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Experimental Investigation on Mechanical Properties of Cementious Composites by Incarporation of MWCNTS and Nano-Al_2O_3

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Abstract--- **The contemporary building sector intensively depends on the cement based material. As compared to the previous times, the demand for the progress of the new infrastructure is at hike; moreover it demands the construction of efficient, sustainable, and durable building materials. Based on the worldwide screening report, it is apparent that Nanotechnology and Nano materials can intensively affect the material at nano scale which in turn effects the properties at micro and macro level leading to development of cementious composites with improved properties such as mechanical performance, durableness, resistance to brittle failure, ability to resist crack, etc. In the present experimental work a set of cementious composites were incorporated with MWCNTS and their percentage was fixed to 0.75% by weight of cement, while a set of specimens were incorporated with 3wt% Al_2O_3 nanopowder and a composition with combination of both 0.75wt% of cement MWCNTs and 3wt% of cement Al_2O_3 nanopowder. Dispersion of both MWCNTS and Al_2O_3 nanopowder was done using ultrasonic sonicator. These specimens were tested for compression and flexure and their mechanical properties such as Compressive strength, Flexural strength, Toughness and Ductility, Indirect tensile strength were evaluated and these properties were compared with that of plain cement control beams. Microstructure of composite was also assessed using SEM (Scanning Electron Microscopy).From the study it was concluded that there was improvement in mechanical properties and microstructure of the composites.**

Keywords--- **MWCNTS, Nano- Al_2O_3, Cementious composites, Mechanical properties, SEM**

I. INTRODUCTION

Concrete is a vital building material in construction sector with regards to its undeniable properties like high strength, mould-ability, to various profiles, durability despite of its ecological concern. Approximately 20 billion metric ton of concrete is being produced every year. It is a composite material with wide range of length scales from millimetre to nanometre. Generally concrete is considered as one material by taking into account its compressive strength and other engineering properties, but at micro and nanoscale, it is having different type of compositions with multifaceted characteristics. There has been always a correlation between the microstructure and bulk properties and researchers are making efforts to set up this correlation in concrete. [1]

Cement is regarded as the key binding material of concrete, which is extensively used for the progress of current civil infrastructures. As cement is a key constituent of the concrete, its utilization increases enormously in construction. In recent days, as nanotechnology is an emerging field in material science which deals with the study of material structure and properties at nano level the technologists and scientist are trying to utilize nanotechnology in the material world. Hence utilization of the nanotechnology in the production of the cement would have the encouraging impact, which will certainly bring revolution in the cement and concrete research. Hence, if the nanotechnology is incorporated with the conventional construction and building research, then the new materials will possess high value and smart properties.

Screening into the light of literature, it is reported that the nano-concrete can be outlined as a concrete made with Portland cement particles that are less than $5x10^2$ nm as the cementing agent. Moreover, the bottom up nano engineering process viz., amalgamation of nano Al_2O_3, Fe_2O_3, SiO_2, TiO_2, nano fibre or nanotube and nano clay, etc. into cement system during mixing has established itself as a hopeful method, which encompasses the structure at the nanometre scale to develop multifunctional cementitious composites with superior mechanical performance and durability. Therefore, nano material implanted cement based material possesses a range of new and improved properties such as: high ductility, self-healing, self-crack controlling ability, low electrical resistivity, and self-sensing capabilities. Moreover, it is also reported that the application of the nano porous thin film on the aggregate surfaces before mixing of the concrete not only brings a range of novel properties, but also improves the interfacial transition zone (ITZ) in concrete system .[2]

In an attempt to introduce cementious materials with novel and advanced properties the present-day building sector is striving for. Performance augmentation of cementitious system consisting of judicious utilization of materials, developing more durable and sustainable Concrete to reduce the maintenance and life cycle cost. There are two approaches for the performance augmentation of cement based materials, one is to find out suitable substitute materials to replace cementitious materials such as geopolymers and another approach is regulation the performance of cement by admixtures. [1]

A material is called a composite material if it is made of two or more ingredient materials that have different chemical and bodily properties which when pooled produce a material whose characteristics are different from the constituent components. Composite materials are favoured than customary materials because they are lighter, stronger, or less expensive. Typical engineered composite materials include: Composite building materials, Metal composites, Reinforced plastics like fibre reinforced polymer, Ceramic composites etc.

Individual materials present in the composite are referred to as constituent materials. The constituent material includes matrix and reinforcement. The constituent that is continuous in the composite is called as matrix ant it supports the material at their relative positions. The reinforcement helps in improving the matrix properties by imparting their physical and mechanical properties. Concrete is one of the most familiar man-made composite materials. The constituents are aggregates which are held in cement matrix. Concrete can sustain under large compressive force, but cannot survive tensile loading. Hence concrete is reinforced by steel bars.

Nanotechnology is an emerging field of material science dealing with study of matter at nano level. In the year 1959 Richard P. Feynman was the first to make first move to initiate nanotechnology by a famous lecture at Califonia Institute of Technology .However, in the latest two decades the study on this subject has become very bouncy. Yet, nanotechnology still continues to be one of the most bouncy research areas with both innovative science and pioneering applications. The challenges regarding the advancement in technology, instrumentation and broadening of basic scientific knowledge is an aid for advancement in research areas. The major factor that effects significantly in nanotechnology is the size of particles i.e., as particle size changes from macro to micro to nano scale the material properties are significantly affected. At nano level, gravity becomes unimportant, electrostatic forces take over and quantum effects come in. Moreover, as particles become nano-sized, the fraction of atoms on the surface gets amplified in relation to those inside which results in novel properties. Researchers are exploring these properties to produce materials with appreciably new properties.

In accordance with the survey conducted by Canadian Program on Genomics and Global Health (CPGGH), among the most influential fields that impact the developing world nanotechnology in civil engineering was graded 8 of 10 applications. Nanotechnology can be applied for design and construction processes in many areas. As there is a demand for high tech material and technology in construction sector, application of nanotechnology can be a promising solution. [3]

In the previous research works study was conducted on reinforced cement composite beams. The composites were incorporated with MWCNTs and Carbon fibres. The percentage of MWCNTS was varied i.e. 0.25, 0.5, 0.75 and 1% by weight of cement and the percentage of CFs was 0.25% by weight of cement. Dispersion of MWCNTs was done using Ultrasonic energy and three point bending test was conducted. Also a set of cement based cylindrical bars incorporated with MWCNTs and CFs with 0.5%,2.25% by weight of cement were prepared and Direct tensile test was conducted and the mechanical properties such as Flexural strength, Toughness, Ductility, tensile strength, stress-strain behaviour were studied the results obtained represented enhancement in the mechanical properties.[4][5].In the research work of Morteza Bastami et al. effect of elevated temperature on high strength concrete (HSC) modified with nano-SiO_2 and its compressive and tensile strength, Spalling and mass loss (f_c>80Mpa) were studied the results obtained demonstrated that there was improvement in mechanical properties at elevated temperatures .Also due to the presence of Ns there was an increase in residual compressive and tensile strength, and spalling and mass loss were decreased due to increase in permeability.[6].Similarly other research works reported

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that use of Nano-silica in cementious paste had improved Mechanical properties and the SEM (Scanning Electron Microscopy) Results represented that presence of nano-silica results in calcium silicate Hydration which results in improved properties of cement. It also indicates that nano-silica behaves not only as a filler to improve microstructure, but also as an activator to promote pozzolanic reaction [7-13]. The research works of Ali Nazari et al. Reported that use of Al_2O_3 nanopowder in self compacting concrete containing Ground Granulated Blast Furnace Slag (GGBS) resulted in improved Physical, Mechanical and pozzolanic reaction.[14][15].The present experimental work study was conducted on Mechanical properties and micro structure of cement specimens incorporates with MWCNTS and Al_2O_3 nano powder.

II. MATERIALS AND EXPERIMENTAL METHODOLOGY

Materials

The cement used was Ordinary Portland Cement of grade 43 Manufactured by Coromandel Info Tech India Limited in accordance with IS 8112-1989.It was tested as per IS-4031-1988 recommendation for hydraulic cement [16]. The normal consistency and the specific gravity of cement were found to be 33% and 3.12 respectively. The initial and final setting times of cement were found to be 75 minutes and 260 minutes respectively.

The multi-walled carbon nano tubes (MWCNTs) and Al_2O_3 nano-powder were obtained from Sigma-Aldrich. The properties of the multiwalled CNTs ,nano-Al2O3 and Ethanol used are presented in Table 1,2,3.The MWCNTs were of industrial grade with a purity of greater than 95%.

The Properties of MWCNTS used.

Table 1: Properties of MWCNTS used

Manufacturer	Sigma-Aldrich Co, USA
Diameter	10-30 nm
Length	1-2 nm
Purity	95%
Surface area	350 m^2/g
Bulk density	0.05-0.17 g/cm^3

Properties of Al_2O_3 nano particles

Table 2: Properties of Al_2O_3 nano used

Manufacturer	Sigma-Aldrich Co, USA
Particle size	13nm
Assay	99.8% trace metal basis
Melting point	2040℃
Molecular weight	101.96 g/mol
Form	Nano powder.

Properties of Ethanol:

Table 3: Properties of Ethanol.

Product	Ethyl alcohol
Assay	99.9%
Water	0.1%
Residue after evaporation	0.001%max
Alkaline	0.1% max
Methanol	0.5% max

III. DISPERSION OF MWCNTS AND AL_2O_3 NANOPOWDER

MWCNTs and Al_2O_3 nano particles undergo agglomeration due to their Vander wall's bonding which is a critical issue. Therefore MWCNTs were pre-dispersed by sonication method. Ethanol was used as dispersant and sonication was done for 15 to 20 minutes.

The ultrasonic sonicator used was manufactured by SMPS Electronics, Mumbai, and Frequency of oscillation: 30±3 MHz Power 50KW. Nano scale materials like carbon nano tubes etc are dispersed using ultrasonic methods. Nano-sized materials have relatively large-area-to volume ratios as well as the scale of action of the ultrasonic waves. Ultrasonic Sonicator was used to disperse the MWCNTs and Al_2O_3 with dispersant ethanol. The Ultrasonic sonicator was filled with 2000 ml of water .It consists of an ultrasonic water bath and from the bottom of which the generator ultrasonic waves are passed up. The nano material mixed with the dispersant were put to a glass beaker and kept in the water bath of sonicator for sonication. The ultrasonic waves move from bottom and they enter the glass beaker and they give the energy to the nanomaterial .The nano particles get dispersed due to ultrasonic energy. The ultrasonic bath used is shown in figure 1.

Figure 1. Ultrasonic Bath Dispersion of MWCNTs and nano-Al_2O_3

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IV. METHODOLOGY ADOPTED FOR SPECIMEN PREPARATION

For the preparation of specimens the cement used was OPC of grade 43. And also the nano particles to be incorporated i.e. MWCNTs and nano Al_2O_3 were used.
The aspect ratio of the specimen was fixed as pe ASTM D790-02. The mould sizes used were 20x20x80mm for beams (Metaxa et al.2007,Chan and Andrawes 2009), and 20x20x20 mm for cubes and the following steps were followed i.e. the Moulds were cleaned, greased and kept on level surface. Amount of cement required and water required were weighed and kept in containers. The w/c ratio maintained was 0.4.The nano particles were stirred and dispersed thoroughly by sonicater prior to addition to cement. The nano particles after sonication were mixed with the cement paste. The paste was placed in the moulds with proper compaction and demoulded after 24 hours. After 24 hours the specimens were demoulded and kept for curing for a period of 28 days.

V. EXPERIMENTAL TESTS CONDUCTED.

The tests conducted on the specimens were three point bending test, Compression test and split tensile test. And the Mechanical properties evaluated from the three point bending test were Ultimate Load, Flexural strength; Toughness Index, Ductility Index, Compressive strength and tensile strength were obtained from the respective tests.

VI. FLEXURE TESTING DEVICE— ASTM C384 - 02[18]

Flexural testing device was arranged in accordance with ASTM C384-02.
According to these guidelines the apparatus for three points bending test must fulfil these principles.

- Constant distance must be maintained between the point of application of load and the supports.
- The loading must be normal to the specimen surface and eccentricity must be avoided.
- The reactions must have their direction parallel to the direction of applied loads throughout the test.
- The rate of loading must be uniform and in such a manner that avoids shock.

The machine has capacity of 10KN and strain rate was maintained at 0.05mm/min.and least count of the device is 0.206 kg.

Figure 2. Loading frame for 3 point bending test

Compression Testing Device

The UTM used for compression test was of capacity 10 Ton and the loading rate was maintained at 2mm/min. The experimental setup is as shown in figure 3.

Figure 3. Compression testing machine

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VII. RESULTS AND DISCUSSION

Table 4. Specimen particulars

Sl.No	Specimen Particulars	composition
1	PC	Plain cement
2	A1	Plain cement+0.75wt% MWCNTS
3	A2	Plain cement+3wt%Al_2O_3
4	A3	Plain cement+3wt%Al_2O_3+ 0.75wt%MWCNTS

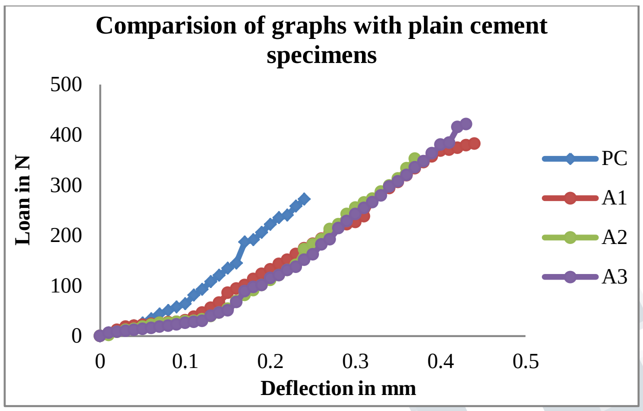

Figure 4. comparison of graphs with plain cement specimens

The graph obtained from the three point bending test shows load vs. mid-span deflection for plain cement specimens and for Specimens incorporated with MWCNTS 0.75wt% of cement and 3wt% of Al_2O_3 and a specimen with mixture of the two. The material behaviour was found to be more plastic and there was no perfect yield point as observed from the graphs.As there was change of mix proportion in the composites, the ultimate load and corresponding deflection increased considerably.

From the results it is concluded that the ultimate load for mix proportion of A3 was maximum i.e.450N as compared to other specimens with respect to plain cement specimen i.e.270N.There was almost 67% increase of ultimate strength with respect to plain cement specimen, and for A1 and A2, it was 41% and 30% increase with respect to plain cement specimens. The increase in strength may be due to dense and compact composition and also Al_2O_3 results in chemical reaction which leads to formation of C-S-H gel.

From the bar chart showing the variation of deflection for different compositions, It can be concluded that the deflection for the mix proportion MWCNTS 0.75wt% of cement was maximum i.e. 76% increase in deflection and also the deflection for A3 was comparably same i.e. 72% .As the deflection for maximum ultimate load increases it indicates that the composite has greater ability to carry loads, as greater amount of energy is needed to produce crack. The reason for increase of deflection for max load carrying capacity is the crack arresting nature of the MWCNTS and the reason for comparably same amount of increase of deflection for A3 composition may be due to improper dispersion of MWCNTS and agglomeration of Al_2O_3 nanoparticles.

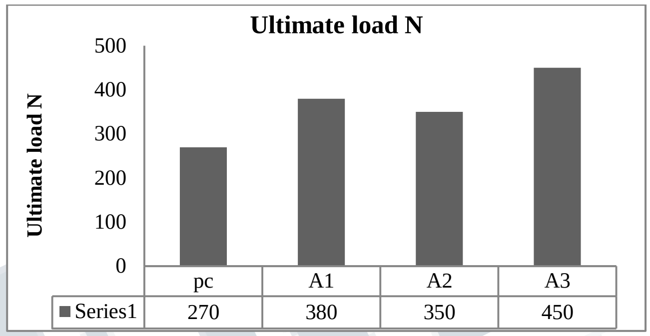

Figure 5. Bar chart representing ultimate load of different compositions.

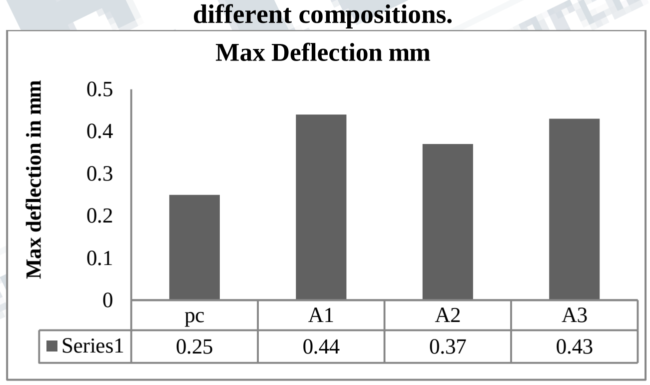

Figure 6 . Bar chart representing maximum deflection of different compositions

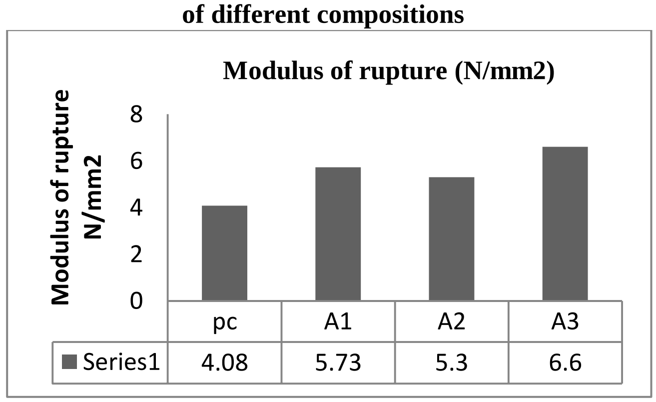

Figure 7. Bar chart representing modulus of rupture of different compositions.

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Flexural Strength

Flexural strength or Modulus of rupture is the max stress or internal resistance at final rupture .From the bar charts it can be noted that there is an increase in flexural strength especially for the specimens containing MWCNTS. The modulus of rupture for the mix A3 was maximum i.e. 61.7% increase with respect to plain cement specimens and the composition A1 also had 40.44% increase with respect to plain specimens and the composition containing A2 also had 30% increase in flexural strength as compared to plain cement specimen.

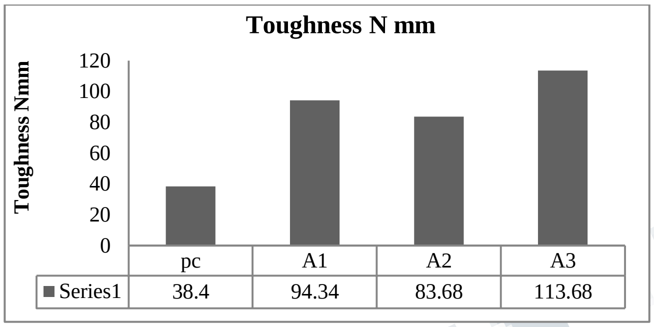

Figure 8. Bar chart representing toughness of different compositions

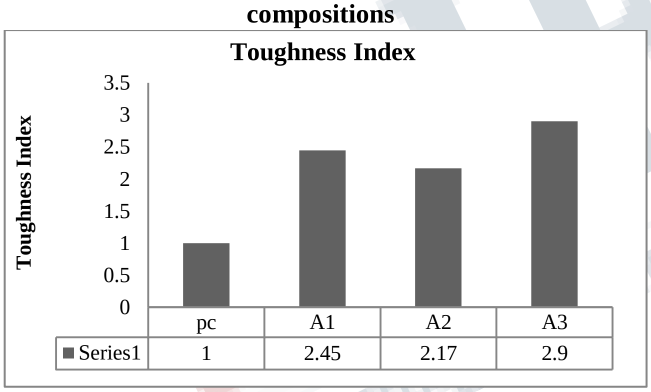

Figure 9. Bar chart representing toughness index of different compositions

Toughness

Energy absorbing capacity of the material is called toughness. Increased toughness means increased capacity to take large load and undergo large deformations before failure. Moreover it indicates increase in resistance to impact, fatigue and impulse loading .It is measure of area under load deflection curve. From the bar chart it can be concluded that the composition of PC+3wt%Al_2O_3+0.75wt%MWCNTs has higher Toughness and toughness index with respect to plain cement specimen i.e.190% increase. Moreover there was 145%, 117% increase in Toughness index as compared to plain cement specimens for 0.75wt%MWCNTs and 3wt%Al_2O_3 respectively. The increase in toughness may be due to reduction of pores in cement paste due to Al_2O_3 nanopowder which results in dense and compact specimen and crack arresting ability of MWCNTs.

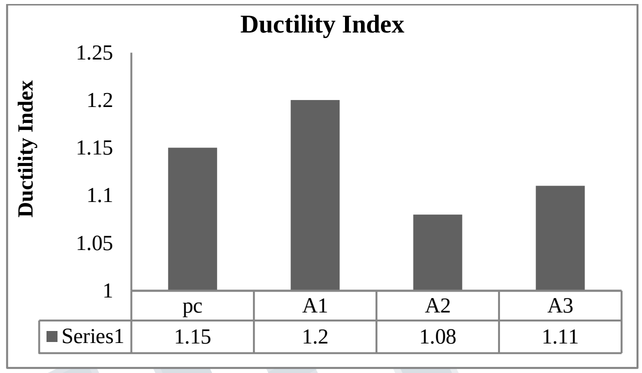

Figure 10. Bar chart representing toughness index of different compositions

Capability of material to experience large deformations with no rupture before failure is termed as Ductility .From the bar graph it is evident that the D.I has improved for the composition A1 i.e. 4.3% as compared to plain cement specimen, however the D.I has not improved for other specimens this may be due to improper binding between the MWCNTs and cement paste and improper dispersion of MWCNTS and agglomeration of Al_2O_3 nano particles.

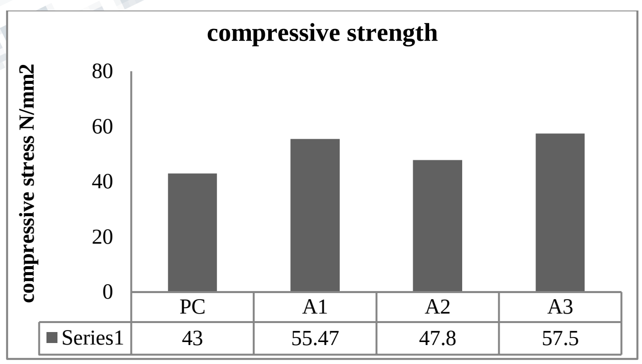

Figure 11. Bar chart representing compressive strength of different compositions

Compressive strength

The bar charts shows the variation of compressive stress for different compositions there was an increase of 33%,29%,11% increase in compressive strength for compositions A3,A1,A2 respectively .The compressive strength for 3%Al2O3 specimens did not have much enhancement of compressive strength this may be due to

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improper dispersion and causing clusters which leads to increased air voids .however the increase of compressive strength for other two specimens maybe due to proper binding between the host matrix and MWCNTS which also results in crack bridging and hence results in increase of strength.

Indirect tensile strength

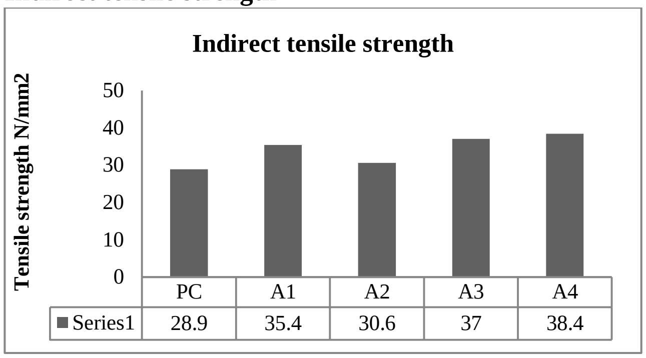

Figure 6.9. Bar graphs representing indirect tensile strength

From the bar graphs it is evident that there is enhancement in the tensile strength of the composites as compared to plain cement specimens. Strong bonding between the MWCNTs and the host matrix and increase of pozzolanic reaction due to nano Al_2O_3 may be the key reason for enhancement of the strength. It is concluded that the specimens containing MWCNTs had enhanced tensile strength i.e. 22.2%, 28%, 33% as compared to specimens incorporated with nano Al_2O_3 which is 6% enhancement with respect to plain cement specimens; this may be due to clustering of Al_2O_3 in the composites.

VIII. SEM RESULT

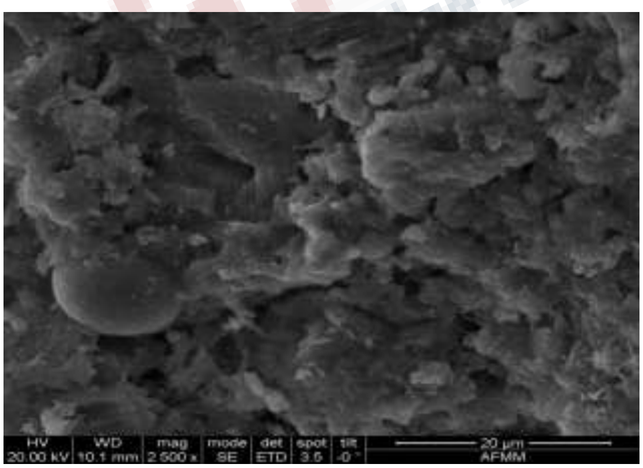

Figure 12. SEM image of cement paste modified with Al_2O_3 nano particles.

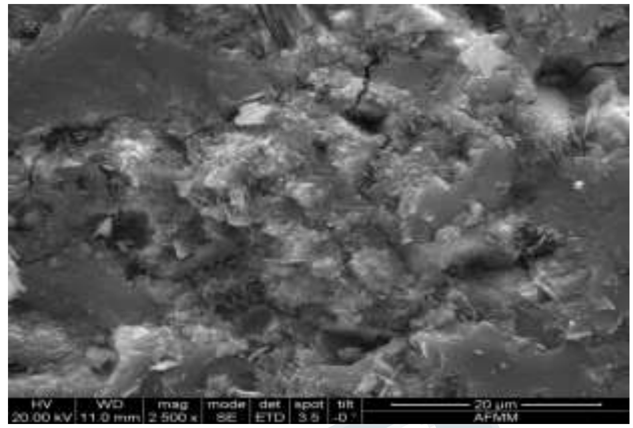

Figure 13. SEM image of cement paste modified with MWCNTs nano particles.

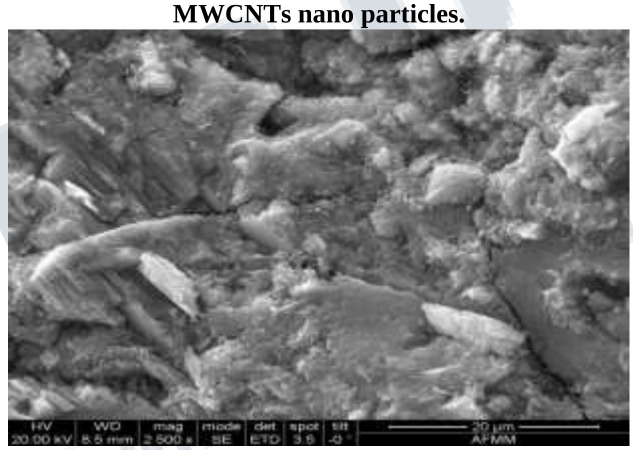

Figure 14.SEM image of cement paste modified with MWCNTs+Al_2O_3 nano particles.

The SEM images for different compositions is showed in fig 4,5,6 .Figure 4 shows the SEM images of Al_2O_3 nano powder with cement paste as host matrix from the figure it can be inferred that there was good pore filling in the matrix; that's why it may be the reason for increased strength Also it is observed that there is good dispersion and from fig 5 and 6 it can be deduced that the dispersion of MWCNTs was effective for a greater extent and there was an effective bonding between the MWCNTs and cement paste and also there was reduction of pores and improved homogeneity by C-S-H gel formation, this can provide more adhesion at interfacial zone which is an aid for enhancement of mechanical properties. However at some places it was observed that there was clustering of MWCNTs and Al_2O_3 nano particles for a little extent which can be reduced by using effective dispersion techniques.

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IX. CONCLUSIONS

The results obtained in this study can be summarized as follows.

- From the results it is concluded that the ultimate load for mix proportion of A3 was maximum i.e. there was almost 67% increase of ultimate strength with respect to plain cement specimen. The increase in strength may be due to dense and compact composition and also Al_2O_3 results in chemical reaction which leads to formation of C-S-H gel.
- As the deflection for maximum ultimate load increases it indicates that the composite has greater ability to carry loads, as greater amount of energy is needed to produce crack. The reason for increase of deflection for max load carrying capacity is the crack arresting nature of the MWCNTS.
- There was an increase in flexural strength especially for the specimens containing MWCNTS.
- From the results it can be concluded that the composition A3 has higher Toughness and toughness index with respect to plain cement specimen i.e.190% increase. Moreover there was 145%, 117% increase in Toughness index as compared to plain cement specimens for A1and A2 respectively. The increase in toughness may be due to reduction of pores in cement paste due to Al_2O_3 nanopowder which results in dense and compact specimen and crack arresting ability of MWCNTs.
- From the bar graph it is evident that the D.I has improved for the composition containing 0.75 wt% MWCNTs i.e. 4.3% as compared to plain cement specimen; however the D.I has not improved for other specimens this may be due to improper binding between the MWCNTs and cement paste and improper dispersion of MWCNTS and agglomeration of Al_2O_3 nano particles.
- From the SEM images it was concluded that there was improvement in micro structural properties of the specimen.

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