Structural Behavior of Nano Composite Materials ($Cu_{0.5}Ni_{0.5}Fe_2O_3$)

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***Abstract*—The intention of this work is to examine synthesizing of Copper-Nickel ferrites nanoparticles using suitable local materials and by applying simple laboratory operations. Output was analyzed to make sure it conformed to the required using X-ray diffraction (XRD) and X-ray Fluorescence (XRF). The Transmission Electron Microscopy (TEM) used to determine the shape of the resulting nanoparticles. This nanoparticles was used as an addition in the cement mortar as a replacement percentage of cement weight. The specimens of ordinary Portland cement mortar used contain, sand: cement ratio of 2:1 and water: cement ratio was 0.35. Percentage 0.07% cement wt of high range water reducer. Also, 15% cement wt was silica fume. The compressive strength was evaluated at age of 7 and 28 days to determine the optimum ratio of nanoparticles which found 0.055% of cement wt. Then models of structural elements were casted to study the behavior of the composites. Also, the microstructure was characterized by means of Scanning Electron Microscope (SEM) analysis.**

***Index Terms*—ferrite, nanoparticles, nano, TEM, SEM, cement mortar, structural behavior, nano composite**

I. Introduction

Due to progressive reaches to develop new composite materials. It is our motives to produce innovative composite materials could be used in developing and developed countries alike. The finished product is a passive machine. In the same vein, nanotechnology is not a new science and it is not a new technology either. It is rather an extension of the sciences and technologies that have already been in development for many years. The size of the particles is the critical factor. At the nanoscale (anything from one hundred or more down to a few nanometres, or 10-9m) material properties are altered from that of larger scales. Another important aspect is that, as particles become nano-sized, the proportion of atoms on the surface increases relative to those inside and this leads to novel properties. It is these "nano-effects", however, that ultimately determine all the properties that we are familiar with at our "macro-scale" and this is where the power of nanotechnology comes in – if we can manipulate elements at the nanoscale we can affect the macro-properties and produce significantly new materials and processes [1].

The construction industry was the only industry to identify nanotechnology as a promising emerging technology in the UK Delphi survey in the early 1990s [2]. The importance of nanotechnology was also highlighted in foresight reports of Swedish and UK construction [3]-[4]. Furthermore, ready mix concrete and concrete products were identified as among the top 40 industrial sectors likely to be influenced by nanotechnology in 10-15 years [5]. However, construction has lagged behind other industrial sectors where nanotechnology R&D has attracted significant interest and investment from large industrial corporations and venture capitalists. Recognizing the huge potential and importance of nanotechnology to the construction industry, the European Commission in late 2002 approved funding for the Growth Project GMA1-2002-72160 "NANOCONEX" - Towards the setting up of a Network of Excellence in Nanotechnology in Construction.

Since the discovery of ferrites, about 80 years ago, much basic and applied research has 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, as radio and television sets spread, ferrites established a significant position in the industry, and now ferrites are one of the most essential materials in the electronics industry [6].

Ferrites constitute a special branch of ferrimagnetics. The term ferrite denotes a group of iron oxides, which have the general formula MO. Fe_2O_3, where, M is a divalent metal ion such as Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Mg^{2+} or Cd^{2+}.The typical ferrite is magnetite, Fe3O4 (on FeO.Fe_2O_3), which has been a well known magnetic oxide since ancient time. The ferrites were developed into commercially important materials chiefly during the year 1933-1945 by Sonek [7] and his

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associates at the Philips Research Laboratories in Holland. In a classical paper published in 1948, Neel [8] provided the theoretical key to an understanding of the ferrites.

There are many types of nanoparticles or nano-sized materials produced and applied in the cement-based materials matrix include: silica nano-particles (nano-SiO_2) [9], nano-iron oxide (nano-Fe_2O_3), nano-alumina (nano-Al_2O_3), nano-clay, nano titanium oxide (nano-TiO_2), nano-calcium carbonate (nano $CaCO_3$), and nano-cement. A nano-particle in the structure of the cement-based materials is not used only in terms of nanoparticle reaction. Other considerations should be studied to make a decision considering performance, lower prices, and better accessibility. Accordingly, the silica nano-particles, whether in terms of performance or of price and accessibility, are the most appropriate nano-particles used in the concrete industry. Nano-particles are used to improve the mechanical properties and durability of cement based products according to their different behaviours. Nano-particles improve the performance of cement-based materials matrix according to possession of one or more of the following categories [10].

- To increase the production of calcium - silicate - hydrate (CSH) gel due to pozzolanic reaction and reduce amounts of $Ca(OH)_2$ crystals;
- To prevent the excessive growth of crystals in the matrix and control crystallization;
- Micro and nano-filling;
- Development of hydration reaction.

The second type of nano-materials applied in the structure of cement-based materials is nano-chemical additive. These materials improve the rheological performance of concrete products and contribute major support in order to produce specific concretes. Nano viscosity modifying agents [11], nano super plasticizers [12], and nano-polymers are some examples of these materials [13]. Finally, the third group of nano-materials is nano fibers used in the matrix of concrete materials. According to the types of fibers in the structure of cement-based materials, carbon nano-fibers (CNF) [14], carbon nanotubes (CNT) [15] and nanocellulose fibers can be indicated [16]. Nano-fibers improve the mechanical characteristics, create higher impact capacity, and reduce cracks in concrete specimens. These materials increase tensile and bending strengths by creating a bridge between micro cracks in the matrix of cement-based materials and also acting as nucleating sites for the hydration products [17].

However, if these nano-particles are not being dispersed appropriately and agglomerate in the matrix of cement-based materials, their performance in the matrix will be reduced. As a result, high range water reducer supperplasticizers should assist nano-particles, and also usage of low dosage nano-particle contents would aid the fabrication process [18]. Moreover, using lower amounts of these types of materials are preferred due to reducing the costs. The other notable point is the increase in shrinkage while the nano-particles are added [19]. This issue can be effective in serviceability of the structure. Low dosage application of nano-particles can prevent this defect in an acceptable manner; however, mostly we have to utilize the shrinkage reducing agents.

The intention of this work is to examine synthesizing of Copper-Nickel ferrites ($Cu_{0.5}Ni_{0.5}Fe_2O_3$) nanoparticles using suitable local materials and by applying simple laboratory operations. These nanoparticles were used as an addition in the cement mortar as a replacement percentage of cement weight. The compressive strength was evaluated at ages of 7 and 28 days to determine the optimum ratio of nanoparticles. These models of structural elements were cast to study the behavior of the composites and to investigate other properties such as static properties.

II. Experimental

A. Materials

$Cu_{0.5}Ni_{0.5}Fe_2O_3$ or (Cu-Ni ferrite) naonoparticles were prepared from $CuSO_4$, $NiSO_4$ and $Fe_2(SO_4)_3$ by applying simple laboratory operations according to the following equation: $0.5CuSO_4 + 0.5NiSO_4 + Fe_2(SO_4)_3 + 8NaOH \rightarrow 0.5Cu(OH)_2 + 0.5Ni(OH)_2 + 2Fe(OH)_3 + 4Na_2(SO_4) \rightarrow Cu_{0.5}Ni_{0.5}Fe_2O_3$ + sodium sulfate solution.

The sulfates were mixed in the required stoichometric ratios in de-ionized water. Sodium hydroxide (NaOH) solution was then added drop wise, while stirring, until the measured pH value was 12. The mixture was continually stirred at 700rpm for two hours while being heated at 80 ℃. A dark color was observed due to the formation of the ferrite particles. The nanoparticles were allowed to settle and the mixture was washed several times until the measured pH value was about 8.6 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 5.2 with a brownish- red color.

High-Resolution Transmission Electronic Microscopic (HRTEM) was carried out in Nano Tech Egypt Research Center using type JEOL - JEM-2100 of magnification up to 60000 was employed to test the $Cu_{0.5}Ni_{0.5}Fe_2O_3$ powders. Fig. 1 shows the morphologies of the obtained Cu-Ni ferrites nanoparticles.

The x-ray powder diffraction (XRD) data was collected using Kα 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. Fig. 2 shows the X-ray diffraction pattern of $Cu_{0.5}Ni_{0.5}Fe_2O_3$ nanoparticles, which clearly shows the single phase of a spinal structure. Also, The X-ray Fluorescence (XRF) analysis carried out to ensure that the chemical composition conformed to the required. Table I shows the X-ray Fluorescence analysis results.

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 II. Natural siliceous sand was used in preparing the mortar specimen. The sieve analysis for the sand used can be shown in Fig. 3. The sand had a specific gravity of 2.6 and an absorption ratio of 0.75 percent. A new product of high range water reducer (HRWR) of modified polycarboxylates was used as Percentage of cement wt.

To increase the strength of the mortar as possible, condensed silica fume was used as a partial replacement of the cement. It was delivered in a powder form with a light-gray color. It gives black slurry when it is mixed with mortar. The chemical composition of silica fume is given in Table III.

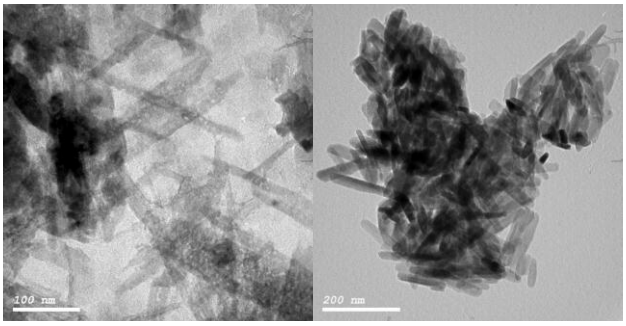

Figure 1. HRTEM photographs of $Cu_{0.5}Ni_{0.5}Fe_2O_3$

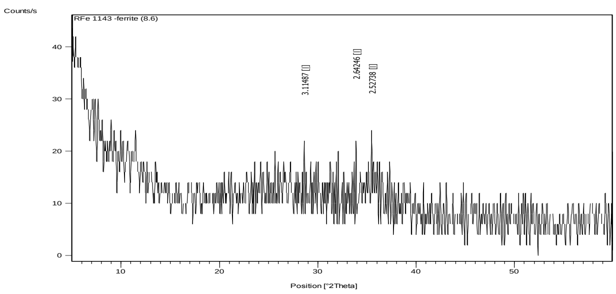

Figure 2. XRD patterns of the $Cu_{0.5}Ni_{0.5}Fe_2O_3$

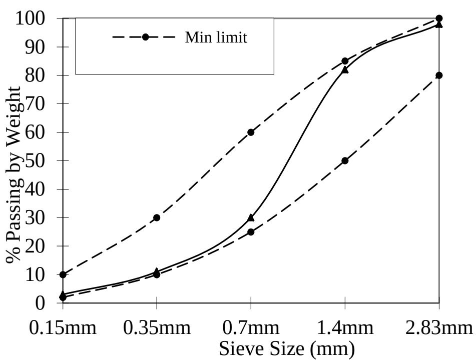

Figure 3. Sieve analysis of the sand used.

TABLE I. XRF ANALYSIS OF THE $CU_{0.5}NI_{0.5}FE_2O_3$

Component	% by volume
SiO_2	0.31
Al_2O_3	0.11
Fe_2O_3	43.29
CaO	0.74
Na_2O	0.21
SO_3	0.25
P_2O_5	2.99
NiO	25.08
CuO	26.67
ZnO	0.24
Total	99.89

TABLE II. CHEMICAL COMPOSITION OF ORDINARY PORTLAND CEMENT

Component	% by mass
SiO_2	19.8
Al_2O_3	4.9
Fe_2O_3	3.30
CaO	64.00
MgO	1.00
SO_3	3.30
CaO	2.00
Na_2O	0.68
Loss of Ignition	2.00

TABLE III. CHEMICAL COMPOSITION

Component	Weight %
Si O_2	92-94
Carbon	3-5
Fe_2 O_3	0.1-0.5
Ca O	0.1-0.15
AL_2 O_3	0.2-0.3
Mg O	0.1-0.2
Mn O	0.008
K_2 O	0.1
Na_2 O	0.1

B. Proportions of Specimens

The prepared Nano Cu-Ni Ferrite (NCNF) was a solution in the water after washing to insure the homogeneity of the suspension before the application. The amount of the suspension used in each mix contained the required weight of the NCNF and the water was a part of the total mixing water. In all mixes the water: cement ratio was 0.35 and sand: cement ratio was 2. Also, percentage of Fly ash replacement was 15% of cement wt. A new product of high range water reducer (HRWR) of modified polycarboxylates was used as Percentage of 0.07% cement wt.

Fourteen trial cement mortar mixes were prepared including a control mix with NCNF replacing different percentages by weight of cement from 0.0% to 3% but none equal division to determine the optimum percentage of NCNF. The mortar specimens were 70- mm cubes. The NCNF suspension and the additional water, if needed, were added to the cement, silica fume and sand. All components were mixed using an electrically driven mechanical mixer of an epicyclical type for at least three minutes to prepare the mortar specimens. The mortar was directly cast into the mold on three layers. Each layer was compacted well. All cubes were cured in the water until the test time. The mortar cubes tested after 7 and 28 days to determine the compressive strength.

The compressive strength was compared to the corresponding content of NCNF to determine the optimum content of NCNF.

Then, six columns (3X3X30) cm of mortar were cast. These elements were equally between control mix (without NCNF) and optimum ratio of NCNF mortar mix. All elements reinforced by mild steel with ϕ 2.4 mm only. All elements were cured by the water until the test time. The elements main sections and reinforcement were chosen as follow in Fig. 4.

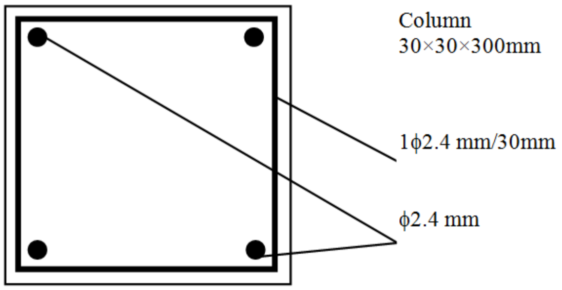

Figure 4. Columns and its reinforcement.

After 28 days, all columns were tested and the deformations were measured. The results were compared and drown in charts.

C. Testing Procedure

Mortar specimens were tested in a 500kN universal compression testing machine. The results were compared, and after three cycles of trial and error to determine the optimum dose of NCNF. The optimum NCNF ratio was 0.055% of cement weight. The results of 7 and 28 days were taken as average of 6 cubes for each point and are shown in Fig. 5. Then, after determining the optimum ratio of the NCNF and casting of columns, the columns were cured in the water during 28 days. Columns were tested by compression testing machine. To facilitate centering of the test specimen, a special compression test rig with a spherically seated upper bearing was used to test the columns.

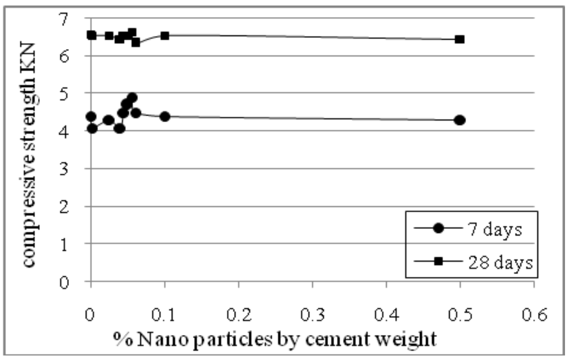

Figure 5. Results of mortar specimens

III. Results and Discussion

The results of the mortar specimens show that adding of NCNF is useful in acceleration the cement reactions and hydration in the range of 7 days by 15-20% at the ratios of 0.45-0.55% as shown in Fig. 5. Also when the ratio of NCNF increases above 0.5% the compressive strength is not affected in the same conditions. But after 28 days the compressive strength was improved by approximately 10% only.

The results of compression tests after the columns were cured by the water along 28 days are shown in Fig. 6a and Fig. 6b. Fig. 6a for the columns of mortar without NCNF (control) and it can be seen that the maximum axial load on the column was 24KN. The contraction behavior and horizontal displacements X and Z are shown in the figure. The figure shows the elasticity of the column which loaded by axial load in the three directions.

Fig. 6b shows the results of columns with NCNF content of 0.055% replacement of cement weight and its behavior under axial loads. The maximum axial load was 37KN which indicates that that there is more than 54% strength gain in the compressive strength comparing with control columns. The figure also shows the contraction behavior and horizontal displacements X and Z. the elasticity behavior of contraction shows the yield stage of the reinforcement. Adding of NCNF dissipate the cracks through the mortar witch improve the compressive strength of the tested columns by redistribute the stresses come from load in the column and make the crack path longer. The bacillary shape of the NCNF is the effective factor because it works as nano fibers.

The related studies showed that Nano-fibers improve the mechanical characteristics, create higher impact capacity, and reduce cracks in concrete specimens. These materials increase tensile and bending strengths by creating a bridge between micro cracks in the matrix of cement-based materials [20].

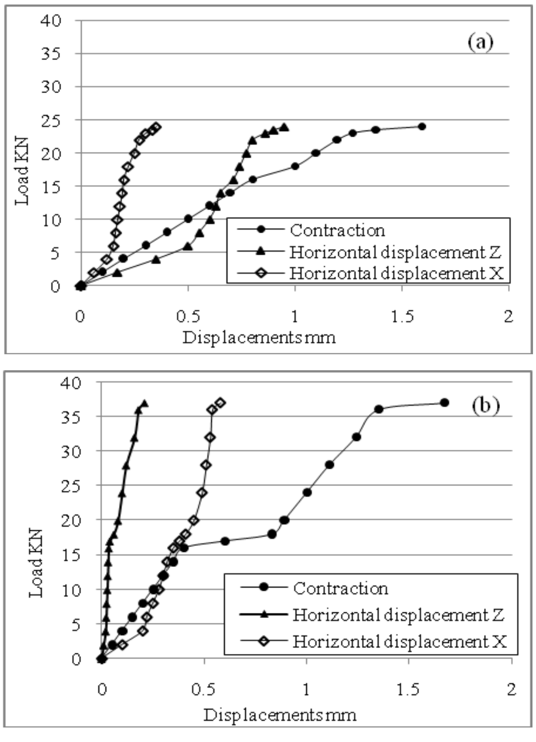

Figure 6. Results of tested columns

A. Microstructure Study

Scanning Electronic Microscope examination (SEM) was performed to verify the mechanism predicted by the compressive strength tests for columns. The addition of NF particles was found to influence the hydration behavior and influence the microstructure of hardened cement mortar. SEM examination was carried out for the mix with the optimum content of NCNF (0.055%) which achieved maximum increase in compressive strength. Fig. 7 shows the microstructure of the mortar in one of tested columns. It can be seen the randomly distributed fibers in the mortar and around the cracks. Also the fibers work as bridges between both sides of the cracks which lead to increase the compressive strength.

Figure 7. SEM micrograph for mortar include 0.055% NCNF

IV. CONCLUSIONS

This work is to examine synthesizing of Copper-Nickel ferrites ($Cu_{0.5}Ni_{0.5}Fe_2O_3$) nanoparticles was used as an addition in the Portland cement mortar as a replacement percentage of cement weight. The mortars were proportioned applying different ratios of NCNF. The influence of the added extremely fine material on the microstructure and compressive strength of the cement mortars and reinforced mortar columns model was studied. Based on available test results, the following conclusions can be drawn:

- The XRD, XRF and TEM analysis show that it possible to synthesize Copper-Nickel Ferrites in the nano size by applying simple laboratory operations.
- Cement pastes containing nano-ferrite needed prolonged mixing time to become consistent and tended to be less workable due to the bacillary shape of the nano Cu-Ni Ferrite. The use of a superplastizer was necessary in the mortar mixes to improve the workability.
- Adding the Cu-Ni Ferrite to the mortar mixes accelerate the hydration operation witch lead to gain early compressive strength higher than the normal mixes by 15-20 % at age of 7 days.
- The optimum ratio of nano Copper-Nickel Ferrites replacement by weight of the Portland cement was 0.055% due to compressive strength witch increased by 10% at 28 days.
- The results of tested columns that contain 0.055% of nano Copper-Nickel Ferrites showed good behavior in the axial load test by 54 % compared to those that do not contain.
- Bacillary shape of the Cu-Ni Ferrites increase tensile and bending strengths by working as bridges between micro cracks in the matrix of cement mortar.

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