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Surface Analysis of Magnesium Alloy Corrosion in 3% NaCl Solution

S.Babu[1], A.Sivarajan[*1],R.Sudhakaran[1], T.Kasilingam[2], T.Deepa[3] and T.Asokan[1]
[1]PG and Research Department of Chemistry, Govt. Arts College, Tiruchirappalli-22.
(Affiliated to Bharathidasan University), Tamil Nadu, India
[2]Department of Chemistry, Sacred Heart College, Villupuram (Affiliated to Thiruvalluvar University), Tamil Nadu, India.
[3]PG and Research Department of Chemistry, Govt. Arts College (A), Karur (Affiliated to Bharathidasan University), Tamil Nadu, India
Email: sivarajan64@gmail.com

ABSTRACT
Corrosion protective performances of pectin were exploring as magnesium alloy corrosion in 3% NaCl solution. Impedance spectra and the measurements of contact angle for water. The studied inhibitor molecules on adsorbed magnesium alloy surface it's confirmed through Nyquist plots analysis due to increased charge transfer resistance and decreased double-layer capacitance. Water contact angle and Atomic force microscopy (AFM) analyses confirmed the reality of an inhibitive film of the inhibitor molecule onto the surface of magnesium alloy. The advantage lies with this study is pectin molecule's eco-friendly and the reduced corrosion rate in magnesium alloy in 3% NaCl solution.
KEYWORDS: Electrochemical analysis, Water contact angle, AFM, Magnesium Alloy, 3% NaCl medium.

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INTRODUCTION
Materials corrode with this electrochemical processes have colossal implication onto their end- use with consequently, economics of protection and repairs for the industrial applications. To control the corrosion effect, inhibitor is frequently added to corrosive medium during these processes. Primarily sulfur-, oxygen-, and nitrogen-containing compounds are reported in the literature as superior corrosion inhibitors [1–6]. In most of the cases, adsorption of inhibitor molecules on the metal surface is the root cause of corrosion inhibition. The adsorption of inhibitor on metal surface depends upon the nature in addition to surface charge of the metal, the structure of the inhibitor, the type of aggressive media, and the nature of its relations with the metal surface [7].
Magnesium alloys have numerous properties, like lightweight, dimensional stability, impact resistance, machinability and shock absorption[8]. Consequently, magnesium alloys are extensively used in a spacious range of fields, for example transportation, medical, electronics, military, etc.[9]and it is greater than ever gradually. Magnesium alloy pass on casting is the highly competitive automotive lightweight and lightest among all die-casting alloys material. An overprovision of magnesium alloy parts isformed to substitute plastic, steel parts and even aluminum alloy. In this article, the corrosion performance of magnesium alloy inhibition of corrosion by NaCl (3%) was studied through electrochemical study and surface analysis techniques.

MATERIALS AND METHODS
Materials
Specimens of size 1.0cm×4.0cm×0.1cm were press cut from the magnesium alloy sheet, were machined and abraded with a series of emery papers. This was followed by rinsing in acetone and bidistilled water and finally dried in air. Before any trial, the substrates were treated as depicted and newly utilized with no further stockpiling. A stock arrangement of 1000ppm of starch was ready in bidistilled water and the ideal focus was acquired by fitting weakening. The study was carried out at room temperature in open air atmosphere.

Electrochemical studies

Electrochemical impedance spectroscopic (EIS) studies were carried out via the model CHI-660A electrochemical workstation in addition to CHI- 760d as well as the experimental data were analyses by using the Version: 12.22.0.0 of electrochemical software. The measurement was recorded in a usual three electrode cylindrical glass cell through saturated calomel electrode as reference electrode and platinum electrode as auxiliary electrode.

The working electrode was magnesium alloy embedded in epoxy resin of polytetrafluoroethylene so that the flat surface of 1cm2 was the only surface exposed to the electrolyte. The three anodes set up was submerged in charge arrangement of volume 100ml both in the nonappearance and presence of the inhibitors details and permitted to accomplish a steady open circuit potential (OCP).

Electrochemical impedance spectra as Nyquist plots were recorded at OCP in the recurrence range from 60 KHz to 10MHz with 4 to 10 stages each decade. A sine wave, with 10mV adequacy, was utilized to bother the framework. The impedance parameters viz., charge transfer resistance (Rct),double layer capacitance (Cdl) were obtained from the Nyquist plots. The protection efficiencies (IEim) were calculated using the equation,

$$(\%)\ IE_{im} = \frac{R_{ct} - R'_{ct}}{R'_{ct}} \times 100$$

Where Rct and R'ct are the charge transfer resistance values in the presence and absence of the inhibitor respectively.

Atomic Force Microscope (AFM)

Atomic force microscopy isan extraordinary technique for the get-together of brutality estimations from a grouping of surfaces. These charming new methods that licenses surface to be imaged at more significant standards and exactnesses than any time in late memory. The cautious motion pictures are dissected for a separated district. AFM is transforming into a recognized system of obnoxiousness examination[10-13]. AFM given direct knowledge into the progressions in the surface morphology happens at a few hundred nanometers when geographical changes inferable from the inception of erosion and development of defensive movie onto the metal surface in the with and without expansion of inhibitors separately. All the AFM pictures were recorded on a Pico SPM2100 AFM instrument working in contact mode in air. The output size of all the AFM pictures is 10μm×10μm regions at a sweep pace of 0.20(Hz) lines each second.

Water Contact Angle

To assess the contact point tentatively, the sessile drop technique was utilized. The sessile drop laid on an even substrate by a needle.The substrate was illuminated by a light source, and then a picture was taken by using a high resolution camera (10.1 Mpixle SONY camera). The image was processed by computer by software made for this reason.

RESULTS AND DISCUSSION

Impedance Spectra

Nyquist plots for the corrosion inhibition of magnesium alloy immersed in 3% NaCl solution in the absence and presence of inhibitor are shown in Figure1. The impedance parameters, i.e., charge transfer resistance (Rct), double layer capacitance (Cdl) and inhibition efficiency (IEimp) calculated from the Nyquist plots are shown in Table 1.

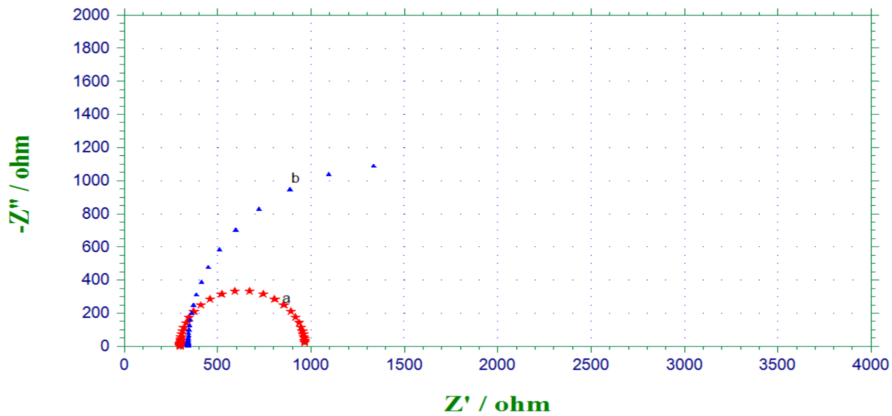
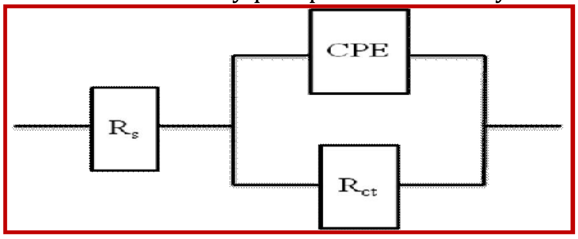


Figure 1. Nyquist plots of magnesium alloy a) 3% NaCl Solution (Blank) b) 150ppm pectin

Nyquist plots acquired are not ideal semicircles along with this distinction has been attributed to frequency dispersion due to roughness in homogenates of the metal surface [14]. Charge transfer resistance values are calculated from the difference in lower and higher frequencies, as suggested by Tsuru and Haruyma [15]. The double layer capacitance, the frequency at which the imaginary component of the impedance maximum, (-Zmax''), is found and Cdl values are obtained from the equation

$$f(-Z_{max}'') = \frac{1}{2\pi C_{dl} R_{ct}}$$

The experimental data obtained from Nyquist plots are fitted by the equivalent electrical circuit



Such an equivalent circuit was also discussed by several researchers who obtained similar depressed semicircles with a single time constant [16-18]. In this case, the constant phase element, CPE is introduced in the circuit instead of a pure double layer capacitor to give a more accurate fit [17].

In the blank, a small semicircle with Rct value of 348 Ω is observed. The value of Rct is increased and Cdl value is decreased with enhance in the value of inhibition efficiency, when 150 ppm pectin is added to the blank. These observations can be attributed to the presence of organic inhibitor in the double layer and control of the corrosion processes to some extent. When the addition of 150 ppm pectin with the blank a large depressed semicircle is observed from high frequency to low frequency directions in the Nyquist plot, indicating that the charge transfer resistance becomes dominant in the corrosion processes due to confirm the thin film on the magnesium alloy surface.

From Table1 the decreased in Cdl and increased in Rct values. The semicircle obtained in the presence of (150ppm pectin) represents an Rct value of 2820Ω, which is greater than that observed in the blank(Figure4.10). Higher value of Rct implies a lower Icorr and hence, lower corrosion rate [19]. The Cdl value at the metal /solution interface is found to decrease from 2.1789μF/cm2 in blank to 0.0406μF/cm2 in the presence of inhibitor. The high, Cdl value is indicated to increase in the surface area with the presence of corrosion product onto the metal surface [20]. It is well known that the capacitance is inversely proportional to the thickness of the double layer [21].

Decrease in the capacitance, which can result from a decrease in the local dielectric constant and or an increase in the thickness of the electrical double layer, strongly suggests that the inhibitor molecules are adsorbed at the metal/ solution interface [22].

The value of inhibition efficiency is considerably improved in the existence of the inhibitor system due to decrease inhomogeneity of the interface through inhibition process. These results indicated that there is formation of protective film in the presence inhibitor. The inhibition efficiency obtained from impedance studies is found to be 87%.

Table 1. Nyquist parameters of magnesium alloy in 3% NaCl solution in the absence and presence of inhibitor

Conc. of pectin (ppm)	Rct ohm.cm2	Cdl F.cm-2×10-6	% IEim
Blank	348	2.1789	-
150	2820	0.0406	87

ATOMIC FORCE MICROSCOPE

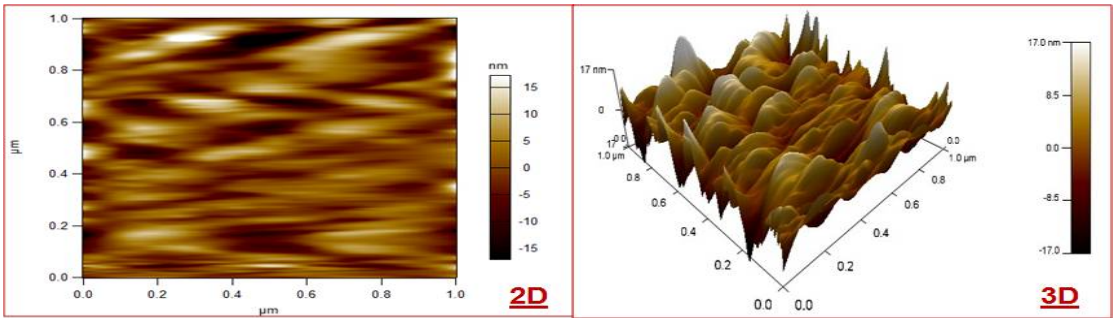


Figure 2. AFM images of magnesium alloy in polished metal

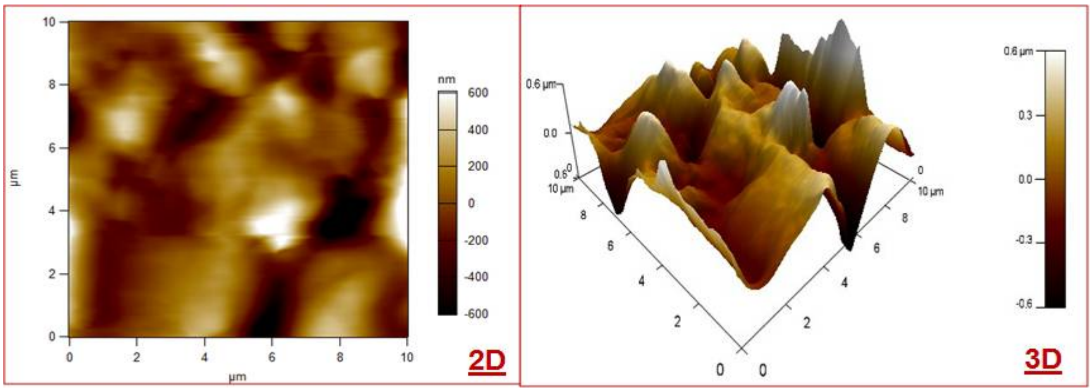


Figure 3. AFM images of magnesium alloy immersed in 3% NaCl Solutions

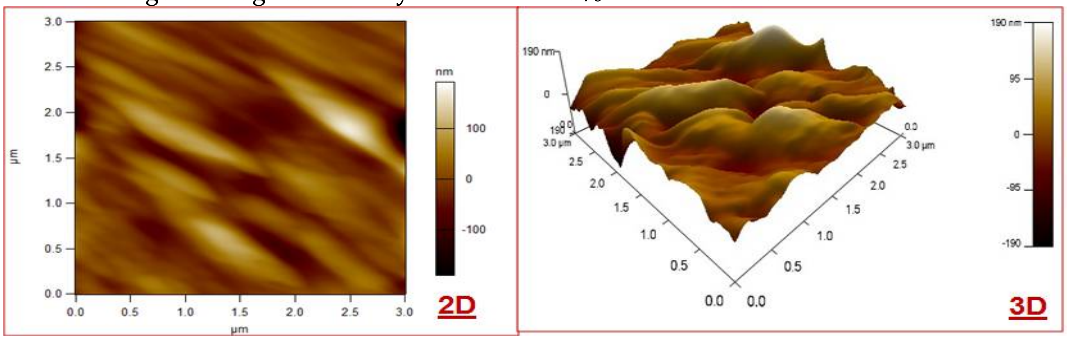


Figure 4. AFM images of magnesium alloy immersed in 150 ppm pectin (3% NaCl Solutions)

High resolution type of scanning probe and it is considered to one of the most powerful techniques to investigate surface morphology. Investigate the morphology of surface from nano scale to micro-scale as well as become a new preference to study the manipulation of inhibitors on the generation and the development of the corrosion at the interface of metal/solution[23]. The image of the surfaces recorded in 2D as well as 3D images was examined and surface roughness (RMS), average roughness (Ra), maximum peak to valley height were determined from the respective images. Table 2shows various AFM parameters acquired for the magnesium surface immersed in different environments.

Figure 3 is observed after immersion in 3% NaCl solution, with an increased Ra value 185.6 nm, RMS value 260.1 nm and maximum peak to valley height value of 615.0 nm, indicating the formation of corrosion product. The root-mean-square (RMS) roughness is found to be 260.1 nm, the high RMS value clearly indicates the high roughness of the corroded magnesium surface. The surface shows many smaller and larger corrosion product deposits. Figure 2shows the AFM images and cross section analysis of the polished metal surface, with an Ra value 94.1 nm, RMS value 138.1 nm and maximum peak to valley height value of 360.0 nm Which indicate the absence of corrosion product on the smooth surface and good surface properties compared to the blank.

Figure 4shows that the magnesium alloy immersed in 150ppm pectin showed a decreased Ra value 67.5 nm, RMS value of 102.7 nm and maximum peak to valley height is 160.1 nm, this value indicates the formation of a protective film on the surface.

RMS roughness from 260.1 nm in the blank to 102.7 nm observed in the inhibitor system, obviously infers the high homogeneity and smoothness of the surface film produced by the inhibitor. Further, these outcomes are affirmed by the obviously apparent contrasts among the optical cross area examination. The metal surface was covered with a defensive film, accordingly, framing an obstruction against assault by forceful particles from the destructive climate. With the expansion of inhibitor, the normal harshness was decreased to 67.5 nm, which recommended the film development of the inhibitor over the surface[24].

Table2. AFM data for magnesium alloy immersed in the presence and absence of inhibitor systems

Samples	RMS(Rq) Roughness (nm)	Average(Ra) Roughness (nm)	Maximum Peak – to – valley Height (nm)
3%NaCl solution (blank)	260.1	185.6	615.0
Polished magnesium alloy (Reference)	138.1	94.1	360.0
150ppm pectin	102.7	67.5	160.1

Water Contact Angle Technique

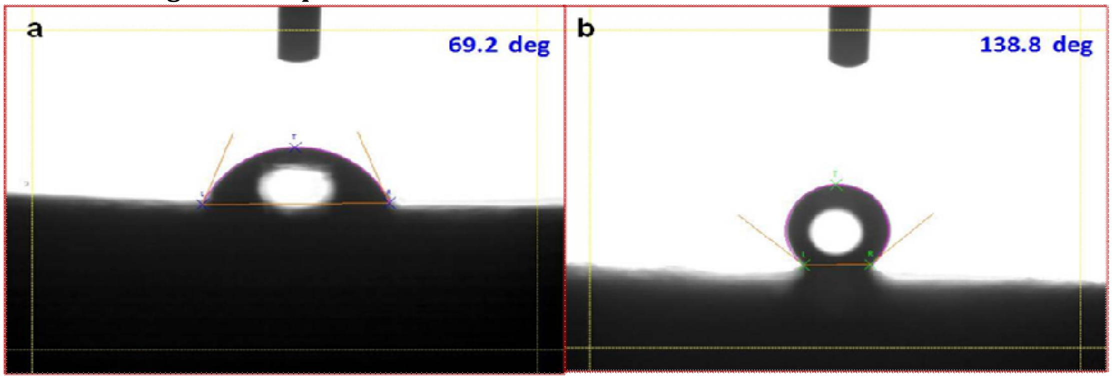


Figure5. Water contact angle image of magnesium alloy surface immersion of a) 3% NaCl solution b) 150ppm pectin

Nature of wettability of magnesium alloy measured through the water drop interacts on the metal surface whether it is a hydrophobic or hydrophilic. Figure 5a shows magnesium surface immersed in 3%NaCl solution, surface highly porous, more roughness (contact angle 69° ± 2°) due to magnesium surface get hydrophilic nature. Figure 5b shows magnesium surface immersed in 150ppm pectin, smoother surface appear (contact angle 138.80 ± 4°) beside the surface gets hydrophobic nature. This confirms the protective film onto the magnesium surface in the presence of inhibitor.

CONCLUSION

The formation of effective protective film on the magnesium alloy is surface which protects from corrosion in 3%NaCl solution. EIS studies showed the significant improvement of the magnesium alloy surface/solution interface through the formation of protective film in presence of the inhibitor. AFM analysis revealed the magnesium alloy surface covered with a protective film, thereby, forming a barrier against attack by aggressive ions from the corrosive environment and water contact angle studies corroborated the protective film formation on the metal surface of magnesium alloy and adsorbed surface film in super hydrophobicity nature.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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