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A Study on Physico-Mechanical and Electro-Chemical Behavior of Nano material in Ordinary Portland Cement

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Abstract: Concrete is the most used construction material on Planet Earth and presents a higher permeability that allows water and other aggressive elements to enter, leading to carbonation and chloride ion attack, resulting in corrosion problems. Therefore, the nanoscale study of the hydration products, as a form to overcome durability issues, is a crucial step in concrete sustainability.In this work, nanomaterials such as Nano Silica (NS) and Carbon Nanotubes (CNTs) has been incorporated along with cement and the effect of physical, mechanical and corrosion resistant properties were evaluated using different electrochemical tests. In this project, nanomaterialslike Multi Wall Carbon Nanotubes (MWCNTs) and Nano-silica have been used as a trial mix. The physico-mechanical and Electro-Chemical behavior of these nanomaterials in ordinary Portland cement (OPC) was studied.The blended cement used in this investigation consists of ordinary Portland cement, carbon nanotubes and synthesised nanosilica.

Keywords: Anodic Polarization Technique, Carbon Nanotubes, Compressive Strength, Fine Aggregate, Flexural Strength Test, Impressed Voltage Test, Nano Silica, Open Circuit Potential, Ordinary PortlandCement, Sorptivity Test, Split Tensile Strength

I. INTRODUCTION

The field of nanotechnology is rapidly maturing into a fertile and interdisciplinary research area from which new multifunctional and smart materials can be developed. Since carbon nanotubes (CNTs) were discovered and they have been widely used for a variety of applications due to their excellent physical properties: high strength and young's modulus (the tensile strength and young's modulus of CNTs are 20 times and 10 times that of CFs respectively), high bonding force with matrix (the interlaminar shear strength of CNTs reinforced epoxy materials is 10 times that of CFs reinforced epoxy materials), large deformation and high ductility (the elongation at break of CNTs is 18% and is 18 times that of CFs), high aspect ratio (>500), and excellent electrical conductivity. In particular,the extremely high aspect ratio and low density of CNTs makes them easy to form a conductive and mechanical reinforcement networks with a CNT doping level as low as 0.05% CNTs also have interesting piezoresistive properties. In recent years,more and more researchers have started investigating on utilizing CNTs in multifunctional and smart cement-based materials.

In addition to that, the use of nanosilica particles increases the compression strength of cement pastes. The use of nanosilica on sludge/fly ash mortars, compensate the negative effects associated to sludge incorporation in terms of setting time and initial strength. Nanosilica addition led to an increase of strength by 15–20%. The result shows that nanoparticles improved the reduction of water absorption and led to an increase of abrasion and impact strength. The use of nanoparticles (nanotubes, nanofibers, nanosilica or nanoclay) is responsible for a higher hydration degree of cementitious compounds as long as higher nanoparticle dispersion can be achieved. Nanoparticles have increase in strength by 2 times and 40 times for electric conductivity, which means a high potential for sensing ability. 1% of carbon nanofibers (by binder mass) can compensate the strength reduction associated with the replacement of 20% fly ash. The effect of carbon nanofibers on cement pastes (0.08% by binder mass) observing an increase in strength.

In this work OPC was substituted by 1 wt. % of cement by nano silica (Sio_2) and the carbon nanotube was added by ratios of 0.01, 0.03, 0.05 and 0.07 wt. % of cement as a trial referred by past journals. Sand ratio used in this investigation was 1:3 wt. %. The blended cement mortar was prepared using water/binder ratio of 0.5 wt. % of cement. Compressive strength, split tensile strength and flexural strength of cement mortar were investigated.

II. MATERIALS AND ITS PROPERTIES

Material used

- Ordinary Portland Cement : Grade 43
- Fine Aggregate : Natural sand of Size <0.5mm
- Multi Walled Carbon Nanotubes : 3nm to 8nm

- Nano-Silica: Sizes ranging from 20nm to 50nm.

2.1 Ordinary Portland Cement

The OPC was classified into three grades, namely 33 grade, 43 grade and 53 grade depending upon the strength of the cement at 28 days when tested as per IS 4031-1988. OPC 43 grade cement was used for this project and the physical and chemical properties of 43 grades OPC was given in table 1 & 2.

Table.1 Physical Properties of OPC

Type of Cement 43 Grade(IS:8112-1989)		
Minimum Compressive Strength (N/mm^2)		
3 Day		23
7 Day		33
28 Day		43
Fineness		
Minimum specific surface		225
Setting times (minutes)		
Initial, minimum		30
Final, maximum		600
Soundness, expansion maximum		10.0

Table.2 Chemical Properties of OPC

Type of Cement		43 Grade(IS:8112-1989)
Loss on ignition,percent maximum		5
Insoluble residue,percent maximum		2
Magnesia MgO,percent maximum		6
$C_3A>5$ percent		2.5
$C_3A<5$ percent		3
Lime saturation factor (LSF)		0.66 to 1.02

2.2 Fine Aggregate

The sand which was locally available and passing through 4.75mm IS sieve is used. The specific gravity of fine aggregate was 2.60. Locally available river sand conforming to Grading zone I of IS: 383 – 1970.Sand passing through IS 4.75mm Sieve will be used for casting all the specimens.

2.3 Multi walled Carbon Nano Tubes

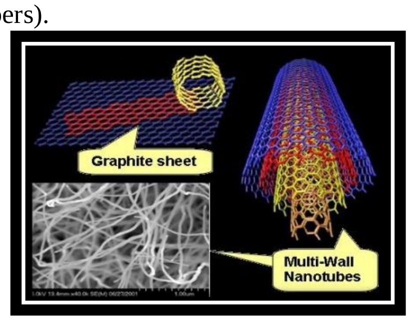
CNTs are extended tubes of rolled graphene sheets. Here, Multi-walled nanotubes (MWNTs) consist of multiple rolled layers (concentric tubes) of graphite were used for this project as shown in Fig.1 &2.

2.3.1 Physical Properties of Multi walled CNTs

- Strength Carbon nanotubes are the strongest and stiffest materials yet discovered in terms of tensile strength and elastic modulus respectively.
- Carbon nanotubes have a low density for a solid of 1.3 to 1.4 g·cm−3, its specific strength of up to 48,000 kN·m·kg−1 is the best of known materials, compared to high-carbon steel's 154 kN·m·kg−1.

2.3.2 Mechanical Properties of Multi walled CNTs

The strength of the sp^2 carbon-carbon bonds endues CNTs amazing mechanical properties (e.g. ultra–high strength and stiffness, and elastic stress-strain behavior). The young’s modulus of the best CNTs can be as high as 1000GPa which is approximately 5 times higher than steel. The tensile strength or breaking strain of CNTs can be up to 63GPa or 10%, around 50 times higher than steel. In addition to their high strength and elastic constant, CNTs have extremely high aspect ratios,with values typically higher than 1000:1 and reaching as high as 2,500,000:1. As a result of these properties coupled with the lightness, large surface area (typically 200-300m^2/g), and excellent chemical and thermal stability, CNTs reinforcements are expected to produce significantly stronger and tougher cement-based materials than traditional reinforcing materials (e.g. glass fibers or carbon fibers).



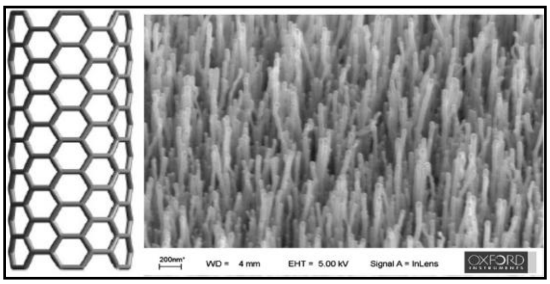


Fig.1 Carbon Nano Tubes Fig.2 Carbon Nanotubes
Right Schematics; Left Microstructure

2.4 Nano-Silica

A cement paste is composed of small grains of hydrated calcium silicate gels, nano-sized individual pores, capillary pores, and large crystals of hydrated products. Thus, there should be room for nano-phase materials to fill the pores of the cement paste. The amorphous or glassy nano-scale silica, which is the major component of a pozzolanos, reacts with calcium hydroxides formed from the hydration of calcium silicates. The rate of the pozzolanic reaction is proportional to the value of Blaine fineness. Therefore, the nano-SiO_2 used was of particle form with 99.9% SiO_2as shown in table.3, a particle size of 40 nm, and 60 m^2/g Blaine fineness. Fig.3, 4&5 shows the Flow diagram and picture of the Rice Husk Ash and Nano Silica.

Table.3 Properties of Nano-SiO_2

Properties	Content
Content of SiO_2-x (%)	99.9
Phase	Non-crystal white powder
Compaction density (g/cm^2)	0.14
Average particle size (nm)	10
Specific surface area (m^2/g)	670
Impurity (%)	
Cl	0.028
Cu	0.003
Al	0.002
Ca	0.002
Fe	0.001
Mg	0.001

Fig.3 Rice Husk Ash

Fig.4 NanoSilica

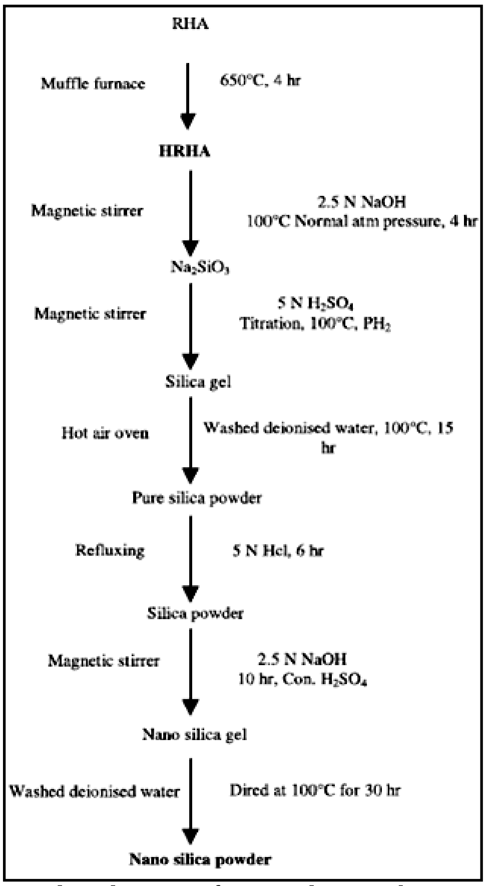


Fig.5 Flow diagram of Nano silica synthesis

III. EXPERIMENTAL INVESTIGATION

3.1 MECHANICAL PROPERTIES

3.1.1 Compressive Strength Test

The compressive strength of concrete is one of the most important and useful properties of concrete. In most structural applications concretes are employed primarily to resist compressive stresses. The compressive strength is frequently used as a measure of these properties. Mortar cube specimens of 50mmx50mmx50mm were cast using OPC and with the various ratios of CNT 0.01%, 0.03%, 0.05% and 0.07%by the weight of cement. Mortar specimens were prepared using mix ratio 1:3 having water cement ratio as 0.50. Quantity of cement and sand for one cube 75g and 225g respectively. The specimens were mechanically vibrated. After 24hrs of setting the specimens were demoulded and they cured for 28 days in normal water. After the curing period was over the specimens were tested for compression strength. The measurements were taken by UTM compression concrete testing machine as shown in Fig.6.

Fig.6 Compressive Testing Machine

Fig.7 Split Tensile Testing Image

3.1.2 Split Tensile Strength Test

Split tensile Strength was carried as per ASTM C496-90. Mortar cylinder of size 75mm diameter and 150mm height were casted with different systems. After 24 hrs specimens were demoulded and cured. During casting it was compacted by table vibrated. For each system three specimens were cast. After curing the cylinders are tested in the universal testing machine of 1000 KN capacity. The test setup is shown in Fig.7.

3.1.3 Flexural Strength Test

Flexural strength is the one of the measure of tensile strength of concrete. It is the ability of a beam to resist failure in bending. It is measured by loading un-reinforced beam or prism of size 100 mm x 100 mm x 500 mm. The specimens were tested for its flexural strength as per IS: 516-1959 using a calibrated flexural testing machine as shown in Fig.8.

Fig.8 Split Tensile Testing Image

3.2 ELECTRO-CHEMICAL PROPERTIES

3.2.1 Open Circuit Potential (OCP) Measurements

The OCP values for the different systems were periodically monitored using a voltmeter with a high input impedance of 10 mega ohms. A saturated calomel electrode (SCE) was used as the reference electrode. The positive terminal of the voltmeter was connected to the working electrode i.e., CTD rods. The common terminal was connected to the reference electrode as shown in Fig.9. The corresponding potentials were recorded. The OCPs for all of the specimens were monitored over an exposure period of 120 days. In this study, triplicate specimens were used for each system and the average of these values were reported and interpreted based on ASTM C -876-1994.

Fig.9 OCP Test Setup

3.2.2 Impressed Voltage Test

Cylindrical concrete specimens of size 20 mm diameter and 25 mm height were cast using 1:3 mix mortar with NS and CNTs at various percentages containing W/C = 0.50, with centrally embedded rebar of 12 mm diameter and 20 mm height, containing ordinary Portland cement (control) and nanosilica. After 24 h from casting, the cylindrical specimens were demoulded and subjected to water curing for 28 days. After curing, the specimens were subjected to impressed voltage test as shown in Fig.10 &11. In this technique, the specimens were immersed in 5% NaCl solution and embedded steel in concrete is made anode with respect to an external stainless steel electrode serving as cathode by applying a constant positive potential of 1V to the system from a DC source. The variation of current is recorded with time. For each specimen, the time taken for initial crack and the corresponding maximum anodic current flow was recorded. Triplicate specimens were used for this technique.

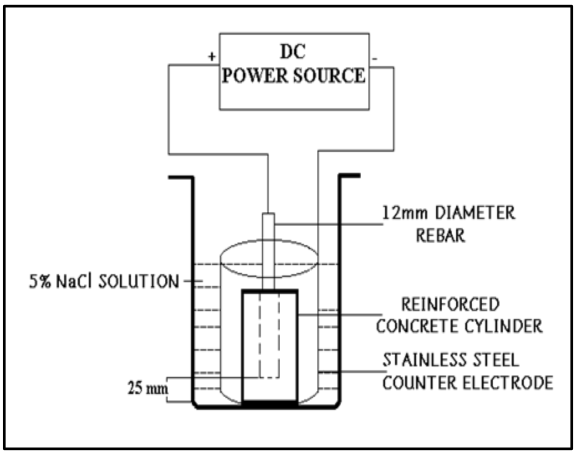


Fig.10 Impressed Voltage Test Setup Fig.11 Schematic Representation of Impressed Voltage Test

3.2.3 Anodic Polarization Technique (Potentiostatic Method)

Mild steel rods were embedded in cylindrical mortar (1:3) specimens of size 20 mm diameters and 25 mm height using w/c ratio of 0.52. The mortar specimens were subjected to anodic polarization studies using a three electrode system which consists of embedded steel in mortar as anode, stainless steel ring electrode as cathode and saturated calomel electrode as reference electrode. Anodic polarization studies have been carried out in 3% NaCl solution. The current flowing at +260 mV was recorded for all the specimens as shown in Fig.12.

Fig.12Anodic Polarization Test Setup

3.2.4 Sorptivity Test

Sorptivity is a measure of the capillary force exerted by the pore structures causing fluids to be drawn into the body of material. Determination of the Sorptivity of a concrete specimen in the laboratory is a simple technique which requires a scale, stop watch and a shallow pan of water. The initial mass of the sample was taken at time zero, the specimen was kept partially immersed to a depth of 5mm in the water as shown in Fig.13. At selected times; the specimens were removed from the water, then note the time and weighed the specimen. Again replace the specimen in the water and note the time. The gain in mass per unit over the density of water is plotted versus the square root of the elapsed time as shown. The slope line of best fit of these points is reported as the Sorptivity.

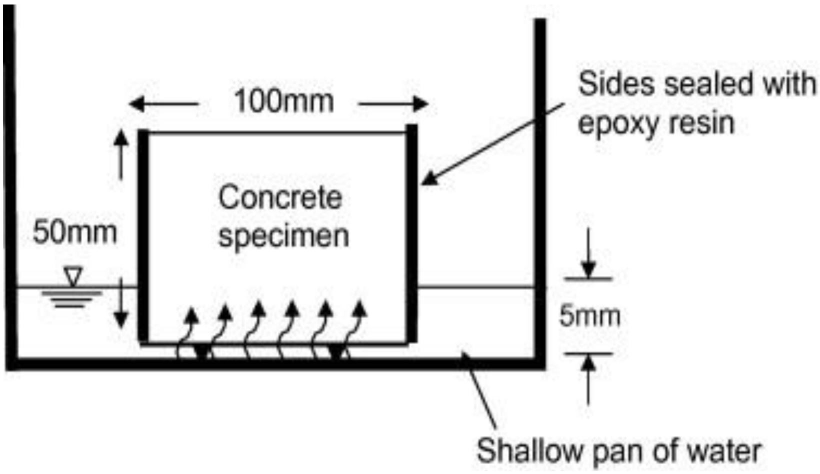


Fig.13Schematic Representation of Sorptivity test Setup

IV. RESULTS AND DISCUSSION

4.1 MECHANICAL PROPERTIES TEST RESULTS

4.1.1 Compressive Strength Test

Table.4 Compressive Strength of Mortars

Sp No.	Mix Type	Load (kN)	Compressive Strength (N/mm^2)
	OPC (Control)	42.7	17
1	OPC+1%NS	54	21.6
2	OPC+1%NS+0.01%CNT	117	47
3	OPC+1%NS+0.03%CNT	**135**	**54**
4	OPC+1%NS+0.05%CNT	103	41
5	OPC+1%NS+0.07%CNT	101	40

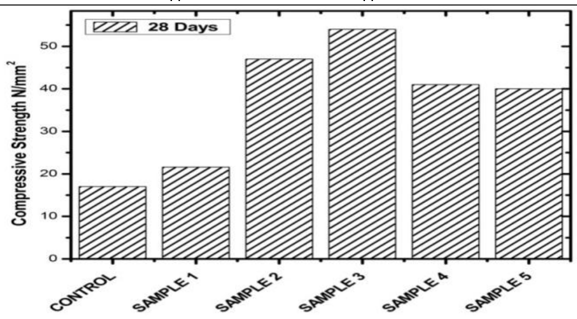


Fig.14Compressive Strength of Mortars

Table.4 & Fig.14 shows the compressive strength results obtained for different systems after 28 days of curing. From the results it is observed that OPC with 1% NS and 0.03% CNT has higher compressive strength when compared to the other systems. In general all the composite systems have higher compressive strength than the OPC. The composite systems have shown 27 to 217% increase in compressive strength, which indicates that composite system performs like ultra-high strength concrete.

4.1.2Split Tensile Strength Test

Table.5 Split Tensile Strength ofMortars

Sp No.	MixType	Load (kN)	Tensile Strength (N/mm^2)
	OPC	10.6	2.04
1	OPC+1%NS	13.3	2.55
2	OPC+1%NS+0.01%CNT	13.7	2.54
3	OPC+1%NS+0.03%CNT	**18.8**	**3.62**
4	OPC+1%NS+0.05%CNT	17.8	3.43
5	OPC+1%NS+0.07%CNT	16.33	3.15

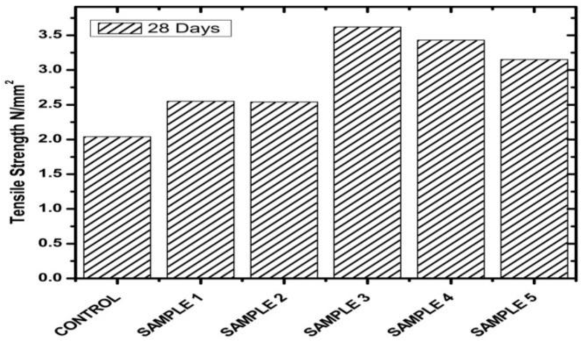


Fig.15Split Tensile Strength of Mortars

Table.5 & Fig.15 shows the split tensile strength results obtained for different systems after 28 days of curing. From the results it is observed that OPC with 1% NS and 0.03% CNT system has 77% higher split tensile strength when compared to the other systems. In general all the composite systems has higher split tensile strength, which is due to the formation of denser Calcium Silicate Hydrate (CSH) gel formation and also reduced $Ca(OH)_2$ content within the hydration products.

4.1.3 Flexural Strength Test

Table.6 & Fig.16shows the flexural strength results obtained for different systems after 28 days of curing. From the results it is observed that OPC with 1% NS and 0.03% CNT system has 233% higher flexural strength when compared to the other systems. Increase in flexural strength may be due to the composite action of NS and CNT. Addition of NS acts as a filler effect and enhanced the formation of CSH. Addition of CNT acted as bridging effect and hence increased the flexural strength.

Table.6 Flexural Strength Test of Mortors

Sp No.	MixType	Load (kN)	Flexural Strength (N/mm^2)
1	OPC	0.3	2.7
2	OPC+1%NS	0.4	3.6
3	OPC+1%NS+0.01%CNT	0.8	7.2
4	OPC+1%NS+0.03%CNT	**1.0**	**9.0**
5	OPC+1%NS+0.05%CNT	0.9	8.1
6	OPC+1%NS+0.07%CNT	0.4	3.6

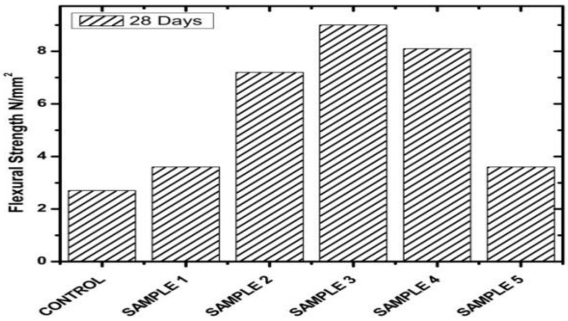


Fig.16Flexural Strength of Mortars

4.1.4 Water Absorption Test

Table.7 Water Absorption Test

Sp No.	Mix Type	Voids in %
1	CONTROL MIX	9.59
2	1% NANO SILICA	9.11
3	1% NS + 0.01% CNT	7.34
4	1% NS + 0.03% CNT	**5.83**
5	1% NS + 0.05% CNT	6.17
6	1% NS + 0.07% CNT	7.12

From the table.7 it is found that, all the systems containing NS and CNTs composites have shown lesser voids, when compared to the control mix. It was found that up to 0.03% CNT addition, the percentage of voids was found to increases, but beyond 0.03 % there is an increase in percentage of voids has been observed. From the above studies it has been concluded that 1%NS and 0.03% CNT was found to be the optimum percentage of addition in reinforced concrete for increasing the durability in terms of corrosion resistance. These effects are responsible for the improvement of mechanical properties of mortars with nano-particles.

4.2 ELECTRO-CHEMICAL PROPERTIES TEST RESULTS

4.2.1 Sorptivity Test

Table.8 Sorptivity Coefficient for CNT Specimens

Mix Type	$K_a = [Q/A]^2 \times (1/t)$
CONTROL MIX	3.0826×10^{-4}
1% NANO SILICA	1.1872×10^{-4}
1% NS + 0.01% CNT	6.4896×10^{-5}
1% NS + 0.03% CNT	$\mathbf{4.5067 \times 10^{-5}}$
1% NS + 0.05% CNT	6.2424×10^{-5}
1% NS + 0.07% CNT	6.3242×10^{-5}

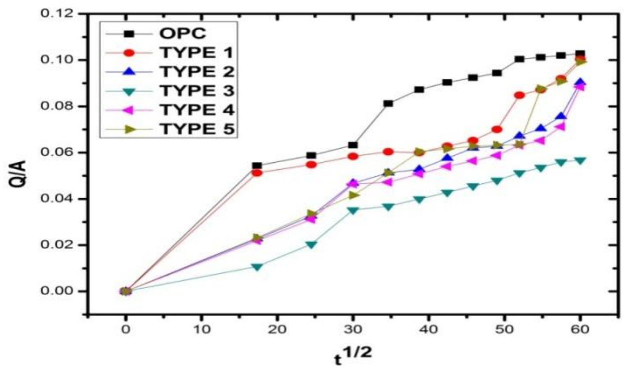


Fig.17Sorptivity for CNT Specimens

Table.8 & Fig.17shows the sorptivity of water cured specimens after 28days of curing. From the results it is observed that 0.03% CNT samples have lesser sorptivity values when compared to other specimens. This is due to the complete hydration reaction of particles while immersed in water.

4.2.2 Impressed Voltage Test

Table.9 Impressed Voltage Test

TIME (Hrs)	CONTROL	1%NS	1%NS + 0.01%CNT	1%NS + 0.03%CNT	1%NS + 0.05%CNT	1%NS + 0.07%CNT
0	2.6	0.13	0.68	**0.19**	0.71	0.7
12	3.1	0.4	0.9	**0.2**	0.7	0.9
24	3.5	0.35	1.2	**0.25**	1	1
36	3	0.35	1	**0.2**	0.9	0.85
48	2.5	0.35	1	**0.2**	0.85	0.95
60	2.8	0.3	0.8	**0.25**	0.55	0.65
72	2.7	0.25	0.45	**0.15**	0.55	0.55
84	2.9	0.35	0.7	**0.2**	0.65	0.65
96	3	0.4	0.65	**0.2**	0.85	0.5
108	2.65	0.4	0.6	**0.15**	0.8	0.45
120	3.8	0.7	0.8	**0.55**	1.1	0.65
132	3.95	0.65	0.75	**0.4**	1.1	0.65
144	3.8	0.95	0.45	**0.55**	1.2	0.3
156	4.2	0.9	0.75	**0.6**	1.2	0.65
168	4.8	0.3	0.45	**1.5**	1.2	0.3
180	3	0.9	0.75	**1.15**	1.2	0.65

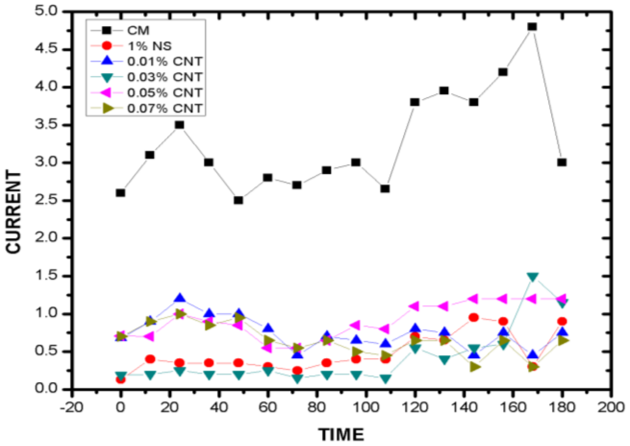


Fig.18Graph for impressed voltage

Table.9 & Fig.18shows the impressed voltage test results obtained for different systems after 28 days of curing. From the results it is observed that OPC with 1% NS and 0.03% CNT system has less current value when compared to the other systems. In general all the composite systems containing NS and CNT have shown lesser current value of 1.5mA after 180hours of exposure. But the control system has shown a maximum current value of 4.8mA Addition of NS and CNT filled the pores and reduced the permeability and hence increased the corrosion resistance.

4.2.3 Open Circuit Potential Test

Table.10 and Fig.19 shows the Open circuit potential measurement taken for different systems. From the figure it is observed that, the mix containing 0.03% CNT material have shown less potential of below -300mV (Passive state) than the Control mix which have potential up to -450mV (Active state).

Table.10 Open Circuit Potential Test

No. of Days	OPC	OPC + 1%NS	OPC+1%NS +0.01%CNT	OPC+1%NS +0.03%CNT	OPC+1%NS +0.05%CNT	OPC+1%NS +0.07%CNT
0	-0.48	-0.465	-0.45	**-0.301**	-0.495	-0.505
3	-0.49	-0.475	-0.46	**-0.325**	-0.5	-0.51
6	-0.501	-0.481	-0.471	**-0.38**	-0.51	-0.515
9	-0.51	-0.5	-0.49	**-0.426**	-0.515	-0.52
12	-0.525	-0.51	-0.5	**-0.465**	-0.535	-0.54
15	-0.535	-0.52	-0.511	**-0.498**	-0.555	-0.56
18	-0.54	-0.535	-0.52	**-0.512**	-0.56	-0.565
21	-0.57	-0.54	-0.525	**-0.516**	-0.59	-0.6
24	-0.595	-0.56	-0.545	**-0.52**	-0.6	-0.61
27	-0.641	-0.631	-0.601	**-0.575**	-0.65	-0.661

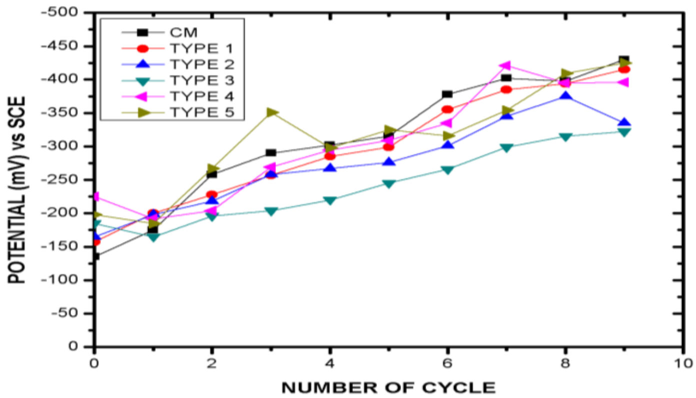


Fig.19 Open Circuit Potential Measurement

4.2.4Anodic Polarisation

Table.11 Anodic Polarisation

TIME (Hrs)		OPC	OPC + 1% NS	OPC + 1% NS + 0.01% CNT	OPC + 1% NS + 0.03% CNT	OPC + 1% NS + 0.05% CNT	OPC + 1% NS + 0.07% CNT
0	CURRENT/ mA	0.33	0.49	0.091	**0.002**	0.389	0.014
12		0.453	0.64	0.35	**0.003**	0.284	0.056
24		0.568	0.68	0.658	**0.002**	0.012	0.098
36		0.698	0.7	0.683	**0.003**	0.036	0.086
48		0.896	0.75	0.669	**0.005**	0.225	0.128
60		0.96	0.79	0.653	**0.003**	0.22	0.542
72		1.06	0.83	0.592	**0.1**	0.219	0.748

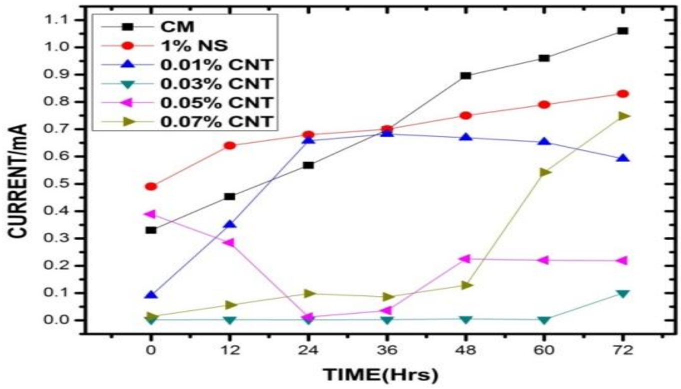


Fig.20Anodic Polarisation

Table.11 & Fig.20shows the current Vs. Time behavior of different composite systems subjected to impressed voltage test under anodic polarization. From the results it is observed that all the composite systems have shown lesser current value when compared to the control. In the composite systems, 0.03% CNT was found to have the lowest corrosion current of 0.1mA and the control was found have the corrosion current of 1.06 mA which is 10 times higher than the composite system. From the results it was concluded that systems which is having corrosion current lesser than 1.06 mA is said to be the best system. Interestingly, from this investigation all the composite systems have lesser corrosion current than control system which is due to the filler of the presence of nano particles which reduced the pores and enhanced the CSH gel formation which is very well evidenced from the other results.

V. CONCLUSION

Based on the available data, the beneficial action of the Nano-particles on the microstructure and performance of cement based materials can be explained by the following factors:

- Nano-particles fill the voids between cement grains, resulting in the immobilization of "free" water thereby causing "filler effect".
- Well-dispersed nano-particles act as centers of crystallization of cement hydrates therefore accelerated the hydration and hence increased the compressive, tensile and flexural strength.
- Nano-particles favored the formation of small-sized crystals such as $Ca(OH)_2$ and small-sized uniform clusters of C-S-H.
- Nano-SiO2 participates in the pozzolanic reactions, resulting in the consumption of $Ca(OH)_2$ and formation of an "additional C-S-H".
- Nano-particles improve the structure of the aggregates contact zone, resulting in a better bond between aggregates and cement paste.

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