THE EFFECT OF NANO SILICA ON THE PROPERTIES OF CEMENT MORTARS CONTAINING MICRO SILICA AT ELEVATED TEMPERATURES

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This study investigates the effects of nano silica admixture on the behavior of cement mortars containing micro silica exposed to elevated temperatures. The cement mortars were incorporated with an optimized ratio of 5 wt% micro silica and 0,1,2,3wt % of nano silica admixtures. The specimens were exposed to elevated temperatures of 300 and 600 °C. After cooling, the mass loss, ultrasonic pulse velocity and compressive strengths of the specimens were determined. The investigations undertaken in this study were supported by scanning electron microscopic images. As a result, there is an optimum nano silica content which can be beneficial for improving the thermal resistance of cement mortars. Nano silica (2 wt%) improved the resistance of cement mortars containing 5 wt% micro silica at elevated temperatures.

Keywords: *Cement mortar, nano silica, micro silica, elevated temperature*

1. Introduction

Nano particles refer to the particles with the size less than 10^{-9} m. Particles of this size have distinctive mechanical, optical and electronic properties which have been a cause of interest to researchers around the world. Of late, the effect of nanoparticles in the field of Civil Engineering has been attracting attention and has led to various researches being conducted in that area. The various application areas include mortar, concrete, steel, wood, asphalt, coatings and water filtration among others [1].

The potential of nanotechnology to improve the performance of concrete and to lead to the development of novel, sustainable, advanced cement based composites with unique mechanical, thermal, and electrical properties is promising and many new opportunities are expected to arise in the coming years. The advances in instrumentation and computational science are enabling scientists and engineers to obtain unprecedented information about concrete, from the atomic through the continuum scale, and the role of nanoscale structures on performance and durability. This information is crucial for predicting the service life of concrete and for providing new insights on how it can be improved [2]. Many researches have conducted experiments on the physical, chemical, mechanical and microstructure properties of cement paste, mortar and concrete impregnated with nano particles. One of the most widely investigated is nano silica (NS) [3-12].

Nano materials are being added to the cement mixtures in order to improve the mechanical properties of mortar and concrete. Research is then being carried out to determine the most efficient and economical way in which they can be used [1]. Optimal NS content have been inconsistently reported by previous studies. Shahrajabian and Behfarnia (2018) investigated the effect of nanoparticles including NS, nano alumina, and nano clay on the resistance of alkali-activated slag concrete against freeze and thaw cycles [13]. It was proved that due to the greater impact of NS and its lower price, in order to improve the compressive strength and durability of concrete under freezing-thawing cycles, it is recommended to use NS up to 1%. In partially replaced cement based mortar and paste, the optimum content of nano-materials is 2-3% by the weight of binder materials [14]. The NS up to 5% proved to be an effective mineral addition for blending with OPC to improve its chemical, physico-mechanical and thermal properties [15]. Nazari and Rihai (2010) investigated flexural strength, thermal properties and microstructure of self-compacting concrete with different amount of NS. They founded that NS up to 4 wt% could improve the mechanical and physical properties of the specimens [16]. Najigivi et al. 2013 founded that the cement can be advantageously replaced by both types of NS up to a maximum limit of 2.0% to improve the strength of binary blended concrete after curing in lime solution, although, the optimal incorporation level of NS particles for maximum strength was gained at

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1.0% cement replacement after curing in water [17]. Experimental results showed that partial replacement of cement by NS decreases the workability of fresh concrete; therefore, it can be concluded that the addition of superplasticizer can be advantageous for high-volume nanoparticle blended concretes. According to previous studies experimental results indicated that the durability of cement based composites is generally improved by adding NS [18]. Taengua et al. 2015 have reported that the addition of NS increased compressive strength and density of the mortar, and a quadratic trend was observed in these relationships. Maximum compressive strength values were observed at an NS content of 1.63% [19]. Li et al. 2004 carried out that the compressive and flexural strength of the cement mortars with NS were higher than that of the plain cement mortar [20]. NS reacts with calcium hydroxide (CH) and increases the amount of calcium silicate hydrate (C–S–H) produced, leading to a compact microstructure and, consequently, improving the mechanical properties of hardened [21]. The pozzolanic activity of NS was much greater than that of micro silica (MS). The reaction rate of Ca $(OH)_2$ with nano SiO_2 and the velocity of C-S-H gel formation from $Ca(OH)_2$ with NS were much quicker than that of $Ca(OH)_2$ with MS [22].

Li et al. 2017 pointed out that NS should not be added alone but should be added together with MS so that the MS and NS would successively fill into the voids to increase the packing density of the cementitious materials and densify the microstructure of hardened cementitious paste. This is the best way to utilize the good filling effect of MS and the high pozzolanic reactivity of the NS so as to further increase the strength and durability of concrete for the production of the next generation high performance concrete [23].

Li et.al 2017 investigated the combined effects of MS and NS on the durability of concrete. İt was founded that the additions of MS and/or NS would significantly increase the cube strength, sulphate resistance and carbonation resistance of mortar. Basically, the addition of just 1% NS is almost as good as the addition of 10% MS, and the combined addition of MS and NS could offer certain synergistic effects in the sense that the combined effects of MS and NS are larger than the respective sums of the individual effects of MS and NS [23].

Li et al. 2018 studied the combined effects of MS and NS on the compressive behaviour of concrete. With both MS and NS added together (%5 MS+1% NS), the compressive strength and elastic modulus could be increased to higher than those achievable with only MS added or only NS added [24].

The authors [25] investigated on the cement composite with MS and with 9% of NS. It was found that the microstructure of control cement paste and cement paste with MS was similar. MS has lesser pozzolanic activity and filling ability compared to NS. Also, the MS has larger particles that reduce the packing ability as well as pozzolanic activity.

It is well established that mechanical properties of cement based composites are adversely affected by thermal exposure. Concrete typically loses between 10 to 20% of its original compressive strength when heated to 300 °C and between 60 to 75% at 600 °C [26]. Aggregate type, cement type, mineral admixtures affect cement based composites at elevated temperatures. These contents have investigated by several researches [27-32].

Bastami et.al 2014 studied effect of elevated temperature (400, 600, 800 C) on of high strength concrete modified with NS and on its compressive and tensile strengths, spalling, and mass loss. They observed that the presence of NS increased residual compressive and tensile strengths, and spalling and mass loss are decreased as penetrability increased [33].

Horszczaruk et.al 2017 explored that there is an optimum NS content which can be beneficial for improving the thermal resistance of cement mortars. NS (up to 3 wt%) improved the resistance of cement mortars at elevated temperatures, especially to temperatures up to 200 °C. For higher temperatures (from 400 °C) the effect is less pronounced or not significant [34].

The study aimed to evaluate the effect of nanosilica on the performance of cement mortars containing 5wt % micro silica at elevated temperatures.

2. Materials and methods

2.1. Materials

CEM I 42.5R Portland cement from Turkey, MS, NS, water and standardised sand were used in the study. Physical and mechanical characteristics of the used cement are shown in Table 1, Chemical analysis is shown in Table 2.

MS was obtained from Antalya Eti Metallurgical Business in Turkey and its grain

Table 1

Physical and mechanical properties of the Portland cement

Compressive strength (MPa)			Initial setting time (h)	Final setting Time (h)	Le Chatelier (mm)	Specific gravity (g/cm^3)	Blaine (cm^2/g)
32,5	43,4	53,6	150	210	2.0	3,12	3269

Table 2

Chemical analysis of the portland cement

Compound	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	Loss of ignition
Weight %	62,64	19,05	4,56	3,36	2,98	2,88	0,15	3,02

Table 3

Chemical properties of MS

Compound	SiO_2	Al_2O_3	Fe2O3	CaO	MgO	SO_3
Weight %	81,40	4,47	1,40	0,82	1,48	1,30

Table 4

Amounts of materials used in the preparation of the mortar mixtures

MS content %	NS content %	Mix code	Cement, g	MS, g	NS, g	Standart sand, g	Water, g
5	0	5S	427,5	22,5	0	1350	255
5	1	5S+1NS	423,0	22,5	4,5	1350	255
5	2	5S+2NS	418,5	22,5	9,0	1350	255
5	3	5S+3NS	414,0	22,5	13,5	1350	255

Table 5

Quality of concrete as a function of the ultrasonic pulse velocity

Ultrasonic pulse velocity (km/s)	>4.5	3.5–4.5	3.0–3.5	2.0–3.0	<2.0
Concrete quality	Excellent	Good	Doubtful	Poor	Very Poor

density was 2.32 g/cm^3. The specific surface area of MS is 3350 cm^2/g and its chemical properties are shown in Table 3.

NS mean particle size was 30 nm, with 99.9% purity. Surface area was 440 m^2/g, color was white. The mass of the NS in spherical form was 2.2-2.6 g/m^3.

2.2. Method

The proportions of the mortar components according to TS EN 196-1 are in mass; one part of cement (450 g) is prepared as three parts of standard sand (1350 g) and ½ part of water (225 g). In this study, four different mortar mixtures were prepared with cement, MS, NS, standardised sand and water. Mortar specimens were prepared according to TS EN 196-1 [35] in a cement laboratory with 50–60% relative humidity. Mortar mixtures were prepared in a laboratory mixer with a capacity of 5 L. Cement and sand were mixed at low speed for 1 min to provide homogeneity. After adding water to the mixture, it was mixed at low speed during the first minute, at a high-speed for the second minute. After the mixing process, three mortar samples were molded into 5 cm cubes for the high temperatures. Amounts of materials used in the preparation of the mortar mixtures are given in Table 4 (in grams).

Cube specimens were kept in the room with 20 °C temperature and 90% relative humidity for 24 h. The specimens were removed from the molds after 24 h. They were placed in pools at a temperature of 20 °C until 90 days. The pool water was constantly ventilated and changed once every 14 days.

The hardened cement mortars were dried at a temperature of 105 ± 5 °C for 24 h in an oven to remove the free water. After that, mortar specimens were thermic treated in the oven at 300°C and 600°C for 3 hours. The specimens were allowed to cool gradually inside the furnace to room temperature.

Cube specimens after cooling down the specimens to room temperature were used for determination of mass loss, ultrasonic pulse velocity and *compressive strength*. Results were compared with data obtained for the control specimens, which were stored at 20 ± 2 °C in the laboratory, with the specimens having been dried at 100±5 °C.

One of the important changes in cement based composites when exposed to high temperatures is a reduction in mass that is usually due to the evaporation of water in the concrete [36]. To assess the extent of mass loss, the mass of cement mortar specimens was measured before and after exposing to heating, under the same conditions, and the mass reduction was reported as the mass loss.

The *Ultrasonic Pulse Velocity* System has been used for more than 50 years. It is a very suitable method for the study of the quality of cement based composites by measuring the speed of propagation of ultrasound waves [37]. Measurements were taken for each test specimen by switching the position of the transducers between the two opposite faces of cubes. For all mortar mixes (5S, 5S + 1NS, 5S + 2NS and 5S + 3NS) ultrasonic pulse velocity was measured at 90 days. Also all mixes ultrasonic pulse velocity was measured after exposing to heating. The ultrasonic pulse velocity of mortars was determined according to ASTM C 597 [38]. Table 5 shows the use of velocity obtained to classify the quality of concrete [39,40].

Three 5 cm cube specimens were tested for determination of the compressive strength according to the TS EN 196-1.

SEM analyzes were carried out in order to have knowledge about internal structures of hardened mortars after physical and mechanical tests after heating of exposing 600 ºC. Gold was sputtered on samples, images were obtained at 5000 magnifications. Mortar samples kept at 20 °C as control temperature were used.

3. Results and Discussion

On the 90th day, mortar specimens were exposed to 300°C and 600°C for 3 hours and cooled gradually inside the furnace to room temperature. Mass loss, ultrasonic pulse velocity and compressive strength tests carried out.

Mass loss, which is usually caused by the evaporation of water in concrete, is one of the important changes occurring in specimens when exposed to high temperatures [36]. Figure 1 shows that the mass loss is reduced in the mixtures containing 1% and 2% NS compared to the 5S mortar mix, and increased in the mixture containing 3% NS. The mass loss at 600 °C was found to be at least 6.88% in the 5S+2NS mixture, and the most mass loss was seen in the 5S + 3NS mixture with 8.10%.

In general, mass loss due to an increase in temperature from ambient to 200 °C could be attributed to the elimination of evaporable water and the release of free water in the capillary pores. Mass loss at 400 °C could be due to the elimination of water in the chemical bonds as a result of the decomposition of carboaluminate hydrates. At 400 °C, all the capillary water is lost. Mass loss was inevitable because most bonds could breakdown in the C-S-H gel at temperatures above 600 °C [36, 41]. Mass loss was governed by the decomposition of other compounds through processes such as portlandite decomposition or decarbonation [42].

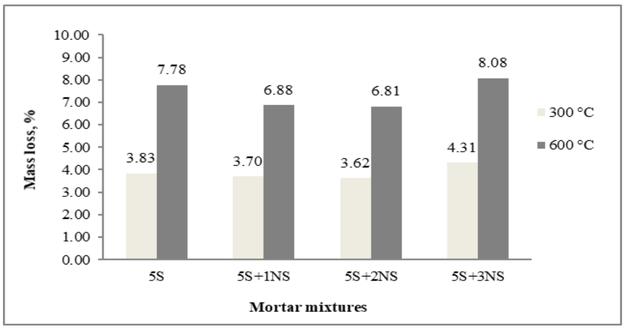

Fig. 1- Mass loss of the mortar specimens after exposed to 300 and 600 °C.

On 90th the ultrasonic pulse velocity values of the mortar mixtures kept at 20 °C as the control temperature are shown in Figure 2. The ultrasonic pulse velocity values of all mortar mixtures are greater than 4.5 km /s. This describes that the quality of specimens are as "excellent" according to Table 5. The highest ultrasonic pulse velocity belongs to the 5S+2NS sample with a value of 5.0 km/s. The effect of using NS on the ultrasonic pulse velocity is presented in Fig. 2 which indicated that the effect of adding 1% and 2% dosages of NS increase the ultrasonic pulse velocity more than 5S mix with 3,37% and 5,26% for 90th days. The ultrasonic pulse velocity of a homogeneous solid can be easily related to its physical and mechanical properties [43]. İt is known that the nano-materials plays a vital role in filler effect [14].

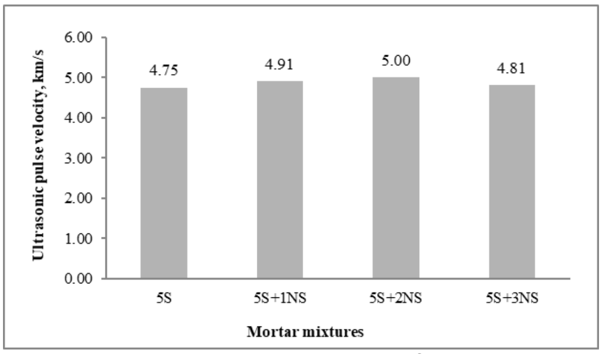

Fig. 2- Ultrasonic pulse velocity values of mortar mixtures

The rates of change from the initial to elevated temperature and cooling and the ultrasonic pulse velocity rate readings in the mortar specimens were calculated and presented in Figure 3. At 300 and 600 °C and after self-cooling in the oven, the ultrasonic pulse velocity values of the mortar mixtures decreased. $Ca(OH)_2$ in the cement may be transformed into CaO by changing at 600 °C. As a result of this change, a porous structure was formed in the specimens Also, it is a well-known fact that the thermal coefficients of cement paste and aggregates are different from each other. Therefore, shrinkage occurs in cement paste while expansion occurs in aggregates [44, 45]. The mortar mixture 5S+2NS least affected by both high temperature values; the most affected mortar mixture is the 5S+3NS mix. Ultrasonic pulse velocity values decreased by more than 50% in all mortar types.

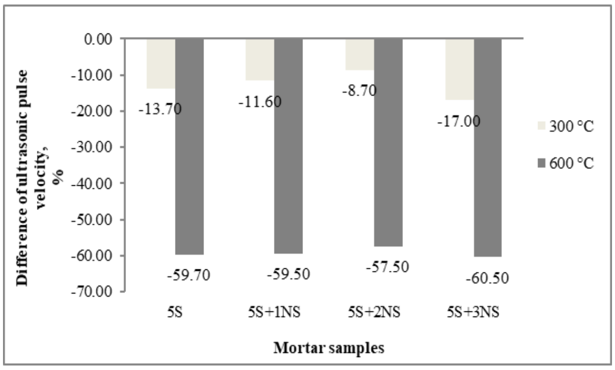

Fig. 3- The impact of elevated temperatures on the loss of ultrasonic pulse velocity.

Table 6

The compressive strength values of mortar mixtures after high temperature and cooling, MPa

Temperatures	Mortar mix			
	5S	5S+1NS	5S+2NS	5S+3NS
20 °C	46.40	48.10	48.40	43.20
300 °C	40.65	50.26	53.72	38.06
600 °C	29.37	29.37	30.68	24.58

Moreover, concrete is not particularly affected physically at temperatures below 300 °C. Previous studies reported drastic chemical and physical changes occurring in concrete at temperatures above 300 °C, which is consistent with the decrease in compressive strength and elastic modulus of concrete at high temperatures [46]. Because of this, temperatures below 300 °C haven't used in this study.

The effect of using NS on the compressive strength is presented in Fig. 4 which indicated that the effect of adding 1% and 2% dosages of NS increases the compressive strength more than 5S mix with 3.66% and 4.31% for 90 days. The use of NS can be assertively suggested to improve the structural characteristics and mechanical performance of cement mortars and concrete when compared to MS [47]. Table 6 shows the compressive strength values of mortar mixtures after high temperature and cooling. Therefore, 2% can be considered as the optimum mix amount of NS in this study. 2% NS substitution positively affected the compressive strength by compacting the structure and improving the bond between aggregate and cement matrix [48]. According to Ghafari et.al. this positive property can be mainly attributed to the size and large surface area, which has pozzolanic and filler effects on the cementitious matrix [49]. On the other hand, excessive dosage of NS would result in more serious agglomeration of nano particles, with decline in nano effect [50]. The use of nano-powders does not apparently accommodate good dispersion; and while sonication with or without dispersing agents improves dispersion, nanoparticles remain in large agglomerates [51]. Probably, 3% NS would negatively affect 90th compressive strength.

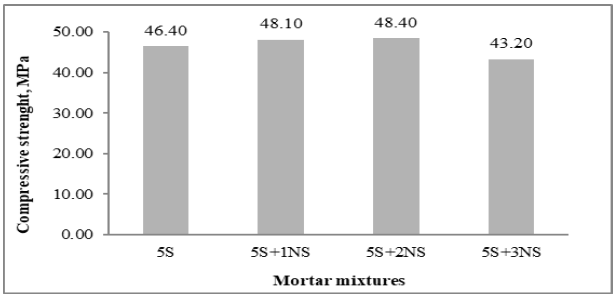

Fig. 4- Compressive strength values of the mortar mixtures.

Compressive strength values at 300 and 600 °C for the mortar mixtures and after self-cooling in the oven at 20 °C are compared with the compressive strengths of the mortars at the control temperature and the changes in the compressive strengths are calculated and the obtained values are given in Figure 5.

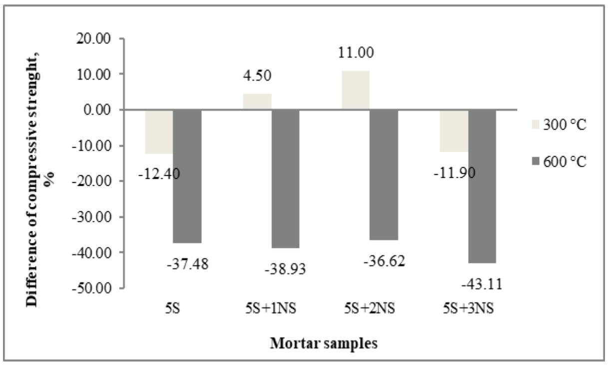

Fig. 5- The impact of elevated temperatures on the loss of compressive strength.

As can be seen in Figure 5, the increase in compressive strengths of the 5S+1NS and 5S+2NS mixtures at 300 ° C was 4.5% and 11%, respectively. After exposure to 300 ºC, 5S+1NS and 5S+2NS showed an improvement in compressive strength. This phenomenon has been observed by several authors [41, 42, 52, 53] and has sometimes been interpreted as a consequence of CSH re-hydration at 300 ºC, when water migrates and condenses within the colder areas of the sample. Such an effect is quite common even for cement-based materials and can be attributed to the stiffening of the gel and the increase in surface forces between the gel particles due to the release of adsorbed moisture [54].

Moreover, increase in compressive strength can be observed by the hydration of anhydrated pozzolan particles which are activated with temperature rise. Therefore, compressive strength of concrete keeps constant, or even slightly increases up to 300 °C [55, 56].

At 600 °C, all mortar mixtures showed a decrease in compressive strength. The corresponding loss in compressive strength at 600 °C is 37.38%, 38.93%, 36.62%, and 43.11% for mixes 5S, 5S+21S, 5S+2NS and 5S+3NS, respectively. In the temperature range of 400-800 °C both concrete lose most of their original strength, especially at temperatures above 600 °C due to the decomposition of the calcium silicate hydrate gel (C-S-H) that is the responsible for the mechanical strength of the cement [57].

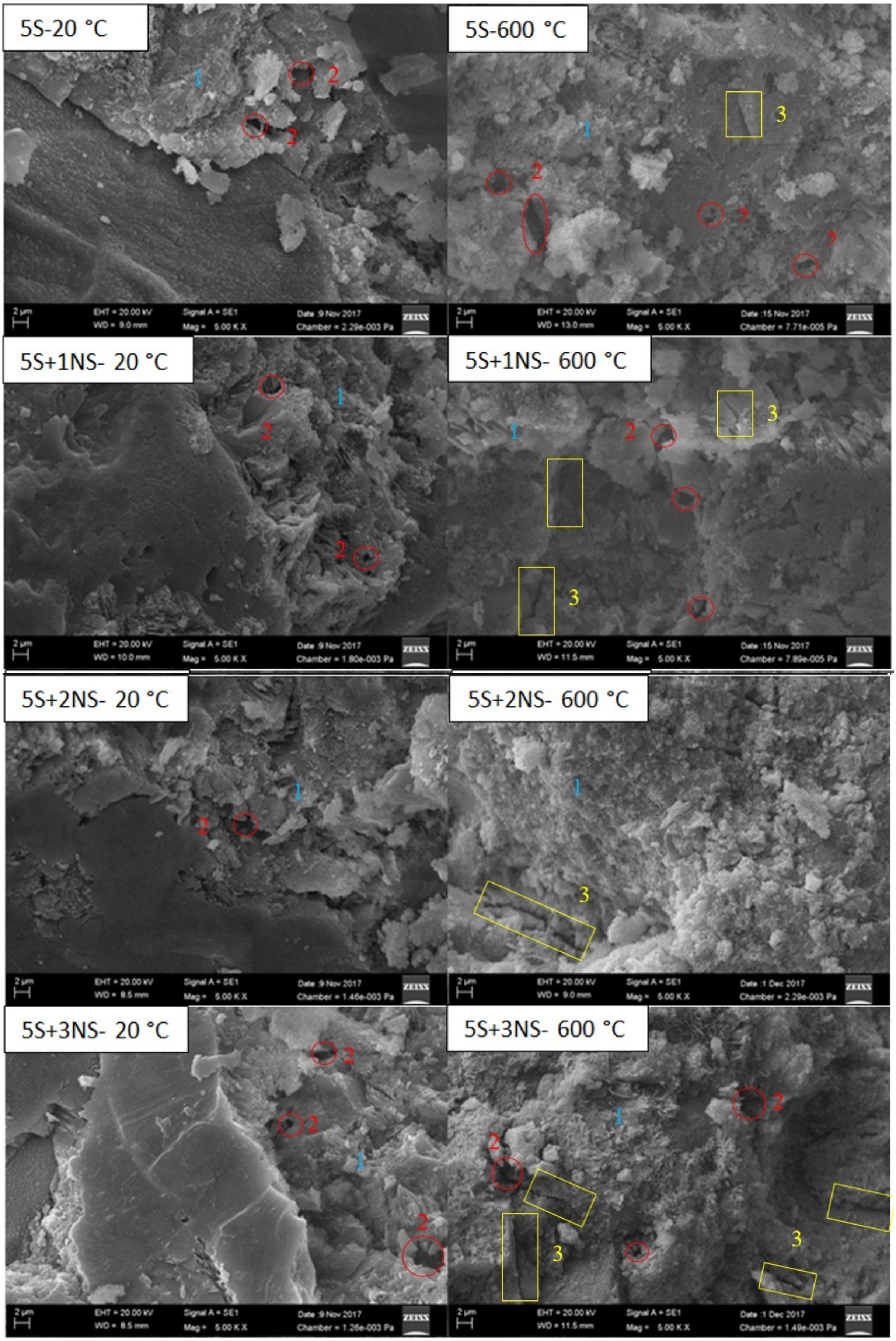

Fig. 6- SEM images of mortar specimens before exposed temperature and after exposed 600 °C (1: CSH, 2: Voids, 3: Cracks).

2% NS significantly improves the resistance of cement mortars added %5 MS to elevated temperatures, as a result of the retention of higher residual compressive strength. These phenomena are attributed to the improved amount and stability of the C-S-H phase, as well as the filling effect of NS.

In order to understand the microstructure of the cement mortars after the elevated temperature exposure, SEM was conducted on the specimen fragments. Specimens fragments without exposure and with the exposure temperature of 600 °C were examined by SEM shown in Figure 6.

According to SEM images of all cement mortars cured under laboratory conditions (20 °C), the C-S-H phase, which dominates the structure, has been observed. The amorphous gel formed the dominant phase in the whole structure, formed the bond between the hydration products and tried to fill the gaps. This phase tried to fill the gaps in the 5S mortar, but there were only a few gaps in the area of 1-2 microns in length. In addition, cracks were found in very small sizes. In addition, 5S+1NS and 5S+2NS cement mortars were found to have a more gapless and stable structure. However, 5S+3NS coded cement mortar can reach up to 3 micrometers and it is determined that there is more space structure than other samples. It is known that when the NS is not well dispersed, as in the case for excessive NS content, the aggregated NS can create weak zone in form of voids and lowered the compressive strength [58]. The SEM image of 5S+3NS supports the compressive strength value.

According to the SEM images of all the cement mortars exposed to the temperature of 600 °C, the C-S-H phase, which dominates the structure, has been observed. All cement mortars exposed to a temperature of 600 °C were found to have a more intense space and long cracks were observed according to the cement mortars exposed at 20 °C. This structure of the gap and cracks, the dominant phase of the bond structure between the thought to be weakened. Matrix of cement mortars are damaged at 600 ºC.

4. Conclusions

The study aimed to evaluate the effect of nanosilica on the performance of cement mortars containing micro silica at elevated temperatures. From the results obtained, it can be concluded that:

- The mass loss of specimens at both types of cement mortars was decreased as the temperature was increased. The percentage of mass reduction in the 5S+2NS mortar was lower than other mortar specimens.
- The ultrasonic pulse velocity decreased at all mortar mixtures at 300 and 600 ° C. At 600 ° C the mortar mixture with the highest rate of ultrasound transit was the 5S+3NS samples with 60.50%. The decrease in the rate of ultrasonic pulse velocity by 57.50% is attributed to the 5S+2NS samples.
- At 300 °C, the compressive strength values of 5S+1NS and 5S+2NS mortar mixture increase.
- The compressive strength values of the cement mortars were decreased as the temperature was increased 300 to 600 °C. By increasing the temperature up to 600 °C, the compressive strength of the 5S, 5S+1NS, 5S+2NS and 5S+3NS was decreased by 37.48%, 38.93%, 36.62% and 43.11% respectively. The reduction in compressive strength may be due to the occurrence of micro- and macro-cracks in mortars because of high temperatures.
- The highest compressive strength at 600 °C was observed in 5S+2NS mortar specimens with 30.68 MPa.

The results demonstrated that is the combined effects 5 wt% micro silica+2 wt% nano silica improve residual compressive strength in cement mortars exposed to elevated temperatures.

In future work, it is thought that it is useful to apply elevated temperature applications to the concrete specimens made of micro silica and nano silica and to apply different cooling conditions (air cooling/water spray cooling).

ACKNOWLEDGEMENTS

This study was supported via 2016.F94.02.02 numbered Project by Artvin Coruh University Scientific Research Projects Fund. The author would like thank.

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