Characterization of Nano Powder Incorporated for Building Applications

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Abstract

In this paper Nano powders were used a mono; copper oxide CuO and di metal oxides powder; titanium dioxide (TiO_2) in fabrication of mortar. Powder particle size about (67.8 nm) for TiO_2 and (55.7nm) for CuO with crystal structure tetragonal and monoclinic system respectively have been used. These Nano powders were used as additives to the mortar material (0.5, 2.5, 5, 7.5 wt %) in order to be used in construction application including covering building, and studies on these mortar. Investigation was done on the mortar including XRD, AFM, optical microscopy, physical and some mechanical properties, micro hardness and wear rate). Results shows that where there an increase in micro hardness and decrease in wear rate in the mortar with the addition of Nano powder. Also be reduced $Ca(OH)_2$ soft crystals and formation fine grains structure and homogeneous and the abolition of flaws or cracks. And the mortar with the Nano addition has been improved, which make the addition of Nano material beneficial and have a promising future in modern construction application.

Key Word: Nano powder, TiO_2, CuO, mortar, micro hardness, wear rate.

توصيف مسحوق نانو المدمج في تطبيقات البناء

الخلاصة

في هذا البحث مساحيق نانويه استخدمت. مساحيق أكاسيد أحاديه واوكسيد النحاس أكسيدCuO وثنائيه المعدن ثنائي أوكسيد التيتانيوم (TiO_2) في تصنيع المورتر(الملاط) . كانت الحجوم الحبيبية للمساحيق حوالي (67.8 nm ل TiO_2 و (55.7) nm لل CuO ,مع تراكيب بلورية رباعي الزوايا و أحادي الميل على التوالي قد استخدمت.وقد استخدمت هذه المساحيق النانويه كأضافه لماده المورتر (0.5،2.5، 5 ، 7.5٪ بالوزن) من وزن المورتر لأجل استخدامها في أغراض البناء بما في ذلك تغليف المبناني، و دراستها.تم اجراء الفحوصات على المورتر بما في ذلكXRD ، AFM ، المجهري الضوئي والخواص الفيزيائية و بعض الخواص الميكانيكية ،(فحص الصلاده المايكروية وفحص معدل البلى) .بينت النتائج أن حيث هناك زيادة في الصلادة الدقيقة وانخفاض في معدل البلى في المورتر مع إضافة مسحوق النانو. كما أن انخفاض تكون بلورات هدروكسيد الكالسيوم $Ca(OH)_2$ وتشكيل حبيبات الدقيقة بلورات ناعمة هيكل ومتجانس وإلغاء عيوب أو الشقوق. والهاون مع إضافة نانو قد تحسنت، والتي تجعل من إضافة مواد نانو مفيدة ولها مستقبل واعد في أغراض البناء الحد.

INTRODUCTION

Nanotechnology is a highly developed technology with great potential to produce new materials with exceptional characteristics and to fabricate new and enhanced products for a wide variety of applications. Numerous nano-centered applications have a great share in the global market; these products include electronics, Construction, health care, sporting goods, and automotive parts. The products fabricated by nanotechnology products are expected to represent a tremendous blooming in the near future with pharmaceutical and healthcare, chemicals development, construction, and many others [1].The fabricated nanomaterial's must be flexible in many aspect such as being used in for renewed clean energy, complicated drug delivery systems, bio-monitoring apparatus, and ultra-quick computing architectures.

Nanotechnology, the manipulation of matter at the Nano scale plays a key role in this matter where it provides answers for current construction related problems, Thus the potential for energy conservation and reduction of resource consumption, waste, toxicity and carbon emissions towards more sustainable "Green Practices" is significant [2]. Mortar properties and efficiency can be enhanced by adding nanomaterial into it, Nano powders can greatly improve the properties for mortar such as enhance mortar compressive strength and fill the porosity leading to denser mortar thus reducing water absorption due to the filling of mortar pores an example is the addition of silica in nano range to enhance resistance for segregation in mortar and concerete[3,4].Cement mortars containing nano-particles have reasonably higher strength, low water absorption and denser interfacial transition zone compared to those of the OPC ferrocement mortars [5]. The addition of the nanosized particles into mortars and concerete materials has surfaced as a bright solution for improving the characteristic, efficiency, cost and reliability of cementeous materials with variety of added Nano sized powders such as titania, silica, alumina …etc. [6] . Many researchers have studied the subject where: In 2014, B. Kartikeyan, et.al., studied the effect on using nano-sized mineral admixtures in concrete as a partial replacement of cement , compressive strength of the concrete cubes cast with partial replacement of ground micro silica GSF (Ground Silica Fume) for cement in 10% showed an increase by 7.5% compared with control concrete cubes. The split tensile of concrete cylinders cast with 10 % GSF an improved strength of 19.2 % compared with cylinders cast with control concrete cylinders. [7].In 2012, S. Maheswaran, et.al. studied the influence of Nano SiO_2 with particle size range from (12-50)nm in concrete and to study the pore filling effect and its pozzolanic activity with cement towards improvement of mechanical properties and durability aspects. Considerable improvement in the properties of permeability, pour filling effects, reduction of CH(calcium hydroxide) leaching, rheological behavior of cement pastes, heat of hydration, micro structure analysis, the pozzolanic activity or reactions and workability, strength and durability were reported[8].In 2012, Z. He, et.al., studied the influence of nano-SiO_2 (NS), super-fine slag (SS) and rubber powder (RP) on the abrasion resistance of concrete ,Concrete incorporated with 5wt.% NS, 40wt.% SS were investigated respectively, as well as 40% super-fine slag combining 20% replacement of sand volume with RP. The comparison experiment results showed that the reference concrete had the lowest compressive strength, modulus of elasticity and abrasion resistance, the concrete containing SS had the highest compressive strength and modulus of elasticity, the concrete containing SS combining RP had the highest abrasion resistance [9]. The main purpose for this work is to addition of the Nano powders to the mortar in different weight ratios and studying the effect of these additions on structural, mechanical and thermal properties.

Experimental Part

Experimental work consists of:

X-ray diffraction (type XRD-6000, Shimadzu) was used and this test was carried out in Nanotechnology and Advanced Materials Research Center /University of Technology. The XRD apparatus with X-ray tube Cu(1.5406A^o),voltage 40KV Current 30m,scan range 20-60 deg .In this research ordinary Portland cement (OPC) and sand were used ,and particle size distribution and chemical analysis was carried for both of them, particle size distribution was carried in the sieve shaker device by (FRITSCH) as shows in fig. (1) and The chemical composition of sand and cement is determined u was done in Iraqi Geological Survey Department / Ministry of Industry, Baghdad / Iraq, and the result are shown in table (1). Size distribution through the sieves ,for the cement and sand the sieves opening diameter for cement and sand particles was 63, 53, 38, 25 (µm) respectively and the sieve opening for sand was 300,212,150,106 (µm) respectively. The inspection of particle size is worked in Nanotechnology and advanced research Center. (Models: Brookhaven Nano Brook 90 Plus USA) Particle Size Analyzer ,ISO 13321 & ISO 22412 ,used for Most nanoparticle, and colloidal-sized materials, in any non-absorbing liquid Range: 2 nm to 6 µm, Scattering Angle 90°.The Principles of the operation is a Dilute suspensions, on the order of 0.0001 to 1.0% v/v are prepared, using suitable wetting and/or dispersing agents. The result were show in fig.(2) and it were(67.8 nm) for TiO_2 and(55.7nm) for CuO.

All the mortars had a water-to-cementations materials ratio (w/c) of 0.48 and a sand-to-cementations materials ratio of 1:3(one part of the Ordinary Portland cement (OPC) and 3 parts of sand) according to ASTM Specifications(C109) [10]. The amounts of cement and sand and Nano powders are shown as in table (2).A mixer of (150) m^3 capacity was used to prepare the mortar mixtures. Sand, cement and the Nano powder were mixed. The mixture in the container is placed into the ultrasonic device before the addition of water in order to produce a perfect incorporation of the particles then, water were added to the dry mixed powder then mixed. For about 20 minutes. The type polymeric cubic and mold was well cleaned, and the internal faces were thoroughly oiled before use, to avoid the adhesion with the mortar after hardening, the mortar casts was left for 24 hour and then de-molded. After that, the specimens marked and then completely immersed in curing tank until the date of test.

For characterization of mortar specimens microstructure test was carried out in specimen preparation lab in the Mat. Eng. Dep./University of technology, the microstructure of the specimens was found by a stereo microscope Unlike compound microscopes that give a (2-D) flat image, stereo microscopes give the viewer an "erect" (upright and un-reversed) stereoscopic (3-dimensional) image.In order to observe the surface roughness and topography of the mortar, Atomic Force Microscopy (AFM) micrographs were taken with a digital Instrument**.** Typical data has been taken from AFM height images include root mean square (RMS) and roughness, Made in USA, model AA3000 220V.

Micro hardness was carried out in the Mat. Eng. Dep./University of technology, according to ASTM E384. This device is featured by two grades micro-reading magnification with 100X and 400X times and Large LCD screen can directly show measurement methods. The test force that was used is 9.8N and the dwell time of the test force was 15 Sec and the number of measurement was 4times.it has been measured according to [11].

$$HV = \frac{F}{A} \quad \text{... (1)}$$

Where:

F: the force applied, A: is the surface area of the resulting indentation.

Wear test were conducted on a mortar specimen on wear device. The wear specimens were cubic with dimension of(5x5x5)cm. The specimen slide on a steel disc .Wear rate is calculated from the following formula[12], the applied load used in this test was 5N :

$$Wr = \frac{\Delta W}{2\pi N t} \quad \text{.... (2)}$$

And

$$\Delta W = W1 - W2 \quad \text{...(3)}$$

Where

Wr : wear rate

W1 : specimen weight before the wear test (gm)

W2 : specimen weight after the wear test (gm)

r : the distance from the center of the specimen to the center of the steel disc = 6cm

n : number of cycles for the steel disc = 950 rpm

t : sliding time

Results and discussion

The results of X-ray diffraction pattern for cement powder is shows in Fig.(3) , gives the presence of the following phases:

-Ca_3SiO_5 : C3S Alite, (monoclinic crystallized, JCPDS (42-0551);

-Ca_2SiO_4 : C2S Larnite (monoclinic crystallised, JCPDS(33-0302);

-$Ca_3Al_2O_6$: C3A Celite (cube crystallized, JCPDS(38-1429);

-$Ca_2(Al,Fe)_2O_5$:C4AF Brownmillerite (orthorhombic crystallized, JCPDS(30-0226).

Figure (4) shows the XRD patterns of the ordinary Portland cement paste with angular range of 2θ = 0 – 70 degrees , fabricated Portland cement paste modified with TiO_2 and CuO nanoparticles at 7.5 wt %, using Portlandite as the reference.

In mortar (after curing) the phases shows in fig.(4) a ,b and c, the component were:

-Portlandite : $Ca(OH)_2$,Hexagonal crystallized, JCPDS (00733)

-Tobermorite :$Ca_5Si_6(O,OH,F)_{18}.5H_2O$,Orthorhombic crystallized ,JCPDS (45-1480)

-Ettringite :$Ca_6Al_2(SO_4)_3(OH)_{12}.26H_2O$, Hexagonal crystallized, JCPDS (41-1451)

Furthermore the phases were shows:

-CSH :$CaO.SiO_2.H_2O$,poor crystallized, JCPDS (30002)

-CS :$CaSiO_3$, Monoclinic crystallized JCPDS (43-1460)

The C-S-H , CS phases in the set of Portland cement . The diffraction spectra show that alite represents the majority in the sample, a part of it is hydrated and the hydrated calcium silicate is produced. It is noticed that etringite and portlandite are present in all the hydration stages. The changes occurring in the mineralogical components during the hydration processes were also noticed, where hydrosilicates and hydroaluminates were present (to bermorite, portlandite and ettringite), After 28 days, the highest peaks of the diffractogram correspond to alite and tobermorite jells[13].The direct reaction of $Ca(OH)_2$ and TiO_2 to form $CaTiO_3$ normally occurs only at high temperature over 1000 K. As such, no obvious $CaTiO_3$ peak was detected from the XRD data.

Optical Microscopy results were shows in fig. (5, 6) ,for mortar with TiO_2 and with CuO respectively compared with mortars without Nano additions. Mechanical stirring distributes particle aggregates all over the forming material. Once the cement has completely hardened, the dispersion of the two type of distribution can be predicted according to A. Folli et.al. (Flocculating/deflocculating sedimentation models)[14]. For an equal mass of nano powder introduced into cement mortar , the first one is expected to have smaller and better dispersed aggregates than other one for which aggregates are bigger and more difficult to spread [108].

As shown in the figure, adding nanoparticles causes difference in the microstructure samples closing cement pore. In microstructure samples of ordinary cement mortar there exists large crystals of $Ca(OH)_2$. Microstructure of cement mortar is not dense and voids can be seen. As shown in figure (b), in sample containing 0.5 wt% nanoparticles relative to sample of ordinary cement mortar the structure of cement mortar has become denser and the voids decreased but still large crystals of $Ca(OH)_2$ are observed. But, with increasing quantity of nanoparticles to 2.5 percent in (c), large crystals of $Ca(OH)_2$ are eliminated and microstructure of cement mortar is completely denser.As shown in figure (d), in samples containing 5 percent nanoparticles because of the agglomeration of nanoparticles voids are formed. These microstructures with the reduction of mechanical properties in these samples are appropriate. First, the nanoparticles acted as nuclei to form needle-shaped hydration products, which can bridge the micro-cracks and fill in the micro-pores. Second, the nanoparticles altered their adjacent environment for cement hydration reaction and facilitated the formation of high- density C−S−H over low-density C−S−H. Finally, the nanoparticles led to more evenly distributed hydration phases. Note that fully achieving the potential of nanomodification hinges on the proper dispersion of the nanoparticles in the heterogeneous (or agglomeration) in matrix of a cement-based composite.

AFM is a powerful technique to investigate the surface morphology at nano to microscale. The surface of the mortar are shown in Figs.(7,8) for TiO_2 and CuO respectively all mixing ratios compared with mortars without Nano additions. The surface morphology as observed from the (3-D) AFM micrograph in the figures, presents the AFM data of the regular uniformly distributed cement paste and the cement paste modified with nanoparticles at (0.5 ,2.5,5 and 7.5) weight mass %, in a $3 \times 3\ \mu m^2$ scanning area. As can be seen in this figure (top view), the regular cement paste and nanomodified cement paste feature a microstructure with distinct characteristics. Addition of the metal oxides with higher hardness enhances the hardness and mortar as observed in Fig.(9). In Fig observe that the addition ratio (0.5 - 7.5) wt% of titanium dioxide nano powder raising of micro-hardness by (0.34 - 1.38) time while the presence of the same amount of copper oxide nano powder increased the percentage of (1.2- 4.6) times. In addition an apparent of hard smooth grain (as seem in microstructure) also decreased and evanescence the large $Ca(OH)_2$ hard grain responsible of softening[14,15].

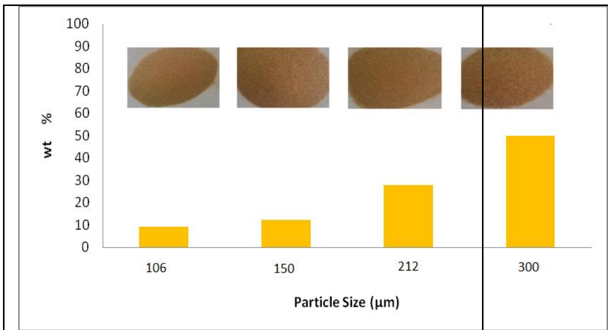
Fig.(10) shows the effect of nanopowder of wear rate .It shows that wear rate decreased with an increased in weight ratio**.** This is the consequence of the attractive electrostatic interactions promoted by ion–ion correlations, already observed also for

C–S–H (calcium silica hydrate gel) particles. This is the consequence of the attractive electrostatic interactions promoted by ion–ion correlations, already observed also for C–S–H (calcium silica hydrate gel) particles[16,17].This interconnection reduced wear rate and increased hardness as it can seem above in fig.

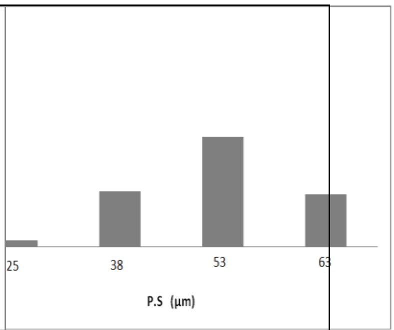
Conclusion

Using metal oxide was useful, for building application For all the mortar XRD it showed several peak belonging to the nanomaterials in the crystal structure within the mortar, which identify that these nano powder completely blended within C-S-H, as well as filling the pores and increase in the apparent density, smoothening of the grain which belong to CH in general.

All the mortar samples had a reduction in it roughness after the addition of the nanopowder in which the addition of CuO lead to a decrease by One eighth of the free of addition mortar, and TiO_2 decreased the roughness by one quarter of the as received mortar, due to CuO particles being smaller than TiO_2 particles, thus leading to an improvement in the surface characteristic of the mortar.



a: sand



B:cement

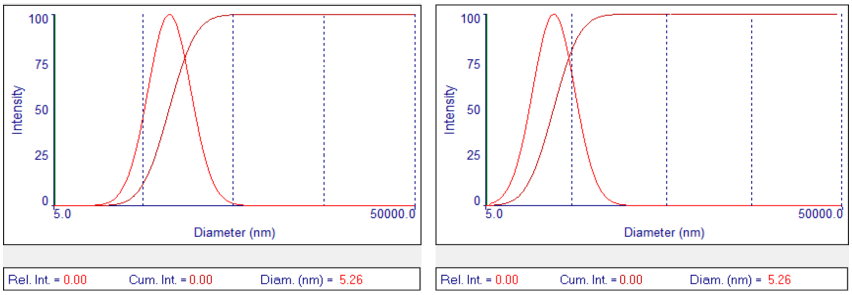
Figure (1) a:Analyzing of particle size for

Table (1):Chemical composition of sand and cement

%	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	Na_2O	K_2O
Sand	99.2	0.092	0.66	< 1	0.04	0.04	0.04	0.03
Cement	17.75	4.28	5.29	63.37	2.35	2.77	0.31	0.84

Table (2) : Weight ratio for each Components.

Water ml	Sand wt%	Cement wt%	N.P. wt%
2.5	75	25	0
2.5		24.5	0.5
2.5		22.5	2.5
2.5		20	5
2.5		17.5	7.5



a. **b.**

Figure (2) P.S Spectra of a: TiO_2 , b:CuO

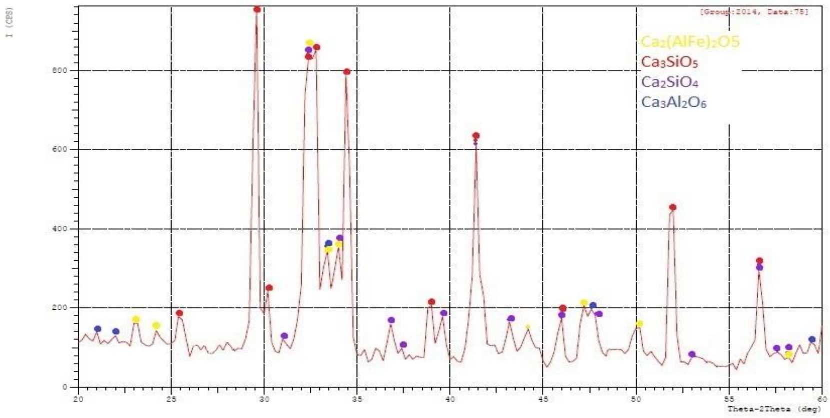
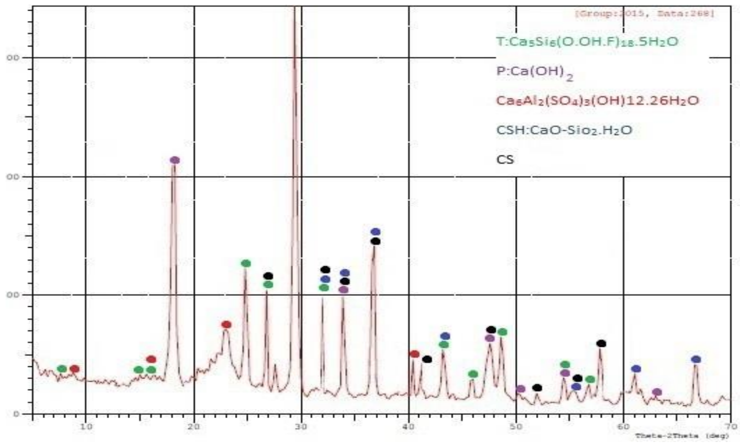
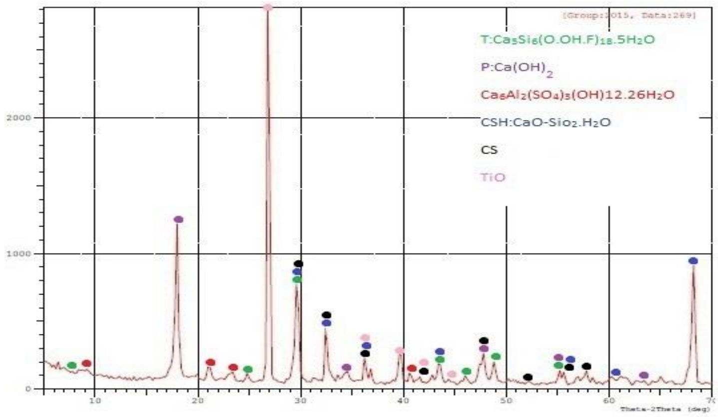


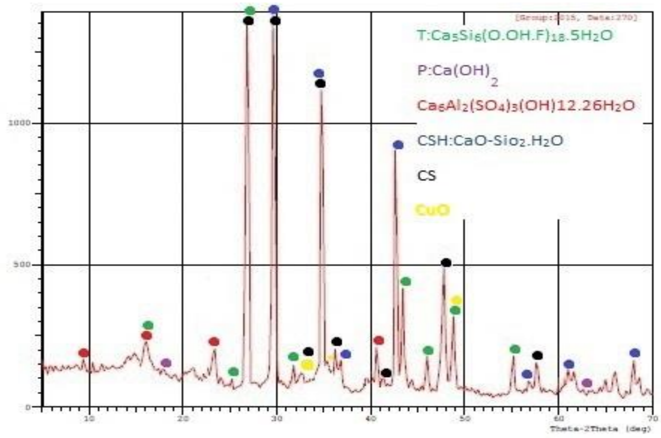
Figure (3) The XRD Spectra of cement powder



a

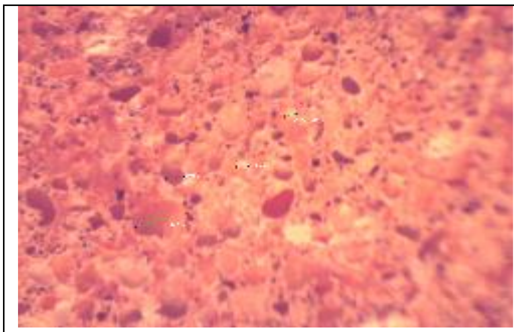


b

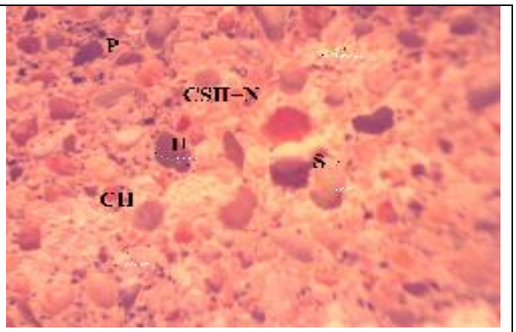


c

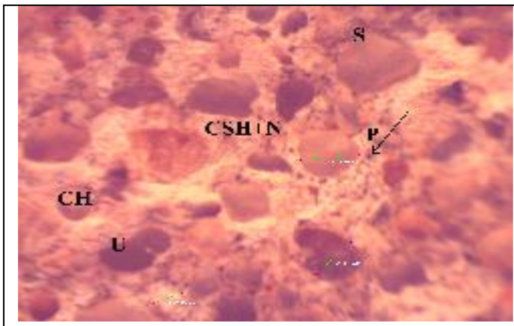
Figure (4)The XRD Spectra of cement Mortar (a: Mortar ,b: Mortar +TiO_2 ,c: Mortar + CuO)



5%TiO_2

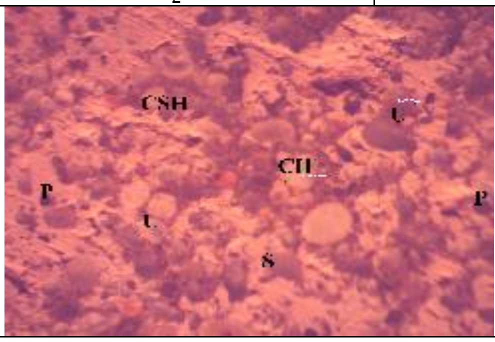


7.5% TiO_2



0.5% TiO_2

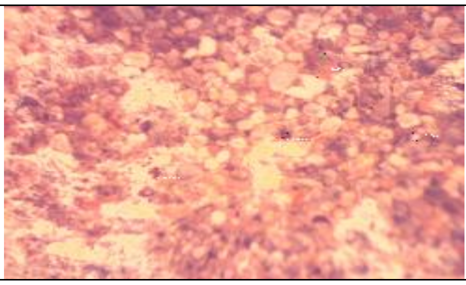
2.5% TiO_2



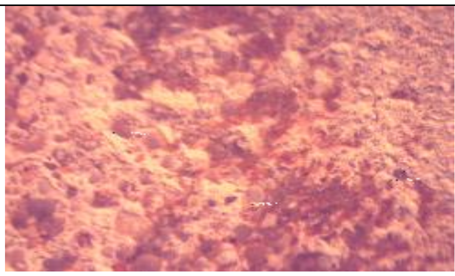
0%

S : sand
CSH:
U :Unreacted cement
P :Pore
CH:
N:Nanopowder

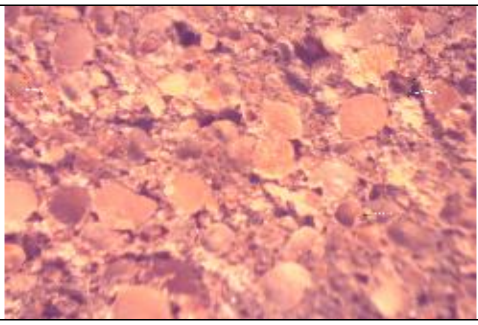
Figure (5) Optical microscopy image for TiO_2



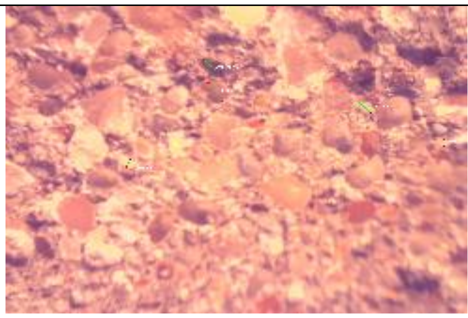
5%



7.5%



0.5%



2.5%

Figure (6) Optical microscopy image for CuO

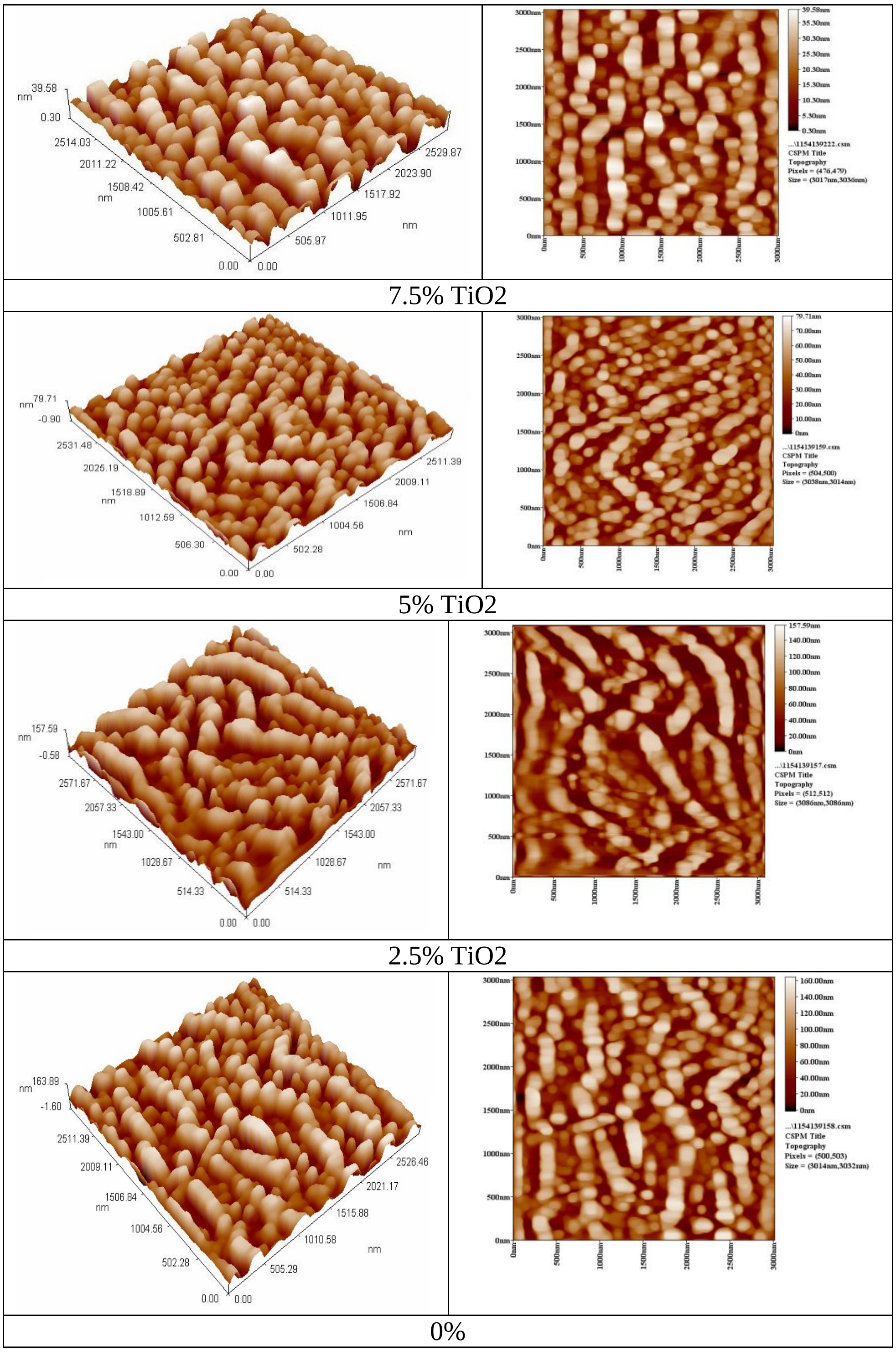
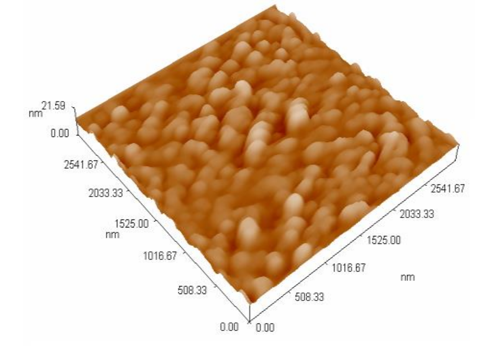
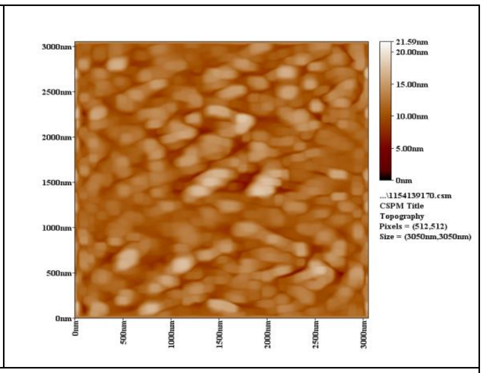
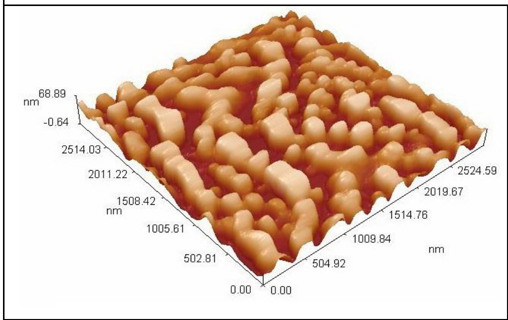
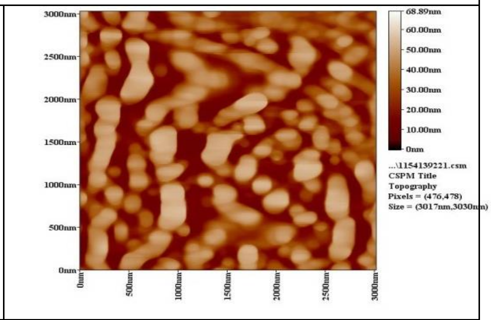


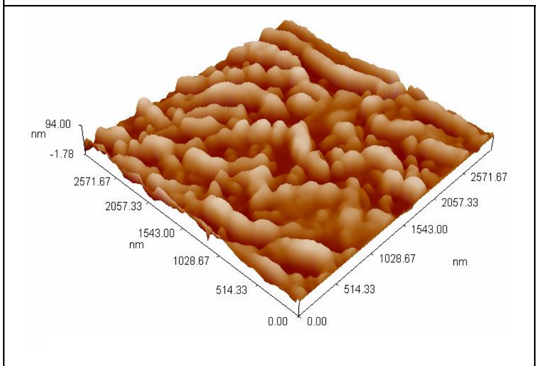
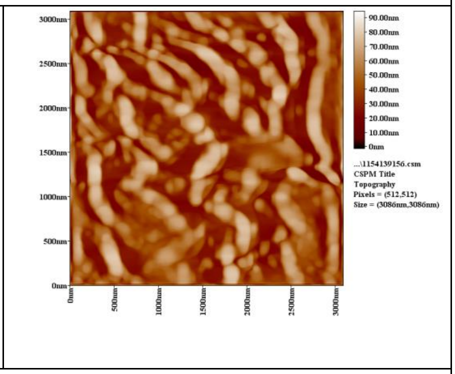
Figure (7) 3-D AFM images of the Mortar mixed TiO_2 at different ratio.

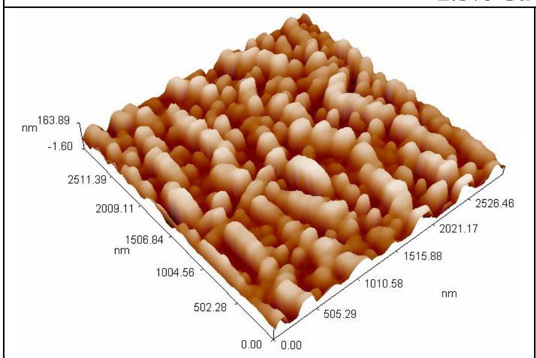
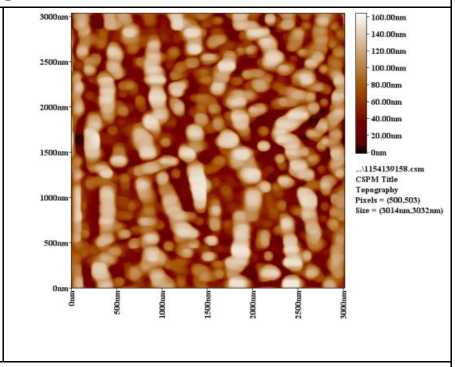
7.5% CuO

5% CuO

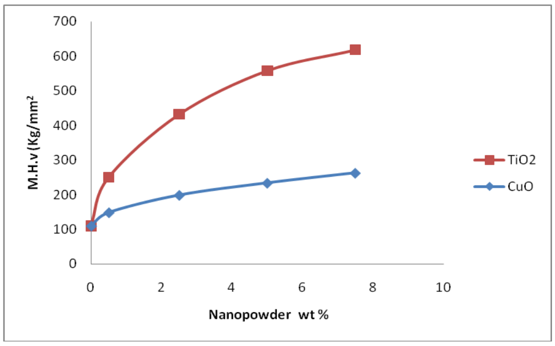



2.5% CuO

0%

Figure (8) 3-D AFM images of the Mortar mixed CuO at different ratio.



Figure(9) Microhardness of prepared mortar at different weight ratio.

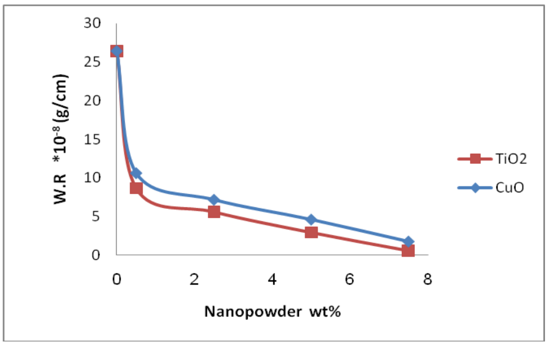


Figure (10) Wear rate of prepared mortar at different weight ratio.

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