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Research Paper

Role of nano-Al2O3 in modifying the properties of ultra-high-performance concrete

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ABSTRACT

The purpose of this study is to use cutting-edge nano modification technology to provide a crucial foundation for utilizing new-generation cementitious materials with extremely high strength and extremely extended service life. This research addresses the effects of nano Al2O3 particles replacement on the UHPC. In this research, nano Al2O3 particles mixes were used as additive ranges from 0, 0.5, 1, 1.5, 2, 2.5, and 3 percent from cement weight in UHPC. Concrete was prepared to perform slump cone, strength, modulus of elasticity, water permeability, and SEM analysis. The results indicated that the nano Al2O3 particles enhanced the flowability and increased the heat of hydration with the increase of their contents. It shows that nano Al2O3 particles can significantly accelerate the setting and hardening process of UHPC. The optimal dosages to enhance mechanical and durability properties were 2% nano Al2O3 particles. The compressive strengths of specimen increased by 26.86% compared to the control mix., it also reduced the 37.04% depth of water penetration under pressure, respectively in comparison to control concrete. The results exhibit that the nano Al2O3 particle is an excellent filler material, reducing porosity regions and accelerating cement hydration via the pozzolanic effect. The SEM analysis demonstrated that adding nano Al2O3 particles may also effectively enhance the interfacial transition zone.Click here and insert your abstract text. Click here and insert your abstract text. Click here and insert your abstract text. Click here and insert your abstract text. Click here and insert your abstract text. . Click here and insert your abstract text. . Click here and insert your abstract text. . Click here and insert your abstract text. . Click here and insert your abstract text.

1. Introduction

The concrete was developed through a continuous procedure, which may have started under the Roman Empire. The modern history of concrete was developed after the invention of Portland cement in 1824. It was produced by mixing the cement with aggregate and water. In 1849, steel reinforcement was introduced into the concrete to achieve greater strength and ductility, which was named "Reinforced Cement Concrete". During the 19th and 20th centuries, concrete technology continued to advance, as many people were interested in optimized strength and particle packing density. In the 1980s, High-Performance Concrete (HPC) and High Strength Concrete (HSC) were both invented for compressive strength up to 100 MPa with improved durability [1–5]. Shortly after, fibre-reinforced concrete (FRC) was developed to counteract brittle matrix, increasing ductility, strength, and resistance by applying fibres. Incorporating fibres into the concrete leads to controlling the cracks and improves the impact performance. The focus of concrete development in the 1990s was on increasing compressive strength and durability. Ultra High-Performance Concrete (UHPC), whose compressive strength varies from 100 to 200 MPa, was first employed in North American applications in 1994. Extensive research on UHPC compressive strength over 200 MPa, namely called Reactive Powder Concrete. The UHPC is a new type of concrete; according to the ASTM C1856/C1856M – 17 guidelines, it requires a minimum of 120 MPa compressive strength, the slump flow between 200 to 250 mm, and the maximum size of aggregate is limited to 5 mm. It has superior performance in strength and durability with a low life-cycle cost [6, 7]. The homogeneous mix was developed by removing coarse aggregate from the mixture and the effectiveness was increased by optimizing the particle packing density of the mix. The combination of high-range water reducers and new-generation polymer-based super-plasticizers optimizes the particle packing density of the UHPC mix, which enhances the rheological characteristics of the fresh mix with a low w/b ratio [8–10]. The present scenario increases the demand for the use of UHPC in structure because the developed and developing nations are mainly focused on conserving natural resources to prevent the changing environment. Energy conservation has become a priority for designing sustainable buildings with low life cycle costs [11–15]. The alternate binders are necessary to develop sustainable construction materials and minimize the global CO2 emission to the environment. The fabrication of UHPC consumes a large amount of cement in the mix [16–20]. Supplementary cementing materials (SCMs) are essential and contribute to improving the properties of UHPC through hydraulic and pozzolanic activity. To generate sustainable building materials and lower global CO2 emissions to the environment, alternative binders are crucial. These can be used individually with Portland or blended cement or in different combinations [21–24]. The micro-sized SCMs are frequently added to UHPC to make mixtures more economical, increase strength, reduce permeability, and influence other concrete properties[25–31].

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Nomenclature:

List of variables:

HSC	High strength concrete
FRC	Fibre-reinforced concrete
HPC	High performance concrete
SCMs	Supplementary cementing materials
IS	Indian standards
SSA	Specific surface area
LOI	Loss on ignition
ASTM	American Society for Testing and Materials
CS	Compressive strength
MoE	Modulus of elasticity
SEM	Dimensionless temperature

Greek Symbols:

G	Specific gravity
μ	Particle size in micrometer ($X \times 10^{-6}$)

Nanotechnology is a multidisciplinary science and engineering subject concerned with understanding and directing materials with dimensions ranging from one to one hundred nanometers. Nanomaterials are employed in a variety of unique applications such as agriculture, medical, electronics, infrastructure, food, fuels, air and water cleaners, chemical sensors, fabric, and so on. Depending on the nanomaterial dimensions, they can be separated into four different groups, namely called Zero-dimensional (0D), One-dimensional (1D), Two-dimensional (2D) and Three-dimensional (3D). A recent and interesting area of research is the incorporation of nanoparticles in concrete matrix. To enhance the characteristics of concrete, several nanomaterials have recently been used. Nanomaterial uses have a higher potential to diminish the environmental impact of energy intensity and enhance the structure's lifespan. Hence, the research on nanomaterials in concrete is crucial to making concrete more environmentally friendly. It is crucial to study nanomaterials' application to improve cement-based composites' properties. Still, only a few studies are available on nanomaterial use in ultra-high-performance concrete. Over the past two decades, the growing application of nano Al2O3 particles as SCM only in cement paste, mortar, shotcrete, normal strength concrete, and high strength concrete has been widely investigated. Now, there are only a few studies are available on the use of nanomaterials in UHPC. Blending nanomaterials in UHPC would be a breakthrough in the vision for future sustainability needs, creating extraordinary applications in the construction sector. The proposed research will cover a knowledge gap in the impacts of nano Al2O3 particle substitution on the slump cone, strength test, modulus of elasticity, water permeability and microstructural properties features of UHPC.

2. Materials

In the current study, OPC53 grade cement was used in accordance with IS: 12269-2013 [32]. The river sand is used as fine aggregate and has a specific gravity of 2.64, bulk density of 1650kg/m3and water absorption of 0.55%, respectively, according to IS 2386–Part 1 [33]. Elkem Microsilica® 940 type densified silica fume obtained from Elkem Metallurgy (P) Ltd is utilized, which conforms to IS 15388: 2003 [34]. Figure 1 depicts the morphology, elemental content, and particle size distribution of silica fume as described in Table 1.

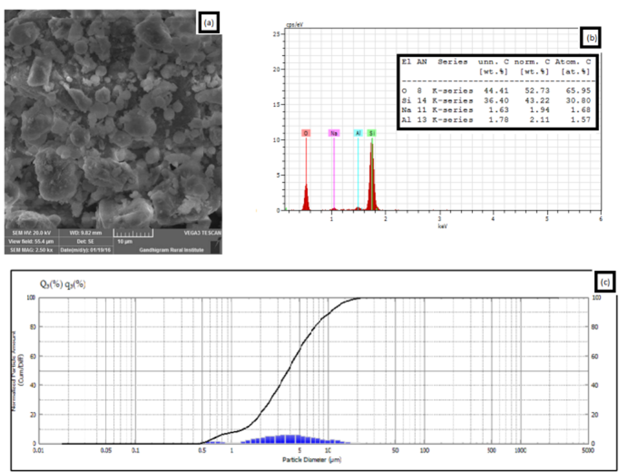

Figure 1**.** The characterization silica fume.

Nano-Al_2O_2 particles with a SSA of 180 m^2/g is used in this study. Table 2 illustrates the properties of the nano-Al_2O_2 particles and their appearance, elemental composition and XRD spectra in Fig. 2.

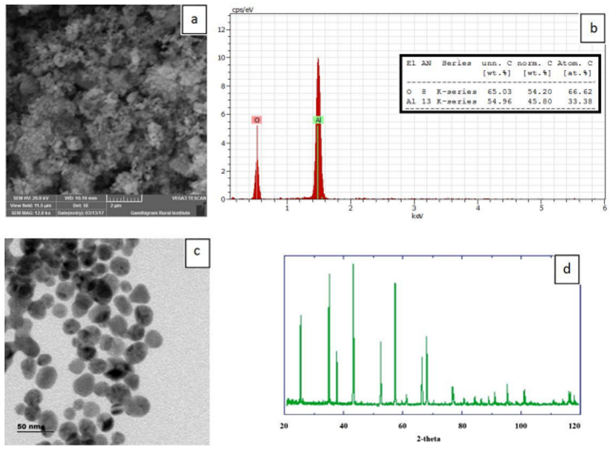

Figure 2**.** The characterization of nano-Al_2O_2 particles (a) SEM, (b) EDAX elemental composition and (c) TEM and (d) XRD.

Table 1**.** The properties of the silica fume.

An example of a column heading	Property	Values
Chemical Properties	Moisture Content	00.63%
	SiO_2	96.12%
	LOI	02.42%
Physical Properties	Specific Gravity	02.31
	Visibility	Grayish Powder
	State	Amorphous
	Bulk Density	700 (kg/m^3)
	SSA	23.18 (m^2/g)
	Solubility	Insoluble

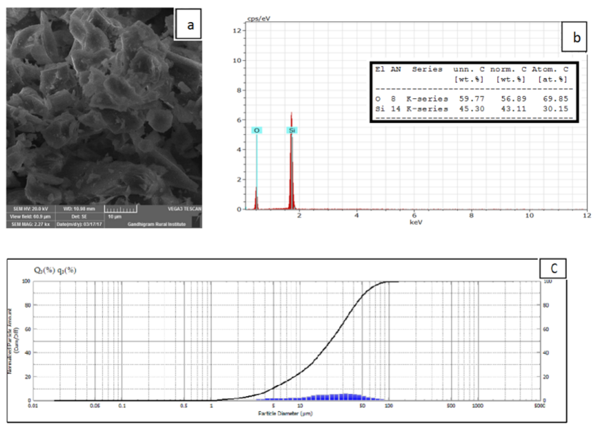

Figure 3**.** The categorization of quartz powder.

The quartz powder that was obtained from Gayathiri Enterprise (P) Ltd. has an average particle size of 19 μm. The manufacturer's disclosure of the quartz powder's physical and chemical characteristics is displayed in Table 3. Also, the Fig. 3 displays the powder's morphology, elemental content, and particle size distribution curve. The Polypropylene fiber procured from the Fibre zone Company is used, conforming to BS EN 14889-2: 2006 [35]. The physical properties are presented in Table 4.The Auramix 400, a modified polycarboxylic ether-based new generation superplasticizer is utilized and its properties are presented in Table 5. Potable drinking water accessible in the laboratory is used for conforming to IS 456-2000 [36], which is used for the casting and curing of specimens.

Table 2. The physical and chemical properties of the nanoparticle (quartz powder) of Aluminum Oxide.

Density (g/cm^3)	**Purity** (%)	**Size** (nm)	**SSA** (m^2/g)	**Morphology**	**MW**	**Color**
3.7	99.99	20-30	180	Spherical	101.96	White powder

Table 3. The properties of the quartz powder.

Grades	SiO_i	Al_2O_3	Fe_2O_3	**Boiling Point**	**Specific Gravity**	**State**
300 MESH	99.6%	0.1%	0.01%	Above 1300C°	2.65	White powder

Table 4. The polypropylene fiber physical properties.

Colour	White
Density (g/cm^3)	0.91
Burning Point, C°	> 360
Melting point, C°	160-180
Fiber diameter, μm)	38
Acid resistance	Fine
Tensile Strength, (MPa)	> 500
Elongation rate	$> 15\%$

Table 5. The properties of superplasticizers.

Superplasticizer type	ASTM C494 Type F
PH	> 6
Aspect	Reddish brown liquid
Chloride ion content	$< 0.2\%$
Relative Density	1.08 at 25 C°

3. Mix proportions

There is a lack of design code for the mix design of UHPC, so the mix UHPC mix is optimized according to the literature and prepared as per the requirement of ASTM C1856 [37]. Based on the laboratory trails, the totally seven UHPC mixtures are optimized and shown in Table 6. The water to binder ratio is fixed at 0.24 continuously for all mixes. Seven types of UHPC mixtures are divided into two series, each with a different dosage of cement and nanoparticles while always totalizing low cement content of 570.45 kg/m^3. These include one-control mixtures (CON mix -without nanoparticles), and the other six mixtures are produced with different dosage levels of nano Al2O3 particles 0.5% to 3% replacement with an increment of 0.5%, respectively. The content of polypropylene (PP) fibers amount is added by 0.4% to the volume of concrete. Silica fume, Quartz powder and sand content water were fixed in all mixes at 171.20 kg/m^3, 245.28 kg/m^3 and 1245.38 kg/m^3, respectively, respectively. All seven mixtures are prepared in a mortar mixer with a total mixing time of 30 minutes. Furthermore, mixing is always done in a laboratory environment using dried, tempered aggregates and powder components. The room temperature is $24C°$for mixing, testing, and concreting. The mixing, casting and curing procedure are adopted as summarized below:

- The nano Al2O3 particles are dispersed in 20% PCE and 20% water using a bath sonication technique for 30 min.
- Cement, quartz powder, sand, and Silica fume are placed in a mortar mixer and mixed slowly for 5 min.
- 60% of PCE is mixed with 80% water are added slowly to the dry mixture. The mixing continues up to granules' transformation (about 10 min).
- After forming the granules, the dispersed nano Al2O3 particles mix is added slowly and mixing is continued for another 5 min. Subsequently, the remaining 20% of the PCE is added to the mixture and mixed for 5 min at high speed until the transformation of the mixture into a homogenous mix.
- Then polypropylene fibers are added to the mixture slowly and mixed for 5 min. After that, the mixing is continued for a further period of 5 min to well dispersion of the UHPC mixture.
- The produced UHPC mixture is partially taken for fresh concrete tests. The remaining concrete is filled with moulds, with the plastic sheets covering the surface and kept for 24 hours at laboratory temperature.
- After that the demoulded specimens are placed in normal water curing at room temperature.

4. Testing of Concrete

4.1 Mini slump flow

The flow of fresh concrete mix is measured by mini-slump cone according to the ASTM C1437-07 [38]. The freshly prepared concrete mix is placed into the mini slump cone having bottom diameter100 mm and a top diameter of 70 mm and height 50 mm. The concrete mix is filled with a single layer without compaction, which is specified by ASTM 1856/C1856-17 [39]. Then the cone is lifted slowly which allows the concrete to flow. The largest width of concrete spread is noted as d1and then the d2 is measured by right angles to d1.

4.2 Compressive strength

The compressive strength of specimens is tested as per the IS: 516-1959 [40] at the age of 1, 3, 7, 14, 28and 56 days of curing. The saturated surface dried 50mm cube specimens are tested in 3000KN AMIL digital compression testing machine at a rate of 900 N/s.

4.3 Modulus of elasticity

As per ASTM C 469 – 02 [41], the static modulus of elasticity of concrete is measured by using of deflection gauge method in cylindrical specimens having a size of 100 mm diameter and 200 mm height at the age of 28 and 56 days. In this method of testing, the specimen is placed between the loading plates of digital CTM and the dial gauge is fixed on both sides of the bottom plate. Then apply the axial load continuously without shock at constant rate 0.25 N/mm2. The deflection gauge is used to record the length changes of the specimens which is used for the axial strain measurement of specimens.

4.4 Water permeability

The depth to which water may penetrate specimens (100mm cubes) under pressure is determined after 28 and 56 days using a water permeability device, following the guidelines provided in BSEN 12390-8:2009 [42]. Apply a water pressure of 500 kPa for a duration of 72 hours, and thereafter remove the specimen from the device. Clean the surface where water pressure is exerted to eliminate any remaining water. Divide the specimen into two equal halves at a right angle to the surface where the water pressure is exerted. Determine the greatest extent of penetration under the designated region and document using a Vernier calliper. The greatest depth of penetration, measured in millimetres.

4.5 Micro-structural Properties

The broken concrete samples after compression testing are collected and performed SEM.

5. Results and Discussion

5.1 The Mini Slump Test

The mini-slump flow was tested according to the ASTM C1437-07 [38]. The Table 6 listed the flow properties of UHPC with and without nano Al2O3 particles. It shows that the lowest slump flow spread of 240 mm is observed in the control mix without nanoAl2O3 particles, and for the nano Al2O3 admixed mixes the slump value ranges from 242mm to 251 mm. From the results, it is understood that the inclusion of nano Al2O3particles has improved the flow of mix without adjusting the superplasticizer dosage. However, 3% of nano Al2O3 particles added to mix is reached largest spread of 251 mm, which increased flow by 3.33%, in comparison to the control mix. Kazempour et al. 2015 [43] and Khorasani et al. 2015 [44] reported that the higher dosage of nano alumina are decreased the flow of mortar, due to increasing the cohesiveness of the

Table 6. The mix proportions.

Mix ID	**Cement** (kg/m^3)	**Nano Al2O3** (kg/m^3)	**Silicafume** (kg/m^3)	**Sand** (kg/m^3)	**QP** (kg/m^3)	**Water** (kg/m^3)	**PCE** (kg/m^3)	**PP Vol.** (%)
CON	570.45	00.00	171.20	1245.38	245.28	178	22.818	0.4
0.5AL	567.60	02.85	171.20	1245.38	245.28	178	22.818	0.4
1.0AL	564.75	05.70	171.20	1245.38	245.28	178	22.818	0.4
1.5AL	561.89	08.56	171.20	1245.38	245.28	178	22.818	0.4
2.0AL	559.04	11.41	171.20	1245.38	245.28	178	22.818	0.4
2.5AL	556.19	14.26	171.20	1245.38	245.28	178	22.818	0.4
3.0AL	553.33	17.12	171.20	1245.38	245.28	178	22.818	0.4

mixtures. When the nano alumina particles were directly added in mix without dispersing, it has increased the yield stress and viscosity of mortar, due to their high reactivity and surface area. Nazari and Riahi (2011a; b) [45,46] has reported that the nano alumina particles blended concrete mixtures have low slump values, the cement substitute with nano alumina particles will increase the water demand due to the increase in surface area. But in this study, the addition of nano Al2O3 particles has increased the fluidity of the mix and become more workable and easier to placement, in all nano Al2O3 particles blended mixtures. This may due to thewelldispersionofnanoAl2O3particleshasreduced the yield stress and viscosity of the mix, which leads to enhance the workability of the fresh concrete.

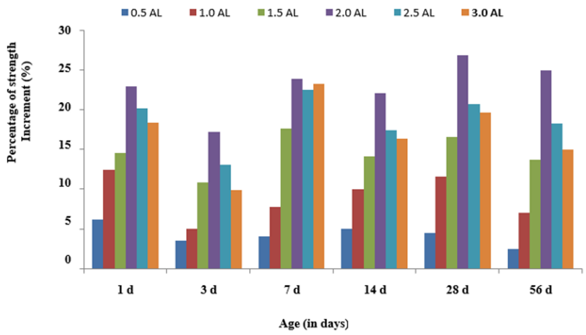

Figure 4. The percentage of enhancement in compressive strength.

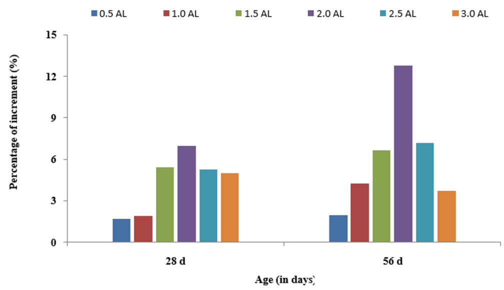

Figure 5. The percentage of enhancement in modulus of elasticity.

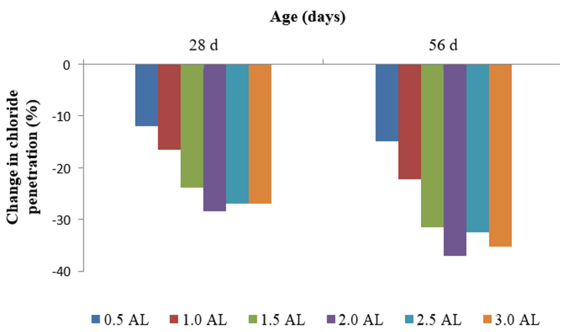

Figure 6. The percentage of reduction in water penetration of depth under pressure.

5.2 Compressive Strength (CS)

The CS of specimens was tested as per ASTMC109/C10M – 07 [47] at the different ages of curing. The effect of nano Al2O3 particles replacement on the compressive strength and percentage of strength enhancement are depicted in Fig. 4. It could be seen that the minimum CS noticed in the control mix was 55.05 MPa, 71.28 MPa, 91.41 MPa, 103.32 MPa, 122.65 MPa and 129.57 MPa at the age of 1, 3, 7, 14, 28, and 56 days respectively. However, the CS of UHPC is increased with every case of nano Al2O3 particles replacement percentage and it has higher than the control mix strength. Whereas, the highest CS observed in 2.0 AL mix was 67.65 MPa, 83.57 MPa, 113.28 MPa, 126.13 MPa, 155.59 MPa and 161.92 MPa at an age of 1, 3, 7, 14, 28 and 56 days respectively. The 2% of Al2O3 particles replacement is found as optimum level for efficiency and economical point of view, which has increased CS by 22.89%, 17.24%, 23.93%, 22.09%, 26.86% and 24.97% at 1, 3, 7, 14, 28, and56 days respectively in comparison to control mix. This strength improvement is mainly attributed due to the filler effect and pozzolanic reactivity of nano Al2O3 particles. The nano Al2O3 particles acts as a dynamic reactive substance in the arrangement of nuclei cite during C-S-H gel formation as well as it's promoting hydration process, which leads to refined pores and densifies the microstructure. In addition, the nano Al2O3 particle has reduced the distance between cement grains and SCM particles, which makes the cement matrix as more homogenous and compact microstructure. As a result, it enhanced the CS rapidly at early ages and later it increased gradually with curing days.

5.3 Modulus of Elasticity (MOE)

The MOE test was performed in accordance to ASTM C 469 – 02. The influence of nano Al2O3 particle replacement on the MOE of UHPC mixes is depicted in Fig. 5. It is observed that the modulus of elasticity is increased with nanoAl2O3 particles content up to 2% and beyond that dosage, it has decreased the MOE but those values still higher than the control mix. The maximum modulus of elasticity value of 38.98 GPa is observed for 2.0 AL mix and the minimum of 36.43 GPa for the control mix at 28 days. Similarly for 56 days, the minimum and maximum modulus of elasticity achieved by control mix and 2.0 AL mix, which is 38.29 GPa and 43.19 GPa respectively. The 2% of Al2O3 particles were enhanced the modulus of elasticity by 7.00% and 12.80% at 28 and56 days. The compressive strength and porosity of specimens play the vital role on the modulus of elasticity of concrete. The nano Al2O3 particles inclusion has enhanced the compressive strength of concrete and reduced the voids of UHPC. Furthermore, the nano Al2O3 particles has arrests cracks and restrict the continuity of slip plans which lead to improve the toughness of cement matrix. This might reason for increasing of modulus of elasticity of UHPC samples.

5.4 The water permeability

The water infiltration depth under pressure of UHPC mixes was examined conferring to the standards outlined in BS EN 12390-8:2009. Figure 6 shows the impact of substituting nano Al2O3 particles on the water penetration depth in UHPC samples after 28 and 56 days. after 28 days, the control mix showed a maximum water penetration depth of 1.34 mm, which decreased to 1.08 mm after 56 days. On the 2.0 AL mix, the minimal depth of water penetration was described to be 0.96 mm at 28 days and 0.68 mm at 56 days. The minimum water permeability is observed in the 2.0 AL mix is represented the less amount permeable voids in the mix, which is due to the filler effect of nano Al2O3 particles. Similar to the water absorption results, the inclusion of nano Al2O3 particles has reduced the volume of permeable voids in UHPC, which has reduced of the water penetration depth at all ages. Similar results were found in the previous research work of Arefi et al. 2011 [48].

5.5 SEM analysis

The samples are collected after the testing compressive strength of concrete and immersed in anhydrous ethnonal to stop further hydration process. These samples are utilized for studying micro-structural analysis by SEM test. The microscopic images of UHPC surface is captured at an age of 3, 14, and 28 days as per the ASTMC1723–16 [49]. Figure 7 displays SEM images of the CON mix and 2.0 AL mix, taken on the 28th day. The micrographs in Fig. 7a and b clearly demonstrate the presence of a significant number of micro pores in the cement matrix. The micrographs in Fig. 7c and d show that a homogenous densified microstructure is found in the cement matrix of the 2.0 AL mix specimens. The sample containing 2% nano Al2O3 particles exhibits a denser micro-structure, with fewer spaces being filled by the nano Al2O3 particles. This is attributed to the quick processing of the C-S-H gel. The Ca(OH)2 crystals have been reduced and transformed into more densely packed hydration products. The acquired results are consistent with those published by other studies.

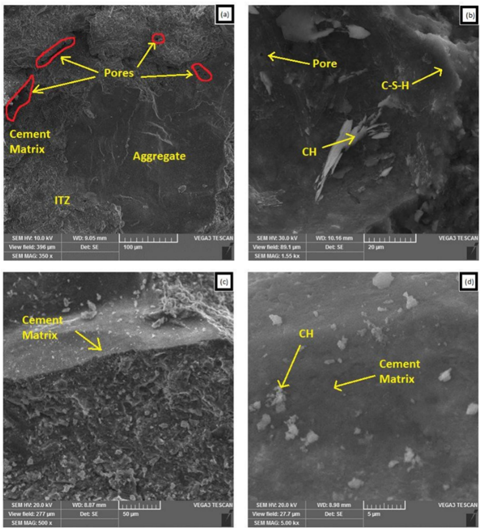

Figure 7. The SEM micrographs (a) & (b) CON mix, (c) & (d) 2.0 AL mix

6. Conclusions

The results are confirmed that the inclusion of nano Al2O3 particles is improved workability properties of the fresh UHPC and following results are observed while compared with control concrete without nano Al2O3 particles. 3% of nano Al2O3 particles is found optimum for enhancement of workability. The inclusion of 2% of nano Al2O3 particles were improved the mechanical properties of UHPC and following conclusion are drawn control concrete without nano Al2O3 particles. The compressive strength enhanced up to 26.86%. The modulus of elasticity enhanced by 7%. The SEM micrographs reveal the increase of nano Al2O3 particles were led to increase the hydration process and acts as a nucleation cites of CSH gel formation and pore-filling material in a cement matrix, therefore densified microstructure was produced compared to the control concrete. Also, the Ca(OH)2 crystals were declined and a compact development of hydration products was perceived by exploiting this micrograph. The inclusion of 2% of nano Al2O3 particles were found as optimum percentage for durability enhancement. The water penetration depth, under pressure and sorptivity is reduced up to 37.04% and 15.34%, respectively.

Authors' contribution

S. Hariprasad Reddy: conceptualization of research, drafting the research manuscript, reviewing, editing, and preparation of the final draft. K. Mahaboob Peera: Data collection, manuscript review, and editing. A. Surendra, C. Arvind Kumar, M. Leelakar: contributed to various aspects of the research work as needed.

Declaration of competing interest

The authors declare no conflicts of interest.

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Data availability

The data and images used in this study were collected and processed directly by the researchers as primary research materials. Requests for data sharing can be fulfilled upon reasonable inquiry to the research team.

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