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Effect of Nano Alumina on Mechanical Properties of Reactive Powder Concrete

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Abstract. Reactive Powder Concrete (RPC) is a cement-based substance that's considered as one of the ultrahigh performance concrete types. In fact, it is an ultrahigh strength concrete with excellent toughness. It can somewhat be referred to as a mortar than a real concrete mix due to the substitution of fine and coarse aggregate in conventional concrete by extremely fine sand having a particle size within the range (150-400 μm). In this investigational work, the compressive as well as the flexural strength of control mixture ((RPC) and (RPC) with three different nano alumina contents (1.5, 3 and 5%) replaced from the weight of silica fume was obtained, and the outcomes were compared. An experimental work was conducted on these four groups of mixtures, consisting of 12 cubes of (50 \times 50 \times 50) mm and 12 prisms of (50 \times 50 \times 300) mm. The microstructural analysis was performed using Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). The results manifested that the ultimate compressive strength is 140.32 MPa, and the maximum flexural strength is 30 MPa in mixes with 3% nano alumina. Mixes with 1.5% and 5% nano alumina also enhanced the compressive and flexural strength compared with the plain concrete mix. The XRD and SEM results elucidated that the nano alumina addition enhanced the microstructural bonding and augmented the hydration of the cement, which led to improving RPC mechanical properties.

Key words: RPC, Nano alumina, Compressive strength, Flexural strength.

INTRODUCTION

Concrete is the utmost broadly spent building material. Nevertheless, it characteristically displays less tensile strength and deprived deformability. By the infra-structure scale and the intricacy of the working environment, the difficulties, such as the inadequate mechanical characteristics and deprived toughness of the concrete have got progressively protuberant, needing further care for improving the fineness and reducing the pores and microcracks in the concrete ^{1, 2}.

The Reactive Powder Concrete (RPC) belongs to the Ultrahigh-Performance Concrete (UHPC) family. It's a highly effective cementitious substance with a high strength as well as exceptional toughness. The RPC's high characteristics are primarily attributed to its optimized size of the particle, low porosity, and enhanced microstructure ³⁻⁵.

RPC is a type of concrete with no coarse aggregate, but it contains sand, cement, quartz powder, steel fiber, silica fume (SF), too low ratio of water: binder, and superplasticizer. The coarse nonexistence of aggregate was regarded by designers to become a crucial feature for the performance and microstructure of RPC for reducing the inhomogeneity between the aggregate and the matrix of cement.

Also, it was approved that the fine steel fibers inclusion enhances the ductility ⁶⁻⁸. Furthermore, because of the high pozzolanic reactivity and the small size, such pozzolanic particles, particularly the fume of silica, can efficiently encourage the strength evolution of RPC ⁹⁻¹¹.

Nowadays, construction engineers are more interested in enhancing the mechanical characteristics of the cement-based composite materials in the current works using concrete. An example, the concrete constructions' compressive, tensile, and flexural strength of concrete are the most desired mechanical properties to be enhanced. The nanomaterials can be blended with the cementitious matrixes for obtaining the mixes of concrete with an elevated mechanical strength^{12, 13}.

Besides, the nanotechnology possesses a high potential in the building and construction materials. The fundamental idea beyond utilizing nanomaterials is that they have an enormous surface area that may enhance the strength at premature ages and the hydration features of the cementitious substances.

Moreover, they assist in decreasing the water absorption and porosity compared to the traditional cementitious substances. Cement-based materials can be blended with the nano materials, like Nano Alumina (Al₂O₃), Nano Silica (SiO₂), Nano Titanium Oxide (TiO₂), Nano Ferric Oxide (Fe₂O₃), Graphene and Graphene Oxide, and Carbon Nanotubes (CNTs)¹⁻¹⁶.

Among all nanoparticles' types, the Nano Al₂O₃ addition to cement materials was investigated by several researchers. The Nano Al₂O₃ usage can speed up the development method of the (C-S-H) gel, particularly at premature periods, which improves the composites strength¹⁷⁻¹⁹.

Zahraa et al.²⁰ studied the effect of various percentages from Nano Al₂O₃. The mortar's compressive strength that augmented slowly with the rise of the content of nano alumina till 5% via the weight of cement was obtained.

E. Mohseni et al.²¹ performed an investigation upon the hybrid influence of nano silica, nano alumina, and nano titanium oxide upon the rheology, toughness, and mechanical properties of the mortar's self-pressing with additive (fly ash).

Results indicated that (1%, 3%, and 5%) from the nanoparticles of nano Al₂O₃, nano SiO₂, or nano TiO₂ provided the best compressive strength. Guangcheng Long et al.⁹ concluded that the nanoparticles supplement causes the increment of the surface area of pores and decreases the entire pores' volume and the dia. of the reactive powder concrete. Amid the specimens, the reactive powder concrete with the supplemented nano silica possesses the minimum volume of pore and the most significant pore surface area.

Also, the optimum quantity for every nanoparticle in the investigated area is around (1.5-2.0%) for each nano powder (CaCO₃, Fe₂O₃, Al₂O₃ and SiO₂). At such percentage, the compressive or flexural strength of the prepared reactive powder concrete can be increased by around (30%). On the other hand, the inconsistent dispersal or the supplement above the optimal quantity reduces the compressive strength owing to the agglomeration for the Nano silica and alumina.

The current paper investigates the influence of Nano alumina upon the RPC properties. The Nano Al₂O₃ has been selected as filler due to its typical combination of the physical and chemical properties. The Nano Al₂O₃ influences upon the hydration method, flexural strength, and compressive strength of the reactive powder concrete have been studied.

EXPERIMENTAL WORK

Used Materials

The ordinary limestone cement, as locally known as Karasta, being utilized for the all mixes of concrete examined in the current work. This type of cement satisfies the Iraq Specification (3868) according to the international standard (EN 197-1:2011 CEM II/A-L 42.5R) 22.

Chemical analysis of the employed cement is shown in the TABLE 1. In addition, the normal sand acquired from a local area was utilized for the concrete mixes.

The grading of fine aggregate agrees with the Iraqi specification (IQS No.45/1984) (Zone 4)²³. The grading curve for the sieved sand compared with its specification is depicted in Fig. 1, while the physical properties of the fine aggregate are listed in the TABLE 2. The chemical and physical properties for materials had done in laboratory of Babylon University.

As for the nano materials, the dense micro SiO₂ (SF) (Conmix Co., United Arab Emirates) was used in this work. The employed silica fume chemical composition confirms with the standard ASTM C1240/2015 24. TABLE 3 and TABLE 4 display the chemical and physical properties of SF utilized, respectively.

Alfa nano alumina (Al₂O₃) powder was used in this study. TABLE 5 lists the analysis for the typical impurities of the alumina oxide nanoparticles (ppm).

TABLE 1. Chemical analysis and physical properties of the used cement.

Compound	Chemical Composition	Percentage by Weight	Limit of (EN 197-1:2011)
Lime	CaO	62.79	/
Silica	SiO ₂	20.58	/
Alumina	Al ₂ O ₃	5.6	/
Iron oxide	Fe ₂ O ₃	3.28	/
Sulfate	SO ₃	2.35	2.5 if C ₃ A ≤ 5 2.8 if C ₃ A > 5
Magnesia	MgO	2.79	5% max
Free Lime	Free CaO	0.9	/
Loss upon Ignition	L.O.I.	1.94	/
Insoluble Residue	I.R	1.00	/
Lime Saturation Factor	L.S.F	0.9	/
Main Compounds (Bogues Equis.)		Percentage via cement weight	
Tricalcium Silicate (C ₃ S)		50.12	
Dicalcium Silicate (C ₂ S)		21.26	
Tricalcium Aluminate (C ₃ A)		9.29	
Tetracalcium Aluminoferrite (C ₄ AF)		9.98	
Physical properties		Test Results	
Setting time:			
Initial hrs; min		2; 05	≥ 45 min
Final hrs; min		4; 00	≤ 10 hrs

TABLE 2. Fine aggregate physical properties

Properties	Result of Test	Iraqi Specification (No.45/1984) Limits
Specific Gravity	2.65	/
Fineness Modulus	2.6	/
Absorption	1.5 %	/
Sulfate Content	0.24 %	≤ 0.5 %

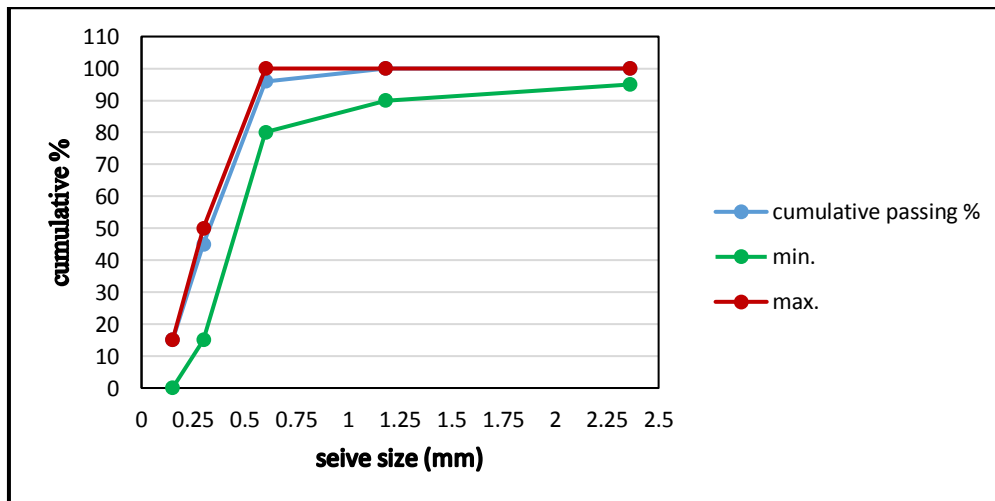


FIGURE 1. Grading curve of used sand.

TABLE 3. Chemical properties of the utilized SF

Oxide Composition	Oxide Content %	ASTM C-1240 Limitations ²⁴
SiO ₂	92	≥ 85
Al ₂ O ₃	0.50	---
Fe ₂ O ₃	1.25	---
Na ₂ O	0.35	---
K ₂ O	1.10	---
CaO	0.37	< 1
MgO	0.99	---
SO ₃	0.29	< 2
L.O.I.	3.15	≤ 6.0

TABLE 4. Physical properties of the utilized SF

Physical Properties	SF	Specification Requirements (ASTM C-1240) Limit ²⁴
Percent retained upon 45 μm (No. 325) Sieve, max, (%)	1.7	≤ 10
Specific surface, Min., (m ² .g ⁻¹)	23	≥ 15

TABLE 5. Analysis for typical impurities of alumina oxide nanoparticles (ppm)

Element	%
Ca	< 100
K	< 100
Cu	< 10
Mg	< 50
Fe	< 100
Mn	< 50
Zn	< 50
Si	< 100

The high-range water-reducing admixture (Hyperplast PC200 type) being utilized through such investigation and confirmed the standard (ASTM C494/C494 M/2017) ²⁵. Tap water available in the laboratory was used in mixture and specimens curing, and it had a temperature of (20±6°C). The properties of micro steel fibers (Chinese origin) utilized in the present research being tabulated in the TABLE 6.

TABLE 6. Properties of the utilized steel fibers

Property	Specifications
Surface	Brass coated
Density	7860 Kg.m ⁻³
Tensile Strength	> 2300 MPa
Form	Straight
Melting Point	1500°C
Average Length	13 mm
Diameter	0.2 mm ± 0.05 mm
Aspect Ratio (L _t / D _t)	65

Mix Proportions

RPC developed by Al-Sultan mix proportions²⁶ was considered for the current research's work. Silica fume was replaced by different percentages of Nano alumina. TABLE 7 shows the mix proportions for RPC with various percentages of Nano alumina.

After completing the process of weighing the materials, the cement, sand, silica fume and nano alumina are placed in the mixer. The dry materials are mixed at a slow speed for three minutes in order to sure the homogeneous. The plasticizer is mixed with water and gradually added to the dry mixture while increasing the mixing speed.

When ensuring that the materials are homogeneous, the steel fibers are added and the mixing continues for several minutes, and then the mixture is poured into the moulds.

TABLE 7. The mix proportions for RPC (Kg/m³).

Mix symbol	RPC ₀	RPC _{1.5}	RPC ₃	RPC ₅
Cement	950	950	950	950
Sand	1010	1010	1010	1010
Silica fume	250	246.28	242.5	237.5
Steel fiber	157.2	157.2	157.2	157.2
Water content	204	204	204	204
Superplastizers	84	84	84	84
Nano alumina	0	3.75	7.5	12.5

Tests

Compressive strength Test

Compressive test for the RPC has been conducted following the ASTM C109 standard²⁷. Three cubes having dimensions (50 mm × 50 mm × 50 mm) dimensions have been employed to obtain the compressive strength. The average magnitude of compression strength for such cubes was taken to determine the compressive strength. These cubes have been examined at (28 days) age utilizing digital hydraulic compression equipment.

Flexural Strength Test

The flexural strength test for the RPC samples has been carried out according to the standard (ASTM C78)²⁸. Three prisms having a size of (50mm × 50mm × 300mm) with (240 mm) testing span have been utilized to perform the flexural strength test. In addition, the average flexural strength value for these prisms has been taken to determine the flexural strength.

XRD and SEM Characterization

XRD and SEM characterization has been achieved for determining the composition of the phase's composition of material and the degree of crystallization. Broken pieces from compressive cubes (0%, and 3% nano Al₂O₃ mix) have been used as samples for the (XRD) and (SEM) tests. The Intel diffractometers scan range (20 to 80) degrees in the XRD test has been employed to obtain the X-ray diffraction patterns.

As for the scanning electron microscopy (SEM), the samples (i.e., the broken pieces) have been coated by conducted materials, like gold, before testing. This is due to the fact that concrete doesn't conduct the ray of electron. The SEM test has been achieved in the Collage of Science/University of Al-Nahrain. The XRD test has been done in the Laboratory of Nanotechnology/Ministry of Science and Technology.

RESULTS AND DISCUSSION

Results of the Mechanical Properties

The mechanical properties of RPC with nano alumina have been studied via performing the compressive and flexural tensile strength tests. The outcomes have been inferred in the present section.

Compressive Strength

Figure 2 illustrates the RPC compressive strength outcomes of the specimens with and without nano alumina. It is noticed that the high value of compressive strength being when the Nano Al₂O₃ was reached to 3%. These increments were 6.4% and 16.7% when the Nano alumina percent was 1.5 and 3% compared with the compressive strength for control mixture (nano alumina zero percent), and the compressive strength decreased when the Nano Al₂O₃ was 5% ,but it is still higher than plain concrete mix²⁹. This increment is due to the nanoparticles that enable filling the pores. The nanoparticles reduced the size of voids of RPC, and this improved the strength and reduced the porosity¹².

Also, the rise into the compressive strength of the mixtures with nano Al₂O₃ can be ascribed to rapid ingesting of the Ca (OH)₂ developed through the hydration of Portland cement as well as the additional formation of C-S-H. This is more pronounced when it is connected to the high reactivity of the nano particles of alumina³⁰ that enhance the bonding between the aggregates (sand) and cement¹³.

The decrease in compressive strength at 5% occurred owing to the extreme quantity of alumina nanoparticles that reduced the ratio of water to binder, resulting in insufficient hydration evolution because of the high surface area that required higher w/c and the RPC having low w/c. So, the inconsistency dispersal of nanoparticles into the matrix of concrete also reduced the compressive strength³¹.

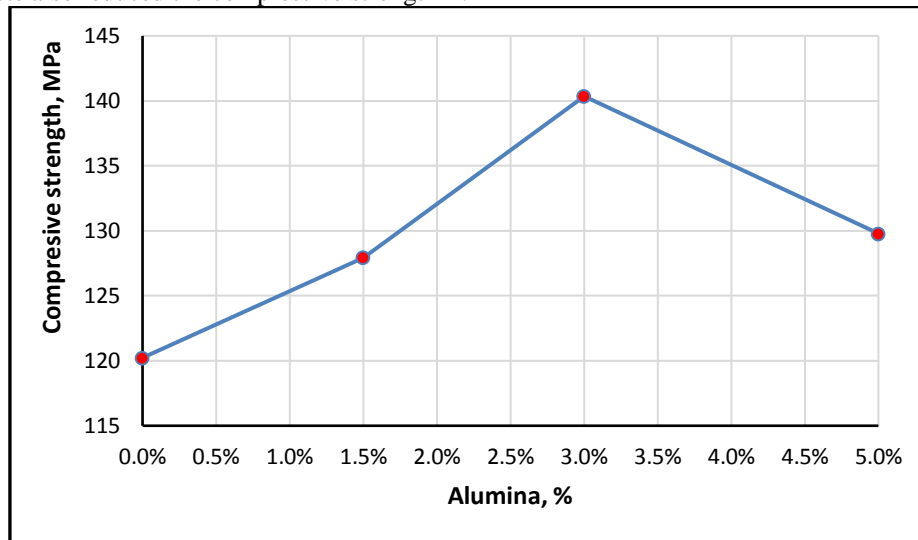


FIGURE 2. The compressive strength of RPC specimens.

Flexural Strength

Figure 3 demonstrates the test outcomes of flexural strength. Improvement with the addition of nanoparticles was recognized in all mixes, and the maximum increment when the Nano alumina percentage was up to 3% (29.6 MPa) was compared to the plain RCP (20 MPa).

This increment is due to the interfacial adhesion between Nano alumina and steel fiber that are more compatible and adherent to each other. On the other hand, when the Nano alumina was 5%, the flexural strength decreased due to the possibility of agglomerate formation, which caused a weak bond between Nano Al₂O₃ and the surrounding matrix³². Also, this is because of the gaps formed by the agglomeration of Nano alumina particles and the other component in the RCP mix^{33,34}.

Results of the XRD Analysis

Figure 4 manifests the outcomes of the X-ray diffraction analysis for the mix of control mortar and the alumina adapted mix of mortar after curing for (28 days) age. The outcomes of X-ray examination evinced the following created phases: SiO_2 , $\text{Ca}(\text{OH})_2$, CaCO_3 , and CSH gel.

The two dominant and most distinct phases in all schemes are SiO_2 and CaCO_3 , which completely agree with the research results 35. The researchers confirmed that the X-ray examinations of concrete samples revealed mainly silica and lime compounds.

Another vital phase that appeared in the two diagrams is the $\text{Ca}(\text{OH})_2$ compound, as a product of the hydration process, which confirms the successful hydration process.

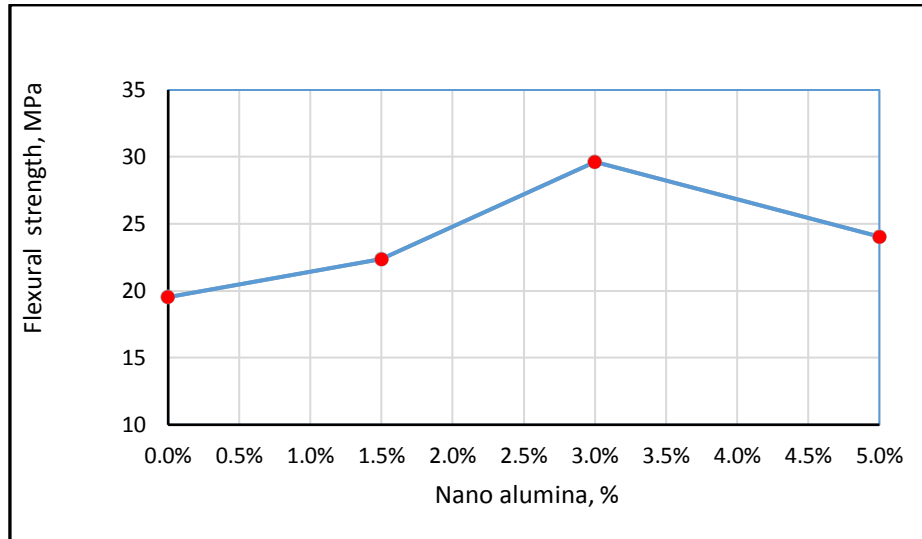


FIGURE 3. The flexural strength of RPC specimens.

Furthermore, one of the compounds that also appeared is Ettringite, which is the product of the hydration reaction of aluminate in cement. Finally, the highest chart in fig 4b of the presence of nano porous alumina, which plays a role similar to the role of silica in the interaction with calcium hydroxide and the formation of alumina compounds (C3ACSH), has appeared.

Although there is a difficulty in determining all the picots of CSH and C3ACSH compounds—glass-phase compounds with slight crystallization with a low picnics intensity, X-ray diffraction remains one of the essential tests. Such test assists in inferring the changes in the chemical compounds resulting from the hydration process and their percentage when using alternative materials for cement, whether they are nanoparticles or other materials.

Results of the SEM Analysis

Figure 5 portrays the images of SEM of the mortar control mix as well as the alumina modified mortar mix. In analysis of the SEM of the specimens of cement mortar paste, it is obvious that no clear nanoparticles being observed; therefore, the whole nano materials participated into the hydration procedure to variable levels are verified. It can be seen via the comparison of Fig. 5a with the control mortar mixture shown in Fig. 5a that the mortar's microstructure with the Nano Al_2O_3 is considerably denser than the reference mixture.

The dominant and most distinctive phase in both images is CaCO_3 .

Such observation completely agrees with the XRD results. In Figure 5b, it can be observed how the Nano alumina speeded up the hydration rate. Thus, it was adequate for enhancing the mortar strength owing to the responded grains that exist into the hydrated cement paste ³⁶.

When comparing these two Figures 5a and 5b, the evolution of the further outputs of hydration, such as the (CSH) and the Ettringite, can be exhibited.

A similar microstructure condensation can also be elucidated in the paste owing to the Nano alumina powder supplement that illustrates earlier development of (CSH) Compared to the reference mixture. In relation to the suggested mechanism via Shaikh and Hosan ³⁷, the most significant rise into the creation of hydration product as well as the strength evolution can be elucidated when adding the nanoparticles of alumina to the cement mortar.

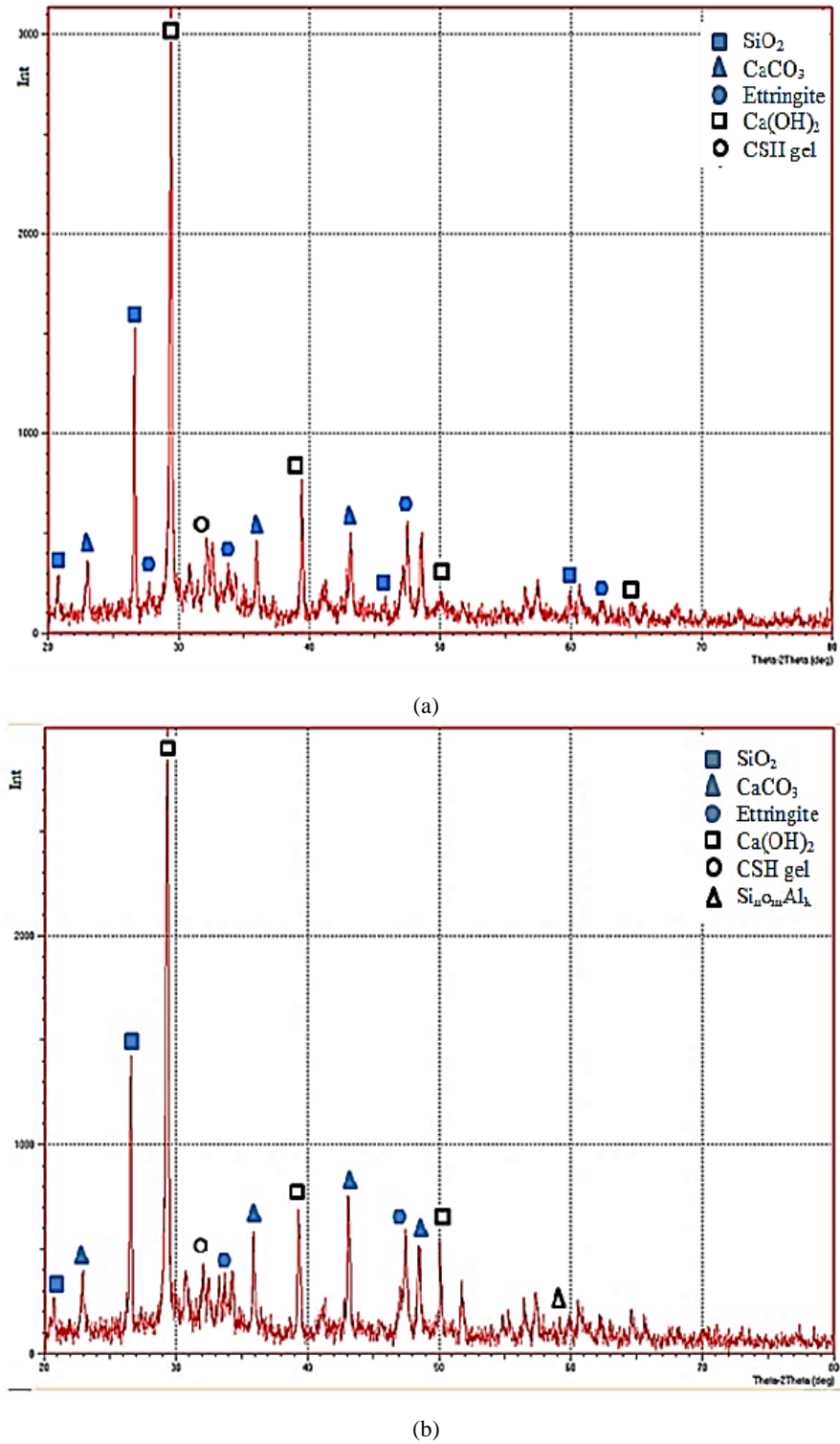


FIGURE 4. The X-ray diffraction analysis for: (a) The mixture of control mortar and (b) The mixture of alumina adapted mortar.

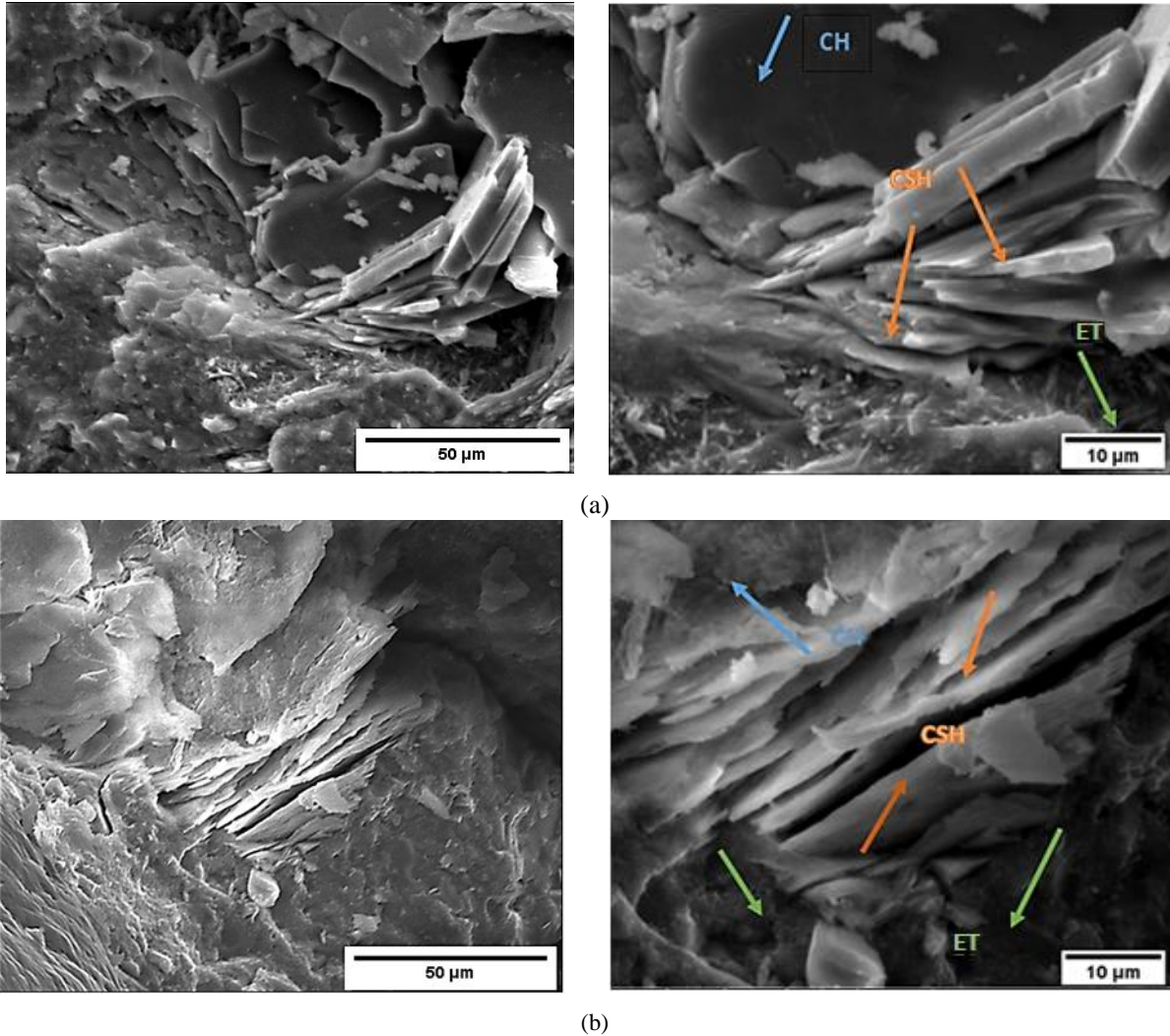


FIGURE 5. The SEM image for: (a) The control mortar mix and (b) The alumina modified mortar mix.

CONCLUSIONS

The current study investigated the Nano Al_2O_3 effects as replacement of silica fume on the mechanical properties and microstructural of RPC. The chief drawn conclusions include the followings:

- It was observed that the RPC compressive strength as well as the flexural strength of the RPC progresses via raising the nano Al_2O_3 particles' content. Similarly, it was noted that the silica fume could be beneficially substituted by the nanoparticles of Al_2O_3 to an ultimate limit of (3%). Such substitution can notably enhance the RPC compressive strength and flexural strength.
- According to the results of the SEM and the XRD analysis, the nano alumina not only speeds up the cement premature hydration and encourages the creation of further (C-S-H) throughout the influence of nucleation, but also causes a broadly dispense in the matrix of cement for filling the pores and preventing the (CH) crystals growth. Therefore, it causes a more solid microstructure.

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