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ASSESSMENT OF THE DURABILITY OF NANOSILICA / FLY ASH CEMENT PASTES EXPOSED TO NORMAL AND AGGRESSIVE CURING CONDITIONS

The aim of this work is to study the effect of fly ash (FA) and nanosilica (NS) on the durability of cement pastes under normal and aggressive curing condition. Different mixes were made with various amounts of NS and constant value of FA (10% by weight of cementitious materials). First group of samples were subjected to normal curing and tested after 3, 7, 28 days and after one year, while the second group of samples were subjected to sulphuric acid of concentration 0.2 N and tested after the same ages. The hydration process and durability of cement pastes were monitored using scanning electron microscope (SEM). The results of this study indicated that FA of percentage 10% and nanosilica of percentage 1% of cementitious materials improve the durability and microstructure of cement pastes against both normal and aggressive curing conditions.

Key words: fly ash, nanosilica , sulphuric acid, aggressive conditions, durability and microstructure.

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1. Introduction

Previous research works concerning the effect of NS on the durability of cement pastes, mortars and concretes are so limited, as most of the previous research summarized below deals mainly with the dispersion issue and the influence of NS on the properties of fresh and hardened states subjected to normal curing condition.

Hongjian D. et al [1] studied the durability performances of concrete with NS at dosages of 0.3% and 0.9% of cementitious materials exposed normal curing and found that due to the nano-filler effect and the pozzolanic reaction, the microstructure become more homogeneous and less porous, especially at the interfacial transition zone (ITZ), which led to reduced permeability and beneficial effects of nanosilica on durability.

Kong et al [2], studied the influence of NS agglomeration on microstructure and properties of the hardened cement based materials and found that the addition of NS up to 1% increased the resistance of mortar to chloride penetration.

Quercia et al [3] carried out a study on the durability of self-compacting concrete (SCC) incorporating NS in both colloidal and powder forms at dosage 3.8% and found that all the durability performances of SCC were enhanced due to addition of NS.

Abd El-Aleem S. et al [4] studied the durability of blended cements containing NS and found that the use of 4% NS produces a highly durable mixed cement.

Rudelf H., and Jana M. [5] studied the possibilities of Nano-Technology in Concrete, and found that concrete with FA has higher porosity at early ages and lower porosity after two years in compassion with concrete without FA, also they found that concrete with fly ash and nanosilica showed small sizes of pores even in early stages of hydration, pores 10–100 μm were significantly reduced in concrete with nanosilica , which enhances properties of mastic cement, makes it denser and less permeable for water and corrosives.

Lin et al [6] demonstrated the effect of nano-SiO_2 addition on permeability of eco-concrete, they found that the relative permeability and pores sizes decrease with nano Sio2 addition (1 and 2%).

Gengying Li [7], studied the properties of high volume FA concrete incorporating nano-SiO_2 and found that the addition of FA leads to higher porosity at short curing time, while nano-SiO_2 acting as an accelerating additive, leads to more compact structures even at short curing times.

Quercia G. et al [8] studied the application of nanosilica in concrete mixtures and mentioned that the microstructural analysis of concrete by different electronic microscope techniques (SEM, TEM, and others) revealed that the microstructure of NS concrete is more uniform and compact than that for normal concrete.

Ji [9], added 3.8% NS to the concrete mixture and observed a more uniform and compact microstructure in the nanosilica concrete and attributed this due to: NS can react with $Ca(OH)_2$ crystals, and reduce the size and amount of them, thus making the interfacial transition zone (ITZ) of aggregates and binding cement paste more denser.

Gaitero J.J. et al [10], found that the addition of 6% NS reduces the calcium leaching rate of cement pastes and therefore increasing their durability.

Verma A., et al [11], studied the influence of aggressive chemical environment on high volume FA concrete, and found that utilization of FA reduces the loss in compressive strength, and has been proved that FA is an effective material for making durable concrete in aggressive environmental conditions.

Salemi N., et al [12], studied the effect of nanoparticles on frost durability of concrete, they found that the contribution of nano-TiO_2 on the improvement of durability was more than the other nano particles which were nano-Fe_2O_3, nano-AL_2O_3 and nano-ZnO_2.

Celik Q. et al [13] carried out laboratory investigation of nano materials to improve the permeability and strength of concrete, and found that the nanosilica has the largest improvement in both compressive strength and permeability among the other nano materials used.

Amin A. A. et al [14], studied the durability of some Portland cement pastes in various chloride solutions and fund that the penetration of chloride into Portland pozzolanic cement is the lowest, followed by ordinary Portland cement, whereas sulfate resisting cement has the highest depth of penetration.

Shi L. et al [15], investigated the influence of sulphuric acid and sulfate on concrete cementitious system performance and found that the presence of mineral admixtures are beneficial to the system mechanical properties remain at later age (90-days) after exposure to acidic erosion.

To date, there was no comprehensive study relating the influence of different dosages of nanosilica /fly ash on the durability of cement pastes, mortars and concretes.

The main objective of our study will be to investigate the durability of cement pastes containing different dosages of nanosilica in addition to constant dosage of fly ash, and exposed to normal and aggressive curing conditions.

2. Experimental Work

In this work the performance of fly ash/nanosilica cement pastes under normal curing conditions was compared with that under aggressive curing conditions at different ages from 1-day up to one-year.

2.1. Materials

The cementitious materials used during this study were OPC, fly ash, and nanosilica .

2.1.1. Ordinary Portland cement (OPC)

Ordinary partland cement (OPC) was used of type (CEMI, 52.5N), obtained from Beni-Suef cement factory in Egypt and complies with ESS (4756-1-2006) [16], the chemical analysis of OPC and fly ash are shown in table 1.

2.1.2. Fly Ash (FA)

Fly ash used in this study was imported from India through Goise Company in Egypt. The blane fineness of it is 3200 cm^2/gm and its specific gravity is 2.25 as shown in table 2, it complies with ASTM C618 class F [17].

2.1.3. Nanosilica (NS)

Nanosilica used in this investigation is synthetic product with spherical particles in the range of (24–35) nm, imported from Sigma – Aldrich (Germany), its physical properties are given in table 3, its chemical analysis showed that it consists mainly of pure silica, 97%, x-ray diffraction

(XRD) was carried out in HBRC at Raw Building Materials Institute and its pattern is shown in fig 1, from which it can be seen that the used NS is highly amorphous materials and has low crystallinity.

Transmission Electronic Microscopic (TEM) of nanosilica was carried out in National Research Center (NRC) at physics department, fig. 2 shows the morphology of NS, it can be seen that NS particles are highly agglomerated clusters with size (24–35) nm.

2.1.4. Superplasticizer (SP)

Polycarboxylic ether polymer based PCE sky, gelenium ACE 30 obtained from passive company in Egypt was used during this study.

2.2. Mix design and preparation of specimens

Standard cement paste was prepared according to ESS (2421-1993) [18] to determine the water – cement ratio, which was found in this study 26% for mixes M_1, M_2 without superplasticized, while was 23% for other mixes (with super plasticizer) as shown in table 4, from which it can be seen that mix M_1, and M_2 are control mixes, while mixes M_3, M_4, M_5, M_6, M_7 are main mixes with different values of NS (1% , 2% , 3% , 4% and 5% respectively).

2.2.1. Mix design

Cement pastes were prepared, mixed and tested in HBRC (Quality Control and Material Institute), seven cement paste mixes are designed as shown in table 4, thirty cube of size 20 x 20 x 20 mm were cast for each mix.

2.2.2. a) Wet Mixing Procedure

The cement and FA were mixed in the electric pan mixer for 30 sec., then 50% of mixing water was added and mixed for 30 sec. Ready – mixed liquid including superplasticizer and NS was added to the 50% remained water and sonicated in ultrasonication bath for 5 minutes at temperature 38°C for good dispersion of NS in the superplasticizer. The ready mixed and then the liquid was poured into the mixer slowly and mixing continued for additional 2 minutes until the mix becomes homogenous.

2.2.2.b) Casting and Curing

The cement paste was then cast into steal mould which contains 30 cube of dimensions 20 x 20 x 20 mm, and the surface of the paste was leveled in one direction and covered with plastic sheet and left in standard curing conditions (20°C and 90% R.H.) for the first 24 hours. All samples were demolded after 24 hours numbered and weighed, then 1st half of them was cured in potable water and the second half was cured in sulphuric acid of concentration 0.2 N (7.35 ml/lit), after 24 hours until the age of testing (3 days, 7 days, 28 days and one year), curing water and the sulphuric acid solutions were changed every month.

2.2.3. Testing Procedure

At the date of testing three samples of each group were extracted from curing conditions, weighed and measured their dimensions, then tested in compression testing machine and the average result is recorded.

The compressive strength loss of specimens due to immersion in acid solution was monitored at ages 3-day, 7-day, 28-day and one year.

The compressive strength loss is calculated according to the equation:

$$SL = (Sa - Sb) / Sa \times 100\% , \qquad (1)$$

where, Sa – average compressive strength of specimens cured in potable water as shown in table 5; Sb – average compressive strength of specimens after curing tests in acid solution for the required time as shown in table 6; SL – the compressive strength loss due to the exposure to acidic curing for the required time [19] as shown in table 7.

3. Analysis and Discussion of Test Results

3.1. Compressive Strength

3.1.1. Normal Curing

Fig. 3 show the development of compressive strength of cement pastes containing NS/FA at different ages exposed to normal curing con-

Table 1.

Chemical Analysis of FA and OPC, wt. %

Oxide content	OPC	FA
SiO_2	19.8	61.7
Al_2O_3	5.5	21.3
Fe_2O_3	3.4	7
CaO	63	4.5
MgO	1.2	1.99
Na_2O	0.46	–
K_2O	0.19	–
SO_3	3	0.35
Less on ignition	2.5	1.9

Table 2.

Physical characteristics of FA and OPC

Description	OPC	FA
Specific gravity	3.15	2.25
Specific surface area (cm^2/gm), blaine	3100	3200
D 50 µm	17	14

Table 3.

Properties of nanosilica

Description	Results
Diameter (nm)	24–35
Purity (%)	97
Surface area (m^2/g)	220
Density (g/cm^3)	0.55
Molecular	SiO_2
Molecular weight	60.08

Table 4.

Mix composition, mass % of cementutious materials

Mix. No.	OPC	FA	NS	w/p%	SP%
M_1	100	0	0	26	0
M_2	90	10	0	26	0
M_3	89	10	1	23	3
M_4	88	10	2	23	3
M_5	87	10	3	23	3
M_6	86	10	4	23	3
M_7	85	10	5	23	3

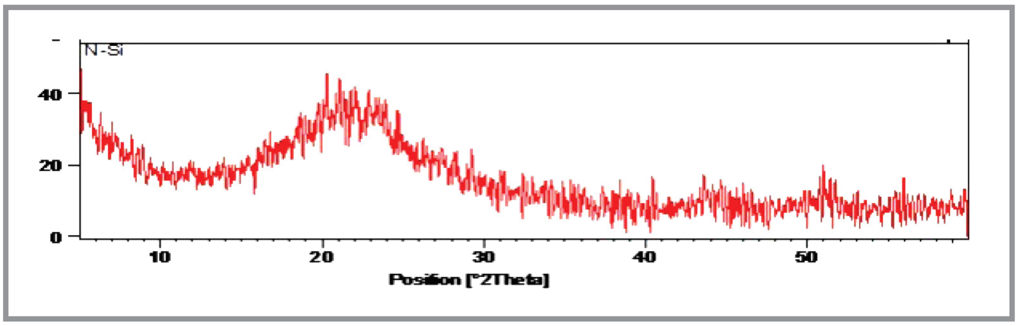

Fig. 1. **XRD analysis of Nanosilica**

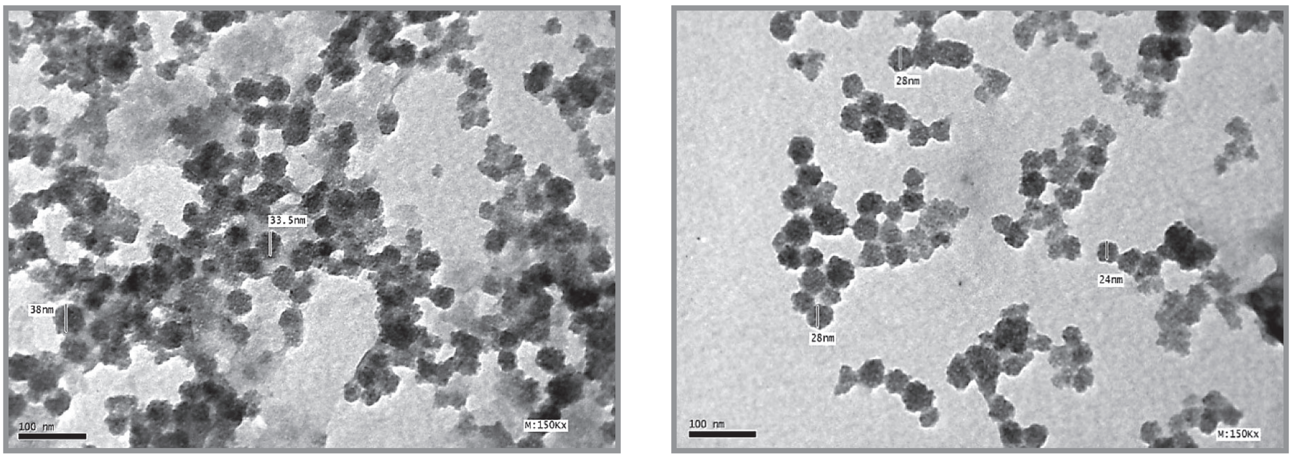

Fig. 2. **TEM micrograph of Nanosilica**

ditions. It can be seen that compressive strength of cement paste mix no. M_2 which contained 10% fly ash as replacement of cement was lower in early ages up to 28-days than mix M_1, while after one year approached it, this can be attributed to the pozzolanic effect of FA which appears at later ages.

The percent of FA during this study was chosen 10% as replacement of OPC, this is was optimum replacement followed in most research works investigated the influence of aggressive suphuric acid on concrete or cementing materials [15] & [19].

It can be seen also that nanosilica mix of percentage 1% enhanced compressive strength from 3-days up to one year in comparison with control mix (without nanosilica), the % of enhancement was 1.4% after 3-days, 5.7% after 7-days, 26.8% after 28-days, and 13.5% after one year.

This can be attributed to the effect of NS, which behaves not only as a filler to improve microstructure, but also an activator which promote pozzolanic reaction as well as acts as nucleating sites to form more accumulation and precipitation of calcium silicate, aluminate and alumine-silicate hydrates in open pores originally filled with water, leading to formation of homogeneous, dense and compact microstructure [20].

It can be seen also that when the amount of NS increased than 1% , the compressive strength decreased at all ages (from 3-days up to one year) in comparison with 1% NS mix and control mix (without nanosilica).

This is can be attributed to the common characteristic of NS and FA is that they both are pozzolanic materials, and both adsorb, react with, or consume calcium hydroxide (CH) that is generated from cement hydration. Considering the great difference of the reactivity of the two pozzolans (NS and FA), since the capability of NS for consuming CH is much greater than the capability of FA, which could be one of the features that govern the maximum dosage of NS in the mix [21].

Note: Many researches mentioned that 2% NS or more is the best replacement ratio of OPC, this only for:

1. Research works did not add FA to NS in the same mix.

2. Research works used dry mixing procedure without dispersion of NS in superplasticizer (i.e. without sanication).

3.1.2. Aggressive Curing

Fig. 4 show the development of compressive strength of cement pastes containing NS/FA at different ages exposed to aggressive curing conditions (0.2 N sulphuric acid). It can be seen that compressive strength of cement paste mix no. M_2 which contained 10% fly ash as replacement of cement was lower at early ages up to 28-days than mix no. M_1 without fly ash), while after one year the behaviour was reversed, this can be attributed due to pozzolanic effect of fly ash which appears at later ages. It can be seen also that 1% nanosilica enhanced compressive strength from 7-days up to 28-days although the samples were subjected to sulphuric acid of concentrations 0.2 N, this is in comparison with control mix without nanosilica (M_2), also it can be seen that when the amount of NS increased to 2% (Mix No. M4), the compressive strength decreased than 1% NS mixes.

It can be seen also that, when the amount of NS increased than 2%, the compressive strength decreased at all ages (from 3-days up to one year) in comparison with control mix without nanosilica, this can be attributed to more agglomeration of additional NS which negatively affected the cohesion of formed binding materials.

Fig. 5 shows the losses in compressive strength for different mixes exposed to aggressive curing (0.2 N sulphuric acid), it can be seen that in all mixes the pattern of losses taking place show a similar trend and the losses in compressive strength which increases with time exposure to aggressive conditions. Also, it can be seen that the percentage of losses for mix no. M_2 which contained 10% fly ash as replacement of cement was lower at all ages than mix no. 1 (without fly ash). It can be seen also that 1% NS mix no. M_3 showed minimum losses at any stage of exposure which means that the residual strength of NS/FA mixes was found to be more than that of plain concrete and the maximum residual strength of NS/FA mixes was found at mixes M_3 which contained NS 1% with amount of fly ash (10%).

Also, it was found that the rate of increase of losses of nanosilica mixes is less than that in the case of plain mix (without nanosilica) especially in the stage of exposure time after 28-days up to one year, which proves that the behaviour of NS/FA mixes is enhanced than the plain mixes when exposed to aggressive curing conditions for long time of exposure, this is can be attributed to: when sulphuric acid contacted plain cement paste mixes, it reacted with Portlandite in cementing paste forming gypsum along with

Table 5.

Compressive strength of Nanosilica / Fly Ash mixes at different ages in normal curing

Mix No.	Mix Type	3-Days	7-Days	28-Days	one - year
M1	Control FA- NS-	71.6	71.9	93.3	112.5
M2	FA10% , NS--	62.7	63	82.8	111.3
M3	FA10% , NS1%	63.6	66.6	105	126.4
M4	FA10% , NS2%	56.7	59.5	77.6	114.2
M5	FA10% , NS3%	43.8	46.6	63.6	86.0
M6	FA10% , NS4%	22.2	25.6	45.4	65.7
M7	FA10% , NS5%	21.6	23	25.7	57.7

Note: Units of compressive strength MPa.

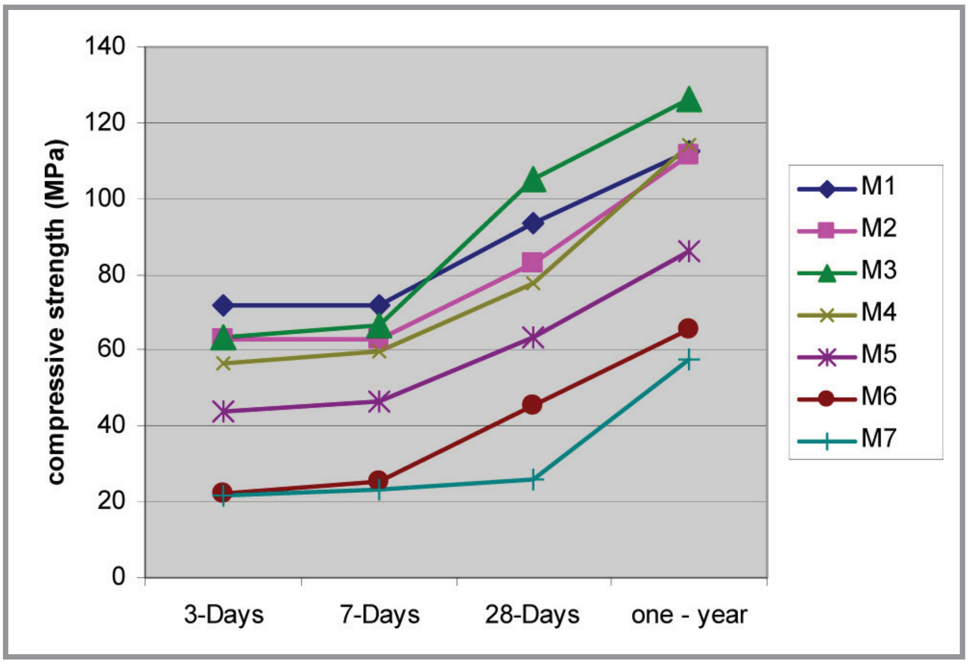

Fig. 3. **Compressive strength of cement pastes containing NS/FA at different ages exposed to normal curing conditions**

Calcium Silicate Hydrate (C.S.H) decomposed, which lied on the stability of plain cement paste, on the other hand, the high reactivity of NS particles produces more refined microstructure which decreases the accessibility of

Table 6.

Compressive strength of Nanosilica / Fly Ash mixes at different ages in normal curing

Mix No.	Mix Type	3-Days	7-Days	28-Days	one - year
M1	Control FA- NS-	67.3	67.7	78.6	49.1
M2	FA10% , NS--	60.0	60.3	69.8	55.2
M3	FA10% , NS1%	61	64.1	94.3	85.4
M4	FA10% , NS2%	54.3	56.9	79.6	76.3
M5	FA10% , NS3%	41.9	44.6	56.5	45.0
M6	FA10% , NS4%	21	24.5	40.4	23.3
M7	FA10% , NS5%	20.6	22	24	34.3

Note: Units of compressive strength MPa.

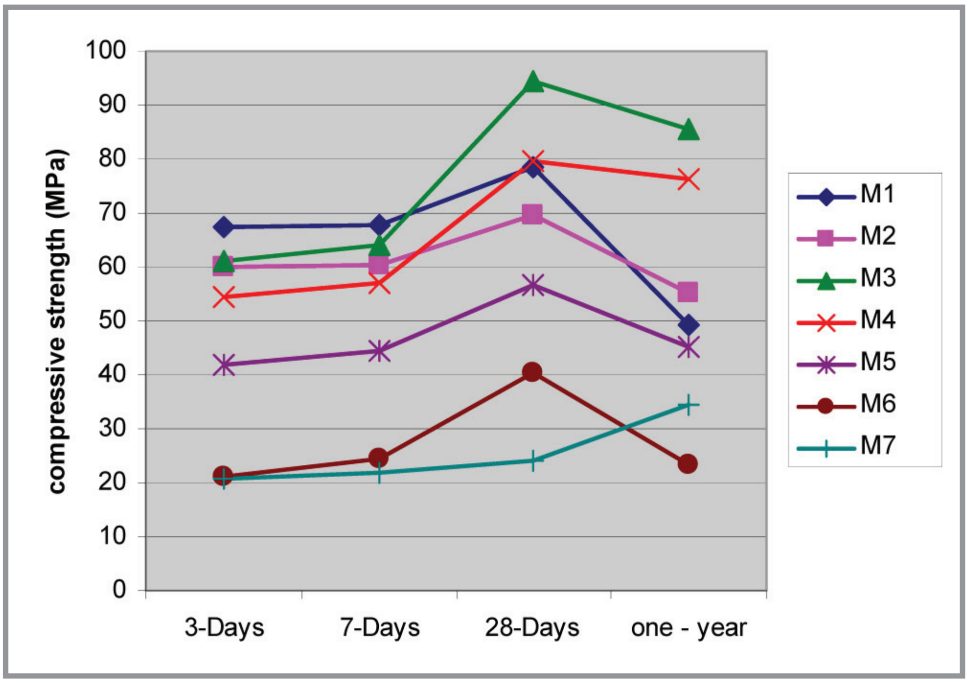

Fig. 4. **Compressive strength of cement pastes containing NS/FA at different ages exposed to aggressive curing condition (0.2 N sulphuric acid)**

sulphuric acid towards the more compact structure which improves the residual strength of NS/FA mixes[19].

Table 7.
Losses in compressive strength for different mixes exposed to aggressive curing (0.2 N sulphuric acid)

Mix No.	Mix Type	3-Days	7-Days	28-Days	one - year
M1	Control FA- NS-	6%	5.8	15.7	56%
M2	FA10%, NS--	4.3%	4.3	15.7	50.8%
M3	FA10%, NS1%	4%	3.7	10.5	32.4
M4	FA10%, NS2%	4.2%	4.4	–2.5	33.2
M5	FA10%, NS3%	4.3	4.3	11.2	47.6
M6	FA10%, NS4%	5.4	4.3	11	64.9
M7	FA10%, NS5%	4.6	4.5	6.6	40.5

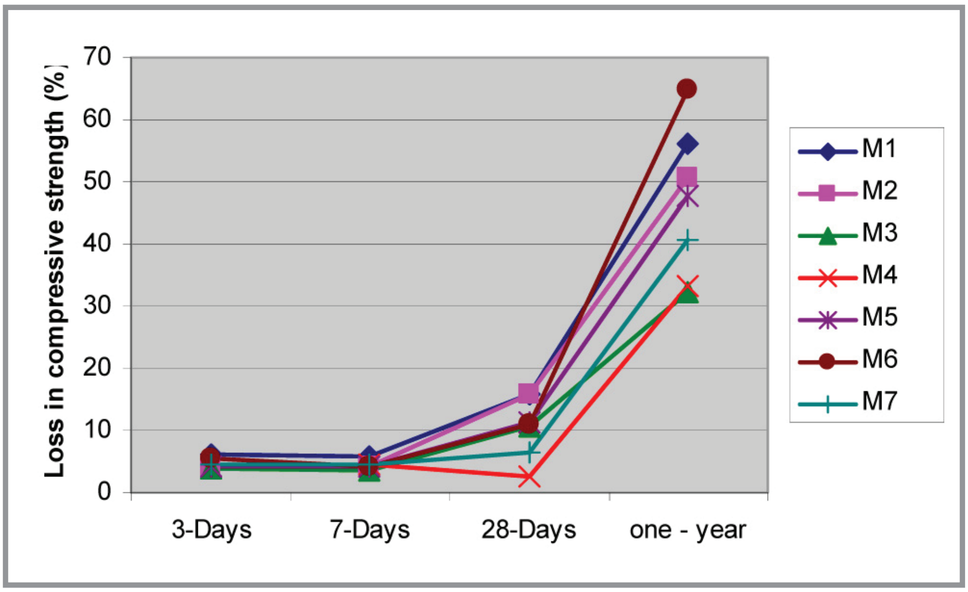

Fig. 5. **Variation in losses in compressive strength for different mixes exposed to aggressive curing (0.2 N sulphuric acid)**

3.2. Microstructure

Scanning electronic microscope (SEM) examination was carried out in the laboratories of Building Physics Institute in Housing and Building National Research Center Using ESEM(Environmental Scanning Electronic

Microscope) FEI inspect S, made in Holland). The specimens were taken directly from cement paste samples after testing in compressive strength, the magnification power was 1500.

3.2.1. Normal Curing

Fig. 6 shows the SEM micrograph of cement paste samples subjected to normal curing for one year with and without NS.

Fig. 6 A shows the SEM of control mix after one year (without NS) in normal curing, it can be seen that: The matrix is heterogeneity in addition to the presence of wide cracks within the matrix, this is accompanied by the spreading of free hydrated lime which liberate from the hydration reaction all over the surface; which is more susceptible to carbonation and increase porosity.

Fig. 6 B shows the SEM of 1% NS, from which it can be seen that: Addition of 1% nanosilica results in the formation of dense and compact microstructure with lesser amount of calcium hydroxide crystals, as the added

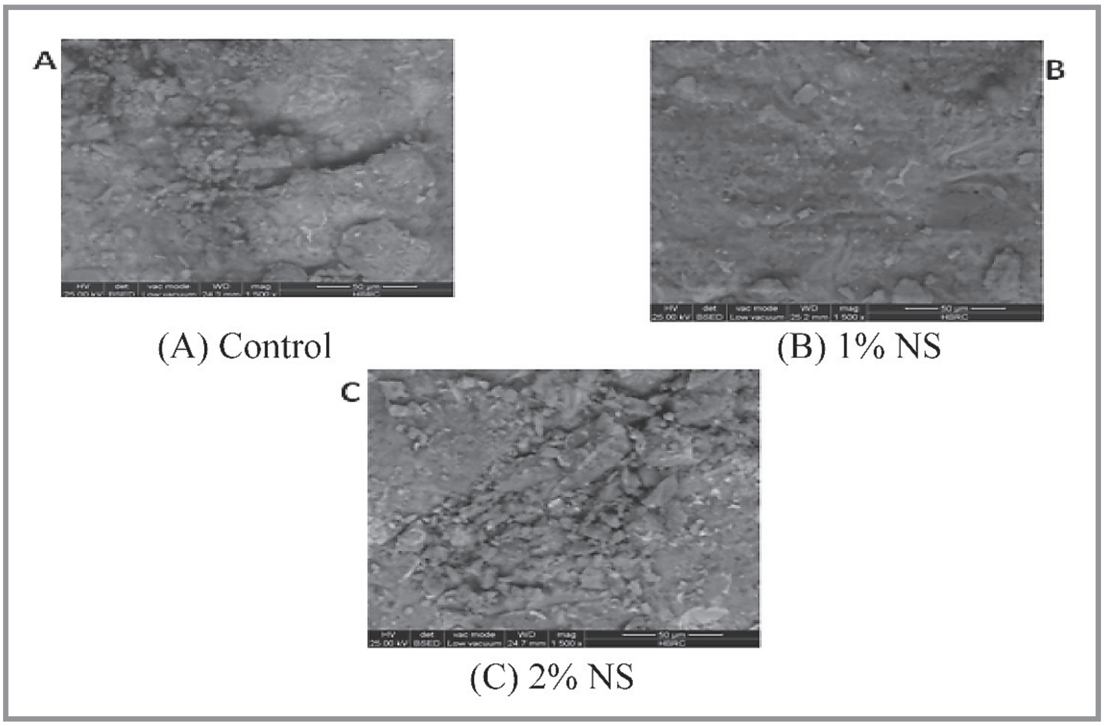

Fig. 6. **SEM micrographs of fracture surface of hardened cement pastes with and without NS cured for one year in normal curing conditions**

nanosilica digests the free lime forming C–S–H which in turn participates in additional strengthening and homogeneity of the microstructure.

Fig. 6 C Shows the SEM of 2% NS, from which it can be seen that: Increasing the nanosilica to 2% results in the agglomeration of the added nano materials which negatively affects the cohesion of the formed binding materials leading to the formation of high pore volume and heterogeneous structure.

3.2.2. Aggressive Curing

Fig. 7 shows the SEM micrograph of cement paste samples subjected to aggressive curing conditions (0.2 N sulphuric acid) for one year with and without NS.

Fig. 7 A shows the SEM micrograph of control mix (without NS) after one year in aggressive curing conditions, it can be seen that:

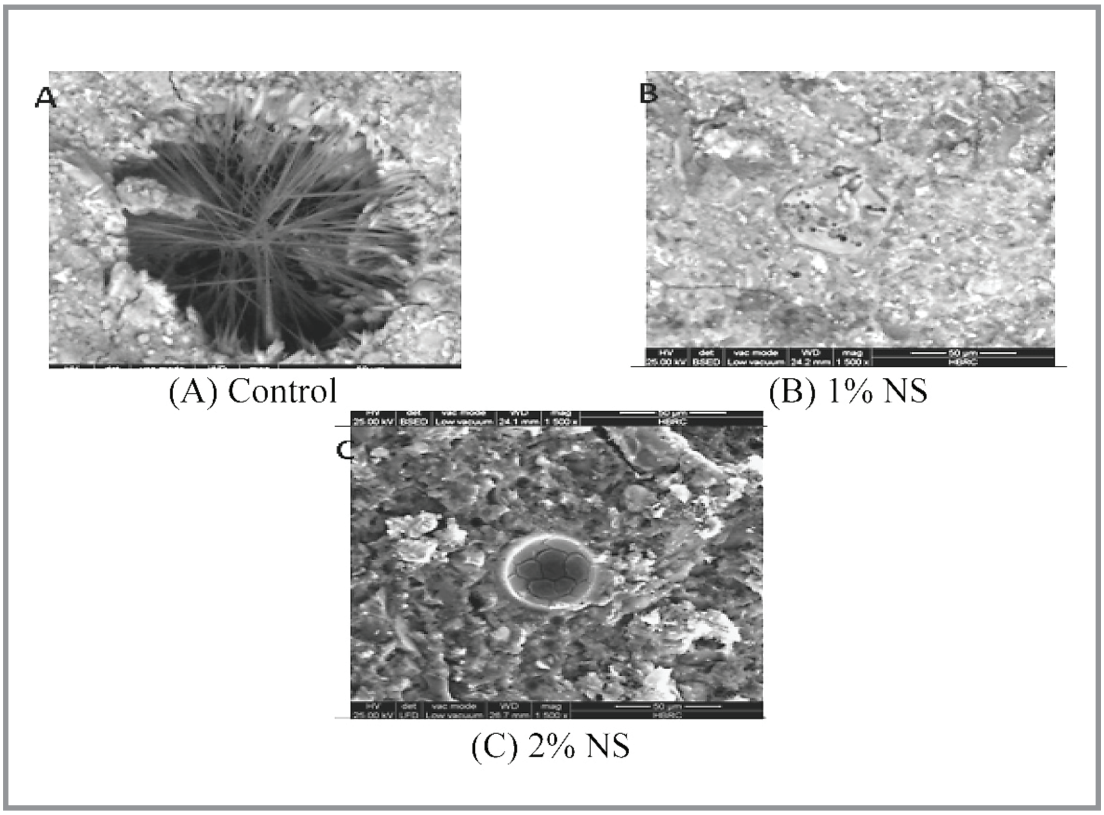

Fig. 7. **SEM micrographs of fracture surface of hardened cement pastes with and without NS cured for one year in aggressive curing conditions**

The spreading of ettringite needles appears within the wide pores which reflects the low stability of Portland cement against acid attack as the pH decreases sharply leading to destabilization of hydrating materials, also the free lime interacts with sulfuric acid forming gypsum and so ettringite with its expansive characteristics which comes clearly from the intensity of the speeded ettringite bundles and the expansive characteristics which increase the gap within the pores.

Fig. 7 B shows the SEM micrograph of 1% NS, after one year in aggressive curing conditions, from which it can be seen that:

The addition of 1% nanosilica results in better stability against acid attack as mostly all the free lime consumed by the added nanosilica and forms C–S–H with fibrous, dense and compact microstructure with lesser amount of calcium hydroxide crystals and better formation of cohesion structure.

Fig. 7 C shows the SEM micrograph of 2% NS after one year exposure in aggressive curing conditions, from which it can be seen that:

Increasing the nanosilica to 2% results in agglomeration of the added nano materials which negatively affects the stability of matrix against acid attack but the microstructure still cohesive as compared with the plain cement mix where more honeycomb C–S–H structure (with high Ca/Si ratio) spreads over the surface, which known by its lower stability against acid attack as compared with fibrous C–S–H one illustrated in 1% NS.

4. Conclusions

a) The properties of cement pastes are improved by the use of NS, since nanoparticles fill the voids between cement grains and also consume a part of calcium hydroxide which results in additional formation of calcium silicate hydrate (C.S.H) and more improvement of microstructure in both normal and aggressive curing conditions.

b) The application of NS particles with newly developed super plasticizer (polycaboxylic ether polymer based PCE sky) improved the durability and strength of cement pastes since nano-SiO_2 interpenetrates polymer network and causes the above improvements.

c) The efficiency of NS depends on its morphology as well as the application of effective superplasticizer which helps to disperse formation of agglomerates and improve cement strength and durability.

d) Nanosilica found to be very effective when mixed separately with superplasticizer polycarboxylate base) and then added to the 50% remained of mixing water as mentioned in mixing procedure.

e) NS was addition, results in significant increase in compressive strength and durability after 28-days up to one year for both normal and aggressive curing and the optimum amount of nanosilica is 1% by weight of cementitious material in addition to 10% fly ash (wet mixing procedures).

f) The mechanical properties of NS/FA cement pastes exposed to aggressive curing conditions maintained a growth within a certain age range up to 28 days, followed by server losses up to one year, but still better than control samples without NS and FA at all ages.

g) Resistance of cementing materials exposed to sulphuric acid attack can be improved by the addition of 1% NS and 10% FA by weight of cementitious materials, which concluded to be optimum mix [Mix No. 3].

h) Addition of NS 2% or more and 10% FA resulted in more agglomeration which negatively affected the cohesion of the formed binding materials leading to the formation of high pore volume and hence heterogeneous structure and lower strength for both normal and aggressive curing conditions.

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Dear colleagues!
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ОЦЕНКА ПРОЧНОСТНЫХ ХАРАКТЕРИСТИК ЦЕМЕНТНЫХ РАСТВОРОВ НА ОСНОВЕ НАНОКРЕМНИЯ И ЗОЛЫ-УНОСА В НОРМАЛЬНЫХ И АГРЕССИВНЫХ УСЛОВИЯХ ВЫДЕРЖИВАНИЯ БЕТОНА

Цель исследования состоит в изучении влияния золы-уноса (ЗУ) и нанокремния (НК) на прочностные характеристики цементных растворов в нормальных и агрессивных условиях выдерживания бетонов. Были приготовлены смеси с различным количеством НК и постоянным содержанием ЗУ (10% от массы вяжущих веществ). Первая группа образцов была подвержена нормальным условиям выдерживания и испытана через 3, 7, 28 дней и 1 год, вторая группа подвергалась воздействию серной кислоты концентрацией 0.2 N и протестирована через те же интервалы времени. Наблюдение за процессом гидратации и прочностью цементных растворов производилось с помощью сканирующего электронного микроскопа. Результаты исследования показали, что ЗУ и НК, взятые в количестве 10% и 1%, соответственно, от содержания вяжущих веществ, улучшают прочность и микроструктуру цементных растворов как в нормальных, так и в агрессивных условиях выдерживания бетона.

Ключевые слова: зола-унос, нанокремний, серная кислота, агрессивные условия, прочность, микроструктура

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