Influence of Nano-Materials in High Strength Concrete

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ABSTRACT

The recent research in nanotechnology has demonstrated that nanomaterials can improve the characteristics of concrete such as mechanical and durability properties (Shih, 2006). This experimental study compares the strength and durability of different nanomaterial concrete. When nanomaterials are added to concrete it fills pores present in the matrix to provide an exceptional surface area to volume ratio and improves the basic property and reactivity of the material. This in turn enhances the strength and durability of concrete. The influence of nano silica, nano alumina and nano titanium dioxide on M60 high strength concrete were discussed in this experimental study. The cement was partially replaced with 40% ground granulated blast furnace slag and different percentage of nanomaterial like 2% to 6% for preparing the high strength concrete. Mechanical Properties like compressive strength, tensile strength and flexural strength of control mix, concrete with pozzolanic material and nanomaterial concrete were compared. Accelerated corrosion test was conducted in saline and acidic conditions to determine the durable nanomaterial concrete. The durability of these concrete was confirmed using corrosion analysing instrument and corrosion resistivity meter. From these test results the best nanomaterial concrete in terms of mechanical properties and durability was determined.

KEY WORDS: Nanotechnology, Nanomaterial, Nano Silica, Nano Alumina, Nano Titanium dioxide, Accelerated Corrosion Test.

1. INTRODUCTION

There had been a lot of developments in the field of concrete technology in the past years; varieties of concretes have been developed to meet the specific functional requirements of concrete. The manufacturing of concrete needs massive amount of cement as a binding material and the production of cement is considerably increases the emission of greenhouse gases to the atmosphere from the cement manufacturing industries. Hence, various waste products form industries have been consumed in concrete as partial replacement to binder (cement) to attain better properties than ordinary Portland cement and have proved to be quite beneficial over concrete containing no mineral admixture.

Recently, the research has focused on nano materials and it is paving a new avenue to the feature in the field of science and technology. A proper usage of nano-particle material in cement mortar and concrete leads to generate of new material product with entirely different enhanced properties. Available experimental research studies indicated that incorporation of nano-silica (nano-SiO_2) with different dosages in cement based composite materials has established in strength gain due to hydration seeding effect and high pozzolanic activity (Shih, 2006; Ali Nazari, 2011). On the other hand Shekari (2011), observed an improvement in the mechanical properties of high performance concrete by incorporating nano alumina in concrete. Nazari (2010), determined that nano titanium oxide act as nanofillers and improve the pore structure of self-compacting concrete. They also found that there is an increase in the compressive strength, split tensile strength and flexural strength of self-compacting concrete at a particular dosage and afterwards it decreases. Bhuvaneshwari (2016), achieved improvement in mechanical properties of concrete by incorporating nano oxides. They observed that there is a delay in setting time of concrete when nano particles are blended in concrete and also observed that the nano oxides increase the density, reduce porosity and improves the bond between cement matrix and aggregate. Flores-Vivan (2013), found that nano Titanium oxide act as photocatalytic material and destroys the organic pollutants and helps in removing inorganic matter from the surface of architectural concrete. According to Ozyildirim (2010), examined how nanomaterial improved permeability and hardened strength of concrete and found that very little amount of nano materials like nano silica and nano clay can improve the compressive strength and permeability in concrete and also stated the permeability reduces with the addition of nano silica. Bolhassani (2015), have found that the overall performance of concrete with or without fly ash was significantly improved with the addition of variable dosage of nano silica. They concluded that nano silica led to significant consumption of portlandite (CH) in the pozzolanic reaction.

The majority of structures are being constructed as reinforced concrete structures. Corrosion of steel in concrete is source of deterioration of reinforced concrete structures, leading to premature structural failure. Many corrosion studies had conducted on variety of normal and high strength concretes. Saeid Kakooei (2012), investigated the corrosion of rebar embedded in the fibers reinforced concrete. Faizal (2015), has studied Effect of Clay as a Nanomaterial on Corrosion Potential of Steel Reinforcement Embedded in Ultra-High Performance Concrete. Now-a-days the nano-materials also used to inhibits the corrosion reactivity on steel embedded in concrete (Saraswathy and Ha-Won Song, 2007). Still the properties of nanomaterial in concrete have not been completely explored and there is a huge scope of research to study the effectiveness and chemical reactivity of nanomaterial with concrete. Hence, this paper aimed to compares the mechanical properties, ultra-sonic pulse velocity (UPV), durability by accelerated corrosion test and confirming it using corrosion analyzing instrument and corrosion resistivity meter. The change in pH of acidic and saline solution before and after corrosion was also observed in this experimental study.

2. EXPERIMENTAL PROGRAM

In this research program the cement is partially replaced with fly ash (FA) and ground granulated blast furnace slag and three different nanomaterials. GGBS was used because it shows good pozzolanic activity and mainly improves the microstructure of cement concrete. Moreover it attains mechanical properties much faster than Fly Ash (FA) and can be replaced for higher percentage of cement. The nanomaterials like nano silica, nano alumina and nano titanium dioxide are used in this research paper to compare the mechanical properties and chemical reactivity of these material in concrete. All these nanomaterials are used by replacing small percentage of cement from 2% to 6%. A total of 17 mixes was prepared for the tests. The first one was a control mix, in second mix the cement was replaced by 40 % GGBS and in rest of the mix it was replaced by 40% GGBS and 2% to 6% nano silica (SiO_2), nano alumina (Al_2O_3) and nano titanium oxide (TiO_2). For each mixes 6 cubes, 3 cylinders and 3 prisms was casted. 6 cylinders with steel rebar was casted for corrosion test for control mix, 40% GGBS and for optimum dosage of three nanomaterials.

Materials: Commercially available 53 grade Ordinary Portland Cement was used in this experimental research. The M60 grade high performance concrete was developed in this experiment. The mineral admixture GGBS was used as partial replacement of cement. It was found that 40% GGBS is giving better mechanical properties compared to other percentage of replacement. Moreover higher percentage of GGBS may lead to segregation. 12.5 mm coarse aggregate was used.

The locally available fine aggregate was used and confirms to Zone 2 as per IS Code 383. The superplasticizer used belongs to poly-carboxylate ether based family. The nano oxides used are nano silica (SiO_2), nano alumina (Al_2O_3) and nano titanium dioxide (TiO_2). The physical properties of these nano oxides are shown in Table 1 and chemical compositions of nano SiO_2, nano Al_2O_3 and nano TiO_2 are shown in Table 2, Table 3 and Table 4 respectively.

Table.1. Physical Properties of Nanomaterials

Properties	Nano SiO_2	Nano Al_2O_3	Nano TiO_2
Morphology	Porous	Spherical	Spherical
Color	White	White	White
Purity	99.5%	99.5%	99.5%
Particle Size	50-80 nm	30-50 nm	10-20nm
Specific Gravity	2.1	3.9	4.26

Table.2. Chemical Compositions of Nano SiO_2

SiO_2	Al	Fe	Mg	Ca
> 99.5%	< 0.02%	< 0.05%	< 0.1%	< 0.08%

Table.3. Chemical Compositions of Nano Al_2O_3

Al_2O_3	CaO	Fe_2O_3	MgO	SiO_2
>99.5%	<0.017%	<0.035%	<0.001%	<0.05%

Table.4. Chemical Composition of Nano TiO_2

TiO_2	S	Si	Mg	Al
>99.5%	<0.05%	<0.02%	<0.01%	<0.01%

Working Procedure: The material for preparing the concrete was mixed properly in a pan mixer. The coarse aggregate used for the mix was thoroughly sieved and was kept in saturated surface dry (SSD) condition. The nanomaterial used was in powder form. The superplasticizer and nanomaterial was thoroughly mixed in water by using a stirrer for 5 minutes. The nanomaterial was mixed along with superplasticizer to avoid the chances of agglomeration. The superplasticizer induces electrostatic dispersion and reduces the chances of agglomeration. First the coarse aggregate was fed into the pan mixer. Small quantity of water was poured and the aggregate was mixed until it was completely wet. Before pouring the water with superplasticizer and nanomaterial each time into the mix, it should be stirred properly. Next the binding material (cement and GGBS) was introduced into the mixer and again a little quantity of water was poured. The mixing was done until the binding material was coated properly on to the coarse aggregate. The binding material coated aggregate was wetted with water and thoroughly sieved dry fine aggregate was fed into the mixer machine. After 2 minutes of mixing the remaining water with superplasticizer and nanomaterial was stirred and poured into the pan mixer. The mixing was done until a homogeneous mixture of concrete was obtained.

Details of Specimens and Tests conducted:

Destructive Tests: The cubic specimens casted for the compressive test were of 100x100x100 mm. The cylinders were of 100mm diameter and 200mm height. The prism was of 500x100x100 mm. The cubes were tested for 7 days and 28 days compressive strength. The split tensile test and flexural test was conducted for 28 days.

Non-Destructive Tests: Apart from destructive tests, non-destructive tests were also performed. Ultra-Sonic Pulse velocity (UPV) was conducted to know the quality of concrete. The UPV was done on cubes. The equipment consists of an electronic circuit for generating pulses, a transmitter transducer and a receiver transducer. These pulses are transmitted through these transducers. The UPV displays the time taken for the pulse to travel from the two transducers and the velocity of the pulse between these transducers. If the equipment is showing higher velocity and less time it indicates that the concrete is of good quality and the constituent material of the concrete are packed densely.

The corrosion analysing instrument measures the corrosion potential in mille Volts (mV) with respect to a standard reference electrode which was copper sulphate solution in this experimental research. In concrete structures with reinforcement the concrete plays the role of an electrolyte and the reinforcement will show a potential depending upon the environmental condition of the concrete structure. The total potential range of the instrument ranges from +200 to -950 mV. The main method of the corrosion detection is the half-cell potential measurement. The probability of corrosion of steel reinforcement is given by ASTM C 876.

The corrosion resistivity meter measures the electrical resistivity of concrete subjected to corrosion. The concrete specimen with high resistivity will show slow and less corrosion whereas the specimen with low resistivity shows fast and high corrosion. AC and DC measurement techniques are used to determine the electrical resistivity of the concrete specimen. The DC measurement can be done by applying an electric field between the two electrodes and measuring the current as a voltage drop over small resistance. The AC measurement was done by two or four pin method. The mostly used surface probe is Wenner array. An AC current was passed between the outer electrode and the potential between the inner electrodes was measured. The corrosion resistivity in this experiment was determined by Wenner's four probe method.

Durability Tests: The durability test conducted in this research was accelerated corrosion test. The specimen used for these test are concrete cylinders of 100mm diameter and 200mm height. The cylinders consist of a 12mm diameter and 200mm height Fe415 steel rebar embedded in it. The initial weight of the steel rebar was taken before placing it in the concrete cylinder. The steel rebar was placed at the center of the cylinder. A cover of 50mm was provided at the bottom of the cylinder. The steel rebar projects 50mm out of the concrete cylinder. 6 cylinders were casted for 5 mixes as mentioned before. The cylinders were cured for 28 days. The cured cylinders were transferred to the solution of accelerated corrosion setup. 3 cylinders were tested in 5% NaCl solution and 3 cylinders were tested in 5% H_2SO_4 solution. The corrosion was carried out for 1 week. Figure 1 shows the acceleration corrosion setup in 5% NaCl and 5% H_2SO_4 solution.

Fig.1. Accelerated Corrosion of Cylinders

The corrosion was carried out using an AC-DC convertor of 24V and 4A. The positive terminal of the AC-DC convertor was connected to the steel rebar of the cylinder and the negative terminal was connected to the steel rod dipped in the solution as shown in Figure 1. The first three rows of buckets consists of 5% NaCl solution and the second row of buckets consists of 5% H_2SO_4 solution. The setup was kept undisturbed for 1 week. After 1 week the cylinders were taken out and the steel rebar was taken out of the cylinders by breaking it. The final weight of the rebar was taken after cleaning the bar properly with emery paper and distilled water. There will be a weight loss due to corrosion in the bar. From this weight loss the corrosion rate can be calculated by using the below given formula.

$$CR = \frac{3.45x10^6\ W}{DAT} \quad (1)$$

Where, CR - Corrosion Rate in mpy; W - Weight Loss in g; D – Density of Steel in g/cm2; A – Area of steel plate in cm^2; T – Duration of corrosion in hours

The pH of the saline and acidic solution before corrosion and after corrosion has been taken to notice the reaction of nanomaterial concrete with these solutions.

3. RESULTS AND DISCUSSIONS

The Table 5 reveals the test results for destructive tests such as compressive strength for 7 and 28 days, split tensile strength and flexural strength for 28 days and non-destructive test such as UPV which was conducted on 100x100x100mm cubes cured for 28 days. CM indicates control mix of M60 grade concrete and CMG stands for

40% replacement of cement with GGBS. Concrete with nano silica (CNS), concrete with nano alumina (CNA) and concrete with nano titanium oxide (CNT) for 2 to 6% replacement of cement and 40 % replacement of cement with GGBS are shown in Table 5. From the Table 5 it can be seen that the compressive strength of CMG for 7 days have increased by 23.46% than CM and it had been increased by 4.78% for 28 days. This shows that the initial rate of hydration is increased by the addition of GGBS. This is due to use of nanomaterial in concrete is because of its higher surface area to volume ratio. The nanomaterial densifies the cement matrix by increasing the C-S-H gel during the pozzolanic reaction of these materials with calcium hydroxide. Also the nano sized particle fills the voids in interfacial transition zone which is present between the cement and aggregate. The optimum dosage of nano SiO_2 and nano Al_2O_3 is at 3% i.e. CNS3 and CNA3 whereas for nano TiO_2 it is at 4% i.e. CNT4. Nano titanium dioxide plays the role of nano fillers and refines the pore structure of the concrete. Moreover it increases the formation of C-S-H gel leading to high strength of the concrete specimen. This is due to the smaller size of nano TiO_2 compared to nano SiO_2 and nano Al_2O_3 as given in Table 1. From Fig.2, Fig.3, Fig.4 and Fig.5 it can be seen that the compressive strength, split tensile strength and flexural strength is giving the same optimum dosages. From Table 5, Fig.2 and Fig.3 it can be noticed that the compressive strength of CNS3 for 28 days had increased 24.25% than CMG and 30.18% than CM whereas compressive strength of CNA3 for 28 days had increased 17.06% than CMG and 22.65% than CM and compressive strength of CNT4 for 28 days increased 23.16% than CMG and 29.05% than CM. Therefore CNS3 gives the maximum percentage of increase in compressive strength, split tensile strength and flexural strength when compared to CMG and CM than CNA3 and CNT4. The high mechanical property of nano SiO_2 is due to the increase in the quantity of C-S-H gel in the paste through pozzolanic reaction, increase in initial hydration rate, reduction of porosity and improvement in mechanical property of C-S-H gel itself. After CNS3 the maximum percentage increase of mechanical properties is given by CNT4 followed by CNA3. The UPV velocity criterion for concrete quality grading is based on IS: 13311-Part 1. From table 5 it can be seen that CNS3 have the maximum velocity. This is contributed to the dense packing of CNS3 and due to which the ultrasonic pulse can pass through the cube with high velocity. It can also be noticed that for all nanomaterial concrete the quality grading is good. Hence nanomaterial when mixed along with concrete results in less porous and impermeable concrete. The decrease in mechanical properties after the optimum dosage is due to the excess quantity of nanomaterial present in the concrete than the quantity which is required for combining with liberated lime produced during hydration and therefore the excess silica leaches out leading to the decrease in strength of concrete.

Table.5. Destructive and Non-destructive Tests Results

Specimen	Compressive Strength (MPa)		Tensile Strength (MPa)	Flexural Strength (MPa)	UPV (28 Days)	
	7 Days	28 Days	28 Days	28 Days	Velocity(km/s)	Quality
CM	38.15	61.76	3.20	8.20	3.7	Good
CMG	47.10	64.71	3.50	9.00	4.3	Good
CNS2	51.20	72.60	3.82	10.50	4.1	Good
CNS3	56.80	80.40	4.36	12.50	4.6	Excellent
CNS4	53.60	77.30	4.23	12.13	4.1	Good
CNS5	52.40	75.21	4.02	11.00	4.0	Good
CNS6	50.50	72.80	3.90	10.25	3.9	Good
CNA2	48.80	71.40	3.62	9.50	3.8	Good
CNA3	52.00	75.75	3.98	10.13	4.0	Good
CNA4	50.20	73.10	3.80	10.00	4.0	Good
CNA5	49.90	72.30	3.75	9.75	3.9	Good
CNA6	48.50	70.50	3.60	9.50	3.7	Good
CNT2	48.20	70.92	3.88	10.00	3.9	Good
CNT3	52.75	77.20	4.16	11.00	4.2	Good
CNT4	55.45	79.70	4.29	11.50	4.0	Good
CNT5	53.20	76.80	4.03	11.00	4.0	Good
CNT6	51.80	74.24	3.90	10.75	3.8	Good

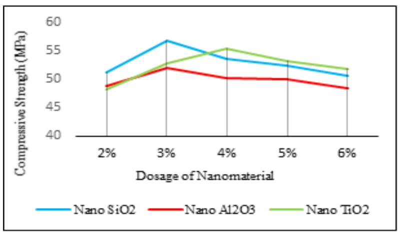

Fig.2. 7 Days Compressive Strength of Nanomaterial Concrete

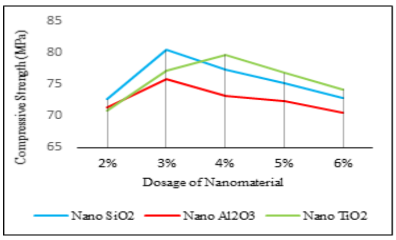

Fig.3. 28 Days Compressive Strength of Nanomaterial Concrete

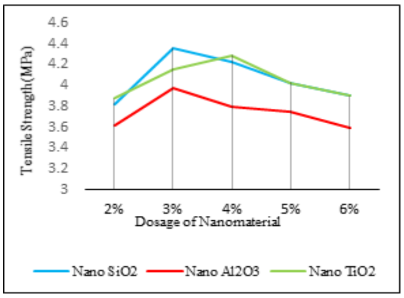

Fig.4. 28 Days Tensile Strength of Nanomaterial Concrete

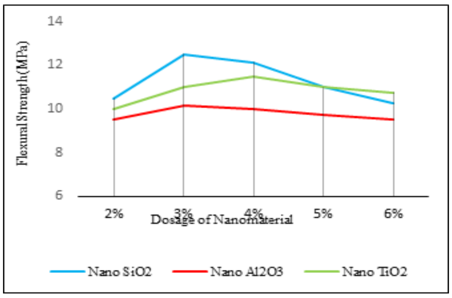

Fig.5. 28 Days Flexural Strength of Nanomaterial Concrete

Table 6 and Table 7 show the result for accelerated corrosion of cylinders. The corrosion rate was measured using weight loss method and using the formula (1). The table also shows the pH of the solution before and after the corrosion process. The accelerated corrosion was conducted for 5 mixes as shown in the table. The optimum dosage of three nanomaterial concrete was taken for corrosion test to determine the most durable concrete among the three. From Table 6 and Table 7 it can be seen that more corrosion takes place in saline condition i.e. 5% NaCl solution. CNT4 shows less corrosion rate compared to CNS3 in saline as given in Table 6 and same in acidic condition shown in Table 7. CNA3 has more corrosion rate compared to CNT4 and CNS3. The less corrosion rate of CNT4 in saline and acidic condition can be contributed to the dense packing of the concrete and reduction in porosity and increase in impermeability of concrete. Moreover the size of nano TiO_2 is small compared to nano SiO_2 and nano Al_2O_3 as shown in Table 1. The pH of water used for curing the concrete cylinders for corrosion was 7.9. From table 6 it can be seen that there is a drastic variation in pH before and after 7 days corrosion in 5% NaCl solution whereas in Table 7 there is a slight variation in pH before and after 7 days in 5% H_2SO_4 solution. Due to this high variation in pH of saline solution before and after the corrosion the corrosion rate of specimen in saline solution is greater than in acidic solution. From Table 6 and Table 7 it can be noticed that higher the difference between pH after and before corrosion in saline and acidic solution greater will be its corrosion rate. The corrosion rate depends upon the high solubility of oxygen and high conductivity of the solution used for corrosion.

Table.6. Accelerated Corrosion Test Results for 5% NaCl solution

Specimen	Weight Loss (gms)	Corrosion Rate (mpy)	pH of solution before corrosion	pH of solution after corrosion
CM	9.33	313.89	8.4	11.8
CMG	6.67	224.39	8.4	11.6
CNS3	3.33	112.03	8.4	11.5
CNA3	3.67	123.47	8.4	11.5
CNT4	2.33	78.39	8.4	11.4

Table.7. Accelerated Corrosion Test Results for 5% H_2SO_4 solution

Specimen	Weight Loss (gms)	Corrosion Rate (mpy)	pH of solution before corrosion	pH of solution after corrosion
CM	2.67	89.83	0.2	1.5
CMG	2.33	78.38	0.2	1.2
CNS3	1.33	44.74	0.2	0.6
CNA3	1.67	56.18	0.2	0.6
CNT4	1.33	44.74	0.2	0.5

The corrosion conditions for corrosion analysing instrument in Table 8 are given as per ASTM C 876. From Table 8 and Fig. 6 it can be observed that the corrosion potential of CNT4 is low compared to other mixes in 5% NaCl solution which indicates that the corrosion condition is intermediate and it is also low in 5% H_2SO_4 solution which indicates that the corrosion condition is low as per Table 4. After CNT4 the next low corrosion potential was for CNS3 followed by CNA3. From Table 8 it was noticed that for higher corrosion potential values the corrosion was high and for lower corrosion potential values the corrosion is low.

The corrosion conditions for corrosion resistivity meter in Table 8 are given as per Song. From Table 8 and Fig. 7 it can be observed that the electrical resistivity of CNT4 is high compared to other mixes in 5% NaCl solution which indicates that the corrosion condition is negligible and it is also high in 5% H_2SO_4 solution which indicates that the corrosion condition is negligible. After CNT4 the next high electrical resistivity was for CNS3 followed by CNA3. From Table 8 it was noticed that for higher electrical resistivity the corrosion was low and for lower electrical resistivity the corrosion was high.

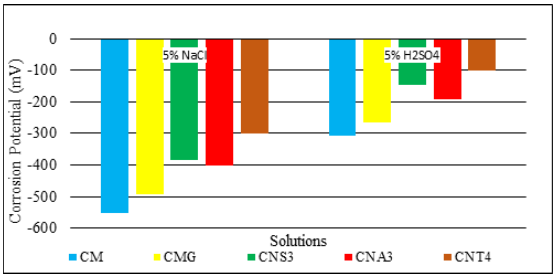

Fig.6. Graph for Corrosion Potential in 5% NaCl and 5% H_2SO_4 solutions

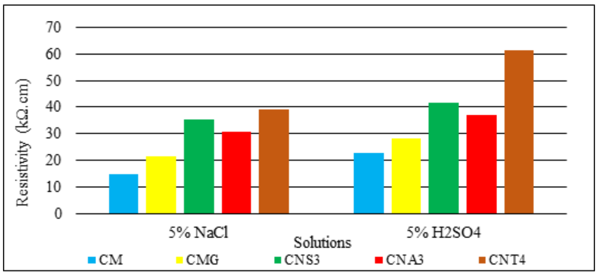

Fig.7. Graph for Electrical Resistivity in 5% NaCl and 5% H_2SO_4 solutions

Table.8. Non-Destructive Corrosion Test Results

Solutions	Mixes	Corrosion Analysing Instrument		Corrosion Resistivity Meter	
		Potential (mV)	Corrosion Condition	Resistivity (kΩ.cm)	Corrosion Condition
5% NaCl	CM	-553.50	Severe	15.00	Low
	CMG	-492.66	High	21.50	Negligible
	CNS3	-385.25	High	35.50	Negligible
	CNA3	-401.75	High	30.65	Negligible
	CNT4	-301.50	Intermediate	39.00	Negligible
5% H_2SO_4	CM	-308.00	Intermediate	23.00	Negligible
	CMG	-266.88	Intermediate	28.25	Negligible
	CNS3	-147.50	Low	41.75	Negligible
	CNA3	-192.75	Low	37.00	Negligible
	CNT4	-100.00	Low	61.25	Negligible

Therefore from accelerated corrosion test, corrosion potential test and electrical resistivity tests it was observed that CNT4 shows better durability than other mixes. In Fig 8, pores can be seen in concrete which results in low strength and less durable concrete as shown in Table 5, Table 6 and Table 7. From Fig 9 it can be clearly seen that the nanomaterial fills the pores present in between cement and aggregate and makes it tightly packed making it less porous which results in better mechanical properties and better durability. From Table 9 it can be seen that the oxygen and silicon content for CNS3 is high compared to CNA3 and CNT4. According to Birgisson and Saloma silica formed will react with the liberated lime formed due to the hydration of concrete to form C-S-H gel making it to gain more strength and mechanical properties. This is the reason for the high mechanical properties of CNS3 compared to CNT4 and CNA3. The silicon content for CNT4 was also high compared to CNS3 and CNA3. CNT4 consists of iron content which combines with oxygen to form iron oxide which imparts strength to concrete. The aluminium element combines with oxygen to form alumina which helps in quick setting of concrete as per Shetty. CNT4 is having aluminium content more than CNS3 and CNA3. Therefore setting of CNT4 is fast compared to CNS3 and CNA3.

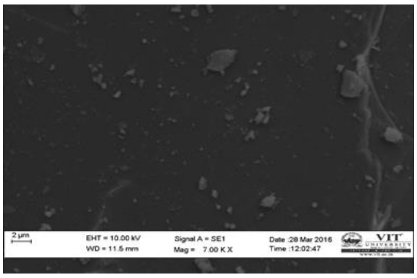

Fig.8. SEM image of CNT4

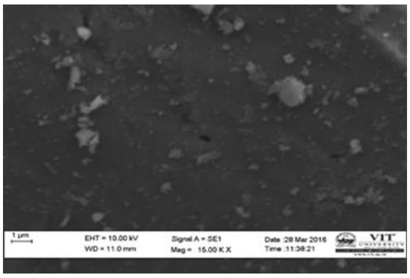

Fig.9. SEM image of CMG

4. CONCLUSION

From all the above results it can be concluded that CNS3 is having better mechanical properties followed by CNT4 and CNA3 and CNT4 is more durable compared to CNS3 and CNA3. When 3% of cement is replaced with nano SiO_2 there is a significant change in the early interfacial transition zone structure. Moreover there is an increase in heat of hydration and decrease of CH content due to the presence of nano SiO_2. Nano TiO_2 can also increase the early age of hydration of cement. Due to the smaller size of nano TiO_2 compared to nano SiO_2 and nano Al_2O_3 it fills the pores present in between cement and aggregate making it more durable to saline and acidic conditions. Moreover CNT4 consists of iron oxide which contributes to the strength of concrete and alumina which helps in quick setting of concrete. Therefore nano SiO_2 and nano TiO_2 can be recommended for concrete structures exposed to sea water and structures susceptible to chemical attack like those near chemical factories.

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