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Original Research Article

Comparative Study on the Durability of Nano-Silica and Nano-Ferrite Concrete

D. E. Tobbala[1*]

[1]Department of Civil and Architecture Constructions, Faculty of Technology and Education, Suez University, Egypt

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Abstract: Adding of Locally prepared nano-silica [1] to concrete improves the mechanical properties and microstructure of SF concrete [2]. Nano-silica was added with different percentages to the SF concrete to study their effect on the durability properties. Seven mixes were prepared with constant cement content of 400 kg/m^3 and additional percentage of silica fume 15% of cement content. Nano-silica and nano ferrite was used at 1-3% and 1-2% of the cement content, respectively. The water permeability, the resistance of the $MgCl_2$ on concrete and the resistance of the electro-chemical of accelerate corrosion. It was found that all the mechanical properties, especially compressive strength of silica fume blended mixes was improved using either nano-silica or nano-ferrite as additional percentages. Also, these properties were improved with increasing the nano-silica or nano-ferrite percentages. The results showed that adding 2% nano-silica was considered the most economical percentage with high properties.

Keywords: Nano-Silica; Concrete; Nano-ferrite; Microstructure; Mechanical Properties.

1. INTRODUCTION

Recent developments in nano-technology and availability have made the use of such materials in improving concrete properties possible started in the early 2000s [3-6]. In general, nano-sized silica (NS), ferrite (NF), Alumina (NA), and other nano powders oxides (such as ZnO_2, CO_2 and TiO_2) were used in the studies. Nano-silica materials are prepared using several methods including, vapor phase reaction, sol–gel and thermal decomposition technique. The powder production of silica from every one of the previous methods is a highly energy intensive process. Unlike this, economically viable and high-grade, nano-size amorphous silica from rice husk can be produced simply by burning under appropriate conditions [1, 2, 7-9]. Also, cobalt ferrite had been prepared by many methods such as auto-combustion, co-precipitation, hydrothermal synthesis and mechanical alloying, etc [10].

As known, NS is more reactive pozzolanic material than silica fume (SF) when reacting with CH because it has a very high content of amorphous silicon dioxide (more than 99%) and consists of finer amorphous particles. NS has very high structural characteristics where silicate anion layers have a net negative charge and are held together by Ca^{+2} cations in the interlayer region, which also contains H_2O molecules. Also, the addition of NS reduces pores, and improves the interface bonding between hardened cement paste and aggregate [11]. Previous studies showed very high mechanical properties of concrete [12-14] by using a small amount of NS up to 5% in their mixtures. Nano-ferrite (NF) powder was used in some researchers to improve mechanical properties of concrete with very small contents. They reported that addition of NF with small content improved the different hardening properties of concrete up to 2% [15]. They reported that the replacement of 1% of cement with nano-ferrite increased the strength with an average of 45 %. A comparison was held between the effect of NF and NS [16, 11] on compressive and flexural strengths of cement mortars. The results showed that, the compressive and flexural strengths of the cement mortars with NS and NF were both higher than that of the plain mortar with the same water to binder ratio (W/B). Moreover, if the nano-particles were uniformly dispersed, the microstructure would be improved because they acted as fillers and as activators to promote hydration. Both studies [17, 18] suggested that the interaction between powders taking place in binary and ternary combinations leads to certain negative effects on the physical–mechanical properties of the mortars, so the use of single powders can be recommended. But there are no sufficient researches about the durability of nano-particles concrete. Kong *et al*. [19] found that the reduction in the blank mortar compressive strength after exposure to ammonium chloride solution after 28 days water curing were reduced from 25.1% and 41.4% to 13.4% and 34.4%, respectively as a result of addition 1% of NS. Ibrahim *et al*. [20] reported that the addition of NS increased the compressive strength of concrete tested after elevated its temperature to 400 ˚C for 2 hr which increased with increasing NS content, but at 700˚C, the comp. strength decreased with increasing NS content as shown in Fig. 2.22 They also reported that after exposure to elevated temperature, the pore size distribution for the materials decreased with the addition of NS. The addition of NF with small percentages reduced the reduction in the compressive strength of concrete after elevated the temperature up to 400˚C. The results showed that of 1% of NF had the lower reduction in strength [6]. Amer *et al*. [21] found that NF was synthesized by thermal decomposition of basic ferric acetate fired at 275, 600 and 800 ˚C. OPC admixed with 1% NF prepared at 275 ˚C gave the higher fire resistance than those admixed with 2 or 3%.

In this paper, NS and NF were prepared from locally available materials with suitable cost. 100% pure NS with 6.5 nm avg. particle size (PS) was prepared by burning treated rice husk [19], while 100% pure NF with 29.62 nm avg. PS was prepared chemically by citrate-gel method. These two types of nano-particles (i.e. NS and NF) were added with small percentages to the concrete constituents to study their effect on the microstructure and mechanical properties and durability of high strength concrete.

2. EXPERIMENTAL WORK

2.1 Nano-Materials

Two different types of nano-particles namely nano-silica (NS) and nano-ferrite (NF) were prepared from locally available materials. NS was locally prepared from rice husk using hot HCL chemical treatment [1]. The resulted NS was an amorphous powder with 100% purity and avg. particle size 6.25 nm. The cost of prepared NS was 400 LE/kg while the available commercial NS was 1000 LE/kg.

$CoFe_2O_4$ nano-crystalline powder (NF) was prepared by citrate-gel method. It was prepared by dissolving a mix of cobalt nitrate (Co $(NO_3)_2.6H_2O$) with molecular weight (M.W) of 291.04, ferric nitrate (Fe $(NO_3)_3.9H_2O$) with M.W 404 and anhydrous citric acid ($C_6H_8O_7.H_2O$) with M.W of 210.14 in distilled water. Ammonia was used to get rid of acidic. The solution was heated on a hot plate until drying and flashing. Finally, it was grounded in an agate mortar. NF main characteristics measured by XRD correlates that the sample chemical composition was pure 100% with 29.62 nm avg. PS and d-spacing [Å], see Fig. 1-a. Vibrated Sample Magnetometer (VSM) which correlates that there was symmetry around origin with Magnetization (M) of 63.521 emu/g and magnetic field (H) up to1660 Oe, see Fig. 1-b. The loop shows the ferromagnetic character with meanly saturation. The values of saturation Magnetization (Ms), remnant Magnetization (Mr) and coercive field (Hc) were illustrated on the Fig.1-b. The molecular weight of $CoFe_2O_4$ was 234.62. The magnetic susceptibility test correlates that the transition temperature of $CoFe_2O_4$ was 835.27˚ K. The cost of prepared nano-$CoFe_2O_4$ was 2000 LE/Kg while the commercial was 3500 LE/kg.

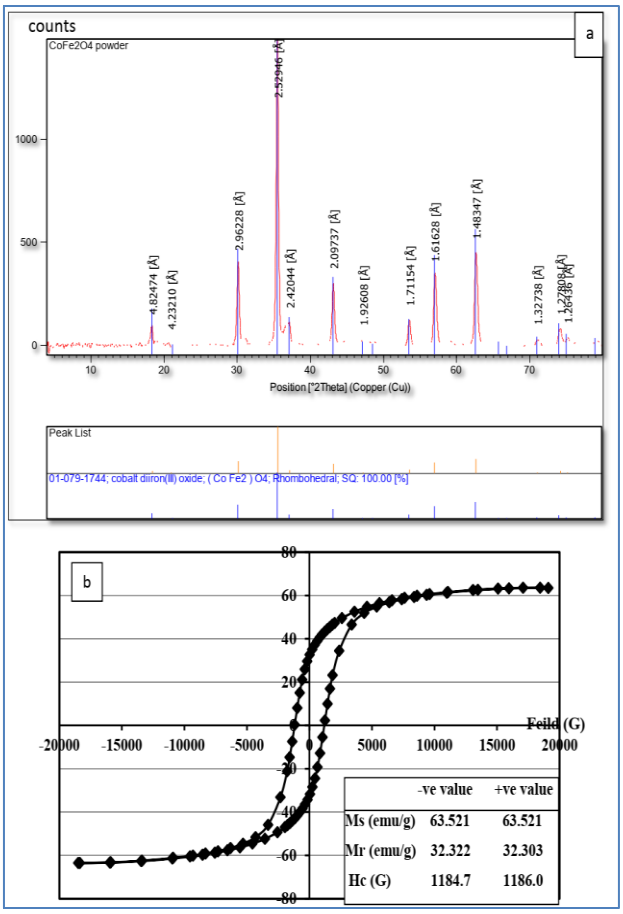

Fig-1: Main characteristics of prepared NF (a) XRD, (b) VSM

2.2. Other concrete materials

Type me ordinary Portland cements (OPC) and SF were used in all mixes. The fine aggregate was natural sand (S) with a fineness module of 2.6 and a specific gravity (S.G) of 2.67. The coarse aggregate was dolomite (D) with NMZ of 12 mm, specific weight of 2.7 and water absorption of 3%. The super-plasticizer (SP) was Sika Visco-Crete 5-930 complying ASTM-C-494 types G and F and had a density of 1.08 kg/m^3. It was used to aid the dispersion of NS in concrete mix and to achieve good workability of mixes.

2.3 Mixes and tests

Seven concrete mixes were proposed, as shown in Table 2. In all mixes the cement content (C**),** the percentages of S/Agg, W/B and SP/B were kept constant at 400 kg/m^3 and 40, 30 and 4%, respectively, where the binder (B) was C, SF and NP. Due to the high surface energy and small contents of nano-particles, their uniform dispersion in the mix is not easy. The concrete constituents of all mixtures were mixed in a rotary mixer with constant procedures as in [2].

The concrete specimens used in different tests were cubes with dimensions of 70.7×70.7×70.7 mm for compressive strength (f_c) and affected specimens of $MgCl_2$, cubes of 100×100 ×100 mm reinforced by center bar of 10 mm diameter and 200 mm length for bond strength (pull out test) (f_b) of the effect of electrochemical accelerate of corrosion and concrete cylinders with dimensions of 75×150 mm for both of splitting tensile strength (f_{sp}) and static modulus of elasticity (E). All specimens were shacked on the vibrating table for 10 second and then they were demolded after 24 hours and cured by immersing in water at laboratory temperature till the testing date except affected specimens of $MgCl_2$ and the electrochemical. The compression test was carried out according to BS 1881: part 116: 1983 and tested at ages 7, 28, 56, 120 and 180 days while all other were tested at 28 days only. The splitting tensile test was carried out according to BS 1881: part 117: 1983. The bond test between a reinforcing bar and the surrounding concrete was carried out by using the pull out test according to BS 1881: part 207: 1992. The static modulus of elasticity (Ec) in compression was carried according to ASTM C469-65-(1975).

Table-3: Mixture proportions of concrete mixes, all the component unit is with kg/m^3

Mix no.	C	SF	NS	NP-type	Sand	Dolomite	W	SP
M_0	400	0	0	0	792.3	1188.4	120.0	16.0
M_{Sf}	400	60	0	0	740.8	1111.2	138.0	18.4
M_{NS1}	400	60	4	NS	737.2	1105.8	139.2	18.6
M_{NS2}	400	60	8	NS	733.6	1100.5	140.4	18.7
M_{NS3}	400	60	12	NS	730.1	1095.1	141.6	18.9
M_{NF1}	400	60	4	NF	737.2	1105.8	139.2	18.6
M_{NF2}	400	60	8	NF	733.6	1100.5	140.4	18.7

To study the change in microstructure of specimens due to NS incorporation, small cubes with 50×50×50 mm dimensions were prepared using cement pastes of mixes M_0, M_{SF}, M_{NS2} and M_{NF2}. These cubes were subjected to compression up to failure, then a very small portions was taken from the core of each cube to carry out the scanning electron microscope (SEM). This test was carried using a Field Emission Scanning Electron Microscope (FE-SEM). The fracture surface of the specimens was scanned at 7 and 28 days ages.

3. RESULTS AND DISCUSSION

3.1 Mechanical properties

Figure 2, presents the compressive strength and their development percentages of the 7 mixes at 7, 28, 56, 120 and 180 days age. The strength of control mix was 37.6, 55.5, 62.5, 67.8 and 73.7 MPa, respectively. The addition of SF improved the strength of control mix with percentages 35.37, 29.55, 23.8, 23.6 and 17.8 %, respectively. Figure 2, illustrated also that extra increase was observed in mixes incorporation nano-particles over SF improvement and increases with the increase of their percentages. This may be due to the very small particle size, high surface area and high purity of used NS and NF specially the very high pozzolanic reaction of NS. For example, the development when adding 1, 2 and 3% of NS were 50.09, 66.67 and 82.98 %, at 28 days age and 41.78, 59.03 and 68.53 %, at 180 days ages, respectively. On the other hand, the addition of prepared NF with the same percentages of 1and 2% improved the strength of control mix with percentages 44.2 and 72.3 %, at 28 days age and 30.12 and 47.5 %, at 180 days ages, respectively. Comparing the compressive strength of 1% of NS with NF concrete, it was found that the concrete strength of NS is higher than NF at all ages. While when compared the strength of 2% of their concrete, it can be noticed that 7 days strength of 2% NF was better than NS. Their difference decreased with increasing time until being marginal difference at 28 and 56 days then at later ages 120 and 180 days the improvement of NS came over NF, see Fig.2. So, the differences in 7, 28 and 56 days compressive strengths between 2% NF and 2% NS were useless when compared with the increasing in cost. Moreover, when the compressive strength of 3% NS was better than 2% NF at all ages and cost of 3% NS addition of one cubic meter of concrete was considered 30% of 2% NF addition.

The improvements of compressive strength (f_{cu}), splitting tensile strength (f_{sp}) and modulus of elasticity (E) of different NS and NF percentages mixes at 28 days age are shown in Fig. 3. This Figure illustrates that an extra increases in the improvements of all the mechanical properties were observed in mixes incorporation NS and NF over SF. Also, these improvements increased with the increase of their percentages. It can be seen that the improvements in f_{cu}, f_{sp} *and* E of M_{SF} were 29.55, 20.31 and 20.83 % and increased to 66.67, 57.84 and 44.17 % MPa with adding 2 % NS, respectively, while increased to 72.32, 59.48 and 44.58 for mixes with adding 2% NF, respectively. But their improvements were 80.9, 70 and 58.33 % for mixes with adding 2% NF, respectively, with smaller cost of 2% NF.

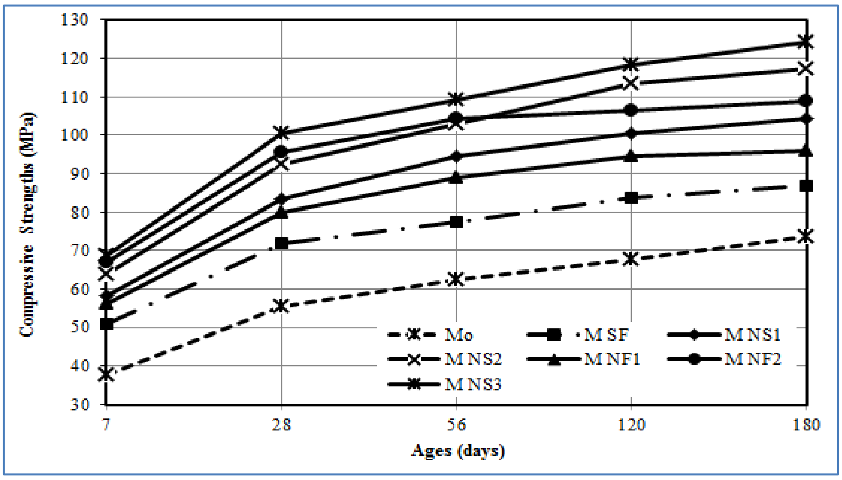

Fig-2: Compressive strength of different mixes at different ages

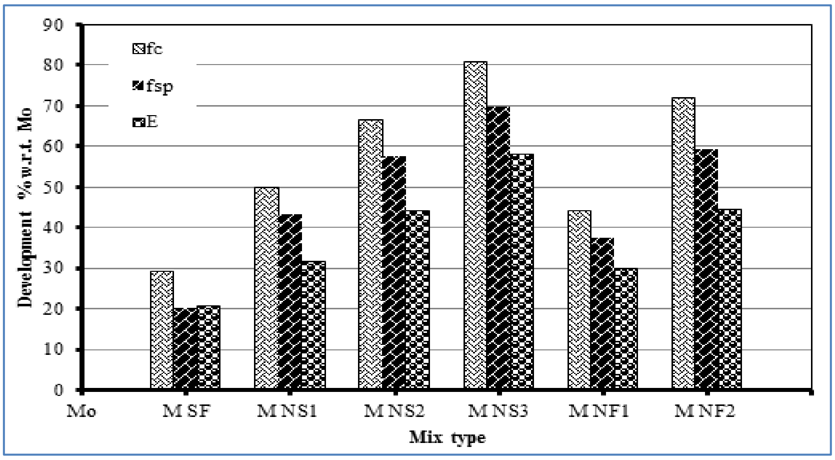

Fig-3: Compressive strength (f_c), splitting tensile strength (f_{sp}) and modulus of elasticity (E) improvements of different types and percentages of nano-particles w.r.t. Mo.

3.2. Water Permeability Coefficient

Figure 4 represents the improvements of the water permeability coefficient (K) of nano-particles mixes at 28 days age. K coefficient of Mo was 4×10^{-11} cm/sec reduced with 59% when adding 15% of SF. As can be seen there were considerable reduction in K coefficient with adding small dosage of NS and NF of only 1% reached 78 and 76.2%. These increases were continued with the increase in NS or NF content. The reduction in K of M_{NS2}, M_{NS3} and M_{NF2} were 81.25, 84.38 and 81.8%. Although the reduction in K% of M_{NF2} was better than of M_{NS2} with small percentage, the reduction of M_{NS3} was better M_{NF2} with smaller cost.

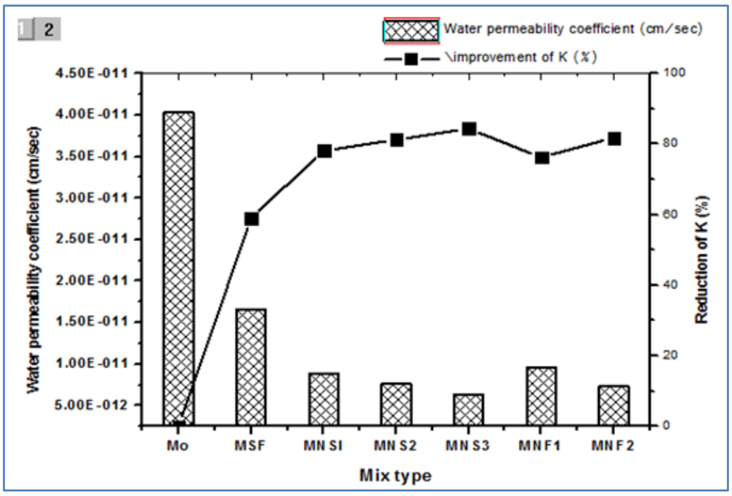

Fig-4: Water permeability coefficient (K) of nano-particles mixes and their reductions percentages

3.3 Durability of nano-particles concrete

3.3.1 Chemical Attack ($MgCl_2$) aggressive environmental

The reduction percentages in the compressive strengths due to the effect of chloride either 30 gm/L or 60 gm/L w.r.t the water curing specimens of all tested mixes were illustrated in Figs. 5 at 28, 56, 120 and 180 days ages. The reduction in the compressive strength of all mixes increased with increasing the concentration from 30 to 60 gm/L. For example the reductions of M_O were 3.4, 6.9, 16.6 and 25.6 % at 30 gm/L and 5, 10.1, 21.7 and 30.4 % at 60 gm/L. The addition of SF reduced the reduction to 2.8, 5.3, 12.6 and 17.3% at 30 gm/L and 4.5, 7.4, 17.4 and 21.2 % at 60 gm/L. All of NS_C and NF mixes continued their improvement with time although exposing them to chloride but their development had a small reduction when compared with which curing in water due to the effect of chloride. It was found that the increasing of NS or NF with small contents aided to reduce the effect of chloride, as illustrated in Figs. 5. When compared M_{NS1} with M_{NF1}, found that NS was better than NF in resistance the two concentration of the Mg Cl_2. For example, the reductions of M_{NS1} and M_{NF1} mixes due to 60 gm/L of chloride at 28 days age were 3.7 and 3.9 % reduced to 2.8 and 2.3 % when increased their percentages to 2%, respectively. While the reductions of M_{NS1} and M_{NF1} mixes due to 60 gm/L of chloride at 180 days age were 11.2 and 11.8% reduced to 9.3 and 9.8 % when increased their percentages to 2%, respectively. The reduction in compressive strength of $M_{NS1}$was smaller than M_{NF1} at all ages. The reduction of M_{NF2} smaller than M_{NS2} at early ages but the reduction of M_{NS2} was smaller than M_{NF2} at late ages. But when compared the reduction of M_{NF2} with M_{NS3} which reduced with 2.2 and 6.8% at 28 and 180 days ages, respectively found that the resistant of M_{NS3} was better than M_{NF2} at all ages with smaller cost which assure with their mechanical properties.

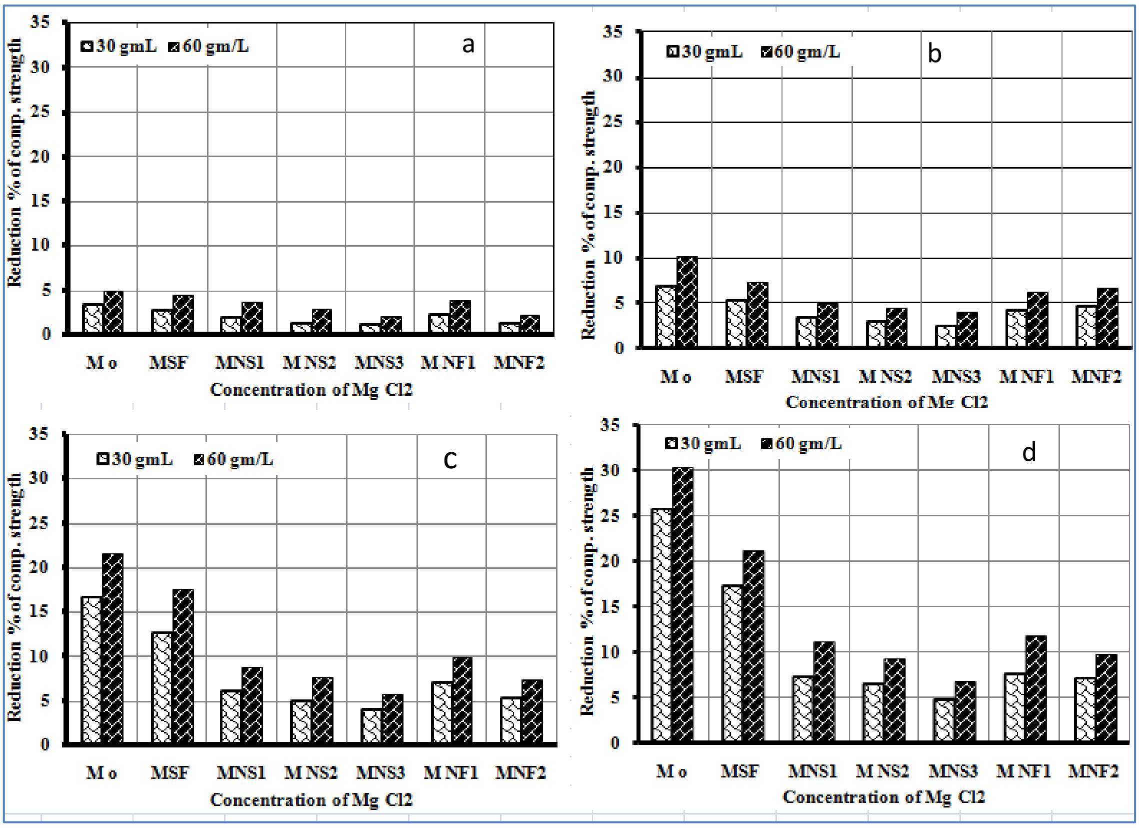

Fig-5: The reduction% in f_c of nano-particles mixes due to curing in aggressive $MgCl_2$ Sol. for: a) 28, b) 56, c) 120 and d) 180 days, w.r.t their water curing.

3.3.2. Fire effect on the compressive strength of concrete

The fire affected compressive strength of tested concrete mixes calculated from fracture load of 70.7×70.7×70.7 mm concrete cube after exposing to fire at constant temperature of 600 ˚C under constant load of 70 KN for 1hr. The fire temperature was raised to 600˚C with rate 50˚C/min then kept constant at 600˚C which was measured on the surface for 1 hr. while the constant load was determined from the initial cracking load of the control mix. The affected compressive strength of tested mixes and their strength reduction % when compared with their main compressive strengths were shown in Fig.6. It was found that the reduction in the compressive strength of M_o reached 6.5% increased to 14.8% when adding 15 % SF which agree with that the reduction in the permeability increasing the effect of fire on the compressive strength [21]. These reductions continued their increasing with the increasing of NS or NF contents, reached 20.4 and 25% at 1 and 2% NS and reached 18.4 and 39.05% at 1 and 2% NF, see Fig. 6. But the compressive strength of NS and NF concrete still better than the main strength of MO and MSF. The affected strength of 1 and 2 %NS concrete was better 1 and 2% NF concrete although the reduction in 1% NF was less than 1% NS, see Fig.22. So, the fire resistance of 1 and 2% NS concrete can be considered better than 1 and 2% NF. The addition of 1 and 2% of NS had a good resistance of fire.

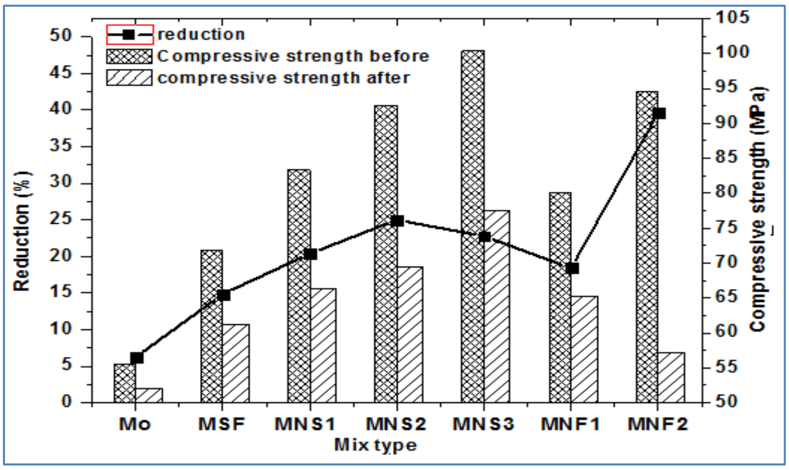

Fig-6: Effect of fire on prepared NS_C vs. NF concrete mixes compressive strength and their reductions %.

3.4 Microstructure of cement pastes

The microstructure results of cement pastes at 7 and 28 days are shown in Figs. 24–27. Fig. 24 shows SEM of plain cement paste in which C-S-H gel exists as separated clusters, lapped and jointed with needle hydrates. At the same time, deposition of calcium hydroxide (CH) with big crystals is distributed among the cement paste. Fig. 7 shows SEM of SF cement paste indicating increment of C-S-H gel, decreasing of CH and pores and disappearing of needle hydrates. Moreover, SF can act as micro filler. Fig. 8 illustrates that the micro-structures of Ns paste were better than SF paste at ages 7 and 28 days ages. That is may be due to its very high surface area compared with SF which accelerated its reaction with CH crystals forming C-S-H filling distributed pores dramatically beside its physical effect in filling nano-pores. When comparing between SEM of NS with NF at ages 7 and 28 days ages, as illustrated in Fig. 9 and 10, shows that the SEM of 2% NF was better than 2% NS at 7days age but with time NS can be improved and be nearly the same as NF at 28 days. That is because NF may has a physical effect which can act as nuclei of hydration which accelerate hydration process of cement and pozzolanic reactions of SF at early ages the hydration processes while the reaction of NS with all CH production of hydration takes time.

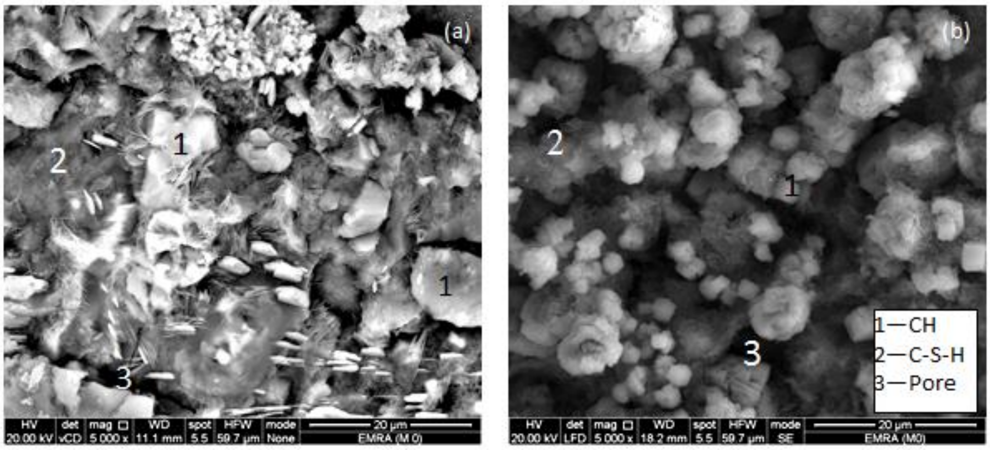

Fig-7: SEM of control mix at a) 7 days and b) 28 days ages

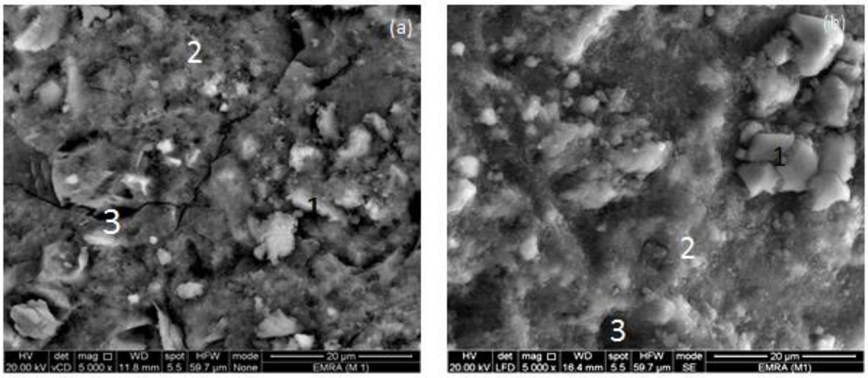

Fig-8: SEM of SF mix at (a) 7 days and (b) 28 days ages.

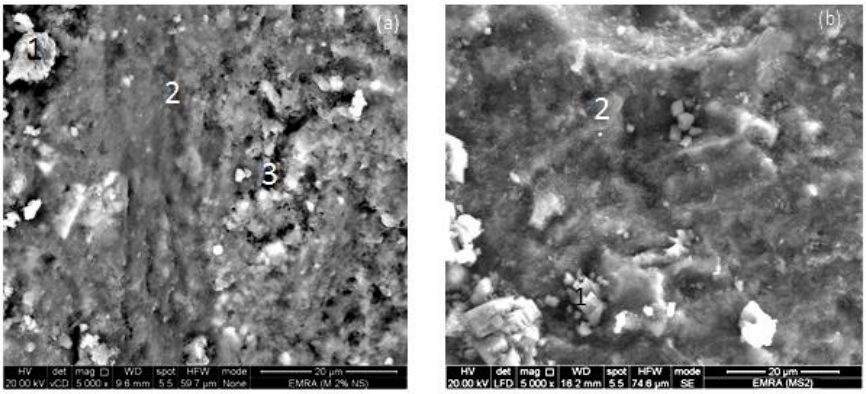

Fig-9: SEM of mix M_{NS2} (a) 7 days and (b) 28 days age

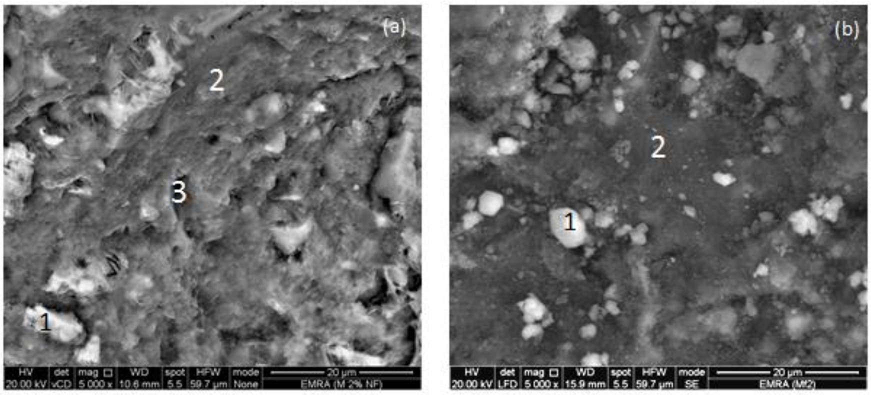

Fig-10: SEM of mix M_{NF2} (a) 7 days and (b) 28 days age

4. CONCLUSION

1. SF addition improved all the mechanical properties of control mix at all ages for example the 28 days age compressive strength improvement was 29.55%.
2. The addition of NS or NF with 1and 2% improved the mechanical properties of control mix over SF concrete. Extra improvement achieved when NS increased from1% to 2%. The strength 1% NS is better than 1% NF at all ages. For examples, at 28 days the compressive strength improvement of M_0 strength over SF was 20.54 and 14.65 % when adding 1% of NS and NF then increased to 37.12 and 42.75 % when their percentages increased to 2%, respectively. So, 2% NF concrete strength was better than 2%NS at 7 days age, the difference decreased with increasing time until NS overcome NS at 120 days age.
3. The cost of NS is 20% of NF, so NS is considered more economical.
4. SEM photos illustrates that SF addition improved paste of plain concrete by reacting with some free CH producing CSH clusters. Extra improvement achieved in the matrix density by adding 2% NS which reacted with more of CH reducing the pores and made paste matrix more impermeable. Moreover, SEM of 2% NF was better than 2% NS at 7days but at 28 days them were be nearly same.

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