell potential is used to measure corrosion probability that can be determined in terms of the potential difference between the steel rebar and the reference electrode according to ASTM C876 [25]. In Table 3, various range of corrosion probability with respect to Cu-$CuSO_4$ electrode, as specified by the ASTM C876 [26], are shown:

TABLE III. CORROSION PROBABILITY WITH RESPECT TO CU-$CuSO_4$ (ASTM C876)

Corrosion Probability	(VS Cu/CuSO4) Corrosion Potential
> 95%	< -0.35
Approximately 50%	-0.2 to -0.35
< 5%	> -0.2

Electrical resistivity test is established on this principle that the chloride ions can penetrate into the concrete and can reduce its electrical resistivity. The higher the chloride ion penetration into concrete, the lower its electrical resistivity. The electrical resistivity of specimens was measured by using a four-electrode device. The test procedure and the recommended limitations have been described in the literature [27-28].

The water absorption test was performed after 90 days of exposure to 10% NaCl solution. The test procedure is explained in detail elsewhere [29]. The water absorption was calculated by the following equation:

$$a = \frac{m_i - m_0}{m_0} * 100 \tag{9}$$

Where m_i and m_0 are the weight of samples (gr) in wet and dry conditions.

The compressive strength of specimens, according to BS 1881: part 116 [30], were determined after 90 days of exposure to 10% NaCl solution.

III. DISCUSSION

There is a general agreement on the gypsum formation caused by the sulfuric acid attack on concrete, but its consequences are rather controversial. Some researchers found that the formed gypsum retarded while some others argued that it accelerated the development of corrosion [31].

Slump Flow: As indicated in Fig. 1, mixture with 5% replacement of cement by NS has the highest slump flow diameter (68.5 cm). This diameter is situated in the class 2 SCC of EFNARC [23] classification. It means that this mixture is suitable for application in various structural concrete members. The mixtures with 2.5% and 7.5% of NS had high viscosity and were situated in the class 1 SCC of EFNARC classification. Nonetheless, no bleeding and no segregation were observed in any of the mixtures.

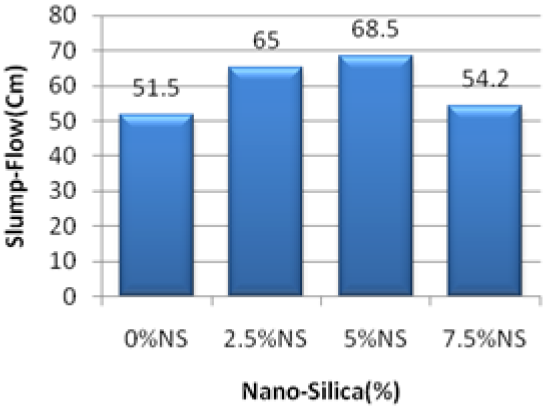

Figure 1. Slump-Flow vs. Nano-Silica (%)

J-ring: The J-ring test determines the passing ability of SCC. The higher the difference in height of the mixture (inside and outside of J-ring), the lower is the passing ability of the SCC. Both the 2.5% and 5% mixtures met the specified requirements of EFNARC [23]. In the case of SCC without NS (0% NS), the little blockage was observed (Fig. 2).

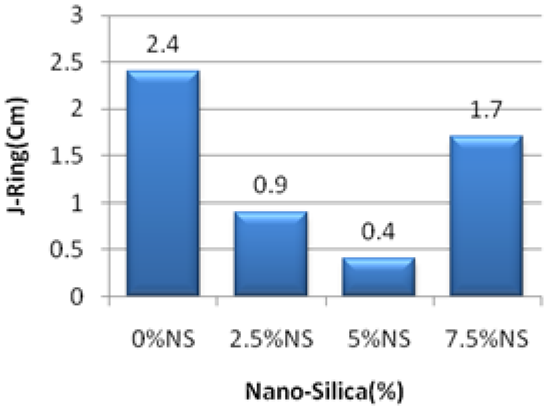

Figure 2. J-Ring vs. Nano-Silica (%)

V-funnel: Filling capability of the SCC can be measured by this test method. Not only the discharge time but also the uniformity and quality of the SCC can be assessed by means of V-funnel test [23]. From the results in Fig. 3, it can be seen that at 5% NS, an optimum content of NS is obtained. Fig. 3 also shows clearly that the highest flow time (13.4 s) belongs to 7.5% NS specimen.

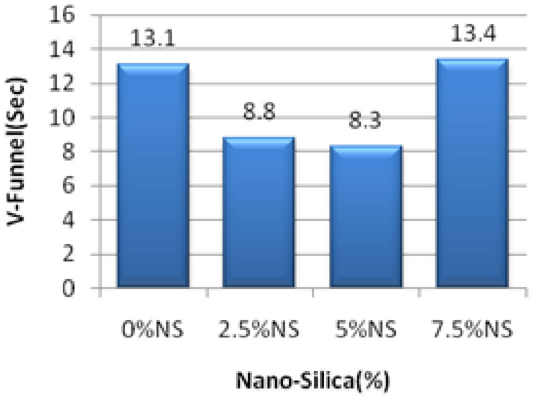

Figure 3. V-Funnel vs. Nano-Silica (%)

L-box: This serves to evaluate the filling capacity of SCC. AS per EFNARC [23] assessment criteria, the h_2/h_1 ratio should be between 0.8 and 1. Among all the mixtures, the one containing 5% NS satisfied the EFNARC [23] requirements. It seems that the lower bound of the EFNARC specifications for L-box can be declined (Fig. 4).

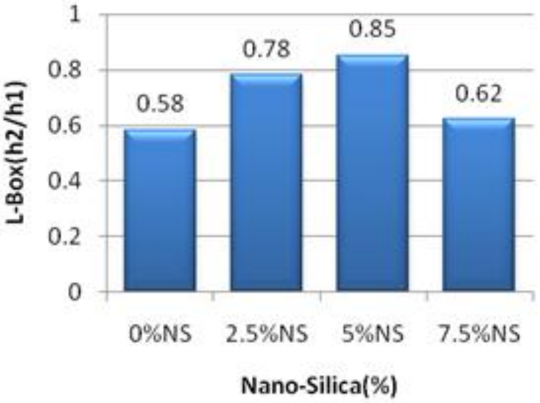

Figure 4. L-Box ratio vs. Nano-Silica (%)

U-box: Filling capacity and passing ability of the SCC can be measured by U-box apparatus. In comparison with the other SCC tests, the unique feature of this test is that the SCC must flow upwards. In accordance with EFNARC [23] recommendation, the maximum difference in height of SCC, between two sides of apparatus, must be less than 3 cm. Fig. 5 represents the results of the U-box test versus NS content. As shown, the filling height of 5% NS mixture is six times lower than that of the control mixture.

Considering the data of SCC tests, the mixture including 5% NS has acceptable self-compactibility. The reason for this behavior is that replacement up to 5% of NS improved the self-compactibility due to filling the voids of aggregates and lubricating effect. Whereas in specimens with 7.5% NS, the self-compactibility decreased because of NS great nucleating Effect which in turn intensifies NS accumulation in the mixes [11-14, 16].

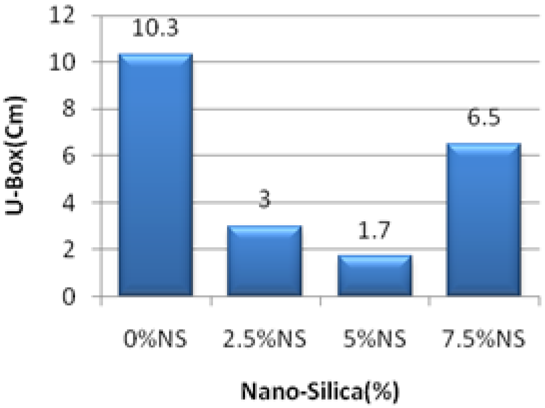

Figure 5. U-Box vs. Nano-Silica (%)

Compressive Strength: The effects of NS content and curing time on the compressive strength of specimens are summarized in Table. 4. It is well known that increasing curing time of the specimens increases the compressive strength. As shown in Table. 4, compressive strength development of the specimens comprising NS is greater than that of other pozzolans, such as silica fume, fly ash, blast furnace slag, and rice husk ash. This can be attributed to the high specific surface area of the NS [10, 13].

TABLE IV. COMPRESSIVE STRENGTH OF SPECIMENS (MPA)

Mixture	14(day)	28(day)	90(day)
0%NS	27.2	29.4	32.03
2.5% NS	23.5	28.84	32.77
5% NS	25.19	30	33.15
7.5% NS	24.51	29.18	32.9

The relationship between the strength loss (%) and the exposure for different dosages of NS are graphically illustrated in Fig. 6. Specimens were washed with tap water; their surfaces were cleaned with a cloth and kept at 20 °C for 24 h before testing. Fig. 6 shows that the largest strength loss belongs to the control mixture after all periods of exposure. The LS for the control mixtures ranged from 20.7% to 29.8%. The LS increases with elapsed time, except for the mixture containing 7.5% NS at 4 weeks of exposure. The fact that the strength loss of mixes with 2.5% NS replacement is smaller than others can be described by the lower PH of the NS (PH=10) than concrete. Further study is required to identify how NS affects the sulfuric acid resistance of SCC.

Loss of Weight: Prior to measuring the initial and final weights, samples were kept at a temperature of 20 °C for 24 h. The initial weight of samples was measured before immersion in 3% sulfuric acid solution. The LW of specimens with respect to the corresponding exposure period is given in Fig. 7. The weight of all mixtures, apart from the control mixture, is enhanced after 2 weeks of immersion. After 4 weeks, the 7.5% NS specimen still continued to gain weight (0.54%) in 3% sulfuric acid solution. This increase in weight stems from the formation of gypsum on the concrete surface [32, 33]. The reaction products were milky white in color. It is quite clear that there is no correlation between the strength loss and the weight loss.

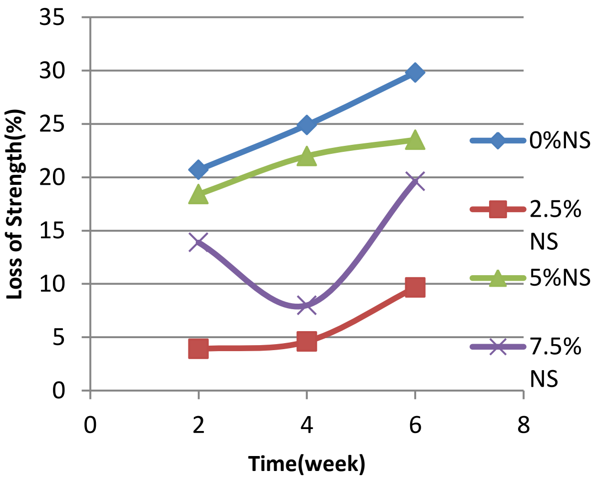

Figure 6. Loss of strength vs. time (week)

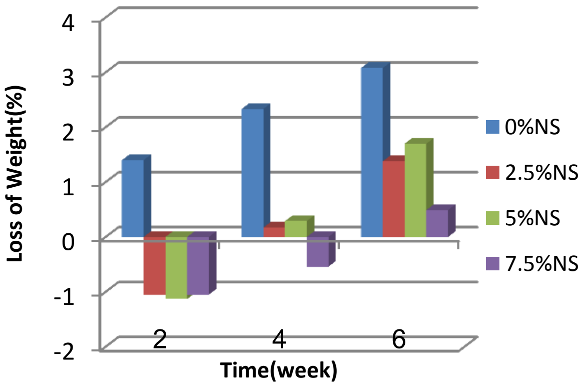

Figure 7. Loss of weight vs. time (week)

Half-cell potential: The influences of time of exposure of specimens with different NS substitution in 10% NaCl environment on the half-cell potential readings are displayed in Fig. 8. The results indicate that corrosion probability of the samples decreased with increasing dosage of the NS. At 90 days of exposure, the potential difference of mixtures containing 0%, 2.5% and 5% NS were -540 mV, -421 mV and -408 mV, respectively. These measurements exceeded the -350 mV threshold value recommended by ASTM C876. The difference in the half-cell potential readings of the mixtures with NS as compared to that of the control mixture (0% NS) is because of the great pozzolanic activity of colloidal nano-silica. This is consistent with the findings of other researchers [10-11, 13, 15].

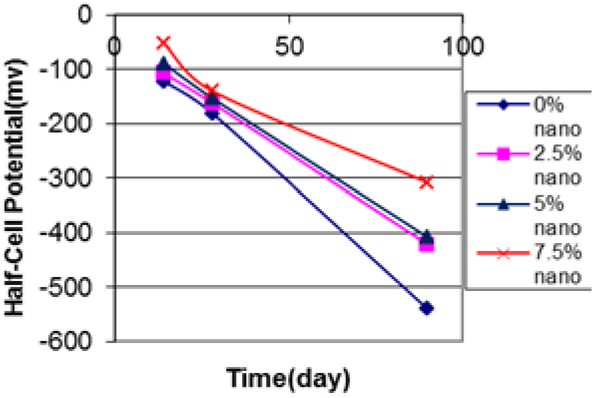

Figure 8. Half-cell potential vs. time (day)

As shown in Fig. 9, the electrical resistivity of the specimens with 0%, 2.5% and 5% of NS is below 120 Ω.m, while in the case of the specimen with 7.5% of NS, the reading is more than 120 Ω.m. This means, according to AASHTO TP-95 that the high chloride ion penetration does not occur in the specimen with 7.5% of NS. This behavior also confirms the appropriate pozzolanic activity of the NS, such as those mentioned in the literature [10-11, 13, 15]. Another interpretation that can be drawn from Fig. 9 is that the electrical resistivity of the samples was improved by the increment of NS content.

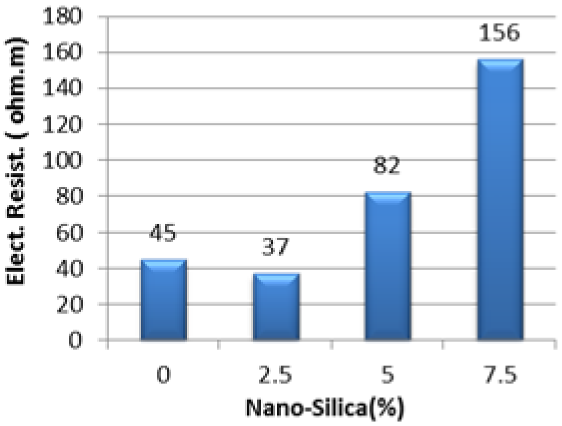

Figure 9. Electrical resistivity vs. Nano-Silica (%)

Compressive strength: The compressive strengths of specimens subjected to 10% NaCl solution for 90 days are plotted in Fig. 10. In comparison with the control mixture (0% NS), the replacement of 2.5%, 5% and 7.5% of NS increased the compressive strength of specimens by about 51%, 53%, and 57%, respectively. It can be clearly seen that the NS, both as a pozzolan and as filler, plays a major role in enhancing the chloride ion resistance of concretes [10-15].

Water absorption: The water absorption test can be used to evaluate permeability of concrete comparatively. As shown in Fig. 11, the replacement of 2.5% of cement with NS had no significant effect on the water absorption ratio, whereas, in mixtures with 5% and 7.5% of NS the water absorption was decreased. This reduction could be attributed to the filler effect of NS [10-11, 13]. The results of the water absorption of the specimens support the findings obtained from the electrical resistivity test. The less the water absorption ratio of the specimen, the more is the electrical resistivity reading.

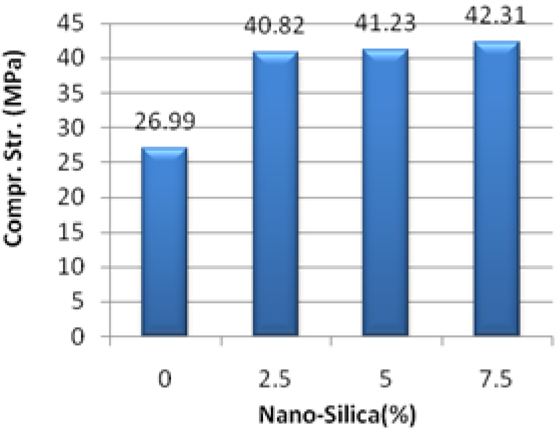

Figure 10. Compressive strength vs. Nano-Silica (%)

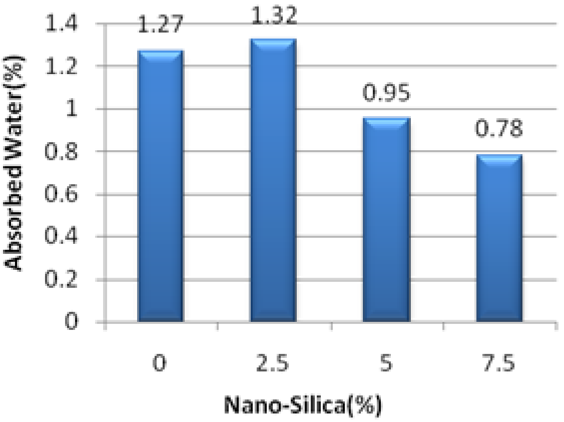

Figure 11. Water absorption vs. Nano-Silica (%)

IV. CONCLUSION

In this paper, firstly, the causes and mechanisms of both chloride and sulfuric acid corrosion of concrete were demonstrated. Secondly, the self-compactibility of four mixtures containing colloidal nano-silica was investigated. Finally, the durability of mixtures subjected to 10 % NaCl solution and 3% sulfuric acid attack was determined. Based on the findings of this study, the following conclusions were drawn:

- The results of the slump flow and the V-funnel tests showed that NS replacement had a positive effect on the characteristics of SCC mixtures, especially at 2.5% and 5% of replacement.

- With regard to J-ring and U-box experiments, it is shown that mixtures containing 2.5% and 5% of NS have the good passing ability and filling capacity.

- In the case of the L-box test, only the mixture with 5% of NS satisfied the EFNARC requirement. It seems possible to reduce the blocking ratio from 0.8 to a lower ratio in order to improve the passing ability for 2.5% NS.

- No obvious signs of bleeding and segregation were detected in mixtures by means of visual inspection.

- At early ages, the strength gaining of specimens including NS was similar to normal concrete, in contrast to other pozzolans. This could be due to the fact that the NS has great specific surface area.

- In the case of the compressive strength loss of specimens in 3% sulfuric acid solution, no important tendency to increase the compressive strength was measured.

- With regard to weight loss data, it should be mentioned that all of the mixtures had a weight development after 2 weeks (14 days) of exposure, except the control mixture. This phenomenon is caused by the production of gypsum on the surfaces of samples. The minimum weight loss was achieved for mixture with 7.5% of NS replacement. There was no obvious relationship between the strength loss and the weight loss.

- The corrosion probability of specimens decreased when the NS replacement level increased. Except the mixture containing 7.5% of NS, more than 90% of corrosion probability was observed in all mixtures, after 90 days of exposure to 10% NaCl solution.

- The electrical resistivity of the mixture with 2.5% of NS was lower and its water absorption was higher than that of the control mixture.

- As expected, the good correlation achieved between the electrical resistivity readings and water absorption values.

- Regarding the results of the compressive strength tests after 90 days of exposure to 10% NaCl solution, with the addition of 2.5-7.5% of NS an improvement in the range of 51-57% was achieved.

- It can be concluded that the NS has a significant effect on the chloride corrosion resistance of reinforcement in SCC.

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