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Tetrahydro-dibenzo[a,d] Annulene-5, 11-Dihydrazone and Magnesium Oxide Used to Control the Corrosion of Aluminium in Chloride Ions Environment

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ABSTRACT

Chloride ions interact with aluminium in marine atmosphere to form corrosion cell. Due to this corrosion reaction occurs on their surface, aluminium is oxidized into Al^{3+} . The corrosion reaction accelerates deterioration in metal and it produces galvanic, pitting, stress, crevice, intergranular corrosion. Chloride ions decrease internal and external strength of aluminium metal. It is a very important metal so used in different appliances for e.g. road, water, air transports, housing, railways and other fields. Nanocoating and electrospray techniques used to check the corrosion of aluminium metal. For nanocoating and electrospray materials applied tetrahydro-dibenzo[a,d] [7] annulene-5, 11-dihydrazone and MgO. Both materials formed composite barrier and developed a passive surface for Cl^- ions. This barrier reduced the corrosion rate of aluminium. Nozzle spray and chemical vapour deposition technique used for coating process. The corrosion rate of metal was determined by gravimetric method. Corrosion potential and current density were calculated by potentiostat. The composite barrier formation was confirmed by activation energy, heat of adsorption, free energy, enthalpy and entropy. These thermal parameters were obtained by Arrhenius equation, Langmuir isotherm and Transition state equation. The adsorption of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electrospray on aluminium surface was depicted by Langmuir, Frundlich and Temkin isotherm. The results of surface coverage area and coating efficiency were noticed that both materials were mitigated the corrosion rate of aluminium in chloride ions environment.

1. Introduction

Metals produce their corroding process in presence of acids [1], bases [2], salts [3], humidity [4], heat [5], temperature [6], pollutants [7], effluents [8] and particulates [9]. They also corrode when their internal morphology [10] can be change. Metals are surrounded by these hostile environments to form corrosion cell [11] and create

dissolution on their surface. Corrosion chemists used several types of techniques to control the corrosion of materials like metallic [12] and nonmetallic coating [13] such coatings have done in acidic or basic medium. Cathodic protection [14] methods applied to protect metals corrosion in remote areas. Anodic protection [15] methods used in to control the corrosion of underground buried pipe. Various types of in-

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hibitors^[16] like organic and inorganic compounds provided ambient environment for corrosive substances. Organic compounds used as anodic, cathodic and mixed inhibitors^[17] as per nature of corrosive of medium. The affect of corrosion mitigated by change the shape and size of metals as surrounding environment. Polymeric lamination^[18] checked corrosion of materials in some corrosive environment. The attack of pollutants on materials protected by the application of paint coating. Conversion coating^[19] can be alter outer face of metal and produced passive layer against effluents or pollutants. Thermal coating^[20] can be done for metal which worked at high temperature. Top layer coating^[21] stopped corrosion reaction in acidic medium. The metals shaved by the utility of hot dip coating^[22], flame spraying^[23], cladding and chemical vapour deposition. Biological corrosion^[24] controlled by sparing of nitrogen and sulphur containing functional organic compounds. The aluminium corrosion suppressed by nanocoating of tetrahydro-dibenzo[a,d]^[7] annulene-5,11-dihydrazone such coating can be developed lot of porosities on the surface of base metal. Pollutants entered inside metal through diffusion or osmosis process and accelerate corrosion reaction. These porosities blocked by MgO by electro spraying method thus nanocoating and electro spray materials formed composite barrier that is worked as passive barrier for chloride ion environment. The coating and electro spray compounds concentration used 40 mM and 20 mM.

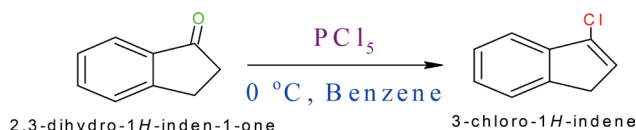
2. Experimental

Gravimetric method used to determination of corrosion rate: The sample of aluminium cut into (3X5X0.1) cm² and its outer layer rubbed with emery paper. It washed with acetone and dried with hot air gun. The corrosion rate of aluminium calculated at 283, 293, 303, 313 and 323 K temperature and these temperatures dipping duration of sample 10, 20, 30, 40 and 50 days in chloride ions solution. The sample kept into 10 M sodium chloride solution and the corrosion rates were determined above mentioned temperatures and days without coating and with coating nanocoating of tetrahydro-dibenzo[a,d]^[7] annulene-5,11-dihydrazone and MgO electro spray.

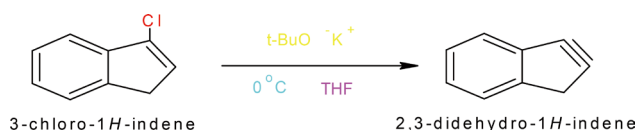
Potentiostat Techniques: Potentiostat 173 model EG & PG Princeton model used to obtain the results of corrosion electrode potential and corrosion current density. Aluminium metal kept between calomel electrode and Pt reference and these electrodes connected with wire thus external current is flowing. The corrosion electrode potential and current density recorded without coating and with coating tetrahydro-dibenzo[a,d]^[7] annulene-5,11-dihydrazone and MgO electro spray in chloride ions medium.

Coating compound tetrahydro-dibenzo[a,d]^[7] annu-

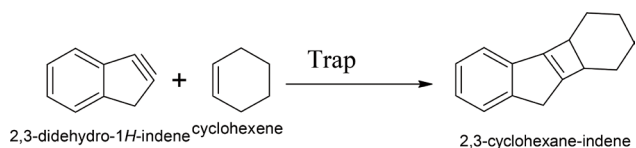
lene-5,11-dihydrazone synthesized by following methods: Indanone was added in cold solution of phosphorus pentachloride in presence of benzene solvent and the reaction mixture was stirred for 1hour. After work up the received yield was 87% of 3-chloro-1H-indene.



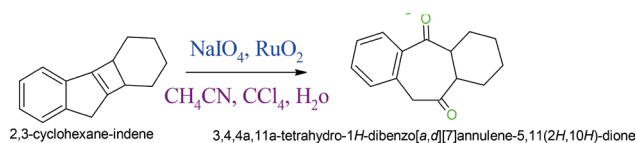
3-Chloro-1H-indene treated with potassium t-butoxide solution and during completion of reaction mixture temperature was maintained 0°C. The reaction mixture was stirred for two hours then cyclohexene was added which trapped 2, 3-didehydro-1H-indene and produced 2,3-cyclohexane-indene of 86% yield.



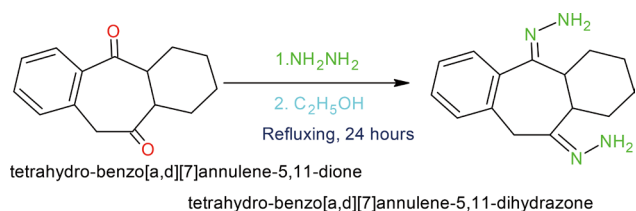
When 2,3-didehydro-1H-indene treated with cyclohexene, it is trapped with cyclohexene to give 2,3-cyclohexane-indene.



2,3-cyclohexane-indene was oxidized with NaIO₄ and RuO₂ in the presence of methyl nitrile, carbon tetrachloride and water to give tetrahydro-dibenzo[a,d]^[7] annulene-5,11-dione.



When tetrahydrodibenzo[a,d]^[7] annulene-5,11-dione was refluxed with hydrazine in presence of ethyl alcohol to give tetrahydrodibenzo[a,d]^[7] annulene-5,11-dihydrazone.



3. Results and Discussion

Aluminium comes in contact of chloride ions to produce galvanic, pitting, stress, crevice and intergranular

corrosion. Such types of corrosion were controlled by the nanocoating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and electro spray of MgO.

Tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO corrosion protection with time: The corrosion rate of aluminium was obtained without coating and coating with tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO by formula K_1 (mmpy) = $(13.56) W_M / D_M A_M t$ (where W = weight loss of metal in kg, A = Area of metal in square meter, D = Density of metal in kg/ m³, t =exposure time in hours) in chloride ions environment. The values of corrosion of aluminium were mentioned in Table 1. Figure 1 graph was plotted between K_M (corrosion rate) versus t (days) found to be straight line. The results of Table 1 and the plot of Figure 1 were shown that corrosion rate of aluminium increased without coating but their values decreased with nanocoating and electro spray of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO compounds. Polymeric-coated Al was nanocoated with tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone thus it developed protection barrier on its surface and on that barrier MgO electro spray used in this methods composite surface film generated. It stopped osmosis of diffusion process of chloride ions.

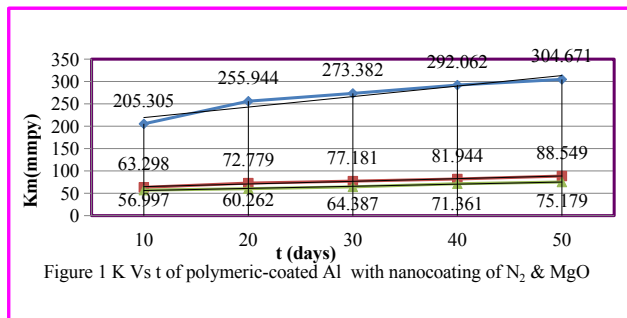


Figure 1 K_M vs t of polymeric-coated Al with nanocoating of N_2 & MgO

Aluminium corrosion rate without and with coating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO at different temperatures: The corrosion rate of aluminium was calculated at 283, 293, 303, 313 and 323⁰K temperatures in absence and presence of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electro spray. The recorded corrosion rate was written in Table1 which indicated that corrosion rate increased at lower to higher temperature but their values were reduced after coating of nanocoating and electro spray materials. Such trends were clearly observed in Figure 2 which was plotted between $\log K$ versus $1/T$.

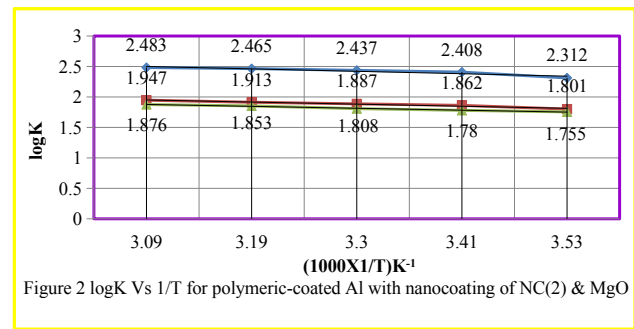


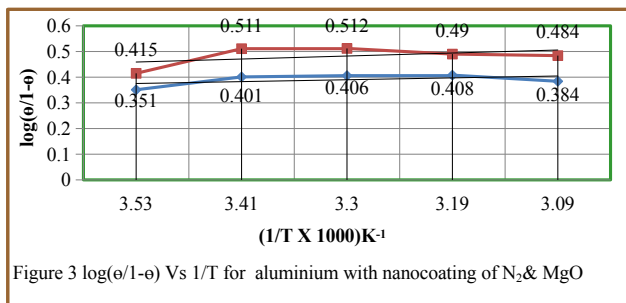
Figure 2 $\log K$ Vs $1/T$ for polymeric-coated Al with nanocoating of NC(2) & MgO

The values of $\log(\theta/1-\theta)$ for aluminium in tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO: The values of $\log(\theta/1-\theta)$ for nanocoating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO were given in table 4.13.1 at 283, 293, 303, 313 and 323⁰K temperature. It observed that their values were decreased as temperatures rising. In case with electro spray material got same types of results. Such types of trends clearly noticed in Figure 3 which was plotted between $\log(\theta/1-\theta)$ verse $1/T$. MgO dispersed into matrix of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihy-

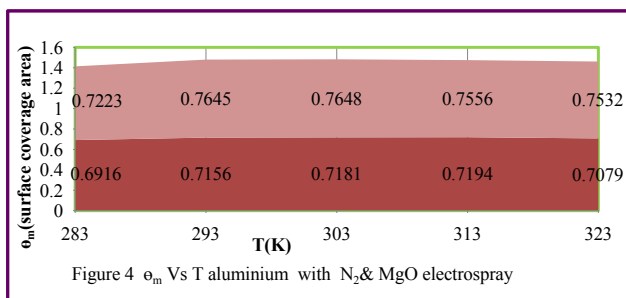
Table 1. Corrosion rate of aluminium without and with coating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electro spraying of MgO in chloride ions environment

N & E	Temp(K)	283K	293K	303K	313K	323K	C(mM)
	Time(days)	10	20	30	40	50	
No	K_{m0}	205.305	255.974	273.798	292.062	304.671	00
	$\log K_{m0}$	2.312	2.408	2.437	2.465	2.483	
N_2	K_m	63.298	72.779	77.181	81.944	88.549	40
	$\log K_m$	1.801	1.862	1.887	1.913	1.947	
	$\log(K_m/T)$	1.253	1.329	1.368	1.409	1.457	
	θ_m	0.6916	0.7156	0.7181	0.7194	0.7079	
	$(1-\theta_m)$	0.3084	0.2844	0.2819	0.2806	0.2921	
	$\log(\theta_m/1-\theta_m)$	0.351	0.401	0.406	0.408	0.384	
	% CE(m)	69.16	71.56	71.81	71.94	70.79	
MgO	K_m	56.997	60.262	64.387	71.361	75.179	20
	$\log K_m$	1.755	1.780	1.808	1.853	1.876	
	$\log(K_m/T)$	1.208	1.247	1.290	1.349	1.386	
	θ_m	0.7223	0.7645	0.7648	0.7556	0.7532	
	$(1-\theta_m)$	0.2777	0.2355	0.2352	0.2449	0.2468	
	$\log(\theta_m/1-\theta_m)$	0.415	0.511	0.512	0.490	0.484	
	%CE (m)	72.23	76.45	76.48	75.56	75.32	

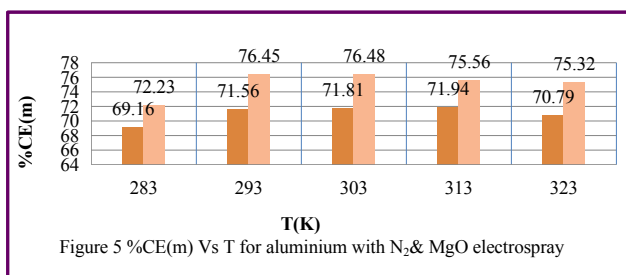
drazone and increased the values of $\log(\theta/1-\theta)$.



Surface coverage area on aluminium by tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO: Surface coverage area accommodated by tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electro-spray were calculated by equation, $\theta_m = (K_{m0} - K_m / K_{m0})$ and its values were recorded in Table1. Figure 4 plotted between θ_m (surface coverage area) versus T(temperature) indicated that electro-spray of MgO enhanced the capability of surface as temperature increased. Nanocoating and electro-spray compounds formed a stable barrier which blocked the osmosis or diffusion process of corrosive pollutants.



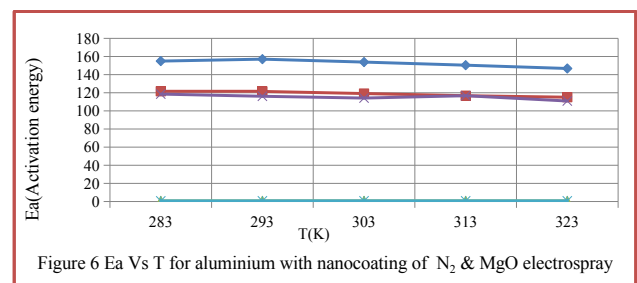
Percentage of coating efficiency of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO: The percentage coating efficiencies tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and electro-spray of MgO were determined by formula $\%CE = (K_{m0} - K_m / K_{m0}) \times 100$ and their values were given in Table1. It observed that percentage coating efficiency enhanced coating with tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone but it coated with MgO percentage coating efficiency developed more as shown in Figure5 which plotted between $\%CE(m)$ versus T (temperature). It noticed that at higher temperatures these compounds produced stable barrier.



Activation energies of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO: Activation energies of these compounds were calculated at different temperatures by the use of Arrhenius equation $\log K_m = \log A - E_{ma} / 2.303RT$ and Figure 2 plotted between $\log K_m$ versus $1/T$ and values were recorded in Table 2. Figure 6 graph plotted between E_{ma} (activation energy) versus T (temperature) noticed that activation energies of aluminium increased without coating after coating of tetrahydro-dibenzo [a,d] [7] annulene-5,11-dihydrazone and electro-spraying of MgO its values were decreased. These results were noticed that both compounds formed chemical bonding with base metal. The results of Table 2 were shown that activation energies decreased as temperature enhanced but surface coverage area increased.

Table 2. Thermal parameters of aluminium without and with coating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO in chloride ions environment

Thermal Parameters	283K	293K	303K	313K	323K
E_{ma}	156.126	157.081	153.845	150.425	146.774
E_{za}	121.619	121.464	119.124	116.740	115.091
q₂	-23.702	-26.158	-25.631	-24.898	-22.698
ΔG₂	-224.454	-220.778	-215.267	-209.678	-211.733
ΔH₂	-84.655	-86.711	-86.423	-86.028	-86.139
ΔS₂	-87.566	-90.173	-91.421	-92.696	-96.398
θ_{2m}	0.6916	0.7156	0.7181	0.7194	0.7079
E_a, MgO	118.513	116.115	114.137	116.740	110.893
q, MgO	-28.024	-33.334	-32.322	-29.902	-28.610
ΔG, MgO	-221.379	-215.432	-210.298	-206.013	-200.893
ΔH, MgO	-81.580	-81.364	-81.454	-82.363	-81.936
ΔS, MgO	-85.824	-87.036	-88.409	-90.400	-91.530
θ, MgO	0.7223	0.7645	0.7648	0.7556	0.7532



Heat of adsorption of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electro-spray: The heat of adsorption of both compounds were obtained by Langmuir equation $\log(\theta_m / 1 - \theta_m) = \log(A \cdot C) - (q_m / 2.303 R T)$ (where T is temperature in Kelvin and q_m heat of adsorption) and Figure3. The negative sign of heat of adsorption depicted that nanocoating and electro-spray compounds adsorbed by chemical bonding. The plot of Figure7 between q_m (heat of adsorption) verse T(temperature) with θ_m (surface coverage area) in-

indicated as temperatures increased surface coverage area also enhanced.

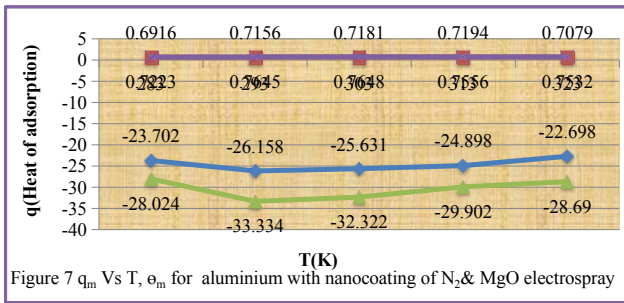


Figure 7 q_m Vs T_m for aluminium with nanocoating of N_2 & MgO electro-spray

Free energy of nanocoating tetrahydro-dibenzo[a,d] annulene-5,11-dihydrazone and MgO electro-spray: The values of free energies of nanocoating and electro-spray were calculated at various temperatures by formula $\Delta G_m = -2.303RT \log(33.3K_m)$ and their values were written in Table 2. It was observed that free energies of nanocoating and electro-spray materials decreased when temperature rose from lower to higher. Free energies of both compounds indicated that they were attached with aluminium by chemical bonding. Figure 8 plotted between ΔG_m (free energy) versus T (temperature), θ_m (surface coverage area) that graph was shown that free energies reduced and surface coverage area was enhanced.

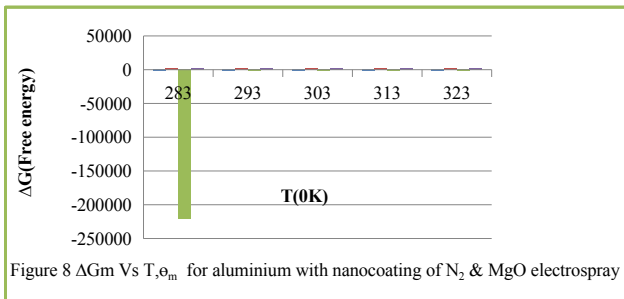


Figure 8 ΔG_m Vs T_m for aluminium with nanocoating of N_2 & MgO electro-spray

Enthalpy of nanocoating tetrahydro-dibenzo[a,d] annulene-5,11-dihydrazone and MgO electro-spray: Enthalpy of both compounds were determined by equation $K_m = R T / N h \log(\Delta S_m^\# / R) \times \log(-\Delta H_m^\# / R T)$ and Figure 9. Their values were recorded in Table 2. The results of Table 2 indicated that both compounds coating process were exothermic. Figure 9 the graph plotted between ΔH_m (enthalpy) versus T , θ_m for nanocoating and electro-spray indicated that enthalpy decreased at lower to higher temperature then surface coverage increased. It confirmed that both compounds were attached with aluminium by chemical bonding. Enthalpy of values nanocoating and electro-spray compounds were decreased so both compounds were formed a composite thin layer barrier. Enthalpy values found to be smaller with nanocoating and electro-spray and increased surface coverage area as shown in Figure 10.

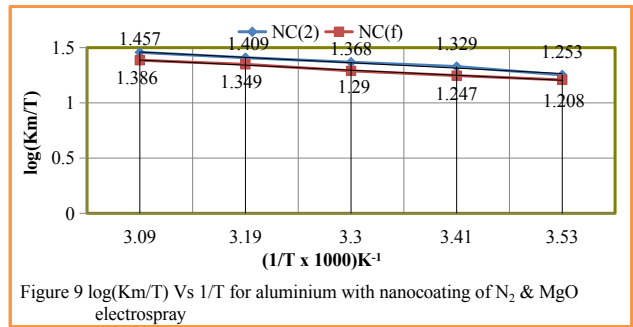


Figure 9 $\log(K_m/T)$ Vs $1/T$ for aluminium with nanocoating of N_2 & MgO electro-spray

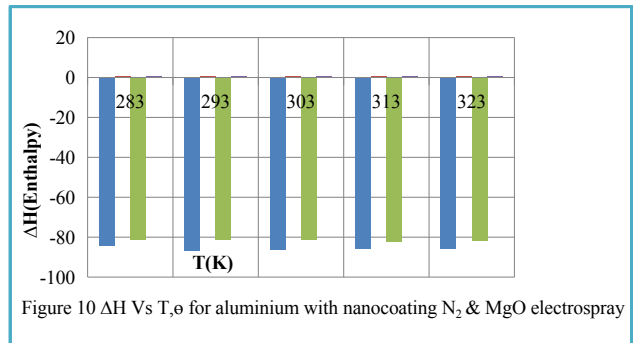


Figure 10 ΔH Vs T_m for aluminium with nanocoating N_2 & MgO electro-spray

Entropy of nanocoating tetrahydro-dibenzo[a,d] annulene-5,11-dihydrazone and MgO electro-spray: Entropy of nanocoating and electro-spray compounds were calculated by $K_m = R T / N h \log(\Delta S_m^\# / R) \times \log(-\Delta H_m^\# / R T)$ and their values were mentioned in Table 2. Both compounds produced negative entropy which depicted a chemical bonding occurred between nanocoating and electro-spray on aluminium metal. Figure 11 plotted between ΔS_m versus T , θ_m , it indicated entropy decreased then surface coverage area increased.

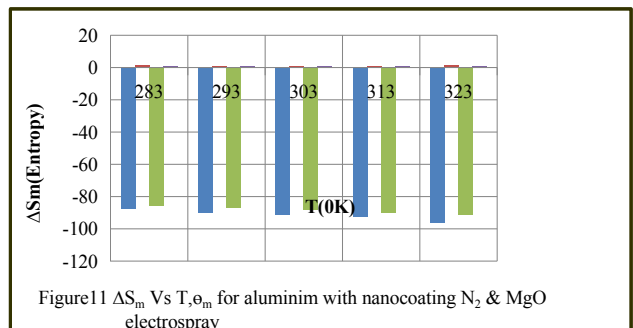


Figure 11 ΔS_m Vs T_m for aluminium with nanocoating N_2 & MgO electro-spray

Potentiostat of nanocoating tetrahydro-dibenzo[a,d] annulene-5,11-dihydrazone and MgO electro-spray: Electrode potential and corrosion current of aluminium, tetrahydro-dibenzo[a,d] annulene-5,11-dihydrazone and MgO electro-spray were calculated by equation $\Delta E_m / \Delta I_m = \beta_a \beta_c / 2.303 I(\beta_a + \beta_c)$ and Tafel plot of Figure 12 plotted between ΔE_m (electrode potential) versus I (current density) and their values were expressed in Table 3. These results were shown that electrode potential, corrosion current density and anodic polarization were enhanced without

coating and decreased cathodic polarization. Nanocoating and electrospray compounds reduced electrode potential, corrosion current density and anodic polarization and increased cathodic polarization. The corrosion current of aluminium reduced by nanocoating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electrospray. MgO electrospray increased more current density with respect of nanocoating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone. Both nanocoating and electrospray materials developed composite passive barrier on the surface of aluminium in chloride ions environment.

The corrosion current density of aluminium, nanocoating and electrospray were obtained by above mentioned equation and the values put in equation, $K_{cr} \text{ (mmpy)} = 0.1288 I \text{ (mA/cm}^2) \times Eq. \text{ Wt (g) / } \rho \text{ (g/cm}^3)$ to produce their corrosion rate. Their values were written in Table 3 which clarified that corrosion increased without coating of aluminium but it reduced with nanocoating and electrospray. Nanocoating tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone increased coating efficiency and surface coverage area where as these values were more enhance by MgO electrospray. The corrosion rates of materials were obtained by weight loss experiment which satisfied the corrosion rate of potentiostat measurement technique.

Table 3. Potentiostat of aluminium nanocoating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO electrospray

N & C	ΔE_m (mV)	ΔI_m	β_a	β_c	I (mA/cm ²)	K_{cr} (mmpy)	η_m	% CE	C (mM)
N(0)	-630	239	286	126	14.43	620.533	00	00	00
N ₂	-255	72	45	157	4.29	184.471	0.7027	70.27	30
EMgO	-221	62	40	167	3.94	169.420	0.7269	72.69	10

Mechanism of composite barrier formation: The organic compound tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone possessed electron rich functional group hydrazone and aromatic ring and they chelated with aluminium to form complex compound. This compound developed lots of porosities on their surface that is blocked by MgO electrospraying. MgO entered into matrix of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and formed a composite barrier. The composite barrier formation confirmed by the results of activation energy, heat of adsorption, free energy, enthalpy and entropy. The composite barrier of aluminium- tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone-MgO worked as noble barrier against chloride ions environment.

4. Conclusion

The results of corrosion rate of aluminium were obtained

by gravimetric and potentiostat techniques which indicated that corrosion rate and electrode potential increased without coating but their values were reduced by coating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and electrospraying of MgO. The surface coverage areas and percentage coating efficiencies produced by both compounds were shown that they had capability to adhere with aluminium. The coating of tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and electrospraying of MgO were an exothermic process. It confirmed by the values of free energy, enthalpy and entropy. Both compounds adsorbed on aluminium surface by chemical bonding to satisfy by the results of activation energy, heat of adsorption, free energy, enthalpy and entropy. The values of entropy indicated that both compounds were high stable on aluminium surface. The results of corrosion rate, surface coverage area, coating efficiency, corrosion potential, current density and thermal parameters values were depicted that tetrahydro-dibenzo[a,d] [7] annulene-5,11-dihydrazone and MgO were mitigated corroding effect of aluminium in chloride ions environment.

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