



Fabrication of Advanced Cement Mortar for Building Anti-Bacterial Applications

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Abstract

In this research, we have added nano anatase TiO_2 as a partial replacement of Portland cement by a weight percentage of (0.25 to 1%) for the development of properties for protection against bacteria. The control mix was made by using "the cement to sand" proportion about (1: 2.75) with the "water to cement" proportion of (0.5) to study the structure, porosity, water absorption, density, mechanical properties, as well as anti-bacterial behavior. Inspections have been done such as scanning electron microscopy (SEM), and atomic force microscope (AFM) for mortar. Experimental results showed that after the addition of Nano powders in cement mortar, the structural properties improved significantly with the development of hydration of cement mortar at early age, reduction of porosity and the increase of density as well as enhancement in compressive and anti-bacteria properties that make the preparation of nano material very suitable for protection against bacteria.

Keywords: Anti-bacteria, Cement mortar, Nano powder, TiO_2 .

1. Introduction

Nanotechnology has a major importance in producing nanoscale materials with excellent properties for many applications, including electronics, healthcare, chemicals development and construction. [1].

The TiO_2 is completely recognized that it is existed naturally at crystallographic with three separate situations, "anatase, rutile and brookite" at the same time as the "anatase TiO_2 " generally uses photo catalysts to oxidative dissolution of natural combination, and very good photocatalyst to photo dissolution and "sun - energy" adaptation according to the rising of photo activity [2].

In 2009, A.R. Jayapalan et. al. studied the effect of addition anatase TiO_2 nanoparticle within 2 dissimilar bit quantities of TiO_2 at substitute stages about (5%, 7.5% and 10%) for the mortar

at premature time hydration of Portland cement. The product showed the quickened rate response at early levels of hydration and improved the heat of hydration relative to the adding percent and the purity of TiO_2 [3].

Ali and Shadi in 2010 studied concrete containing different amounts of TiO_2 nanoparticles by employment a strong point of water absorption efficiency to the highest performance. The result showed the specific strength and impedance to the permeability of water, that improved by adding TiO_2 nano particles within the cement paste to (4 .0 wt %) and TiO_2 nano particles, was enlarged by crystalline $Ca(OH)_2$ quantity. TiO_2 nano particles could enhance mechanical and physical characteristics by speeding the summit form of conduction calorimetry test and weighing

reduction within thermo gravimetric examination [4].

In 2010, Thanongsak Nochaiya and Arnon Chaipanich investigated the compressive strength and hydration of Portland cement-TiO₂ Mixes. The ratio of water used to produce cement mortar is about (0.5), the result showed no influence for TiO₂ in mortar on the compressive strength, but there was a slight increase in the density, the ability of TiO₂ in combination with UV light is to kill various microbials and remove the air pollution [5].

In 2012, N. Maravelaki et al. studied the influence of nano titania of anatase shape in the hydrolysis and mechanical characteristics of the resulting mortars having (first: binder of natural hydraulic lime) and (second: very well collections of carbonate), on the top of binders extensively using the layout of reinstatement mortars, the nanotitania ratio was about (4.5% and 6%) of binders. The physicochemical and mechanical characteristics of the nano titania mortars were considered and compared with the individuals not including the nano titania extension. FTIR and XRD analyses specified the development of (carbonation, hydration and hydraulic complex structure), while a one year treatment showed the effect that enhanced the mechanical characteristics and hydration enhancement of the mortar mix with nano titania when contrast with the sample not including nano titania. The hydrophilicity of nano titania enhanced the moisture kept in mortars as a result of smoothing the hydration procedures, where the existence of humidity controls the mortar for more efficient sticking [6].

At the year (2013), Ming Zhi Guo et al. studied the behavior of TiO₂ photo catalytic dip plated by "self-compacting glass mortars (SCGMs)", at the conditions of weather contaminant deduction and bacteria obstruction, studies improved that "Nitrogen oxide (NO)" and "Escherichia coli K12" could be applied as an aim for weather contaminant and bacteria examination fatigue correspondingly. If "Nitrogen oxide (NO) elimination" is applied, then the effective situation of (no weathering) and (harsh processing) could be used for (EtOH / mortar) equal to (14.33 mg m⁻²h⁻¹) and (8.75 mg m⁻²h⁻¹).

Nitrogen oxide (NO) elimination efficiency by TiO₂ for dip plating the glass model and it has absolutely vanished as a disparity situation. The Nitrogen oxide (NO) elimination efficiency for dip plated, SCGM at a standstill elevated for antibacterial effectiveness the whole deactivation of E.coli, was spotted by TiO₂ dip plated of glass

with SCGM models about (60 minute) of UV irradiations. E coli deactivation of TiO₂ dip plated glass was almost a minor later than scratching procedure while concentrate the situation of E coli residual at the plane of TiO₂ dip plated SCGM just reduction from (105) to (103) CFU – ML. It be able to accomplish photo catalytic bacteria deactivation is a further complicated procedure and the outcome to photo catalytic action of the Nitrogen oxide (NO) elimination cannot for all time be readable to photo catalytic antibacterial action [7].

In 2014, Anne Beeldens studied the photo catalysis basics and relations amid within TiO₂ and concrete at various parameters, such as temperature, light intensity, relative humidity and flow rate. Photocatalysis is a huge probability to handle with growing pollution by traffic, adding TiO₂ to cement mortar is to decrease the polluting of air that is effected by the exhaust of gasses, and the examination appeared the switching of (NO_x) to (NO₃) while uncovering to "UV light" at:

- altitude temperature over than 25C°
- lower relative humidity
- long contact times [8].

2. Experimental Part

The experimental work consists of two parts:

1. The first section is the preparation of the nano powders anatase TiO₂.
2. The control blend was made with a cement to sand of, 1:2.75 with no adding of nanoTiO₂. Cement was then replaced with nano TiO₂ as received and also processed nano TiO₂ at replacement levels of (0.25,0.5,0.75,1%), the ratio of water to cement was kept on 0.5 to every blend and the mortar was then poured into (d=2 cm*h=1cm) cylinder moulds for testing the structural properties (SEM,AFM) and physical properties (true density, porosity, water absorption) Leading such examination require arrange a test model of a suitable density.

In order to learn the effect of nano powder at the appearance of mortar, the inspections of nano powders include:

- a- Chemical composition: This test was performed in the Iraqi Geological Survey Department at the Ministry of Industry, Baghdad Iraq.
- b- Scanning electron microscopy (SEM): The SEM study was carried out by Electron Gun Tungsten, the accelerating voltage was from

200V to 30kV, (Japan). The setting was done in the Department of Production Engineering and Metallurgy at University of Technology.

- c- Density: This test was performed in the Metallurgy Laboratory of Materials Engineering Department at University of Technology rendering for typical (ASTM C373) [15], the valid thickness (ρ_t) was prepared by the technique of tuning the sample in water (Archimedes base) by means of connection [9, 10]:

$$pt = \left(\frac{Wd}{W_s - W_n} \right) * D \quad \dots(1)$$

Where:

Pt: True density or bulk density (gm/cm^3)

D: Density of distilled water (1 gm/cm^3)

Wd: Dry weight of sample (gm)

Wn: Weight of the sample when submerged in water (gm)

Ws: Weight of the sample after saturation in water (gm)

- d- Atomic Force Microscopy (AFM): It was processed by using digital tools, ideal data processed by AFM elevation images as well as the root mean square (RMS), AFM is made in USA model AA3000 220V.

- e- Compressive strength: The compressive strength of cubes was determined by using a universal measurement machine (EVERY DENISON) of 2000 KN capacity, the loading rate was about 0.25 MPa per sec. The average result of three specimens was reported for each mortar sample that was performed for 7 days. This inspection was carried out in the material Laboratory. Of the Building and Construction-Engineering Department /University of Technology using the following relationship [11].

$$\sigma = P/A \quad \dots (2)$$

Were,

σ : compressive strength, (MPa)

P: ultimate compressive load, (N)

A: sample area, (mm^2)

- f- Anti bacteria: It was taken using bio nano technology main lab of highly developed equipment Research Center at Technology University in Iraq, the formula to calculate the bacteria survival rate is as following [12]:

Number of Colonies =

$$\text{(Number of colonies for each dilution) * (dilution 103 factor) / sample volume}$$

$$K = \frac{(A-B)}{A} * 100\% \quad \dots(3)$$

Were,

K: The bactericidal rate.

A and B: are the numbers of bacteria colonies corresponding to the reference sample and TiO_2 incorporation sample, respectively.

3. Result and Discussion

3.1 Chemical Compositions

The chemical composition results of raw materials are given with the American Society for Testing and Material in Table (1) for comparison purpose. By comparing the ratio of cement oxide according to the characteristic of ASTM C150 indicated that the ordinary Portland cement of type I is "Normal" and Silica natural sand free of salts conforming to the standard sand according to the characteristic of ASTM C778.

Table 1,
Chemical compositions of raw materials.

Material	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	CaO %	MgO %	SO ₃ %	Na ₂ O %	K ₂ O %	Total
Sand	99.2	0.092	0.66	<1	0.04	0.04	0.04	0.03	100
Cement	18.75	4.28	5.79	64.37	2.85	2.77	0.31	0.88	100

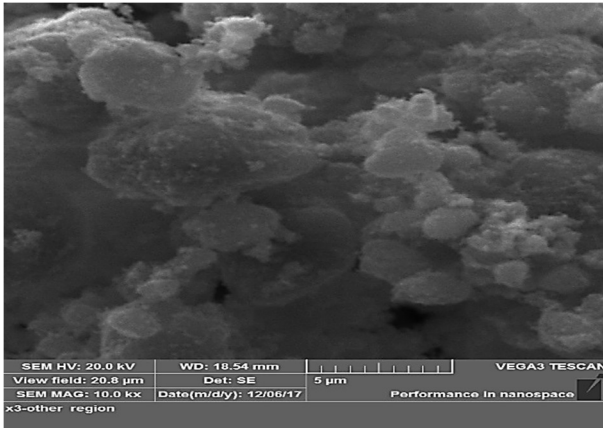
3.2 SEM Results

By adding the percentage of nanoanatase TiO_2 to mortar, it was noticed that a change was made in the microstructure; an increase in the grain size and the gel expanded until it filled all the spaces and for mortar without any additive, as shown in fig (1-b) it was noted that the gel exists and it

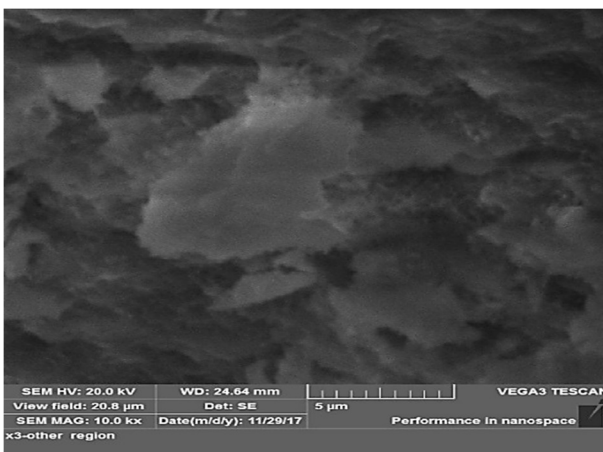
content of porosity because interaction is not complete.

The two figs. (1-c) and (1-d) indicate the addition of nanoparticles causes a difference in the microstructure of samples (as it was seen in the optical test of the microstructure in sample containing (0.25wt) % nano particles compared to the model of usual cement mortar). The cement mortar structure has grown to be more intensive

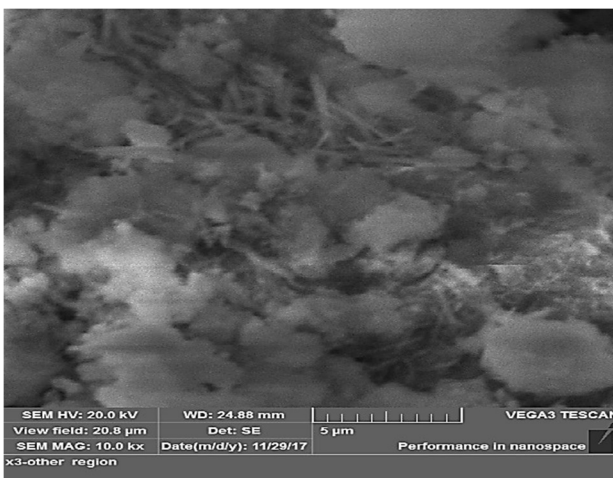
and the spaces decreased until huge crystals of $\text{Ca}(\text{OH})_2$ are spotted. However, at growing the size of nano particles, about 1% large crystals of $\text{Ca}(\text{OH})_2$ are extracted with a complete density microstructure of cement mortar and the appearance of the needle phase at high rate.



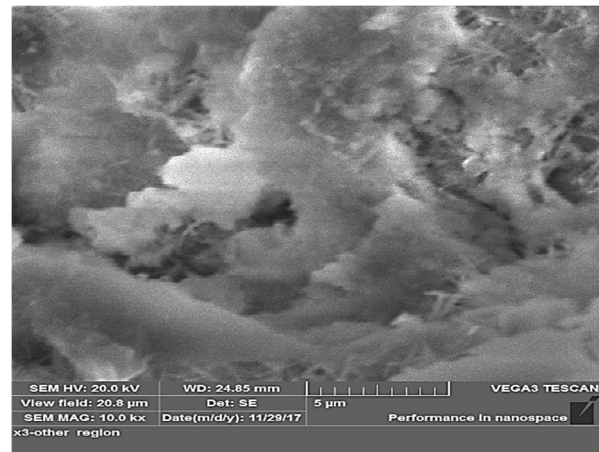
(a)



(b)



(c)

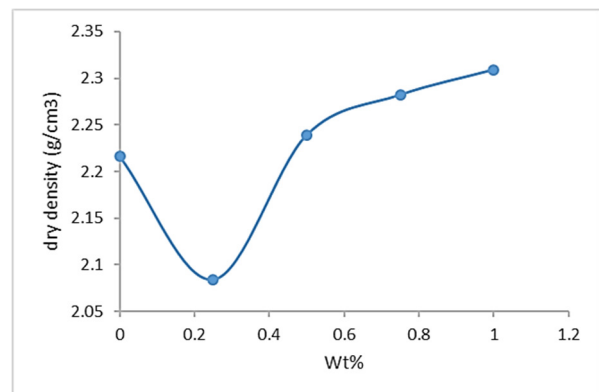


(d)

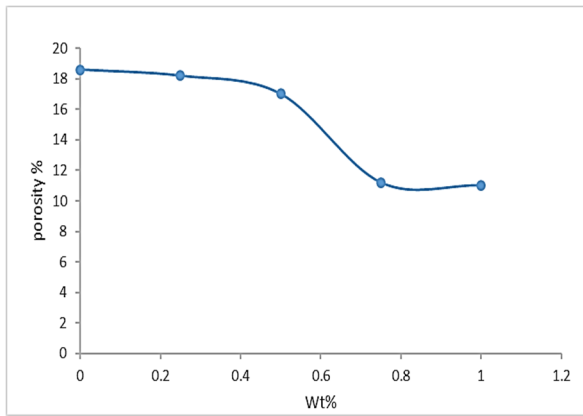
Fig. 1. SEM micrographs of used a: nanoanatase TiO_2 , b: Mortar without addition, in c: Mortar with 0.25 TiO_2 , in d: Mortar with 1% TiO_2 .

3.3 Density, Porosity and Water Absorption Results

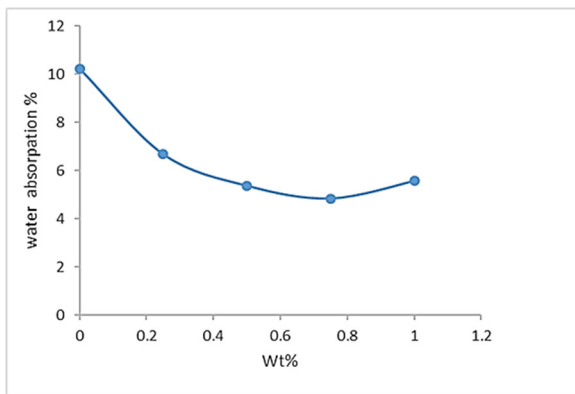
As shown in figure 2A, in sample containing (0.5 wt%) percent nanoparticles relative to the sample of ordinary cement mortar, the structure of cement mortar has become denser and the voids decreased but still large crystals of $\text{Ca}(\text{OH})_2$ are observed. But, with increasing quantity of nanoparticles to (1 wt%) percent, large crystals of $\text{Ca}(\text{OH})_2$ are eliminated and the microstructure of cement mortar is completely denser [13], The two figs. B and C indicate that the absorption of all specimens decreases continuously with the mixing by nano materials. This is may be due to the partial filling of pores by the hydration products, which reduce the capillary porosity. The decreasing in water absorption may be due to enhancement of the cement mortar porosity by both physical and chemical mechanisms of nano materials.



(a)



(b)



(c)

Fig. 2. The physical properties of cement mortar.

3.4 The Compressive Strength

The compressive strength and workability of mortar with nano TiO₂ were strongminded, and the results were associated with these of Normal Cement mortar (NCM) mortar with nano TiO₂ that was created to be more durable than Normal cement mortar and the measurement increases in strength of mortar with nano TiO₂ with deference to NCM which was found to be in the variety of 17% to 50% for various marks of mortar. The workability of mortar with nano-TiO₂ was found to be considerably further than that of NCM.

It is realized that the porosity and pore size distribution are the critical mixing contents by microstructure of hydrated cement paste so that the appearance of strength. to get a high durability should decrease the permeability and hardening cement mortar, the decrease of the cement paste porosity is obtained in the present work due to the influence of nano-TiO₂ on the mechanical properties of cement mortar. Examination outcomes specified that while cement has been replaced with nano - TiO₂, the strong

point about the mortar of cement by near the beginning ages has been more improved, also the liquidity and strength at late ages were certainly reduced [14]. On the other side, Portland cement at (0%, 0.25, 0.5, 0.75 and 1% by weight) was partially used by adding water to cement proportion (w/c) and about 0.5 has been applied completely to the mixture of cement dentifrice.

Whole the porosity and capillary pores have decreased when the gel pore has larger than before as an consequence of the adding nano TiO₂ at every substitute stages examination result consequences point to that the mixing cement dentifrice more than 1% of nano TiO₂ has been designed a dentifrice and high compressive times strength than that with 0.25 nanoTiO₂. The porosity and pore size of mixed cement paste were dramatically affected by the additional of nanoTiO₂.

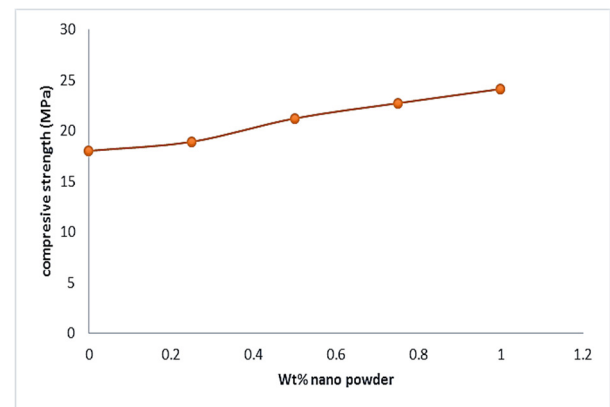
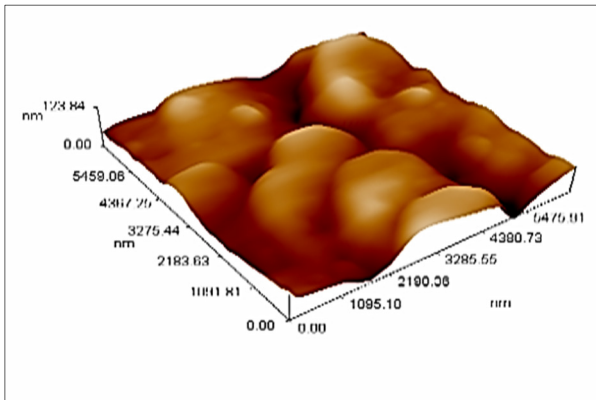


Fig. 3. The Compressive strength of cement mortar.

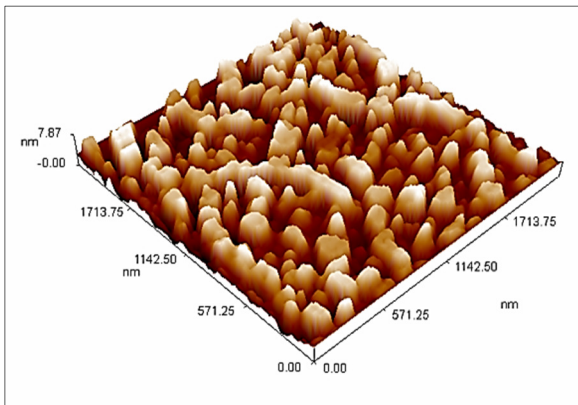
3.5 AFM Result

AFM is an influential method to study the morphology plane by nano microscale, the surface of mortar is shown in fig. (5) for 2D of the surface. The roughness of mortar surface without the addition of nano anatase TiO₂ was 123.84 nm while in the presence of 0.25 Wt% as partial replacement of cement by nanoanatase TiO₂, it was 7.87 nm and in percentage of 1% TiO₂ 3.3 nm. The difference depends on the rate of replacement of cement with nanopowder. From the AFM test, it can also be seen that the mortar without additive, the average diameter was 247 nm, and in 0.25 TiO₂ it was 105 nm and in 1% TiO 92.16 nm. These figures show that the mixed samples are more homogeneous than reference cement mortar because the addition of

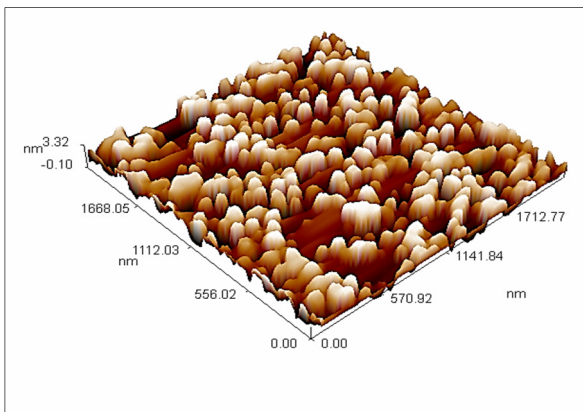
nanoparticles reduced surface roughness and average diameter. The aim of this investigation is to obtain information about the surface morphology and geometry of structures [15]



a



b



c

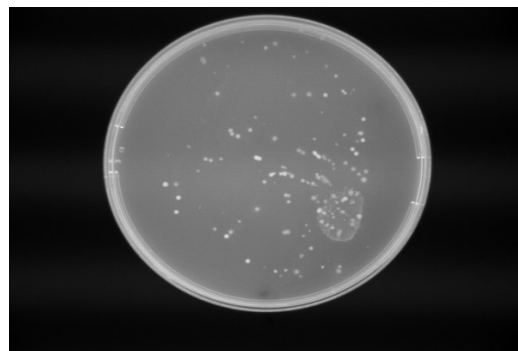
Fig. 4. The AFM results of cement mortar: (a) Mortar without addition, (b) Mortar with 0.25 TiO₂, and (c) Mortar with 1% TiO₂.

3.6 Anti-bacteria

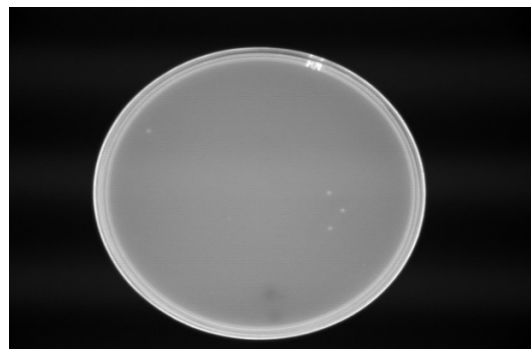
Preparation of Bacterial Culture and Result

Bacteria species that used in this research included Gram negative *E.coli*.

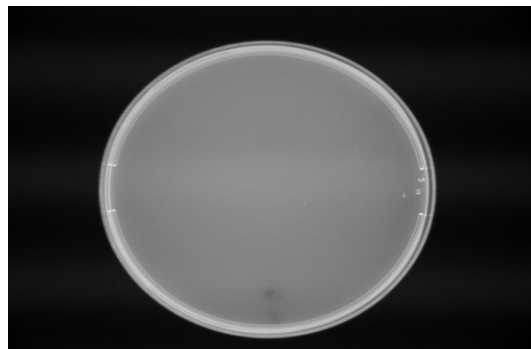
Bacteria were cultured on nutrient agar at 37°C for 24 h, then bacterial suspensions were prepared by taking a single colony from each stock bacterial culture with a loop and impregnated in sterile of specific amounts of 0.9% normal saline. Then, the bacterial suspensions were diluted using a portion of 0.9% normal saline to obtain cell samples with concentration ~10⁷-10⁸ CFU/ml by 0.5 McFarland standards. The result showed that rate of killing of bacteria increased by increasing the substance nanoanatase TiO₂, fig. (5).



Mortar without nano TiO₂.



Mortar with 0.25% TiO₂.



Mortar with 1% TiO₂.

Fig. 5. Effect of anti-bacteria on mortar.

4. Conclusions

1. The hydration of ordinary Portland cement increased at early age and this resulted an increase in the compressive strength of the cement mortar.
2. Nanoparticles fill the voids, reduce the pore, improve the structure and become more homogenous.
3. Anatase TiO₂ nanoparticles have excellent photocatalyst properties and reduce largely the proportions of the bacteria growth and therefore they are suitable to protect the environment from the growth of bacteria.

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تصنيع مونة الاسمنت المتقدمة لتطبيقات البناء المضادة للبكتريا

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الخلاصة

في هذا البحث تم اضافة نانو-بتاينيه طور (anatase) بوصفه كمادة استبدال زئي للسمنت البور-لاندي بنسب وزنية (0.25 to 1%) لأجل تطوير خصائص (الحماية ضد البكتريا) وم إراء-زيج-رعي مع نسبة الأسمنت إلى الرزل (1:2.75). تم الحفاظ على نسبة المياه في الاسمنت عند 0.5 وم إراء فحوصات الكثافة، المسامية، معامل الإلتصاق فضلا عن الى ذلك الخوا التركيبية SEM, AFM, و فحوصات Anti-bacteria بينت النتائج إ الملائم الاسمنتي يمتلك بنية منتظمة وعامل خشونة انخفضت بعد اضافة المادة النانوية وحسنت الكثافة و انخفاض في معامل الإلتصاق والمسامية مما يجعل الملاط المحضر لانما لتطبيقات (الحماية ضد البكتريا).