Analyzing the Microstructural Properties of Nanomaterial in OPC by SEM, TEM, XRD and Corrosion Rate by TAFEL Techniques

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Abstract - *Use of Nano materials increases the compression strength of cement pastes. Some authors states that the phenomenon is not due to the pozzolanic reaction, because calcium hydroxide consumption was very low but, instead, to the increased of silica compounds that contributes to a denser microstructure. In this paper, nanomaterial's such as Nano Silica (NS) and Carbon Nanotubes (CNTs) have been used along with cement and they are analysed for their microstructural properties and Corrosion Resistance properties. The microstructural properties were evaluated using SEM, TEM, XRD technique and Corrosion rate by TAFEL techniques. Nevertheless, the fact that carbon nanotubes are not cost-efficient prevents the increase in commercial applications in a near future. In this work, Ordinary Portland Cement (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 and the research is carried out.*

Key Words: Carbon Nanotubes, Nano Silica, Ordinary Portland cement, Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM), TAFEL Technique, X-Ray Diffraction (XRD).

1. INTRODUCTION

1.1 Nano Materials

First, nanomaterial's have a relatively larger surface area when compared to the same mass of material produced in a larger form. This can make materials more chemically reactive and affect their strength or electrical properties. Second, quantum effects can begin to dominate the behavior of matter at the Nano scale particularly at the lower end - affecting the optical, electrical and magnetic behavior of materials. Materials can be produced that are Nano scale in one dimension (for example, very thin surface coatings), in two dimensions (for example, nanowires and nanotubes) or in all three dimensions (for example, nanoparticles).

Materials at length scale below 100 nm i.e. particles in nanometre (10^{-9}m) scale and quite often they make a comparison with a human hair, which is about 80,000 nm wide. Nanotubes are members of the fullerene structural family, which also includes the spherical buck balls. The ends of a nanotube might be capped with a hemisphere of the buck ball structure. Their name is derived from their size, since the diameter of a nanotube is on the order of a few nanometers (approximately 1/50,000th of the width of a human hair).

1.2 Nano Silica

At present Silica Fume and Nano Silica, because of their price, are only used in the so-called high performance concretes (HPC), eco-concretes and self-compacting concretes (SSC). For the last types of special concretes (eco-concrete and SCC), the application of these materials is a necessity. Also, some explorative applications of nanosilica in high performance well cementing slurries, specialized mortars for rock-matching grouting, and gypsum particle board can be found, but nanosilica is not used in practice yet. The application of these concretes can be anywhere, both in infrastructure and in buildings.

Nano-silica is applied in HPC and SCC concrete mainly as an anti-bleeding agent. It is also added to increase the cohesiveness of concrete and to reduce the segregation tendency. Some researchers found that the addition of colloidal nanosilica (range 0 to 2% by weight of cement) causes a slight reduction in the strength development of concretes with ground limestone, but does not affect the compressive strength of mixtures with fly ash or ground fly ash (GFA). Similarly, use of colloidal nanosilica (2% bwoc) to produce HPC concrete with compressive strength of 85 MPa, anti-bleeding properties, high workability and short demolding times (10 h).

Another application of nanosilica well documented and referred in several technical publications, is the use as additive in eco-concrete mixtures and tiles. Eco-concretes are mixtures where cement is replaced by waste materials mainly sludge ash, incinerated sludge ash, fly ash or others supplementary waste materials. One of the problems of these mixtures is their low compressive strength and long setting period. This disadvantage is solved by adding nanosilica to eco-concrete mixes to obtain an accelerated setting and higher compressive strength. Particulate nanosilica in oil well Cementing slurries in two specific ranges of particles sizes, one between 5 to 50 nm, and a second between 5 and 30 nm. Also they used nanosilica dry powders in encapsulated form and concentrations of 5 to 15% bwoc. The respective test results for the slurries demonstrate that the inclusion of nanosilica reduces the setting time and increases the strength (compressive, tensile, Young's modulus and

Poisson's ratio) of the resulting cement in relation with other silica components (amorphous 2.5 to 50 µm, crystalline 5 to 10 µm and colloidal suspension 20 nm, types silica) that were tested.

1.3 Carbon Nanotubes

Carbon nanotubes (CNTs) are hollow tubular channels, formed either by single wall carbon nanotubes (SWCNTs) or multi walled carbon nanotubes (MWCNTs) of rolled graphene sheets. They have received an increasing scientific and industrial interest due to their physical and chemical properties that is suitable for different potential applications ranging from living matter structure to nanometre-sized computer circuits and composites. Since CNTs exhibit great mechanical properties along with extremely high aspect ratios (length-to-diameter ratio) ranging from 30 to more than many thousands, they are expected to produce significantly stronger and tougher cement composites than traditional reinforcing materials (e.g. glass fibers or carbon fibers). In fact, because of their size (ranging from 1 nm to 10 nm) and aspect ratios, CNTs can be distributed in a much finer scale than common fibers, giving as a result a more efficient crack bridging at the very preliminary stage of crack propagation within composites. However, properties and dimensions of CNTs are strongly depending on the deposition parameters and the nature of the synthesis method, i.e., arc discharge, laser ablation, or chemical vapour deposition (CVD).

Carbon nanotubes used in this investigation was Multi walled carbon nano tubes of diameters from 3 to 8 nm and different length. The high specific strength, chemical resistance, electrical conductivity and thermal conductivity of carbon nanotubes (CNTs) make them attractive for use as reinforcement to develop superior cementitious composites. Kinetic property Multi-walled nanotubes, multiple concentric nanotubes precisely nested within one another, exhibit a striking telescoping property whereby an inner nanotube core may slide, almost without friction, within its outer nanotube shell thus creating an atomically perfect linear or rotational bearing.

This is one of the best examples of molecular nanotechnology, the precise positioning of atoms to create useful machines. Already this property has been utilized to create the world's smallest rotational motor. The toxicity of carbon nanotubes has been one of the most pressing questions in nanotechnology. Unfortunately such research has only just begun and the data are still fragmentary and subject to criticism. Preliminary results highlight the difficulties in evaluating the toxicity of this heterogeneous material. Parameters such as structure, size distribution, surface area, surface chemistry, surface charge, and agglomeration state as well as purity of the samples, have considerable impact on reactivity of carbon nanotubes.

2. REVIEW OF LITERATURES

Mostafa.Khanzadi et al has found that, the influence of Nano silica particles on the mechanical properties and durability of concrete has been studied through measurement of compressive and tensile strength, water absorption, and the depth of chloride penetration**.** The experimental results show that the mechanical properties measured, and the durability of the concrete mixed with the Nano particles were better than that of a plain concrete, also the SEM study of the microstructures showed that the nano particles filled the cement paste pores and, by reacting with calcium hydroxide crystals from calcium silicate hydration, decreased the size and amount of these crystals. Therefore the results indicate that Nano scale silica behaves not only as a filler to improve microstructure, but also as an activator to promote pozzolanic reaction.

Konstantin Sobolev et al has found that, an increase in compressive strength of mortars with developed nanoparticles at early stages of hardening followed by the strength reduction at later age. Addition of super plasticizer was proposed to overcome this obstacle. Super plasticized mortars with selected nano-SiO_2 demonstrated a 15-20% increase of compressive strength, reaching up to 144.8 MPa at 90-day age. The developed high-performance cements demonstrate the 28-day compressive strength at the range of 93 - 115 MPa, which is higher than 72 - 89 MPa, the strength of reference cements.

Zhen-Tian Chang et al has found that, Cements used in the concretes included a Portland cement, binary cement containing ground granulated blast furnace slag, and two ternary cements containing slag and silica fume or fly ash and silica fume. All the concretes had the same water/cement ratio of 0.4, with compressive strengths in the range of 45 MPa and 58 MPa at the age of 28 days. In the experiment, concrete cylinders were immersed in 1% sulphuric acid solution and they were periodically examined for appearance, measured for mass change and tested in compression up to 168 days. The concrete using limestone aggregates and the ternary cement containing silica fume and fly ash performed the best.

Serdar Aydın et al have found that, Cement was replaced with fly ash up to 70%. Sulfuric acid resistance of steam-cured concrete could be improved significantly by incorporation of fly ash. Curing by steam helped to increase the 1-day strength values. Long-term strength values decreased significantly for concrete mixtures over 30% of fly ash replacement levels.

Xiaoqing Gao et al has found that, Carbon Nano tubes resist micro crack formation, Multi wall carbon nano tube (MWCNT) gives greater composites with greater strength and stiffness. Excellent thermal conductivity of MWCNT can be achieved when carbon nano tubes are aligned in same direction and optimal carbon nano tube loading are chosen.

Vladimir Zivica et al has found that, Acidic resistance of cement-based materials prepared with chemically modified silica fume are acidic resistance of the cement-based materials when SF was applied. The changes of chemical composition, pore structure and mechanical properties of the test specimens, high acidic resistance was found on the cement-based materials when SF was applied. This allowed a solution to the problem of insufficient acidic resistance of current cement-based materials which represents a crucial world-wide problem.

M.S.Morsy et al has found that, CNT can improve mechanical performance, durability and sustainability of concrete. When OPC was substituted by 6 wt. % of cement by nano metakaolin (NMK) and the carbon nanotube was added by ratios of 0.005, 0.02, 0.05 and 0.1 wt. % of cement. It reduces the amount of cement needed in concrete in order to obtain higher strength. It also reduces the construction periods and produce high-strength concrete with less curing time.

Alireza Naji Givi et al has found that, SiO_2 nano-particles with two different sizes of 15 and 80 nm have been used as a partial cement replacement by 0.5, 1.0, 1.5 and 2.0 wt. %. It was concluded that concrete specimens containing SiO_2 particles with average diameter of 15 nm were harder than those containing 80 nm of SiO_2 particles at the initial days of curing. When nano particles are uniformly dispersed in the cement paste it promotes cement hydration which is favorable for Strength of Cement mortar. Nano particles fill the cement pores, thus increasing the strength. Nano Sio_2 can contribute to the hydration process to generate more C-S-H through reaction with $Ca(OH)_2$ Result shows Nano Sio_2 particles of blended concrete has higher compressive, flexural and tensile strength at all ages of moist curing..

A. Sadrmomtazi et al has found that, Nano-Sio_2 develops higher mechanical strength and lower water absorption when different amounts of nano-SiO2 (0%, 1%, 3%, 5%, 7%and 9%) were incorporated into the ordinary cement mortar. Here Nano-Sio_2 improves the bond strength of paste-aggregate interface and it was valuable for enhancing abrasion resistance and it accelerates cement hydration due to their high activity.

T. Bakharev et al has found that, Alkali-Activated Slag (AAS) concrete was immersed in an acetic acid solution of pH = 4. The main parameters studied were the evolution of compressive strength, products of degradation, and micro structural changes. AAS concrete of Grade 40 had a high resistance in acid environment, superior to the durability of OPC concrete of similar grade.

S.I Zaki et al have found that, the well-known performance of concrete without nanoparticles was compared with that after the addition of nano-particles for both fresh and hardened states. Nano-Silica concrete requires additional amount of water or super plasticizer to maintain the same workability level. Nano carbon tubes can improve the properties of concrete. Nano-Silica addition results in significant increase in concrete compressive strength after 28-days up to one year and the optimum amount of nano silica is 0.5% by weight of cementitious material.

Ashwani K. Rana et al has found that, the article examines the potential areas where nanotechnology can benefit construction engineering. The data and information collected is from current literature. The purpose is to point out clear cut direction among the nanotechnology development areas where the construction process would immediately harness nanotechnology, by specifying clear recommendations. The information would be beneficial to both construction engineering education and research. In this paper a broad overview of the potential application of various nanotechnology developments in the construction engineering field is discussed, and the potential for further basic research that may lead to improved systems is evaluated. Nanotechnology has been concerned with developments in the fields of microelectronics, medicine and materials sciences. The potential for application of many of the developments in the nanotechnology field in the area of construction engineering is growing.

K. Amutha et al has found that, Nano silica is produced from rice husk ash; an agriculture waste is used to produce nanosilica when rice husk ash is burnt in a muffle furnace at 650°C for 4 hrs amorphous silica is obtained. Pure silica is extracted by titration method using 5N H_2SO_4 solution with constant stirring at controlled temperature. By refluxing method 98% of pure nanosilica can be extracted from pure silica. SEM results show the particles are in agglomeration form and the dimension is 80 mm. XRD powder pattern of nanosilica confirms the amorphous nature of the substance. An FTIR spectrum adds evidence indicating the presence of nanosilica.

F. Pacheco-Torgal et al has found that, the paper reviews current knowledge about nanotechnology and nano materials used by the construction industry. It covers the nanoscale analysis of Portland cement hydration products, the use of nanoparticles to increase the strength and durability of cementations composites, the photo catalytic capacity of nonmaterials and also nanotoxicity risks. Nanotechnology seems to hold the key that allows construction and building materials to replicate the features of natural systems improved until perfection during millions of years.

3. EXPERIMENTAL INVESTIGATION

3.1 Scanning Electron Microscopic (SEM) Test

SEM analyzer is an instrument used for the examination and analysis of the micro structural characteristics of the solid sample. Morphological characteristic is found out by using the instrument Hitachi S-3000H as shown in **Fig-1**. 3-D appearance of the specimen image gained by this test and it gives the direct result of the large depth of field. At greater depth of field much more information about the specimen was found out. This instrument can give the morphological structure of the specimen for various magnifications.

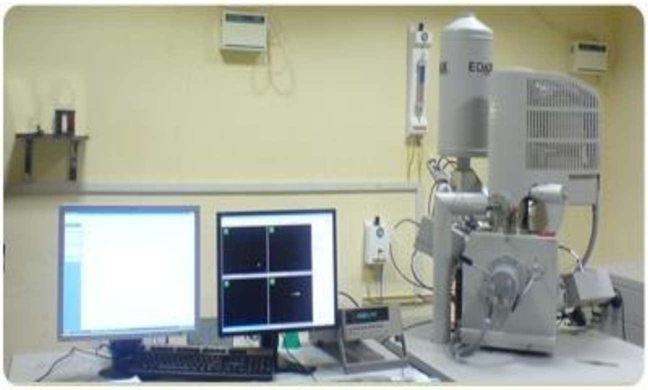

Fig-1: SEM Testing Machine

3.2 Transmission Electron Microscopic (TEM) Test

TEM is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image. The specimen is most often an ultrathin section less than 100 nm thick or a suspension on a grid. An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device as shown in **Fig-2**, such as a florescent screen, a layer of photographic film, or a sensor such as a charge-coupled device.

Fig-2: TEM Testing Machine

3.3 X-Ray Diffraction (XRD) Test

This test is used to find out the qualitative mineral analysis. Using this test has done quantitative determination of glass and crystallization components. The measurement of X-ray diffraction line intensities is the basis for quantitative phase analysis. X-ray diffraction techniques are widely used for determining the identity and structure of crystalline materials. Graph is drawn between 2θ and counts. Thus the quantitative and qualitative measures of the sample have done by using this test. The X-Ray Diffraction (XRD) technique is used to measure atomic spacing between lattice layers in a crystal. This can be calculated from the X-Ray Diffraction test results using the Braggs Law equation, is as follows: $n\lambda = 2d \sin\Theta$, where, λ = wavelength of the incidental X-Ray beam; d = the atomic spacing between the layers; Θ = angle of incidents; n is taken as unity.

3.4 TAFEL Extrapolation Technique

After the electro injection process, the same set-up was used as shown in **Fig-3** for polarization measurements to evaluate the corrosion kinetic parameters such as corrosion current (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (b_c) and anodic Tafel slope (b_a). Both cathodic and anodic polarization curves were recorded potentiodynamically using ACM Instruments, UK. The potentiodynamic conditions correspond to a potential sweep rate of 1 mVs^{-1} and potential ranges of 0.2 to −0.2 V from the OCP. All the experiments were carried out at constant temperature of 32±1°C. For a comparison, polarization was also carried out for the geo polymer and control system.

Fig-3: TAFEL testing machine

4. TEST RESULTS AND DISCUSSION

4.1 Scanning Electron Microscopy Results

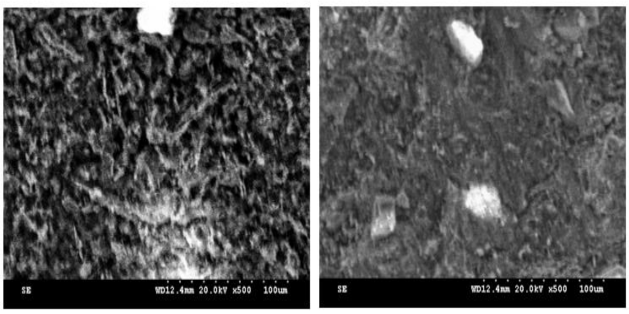

Fig-4: SEM image for Control Mix

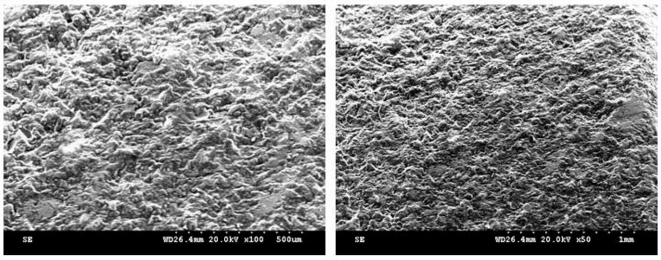

Fig-5: SEM image for 1% Nano Silica

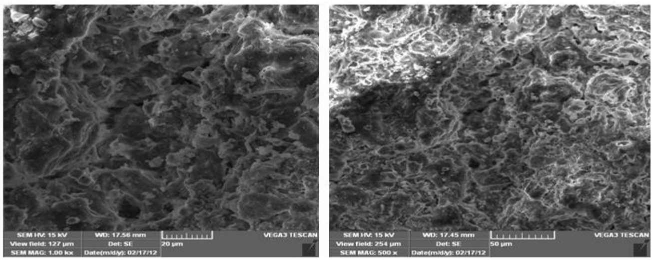

Fig-6: SEM image for 1% NS + 0.01% CNTs

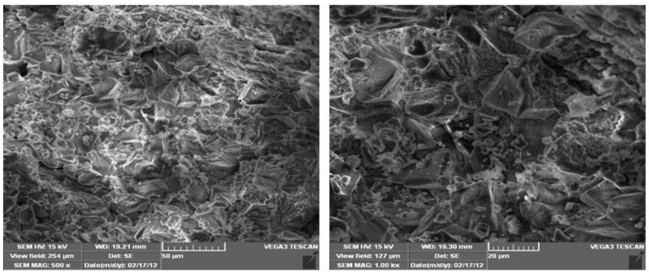

Fig-7: SEM image for 1% NS + 0.03% CNTs

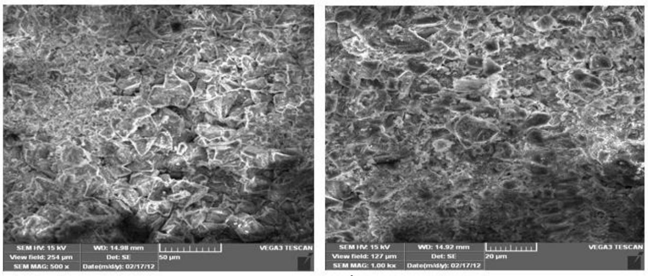

Fig-8: SEM image for 1% NS + 0.05% CNTs

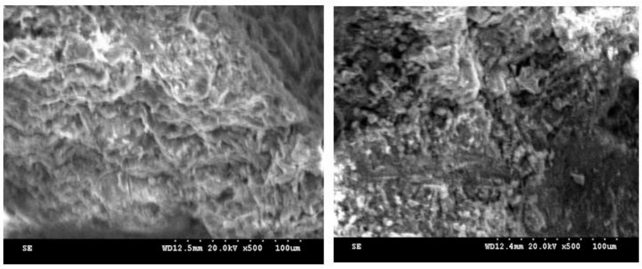

Fig-9: SEM image for 1% NS + 0.07% CNTs

Fig-4 to Fig-9 shows the SEM image for various Nano silica and CNTs admixed systems. From the SEM images it is observed that, Nano-particles improved the structure of the aggregates contact zone, resulting in a better bond between aggregates and cement paste. 1% NS and 0.03% CNT composite system has shown a dense, compact and uniform microstructure indicating the uniform distribution of the Nano particles along with good bonding between cement and sand particles. But beyond 0.03% CNT, there is no uniformity in the distribution of nanoparticles observed which is very well evidenced from SEM micrographs. This shows that 1% NS with 0.03% CNT is found to be the optimum level in making high performance concrete.

4.2 Transmission Electron Microscopy Results

Synthesized Nano silica powder was characterized and conformed using TEM and their microscopy results were shown in Fig-10.

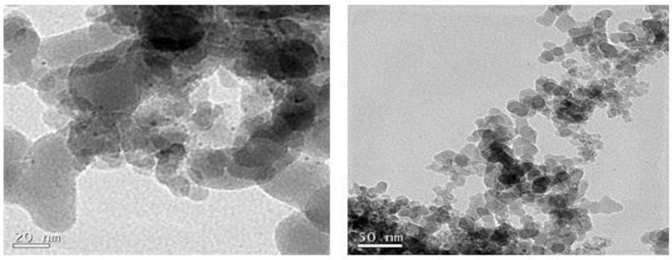

Fig-10 TEM images of Nano silica particle size ranging from 20 to 50 nm

4.3 X-Ray Diffraction (XRD) Test Results

Table-1: XRD results of Nano silica

Pos. [°2Th]	Height [cts]	FWHM [°2Th]	d-spacing [Å]	Rel. Int. [%]
10.3826	43.23	0.6298	8.52033	15.42
19.1605	192.39	0.1574	4.63220	68.64
28.1577	183.14	0.0600	3.16920	65.34
29.1112	145.36	0.5285	3.06754	51.86
32.2550	280.28	0.1771	2.77537	100.00
38.5200	7.00	0.0900	2.33718	2.50
42.1200	3.00	0.0900	2.14538	1.07
48.9161	82.62	0.2880	1.86051	29.48
58.3200	1.00	0.0900	1.58221	0.36

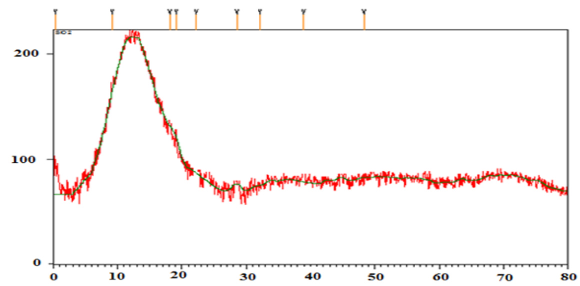

Fig-11: X - ray diffraction pattern of Nano silica

Table-2: XRD results of CNT

D Spacing	Standard D Spacing	Pdf Number	Compound
4.9062	4.9063	841263	$Ca(OH)_2$
4.2532	4.2530	261381	$Ca_3Al_6Si_{11.5}O_{36.18}$
3.3428	3.3423	881130	$Ca_{1.42}(Al_2Si_2O_{12})H_2O_{2.76}$
3.3353	3.3351	811858	$Ca_4(Al_8S_{18}O_{32})(H_2O)_{16}$
3.33505	3.3351	811858	$Ca_4(Al_8SI_8O_{32})(H_2O)16$
3.33247	3.3325	761808	$CaAl_2(Si_2O_7)(OH)_2(H_2O)$
3.33101	3.3306	720467	$Ca\,Al_2Si_2O_8(H_2O)_4$
3.3089	3.3087	722282	$Ca_2Al_3Si_3O_{12}(OH)$
2.6290	2.6280	040733	$Ca(OH)_2$
2.1291	2.1298	740874	$Ca_3(Si_3O_9)$
2.1281	2.1284	510092	$CaSi_2O_5$
1.37233	1.3722	730599	$Ca_3(SiO_4)O$

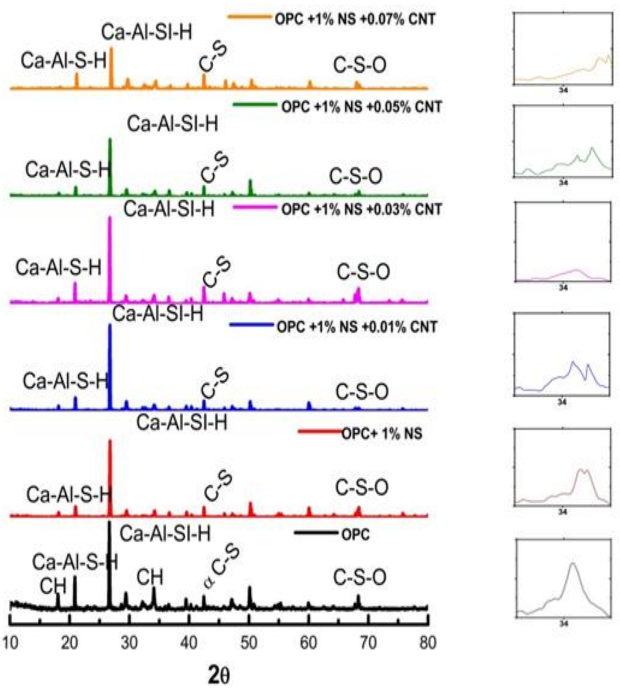

Fig-12: X - ray diffraction pattern of CNT

Fig-11 & 12 and Table-1 & 2 shows the XRD pattern of 1% percentage level of nano silica powder and different percentage of CNT in OPC cement. The XRD peaks were identified by using JCPDF (standard) table. The control system has shown high intensity crystal phase at 26^0-34^0 indicating Calcium aluminum silicate hydrate ($Ca.Al_2.Si_2O_8.4H_2O$), low intensity crystal phase peak at 17^0& 24^0 $Ca(OH)_2$ indicating Calcium hydroxide peaks were noticed. Nano silica and CNT added system has shown a crystal phase of Ca $(OH)_2$ peak were reduced (or peak shifted to calcium silicate hydrate). Lime reacts with water and produces the Ca $(OH)_2$ which reacts with SiO_2 to form C-S-H gel. When SiO_2 nano -particles react with Ca $(OH)_2$ to produce C-S-H gel, which is increased because of high free energy of nano-particles which reduces significantly when reacts with Ca $(OH)_2$. And high intensity of the crystal phase at 26^0-34^0 Calcium aluminum silicate hydrate (Ca_4 (Al_8S_{18} O_{32}) $(H_2O)_{16}$ & $Ca_3Al_6Si_{11.5}$ $O_{36.18}$) peak is observed.

4.4 TAFEL Extrapolation Results

Fig-13 and Table-3 shows the CNT behavior of different systems. From the figure it is observed that 0.03% CNT has lesser Icorr value and lesser corrosion rate indicating the better performance than the other systems.

Table-3: TAFEL Results

SYSTEM	OCP	Ba	Bc	Icorr (mA/cm²) (x10³)	Corrosion Rate mmpy (x10-³)
OPC	-342	91	86	4.0601	46.4302
OPC + 1%NS	-383	48	43	1.987	5.2401
OPC + 1%NS + 0.01%CNT	-425.6	47	45	1.431	16.5856
OPC + 1%NS + 0.03%CNT	-480	41	38.9	0.5748	6.6614
OPC + 1%NS + 0.05%CNT	-589.4	36	34	4.0275	46.6788
OPC + 1%NS + 0.07%CNT	-576.72	41	44	6.4792	75.0937

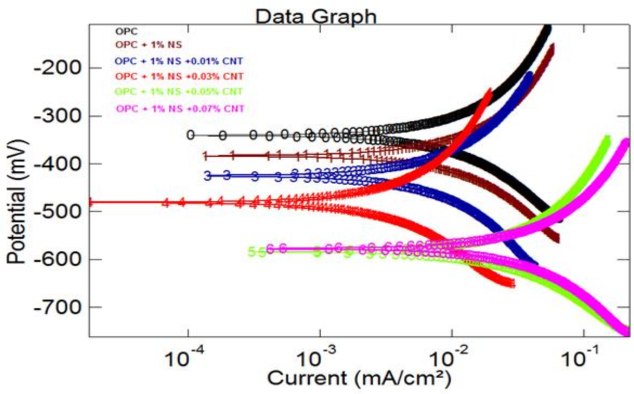

Fig-13: TAFEL chart

5. CONCLUSION

From the above results, it was found that Nano-particles improves the structure of the aggregates contact zone, resulting in a better bond between aggregates and cement paste which is well evidenced from SEM and TEM micrograph and XRD pattern of 1% percentage level of Nano silica powder and different percentage of CNT in OPC cement gives good results.

And also, from the TAFEL Extrapolation corrosion rate determination technique, it was observed that 0.03% CNT has lesser Icorr value and lesser corrosion rate indicating the better performance than the other systems.

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