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Nanomaterials for corrosion protection.

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Abstract

Three different nanomaterials were employed for evaluating the corrosion protection performance of zinc rich primers. These involved nanosilica, nanotalc, multi walled carbon nanotubes (MWCNTs) and their combinations with different weight proportions and at varying pigment volume concentrations (40, 50 and 60%). These materials were also combined with conventional pigment like zinc oxide and zinc phosphate in order to compare the corrosion resistance properties. These primers were tested for performance properties like salt spray, EIS, conductance and film density. Incorporation of these materials was found to be improving the corrosion resistance properties at lower dry film thickness.

Key Words: Corrosion, Nanomaterials, Electrochemical Impedance Spectroscopy, DFT

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1. Introduction

Metals are used to meet our requirements for the purpose of construction, manufacturing and application. In many areas good mechanical properties and high speed of fabrication makes the steel more demandable. [1] The structures made up of steel gets corroded and fouled due to the environment, which results in direct and indirect losses to a country which could be as high as 5% gross national production.[2] retarding corrosion or combating corrosion of metals or alloys is most important.

Coatings with metallic zinc provides the sacrificial as well as barrier protection, therefore, Zinc is more widely used in the primers throughout the world over half a century and has reached a level of in conjunction of the performance with modern coatings system. Zinc rich coatings often called zinc Rich Primer (ZRP) are a thermosetting coatings possessing inorganic or organic binder resin with zinc pigment highly loaded in epoxy, epoxy ester, urethane, vinyl or chlorinated rubber. Coatings system that includes these high performance primer components can provide corrosion protection property as long as 20 year or more, depending upon the severity of the environment. [3] The zinc is unique and widely used for anticorrosion due to its protective property. The use of organic coatings is easier due to their flexibility, other mechanical leads over the inorganic coatings for improving the life of the coatings. [4] In order to provide the best performance, there should Zn-Zn particle contact. This requires very high percentage of zinc.

Partial replacement of zinc other material will help to increase the coating properties for better cathodic as well as barrier protection. The materials which will improve conductivity, particle to particle contact improving overall performance. Nanomaterials like nanosilica, nano zinc oxide, nano talc, multi walled nano tubes (MWNTs), micro-nano composite structures (zinc-nano zinc oxide) and their various combinations will pave new era of developments in the coating industry especially for the corrosion protection. The plate like structure of nano silica and its inertness gives barrier protection and excellent chemical resistance. Same will prove true in case of nano talc due to its plate like and inorganic nature. MWNTs will improve conductivity giving cathodic protection. The combination of all these nanoparticles along with traditional materials

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like zinc, zinc oxide and zinc phosphate will lead to phenomenal and drastic change in the coating end properties will pave to new era of developments in the frontier as well as rear areas of coating industry.

2. Experimental Work

2.1 Materials

Epoxy resin, D.E.R. 331, was obtained from Dow Chemical Company, Mumbai, India. Aromatic amine was obtained from M/s. D.R.Coatings Ltd., India. Zinc and zinc phosphate was gifted by Transpek-Silox Industry Limited, Vadodara, India. ZnO was obtained from M/s. S.D. Fine chemicals, Mumbai, India. Nanosilica was procured from Nabond (NB Nano-Si01), China, having a mean grain size of 10nm and specific surface area of >600m^2/g. MWNT was procured from NTP (grade-1030), China, having length of 1-2 µm and diameter of 10-30nm. Other chemicals such as Xylene and Butanol were procured from M/s. S.D. Fine chemicals, Mumbai, India. All these materials were used without further purification.

2.2 Processing of coatings

2.2.1 Experimental work

Epoxy resin (DGEBA) was dissolved in n-butanol and xylene solvent mixture in 1:1 volumetric ratio. Ball milled lamellar zinc and nano materials were dry blended in sealed packed bag. This dry blended material was added as per recipes on the basis of pigment volume concentration. Theoretical CPVC was calculated on the basis of oil absorption and density of blend of zinc particles and composite material and formulated at PVC close to its combinations CPVC. Therefore, the pigment volume concentration was fixed at 40, 50 and 60%. The percentage weight replacement of zinc was done with nano material in 1, 3 and 5%. This mixture was then stirred vigorously by using high speed disperser (supplied by Tudor Machine, India) for 25-30 minutes or till uniform dispersion was obtained. Some amount of solvent mixture was also added during dispersion, if required, to control the viscosity for the ease of processing. Required amount of the curing agent (i.e. reactive polyamine), calculated on the basis of epoxy functionality, was dissolved in solvent mixture and then added to the system. The panels were

made of mild steel and obtained from a local supplier. Dry film thickness was measured and found to be 30±10 µm. The recipes of the primers are shown in Table No:-2.1 to 2.8.

2.3 Testing and analysis of paint formulations

2.3.1 Critical Pigment Volume Concentration (CPVC) ASTM-D-153 and D-281

Critical Pigment Volume Concentration of pigment mixtures used for various primer formulations were calculated using following formula. It requires pigment densities and oil absorptions (O.A.) values which were determined according to ASTM D-153 and ASTM D-281 respectively.

$$CPVC = \frac{93.5}{93.5 + (\text{Oil Absorption X Pigment Density})}$$

2.3.2 Film Porosity

Film porosity values were calculated using the theoretical formula as given below

$$\text{Porosity} = 1 - (CPVC / PVC)$$

2.3.3 Film Density

Film densities of the primer films on the metallic surface were measured according to the hydrostatic weighing method. The weighing was performed on a Mettler H 20 balance. The film density was calculated through the following equation:

$$\rho = \rho_w (\Delta W_A / (\Delta W_A - \Delta W_W))$$

Where, ΔW_A = difference between the weights of panels plus film and non-painted panel in air.

ΔW_W = difference between the weights of panels plus film and non-painted panel in water.

ρ_w = density of water

2.3.4 Conductance

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Along with good barrier to oxygen and water zinc rich primer should also be able to protect the metal galvanically. For better corrosion resistance the conductivity should be high; approaching to that of metal conductivity. The conductances of the clean primed panels were measured with the help of a millimeter by bringing the pointers of the probes in contact with the coating on the panel surface while distances between two points were 5 cm. Several readings were recorded at various positions on the coating and the average of these readings was then noted.

2.3.5 Electrochemical Impedance Spectroscopy (EIS)

The EIS measurements were carried out using a Gamry Reference 600 Potentiostat/ Galvanostat/ ZRA along with PTC1 cell setup. The working electrode, reference electrode and counter electrode were connected to the Potentiostat. Potentiostat equipped with EIS-100 software was used to run the experiment. The working electrode, which was the coated substrate, was exposed to the salt solution (3.5 wt %). The coating was allowed to attant equilibrium with the salt solution before starting the experiments. The measured frequency range was from 0.002 Hz to 100 kHz, with AC excitation amplitude of 10 mV. The surface area exposed to the electrolyte was 13.2 cm^2. EIS plots were analyzed by Echem Analyst software.

2.3.6 Tafel Scan

The experiments were carried out as per above mentioned procedure. With the difference of Potentiostat with DC-105 corrosion software was used to run the experiment. The Tafel plots were generated by applying a potential of 250 mV in both the positive and negative directions from the open circuit potential against SCE as reference electrode. Gamry Reference 600 equipped with Echem Analyst software was used to analyze the Tafel plots.

2.3.7 Salt Spray Test (ASTM B 117)

The coated mild steel panels were tested for their corrosion resistance properties according to ASTM B-117 method. Two intercepting scribes were made down to the metal surface for observing the protective action of the primers. The panels were placed at an angle of 45° and 3.5% NaCl solution was atomized at 35°C in the salt spray chamber. The panels were continuously exposed to the salty environment for 3000Hr and inspected for corrosion on the scribed area according to ASTM D-1654 at an interval of 250 h along with the signs of

blistering, staining, loss of adhesion. Size and degree of blistering were also evaluated according to ASTM D-714 so that comparative of severity can be judge. Formation of pitting under film was evaluated by careful stripping of coatings and reported in accordance with ASTM D- 1654.

2.3.8 Solvent Resistance Test

Convertible coatings, such as epoxies, vinyl esters, polyesters, alkyds and urethanes, become more resistance to solvents as they cure. These coatings should reach specific levels of solvent resistance prior to being top coated and placing in service; the levels of solvent resistance necessary vary with the type of coating and the intended service. Drop of solvent is placed on the coating surface and were analyzed for the dissolution of resin, change in colour.

2.3.9 Mechanical Tests

1. **Impact Test:** Impact test method for impact resistance has been found to be useful in predicting the performance of organic coatings for their ability to resist cracking caused by impacts. A steel punch with a hemispherical head having a diameter of 0.625 in. (15.9 mm) was released from height of 24 inch. Impact was carried out on the both sides of coated panels and analyzed accordance to the standard. ASTM D-2794
2. **Conical Mandrel Bending Test** (ASTM D 522)**:** The coated panels were bent over a conical mandrel and the resistance of the coating to cracking is determined. They have been useful in evaluating the flexibility of coatings for their ability to resist cracking when elongated.
3. **Crosscut Adhesion Tape Test** (ASTM D3359): This test was used to assess the adhesion of coating films to metallic substrates. Cuts were made on the coating with adequate pressure and speed the cutting tool has an angle between 15 and 30°. The grid area was brushed and a 2.5 cm wide semi- transparent pressure-sensitive tape was placed over it. After 30 sec of application, the tape was removed rapidly and the grid inspected according to the (ASTM D-3359) standards. The amount of coated area retained over the panel corresponds to the adhesion efficiency of the primer. The more coated material removed by the tape, the poorer the adhesion of the coating.

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

4. Results & Discussion

4.1 Critical Pigment Volume concentration (CPVC)

CPVC for all the formulations were calculated. The average of all the formulations CPVCs are very close to 50%; therefore formulations were made with PVCs 40, 50 and 60% that is ±10% of the CPVC.

4.2 Film porosity

The film porosity was calculated theoretically from CPVC & PVC. At 40% PVC film porosity was less than one confirming that there are no free spaces in the coating film. For 50% PVC the film porosity is near about one for all the formulations confirming that film is slightly porous. At 60% PVC the film porosity values are more than one, showing that there is not enough binder in the coating that can completely wet the pigment. .

4.3 Conductance

The incremental rise in PVC in comparison to CPVC of the coating conductance was also increasing. This might be due to exponential jump in the surface area lead to the increased contact between the individual zinc-zinc particles as well as between zinc and nano materials. It was also observed that the conductance of the MWNT was much more superior to that of the other nano materials. This can be attributed to the inherent conductance of the MWNT which is higher than that of the other nano materials. The values of the conductance are shown in the **Table No: - 4.1-4.7** Film porosity is an indicator of the packing ability of the pigments and binder within the coating film. As the PVC gradually increasing the porosity is also increasing. At 40% PVC that is below CPVC there were no porosity and same is the case at 50% PVC, where CPVC and PVC are matching.

4.4 Electrochemical Impedance Spectroscopy

4.4.1 Bode Plot

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Electrochemical impedance spectroscopy this is nothing but the nondestructive, simple, high informative method for analyzing the coating sample.

The Bode plots for all the systems are shown below. Most of them show impedance in the range of 10^5 to 10^3 ohms showing better corrosion resistance. There were some fluctuations in the reading which are to be attributed to the electrical noise present in the surrounding environment of the instrument. These can be minimized by using Faradays cage. Coatings having 40% PVC showed higher values of impedance as compared to the 50% and 60% PVC. These results are in accord with the results obtained from the salt spray test. The results shown by salt spray test after 2000 hours of exposure are in coherence with the results obtained from EIS within 20 days of immersion. Impedance values for 40% PVC for the replacement of zinc with 1, 3 and 5 wt% of MWNT showed increase after 20 days of immersion in 3.5% NaCl solution. This is attributed to the formation of coherent layer of oxide which has been resulted from the corrosion due to the presence of corrosive environment (NaCl). The Bode plots are shown in **Figure No: - 4.1 – 4.4.**

4.4.2 Tafel Plot

From Tafel plots values of open circuit potential (Ecorr), corrosion rate, corrosion current (Icorr) are calculated. All these values were calculated using Stern-Gaery equation and Butler-Volmer equation. Corrosion potential is potential difference between working electrode (metal sample) & reference electrode. The corrosion rate is expressed in mils/ year. Polarization resistance is the ability of the coating to resist the corrosion reaction. The corrosion current is the measure of electrons flow in the corrosion reaction it is in µA.

For all nanomaterials corrosion rates were found to be minimum for formulations based on 40% PVC. The results of Tafel plots are shown in **Table No 4.8- 4.12.**

4.5 Evaluation by salt spray test (ASTM B-117)

4.5.1 Corrosion Rating

The extent of corrosion was represented in an arbitrarily numerical scale from 0 to 10. In this 10 represented no corrosion and 0 represented failure. The results are as shown in

Figure No: - 4.5 -4.10

ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

The samples were exposed for salt spray test for the period of 2500h for nano silica, nano talc and MWNTs and for mix systems for 2000 h. The observations were made intermittingly with the intervals of 250h. Nano silica, nano talc and MWNTs showed better performance with 40% PVC. Plate like structures of silica and talc (magnesium silicate) gave barrier protection lengthening path of corrosive species to reach the substrate. This barrier protection was coupled with the cathodic protection of zinc itself.

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JCSE The Journal of Corrosion Science and Engineering
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Photographs of one of the systems are shown below

Nano Talc 40 PVC 1% Nano Talc 50 PVC 1% Nano Talc 60 PVC

JCSE The Journal of Corrosion Science and Engineering
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Conclusions

It is observed that primers based on MWNT, Nano Talc and Nano Silica showed better corrosion resistance for 40% PVC at DFT = 30±10 µm whereas primers based on MWNT, Nano Talc and Nano Silica along with zinc oxide and zinc phosphate showed better corrosion resistance for 60% PVC at DFT = 30±10 µm. It is found that results obtained from the Salt spray study and EIS are found to be in good agreement. So with the help of nanomaterials, better corrosion resistance properties can be obtained at lower dry film thickness.

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JCSE The Journal of Corrosion Science and Engineering
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JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Tables:-

Table No: -2.1 Nano Silica based Zinc rich primer

% Replacement	1			3			5		
% PVC	40	50	60	40	50	60	40	50	60
	Grams								
Epoxy	11.164	7.883	5.471	12.022	8.528	5.938	12.853	9.157	6.398
Hardener	5.582	3.941	2.735	6.011	4.264	2.969	6.427	4.579	3.199
Zinc	63.401	67.149	69.904	61.159	65.071	67.969	58.988	63.039	66.064
Nano-Silica	0.640	0.678	0.706	1.896	2.013	2.102	3.105	3.318	3.477
Solvent Mixture	19.212	20.348	21.183	18.915	20.125	21.021	18.628	19.907	20.862
Total	100	100	100	100	100	100	100	100	100

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 2.2 Nano Talc based Zinc rich primer

% Replacement	1			3			5		
% PVC	40	50	60	40	50	60	40	50	60
	Grams								
Epoxy	10.872	7.664	5.313	11.162	6.784	5.469	11.449	8.118	5.625
Hardener	5.436	3.832	2.657	5.581	3.392	2.735	5.724	3.788	2.812
Zinc	63.735	67.399	70.084	62.122	67.022	68.494	60.528	64.376	66.911
Nano Talc	0.6438	0.681	0.708	1.921	2.073	2.118	3.186	3.388	3.522
Solvent Mixture	19.314	20.424	21.238	19.213	20.728	21.184	19.114	20.329	21.129
Total	100	100	100	100	100	100	100	100	100

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ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-2.3 MWNT based Zinc rich primer

% Replacement	0.5			1			2		
% PVC	40	50	60	40	50	60	40	50	60
	Grams								
Epoxy	10.867	7.661	5.311	11.009	7.762	5.202	7.813	7.977	5.538
Hardener	5.434	3.830	2.655	5.504	3.884	6.033	5.866	3.988	2.797
Zinc	64.068	67.743	70.441	63.579	67.286	67.599	65.072	66.365	69.101
MWNT	0.322	0.340	0.354	0.642	0.679	0.683	1.328	1.354	1.410
Solvent Mixture	19.315	20.425	21.239	19.266	20.389	20.484	19.920	20.316	21.153
Total	100	100	100	100	100	100	100	100	100

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JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: -2.4 Nano Talc and MWNT based Zinc rich primer.

% Replacement	0.5 %MWNT 1% Nano Talc			0.5 %MWNT 2% Nano Talc		
% PVC	40	50	60	40	50	60
	Grams					
Epoxy	11.01	7.77	5.39	11.16	7.88	5.47
Hardener	5.51	3.88	2.69	5.58	3.94	2.73
Zinc	63.25	66.94	69.64	62.44	66.14	68.85
Nano Talc	0.64	0.679	0.71	1.28	1.36	1.41
MWNT	0.32	0.339	0.35	0.32	0.34	0.35
Solvent Mixture	19.26	20.39	21.21	19.21	20.35	21.18
Total	100	100	100	100	100	100

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: -2.5 Nano Talc and MWNT based Zinc rich primer.

% Replacement	1 %MWNT 1% Nano Talc			1 %MWNT 2% Nano Talc		
% PVC	40	50	60	40	50	60
	Grams					
Epoxy	11.15	7.88	5.47	11.29	7.98	5.54
Hardener	5.58	3.94	2.73	5.65	3.99	2.77
Zinc	62.77	66.48	69.20	61.97	65.68	68.41
Nano Talc	0.64	0.69	0.71	1.28	1.35	1.41
MWNT	0.64	0.69	0.71	0.64	0.68	0.71
Solvent Mixture	19.22	20.35	21.18	19.17	20.31	21.16
Total	100	100	100	100	100	100

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-4.1 Conductance of formulations based on Nano Talc

Nano Talc	1%			3%			5%		
% PVC	40	50	60	40	50	60	40	50	60
Conductance	0.09	5.6	107	7.03	10.9	14.6	0.16	17.86	16.25

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 4.2 Conductance of formulations based on Nano silica

Nano silica	1%			3%			5%		
% PVC	40	50	60	40	50	60	40	50	60
Conductance	0.05	0.06	21	0.12	127	417	0.1	101	272

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-4.3 Conductance of formulations based on MWNT

MWNT	0.50%			1%			2%		
% PVC	40	50	60	40	50	60	40	50	60
Conductance	0.02	32.7	677	10.4	61.5	895	3.21	21	46

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-4.4 Conductance of formulations based on MWNT and Nano Talc

% MWNT	0.5			0.5			1			1		
% Nano Talc	1			2			1			2		
% PVC	40	50	60	40	50	60	40	50	60	40	50	60
Conductance	0.07	4	350	18	22	36	14	29	5	0.1	15	649

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 4.5 Conductance of formulations based on mix Nano Talc

Mix Nano Talc	1 %			3%			5 %		
% PVC	40	50	60	40	50	60	40	50	60
Conductance $S.Cm^{-1}$	0.06	2.43	112	0.05	7	192	0.07	12	1564

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-4.6 Conductance of formulations based on mix Nano Silica

Mix Nano Silica	1 %			3%			5 %		
% PVC	40	50	60	40	50	60	40	50	60
Conductance $S.Cm^{-1}$	0.05	20	563	0.10	75	571	0.09	210	869

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No:-4.7 Conductance of formulations based on mix MWNT

Mix MWNT	0.5 %			1%			2 %		
% PVC	40	50	60	40	50	60	40	50	60
Conductance $S.Cm^{-1}$	0.16	5.88	143	0.08	7.49	357	0.07	5.00	5714

Table No: - 4.8. Icorr, Ecorr and Corrosion Rate for formulations based on Nano silica

Nano Silica Replacement	1			3			5		
% PVC	40	50	60	40	50	60	40	50	60
Icorr, µA	2.189	129.9	1.385	64.62	850.9	1028	68.99	299.0	818.1
Ecorr, mV	-871.2	-1074	-1079	-1131	-1076	-1090	-1082	-1104	-1087
Corrosion Rate, mpy	4.415	5.260	56.12	2.618	34.47	41.63	2.795	12.11	33.14

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 4.9. Icorr, Ecorr and Corrosion Rate for formulations based on Nano talc

% Nano Talc Replacement	1			3			5		
% PVC	40	50	60	40	50	60	40	50	60
Icorr, µA	-	3.302	1088	-	1088	264.3	-	134.4	731.5
Ecorr, mV	-	-983.0	-1076	-	-1078	-1083	-	-1072	-1080
Corrosion Rate, mpy	-	0.3317	44.06	-	5.180	10.71	-	5.445	29.63

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 4.10 Icorr, Ecorr and Corrosion Rate for formulations based on MWNT

%MWCNT Replacement	0.5			1			2		
% PVC	40	50	60	40	50	60	40	50	60
Icorr, µA	-	88.77	3.432	-	308.4	1264	-	208.6	1476
Ecorr, mV	-	-1.104	-965.2	-	-1088	-1082	-	-1101	-1082
Corrosion Rate, mpy	-	3.596	6.9204	-	12.49	51.19	-	8.451	59.79

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Table No: - 4.11. Icorr, Ecorr and Corrosion Rate for formulations based on MWNT and Nano talc

% Replacement	0.5 MWNT/ 1 Nano talc			0.5 MWNT/ 2 Nano talc		
% PVC	40	50	60	40	50	60
Icorr, µA	-	6.365	321.7	-	561.9	735.3
Ecorr, mV	-	-1055	-1073	-	-1079	-1098
Corrosion Rate, mpy	-	12.84	13.03	-	22.76	29.79

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

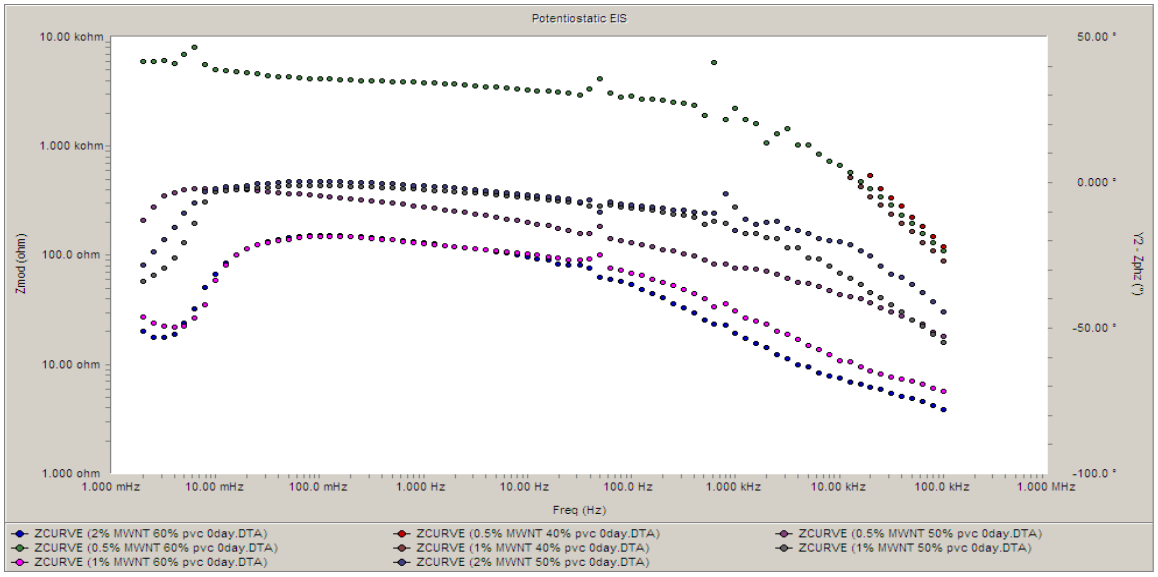
Table No: - 4.12. Icorr, Ecorr and Corrosion Rate for formulations based on MWNT and Nano talc

% Replacement	1 MWNT/ 1 Nano talc			1 MWNT/ 2 Nano talc		
% PVC	40	50	60	40	50	60
Icorr, µA	-	207.0	1109	-	252.0	1.325
Ecorr, mV	-	-1.104	-1.078	-	-1.083	-1.073
Corrosion Rate, mpy	-	8.386	44.91	-	10.21	53.69

JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

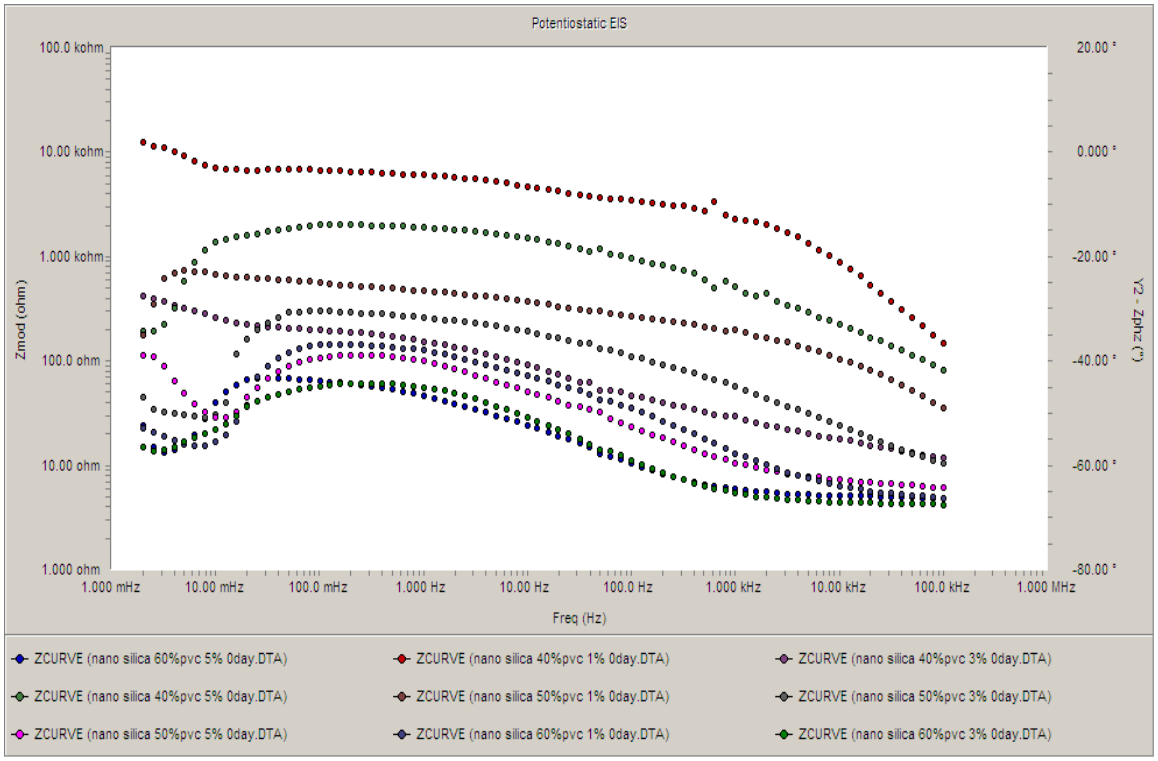
Figures

Figure No: - 4.1 Bode plot for formulations based on MWNT for 0 days for 0.5, 1 & 2% replacement



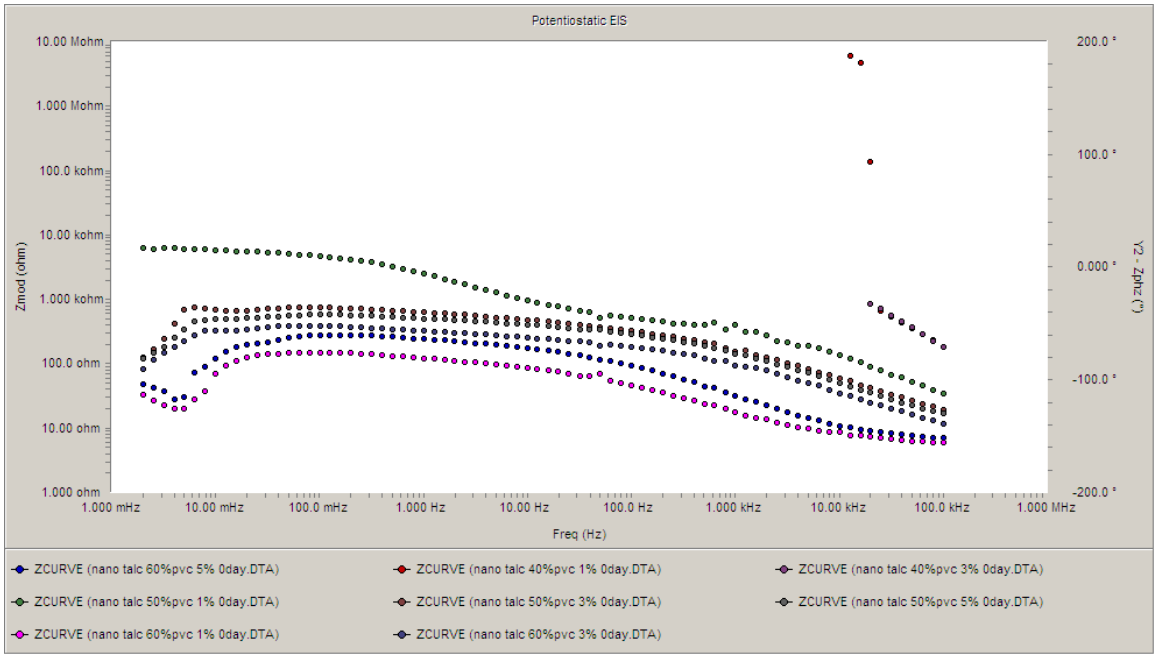
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: - 4.2 Bode plot for formulations based on Nano silica for 0 days for 1, 3 & 5% replacement



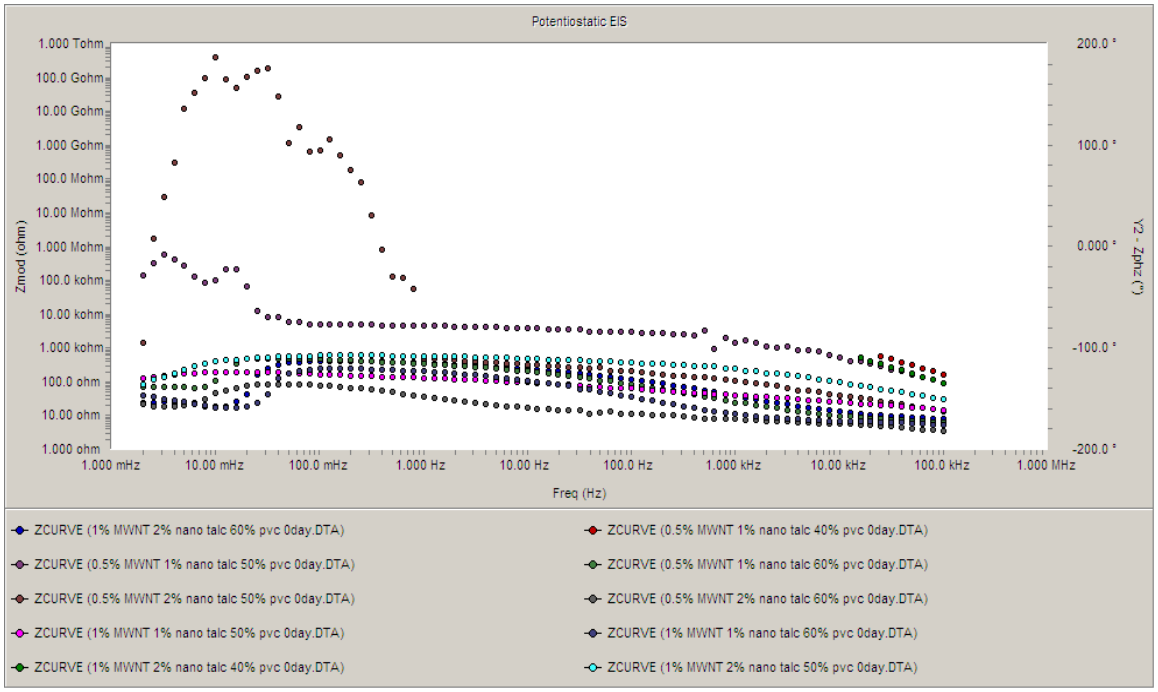
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: - 4.3 Bode plot for formulations based on Nano talc for 0 days for 1, 3 & 5% replacement



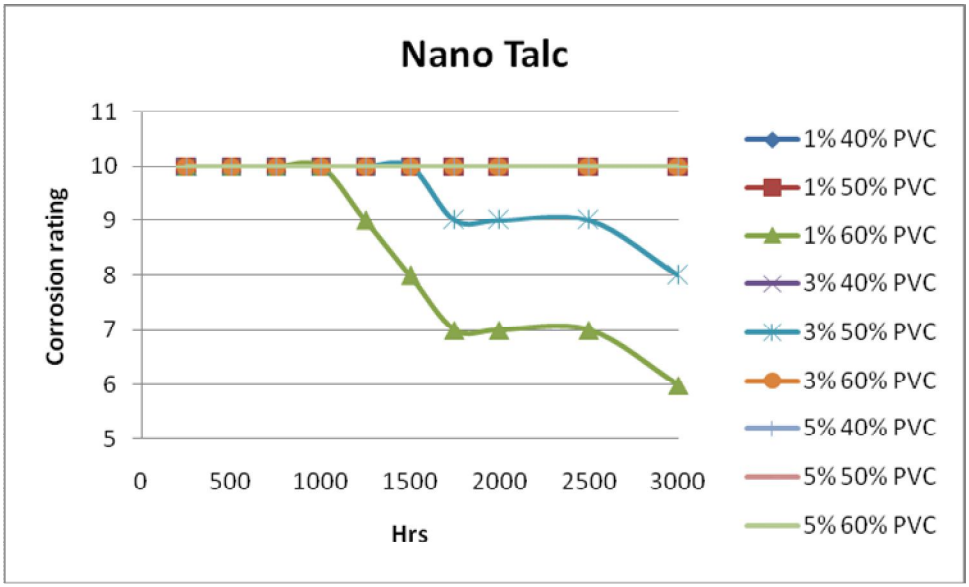
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: - 4.4 Bode plot for formulations based on MWNT and Nano talc for 0 days for various % replacement



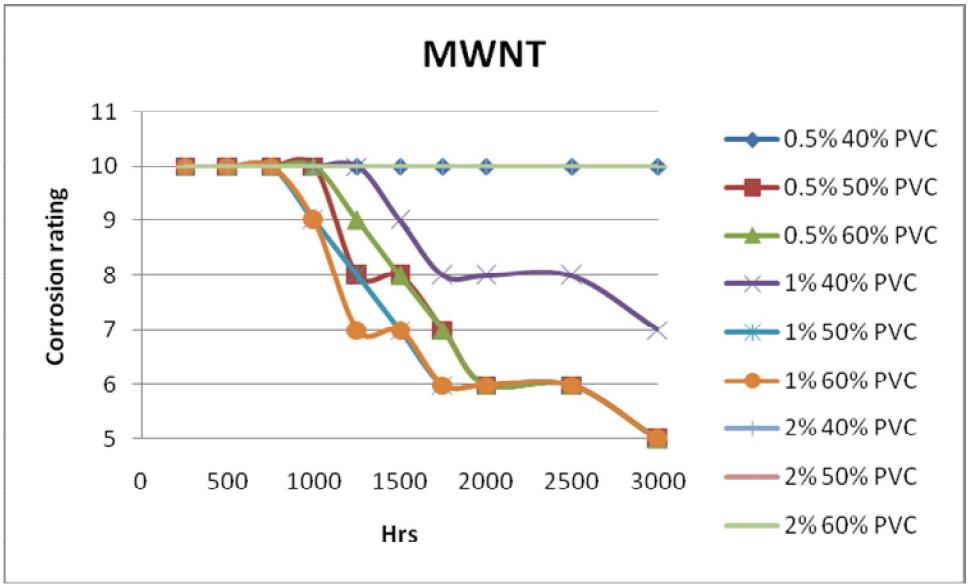
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.5 Corrosion rating for formulations based on nano talc



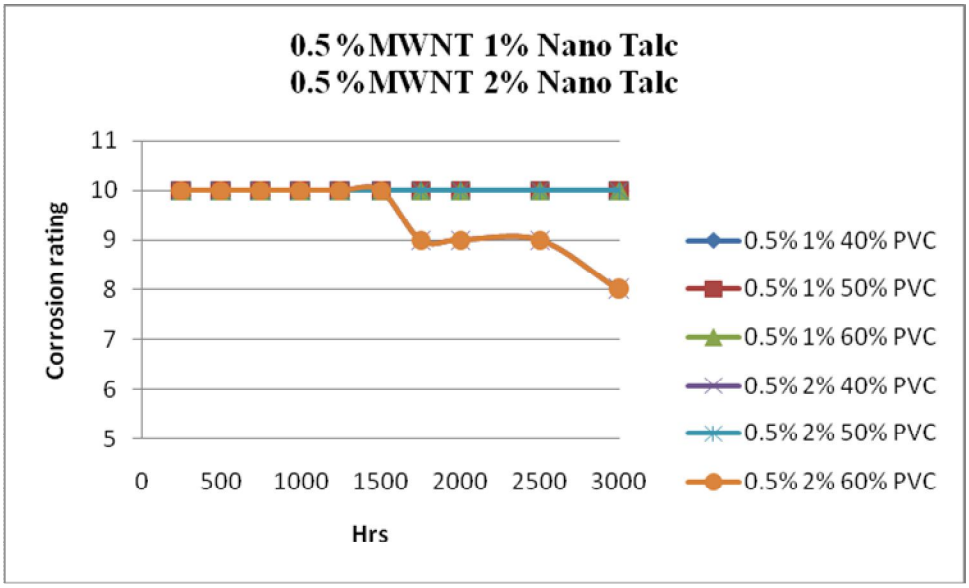
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.6 Corrosion rating for formulations based on MWNT



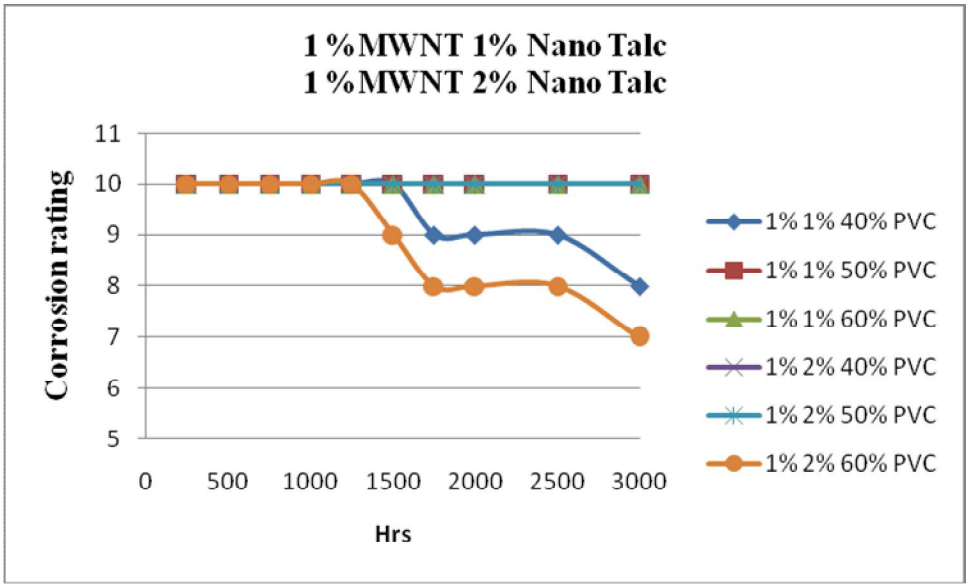
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.7 Corrosion rating for formulations based on MWNT & Nano Talc



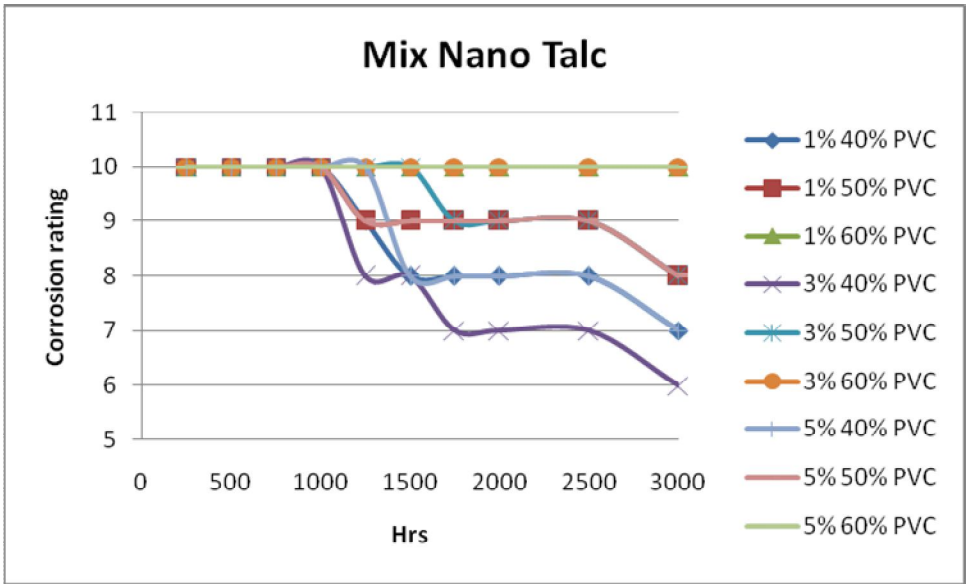
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.8 Corrosion rating for formulations based on MWNT & Nano Talc



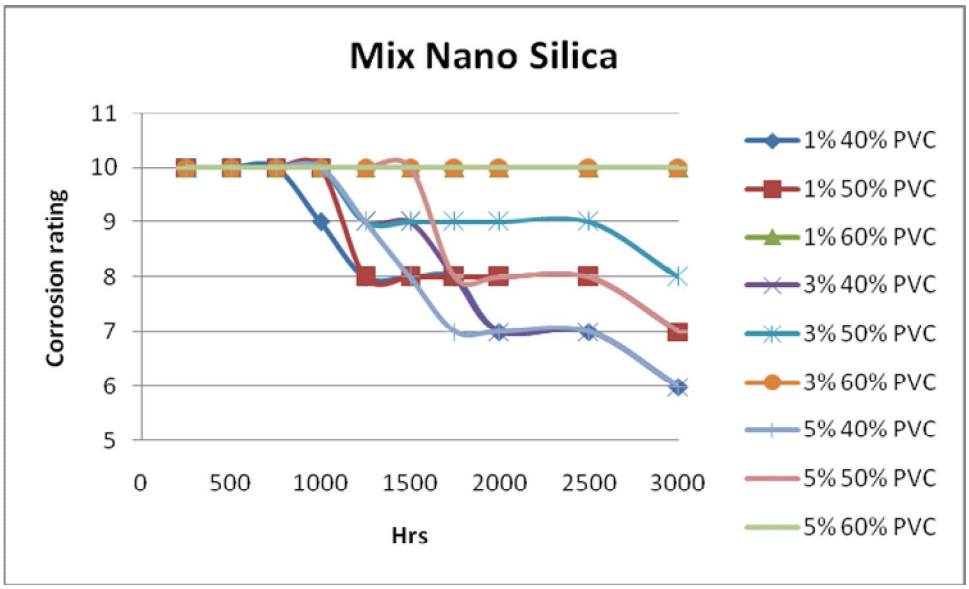
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.9 Corrosion rating for formulations based on Mix Nano Talc



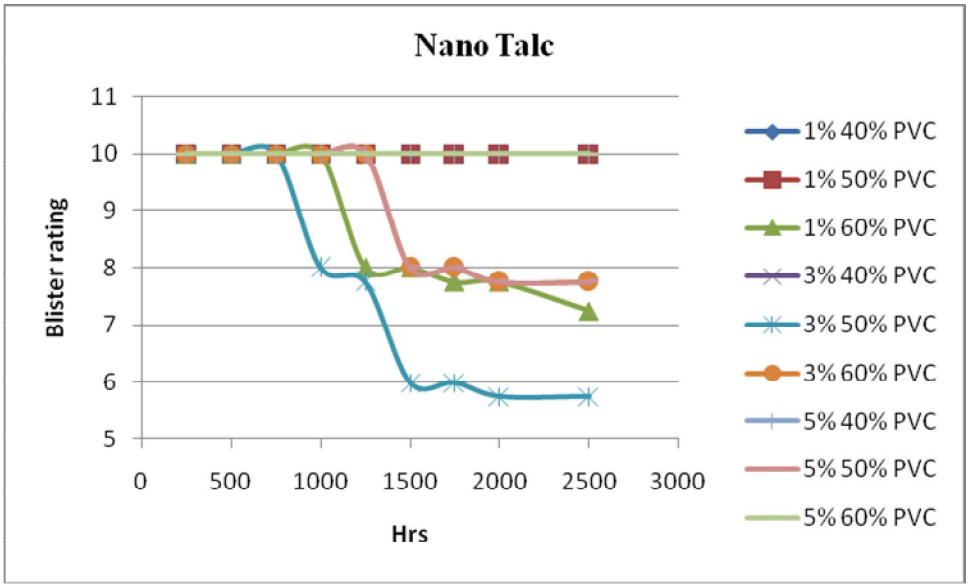
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No:- 4.10 Corrosion rating for formulations based on Mix Nano Silica



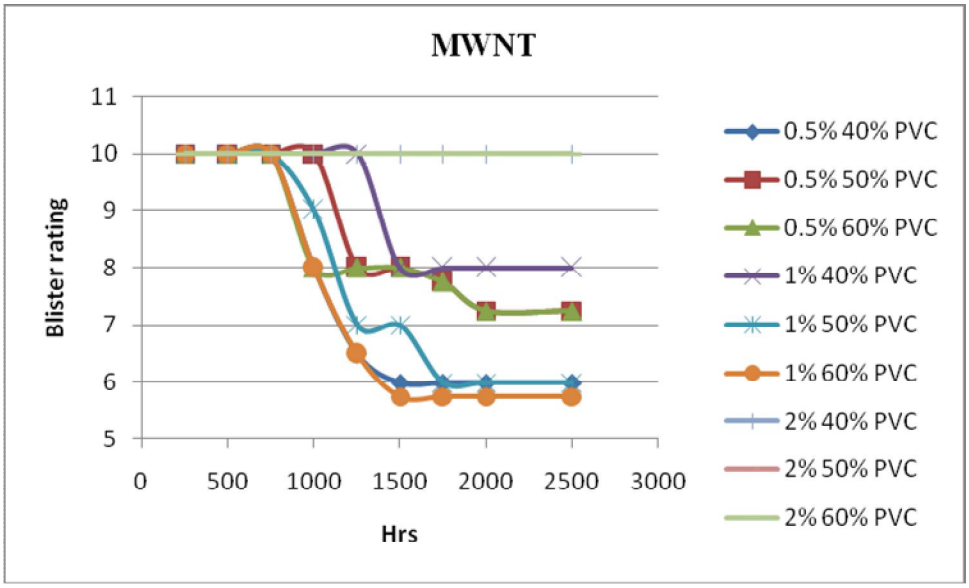
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.12 Blister rating for formulations based on Nano Talc



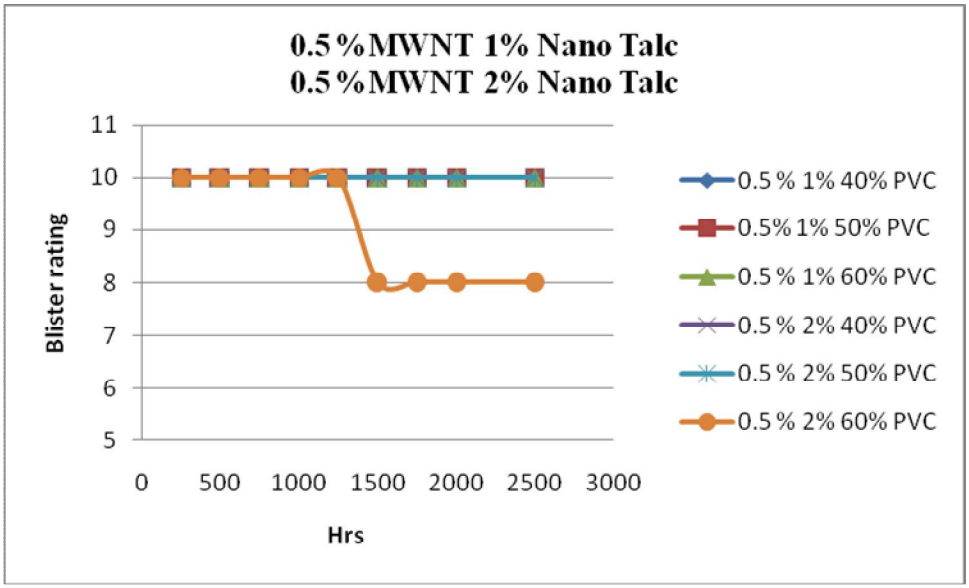
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.13 Blister rating for formulations based on MWNT



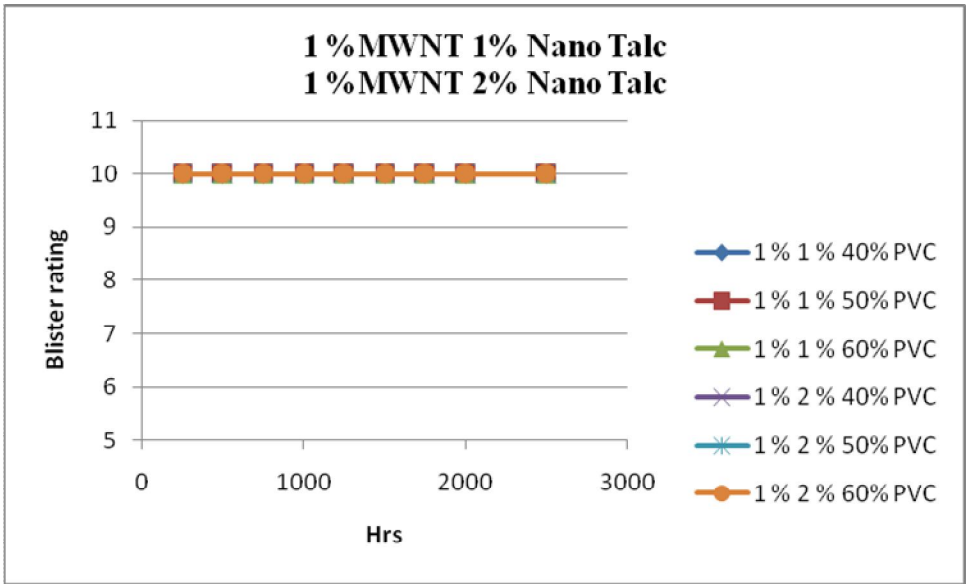
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.14 Blister rating for formulations based on MWNT and Nano Talc



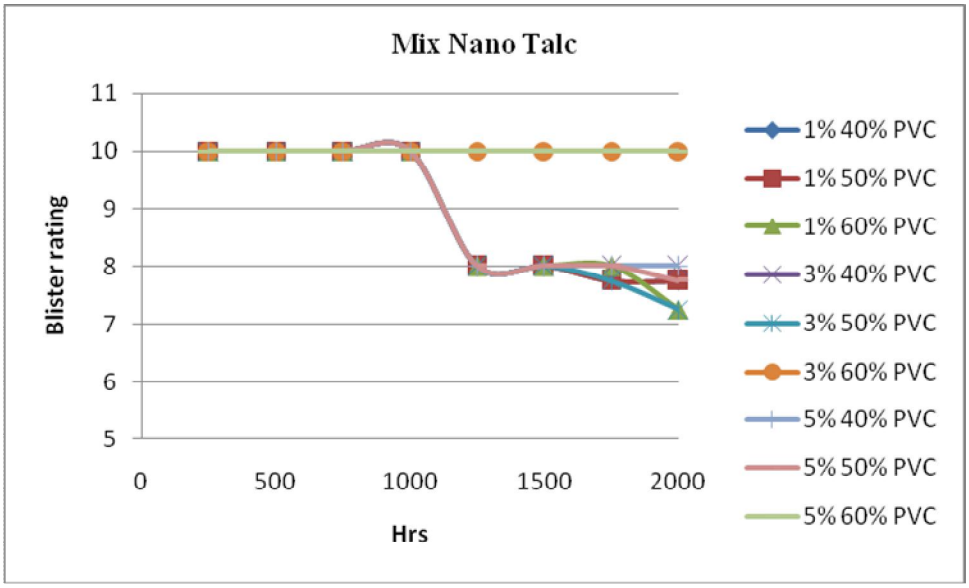
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.15 Blister rating for formulations based on MWNT and Nano Talc



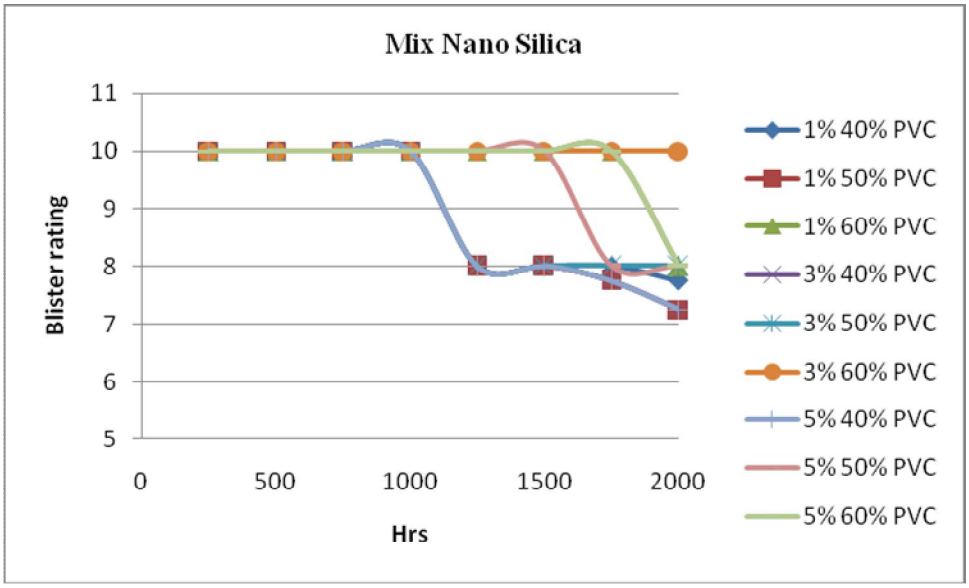
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.16 Blister rating for formulations based on Mix Nano Talc



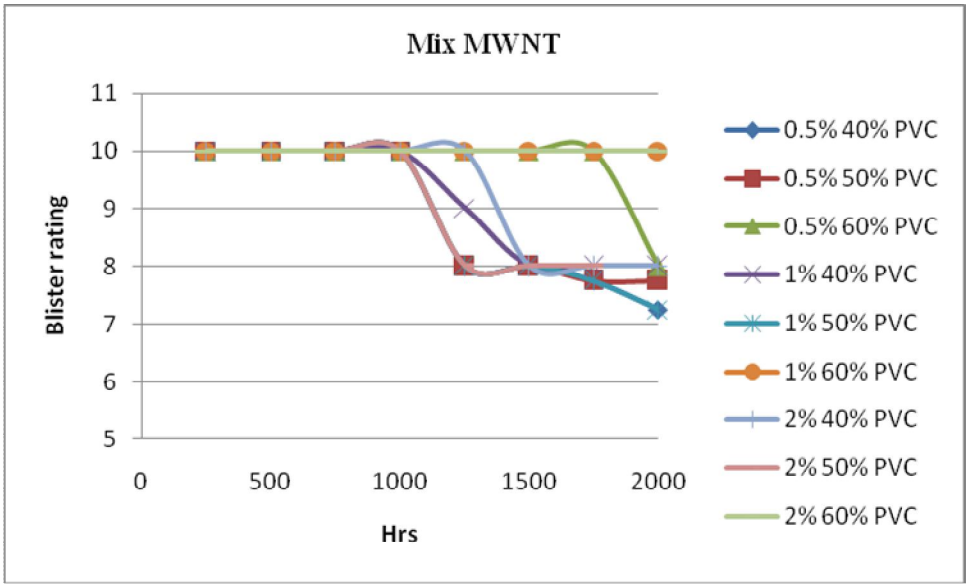
JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.17 Blister rating for formulations based on Mix Nano Silica



JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012

Figure No: -4.18 Blister rating for formulations based on Mix MWNT



JCSE The Journal of Corrosion Science and Engineering
ISSN 1466-8858 Volume 15, Preprint 21 submitted 13 April 2012