

Contents list available at **IJND**
International Journal of Nano Dimension

Journal homepage: www.IJND.ir

Compressive strength of Portland cement pastes and mortars containing Cu-Zn nano-ferrite

ABSTRACT

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Received: 29 October 2011

Accepted: 14 December 2011

This study is concerned with the synthesizing of Cu-Zn nano-ferrite particles in the laboratory applying simple techniques. The morphology of the synthesized nano-particles was analyzed using Transmission Electron Microscopy (TEM), and the minerals were identified using X-ray diffraction (XRD). The nano material was used to replace 1- 4 percent by weight of Portland cement in cement pastes and mortars. The microstructure and compressive strength of the composites were then evaluated. The microstructure was characterized by means of Scanning Electron Microscope (SEM) analysis and the compressive strength was evaluated at 7 and 28 days. While some specimens were tested at normal temperature, other specimens were tested after 2-hour exposure at elevated temperatures of 200°C and 400°C. The results showed that it was possible to synthesize nano-particles having an average size of 12 nanometers. Also, the test results revealed that the optimum content of the nano-ferrite was 1% to produce the maximum increase in the compressive strength. Adding the optimum dose of nano-particles helped to produce a denser microstructure. The paste and mortar specimens showed general tendency to lose strength after exposure to the elevated temperatures.

Keywords: Nano; Ferrite; Cement paste; Microstructure; Strength; Temperature.

INTRODUCTION

Nanotechnology is based on synthesizing nano-particles with specified characteristics to be used in different applications related to the industry, medicine, agriculture, etc. A nano-particle is a microscopic particle whose size is measured in nano-meters (nm). It is defined as a particle with at least one dimension less than 200-nm. During the last ten years, not only the nano-products were utilized to improve the quality and durability of products, but also new approaches were developed to handle traditional problems.

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Fortunately, some research work was directed to improve the strength and durability performance of concrete as one of the most widely used construction materials. The properties of concrete have been well studied at macro or structural level without fully understanding the properties of cementitious mechanisms at the micro level. Better understanding of the structure and behavior of concrete at micro/nano-scale is a basis for improving the concrete properties and performance. Research has been conducted to study the hydration process, alkali-silicate reaction (ASR), and fly ash reactivity applying nanotechnology [1, 2].

Most of the published studies on the use of nano-particles in cement and concrete have utilized nano-oxides, especially SiO_2 and Fe_2O_3 [3–8]. The early work of Li showed that nano- SiO_2 could significantly increase the early compressive strength of concrete containing large amounts of fly ash [3], and improve pore size distribution by filling the pores between large fly ash and cement particles at nano-scale. Also, the slurry of amorphous nano-silica was used to improve the segregation resistance of self-compacting concrete. Another nano-size oxide of interest in construction is titanium oxide (TiO_2) [9, 10]. Nano-titanium and Nano- SiO_2 were also used to increase the abrasion resistance of concrete for pavement [11]. Recently, it has been reported that the TiO_2 nano-particles accelerated the rate of hydration and increased the degree of hydration [12]. Its photo-catalytic characteristics have been mainly used to remove organic pollutants from surfaces directly exposed to ultraviolet radiations such as road pavements and cement-based facade finishing products [13]. Synthetic C-S-H has also been used as seeding agent during the hydration of cement phases [14]. The possibility of controlling the nature of hydration products through nucleation seeding of different types of preformed C-S-H was demonstrated. Nano- $\text{Ca}(\text{OH})_2$ particles have been prepared and their thermal properties were characterized to study the anomalous behaviors of $\text{Ca}(\text{OH})_2$ in cement paste [15]. Other nano to sub-micro inorganic particles, such as zeolite, have been added to cement systems to improve the overall microstructure [16]. This study is concerned with the replacement of Portland cement with Cu-ZN nano-ferrite. The authors thought that such a ceramic material can be attractive for the use in

Portland cement composites to improve the mechanical properties. Since the discovery of ferrites, about 80 years ago, much more basic and applied researches have been carried out to explore their potentials. During the 1940's and 1950's, ferrites were systematized in the academic field, and today ferrite theory is well organized. From the 1950's, ferrites established a significant position in the industry and now ferrites are one of the most essential materials in the industry of electronics. One of these ferrites, Cu-Zn ferrite was synthesized in 1936 as ferrite cores for antennas and intermediate frequency transformers in radios for the first time in Japan. Currently, CU-Zn ferrite has many applications, including rotary transformers, noise filters, and multilayer ferrite chip components with regard to the magnetic semiconductor properties of ferrites [17].

Synthesizing nano-ferrite (NF) particles using simple procedures and commercially available materials is a challenge. The aim of this research was to study the influence of using Cu-Zn ferrite nano-particles as Portland cement replacement in cement pastes and mortars. The nano-particles were synthesized in the laboratory using a simple chemical process. The morphology and mineral composition of the synthesized particles were identified and the microstructure of the cement pastes was studied for different replacement ratios. The compressive strength of the cement composites was evaluated at 7 and 28-day age at room temperature. It was also assumed that the concrete incorporating NF can be very attractive for shielding applications in nuclear power plants with regard to the extreme fineness and the relatively high specific weight of the NF particles. Knowing that such concrete is subjected to aggressive environment of radiation and elevated temperatures, some paste and mortar specimens were tested after 2-hour exposure to elevated temperatures of 200°C and 400°C at 28-day age to explore possible strength degradation.

EXPERIMENTAL

Materials

$\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (Cu-Zn ferrite) nano-particles were prepared using a common co-precipitation technique from CuSO_4 , ZnSO_4 , and $\text{Fe}_2(\text{SO}_4)_3$ according to the following equation:

$0.5\text{CuSO}_4 + 0.5\text{ZnSO}_4 + \text{Fe}_2(\text{SO}_4)_3 + 8\text{NaOH} \rightarrow 0.5\text{Cu}(\text{OH})_2 + 0.5\text{Zn}(\text{OH})_2 + 2\text{Fe}(\text{OH})_3 + 4\text{Na}_2(\text{SO}_4) \rightarrow \text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4 + \text{sodium sulfate solution}.$

The sulfates were mixed in the required stoichiometric ratios in de-ionized water. Sodium hydroxide (NaOH) solution was then added dropwise, while stirring, until the measured pH value was 12. The mixture was continually stirred at 700 rpm for two hours while being heated at 80°C. A dark color was observed due to the formation of the ferrite particles. The nano-particles were allowed to settle and the mixture was washed several times until the measured pH value was about 7.5 due to the removal of the sodium sulfate solution. The powder sample was then allowed to dry at room temperature. The specific weight of the synthesized NF had a specific gravity of 5.32 with a brownish- red color.

The X-ray diffraction (XRD) data was collected using K_α radiation. Approximately 200 mg of powder was transferred to a glass XRD sample holder. This sample holder was then placed inside an X'Pert Graphics X-Ray Powder Diffractometer. Figure 1 shows the X-ray diffraction pattern of Cu-Zn nano-ferrite, which clearly shows the single phase of a spinel structure with typical cubic and octahedral crystals of NF. The mean particle size of around 12-nm was also calculated from the peak (311) of the X-ray diffractogram employing Scherrer's formula [18]. Transmission Electronic Microscopic (TEM) analysis was carried out using a microscope type JEOL-JEM-1230 providing x60000 magnification.

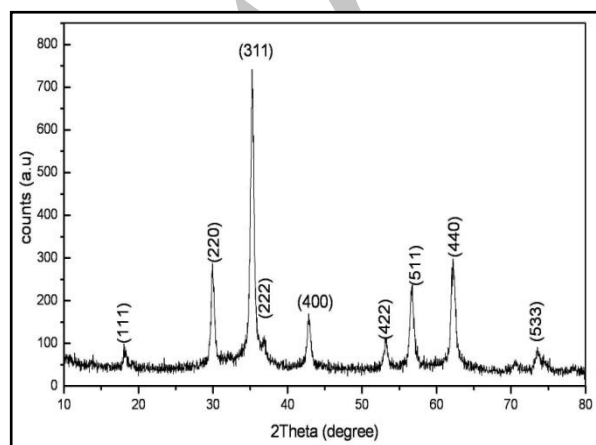


Fig. 1. XRD patterns of the $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$

Figure 2a and 2b show the morphologies of the obtained Cu-Zn ferrite nano-particles. It can be observed that TEM micrographs reveal that the obtained ferrite particles are almost uniform with an average size of about 13-nm which is very close to that being calculated using Scherrer's formula.

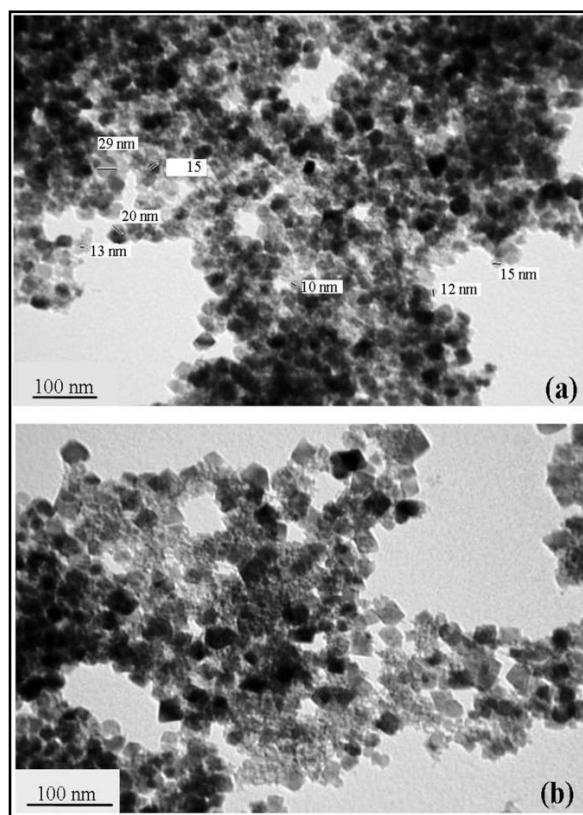


Fig. 2. TEM analysis of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$

The cement used was Portland cement (CEMI 52.5N) conforming to BS EN 197-1:2000. The chemical composition of the Portland cement is given in Table 1. Natural siliceous sand was used in preparing the mortar specimen. The sand passing the 850- μm sieve and retained on the 600- μm sieve was used. The sand had a specific gravity of 2.63 and an absorption ratio of 0.85 percent. A novel high range water reducer (HRWR) of modified polycarboxylates was used. This admixture is a turbid aqueous solution with a specific gravity of 1.08 and conforms to ASTM C494 (types F and G).

Table 1. Chemical composition of ordinary Portland cement

Component	% by mass
SiO ₂	19.8
Al ₂ O ₃	4.9
Fe ₂ O ₃	3.30
Cao	64.00
Mgo	1.00
SO ₃	3.30
Cao	2.00
Na ₂ O	0.68
Loss of Ignition	2.00

Mixing proportions and preparation of test specimens

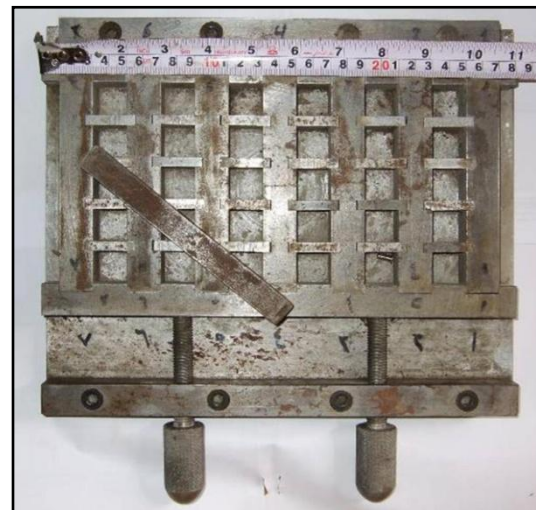
To prevent condensation of the nano-ferrite (NF) particles, water was added to the NF powder and the NF suspension was stored in a glass bottle. The bottle had to be thoroughly shaken to ensure the homogeneity of the suspension before the application. The amount of the suspension used in a specified mix contained the required weight of the NF and the water was a part of the total mixing water. Therefore, the NF-water suspension was prepared by adding 16 gm of NF to 100 gm of water to ensure that the water added from the suspension is no more than that required in the mix.

Five cement paste and five cement mortar mixes were proportioned including a control mix without NF and four mixes with NF replacing 1- 4 percent by weight of cement. In the following text, a specimen denoted Pi or Mi is a paste or a mortar specimen containing (i)% of NF replacing cement. In all paste mixes the water/ powder ratio was taken 0.25. In all mortar mixes, the water/powder ratio was 0.40, the sand content was three times the powder content by weight and the HRWR dosage was 2.0 percent.

The paste specimens were 20-mm cubes. To prepare the paste specimens, the NF suspension and the additional water, if any, were added to the cement and all components were mixed using an electrically driven mechanical mixer of an epicyclic type for at least three minutes. The paste was directly cast into the mold and compacted on a vibrating table. The steel mold was specially

manufactured to ensure easy stripping and allowed casting 30 test specimens at a time as can be seen in Figure 3.

The paste mixes had a stiff-plastic consistency. The cement pastes containing NF were noticed to be sticky due to the extreme fineness of the NF particles. However, those mixes tended to be less workable due to the cubic and octahedral shape of the NF crystals and needed prolonged mixing time to become consistent. Therefore, a HRWR was used in the mortar mixes to improve the workability of the mixes. The mortar specimens were 70-mm cubes. To prepare the mortar specimens, the NF suspension and additional water, if any, were mixed with cement and the sieved sand was added gradually to the mixer. Mixing continued for 3 minutes after which the HRWR was added and mixing continued for additional 5 minutes. All mixes yielded a stiff consistency. The steel molds were filled with two successive layers of mortar. Each layer was compacted using a 500-gm steel bar with a square cross section of 20-mm side length. The paste and mortar test specimens were allowed to cure under wet cloth for 24-hour after which the specimens were removed from the molds and water cured until tested.

**Fig. 3.** Steel mold and tamper for casting the paste specimens**Testing Procedure**

Paste and mortar specimens tested in room temperature were tested within two minutes after being removed from the water curing tank. The specimens tested after exposure to the elevated

temperatures of 200°C and 400°C were first dried for 24-hour in an electrical furnace at 105°C. This procedure was adopted to reduce the tendency of the specimens to spall at elevated temperatures due to vapor pressure. This behavior is common in cement composites containing fine powders due to low permeability [19]. The heated specimens were allowed to cool gradually at room temperature for 24-hour before being tested in compression.

The compression test was performed utilizing a 500 kN universal testing machine which allowed control over the loading range to provide the maximum loading accuracy. To facilitate centering of the test specimen, a special compression test rig with a spherically seated upper bearing was used to test the 20-mm paste cubes, Figure 4. The compression test results for paste and mortar specimens are reported in Table 2. Each test result represented the average strength of three tested specimens.



Fig. 4. Testing of the 20-mm paste cubes

Table 2. Compressive strength (MPa) at different ages and temperatures

NF %	Paste				Mortar			
	7-day	28-day	T 200	T 400	7-day	28-day	T 200	T 400
0	67.4	88.4	67.8	56.9	31.2	40.6	41.0	39.2
1	89.2	126.3	136.6	116.2	34.7	60.0	62.4	51.7
2	98.1	110.1	96.0	70.5	30.1	53.4	51.2	48.9
3	59.2	110.3	78.8	63.6	29.8	49.9	50.3	47.5
4	55.0	97.0	79.7	65.5	--	--	--	--

RESULTS AND DISCUSSION

The results of the compression tests performed at 7 and 28-day at room and elevated temperatures are reported in Table 2. Figure 5a and 5b show the 7 and 28-day compressive strength for cement paste and mortar specimens tested at room temperature. It can be seen that the 7-day compressive strength of the paste specimens gradually increased up to 2% NF content. It is also observed that the paste compressive strength dramatically decreased when the NF content was increased to 3% and 4% and the compressive strength was lower than that of the control specimen without NF. Similar trend was observed for the mortar specimens. While, the paste compressive strength was increased by a maximum of 45% at 2% NF content, the mortar compressive strength was increased by a maximum of 11% at 1% NF content.

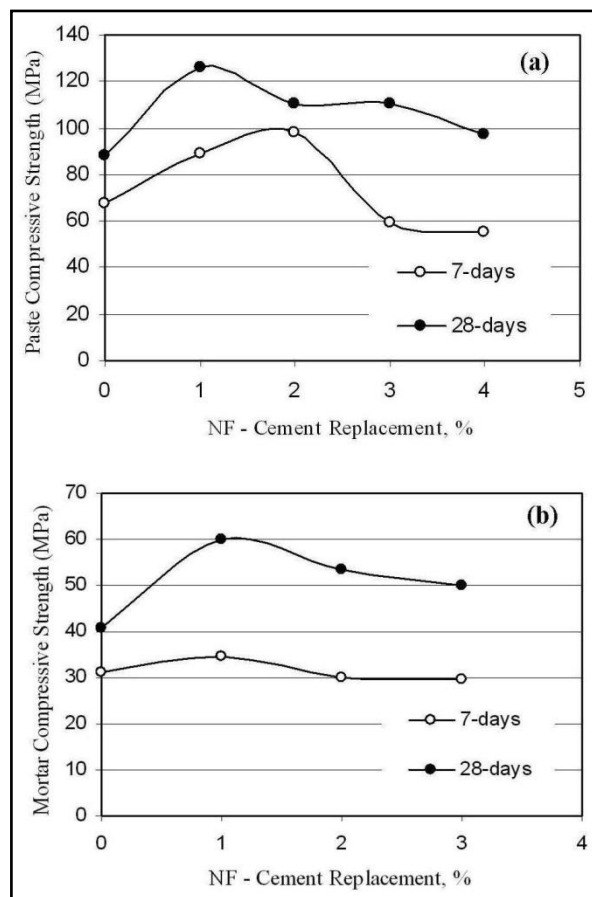


Fig. 5. Compressive strength of cement paste and mortar at room temperature

Figure 5a and 5b show that the 28-day compressive strength of all paste and mortar specimens containing NF was higher than that of the control mix without NF. The maximum increase in the 28-day compressive strength was 43% and 48% for paste and mortar specimens, respectively at 1% NF content. The reduction of the early strength of cement pastes with NF contents higher than 2% of cement weight could be attributed to the negative effects on the early hydration process. On the other hand, mortar mixes M3 and M4 showed minor reductions in the 7-day compressive strength, while the 28-day compressive strength was significantly higher than that of the control mix. This trend was attributed to the use of the HRWR in mixes M1, M2 and M3 that ensured better dispersion of cement particles. The relationship between compressive strength and the NF content showed that the peak strength corresponded to 1% of NF. It should also be noted that the NF-cement combination was not robust as the compressive strength changed considerably within a very limited range for NF content.

Figure 6a depicts the influence of elevated temperatures on the 28-day compressive strength of the paste test specimens after 2-hour exposure at 200°C and 400°C. The paste specimens showed a general tendency to lose compressive strength due to the heat treatment. The only exception was recorded for mix P1 at 200°C that demonstrated strength gain compared to the control mix P0. The initial trend of developing peak strength at 1% NF was the same after heat treatment. The average loss of compressive strength was 20% after heating at 200°C and 30% after heating at 400°C. A similar trend can be observed in Figure 6b showing the variation in the compressive strength of the mortar specimens after heat exposure. After heating at 200°C, the specimens either acquired or lost some strength within 4 percent compared to their counterpart specimens tested at room temperature. On the other hand, all specimens lost strength after heating at 400°C and the maximum strength loss was 14% in mix M1. In all cases, mix M1 achieved the peak strength among all other mixes at different temperatures.

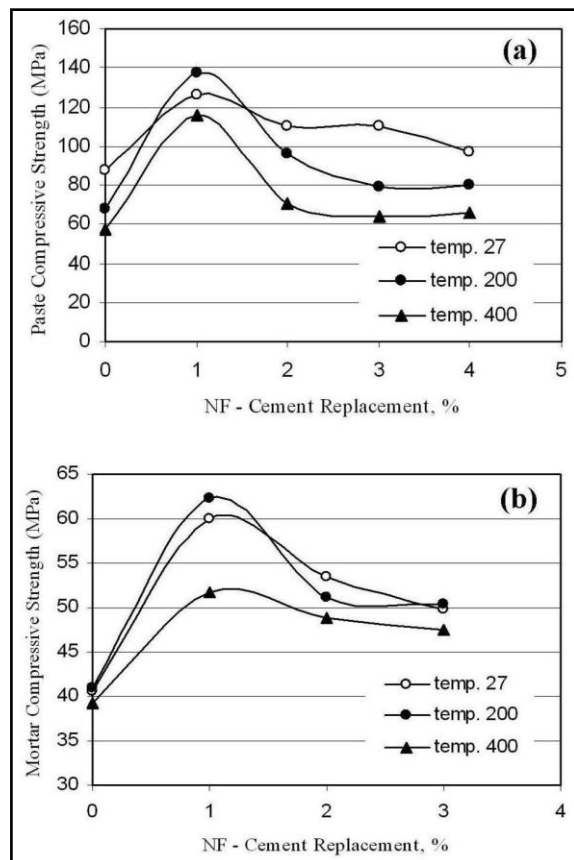


Fig. 6. Compressive strength of cement paste and mortar at room temperature and after 2-hour exposure at elevated temperatures

The related studies showed that hardened cement paste plays a key role in the deterioration of concrete subjected to elevated temperatures [20]. It was found that the loss in structural quality of concrete due to temperature rise is influenced by the changes induced in the basic processes of cement hydration and hardening of the binding system of the cement paste of concrete [21]. Actually, some research results revealed a decrease of the compressive strength of cement pastes after exposure to the elevated temperatures up to 200°C, while other research works gave opposite results. The variation in the compressive strength after heating depends mainly on the mixing water/cement ratio, the permeability and the length of the cooling phase after heating [22, 23].

Different explanations were presented for the variation in the compressive strength after heat exposure. One explanation is that the spatial distribution of Ca(OH)_2 crystals becomes more compact with a rise in temperature. It means

smaller crystals occurring in a unit volume of the cement paste. This is related to the decreased solubility of Ca(OH)_2 at higher temperatures. By contrast, hydrated cement pastes at lower temperature are more likely to contain large masses of Ca(OH)_2 , with a well-developed morphology. A rise in temperature affects the cement paste pore structure by reducing the specific surface of hydration products. Cement paste exposed to high-temperature attack is more heterogeneous in microstructure and coarser in pore structure. The decrease in volume of the hydrate phase and Ca(OH)_2 , and the coarsening of the pore structure of concrete are the main factors influencing the decrease of the compressive strength after heat exposure[24]. Another explanation is that at high temperatures, especially above 100°C, the thermal effect might cause internal stress and thus micro and macro-cracks are generated due to the heterogeneous volume dilation of ingredients and the buildup of vapor in the pores. Therefore, at higher temperature, especially above 400°C, the observed decrease in compressive strength of cement composites containing fine materials may be due to internal thermal stress around pores which generate micro-cracks [25].

Microstructure study

SEM examinations were performed to verify the mechanism predicted by the compressive strength tests. The addition of NF particles was found to influence the hydration behavior and influence the microstructure of hardened cement paste. SEM examinations were carried out for the control paste mix P0 and mix P1 containing 1% NF which achieved the maximum increase in the compressive strength. Figure 7 illustrates the SEM micrographs of cement paste. Figure 7a shows a massive structure of well crystalline C-S-H and well developed sheet-like CH crystals with an elongated shape for the control specimen P0 at room temperature. It can also be seen that the large crystals of C-S-H were surrounded by many needle-like micro-cracks. Figure 7b shows a crack-free amorphous microcrystalline C-S-H structure with dense spread of small CH crystals for mix P1 at room temperature. Such a dense crack-free structure and small size of CH crystals can be related to a regulated hydration process at early ages slowing down the generation of heat associated with the hydration process. After

exposure to an elevated temperature of 400°C, the micrograph of mix P1 shows a coarser structure with extremely smaller CH crystals explaining the reduction of compressive strength.

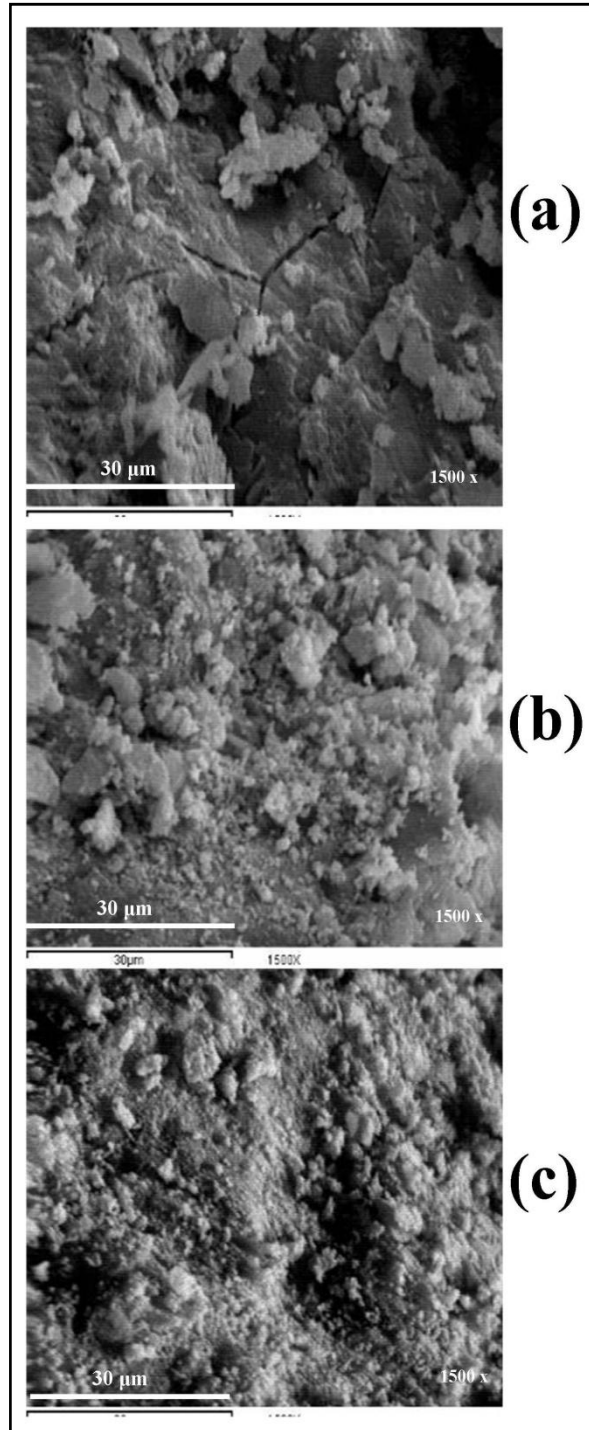


Fig. 7. SEM micrographs for paste mixes P0 (a), P1 (b) and P1 at 400°C(c)

CONCLUSIONS

This work described experiments on the synthesizing of Cu-Zn nano-ferrite and its application as Portland cement replacement. Portland cement paste and mortar mixes were proportioned applying different contents of nano-ferrite. The influence of the added extremely fine material on the microstructure and compressive strength of the cement composites at normal and elevated temperatures was studied. The following conclusions could be drawn based on the available test results:

1. XDR patterns and TEM analysis demonstrated that it was possible to synthesize Cu-Zn nano-ferrite with an average particle size of 12 nano-meters; applying a simple laboratory procedure.
2. Cement pastes containing nano-ferrite needed prolonged mixing time to become consistent and tended to be less workable due to the cubic and octahedral shape of the nano-ferrite crystals. The use of a superplasticizer was necessary in the mortar mixes to improve the workability.
3. The optimum dose of nano-ferrite was one percent of cement by weight. The 28-day compressive strength of cement paste and mortar was increased by an average of 45 percent when nano-ferrite replaced 1% of cement by weight.
4. The compression test results demonstrated lack of robustness of the cement composites containing nano-ferrite as cement replacement. Slight variations in the dose of nano-ferrite around the optimum dose caused significant strength reduction.
5. Paste specimens showed general tendency to lose compressive strength after 2-hour exposure at elevated temperatures of 200°C and 400°C.
6. Mortar specimens showed general tendency to lose compressive strength after 2-hour exposure at 400°C. On the other hand, the compressive strength was not significantly influenced after heating at 200°C.
7. Further studies are needed to explore the hydration process and strength development of cement composites containing Cu-Zn nano-ferrite at early ages.

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Cite this article as: M. A. Safan *et al.*: Compressive strength of Portland cement pastes and mortars containing Cu-Zn nano-ferrite.
Int. J. Nano Dimens. 3(2): 91-100, Autumn 2012