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Study the Effect of Addition nano-TiO₂ by Dispersion Method on the Some Mechanical Properties and Durability of Cement Mortar

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Abstract. The main aim of this study consists of the influence of nano-particle on some mechanical properties of cement mortar with and without nano particles replacement mortar was experimentally studied. The nano-particle used in this study was nano- Titanium dioxide (nano-TiO₂) with the particle size of 30nm. This study involves the mixing method of nano-materials addition and interventions with cement mortar behavior for many mortar samples under variable curing time with constant (W/C=0.45). Some mechanical properties like (specific compressive strength, split tensile strength and water absorption tests) was studied. In this study the main parameters depend include the small amount replacement ratio of nano-particle (nano-TiO₂) and dispersion methods with respect to the Ordinary Portland Cement (OPC) type (I). The percentage of nano-material replacement on the mixture of cement mortar includes (0, 0.5, 1.0, 1.5 and 3%) for nano-materials with constant W/C ratio and also the amount of the fine aggregate use 2.75 from the amount of cement. The results show that, the strength of the mortar consist 1% and 1.5% TiO₂ nano-particles are desired than traditional mortar. The results show that, the strength of the mortar consists of with ultrasonic dispersion of colloidal nano materials give better properties than reference specimens in all test. But the Nano-Titanium dioxide - materials give good properties up to 1.5%.

Keyword: Compressive Strength, Split Tensile Strength, Nano- TiO₂, Water Absorption Tests.

1. Introduction

Recently nano technology is being used in many applications and it has received increasing attention in building materials. Number of works deals with using of nano size particles in cement based materials which is used in constructions [1]. There is a few knowledge about nano particles effects on the properties, consistency, times of setting, rheological, micro structural, workability and mechanical properties of cementitious mixes. Additionally, the literatures appear to be contradictory about of the effects of nano particles on the development of building materials [2, 3]. The effects of nano additives like (nano-Fe₂O₃) and (nano-SiO₂) on cement mortars properties must be experimentally studied. The results of experimental will shows the affected properties measured (for example: at the (7th) day and (28th) day) of the cement mortars mixture with the nano particles addition compared with of a plain cement mortar. The microstructures study (SEM) between two mixture (the cement mortar mixed with the nano particles and the plain cement mortar) illustrates that the (nano-SiO₂) and (nano-Fe₂O₃) filled up the voids and consumed (CaOH₂) compound during the reaction of hydrates. Supreme mechanical performance of the cement mortars with nano particles addition are explained by this mechanism [4]. The permeability and the mechanical properties of (porous-concrete containing (nano-SiO₂)-(PCNS)) were studied, researchers studied also (plain porous concrete (PPC)) and (porous concrete containing (Micro-SiO₂)-(PCMS)) as control materials. Results for (PCNS) specimens cured for (28 day) showed higher compressive and flexural strengths comparing to those of (PPC) with the same (W/C). It was shown that the (Nano-SiO₂ (NS)) were more influence than (Micro-SiO₂ (MS)) in



enhancement of (porous concrete (PC)) flexural strength. Specimens with (5% NS) by weight of binder shows highest mechanical properties in flexural and compressive strength tests for (porous concrete (PC)) and were enhanced by (56% and 48%), respectively. Experimental results showed that the permeability behavior of (PCNS) and (PCMS) decreased, results show that (Nano-SiO₂) (NS)) and (Micro-SiO₂) (MS)) could both reduce water penetration through (porous concrete (PC)); however, (Micro-SiO₂ (MS)) were found to be more effective for equal nano particles and material voids [5]. Casting of ferro cement elements reinforced with (nano-SiO₂ (NS)) particles were investigated and specimens were tested to determine their mechanical properties, durability and microstructural of interfacial transition zone (ITZ). The amounts of replacement ratios of (nano-SiO₂) (NS)) particles were low with respect to cement in (Ordinary Portland Cement (OPC)) mortar mixture. Ratios were (1% to 3%) (Nano-SiO₂) (NS)), water to binder ratio (0.35, 0.4 and 0.5) and sand to binder ratio (2 and 2.5). Results illustrates that the cement mortars consist of nano particles have higher strength, low water absorption and denser (ITZ) compared to those of the (OPC) ferro cement mortars [6]. The effects of other nano particles on the durability and the mechanical properties of concrete had been explained by adding constant content of (nano-ZrO₂ (NZ), nano-Fe₃O₄ (NF), nano TiO₂ (NT) and nano-Al₂O₃ (NA)) to concrete mixtures. Mechanical properties have been explained through the compressive and splitting tensile strength and durability has been explained through chloride penetration test and concrete permeability. Results showed that nano particles can be very influence in development of both mechanical properties and durability of concrete. The results explained that (nano-Al₂O₃) is most effective than other nano particle in the developing the mechanical properties of high performance concrete [7]. Waste ground ceramics as a pozzolan material in concrete was used in two different phases and were investigated. Concrete specimens with substitution percent about (10–40%) of ground ceramic powder were made. Then the simultaneous influence of using (10% to 25%) of ground ceramic powder and from (0.5–1%) of nano Silica were determined. In all status, both compressive strength and water absorption tests were achieved. Results observed when the ground ceramic addition up to (20%) which was found to be don't have a significantly negative effect on the concrete compressive strength. However, water absorption capacity in the concrete reduces by using any amount of ground ceramic. Also when nano-SiO₂ was added improvements in compressive strength and reducing in water absorption capacity occur. Therefore, nano-SiO₂ can be improved the influence of ground ceramic powder on concrete properties [8]. Two types of nano silica (NS) were studied and used in self-compacting concrete, both of these nano additives have the same particle size, but differs in producing processes: one of these are silica fumed powder and the second types was colloidal suspension precipitated silica. The effective of nano silica on (self-compacting concrete) were investigating with respect to the properties of concrete in fresh (workability) and hardened state (mechanical properties and durability). Microstructure of the hardened concrete were inspected by (SEM) and (EDS) analyses. The obtained results shows that nano silica used in (SCC) can improve both the mechanical properties and durability of self-compacting concrete. Taking in account the reactivity of the two additions, the colloidal nano silica type showed a higher reactivity at fumed powder, which effects on the final properties of (SCC) [9]. The mechanical properties and the durability were studied for (self-compacting concrete (SCC)) containing alginate in variety values with artificial stone resin, micro and nano silica. The values of (0.5 and 1%) alginate, (10%) micro silica, (0.5%) nano silica and (0.5%) artificial stone resin were used. Artificial stone resin was used as the super plasticizer. Properties of hardened (SCC) such as compressive, split tensile, flexural strength and water absorption were assessed and represented graphically. In general, the use of alginate improved the performance of (SCC) in fresh state and also avoid for the use of viscosity modifying admixtures. Adding nano silica to samples increased (SCC) and both (workability and the concrete split tensile strength) decreased in (0.5%) alginate in all mixes. Adding micro silica to alginate increases of the split tensile strength while adding nano and micro silica decreases the values of water absorption [10]. The aim of this research involves to study the effect of dispersion methods for nano materials addition to cement mortar, and how it effects on the some mechanical properties and durability of cement mortar when nano material addition by dry and colloidal state.

2. Experimental Work

2.1. Materials

There are many materials which are used to prepare specimens these materials consist of cement, fine aggregate, water and nano material.

2.2. Cement

In this work the main components in the all mixture Ordinary Portland Cement (type I) was used. The most major and minor components of cement used are described in Tab.(1). Cement corresponds to the specification of Iraqi No.5/1984 [11] ordinary Portland cement (type I) from Al Mass Iraqi cement factory, the test was achieved in National Centre for Construction Laboratories (NCCLR) laboratory and research.

Table 1. The most major and minor components of ordinary Portland cement (OPC).

Oxides Composition	Oxide content%	Limits of Iraqi
		Specification No.5/1984
SiO ₂	20.26	-
Al ₂ O ₃	5.50	-
Fe ₂ O ₃	2.19	-
CaO	61.39	-
MgO	1.99	< 5.00
SO ₃	2.7	< 2.8
Free CaO	1.12	-
Loss on Ignition	3.2	< 4.00
Insoluble Residue	0.73	< 1.50
Lime Saturation Factor	0.94	0.66-1.02

Table 2. Chemical Main Component, Weight %, for Cement Type (I).

Component	Formula	% by weight of Cement	Limits
C ₃ S	3 CaO.SiO ₂	55.81	—
C ₂ S	2 CaO.SiO ₂	16.04	—
C ₃ A	3 CaO.Al ₂ O ₃	10.87	0–15%
C ₄ AF	4 CaO.Al ₂ O ₃ .Fe ₂ O ₃	6.66	—

2.3. Fine aggregate

Fine aggregates generally consist of natural sand or crushed stone with most particles smaller than (5 mm). The most desirable fine-aggregate grading depends on the required of work. The sand grading analysis was conforming to the Iraqi Specification requirement of No.45/1984 [12], as illustrated in table (3). AL-Ekadir in Karbala region sand was used as fine aggregate. It was tested to determine the grading and other physical properties. The sand used in this study is according to the standard specifications.

Table 3. Sand grading and requirements.

Sieve Size (mm)	Accumulative Passing (%)	Accumulative Passing (%) According to Limits of I.O.S No.45/1984
4.75	100	90-100
2.36	100	85-100
1.18	87.22	75-100
0.60	67.85	60-79
0.30	28.53	12-40

2.4. Nano materials

The nano- Titanium Oxide particles (TiO_2) is used. It has high purity approach to (99.5%), the particle size rounded range between (10 to 30 nanometer) from Sky Spring Company. Some properties of nano- TiO_2 are shown in table (4):

Table 4. Titanium Oxide nano-particles (TiO_2) properties

Titanium Oxide nano-particles (TiO_2) certificate analysis						
Product Name	symbol	Purity %	particle size nm	SSA (m^2/g)	Color	True Density (g/cm^3)
Titanium Oxide	TiO_2	99.5%	10-30	50-150	white	4.23

3. Mortar Preparation (casting and curing)

The mix proportion of the mortar was forming using (C: S ratio) by 1:275 for Iraqi cement standard (IOS NO.8). The water/cement ratio is ($w/c = 0.45$). The mix of cement mortar was done by replacement addition of nano-materials particles from the weight of cement. In order to reach complete homogeneity all components were homogenized by electric mixer. The cement mortars were molded into 50 mm cubes for compressive strength; the dimensions for the cylindrical (50 x 100) mm are used to splitting tensile strength samples and the molds dimensions for water absorption test was (20*20*20) mm^3 . The specimens are remained in the molds for 24 hours with 100% relative humidity, and then cured in water for (3, 7, 14, 21, 28, 60 and 91 days).

4. Testing methods:

4.1. Compression Strength Test

The compression strength tests were done on 50 mm cube specimens according to ASTM C 109/C 109M – 07 [24]. The compressive machine (ELE machine device) was used with maximum load is 2000 KN. The rate of loading on the cubes was 1.0 mm/min. Three samples were tested for each, and the average strength was recorded.

4.2. Splitting Tensile Test

The flexural test machine was achieved by Tinius Olsen universal material machine device with (100 KN) load is applied in strength of materials laboratory in materials engineering department in materials engineering department/Al-Mustansiriyah University. The recorded final of the all results obtained was represented the average of splitting strength from many samples. The dimension of prism was done according to ASTM C496/M496 [25].

4.3. Water Absorption Test

The absorption test was carried out according to ASTM C642-2010 [26]. A 20 mm cubic specimen used throughout this test. Equ. (1) Used for the absorption calculation.

$$\text{Absorp. \%} = ((B-A) / A) * 100 \quad (1)$$

Where: A= dry weight by oven (gm), B= dry weight at saturated surface (gm).

5. Results and Discussion

5.1. Compressive Results

The results of compressive strength specimen's tests without and with nano-materials replacement to cement mortars are illustrated in figures together in order to show the effect of nano-materials addition on the compressive strength behavior. The control samples in figure 1 show the variation of days (3, 7, 14, 21, 28, 60 and 91 days) with compressive strength. The blended mortar for 0.5% nano-(TiO₂) replacement is show in figure 2. The compressive strength appears little increment in compression strength compare with different curing time. Compressive strength is small increase more than control samples with the same W/C ratio.

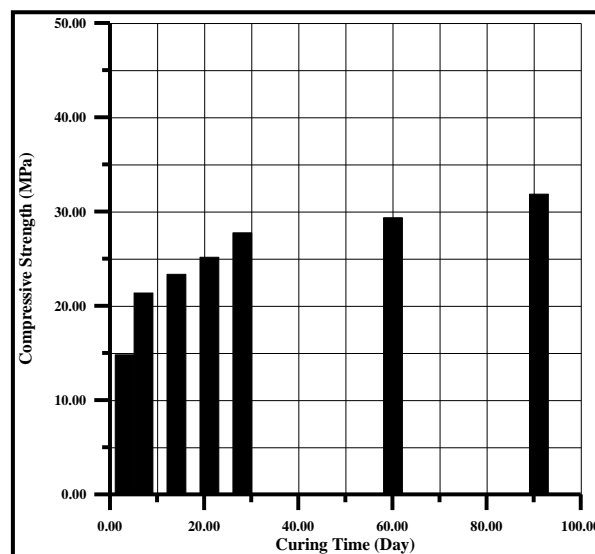


Figure 1. Compressive strength for control mortar

At 0.5% nano-(TiO₂), the compressive strength increases when curing time increase with the same W/C ratio and the compressive strength properties has improved than the control cement mortar at different curing time this behavior was show in figure 2. The compressive strength slow increase of cement mortar with increasing nano (TiO₂) replacements reach to 1.0% the compressive strength behavior can be illustrating in figure 3.

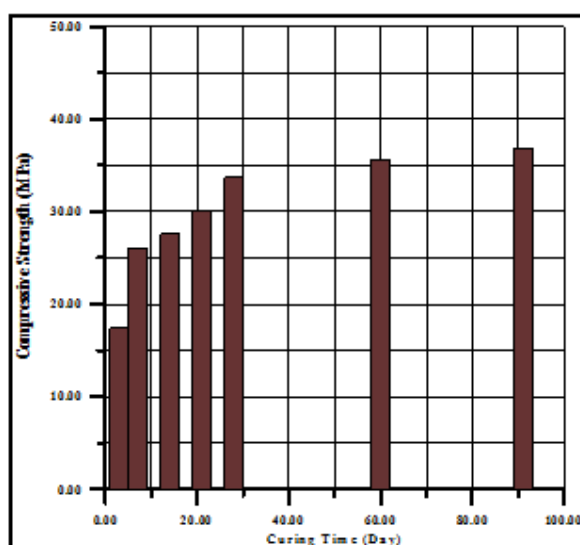


Figure 2. Compressive strength for 0.5%

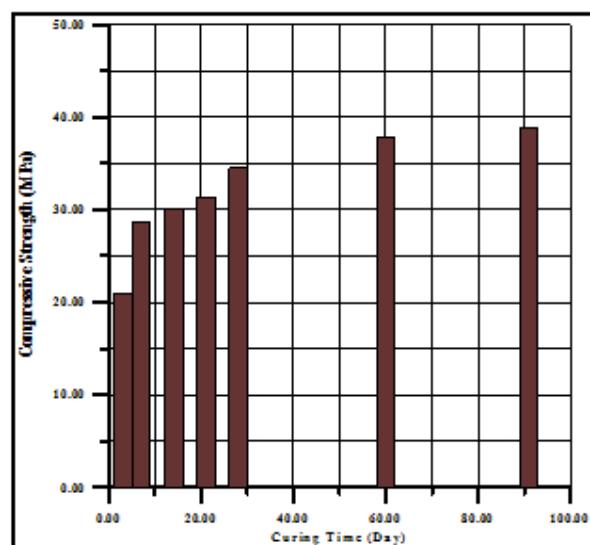


Figure 3. Compressive strength for 1.0 %

But, when the loading of nano-(TiO₂) replacement reach to 1.5% the compressive strength of mortar with nano addition begin to give better compressive strength behavior compared with control. The mechanical property (compressive strength) development at 1.5% colloidal nano-(TiO₂) replacement is can be seen in figure 4. Basically the enhancement in compressive strength because the packing effect of filling the voids that create during the hydration reactions and the materials become more dense and the compressive strength increases.

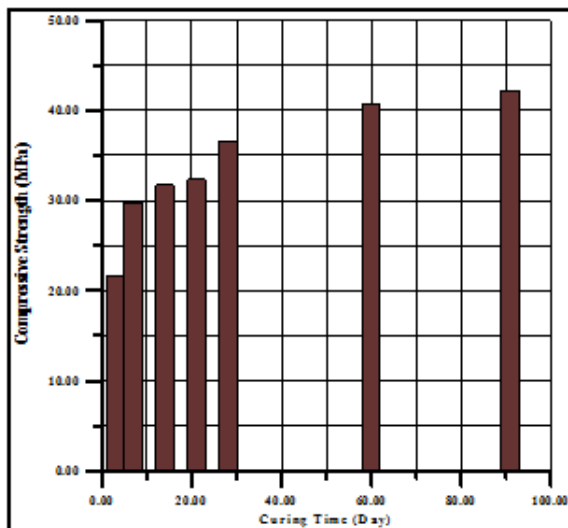


Figure 4. Compressive strength for 1.5 %

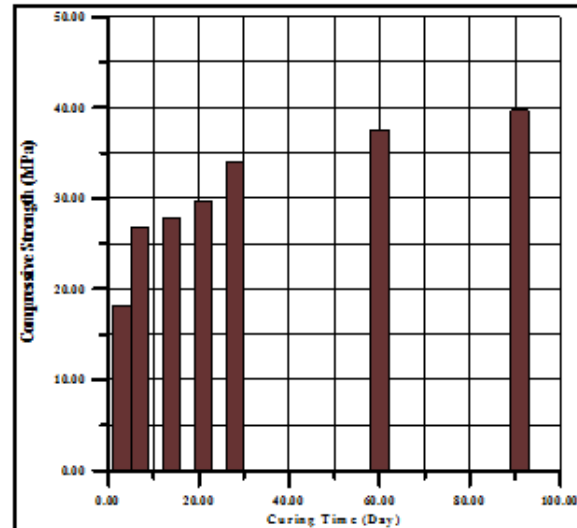


Figure 5. Compressive strength for 3.0 %

The compressive stress it is predictable that effect of TiO₂ nano-particles at 1.5% addition to cement mortar can be has activation effect as a filler material in order to give more in strength of the micro-structure of mixture and also reduces the amount and size of crystals (calcium hydroxide) and the gel structure (C-S-H) was fill the voids and finally, hydrated structure product is compacted and more dens. When nano-particles increase up to 3.0% was illustrate in figure 5 the reduction in nano-particles dimension and the (calcium hydroxide) crystal due to limited space cannot sabuline to suitable dimensions. the agglomerated of nano-particles causes the most properties of the specimens at 3.0% nano-particles is inferior to the control mortar sample.

5.2. Splitting Tensile strength Results

Mortar molds tested in accordance with ASTM C496/M496 [25] which found the Splitting tensile strength and then in compression between control and nano-materials replacement. All the mortars made without nano-oxides showed flexural strength results below. The reference mortar made without replacement shown in figure 6.

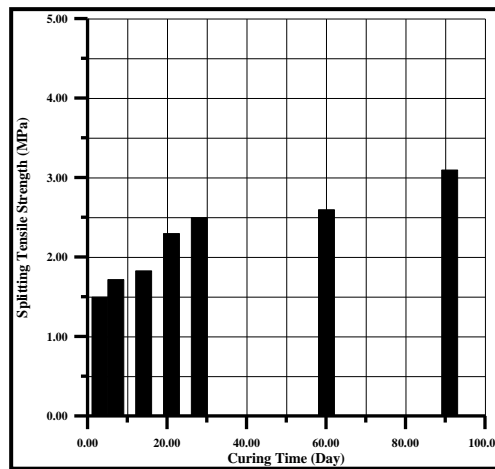


Figure 6. Splitting tensile strength for control samples

Splitting tensile strength of control mortar with 0.5% nano-(TiO₂) (Colloidal) mortar, at the first time of curing the nano-TiO₂ mortar slightly higher than that for control mortar (about 0.5 %) the Splitting strength behavior can be illustrating in figure 7. The effect of nano particles increment 0.5% nano (TiO₂) can be shown in figure 8. with 1.0 % nano (TiO₂) (Colloidal) the Splitting strength improved these behaviors can illustrate in figure (9). Also, more generation of strengthening gel could improve the defects of dispersion method of TiO₂ nano-particles. In addition, TiO₂ nano-particles recovered the particle packing density of the blended cement, directing to a reduced volume of larger pores in the cement paste. These results usually, nano- TiO₂ helps to improve the mechanical performance of cement mortars due to the filler effect of replacement this can be appear in figure 9. Therefore, the results reported in this paper suggest that the mixing conditions play an important role with regard to the splitting strength.

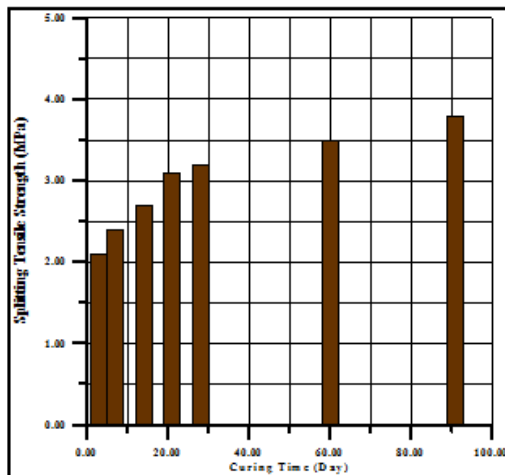


Figure 7. Splitting strength for 0.5 % nano-TiO₂ (Colloidal)

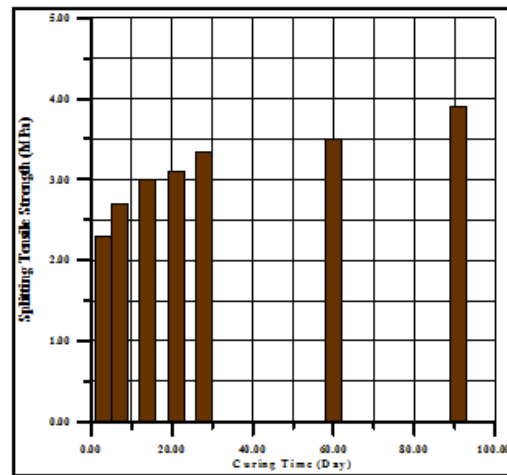


Figure 8. Splitting strength for 1.0 % nano-TiO₂ (Colloidal)

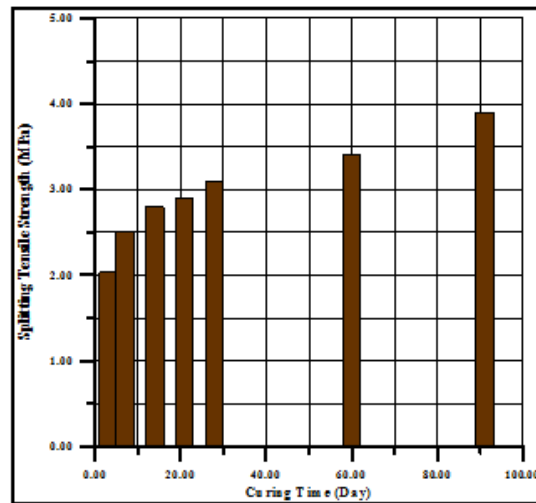
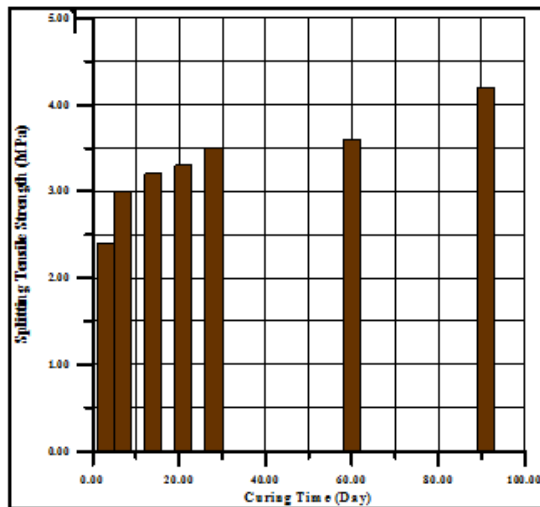


Figure 9. Splitting strength for 1.5 % nano- TiO₂ **Figure 10.** Splitting strength for 3.0 % nano- TiO₂

Then the behaviors of splitting strength decrease when the nano (TiO₂) (Colloidal) (addition increases up to 3.0 %. This case observed in figure 10. The results show that the addition of TiO₂ nano-particles, increasing amount of splitting strength is more than control samples. The agglomeration effect with more nano-(TiO₂) particles will be causes the lower than the control mortar sample in most properties when the replacement reach to 3.0 % nano oxide. Thus, the influence of agglomeration of nano-(TiO₂) particles and some non-desirable on the entire structure will causes some locally cracks and ultimately reduces the most properties.

5.3. Water absorption results

One of the significant tests to measuring the durability of mortar or concrete is water absorption test. The total value of the water absorption will give information about the durability of mortar or concrete were water fill and enter into the voids of mortar and saturates the matrix pores. Figures (11 and 12) can illustrate a comparison for the results of water absorption test between the control mortar and specimen contain replacement of nano- Titanium oxide (TiO₂). It was found that water absorption decreases in specimens contain (0.5% nano-Titanium oxide (TiO₂), compared with control samples.

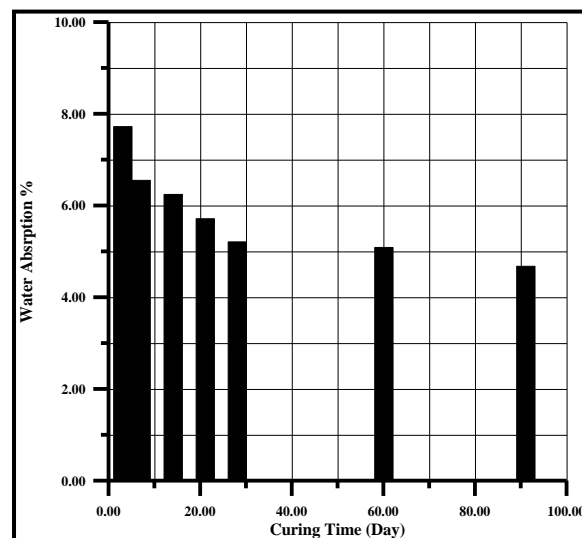


Figure 11. Water absorption for control samples

The results give the indication that, when the titanium-nano particles value increases, the water absorption will be decreases. This behavior can be seen in figures (12, 13, 14 and 15). When the amount of nano-(TiO₂) reaches (0.5%) as seen in figure 12 water absorption decreased more than control samples. At (0.5%) nano Titanium oxide (TiO₂) as seen in figure 12, water absorption values are decreases and becomes less than control samples in all curing time. When the amount of nano-Titanium oxide (TiO₂) increases to (1.0%) replacement this illustrate in figure 13, the water absorption reaches its maximum value, this is illustrated in figure 14, nano Titanium oxide (TiO₂) effect for different curing time. The materials at (1.5%) nano-Titanium oxide (TiO₂) gives the optimum behavior of water absorption, this is because the parameters that controls the decrease on water absorption is the capacity of filling process, activating of pozzolanic reactions and the hydration rate of the cement, all these will create decrease and the porosity and the materials become denser than control specimen. Smaller nano particles react faster than the nano-particle size, the more the heterogeneous nucleation sites gives lower water absorption; therefore, the nano particles must be dispersing as possible as to give the required improvement for the mortar properties in order to prevent the agglomeration. Figure 15 represented the water absorption of nano (TiO₂) replacement at (3.0%). These illustrate the water absorption which begins to increase and near to control specimens. Defects may be generated during the dispersion of nano particles that causes weak area.

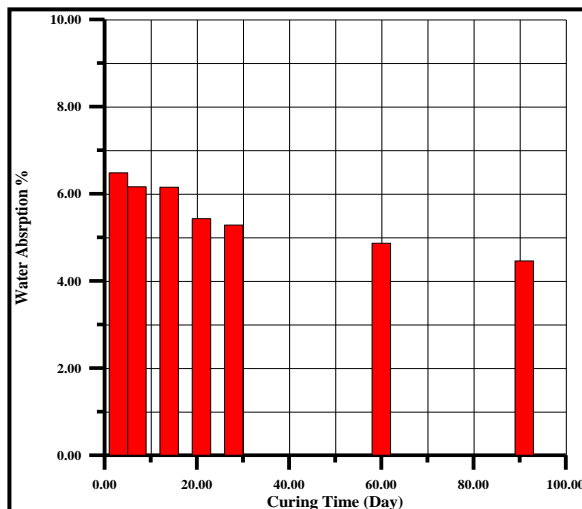


Figure 12. Water absorption for (0.5 % nano- TiO₂)

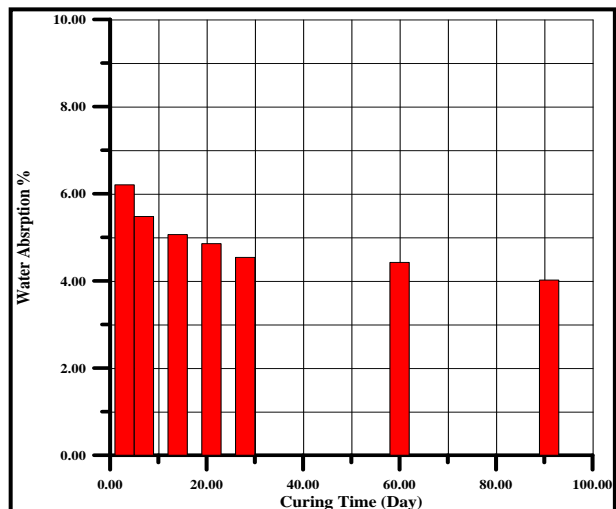


Figure 13. Water absorption for (1.0 % nano- TiO₂)

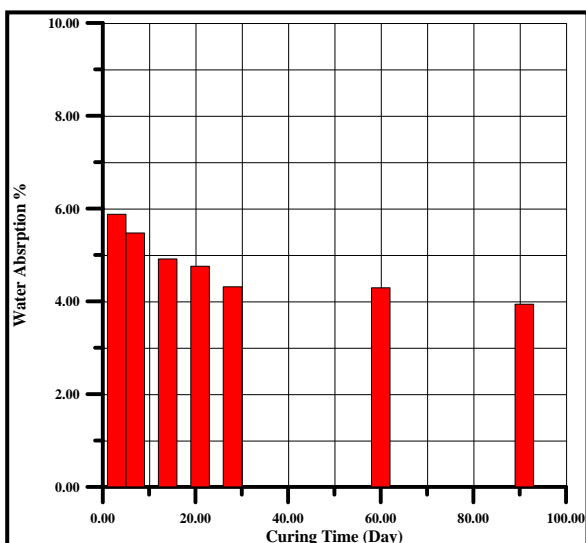


Figure 14. Water absorption for (1.5 % nano- TiO₂)

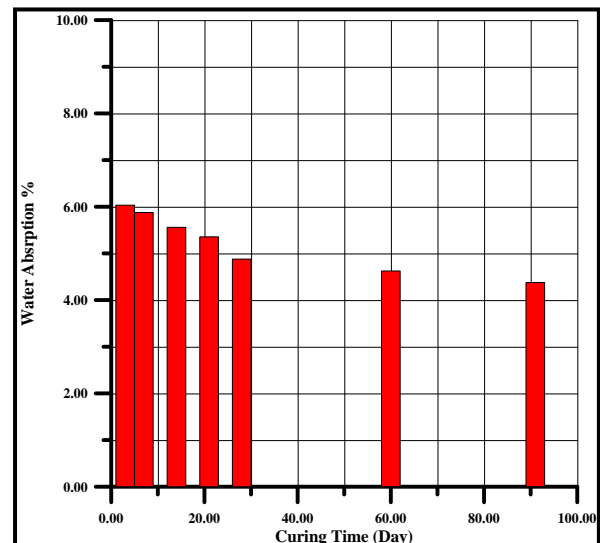


Figure 15. Water absorption for (3.0% nano- TiO₂)

6. Conclusions

Based on the mechanical properties obtained by the tests on a reference cement mortar and nano-replacement mixes containing nano-TiO₂ (colloidal). In general, the present study showed that there is an improvement in the mechanical and some physical properties of mortar. This can be attributed to many factors, like mixing ratio as well as the type of the nano-materials used. The following conclusions can be drawn according to the results from the experimental work, following points can be concluded:

1. The TiO₂ nano-particles have a filler influence which strengthens the microstructure of a cement mortar and the porosity is reduced and the density of the cement mortar is increase. Both, the main products of hydration and the cement hydration rate did not affect by nano-Titanium oxide.
2. Colloidal dispersion method for nano-particles in cement mortar is uniformly each particle has a uniform manner and spacebetween the nano-particles is modifying. Then, the process hydration of cement and the product of hydration are distributed and surround the nano-particles as the nucleus.
3. Titanium oxide nano-particles up to 1.5 % replacement by cement weight can it's have as a filler influence for the micro-structure strengthening of cement and also reduces the amount and measure of Ca(OH)₂ crystals and structure of hydrated product is becomes more compacted and denser because the voids is filled by (C-S-H) gel structure.
4. Because of the size of nano-particles the material has high surface energy this due to tendency towards agglomeration when especially when the amount of nano particles (TiO₂) reach to 3.0% replacement. When nano-particles are more the critical value addition to the cement mortar, it is not have uniformly distribution of nano particles in cement mortar then the agglomeration and weak zone is appear in the cement mortar.
5. The compressive strength of colloidal Titanium oxide improved at 1.5% at all curing time than control samples.
6. The water absorption was enhancement of water absorption by (1.5%) nano-TiO₂ replacement at all curing times above controls mortar.
7. The enhancement of splitting strength by nano-Titanium-oxide replacement was achieved especially at (1.5%) replacement at all curing time.
8. Colloidal nano-particles improve water absorption resistance of all specimens containing nano materials (nano TiO₂) compared with control specimens.

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