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Self-Healing Coating on Aluminium Alloy (AA2014) using a Sol - Gel Process

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Abstract: ***In this paper, a new doping concept has been improved which shows self-healing properties by sustained release of corrosion inhibitors in a carrier system. The anti-corrosive properties of (AA2014) aluminium alloy was tested by electrochemical impedance spectroscopy. The sol-gel coating doped with both inorganic and organic inhibitors gives a satisfying result. Releasing the event of inhibitor depends on the pH value of the corrosion environment. The barrier properties of the best coating divulge by high pore resistance with having high impedance value at low frequency. The immersion test confirmed that double doping concept is valuable also for long immersion test.***
Keywords: sol-gel, zeolite, corrosion inhibitor, immersion test and double doping.

I. INTRODUCTION

Self-healing coating are coating that keep possession of the internal credential to overhaul backed damaged by themselves or with some outside reaction Self-healing coating have various use its main use in automotive industry. For self-healing coating various process are used that is either filing agent in microcapsule or directly incorporate with substrate. The benefit of these type of coating is to intrinsically correct damage, increasing efficiency and preventing cost of material failure. A substrate metal under corrosive medium show degradation due to corrosion. Corrosion is defined as the destruction of metals or alloys with its environment which results in consumption or dissolution through chemical and electrochemical changes [1-6]. In USA according to 1998 data the loss of GDP is 260 billion dollars which is 3.1% of GDP. In India according to 2012 GDP data loss is 2 lakh crores which is 4-5% of GDP (of $2 trillion company). For the cost of corrosion two types of cost included which are direct cost and indirect cost [2]. Direct cost comprises of transportation, framework, applicability and manufacture and production of cost and indirect cost comprises of cost of labor attributed to corrosion management activity, loss of supply chain in revenue, loss of recall ability and cost of equipment [2]. When aluminium alloys placed in an aqueous environment, it can behave in three ways: either it corrodes, show immunity or passivate. In the case of the passivity potential of metal is raised as metal became covered with a protective film so that there will be no direct contact with the environment. The high corrosion resistance of aluminium alloys is due to the formation of a highly protective oxide film. The oxide film has a molecular volume 1.5 times the aluminium [7]. Aluminium alloy is subject to intergranular corrosion due to precipitation or formation of anodic and cathodic phases, concerning matrix it occurs at the grain boundary. Preferential attacks on grain boundary occur due to the formation of $CuAl_2$ .Among the different series of alloy 6xxxx series resist the type of intergranular attack because it contains silicon andMg_2Si approximately the same electrode potential as the matrix. In 7xxxx series alloy it does not contain Cu rather than it contain zinc and magnesium bearing constituents at the grain boundaries [7]. The result of passivation decreases high corrosion current. It requires so much time to evaluate the corrosion resistance of the aluminium alloy. The environmental factor such as temperature and solution turbulence can have a profound effect on corrosion resistance of the aluminium alloy. The deciding factor of aluminium alloy includes a surface to volume ratio, the heat capacity of the surface, alloying additions, and the presence of surface defects. We can protect aluminium alloy by anodizing process. It is an electrochemical method of converting aluminium into aluminium oxide by making aluminium anode in electrochemical method. The oxide layer is thin and non-porous at the metal/oxide interface but possesses a porous outer layer. Its classification done on use of acid electrolyte used. These acids anodized the surface to be sealed by the hydrating the aluminium oxide coating causing the coating to swell and closed the pore. It can be protected themselves by application of an organic topcoat. Most effective method is a Sol-gel method. Sol-gel coating is used for increasing adhesion on metal substrate. These coatings are prepared by a simple process, with less capital investment. Its use in mainly aerospace, decorative and building industry. The main problem we face is the delamination of the coating. It is a challenge for us to develop a coating that increases the adhesion of the coating to the substrate. By forming a dense coating, sol-gel acts as a film barrier for diffusion of aggressive species such as chloride, oxygen. It prohibits the entry of aggressive ions to enter inside. We can also incorporate inhibiting compounds, which provide another mechanism for corrosion protection. It can be prepared at room temperature.

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It is a diverse and complex system with addition it is considered a green method. In the sol-gel formation process, it is seen as phytic acid catalyzing process polysilsesquioxane [9] coating had excellent corrosion resistance performance. Instead of phytic acid if we use hydrochloric acid, acetic acid, and nitric acid, it will remain in the coating as a free molecule causing negative effects and negative influence on the corrosion-resistant properties of polysilsesquioxane. The main advantage is that phytic acid form strong bond and long network chain. It is type of colloidal system in which polymer network form by gelation of sol-gel. Substitution of alkoxide group (OR) by hydroxyl group (OH) called as hydrolysis reaction. First reaction is due to reaction of alkoxide molecule by water. The second reaction is the combination of two hydroxyl group to form (Metal-oxide-metal) network. Experimentally, rate of first reaction is more than second and third reaction. Homogeneity of reaction is achieved by the alcohol as metal alkoxide and water is not miscible. This aprotic solvent increase corrosion resistance. One of the advantages of sol-gel coating is that it has very lower porosity and enhanced mechanical properties. Phytic acid is used as an aprotic solvent, the long chain structure forming capability of phytic acid(C6H18O24P6) with central metal ions is one of the properties of its chelating effect. Phytic acid when react with GPTMS a ring will open of epoxy with hydrolysis and condensation of Methyltrimethoxysilane (MTES), tetraethyl orthosilicate (TEOS), GPTMS to form phytic acid long-chain polysilsesquioxane network. EIS experiment shows that phytic acid catalyzed coating network had better corrosion resistant. It is also seen that two much or two low phytic acid could dignify the anticorrosion property. Sometimes, two low TEOS also decrease corrosion resistant property by decreasing compactness of sols. The huge chelating network help in to stop active ions to enter through pores of substrate. As soon as sol-gel kept in environment it retains moisture which is only one disadvantage of this process. Which result in short period of the solutions, it is due to because viscosity and coating thickness varied with ambient temperature.

A. Sol-gel application [10]

Its use in preparation of Monoliths, powder, grains and spheres, fibers, Composites, porous gel, membrane, thin film coating and nanotechnology.

1) *Monoliths:* It is also called bulk gel. It is easy to form at room temperature. It can be kept at low temperature without melting. Its main application found in optic performance laser and graded refractive index glasses.
2) *Fibers:* Fibers are generated directly from viscous sols. Its potential application includes refractive textile, high temperature super conductors and reinforcement. Ceramic fibers are made from sol-gel.
3) *Powder Grains and Sphere:* Powder is the initiations point for polycrystalline ceramic processing scheme. The main advantage of sol-gel powder is low processing temperature. To make dense fibers reinforcement ceramic it is coated with ceramic slurries.
4) *Composites:* It means, it combines different type of material to obtain synergetic properties. By sol-gel process we can make different phases, fiber phases and reinforcement phases.
5) *Nanotechnology:* For formation of nanostructures material, including nanostructures, nano powders, nanocomposites. Nanocomposites will produce and offer excellent properties. Nano powders have broad application in fuel cells, supercapacitor, and semiconductor due to low k value.

II. EXPERIMENTAL

A. Sample Preparation

Before any application AA2014 alloy abraded with sandpaper of ISO/FEPA grit designation P300 to P1500 in increasing order and degreased with acetone. This process removed a thin oxide layer on a sample. Aluminium samples are known to undergo oxidation forming a thin layer of aluminium oxide on surface hence it needs to be removed before coating. Two process was used degreasing and activation also called as acid cleaning. Acid cleaning processes are usually based on sulphuric acid(H2SO4), phosphoric acid(H3PO4). However, hydrofluoric acid use as an activator. Degreasing solution contains the following composition [12].

Table no I. Degreasing solution composition.

H_2SO_4	3.5ml
H_3PO_4	5.0ml
HF	1.5ml
Triton X 100	0.75ml
H_2O	250ml

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Table no II. Activation solution composition

HNO_3	5.2ml
H_3PO_4	4.2ml
H_2SO_4	0.72ml
H_2O	250ml

After each treatment sample should be washed with running tap water and distilled water. Further, it treated with a Ti-Zr-Va(titanium, zirconium and vanadium) solution for 50 seconds. It composed of tannic acid that increases corrosion resistance. Solution pH is maintained between 2.8 to 4 otherwise it will form black patches. Hexafluoride ions remove the oxide layer on alloy surface [3]. Triton x-100 is a nonionic surfactant that is used for the removal of hydrophobic materials [13]. It is used to remove wax stains and hydrophobic material. The time for degreasing solution treatment is 3 minutes.

Table III: Ti-Zr-Va solution composition [11]

H_2ZrF_6	0.5 ml
H_2TiF_6	1.25 ml
$NaVO_3$	0.75 gm
Tannic acid	0.5 gm
H_2O	250 ml

B. Preparation of sol-gel coating [05]

Sol-gel coating prepared with adding undoped zeolite, cerium doped zeolite and 8-Hydrxoquinoline doped zeolite in a sol-gel solution. The amount of doped and undoped solid samples is 1 weight % (say 0.35 gm in 35 ml of sol-gel solution) and then mixed at high rpm for 10 minutes. Sol-gel solution compromises of two solutions, solution one and two which contains the following quantities:

Table 1V. Solution no 1

3-glycidoxypropytrimethoxysilane (GPTMS)	0.5 ml
Phytic acid	0.5 ml
Ethyl alcohol	23 ml

Table V. Solution no2

Phenyl tri methoxy silane	35 ml
Tetra ethyl ortho silicate	2.2 ml
H_2O	10.8 ml
Ethyl alcohol	11.7 ml

We have to maintain the temperature of solution 1 at 70 degrees Celsius for one hour and after that add solution 2 to solution 1 for 20 minutes dropwise.

C. Preparation of inhibiter(inorganic) doped zeolites

The exchange process of ion occurs between resin zeolite in inhibitor solution (100 ml) of 0.1M cerium nitrate hexahydrate inside a suitable flask with a speed of 300 rpm maintaining a temperature of 70◦C for 1 hour. The doped concentration of inhibitor was calculated by UV-vis (Perkin Elmer lambda 750) is 596 mg/gm of NaY-Ce. It is seen that 0.1M cerium nitrate hexahydrate has the maximum ability of exchange which is approximately 60 %. After proper mixing inhibitor doped solutions where vacuum filtered using Whatman® (125mm Ø) cat no 1442 125. The filtrate that we get is use for calculating doping concentration. The inhibitor doped material firstly washed by water five time and then left for drying in vacuum oven for one day.

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D. Process of doping of organic inhibitor zeolite

The Second doping process performed by the same sequence as above. We will make a 0.15M solution of 8HQ(8-hydroxyquinoline) in 100 ml of alcohol and stir it slowly for 2 hours. This experiment was performed at ambient temperature. 2gm initially prepared Zeolite doped cerium sample mixed with 50 ml of 8-HQ (0.15 M solution) and 2 gm zeolite sample mixed with 50 ml of the above solution.

E. Single dip-coating technique

In this process, the substrate is dipped in solution with constant rate. The substrate remains inside the solution for some time and started to pull off. The thin layer deposits on a substrate, withdrawing of the substrate are carried out at a constant rate to avoid any jitters. It is seen that the higher the withdrawal rate higher the thickness of the coating. This coating technique gives uniform, high-quality films even on bulky, complex shapes. After a single dip coating coated sample dried in ambient temperature for 48 hours and then dried in a vacuum oven at 50 degrees Celsius for 6 hours.

F. Corrosion Testing Procedure

Corrosion parameter is generally tested by Poteionstat. It is the electronic hardware required to control a three-electrode cell. It maintains the potential of the working electrode at a constant level concerning the reference electrode by adjusting the current at an auxiliary electrode. A potentiates a control and measuring device. It comprises an electric current which controls potential across the cell by sensing changing in its resistance, varying accordingly the current supplied to the system. A higher resistance will result decrease in current, while lower resistance will result in an increased current to keep voltage constant [8]. A potentiostat/Galvano stat with a servo control voltage stabilizer connected to the cylindrical cage-like electrochemical cell to not disrupt from any outside effect. This electrochemical setup was used for the potentiodynamic polarization test (PPT) and electrochemical impedance spectroscopy (EIS) test. For arranging the setup of the experiment AA2014 substrate was clamped vertically by clamper which works as a working electrode. The experimental area of the working electrode is approximately 1.0 cm^2, the remaining area should be covered with wax. The wax layer on the substrate is such that it does not disrupt current transfer from instrument to clamp. The uncoated surface of the substrate should be properly scratched for non-hindrance to current. The reference electrode used was the calomel electrode which was placed parallel to the working electrode and graphite gauze counter electrode placed at the side of the working electrode, no electrode should touch each other. Before putting the sample in the electrochemical cell, each sample scratched (5mm) by razor blade diagonally in the middle of the coated layer. It must be ensuring that scratched must be fine and reached the alloy surface. The AA2014 sample immersed in 3.5 weight % NaCl (300 ml) solution with inhibitor doped zeolite for different time intervals. After sample set for experiment open current potential test done for a time interval of 7200 seconds, further on achieving stability potentiostat EIS test performed. For the EIS test same setup require as OCP. The setup contains working (coated sample), reference (calomel electrode) and graphite gauze as the counter electrode. AC voltage kept at 10mv rms and dc voltage 0 volts. The frequency range started from 0.01 Hz to 10^5 Hz. Measured exposed area, equivalent weight, and density put in data before initializing the EIS test. Impedance measurement was performed for bare, single, and double doped samples. After performing the EIS test sample put in the test tube for immersion test using the same NaCl salt concentration. EIS test performed for several days. After 30 days, potentiodynamic test with a scan rate of 0.2(mv/s) performed. Using Tafel plot corrosion current measured so that we can compare it with the bare sample.

III. RELEASE KINETICS OF INHIBITORS FROM ZEOLITE (SODIUM ALUMINOSILICATE, NAY)

Release kinetics from zeolite is calculated by UV- visible spectrophotometry. Absorbance in zeolite is calculated and thereafter by linear calibration released inhibitor concentration plotted. The release profiles of Ce3+ concentration in solution in 0.61 molarity NaCl solution at different pH (3,7 and10) shown below. We know at less pH corrosion rate is triggering. The release of Ce3+ increases rapidly at early stage but after reaching chemical equilibrium Ce3+ release concentration became constant [15]. In the early stage, there is a fast release of Ce3+ followed by stabilization upon longer exposition in salt solution. At pH =10 no Ce3+ detected in solution which we can see from the graph its concentration decreases from 0.0235 mM to 0.0133 mmol. At pH =3 Ce3+ concentration increases from 0.5091 mM to 0.6159 mmol. On Comparing release of 8-HQ (0.9525 mmol to 1.1172 mmol) from NaY at lower pH=3 value it is higher than Ce3+leaching from zeolite, by increasing pH to 10 leaching effect increases from 2.3762 to 3.5444 mmol. Leaching percentage of 8-HQ with cerium ion is the same as 20% at pH =3, but on comparing the loading extent of 8-HQ it is on the higher side. This signifies that when corrosion rate advances after more no days,8-HQ shows it inhibiting property. An increase in chloride ion increases concentration 8-HQ in salt solution.

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In double doped zeolite structure release of the first inhibitor is fast while the second inhibitor release in a controlled manner, it depends on how severe the corrosion attack is, which we see from the graph analysis.

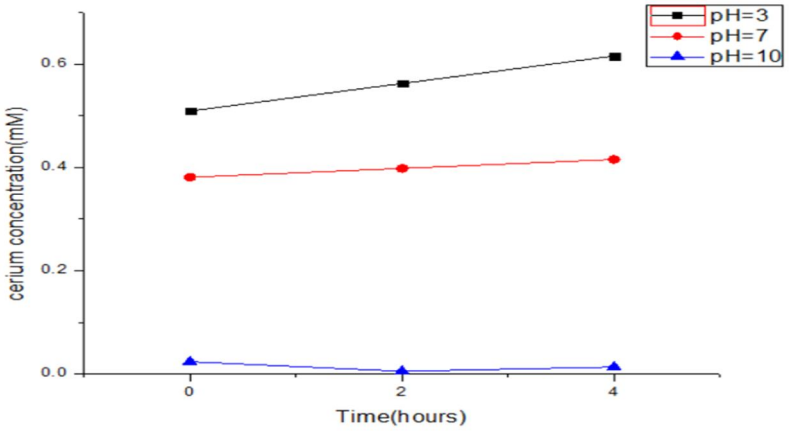

Figure: 1 Ce3+ release profiles at different pH in 0.61 M NaCl solution at 0,2 and 4 hours

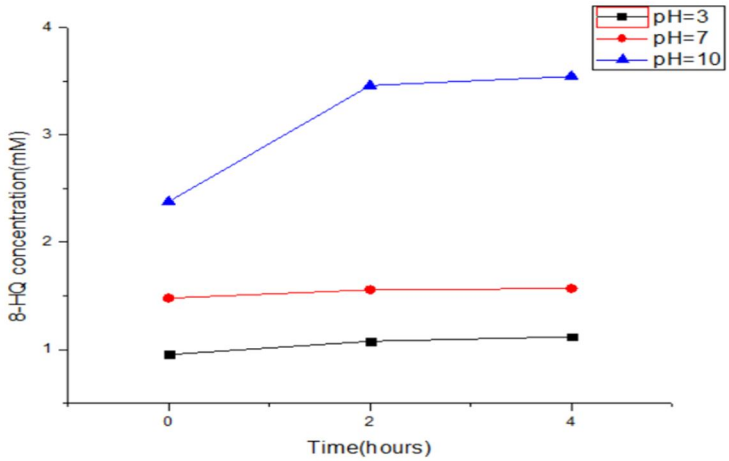

Figure 2 8-HQ release profiles at different pH in 0.61 M NaCl solution at 0,2 and 4 hours.

IV. CORROSION RESISTANCE CHARACTERISTICS OF SOL-GEL COATING SYSTEM

The final test was carried out so that the organic and inorganic doping method could be applied in a self-healing coating method concept, introducing doped zeolites in a sol-gel coating, and protecting a damaged site. Capacities of the coating were to be evaluated. The result is given for five systems bare sample, sol-gel coating, organic coating, inorganic coating, and both organic and inorganic coating. The graph is show as a distribution of impedance against immersion time for several type of coating and EIS tests were performed for the frequency range from 0.01 Hz to 10^5 Hz. Impedance is noted at low frequency of 0.1 Hz because in low-frequency range value of impedance is almost constant concerning high-frequency range where it varied rapidly. In this type of system, as presented in a diagram, if the change in impedance is going negative means that the constraint is reduced over time, if its slope is increasing means, it means that it is increased if it is zero, which signify that there is no change in impedance. It seems like bare sample impedance remains constant for the entire range of frequency after a sudden decrease, most important and Exciting results were obtained with 8 HQ NaY coated samples. This system shows an increase of impedance higher than another coated sample. Whenever the release of inhibitor seen from the carrier system slope increases. It is also seen that impedance variation of sol-gel undoped coating ultimately follows the same path as a bare sample. After a sudden decrement of impedance on the second day, inorganic sol-gel coating (NaY-Ce) shows more positive variation than others. Thus, it is seen that doped sol-gel coating impedance varied more positively than bare and sol-gel coating samples.

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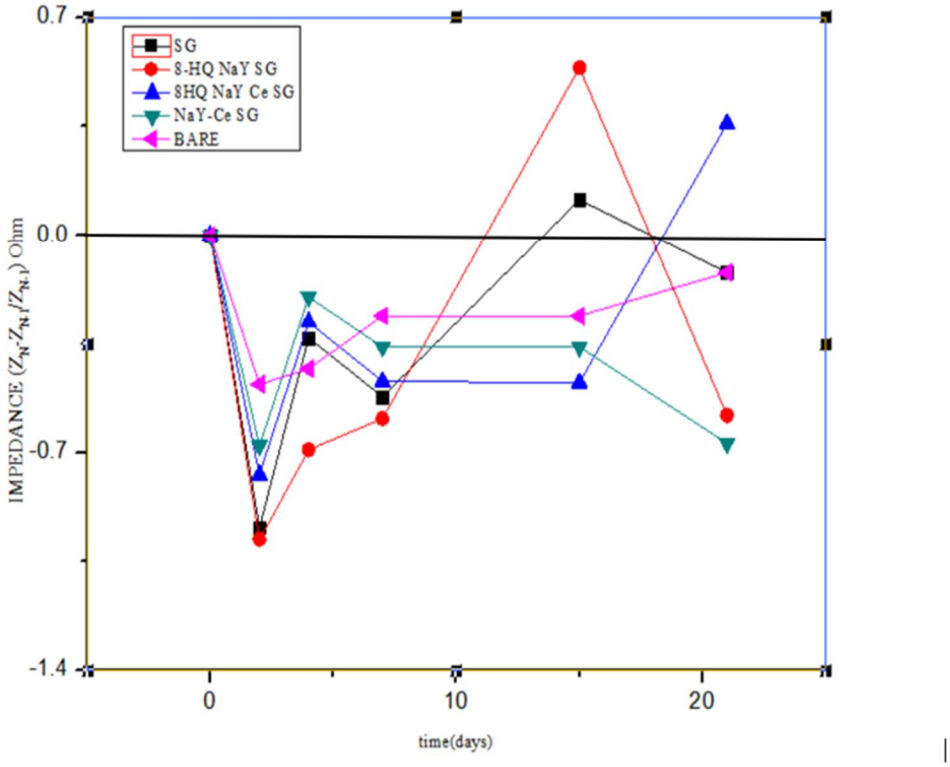

Figure 3 change of impedance by changing immersion time of different doped sol-gel dipped coated sample and bare sample at 0.1 Hz in 3.5 weight % NaCl solution. Positive variation of slope denote increase in impedance and release of inhibitor from carrier system.SG(Coated with sol-gel),8-HQ NaY SG(Coated with organic doped inhibitor),8 HQ NaY Ce SG(Coated with double doped inhibitor),NaY-Ce SG(Coated with inorganic doped inhibitor),Bare means uncoated sample.

V. RESULT AND DISCUSSION

A. Anti-corrosion properties

Aluminium alloy AA2014 sample was coated with sol-gel coating doped with cerium, 8-HQ and both using zeolite as a shape-selective catalyst. Organic and inorganic coating were consistently dispersed in sol-gel before a single dip-coating technique. It must be ensured that the coating is uniform without any accumulation of doped particles at any single point. Coating should be smooth and uniform. Electrochemical impedance tests performed to find the value and coating resistant properties and to know the corrosion-resistant properties of the sol-gel coating. The EIS test was performed in a 0.61 M NaCl solution. EIS test value collected at 1,2,4,7,15 and 21st day for comparison of corrosion resistant. According to circuit analysis, we achieve two coating capacitance one parallel with pore resistance (R coat) and another parallel with polarization resistance (R oxide). Coating capacitance is denoted by CPE coat and double layer capacitance is denoted by CPE oxide. Nyquist plot is used to find these values. It is seen that low impedance recorded for bare(pretreated) samples. We get detail analysis by fitting the equivalent circuit model. It is seen that the value of R coat decreases for each sample for 21 days of immersion mainly for bare pretreated samples. The value of C coat increases for sample which indicates more ion diffusion takes place with an indication of more porosity. R oxide value remains constant for double doped sample which indicates charge transfer resistance remains constant. It means that the double doping process increases corrosion resistance properties. It will stop ion diffusivity in sol-gel solution. An interesting result also saw in the table that except blank sample all other coating charge transfer resistance not decrease so much by comparing from 7th and 21st day. we compare C oxide and C coat; C coat value is more than C oxide thus it shows that more changes occur in the coating side. The constant phase element iconoclasm with capacitance function [17].

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Table no VI. Anticorrosive property

Sample	Immersion time(days)	R coat(Ωcm²)	CPE coat nf/cm²	α coat	R Oxide(kΩ/cm²)	CPE oxide(µf/cm²)	α oxide
S-G	1	1307	1930	0.65	166.3	92	0.9908
	7	1992	644.5	0.63	4.771	243	0.629
	21	1239	297000	0.8	4.099	346.1	0.8
8HQNAYSG	1	7493	6.598	0.8	2724	85.98	0.8
	7	69.41	76.23	0.551	4.046	227.7	0.5059
	21	34.49	53.62	0.9912	2.813	385.1	0.4836
8HQNAYCESG	1	1301	1059	0.5707	8115	446.5	0.8207
	7	5147	160400	0.5398	13.82	45.2	0.9573
	21	194.2	179200	0.3417	10.35	9.18	0.6373
NaYCESG	1	680.5	21510	0.5124	140.16	6.272	0.8347
	7	3443	128100	0.5636	30.82	27.06	1
	21	3451	9835	0.6037	5.784	150	0.616
Bare	1	3760	17180	0.8	5.784	59.16	0.8
	7	20.02	152800	0.3659	3.808	217.6	0.8875
	21	1.957	28900	0.774	1.841	432.2	0.7774

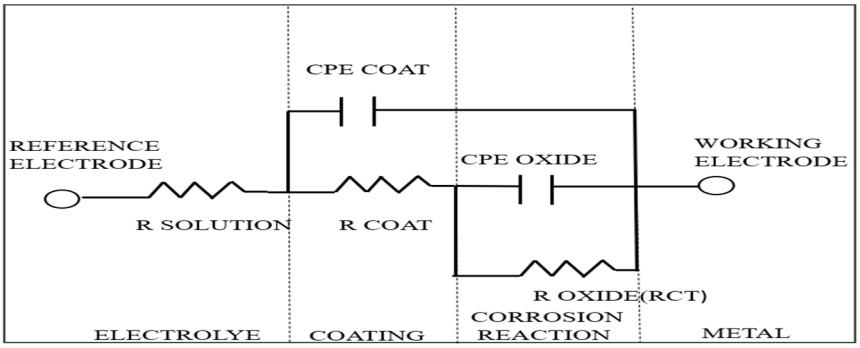

Figure:4 Equivalent circuit of organic and inorganic coating on metal substrate

B. Bode Plot Analysis

We get randles circuit for all samples, it means than all Nyquist plot have semicircular curve [15]. There is shift in phase angle of coated sample for all frequency range but for uncoated sample no such change occurs for all immersion time period. After 7th and 21st day shift seen for coated sample, it means charge transferring reaction occurs at surface of electrode [15-16]. Charge transfer reaction shift the phase angle to 0 for all coated samples. Randles circuit imply two time constant.

VI. CONCLUSION

This paper presented a controlled release of inhibitor in corroding environment in which double doping concept came into picture. This double doping concept is beneficial for both long and short immersion times. The Anti-corrosive properties improve by sol gel coating system doped with both organic and inorganic inhibitor. It is found that zeolite would be best shape selective carrier for doping cerium and 8-HQ and extend the application of other carrier and inhibitor for increasing corrosion resistant.

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VII. ACKNOWLEDGEMENT

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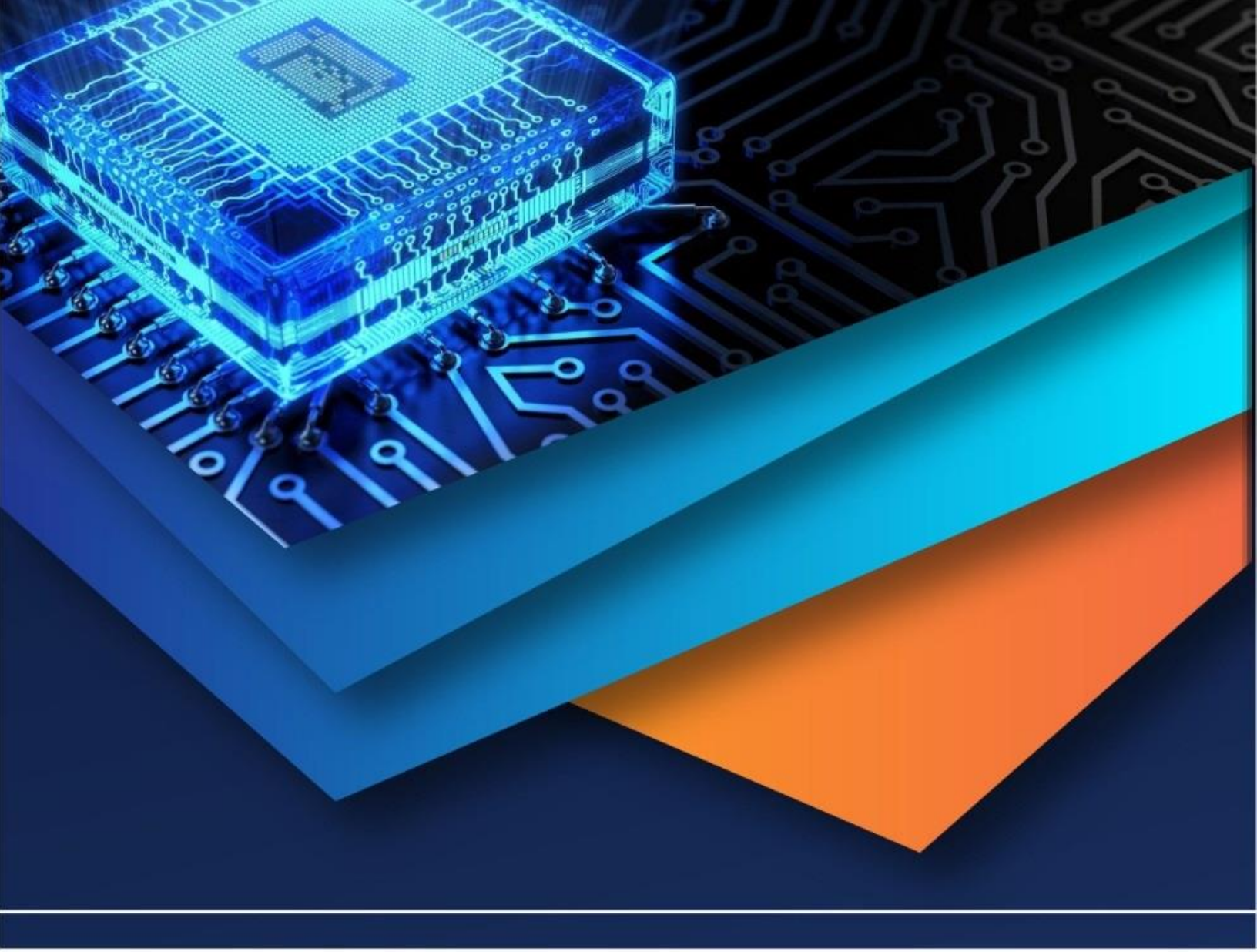

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