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Nano Titania Combined with Micro Silica Reinforced Limestone Cement: Physico-mechanical Investigation

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THE INFLUENCE of different ratios of fillers addition such as limestone and micro white sand, as well as nano-titania particles on the physico-mechanical properties of ordinary white Portland limestone cement (OWPC), has been studied. One mix (10.00 wt. %) of equal ratios from limestone and white sand in micro scale, partially replaced by cement clinker. Mechanical strength, phase composition, and microstructure of blended cement have been investigated. The results showed that filler reduces the setting time and total porosity, improve the strength, free lime content, combined water content and bulk density of cement pastes due to the pozzolanic effect of micro white sand that replaces clinker and enhance an excess amount of hydration products. It can be concluded that limestone fills the pores between cement particles due to the formation of carboaluminate, while micro sand increases the hydration C-S-H product. TiO_2 nanoparticles (i.e., less than 100 nm) shows a great efficiency in enhancing the mechanical strength of cement paste due to the efficient incorporation of nanoparticles as active sites for gel Tobermorite C-S-H fibers growth during hydration,

Keywords: Ordinary White Portland Cement (OWPC), White micro Sand, Pozzolania, Cement hydration, Titania-Nanoparticles and morphology.

Introduction

A lot of research works on adding different materials to cement and concrete has been reported. The physico-chemical and mechanical process of cement hydration is a complex process [1,2]. Topo-chemical theory and through-solution reactions are the two mechanisms which explain the C-S-H gel formation once cement starts hydration [3,4]. The process of cement hydration has five stages starting from initial dissolution period and forming thin protective layer on C3S particles then reaction rate increases continuously through stages two and three reaching its optimum after less than 24 hrs after cement mixed with water leads to growth of gel C-S-H fibers and cement grains become more compacted then decreases the reaction rate into half of its maximum in the remaining stages [5,6].

Limestone filler knowing as inert and when added to clinker might fill the pores or spaces in the granulometric of cement without an increase in water demand, improves cement packing and blocks all the capillary pores. Hence, many researchers proved that addition of limestone to cement slightly decreases water demand, setting times and total porosity; whereas increases combined water content and compressive strength up to 15% of cement paste [7-11]. Whew limestone reacts with aluminate and ferrite phases from cement to form monocarboaluminate it act

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also as a nucleating sites for cement hydration products due to its high specific surface area and incorporates into the calcium silicate hydrates (C–S–H) themselves and accelerating the hydration of clinker particles especially the C3S improving early strength also may lead to the formation of some calcium carbosilicate hydrate, and reduces the potential cementing material causing a decrease in later strength and reacts with C3A forming monocarboaluminates [12-15].

Nanotechnology science shows many advantages in the construction and building materials fields due to the multi-different uses of nanoparticles because of their unique properties [16,17]. Nanomaterials make the building stronger and harder than the traditional building materials giving them enhanced ductility and formability, however materials applications are limited by using nano-TiO_2, nano-SiO_2 or nano-Fe_2O_3. These materials have self-cleaning properties which triggering a photocatalytic degradation of pollutants, such as NO_x, carbon monoxide, a pollutant in the air [18], halides and aldehydes from the vehicle and industrial emissions protecting the environment [19,20]. Test method for air-purification performance of semiconducting photocatalytic materials can be carried out using ISO 22197-1:2016 procedure.

In view of the above-mentioned, the objective of this study is to investigate the effectiveness of limestone and grounded white micro sand used at various replacement levels on the performance of the white Portland limestone cement in terms of compressive strengths, water absorption, drying shrinkage, and density. The normal and high strength limestone pozzolania cement materials were tested at different ages up to 90 days for the aforementioned characteristics. Based on the test results, the threshold of understanding the effect of TiO_2 on cement properties and hydration mechanism as a minor substitution in presence of active pozzolanic materials has been discussed in this paper.

Experimental

Materials

The materials used in this study were Ordinary white Portland limestone cement (OWPC), of Blaine surface area 450 m^2/g and dimensions (200 _ 100 _ 20 nm) ASTM Type I, supplied by Helwan Cement Company, El-Minia plant, Minia Governorate, Egypt. Limestone, of white limestone “Samalot Formation” supplied by Samalot limestone quarry, El-Minia Governorate, Egypt. White sand was ground till reaching the microscale i/e: 0.30 µm; brought from Rass Garieb Area, Sinia Governorate, Egypt. The mix composition of Ordinary white Portland limestone cement, limestone and white micro sand were homogenized together. Titanium oxide nanoparticles pure anatase. (less than 100 nm) was provided by the nanotechnology and materials science department [21], Faculty of Postgraduate studies for advanced sciences, Beni-Suef University, Beni-Suef, Egypt. Mix composition, wt. % of limestone pozzolania cement blended by TiO_2 nanoparticles pastes.

Paste preparation and techniques

One patch of blended cements was prepared by partial replacement of OWPC with 10% equal ratios from limestone and white micro sand in presence of constant clinker/gypsum ratio of 0.95. Cement paste was hydrated with their corresponding water of consistency 0.28%. Freshly prepared cement paste was molded in stainless steel molds of (5x5x5 cm) at about 95% relative humidity and 21±2 ^oC demolded after 24 hours and cured up to 90 days in tap water, (Table 1) illustrates the composition of cement mix blended in wt. %.. Two mix patches of blended cements were prepared by partial replacement of OWPC with 1.0 and 2.0% of TiO_2 nanoparticles Table 2 illustrates the mix composition of blended cements in wt%.

Characterization

X-ray diffraction and X-ray fluorescence analyses

Analysis of X-ray diffraction (XRD) performed by Panalytical x-ray diffractometer, model 2014 PW 1370, Co. using Ni filtered CuKα radiation (1.5406 Å) under 30 kV operating voltage and emission is current 24 mA. The step-scan covered angular range 15-80° (2θ). Mineralogical composition of the investigated samples was identified using the PDF standards. The analysis of X-ray fluorescence (XRF) operates by using ARL 9900 apparatus.

Transmission Electron Microscope

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through it [22]. Transmission electron microscopy is very efficient tools to catching images for particles in nano-scale,

TABLE 1. Mix composition, wt. % of blended cement.

Mix	OWPLC	Limestone	White micro Sand	Total Mix.
LG	80	10	10	100

TABLE 2. Mix composition, wt.% of LG blended by TiO_2 nanoparticles pastes.

Mix	OWPLC	Limestone	White micro Sand	TiO_2 nanoparticles	Total Mix.
LG	80	10	10	0.0	100
LGT1	79	10	10	1.0	100
LGT2	78	10	10	2.0	100

$$d = \frac{\lambda}{2n\sin\alpha} \approx \frac{\lambda}{2NA}$$

Scanning electron microscopy

Scanning electron microscopy (SEM) operated by using Joel-Dsm 5400 LG apparatus to examine the morphology and microstructure of selected samples by mounting on stubs and coating with gold prior to analysis to make them electrically conductive.

Physico-chemical Measurement

Each measurement was calculated through taking the average of the three samples. The following physical and chemical measurements were carried out:

Compressive strength

Measuring the compressive strength of hardened pastes was carried out according to ASTM designation (C 109-150). Compressive strength measurement recorded by using a manual compression strength machine. The broken chips of cement cubes were used for stopping of hydration progress and free water determined then stored in airtight containers for other measurements.

Free Lime Content

The free lime content was indicated by taking 0.5 gm of the remaining hydration chips and well-grounded with 50 ml ethylene glycol in a conical flask which then put in stirring water bath at 70°C for 30 min. and then titrated with HCL (0.1 N) till the color of the solution converts from brown to colorless solution [23].

%F.CaO = V.of HCL (0.1 N) x 0.50

Bulk density

Bulk density was determined by weighing hydrated cement pastes suspended in liquid (weight of suspended sample) and in the air after drying by a wet towel (weight of dried sample). The following equation was used for calculating the bulk density using Archimedes principle [24]:

$$\text{Bulk Density (BD)} = \frac{\text{(Weight of dried sample x density of liquid)}}{\text{(Weight of dried sample - weight of suspended sample)}} \ \text{g/cm 3}$$

Total porosity

The process of hydration characteristic, the total porosity of the hardened cement paste was determined from the values of bulk density, as well as free plus total water contents as follows [25]:

$$\text{Total Porosity, \%} = \frac{0.99 \times We \times BD}{1 - Wt} \times 100$$

Where:

0.99 is the specific volume of free water in cm^3/g.

We is the free water content, %.

BD is the bulk density, g/cm^3.

Wt is the total water content, %.

Results and Discussions

Hydration characteristics

Table 3 illustrates the chemical composition wt.% of ordinary white Portland limestone cement (OWPC), limestone and white micro sand determined by XRF analysis. Also, white micro sand was detected by SEM images (see Fig. 1 & 2), before and after grinding to shows its morphology and the new microstructure of ground sand in micro scale. TiO_2 nanoparticles are prepared as explained elsewhere. Table 4 illustrates the properties of TiO_2 nanoparticles and Fig. 3 illustrates the particle size which proves that TiO_2 nanoparticles in nano-scale which have a size range from 69 – 82 nm. The TiO_2 grains (Anatase) appeared to be with homogenous distribution with a small degree of agglomeration.

TABLE 3. Chemical composition of raw materials by XRF.

Material	SiO_2	Al2O3	CaO	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI	Cl^-	Total
OWPC	22.07	3.00	68.41	0.14	0.31	2.84	0.03	0.06	2.35	0.02	99.23
Limestone	0.28	0.04	54.77	0.03	0.20	0.14	0.04	0.03	43.72	0.02	99.27
White micro Sand	95.16	2.87	0.14	0.08	0.02	0.01	0.001	0.001	0.005	1.30	99.58

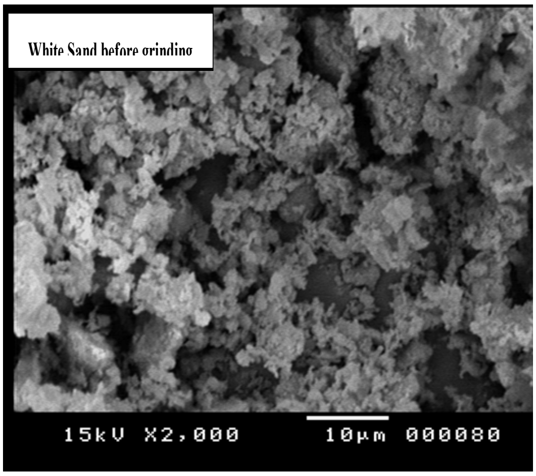

Fig.1. SEM images of white sand before grounded.

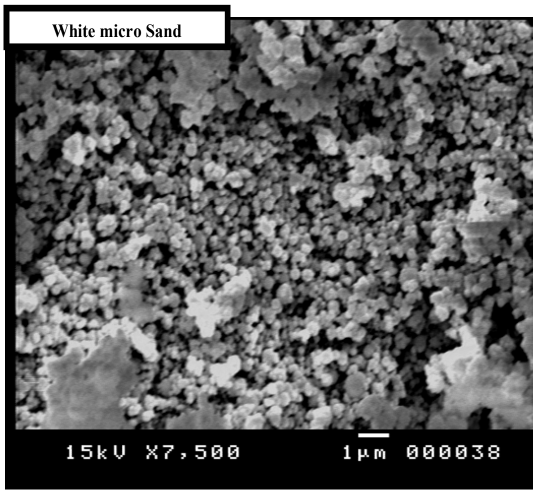

Fig. 2. SEM images of white micro sand after grounded.

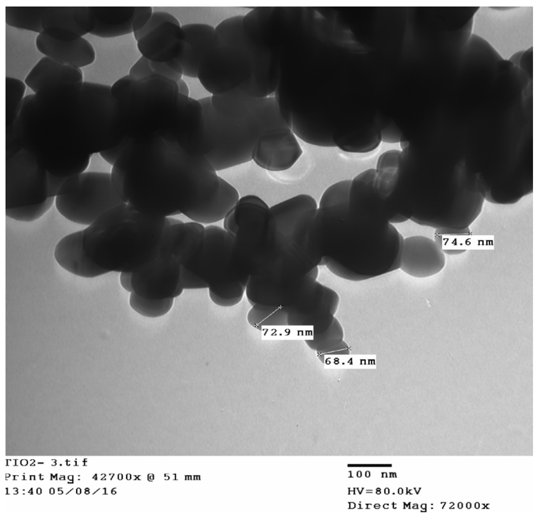

Fig. 3. TEM images of TiO_2 nanoparticles.

SEM Micrographs

Figure 4 illustrates SEM morphology of OWPC and LG hydrated till 28 days. The morphology of limestone pozzolanic cement differs than that of OWPC. This attributed to the formation a lot of crystals adhered to polished clinker surface like the honeycomb; these small crystals are calcium silicate hydrate fibers C-S-H gel as a result of pozzolanic reactions of calcium carbonate with micro sand [1 and 26]. Morphology of LG4 paste differs from other pastes, polished surface with compact ultrapure C-S-H crystals formations which enhance the strength and lower porosity textures. Typical hydration products are identified and they are: a)ettringite (needle-like crystals), b)calcium silicate hydrate (gel-like flocks) c) calcium hydroxide (plant-like crystals).

Figure 5 shows that the morphology of limestone pozzolanic cement pastes blended by nano-TiO_2 differs than other pastes. Huge crystals quantities formed on clinker surface differ than the other crystals formed as result of pozzolanic

TABLE 4. Properties of TiO_2 nanoparticle.

TiO_2 nanoparticle	Average diameter / nm.	Purity %
	79-90	99

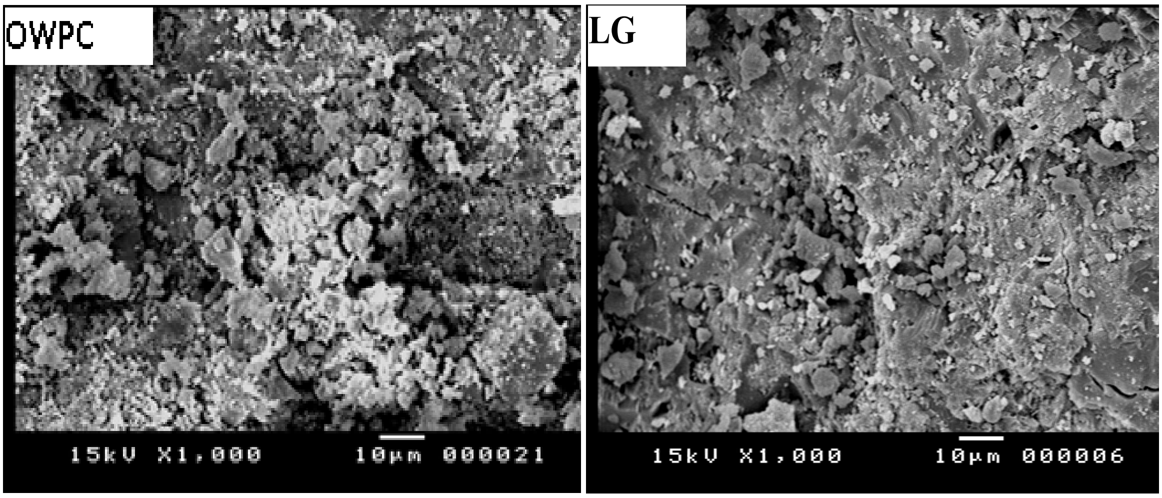

Fig. 4. illustrates SEM micrographs of OWPC and LG hydrated for 28 days.

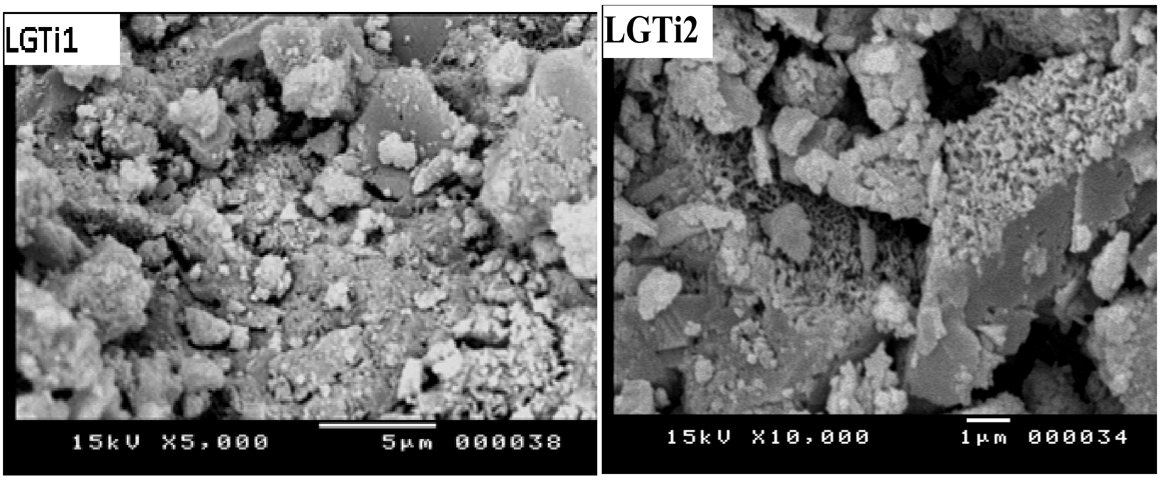

Fig. 5. SEM micrographs of LG sample blended by TiO_2 nanoparticles hydrated up to 28 days.

reactions this can be attributed to the great influence of nano-TiO_2 on crystal morphology during hydration process where C-S-H gel observed as honeycomb-like. The formation of calcium titanium silicate and calcium titanium oxide as a result of heterogeneous nucleation reactions of calcium carbonate with TiO_2 nanoparticles according to nucleation theory [27,28].

XRD Analysis

Figure 6 Illustrates XRD patterns of Ti-1 and Ti-2 hydrated for 28 days. The figure showed that the content of portalndite, calcium di-silicate, and calcium tri-silicate increases with hydration till 28 days as titanium nanoparticles content increases. TiO_2 nanoparticles behave as a complementary filler and nuclei enhancing extra hydration products. So TiO_2 nanoparticles enhance compressive strength forming calcium titanium oxide ($CaTiO_5$), calcium titanium oxide silicate ($CaTiSiO_5$) which has high mechanical strength properties [25]. The XRD patterns (Fig. 6) show that 1.0 % of titanium nanoparticles enhancing the strength better than 2.0% due to the fact that that nano titania will fill the pores of the cement pastes without altering its physical or chemical properties. Therefore mechanical strength becomes stable after early hydration (i.e. titanium nanoparticles enhance the strength at early hydration days).

Compressive Strength

The optimum mix percentage is for sample LG since it has high density, lower the porosity consequently lower the free lime content. The increased hardness of LG sample may be attributed to the following:

1) The increase of limestone contents (10.00%) of limestone pozzolania cement mixtures enhance the combination of sand with liberated lime to form excess hydration product

2) The Limestone content 10.00% is an important factor in the hydration process of C3A, C3S and β-C2S in the presence of $CaSO_4$ and white sand.

The compressive strength of LG sample blended by TiO_2 nanoparticles pastes is depicted in Fig. 7 as a function of hydration ages. It's found that at 3 days of hydration there is no remarkable change in compressive strength while after 7 days strength increased more than LG sample and as TiO_2 nanoparticles increased the strength increased as explained in the morphology of nano-TiO_2 which forms gel C-S-H like a honeycomb on the pastes

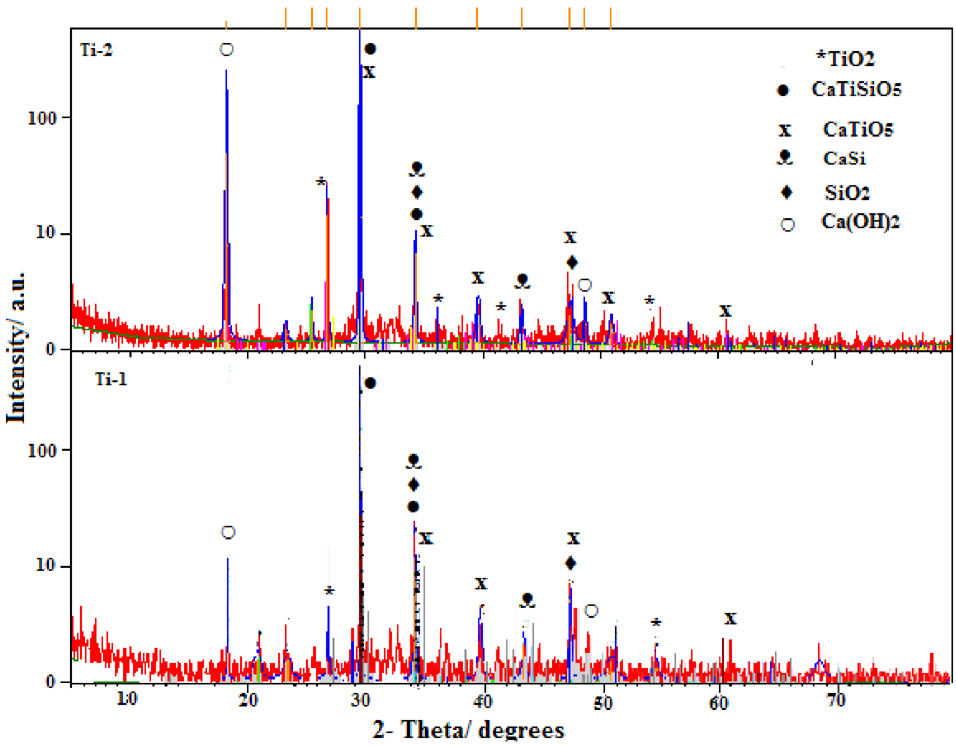

Fig. 6. XRD patterns of LGT-1 and LGT-2 hydrated for 28 days.

surface [29]. But at later days of hydration 28 days the compressive strength increased for LGT-1 sample inspire of LGT-2 sample shows stable behavior this due to the amount of hydration product $Ca(OH)_2$ are not equivalent to the excess amount of TiO_2 nanoparticles (Wt.%) so strength reaches its optimum level at 1.0% [30].

Free Lime Content

Figure 8 Illustrates the free lime content of OWPC, L1 T1 and L1T2 samples blended by TiO_2 nanoparticles pastes. The free lime content increases with curing time and limestone content. The increased liberation of Portlandite with time is due to the hydration process of β-C2S and C3S.

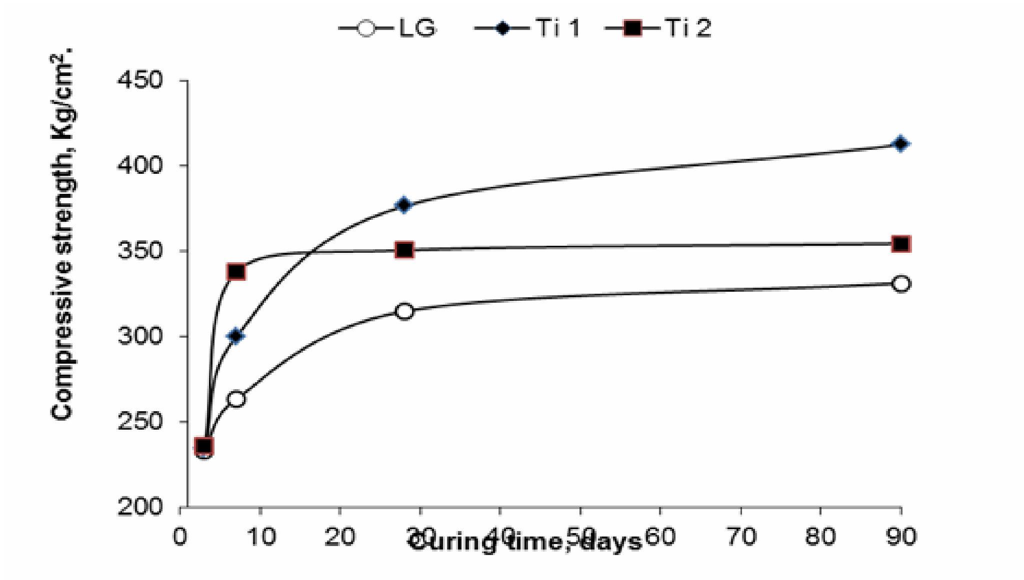

Fig. 7. Compressive Strength of LG sample blended by TiO_2 nanoparticles.

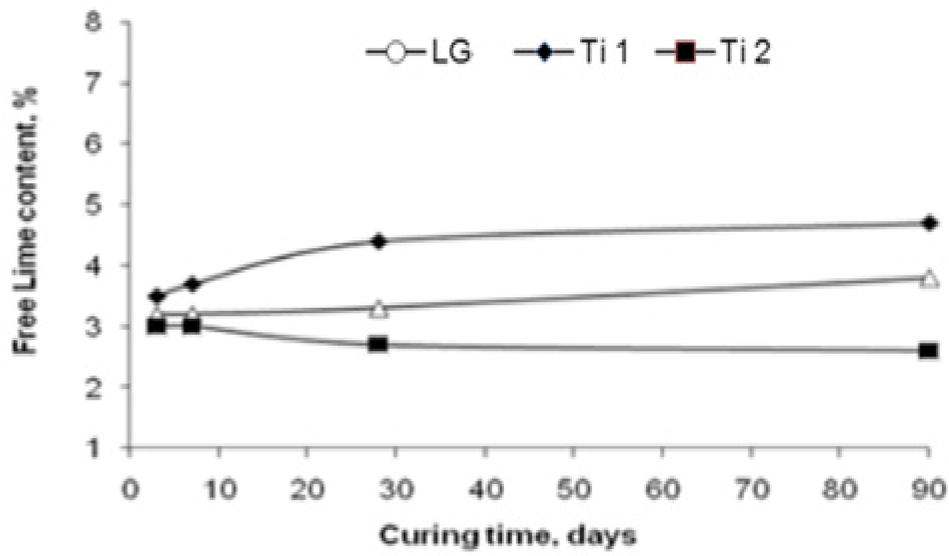

Fig. 8. Free lime content of LG sample blended by TiO_2 nanoparticles pastes.

On the other hand, the increase of Portlandite with limestone content may be also due to some leaching of Ca^{2+} from $CaCO_3$. The decrease in free lime content of limestone cement blended by TiO_2 nanoparticles could be due to the consumption of lime during the formation of more hydration products which dense the paste and decrease its porosity [31].

Bulk density and Total Porosity

In Fig. 9 & 10, the variation of bulk density and total porosity of LG sample blended by TiO_2 nanoparticles pastes as a function of hydration ages are plotted. The bulk density of cement pastes incorporating by TiO_2 nanoparticles increases with curing time for all the cement pastes due to the man-sized particles which act as effective fillers of voids hardened the pastes.

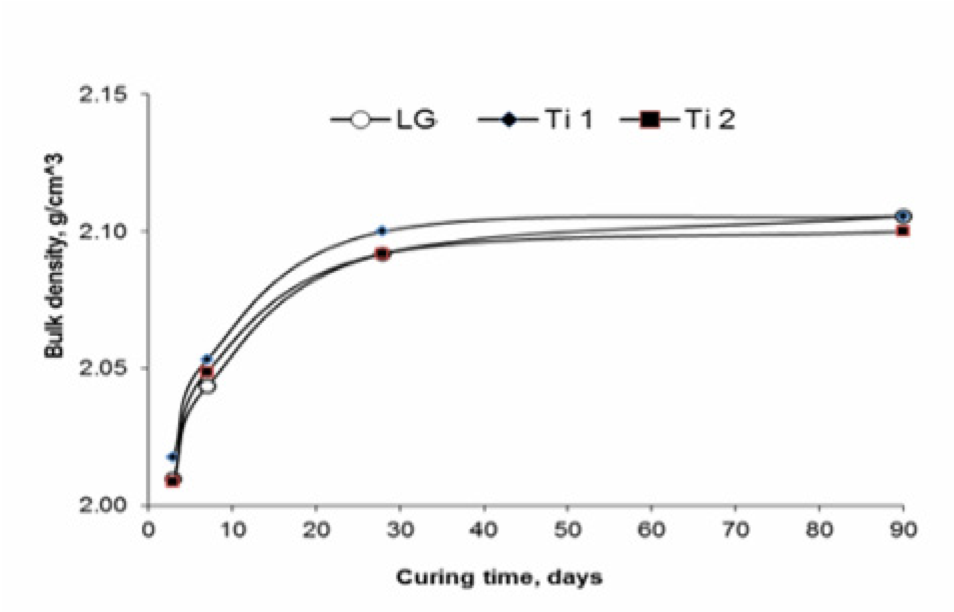

Fig. 9. Bulk density of LG sample blended by TiO_2 nanoparticles.

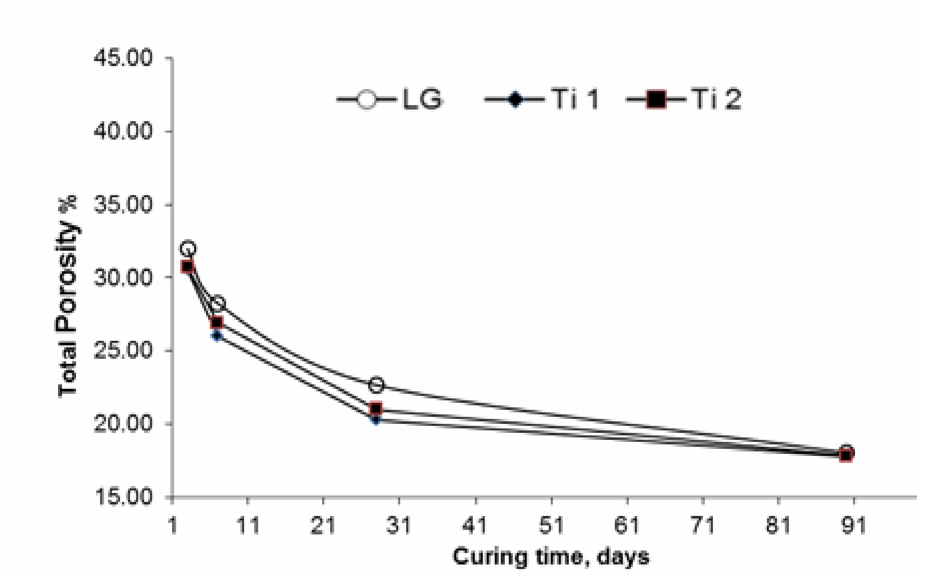

Fig. 10. Total porosity of LG sample blended by TiO_2 nanoparticles.

Whereas the total porosity improved with TiO_2 nanoparticles content due to of TiO_2 nanoparticles were enough to fill all the pores of cement pastes. Continues hydration of pastes nano- TiO_2 act as active nuclei that expand filling the pores inside the cement texture where these nuclei accelerate the hydration and more hydrated products accumulated in cement pastes bulked it [32,33].

Conclusions

Limestone combined with micro sand as fillers reinforced OWPC compressive strength, combined water content and bulk density. This attributed to the pozzolanic effect of micro sand that replaces clinker and increases the amount of gel C-S-H hydration products plus the filling effect of limestone. The morphology of limestone pozzolanic cement pastes blended by nano-TiO_2 differs than other pastes. Huge crystals quantities formed on clinker surface differ than the other crystals formed as result of pozzolanic reactions with nano-TiO_2 during hydration process which as active nuclei site, C-S-H gel observed as honeycomb-like. The formation of calcium titanium silicate and calcium titanium oxide as a result of heterogeneous nucleation reactions of calcium carbonate with TiO_2 nanoparticles according to nucleation theory.

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استخدام النانو تيتانيوم مع جسيمات الميكروسيليكا لتحسين خواص اسمنت الحجر الجيري

التحقيق الفيزيائي الميكانيكي

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تمت دراسة تأثير النسب المختلفة لملئ الفراغات بعجائن الاسمنت مثل الحجر الجيري والرمل الأبيض في حجم الميكرون، بالإضافة إلى جسيمات النانو- تيتانيا ودراسة الخواص الفيزيائية-الميكانيكية للأسمنت الحجر الجيري البورتلاندي الأبيض (OWPC). مزيج واحد (١٠,٠٠٪ بالوزن) من نسب متساوية من الحجر الجيري والرمل الأبيض في حجم الميكرون، تم استبدالهما جزئياً بكلنكر الأسمنت. قد تم التحقق من القوة الميكانيكية، والترابط الكيميائي لعجائن الاسمنت المخلوط والتي أظهرت أن المالئات تقلل من زمن الشك والمسامية الكلية ومحتوى الجير الحر، وتحسن من القوة الميكانيكية للانضغاط، ومحتوى ماء الخلط، والكثافة الظاهرية لعجائن الإسمنت بسبب التأثير البوزلاني للرمل الأبيض التي في حجم الميكرون والتي يحل محل الكلنكر وتعزز من منتجات التهدرد لعجائ الاسمنت. يمكن القول بان الحجر الجيري يملأ المسام بين جزيئات الأسمنت بسبب تكوين الكربو-اليومينات، في حين أن الرمل في حجم الميكرون يزيد من تهدرد منتج الألياف C-S-H ويحسن من خواص الاسمنت. وفي دراسة متقدمة اظهرت الجسيمات النانوية TiO_2 (أي أقل من ١٠٠ نانومتر) كفاءة كبيرة في تعزيز القوة الميكانيكية للانضغاط لعجينة الاسمنت بسبب كفاءة دمج الجسيمات النانوية كمواقع نشطة لنمو الألياف C-S-H أثناء التهدرد للاسمنت.