



FABRICATION OF ADVANCED MORTAR FOR BUILDING APPLICATION

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ABSTRACT

In this research, cement mortar specimens were prepared with sand to cement (1:2.75) and (1, 2, and 3 wt%) of ceramic oxides (MgO, ZnO and Al₂O₃) at different particle sizes (11.8, 38.8, 109.8 nm), respectively. At curing time (7days), the (structural, thermal and physical) characteristics of the mortar specimens were investigated.

The dry density and porosity results show that the values of density increased by (23.4, 15.6, and 9.5%), and the values of porosity decreased by (76.2, 63.8, and 56.2%) for (MgO, ZnO, and Al₂O₃), respectively with respect to the reference mortar specimen. The thermal conductivity results show that the values of thermal conductivity decreased by (47.27, 40.66, and 51.16%) for (MgO, ZnO and Al₂O₃) respectively, with respect to the reference mortar specimen. Results of characterizations tests (XRD and optical microscope) showed that the reference mortar specimen has high roughness with large number of Ca(OH)₂ crystals, as well as the presence of pores, while after adding the nanopowders, it showed a decrease or disappearance of Ca(OH)₂ and produced homogeneous structure of calcium silicate hydrate compound (C-S-H), in addition to a large decrease in the pores and surface roughness. This explains the significant improvement in the properties of cement mortar mixed with nanopowders, hence these mortars were very appropriate for covering walls, buildings and other outdoor building applications.

Keywords: Nanopowder, MgO, ZnO, Al₂O₃, Mortar.

تصنيع مونه إسمنتية متقدمة لتطبيقات البناء

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الخلاصة

في هذا البحث تم إعداد نماذج المورتر بنسبة خلط الرمل إلى الاسمنت (1:2.75) مع (3,2,1 %) أكاسيد سيراميكية هي: اوكسيد المغنيسيوم (MgO) واوكسيد الزنك (ZnO) واوكسيد الألمنيوم (Al₂O₃) بحجوم حبيبية (109, 38.8, 11.8) نانومتر على التوالي. عند الزمن (7 يوم)، تم إجراء فحوصات مثل التوصيل الحراري والفحوصات التركيبية والحرارية والفيزيائية على نماذج المونه المحضرة. اظهرت نتائج فحوصات الكثافة والمسامية زيادة في قيم الكثافة بنسب (23.4, 15.6, 9.5%)، وقيم المسامية زادت بنسب (76.2, 63.8, 56.2%) عند اضافة لوكسيد المغنيسيوم واوكسيد الخارصين والالومينا على التوالي نسبة إلى نموذج المورتر المرجع. أما نتائج التوصيلية اظهرت إن هناك نقصان واضح في قيم التوصيلية الحرارية باستخدام تلك المواد بنسب (51.16, 40.66, 47.27 %) على التوالي، نسبة إلى نموذج المورتر المرجع. اظهرت نتائج الفحوصات التركيبية (حيود الاشعة السينية و المجهر البصري) إن لنموذج المونه المرجع خشونة عالية وكذلك وجود عدد كبير من بلورات هيدروكسيد الكالسيوم Ca(OH)₂ مع وجود مسامات، بينما بعد إضافة المساحيق النانوية، اظهرت نقصان أو غياب للبلورات الكبيرة ل Ca(OH)₂ وانتاج تركيب متجانس من سيليكات الكالسيوم المماهة (C-S-H)، إضافة إلى ذلك نقصان كبير في المسامات والخشونة السطحية. هذا ما يفسر التحسن الواضح في خواص المورتر الأسمنتي المخلوط بمساحيق نانوية، مما يجعلها مناسبة في تغليف الجدران، والبنائات، وتطبيقات البناء الخارجي الأخرى.

الكلمات المفتاحية: مسحوق ناتوي ، اوكسيد المغنيسيوم ، اوكسيد الزنك، اوكسيد الالمنيوم، المورتر.

INTRODUCTION

Nanotechnology is a very advanced technology that is interested with production of new materials and systems at the scale (1-100) nm, which can own extraordinary properties, not obtained in larger scales. This technology can be introduced in large number of applications in our life such as: construction industry, automotive parts, aircraft industry and others. Furthermore, the using of nanotechnology participates in the evolution that take place now, as will participate in the future [Buzea, *et.al*, 2007]. Concerning literature survey about the addition of nano materials with cement mortar, in **2004**, H. Li *et.al*, investigated the flexural strength and compressive strength of cement mortars mixed with nanoparticles of (SiO_2 and Fe_2O_3). From the experimental results, it was observed that the strengths (flexural and compressive) of the cement mortars blended with nanoparticles were better than that of reference cement mortar. The SEM studies showed [Xijun, *et.al*,1997],[Colston, *et.al*,2000] that the nanoparticles of Fe_2O_3 and SiO_2 mixed with cement mortar have filled the pores and contributed in hydration of cement through reducing of (CaOH_2).The mechanical behavior of the cement paste incorporated with nanoparticles was improved [Li, *et.al*, 2004]. In **2008**, X. He and X. Shi, studied the microstructure and permeability of chloride of portland cement mortar blended with nanomaterials at a ratio of (1%) as a weight percentage from cement. From the electromigration test for cement mortars, it was exhibited that the incorporation of nanoparticles (TiO_2 , Al_2O_3 , SiO_2 and Fe_2O_3) with particle sizes (5,10,30,and 30 nm) respectively, and nano clays (montmorillonite) has enhanced the resistance of chloride penetration of the cement mortar. Furthermore, the nanomaterials have reduced the ionic permeability of the cement mortar. Such enhancements were especially considerable when using nano- SiO_2 and nanoclays [He, *et.al*, 2008]. In **2011**, N. Yazdi *et. al.*, studied the tensile and compressive strengths of cement mortar incorporating with nanoparticles of Fe_2O_3 in the amounts of (1,3,and 5%) taken from the weight of cement. The experimental results exhibited that the samples of cement with (1 and 3%) of nano- Fe_2O_3 have better mechanical properties than the reference cement mortar. According to SEM studies on the fabricated samples,it was observed that the nanoparticles of Fe_2O_3 can fill the pores totally, reduce the $\text{Ca}(\text{OH})_2$ large crystals, and also increase the (C-S-H gel) and hence the structure of mortar containing nano- Fe_2O_3 was more uniform and denser than reference mortar. The mechanical properties of the cement mortar incorporating with nanoparticles of Fe_2O_3 are reduced, if the content of Fe_2O_3 nanoparticles exceeded (5%) by weight of cement [Yazdi, *et.al*, 2011]. In **2015**, E. Mohseni *et.al*, investigated the hardened properties of High Performance Concrete (HPC) combining with nano- TiO_2 and Fly Ash. Nano- TiO_2 in ratios of (1, 2 and 3wt%) and fly ash in ratio of (30 wt%) by weight of binder. Compressive strength of HPC samples incorporating with Fly ash and nano titania (NT) was obtained at (7 days) which represented an early age, and also at (28 days). The experimental results on concrete samples containing NT showed improvements in durability performance with increasing the content of NT. Furthermore, it was exhibited that the considerable improvements in the properties of concrete samples incorporating the substitution of cement with (1%) TiO_2 nanoparticles and (30%) Fly Ash [Mohseni, *et.al*, 2015]. The main aims for this work are the addition of nano ceramic oxides (MgO , ZnO , and Al_2O_3) to the cement mortar in different percentages by weight of cement. Finally, studying the impact of these additions on structural, thermal and some physical properties of mortar.

EXPERIMENTAL PART

Materials

Cement and Sand

Ordinary Portland cement, Type I, supplied from (TASLUJA-BAZIAN) factory is used through this study and the chosen cement agrees with Iraqi Specification No.5/1984. Tables (1) and (2) show the chemical and physical properties of the cement used in this research, respectively. **Karbala** natural sand, which was passed from sieve No.4 (4.75 mm), is used as fine aggregate and it is classified to the second zone according to the Iraqi Specification No.45/1984. The grading satisfies the Iraqi specification IQS 45/1984 and ASTM C33-03 as presented in Table (3), while the physical properties of natural sand are presented in Table (4).

Water

Ordinary tap water was used in this research, for mixing and curing of all cement mortar specimens.

Nano powders

Three types of nanopowders were used in this study. These nanopowders are (MgO, ZnO, and Al₂O₃) at particle sizes of (11.8, 38.8, 109.8) nm, respectively. The specifications of the used nanopowders are presented in Table (5).

Particle Size Analysis of the Powders

The particle size inspection for powders was conducted in the Nanotechnology and Advanced Research Center. The device model was Brookhaven NanoBrook 90 Plus USA. ISO 13321 & ISO 22412 were used for most nanoparticles, and colloidal-sized materials, in any non-absorbing liquid range: 2 nm to 6 μm, and scattering angle of 90°.

Preparation of Mixed Cement Mortar Specimens

Many cement mortar mixtures were made for testing. All the mixtures had a ratio of water to cement of (0.5) and the ratio of sand to cement of (1:2.75) according to ASTM Specifications (C305-12) [ASTM,1999]. Percentage of the nano powders were varied from (1, 2 and 3%) by weight of the cementations materials. Mortars with (MgO, ZnO, and Al₂O₃) nanopowders were compared to that of the reference mixture to evaluate the impact of dosage of nanomaterials at particle sizes of (11.8, 38.8, and 109.8) nm, respectively. **Mortars** were mixed at an ambient temperature of (28 °C). To prepare mortar mixtures, the solid materials (cement, sand, and nanopowders) were dry mixed first. The mixtures were then mixed with water using ultra-sonic mixer for ten minutes to form workable pastes, and finally the pastes were cast in lubricate disc molds with dimensions of (40mm) diameter and (5mm) thickness. At 24 hours, the specimens were removed from the molds, and the specimens were cured at a temperature around (28-30°C) till the testing. Structural, thermal, and some physical characteristics were inspected at (7) days.

Characterization Techniques

Optical microscope and X-ray diffractometer (XRD) characterization methods were used to observe the influence of nanopowders on cement mortars. XRD type (XRD-6000), X-ray tube of Cu (1.5406 Ao), V:(40 KV), C:(30 mA), and scan range (20-60 degree) was used. Stereomicroscope was used to exhibit microstructure of mortar specimens. Stereomicroscopes offer the observer an erect (upright and un-reversed) stereoscopic (3-D)

image, which is dissimilar from compound microscopes that view a (2-D) image and the magnification used was (20X).

Dry Density Test

Dry density can be concluded by using the procedure specified according to ASTM Specifications (C642-1997) [ASTM,1997]. It can be calculated from the equation:

$$\text{Dry density (gm/cm}^3\text{)} = W_1 / (W_1 - W_2) * \rho_w \quad (1)$$

Where: (ρ_w): the density of water is equivalent to 1 (gm/cm³).
(W_1):the average weight of dry specimen (gm). (W_2): the average weight of wet specimen (gm).

Density can be considered as an effective indicator of the uniformity of raw materials, mixing, batching, placing, molding, and testing. A considerable change in density signals means a change somewhere in the process [Feldman, et. al,1972].

Porosity Test

This test was performed in accordance with ASTM (C642-1997). The test procedure was similar to that of total absorption test, except that the third weight of specimen was computed by using a sensitive balance. The third weight represented the submerge weight of the specimen. Porosity can be calculated by using the following equation [ASTM,1997]:

$$\text{Porosity (\%)} = (W_2 - W_1) / (W_2 - W_3) * 100 \quad (2)$$

Where: (W_3): the average submerge weight of specimen(gm).

Thermal Conductivity Test

Thermal conductivity for the fabricated mortar specimens was measured by Lee's disc test, in which the conductivity of each specimen can be measured via Lee disk. After the heater is run, the discs of device are begin to heat until the equilibrium state in temperature between discs is reached. Specimens have dimensions of (d:40 mm and t:5 mm). The calculations are subjected to the following equations [Hathal, et.al, 2009].

$$K[(TB - TA) / ds] = e.[TA + 2/r(dA + [1/2] ds) TA + [1/2r]. ds TB] \quad (3)$$

$$H = I.V = \pi^2.e.r(TA + TC) + 2\pi.e.r(dA.TA + \frac{ds}{2}(TA + TB) + dB.TB + dC.TC) \quad (4)$$

where: H (A.V): average time of energy get in heating coil, e:quantity of heat lost in one second of the centimeter cube, d(mm):disk thickness, r(mm):disk radius, K(W/m.⁰C):thermal conductivity, TA,TB(⁰C): temperature of the disks(A and B) respectively, dA, dB, dC(mm):thickness of disks(A, B and C) respectively, V (V): Voltage, and I (A): Electric current.

RESULTS AND DISCUSSION

Particle Size Analysis of the Powders

The Fig. 1, 2, and 3 show the particle size distribution for powders (MgO, ZnO, and Al₂O₃) respectively. The particle size has narrow distribution, for which Table (6) presents the values of median diameters and the mean diameters for (MgO, ZnO, and Al₂O₃).

Optical Microscope

Fig. 4 illustrates the micro-structure for the reference mortar at the age of (7) days, it observes porous texture that is filled with large number of pores and CS, while CH crystals linked to C-S-H gel, and surrounded the sand particles, the hydration is not finished and the reference mortar has low mechanical properties. Fig. 5, 6, and 7 display mixed specimens with nano materials (MgO, ZnO and Al₂O₃) respectively, with percentage of (3%) and curing time of (7 days). The presented figures show that the fabricated specimens mixed with nano powders were denser and more organized than reference mortar, and they have very little number of Ca(OH)₂ crystals and small pores, unlike the reference mortar that contains large number of (CH crystals). Moreover, the mortars mixed with nano powders have a compact texture due to the absence or decrease of un-hydrated compounds and pores, which make the structure more homogeneous with better mechanical properties than the reference mortar specimen. This action was due to the nanopowders with high activity that can enhance the pozzolanic reaction to form more (CSH-gel) to give high mechanical properties in the early age [Quercia, et.al, 2014].

X-Ray Diffraction (XRD)

Fig. 8-a shows the XRD pattern of the cement mortar without nanopowders at curing time for (7 days), while Fig. 8-b, 8-c, and 8-d show the XRD patterns of the cement mortar mixed with (3% MgO), (3% ZnO), and (3% Al₂O₃), respectively. The XRD patterns of the (reference and mixed specimens with nanopowders) have an angular range of $2\theta = 20.0 - 80.0$ degrees. In cement mortar (after curing time) of the (reference and mixed specimens), the components of phases shown in the Fig. from 7-b to 7-d were:

- Portlandite: Ca(OH)₂, hexagonal crystallized, JCPDS (04-0733) (CH).
 - Tobermorite: Ca₅Si₆(O,OH,F)₁₈.5H₂O, orthorhombic crystallized, JCPDS (45-1480) (Tob).
 - Ettringite: Ca₆Al₂(SO₄)₃(OH)₁₂.26H₂O, hexagonal crystallized, JCPDS (41-1451) (CASH).
- Furthermore, the phases shown were:
- Calcium Silicate Hydrate: CaO.SiO₂.H₂O, poor crystallized, JCPDS (34-0002) (CSH).
 - Wollastonite: CaSiO₃, monoclinic crystallized, JCPDS (43-1460) (CS).
 - Magnesium Oxide: MgO, cubic crystallized, JCPDS(30-0794).
 - Zinc Oxide: ZnO, hexagonal crystallized, JCPDS (36-1451).
 - Aluminum Oxide(α): Al₂O₃, cubic crystallized, JCPDS(46-1212).

It was observed that the ettringite and portlandite are involved in all stages of hydration. In addition, the changes that happened in mineralogical components during the hydration processes were observed, where hydro-silicates and hydro-aluminates were present (portlandite, tobermorite and ettringite). After (7) days, the highest peaks correspond to portlandite (CH) and calcium silicate hydrated (C-S-H) gels [Elenal, et.al, 2011]. XRD characterization were done in order to investigate the activity of the incorporated nanopowders with cement mortar specimens after (7) days of hydration. From XRD patterns, it is clear that the (CH) peaks are nearly decreased with mixing by (MgO, ZnO, and Al₂O₃) nanopowders. It is therefore inferred from the figures below that nanopowders can react with the produced CH through the hydration. Hence, the reactivity of nanopowders is significantly high and has developed the microstructure of cementitious materials, thereby it has improved the mechanical properties of cementitious materials. CH compound has a sharp peak in the cement mortar acting as the pure hydration product, which is released from the hydration of cement. Obviously, peak intensity of CH is reduced due to the nanopowders as a cement replacement as shown in Figs. from (8-b) to (8-d), which showed the consumption of CH via pozzolanic reaction [Aly, et.al, 2012].

Dry Density Test

The densities of mixed mortar specimens with nano powders (MgO, ZnO, and Al₂O₃) with percentages of (1,2,and 3wt%) including the reference specimen of mortar are shown in Fig. 9. The comparative densities for the mixed mortar specimens with nano powders are higher than the reference mortar at all ratios. For instance, at (1wt%) of nanopowder, clearly an increase occurs in values of density especially for MgO and less for ZnO, and the lesser value for Al₂O₃ as compared with reference specimen. When the ratio of nanopowders increases, it observes a slightly reduction in value of density for all mixed specimens, but it remains higher than the reference specimen. This increasing in density for mortar mixed specimens can be explained, the very fine particles of nano powders enter the voids into the structure of cement mortar, thus, acting as fillers which improves the microstructure that lead to densification. Thus, the final structure is more denser than the ordinary cement mortar [Mehta, 1986].

Porosity Test

The porosities of mixed mortar specimens with nano powders of (MgO, ZnO, and Al₂O₃) with percentages of (1,2,and 3wt%) including the reference specimen of mortar are shown in Fig. 10. It is observed from this figure, that the values of porosity are decreased clearly for specimens at all ratios of adding the nano powders from the reference ordinary cement mortar. The porosity can be considered as opposite to density, hence, decreasing in values of porosities is due to the increase in densification. The nano powders contribute in pozzolanic reaction for cement mortar which leads to a decrease in CH and an increase of CSH-gel, and filling the pores between particles of cement and aggregate [Mehta, et. al.,2002].

Thermal Conductivity Test

Thermal conductivity of the prepared specimens was measured via Lees disk to identify the influence of nano materials on the cement mortar. Fig.11 shows the thermal conductivity of all cement mixed specimens including the reference mortar specimen. The cement specimens mixed with nano materials of (MgO, ZnO and Al₂O₃) respectively, with percentages of (1,2 and 3%) at a particle size about (12 nm) and curing time of (7 days). The comparison shows that the thermal conductivity for the mixed specimens is lower than the reference specimen, so the mixing decreases the thermal conductivity, especially for Alumina which has the lowest thermal conductivity, and the reduction in thermal conductivity was reached at a rate of (51.16%) from the reference mortar specimen, that may be due to low thermal properties among the others. Therefore, the thermal conductivity of reference specimen is higher than the mixed specimens. This decreasing in thermal conductivity of mortar with the existing of nano powders this agrees with [Zhang, et.al, 2012].

CONCLUSIONS

This study has conducted theoretical and experimental phases of the cement mortar mixing with nanopowders and reached the following conclusions:-

1. The thermal insulation of cement mortar incorporated with nanopowders were generally enhanced, in which the thermal insulation was higher than that of cement mortar reference specimen. The highest value obtained from mixed specimens with (3% Al₂O₃), reaching the increasing ratio of (50.16%), as compared with reference specimen.
2. The addition of nanopowders leads to a significant consumption of portlandite (CH) through pozzolanic reaction according to XRD inspection.

3. The thermal conductivity test reveals that the aluminum oxide nanopowder mixed with cement mortar has lower thermal conductivity at all ratios than that of zinc oxide and magnesium oxide nanopowders.
4. The physical properties (density and porosity) of mortars mixed with nanopowders were generally enhanced through the increasing of density and decreasing of porosity, for which the optimal percentage were obtained by mixing cement mortar sample with a (3%) MgO nanopowder.
5. It was found from this study that the increasing of addition of nanopowders until (3wt%) to cement mortar, leads to a large enhancement of thermal insulation, density, and decreases porosity.
6. In order to reach on the lowest value of thermal conductivity in this research, the quantity of nanopowders per ($1m^2$) and a thickness (0.5 cm) was (40 gm), at a price of (15\$).

Table (1) : Chemical composition of the cement used in this investigation.

Abbreviation of Oxide	By Weight (%)	Limits of Iraqi Specification No.5/1984
SiO ₂	17.75	-
CaO	63.37	-
MgO	2.35	≤5.0
Fe ₂ O ₃	4.28	-
Al ₂ O ₃	5.29	-
SO ₃	2.25	≤ 2.8
Loss on ignition	1.55	≤ 4.0
Insoluble residue	1.15	≤1.5
Lime saturation factor	0.90	0.66-1.02

Table (2):Physical properties of the cement used.

Physical property	Test Results	Limits of Iraqi Specification No.5/1984
Specific surface area (Blaine method), m ² /kg	321	≥ 230
Setting time (vicat's method) Initial, hour: minutes Final, hour: minutes	1:45 4:20	≥ 1 hr. ≤10.00 hr.
Compressive strength (70.7mm cube) (N/mm ²) 3days 7days	19.20 28.50	≥15 ≥23
Soundness (autoclave method) %	0.24	≤0.8

Table(3):The grading of sand used.

Sieve Size (mm)	Passing by weight (%)	Iraqi specification No.45/1984
4.75	100	90-100
2.36	92.22	85-100
1.18	85.37	75-100
0.60	68.25	60-79
0.30	27.53	12-40
0.15	9.11	0-10

Table (4):Physical properties of the sand used.

Properties	Result	Iraqi Specifications No.45/1984
Specific gravity	2.64	-
Absorption(%)	0.77	-
Dry loose- unit weight(kg/m ³)	1591	-
Sulfate content (%)	0.09	≤ 0.5
Material finer than 75µm sieve(%)	3.9	≤ 5

Table(5): Specifications of nanopowders used.

Nano powder	Assay(%)	Density (g/cm ³)	Melting point (°C)	Manufacturer
MgO	99.9	3.6	2800	Nanjing Nano-technology
ZnO	99.7	5.6	1980	
Al ₂ O ₃	99.9	3.8	2900	

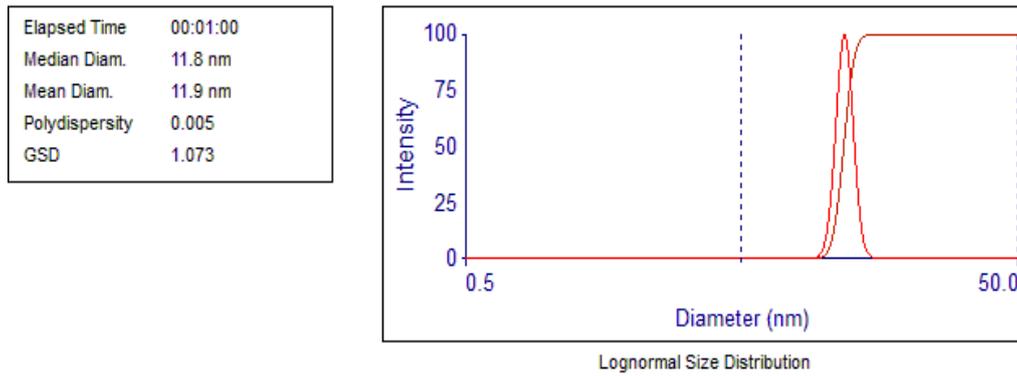


Fig.1: Particle size distribution of MgO powder.

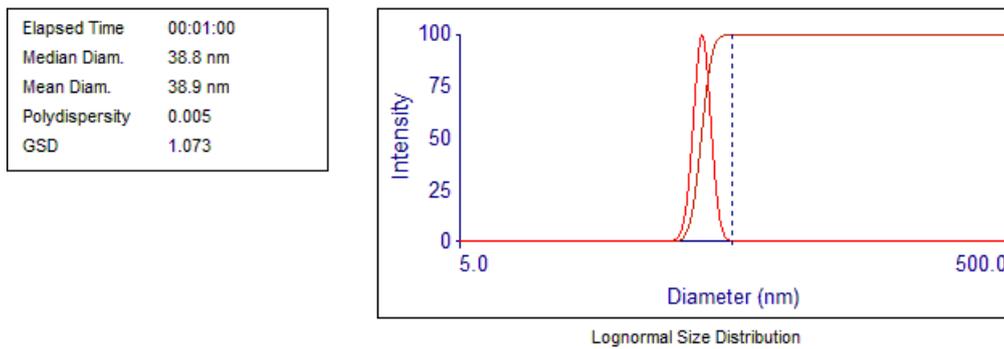


Fig.2: Particle size distribution of ZnO powder.

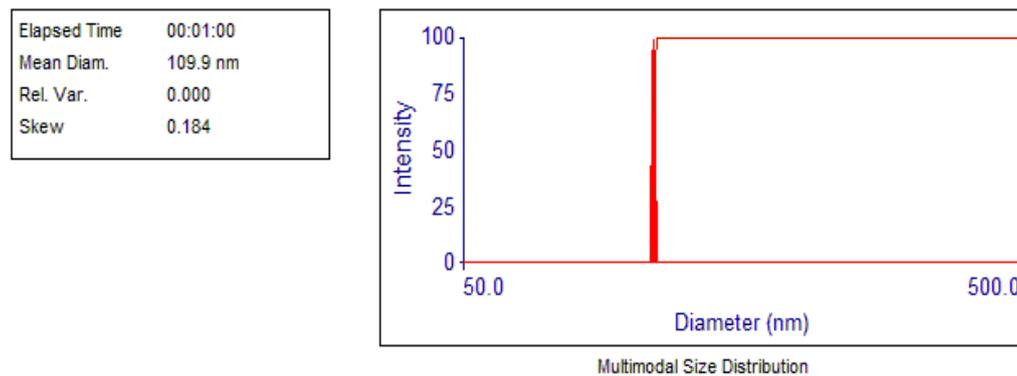


Fig.3: Particle size distribution of Al₂O₃ powder.

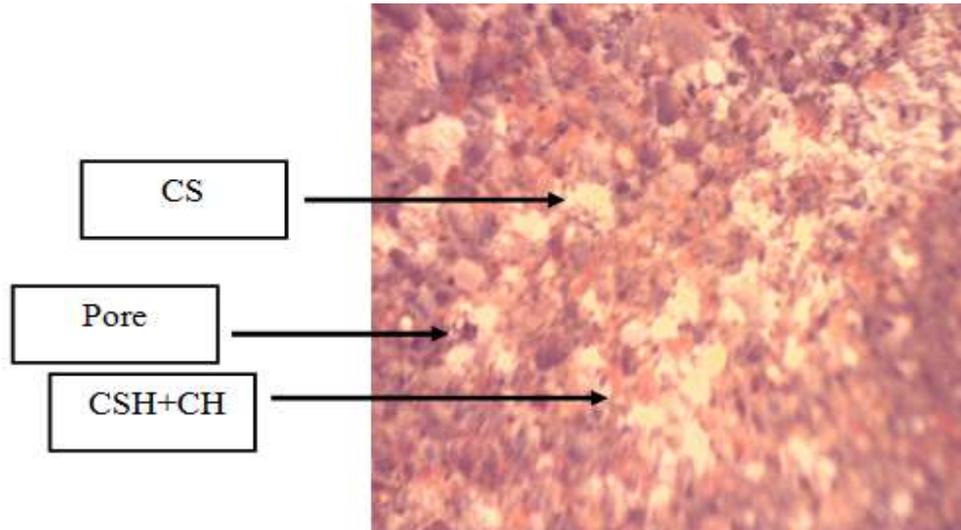


Fig.4: Microstructure of cement sample without nanoparticles.

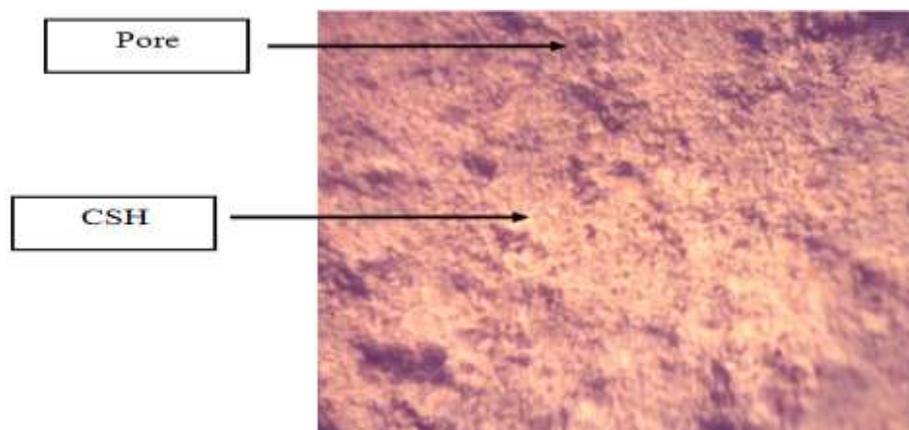


Fig. 5: Microstructure of mortar sample with MgO nanoparticles at (3wt%).

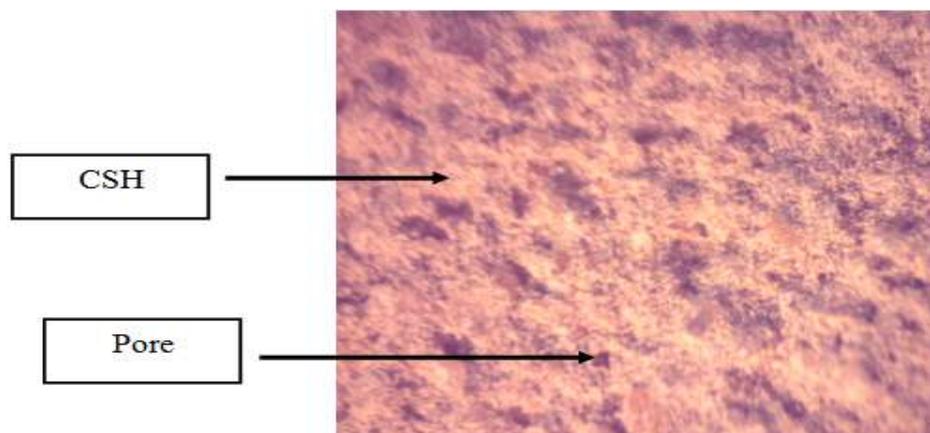


Fig. 6: Microstructure of mortar sample with ZnO nanoparticles at (3wt%).

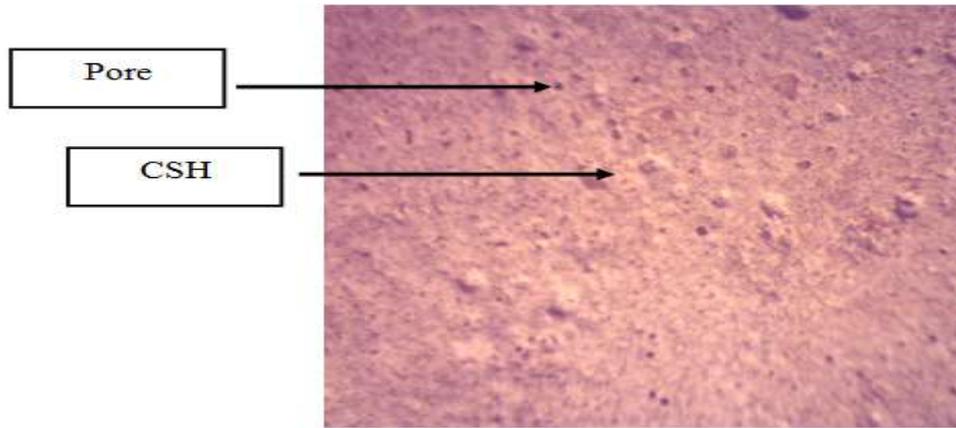


Fig. 7: Microstructure of mortar sample with Al₂O₃ nanoparticles at (3wt%).

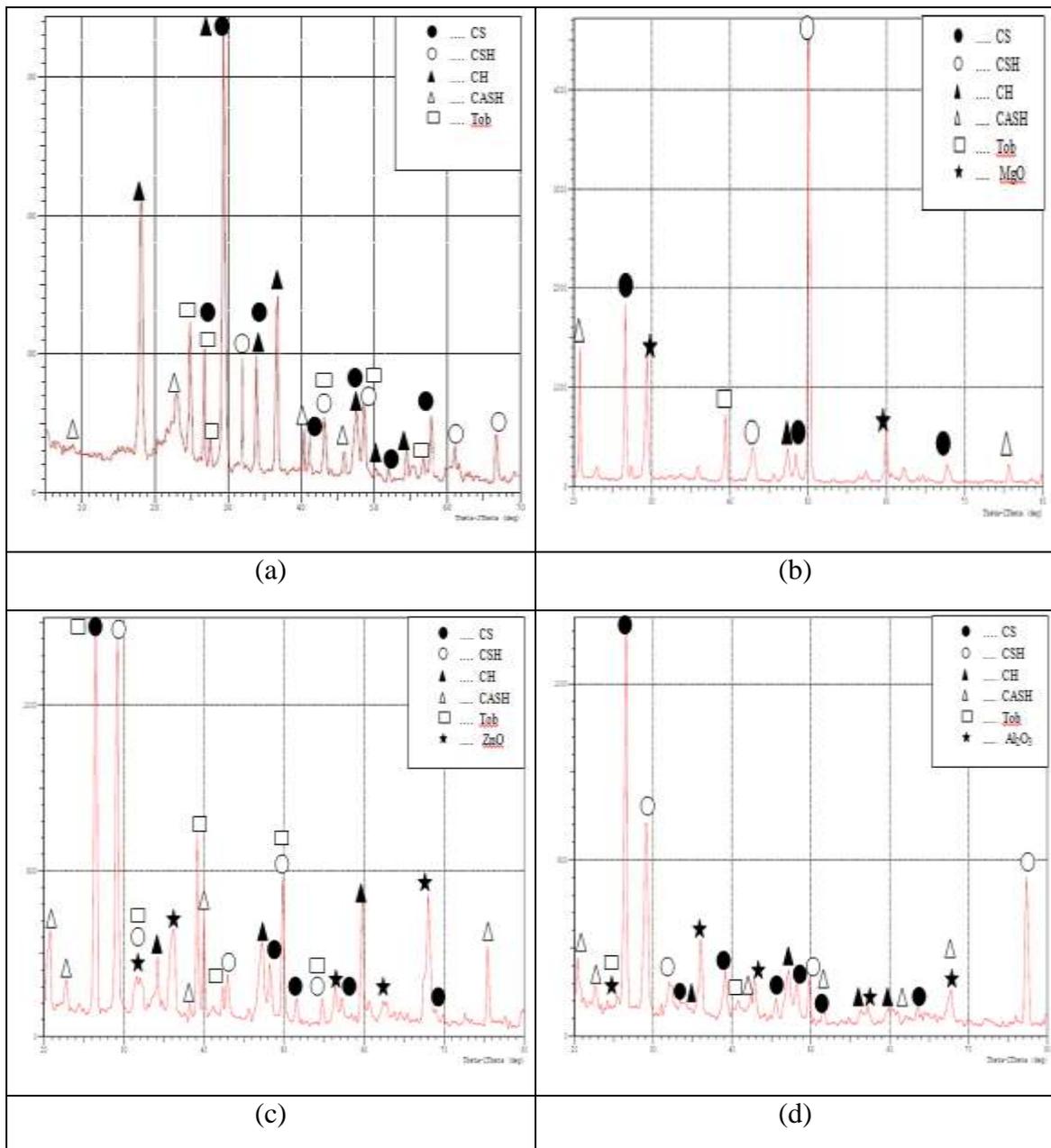


Fig. 8: XRD of cement mortar specimens: (a) without nanopowders. (b) with MgO nanopowder. (c) with ZnO nanopowder. (d) with Al₂O₃ nanopowder.

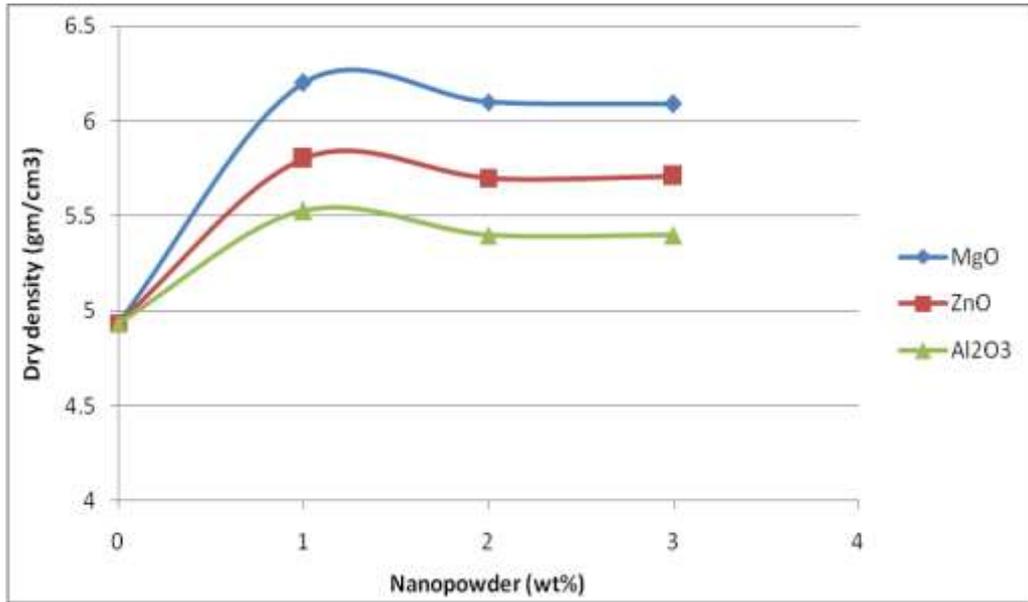


Fig. 9: Influence of nano powders on density for the prepared mortar samples.

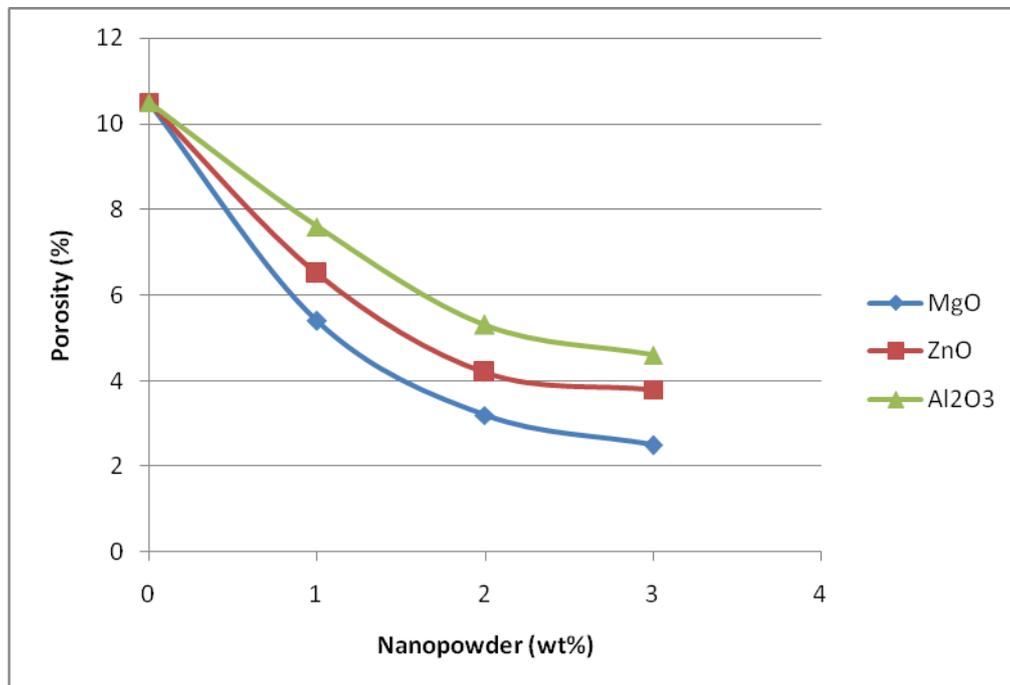


Fig. 10: Influence of nano powders on porosity for the prepared mortar samples.

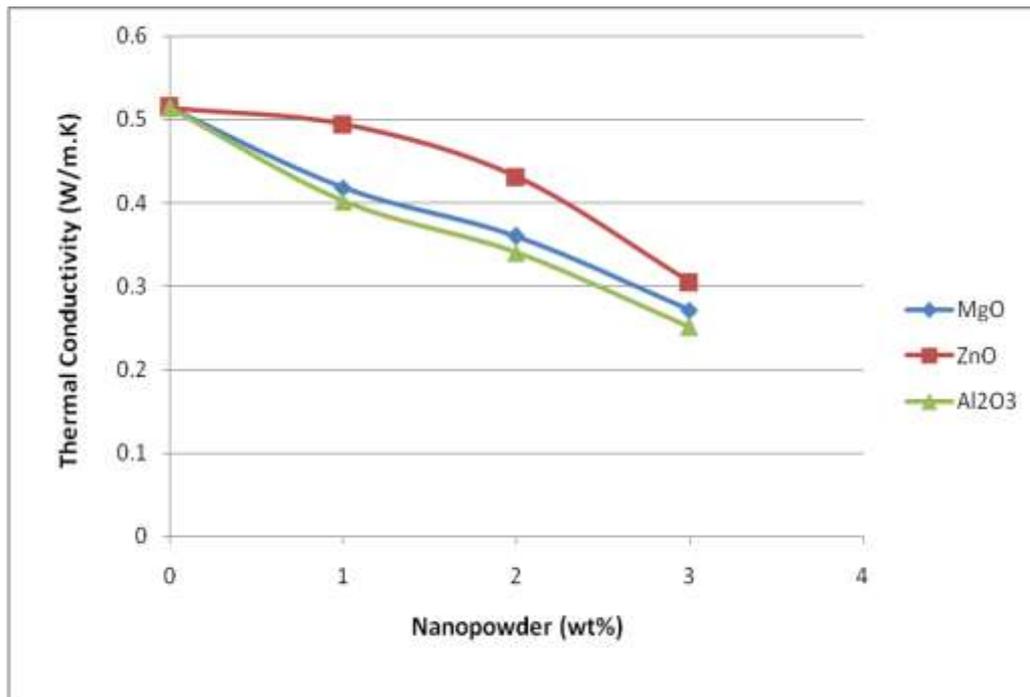


Fig. 11: Influence of nano powders on thermal conductivity for the prepared mortars.

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