Journal of Engineering and Sustainable Development

المجلد 22، العدد2 (الجزء-1)، آذار2018
ISSN 2520-0917
10.31272/jeasd.2018.2.8

EFFECT OF NANO-ALUMINA ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF RECYCLED CONCRETE

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Abstract: In this paper, the effect of using recycle mortar as fine aggregate replacement and nano Alumina (Al_2O_3) powder replaced of cement on the concrete microstructure and their effect on the mechanical properties is investigated. The nano powder was used as additive to replaced (0.5, and 2. 5 wt %) of cement with recycle fine aggregate(RFA) to replacement (10,and 50 wt%) of natural fine aggregate in concrete mixture. The compressive strength and microstructure analysis tests were carried out on the concrete samples. Microstructure analysis tests were conducted using X-Ray Diffraction(XRD), Scanning Electron Microscopy(SEM), and Atomic Force Microscopy(AFM). Results shows that the nano-oxide and recycle aggregate addition leads to different microstructure of concrete. The high specific surface area of nano-oxide causes reduce calcium hydroxide $Ca(OH)_2$ (CH) crystallite in the cement hydration process and the pores structure of calcium silicate hydrated gel lead to products denser and compacted microstructure, also the roughness of concrete surface was reduces which caused enhancement in the compressive strength with the addition of nano- Al_2O_3

Keywords: *Concrete; Nano-Al_2O_3; Recycle fine aggregate; Microstructure, Compressive strength.*

تصنيع الخرسانة من المونة المعادة وتحقق في تأثير الإضافات النانوية على خواص البنية المجهرية والميكانيكية

الخلاصة: في هذه البحث تأثير استعمال المونة معادة كبديل عن الركام الناعم ومسحوق النانو ألومينا كبديل عن الاسمنت على البنية المجهرية و الخواص الميكانيكية للخرسانة تم التحقق منه. المسحوق النانوي تم استعماله كإضافات لتبديل (0.5، و 2.5)% من وزن الاسمنت مع الركام الناعم المعاد كبديل عن(10، و50)%من وزن الركام الناعم الطبيعي في خلطة الخرسانة. اختبارات مقاومة الانضغاط والتحليل البنية المجهرية التي تم أجراها لعينات الخرسانة. تحليل البنية المجهرية تضمن فحص انحراف أشعة كاما، فحص بالمجهر الالكتروني الدقيق،و فحص بالمجهر قوة الذرى. النتائج أظهرت إن إضافة النانو وكسيد و الركام المعاد يودي إلى اختلاف في البنية المهجرية لعينات الخرسانة. المساحة السطحية النوعية العالية للنانو وكسيد تسبب تقليل تكون بلورات هيدروكسيد الكالسيوم خلال عملية اماهة الاسمنت و تركيب الفجوات في جل الكالسيوم سيليكات هيدريات مما يؤدي إلى تكوين بنية ألمجهريه كثيفة ومضغوطة،وكذلك خشونة سطح الخرسانة تنخفض مما يسبب تحسين في مقاومة الانضغاط تزداد مع إضافة النانو ألومينا.

1. Introduction

Recycling process as branch of environmental considerations has become a familiar attribute in the construction business because the building and construction is a most important consumer of world's materials and energy resources which accounts about

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40% of custom and large waste materials amounts that results from the construction, renewal, or destruction of any erection, such as buildings, roads, and bridges.[1]

Recycling concrete has many benefits rather than removing it or burying it in a landfill keeping waste of concrete away from landfills saves landfill space, recycled material that could be used as aggregate diminishes the need for aggregate mining and using recycled concrete as the base material for roadways.[2]

Nano materials are important due to their special properties such as high surface to volume ratios, as the surface area per mass of a material increases, that lead to a larger amount of the material can arrive into contact with neighboring materials, therefore; nano materials are very reactivity. [3]

The recycled concrete aggregate could be classified by use into coarse aggregate and fine aggregate. The utilization of recycled concrete as a coarse aggregate (RCA) for the production of construction (about 85%) was used as road base and RCA was being increasingly used to replace natural aggregate in such road construction applications as concrete mix [4] .

While, Used recycled fine as 15%-20% replacement of the fresh cement is cost effective. The most effective scope to use the recycled fine (before sieving) is as the replacement of sand in the concrete mix. In that case , some additional cementing strength can be gained from the cementing property of recycled fine.[5]

The effect recycled fine aggregate (RFA) product from concrete waste on mortars properties were the mortars with dried recycled sand have higher slump than that of comparable mortars with saturated recycled sand. This is due both to higher initial free water and paste quantity in the mortars product with dried RFA. But the rate of slump loss is more rapidly when using dried RFA.

This may be because the higher initial slump value, and in addition due to the decrease of free water by the absorption of RFA. The mortars made with dried RFA have higher compressive strengths than the mortar with saturated aggregates. The fresh density of mortar decreases with RFA increase because the lower density of RFA. When RFA containing increased in mortar their compressive strength decreases compare with mortar made with natural fine aggregate.[6]

The microstructure examination was allows us to detect the presence of hydrate product on the aggregate surface such as calcium silicate hydrate (C-S-H) and aluminates hydrate products. Three different mechanisms can use to explain the reason for increase compressive strength when very fine recycled aggregate (RA) using as cement replacement. First mechanism, the (RA) small size help the particles to fill the interfacial transition zone between natural aggregates and the cement paste.

Second mechanism, the bond strength with the new cement paste and the hydrate product can increasing due to the dicalcium silicate (C_2S) and tricalcium aluminate (C_3S) present on the RA. Third mechanism Hydrate products on the aggregate surface result more bond between aggregate and new cement paste.[7]

The addition of ultrafine particles of nano materials was lead to fill the voids of concrete microstructure. Nano-powder improved the pozzolanic action by providing more surface area and produced the more cementitious product.[8, 9] Nano-silica(NS) was improves the recycled concretes microstructure. Nano-particles(NS) were using in recycled concrete blended develop the interfacial transition zone (ITZ) and cement paste in all features, may be produce a compact gel with smallest size voids which cause improvement in mechanical properties and recycled concretes permeability, and increase rates hydration and fill the voids of the C-S-H structure leading to a dense concrete.[10, 11]

In the present research the effect of different nano-oxide (Alumina) and recycled fine aggregate on concrete microstructure and mechanical properties are investigated.

2. Experimental Work

2.1. Materials

In this research Ordinary Portland Cement (OPC) was used which was produced by Iraqi Cement Factory (Tassloja)) was conformed to Iraqi Specification No.5/1984.

Natural fine sand was used and its grading is shown in Table 1 and it is within the limits Iraqi Specification No. 45/1984. Coarse aggregate and recycle fine aggregate were used to prepared concrete samples. All concrete samples were produced with same a water-to-cemented materials(cement and nano-Alumina (NA)) ratio (w/c) of 0.4. The concrete ingredients were mixed using 1:2:4 mixing ratio for cement, fine aggregate, and coarse aggregate materials, respectively.

The mortar waste was used as recycle fine aggregate to replace partially natural sand in the concrete. Two different percentages (10, and 50%) by weight of sand were used to replace fine aggregate. Natural fine aggregate (NFA) and recycle fine aggregate (RFA) (i.e. mortar waste) particle size analysis were made using sieving device as shown in Fig.1.

Table 1 Natural fine sand Grading

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
0.15	8.91	0-10

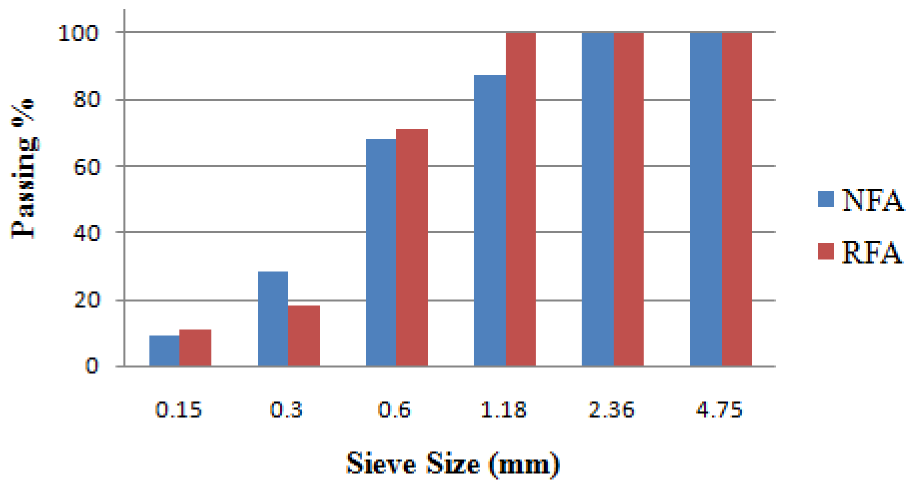


Figure 1. Partical size analysis for siveing for natural and recycle fine aggregate.

The nano ceramic oxide (Al_2O_3) was used for partial replacement of cement. the nano Al_2O_3 was purchased from (HWNANO brand, Hongwu international Group Ltd) and its physical analysis of was shown as in Table 2. Two different percentages of (0.5 wt%, and 2.5 wt%) cement were used.

Table 2 Nano oxide physical Analysis

Purity %	Particle size nm	SSA m^2/g	Color	Crystal lattice
99	20-30	135.1	White	Cubic

2.2. Specimens Preparation and Curing

Concrete are mixed at room temperature and it was 30^{o}C. To prepare concrete, the powder state materials were dry mixed first. Nano oxide material and water were blended first using ultrasonic mixer was used for 15min at 750W power. The Ultrasonic generator device was type (KQ 3200E) was used Ultrasonic of nano-materials with water was most likely a better means for right dispersion of nano materials than used mechanical mixing method. Then the solution of nano oxide and water was add to the dry concrete composition and mixed for about two minutes. before placing the paste inside oiled mold. The molded concrete samples were wrapped with plastic foil for about 24 hr to avoid moisture loss at ambient temperature. After removing from mold, the concrete specimens were cured in tap water at temperature of an around 28–30 ^{o}C for 28 day.

2.3. Characterization Analysis

Atomic force microscope (AFM) is a extremely high-resolution and one of the leading tools for measuring, manipulating matter at the nano-scale, and imaging. The AFM that used is type Angstrom, Scanning Probe Microscope, Advanced Inc, AA 3000A°, USA. Scanning electron microscopy (SEM), (TESCAN)type was used as well and it is useful technique for analyzing the prepared concrete specimens morphology with nano-particles and recycle cement mortar. X-ray diffraction(XRD) ,(MAXima) type, was used to identify the crystal structure of materials powder by (XRD) after reaction with atoms of crystalline substance which were distributed uniformly and repeatedly in the routine. This device provide information on materials phases , composition the crystallization degree. XRD scan range is (5.0 – 80.0) deg and threshold (3.0) , CSC(30KV/30 m A) . The scan mode is a continuous scan with scan speed of (5deg/min). The X-ray diffraction apparatus has Cu(1.5406A^o) as X-ray tube.

2.4. Compressive Strength

Compressive strength of concrete samples was determined in accordance to the BS EN 12390-3: 2009 [9] for different curing ages (7, and 28 days). Tests were done on samples with 100 x 100 mm in dimensions and the average compressive strength values were determined.

3.Result and Discussion

3.1. Atomic Force Microscope (AFM)

Fig. (2 , and 3) show the topographic structures in 3D views for the concrete samples. The reference concrete specimen(Co) was found to have less homogeneous and high roughness surface (22.62 nm) with an average diameter of (372.67nm) . Fig. 3(a) and Fig 3(b) demonstrate the topographic structures in 3D views of mixed samples with (0.5% Al_2O_3) nano material (NA) and (10% and 50%) RFA. Fig. (3(c)) and Fig. (3(d) illustrate the topographic structures in 3D views of samples with (2.5% Al_2O_3) nano material(NA) and (10% and 50%) RFA. These samples showed that the specimens were more uniform and lower average diameter and surface roughness than reference concrete due to addition of nano-particles.

The specimens surface roughness is shown in Fig. 3(a) to Fig.3 (d) was (7.17nm),(7.2nm),(7.24nm), and (13.79nm), Whereas the average diameter was (268.49 nm), (148.19nm), (156.91nm), and (123.53nm), respectively. Fig. 3(a) to Fig.3 (d) illustrate that the specimens are more uniform than reference concrete sample due to the addition of nano-alumina (NA) and recycled fine aggregate (RFA), surface roughness and average diameter was reduced with nano-oxide.

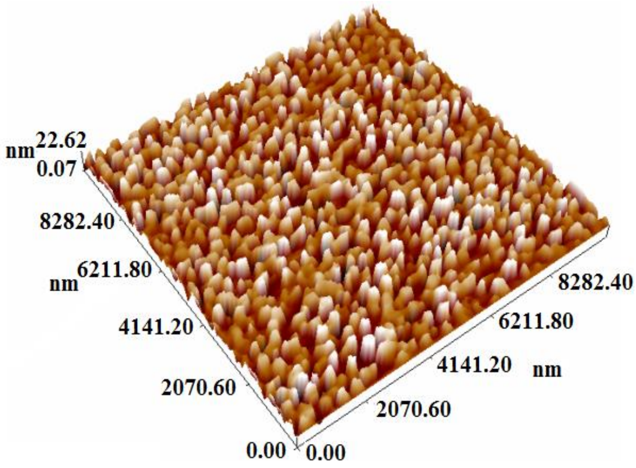
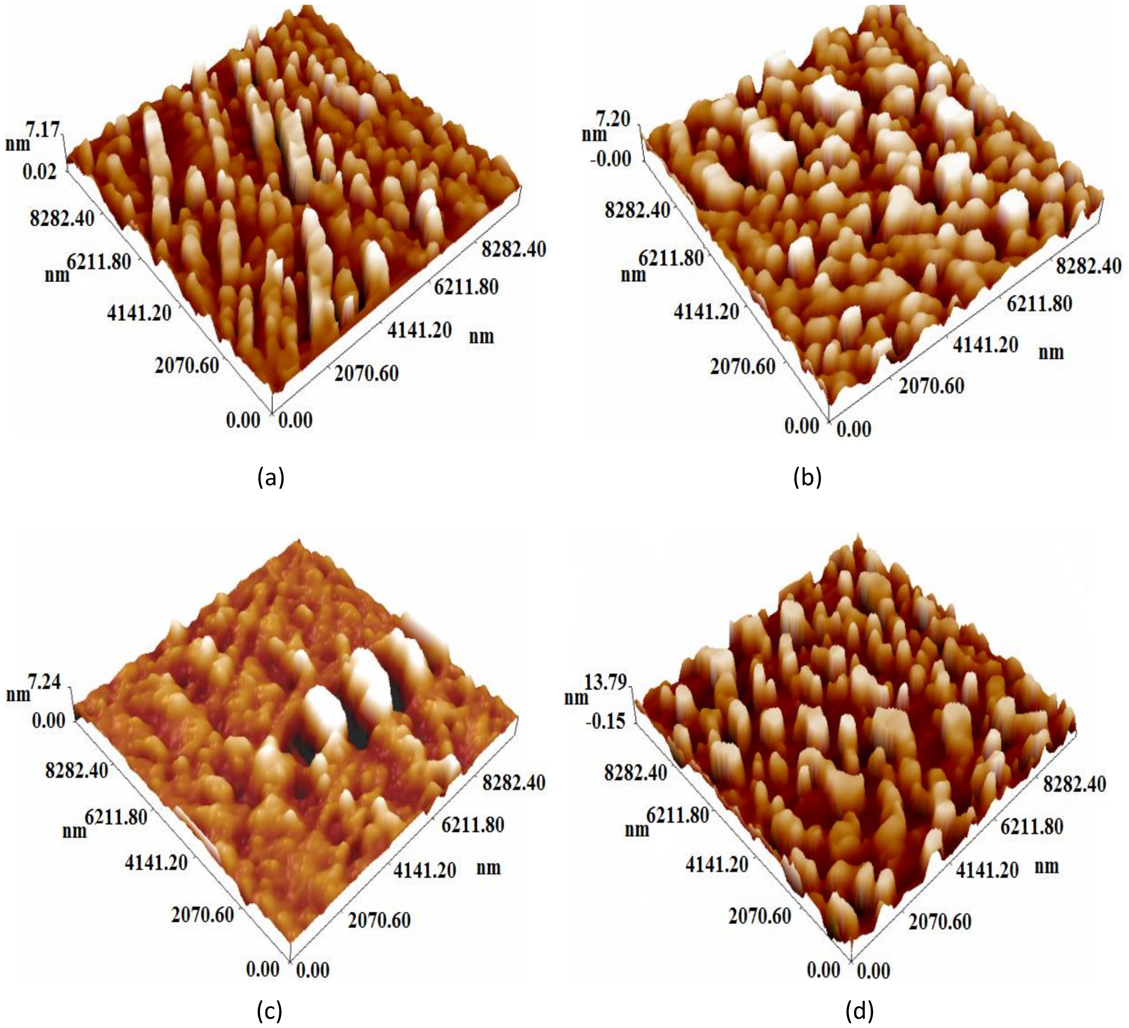


Figure 2. Surface roughness for reference concrete .



(a) (b)

(c) (d)

Figure 3. Surface roughness for concrete with additives (a) 10%RFA-0.5%NA,(b) 50%RFA-0.5% NA, (c) 10%RFA-2.5%NA, (d)50%RFA-2.5%NA.

3.2. Scanning Electron Microscopy (SEM)

Fig. 4 shows the SEM micrograph of the reference concrete mixture at 28 days without any additive. Fig. 4 is showed big size pores with large crystal of $Ca(OH)_2$ with a few ettringite phase hydrate needles in microstructure . These porous structure and existence of many CH crystals connected to the C-S-H gel indicates that the process of cement hydration was not finished and the microstructure of concrete was less compacted with big voids can be seen that which lead to reduce concrete strength.

Figs. 5 illustrates the SEM of concrete samples with (0.5%, and 2.5%) (NA), and with percentages 10 and 50 % of recycle aggregate. In this the samples prepared with (NA and RFA) were more dense C-S-H gel and organized than the reference sample(Co) with a little quantity of $Ca(OH)_2$ (CH)crystals and tiny sized pores, It can also be noted from the same figures that is a compressed structure with the nonappearance of the CH needles, a little non-hydrated crystals, fill voids and more homogeneous than that of the reference cement concrete sample and that may be the reason for increase concrete strength with addition nano-particles as shown in previous studies.

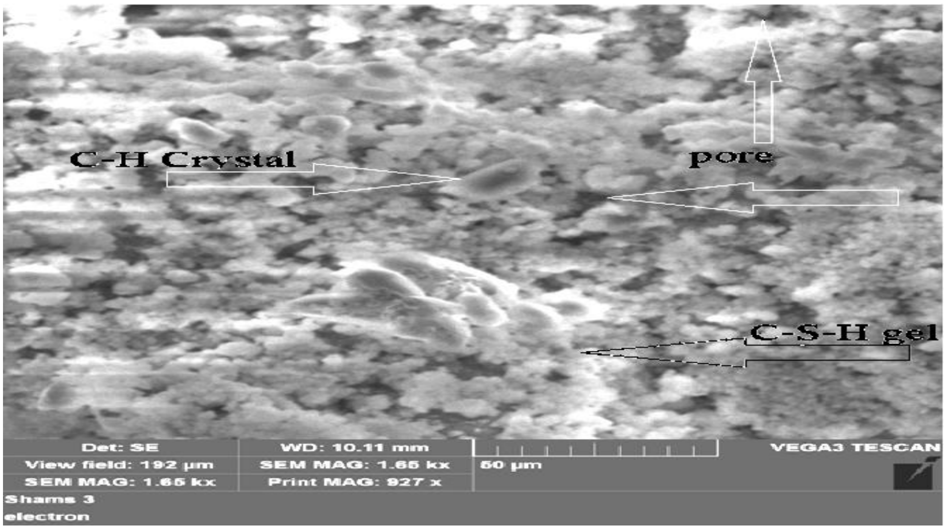


Figure 4.The SEM micrograph of reference concrete.

The enhancement in strength may due to high activity of nano- Al_2O_3 that causing the pozzolanic reaction to create more calcium silicate hydrate (C-S-H) gel. Nano materials consume calcium hydroxide crystals, heap pores to increase the strength, reduce the crystals size at the interface zone(IZ), transform the calcium hydroxide(CH) weakly crystals to the calcium silicate hydrate (C-S-H) crystals, and improve (IZ) and structure of cement paste.

The mechanisms of the Al_2O_3 nano-particles have high specific surface area lead to the depreciation of crystalline calcium hydroxide $Ca(OH)_2$ (CH) which rapidly produced through the cement hydration process and fills the void structure of (C-S-H) gel and lastly the hydrated products were became more denser and compact than that without nano-oxide.[13]

The increment in the percentage of recycle aggregate with remained the percentage of nano-material constant as shown in Fig. 5(b) , and Fig. 5(d) produce more dense structure compared with low percentage, as shown in Fig. 5(a), and Fig. 5(c) that may due to either the micro size of recycle aggregate fill the pore in C-S-H gel of cement mortar matrix. Or the interfacial transition zone between natural aggregates and the cement paste and may be Hydrate products on the surface of aggregate lead to higher bond between aggregate and new cement paste.

The cementitous material in cement concrete structure is increased due to some non reacted cement in fine recycle aggregate produce from cement mortar at different curing age.

The decrement in pore volume and the increment in densification of the transition interfacial zone between old and new cement paste are the factors responsible for the increase in the strength.

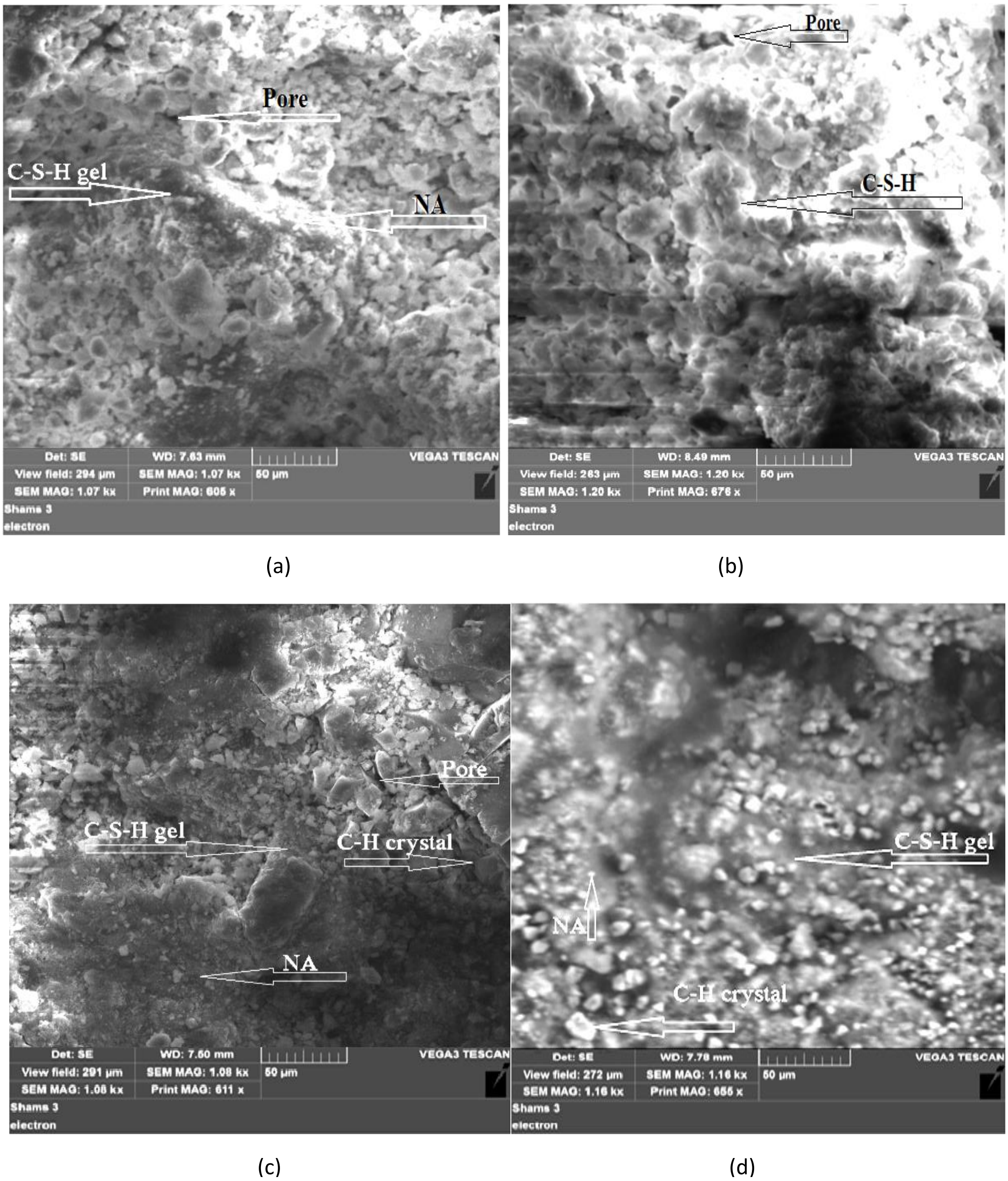


Figure 5. SEM micrograph for concrete with: (a)50%RFA-2.5%NA, (b) 10%RFA-2.5%NA, (c) 50%RFA-0.5%NA, (d)10%RFA-0.5%NA.

Fig.6 shows the XRD pattern of the reference concrete sample(Co), Fig. 7 shows the XRD patterns of the concrete samples with nano material addition (0.5 wt.% and 2.5 wt. % NA) and recycled fine aggregate (RFA) (10 %and 50 %) by weight.

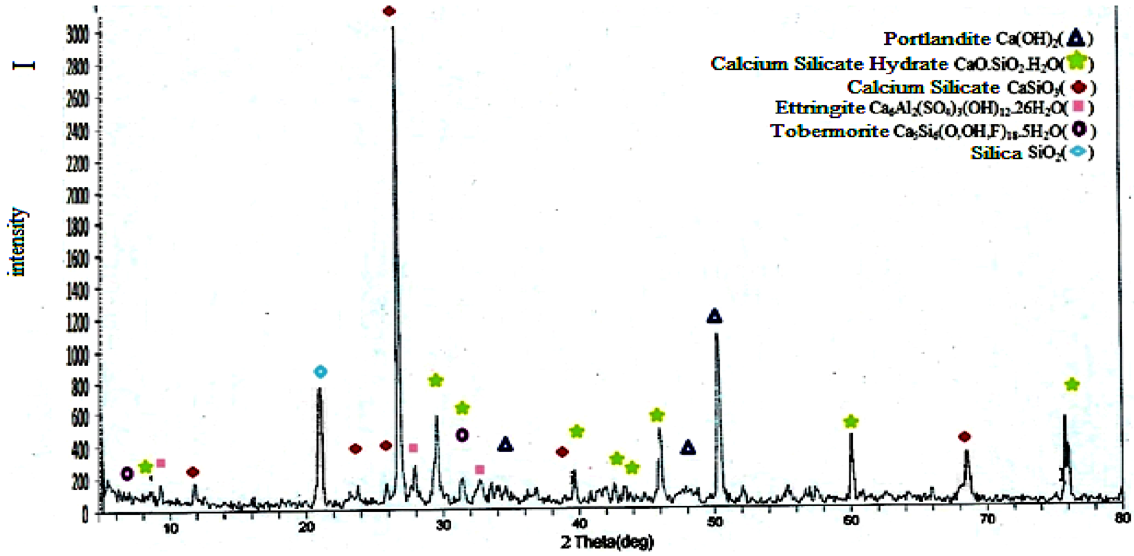
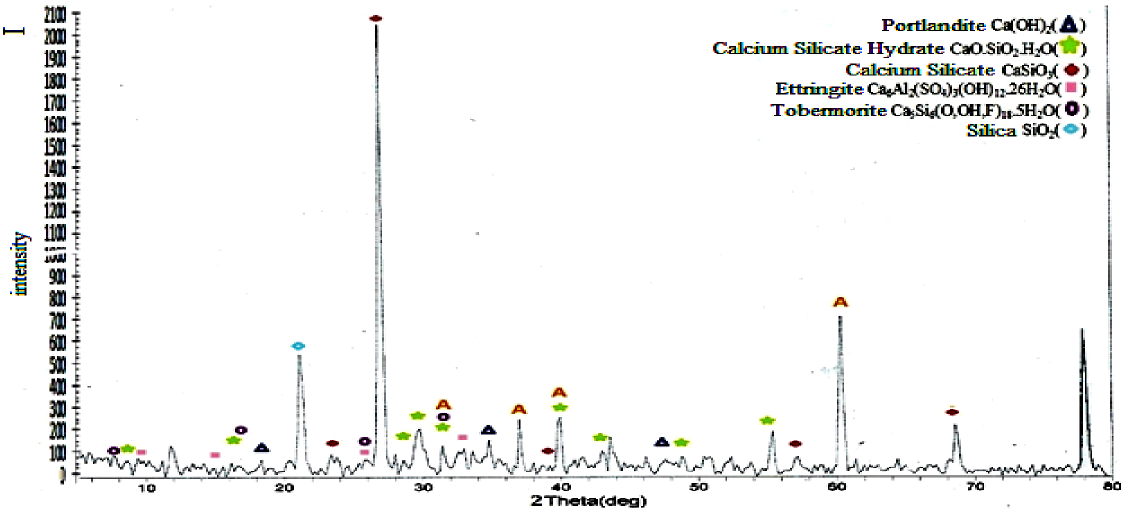
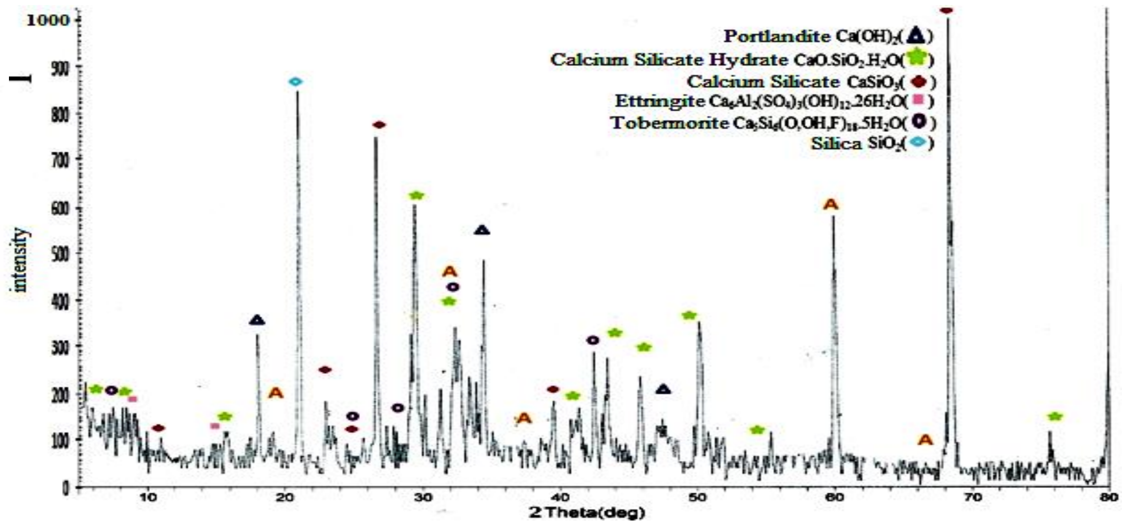


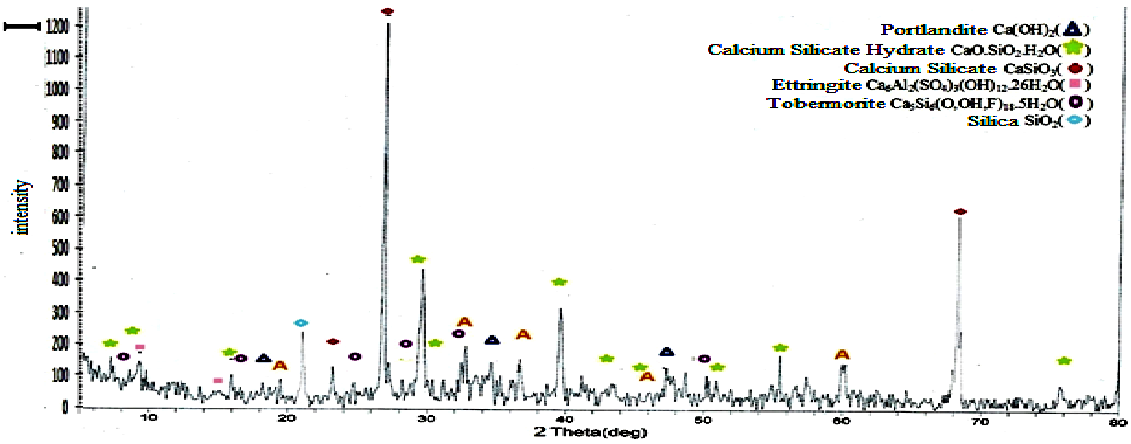
Figure 6. XRD pattern of the reference concrete.



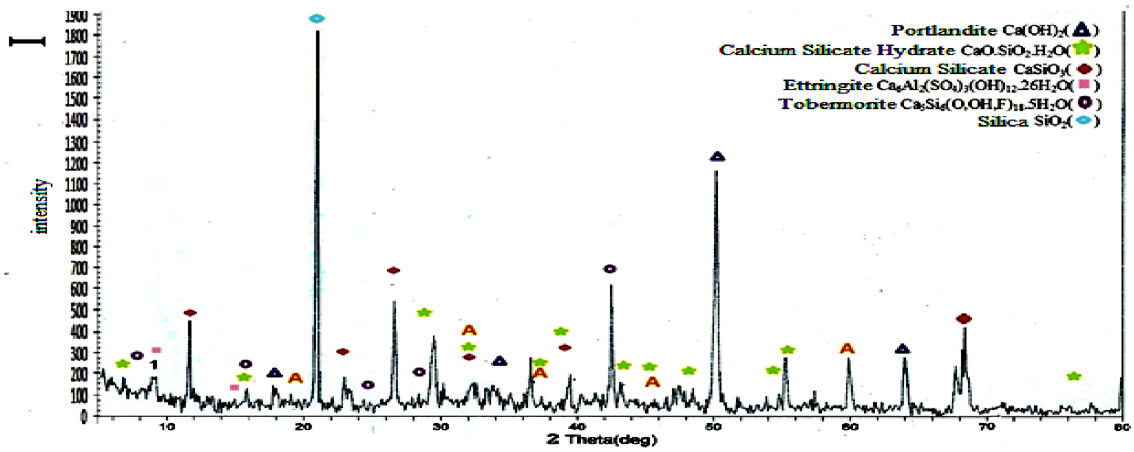
(a)



(b)



(c)



(d)

Figure 7.XRD pattern of concrete : (a) 10%RFA-0.5%NA,(b) 10%RFA-2.5% NA, (c) 50%RFA-0.5%NA, (d)50%RFA-2.5%NA.

From the XRD profiles, it is evident that the (C-S-H)peaks and Tobermorite is increased with mixed by nano-oxide and recycle cement mortar as shown in Fig.7. This increment may due to decrease the effect of Ettringite , calcium silicate (CS) and (CH) phases due to the pozzolanic reaction of NA with concrete composition.

It is may be explained from Fig. 7 that the nano-particles reaction with CH formed through the hydration process, the pozzolanic reactivity of nano-particles were drastically improved the microstructure of cementituos system, thus enhancing the mechanical performance of the cemented materials. A sharp peak in CH was identified in the concrete representing the pure hydration product $Ca(OH)_2$(CH), which is unconstrained from the hydration of cement. which agrees with the SEM results. From Fig. 7 show that the increment in (RFA)% for the same NA % was increased the intensity of C-S-H phase and peak number that may be lead to increase concrete strength.

3.4. Compressive Strength

Fig. 8 and Fig. 9 show the comparison between the compressive strength of concrete samples with and without replacement of NA at 7 and 28 day curing, with 0%,10%,and 50% RFA. It is found that the recycled concrete had lower compressive strength than the reference concrete. The compressive strength decreased with increase the RFA replacement , but at the design range for 1:2:4 concrete ingredients. The compressive strength reduction for samples with the replacement of (10 wt%, and 50 wt% RFA) was (4.5%, and 10.8%) at 7 day, respectively, while it was (1.8%,and 14.1%) at 28 day, respectively. These lower compressive strength of recycled concrete may due to the higher water absorption and porosity when compared with the natural sand .

The compressive strength result of samples with NA with RFA showed that the combination of nano-particles (NA) and micro-particles (RFA) increased the compressive strength of concrete samples compared with C_0 compressive strength . This observation indicates the better action of RFA in presence of NA that can be due to better filler effect and further distribution of the particles in the remaining voids, leading to homogeneous concrete matrix, i.e. the nano-particles play filler action to fill the void in recycled concrete. [14]This leads to narrow and stronger ITZ of recycled concrete with nano-particles as compared to that of recycled concrete without nano particles additive and increased compressive strength as shown in Fig. 5.

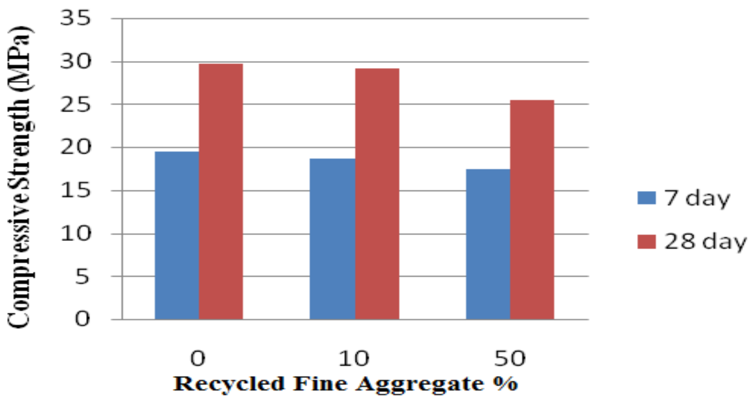


Figure 8. Concrete samples compressive strength with different recycled aggregate percentage.

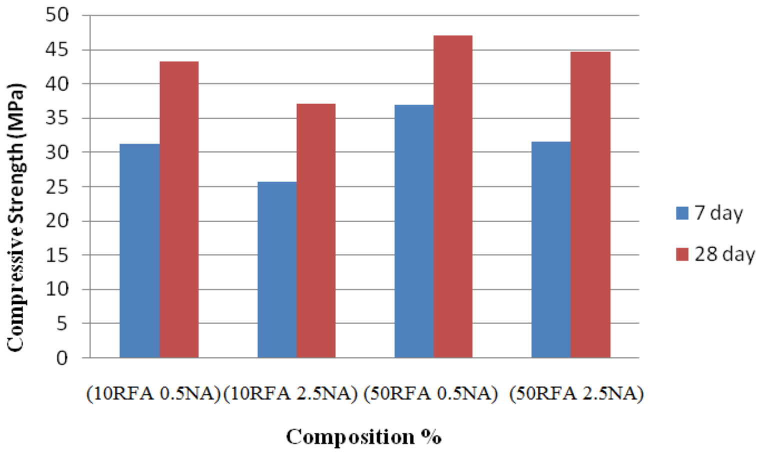


Figure 9. Concrete samples compressive strength with different recycled aggregate and nano-alumina percentage.

5. Conclusions

1. Concrete samples had a reduction in surface roughness after the addition of the (NA) nano-powders and recycle aggregate comparison with reference concrete. The surface roughness for specimens was decreasing by (68.3%,and 68.1%) for(0.5% Al_2O_3 with (10%, and 50%)RFA, respectively, while, by (67.9%, and 39.03%)for (2.5% Al_2O_3 with (10%, and 50%)RFA.
2. Homogeneity was found to be increased with nano addition more than reference samples. This is due to particles being smaller, thus leading to an improvement in the surface characteristic of concrete by smoothening of the grain and closing the pores that increase concrete strength.
3. Increment the recycle fine aggregate was found to reduce the homogeneity of concrete matrix which lead to concrete strength decrease.
4. XRD results show the nano-particles had higher specific surface area than macro-particles and the pozzolanic reaction causes decrease the effect of Ettringite ,CS and CH phases concrete. The hydrated products were became more denser and packed together than the concrete without nano-oxide which agrees with the SEM results.
5. The addition of nano-Alumina (NA) with recycle fine aggregate (RFA) (from cement mortar) to the concrete mixture were found to improved concrete

microstructure (surface roughness, matrix dense, and compressive strength) comparison with reference concrete.

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