Ability to Use of Robinia Pseudoacania L Fruit Extract as a Natural Corrosion Inhibitor in the Protection of Historical Bronze Objects

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Research article

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Abstract

Background: The phenomenon of bronze disease is considered as the most important factor in the destruction of bronze objects. Different methods have been proposed to cope with it. The most important inhibitors used in this regard are BTA and AMT. While these inhibitors control the corrosion, they are toxic and cancerous. In the ideal conditions, these inhibitors are able to slow down the activity of chlorine ion, but they leave some side effects after a period of treatment. Today, plant extracts are used for this purpose. In this study, *Robinia pseudoacania* L extract was selected for this purpose.

Material and methods: Natural inhibitor of Robinia fruit at concentrations of 200 ppm to 1800 ppm was evaluated in a corrosive solution of sodium chloride 0.5 M on a bronze alloy with a percentage similar to ancient alloys (Cu-10Sn) using potentiostat, weight loss method, and humidifier area.

Results: Given the data derived from potentiostate device showed that *Robinia pseudoacania* L Inhibitory power at 1000 ppm with corrosion rate of 12.78% is 55% and the classic method of weight loss inhibitory power after four week at 1800 ppm *Robinia pseudoacania* L in contrast a corrosive solution of sodium chloride 0.5 M is 92% for bronze alloy (Cu-10Sn). In addition, SEM images suggest that the formation of film on the coupon has been flacked.

Conclusion: While the results of the analyses suggest the inhibitory power of *Robinia pseudoacania* L, granular corrosion is evident on the coupons surfaces in SEM-EDX images and analysis.

Introduction

Inhibitors are generally substances, reducing the level of chemical reactions at appropriate concentrations. These substances can inhibit the growth of biological agents and stop the physiological processes. The word “inhibitor” is rooted in the Latin word of “inhibere”, which means to prevent, protect or preserve. The inhibitor at low concentrations in corrosive medium delays the corrosion of metals. These substances can be solid, liquid or gas, and used in closed, gaseous, and aqueous mediums [1, 2].

Much attention has been paid to use of corrosion inhibitors in protecting metal works [3-5]. Corrosion inhibitors in the form of non-soluble compounds at the metal surface can provide better stability for metal corrosion and using it is very common in protecting metal works. By forming a thin impermeable layer of the work, inhibitor compounds slow down the anodic and cathodic activities. This protecting method can be used as the latest and most commonly used solution to cope with bronze disease.

In investigating the effect of inhibitors on historical bronze works, it performed experiments on BTA inhibitor and Jajen Degsed [6, 7] and it was conducted studies on AMT [8, 9], which results were suggested the inhibitory power of these compounds on historical bronze alloys. While these inhibitors have high efficiency, they leave toxic and cancerous impacts on environmental factors. For this purpose, natural inhibitors such as honey, fig juice[10], the extract of salvia [11] and green tea extract [12] have been examined and evaluated in recent years.

It is related that The *Robinia pseudoacania* L fruit extract was used to anti-corrosion properties on steel in drinking water networks by potentiodynamic [13-15]. In this experiment, *Robinia pseudoacania* L fruit extract was used to evaluate the inhibitory effect on bronze alloy (Cu-10Sn).

The acacia plant, scientifically named *Robinia pseudoacacia* L (Fig. 1) from the *Papilionaceae* family, is one of the two-celled plants whose beautiful and ornamental flowers are cultivated by beekeepers to produce fragrant honey. Flowers also have soothing, stomach tonic, astringent and biliary properties[16]. A fast-growing, deciduous tree with a broad crown and leaves consisting of 11-23 dark green oval leaflets whose hanging clusters of white and pink fragrant flowers appear in mid-spring and early summer. The flowers resemble peas and their fragrance it spins in space. In the roots, bark, and seeds of the *Robinia pseudoacacia* L tree, there is a substance called Description Robin, and in the leaves and flowers, there is also a glucoside called Description Robinin. *Robinia pseudoacacia* L wood is hard and durable and peels late. For this reason, it is of industrial and commercial importance and is used to build columns and scaffolding mines, as well as to make sofas and chairs [16].

The general compounds of The *Robinia pseudoacania* L fruit extract contain natural sugars of ramenoz, arabinose, and galactose, as well as gluconic acid, 4 methoxygluconic and rubinin. In addition, some amounts of lignin and other substances such as calcium, magnesium, potassium, sodium are seen this compound [17, 18].

Methodology

Robinia pseudoacacia L fruit was obtained from Agricultural and Natural Resources Research and Training Center of Isfahan. *Robinia pseudoacacia* L fruit extract were achieved from Art University of Isfahan in 2019.

Fruit samples collected were dried on a clean cloth and ground under appropriate conditions. 30 g of the resulting powder was soaked in 100 cc of double distilled water and shaken on a shaker for 24 hours at room temperature. The obtained liquid was then passed through sterile filter paper and finally the extract and powder were separated. The remaining particles in the extract were separated using a refrigerated centrifuge (2500 rpm) at 4 ° C for 20 minutes. The extract was dewatered using a vacuum rotary device. The obtained extract was turned into powder and stored in a dark glass at a temperature of 4 ° C. During the experiment, dilutions of 200 to 1800 ppm were prepared from the extract[19].

In this paper, potentiostat device, SAMA 500 electro-analyzer system model (SAMA Research Center, Iran), was used to perform experiments to determine the inhibitory power of the *Robinia pseudoacania* L fruit. It included three electrodes, a platinum auxiliary electrode, a reference electrode of the saturated chloride mercury (calomel) and a bar working electrode [20, 21] with length of 7.5 cm and diameter of 0.73 cm with compound of Cu-10Sn). Then, it was polished with sandpaper with grades of 400 to 2200. To calibrate the device, the LSV Tafel plot technique was used. Additionally, the classical weight loss method, the humidifier area, and finally, SEM-EDX, manufacture by Philips Company of Netherlands, and the XL30 model were used to evaluate the surfaces engineering as well as inhibitory power of the *Robinia pseudoacania* L fruit [22].

Result And Discussion

Preparation of control solution

Sodium chloride Merck M 0.5 was used to make a control solution. This solution was poured into a special container at volume of 100 ml. After calibrating, the device begins to plot polarization curve. An important point in the curves plotted by this device is that device records one Ecorr at each time. These operations needed to be repeated for several tomes to be able to record the relatively fixed corrosion potential. In the polarization curve (Fig. 2), the corrosion potential of the control solution (Sodium chloride M 0.5) was recorded -243 mV

Fig. 2 Tafel polarization curve of sodium chloride solution 0.5 M

Preparation of the initial solution of *Robinia pseudoacania* L fruit

The considered *Robinia pseudoacania* L fruit was prepared from Isfahan Agricultural and Natural Resources Research Center. To prepare the mother solution, the sample was powdered in the oven. The powder was weighed to the desired size by means of a digital scale and the weighted samples were reached to the volume by distilled water. *Robinia pseudoacania* L solutions were prepared from 200 ppm to 2000 ppm for this test. Then, each ppm was separately mixed and treated with a corrosive solution of sodium chloride 0.5 M with pH=5.5, so that its corrosion power to be examined by potentiostat device [22]. To examine and test the exact corrosion rate, corrosion potential and inhibitory power of the considered solution at each specific ppm, it is necessary to repeat the experiment for several times (Table 1). To analyze some of them, charts with 1000 ppm to 1800 ppm compared to control solution are presented below. The corrosion potential of the control solution is -243 mV. Given the corrosion potential of the sample at the presence of the inhibitor solution to the positive values, -222 mV indicates a shift in the direction of 21 mV to positive values, suggesting the complexity of the inhibitory type (Fig. 3). In addition to change in potential of corrosion, a slight flow is seen in the anodic branch.

Fig. 3 Tafel polarization curve of *Robinia pseudoacania* L solution at 1000 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

The Tafel polarization is seen at 1200 ppm1of *Robinia pseudoacania* L solution at the presence of a sodium chloride 0.5 M, which has an inhibitory corrosion potential of -216 mV. Based on the control solution, inhibitor chart has a shift of direction to positive values (Fig. 4). In addition to change in the corrosion potential, the flow drop in both the anodic and cathodic branches is significant.

Fig. 4 Tafel polarization curve of *Robinia pseudoacania* L solution at 1200 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

The Tafel polarization is at 1400 ppm and the inhibitory solution corrosion potential is -216 mV, which compared to control solution, it has a shift of direction to positive values (Fig. 5). The corrosion has also has had slight drop in the anodic and cathodic branches.

Fig. 5 Tafel polarization curve of *Robinia pseudoacania* L solution at 1400 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

The *Robinia pseudoacania* L solution corrosion potential is -213 mV at 1600 ppm, which compared to corrosion solution, it show displacement of 30 MV (Fig. 6). A slight drop is also seen in the anodic branch.

Fig. 6 Tafel polarization curve of *Robinia pseudoacania* L solution at 1600 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

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The *Robinia pseudoacania* L solution corrosion potential is -213 mV at 1600 ppm, which compared to corrosion solution, it show displacement of 30 MV (Fig. 7). A slight drop is also seen in the anodic branch.

Fig. 7 Tafel polarization curve of *Robinia pseudoacania* L solution at 1800 ppm in the presence of a corrosive solution of sodium chloride 0.5 M

Calculating the corrosion efficiency using potentiostat device calculations

To obtain the inhibitory efficiency percentage, IE% is calculated based on equation (1), in which Icorr is density of the corrosion flow with inhibitory and I^0 is corrosion flow without inhibitory.

$$IE = \frac{I^0 corr - Icorr}{I^0 corr} \times 100 \quad (1)$$

Another method to calculate the IE% is using the equation 2, in which Rp is the resistance of polarization, calculated by using the following equation

$$\theta = 1 - \frac{Rp\ without\ inhibitor}{Rp\ with\ inhibitor} \times 100 \quad (2)$$

In these experiments, corrosion flow density, corrosion rate, and equivalent weight at the presence and absence of inhibitor were calculated by standard (ASTM, G 102-98) [23, 24].

To calculate the density of flow based on the following equation

$$icorr = \frac{Icorr}{A} \tag{3}$$

i_{corr}: corrosion flow density ($\mu A/cm^2$)

I_{corr}: corrosion flow (μA)

A= contact surface (cm^2)

Corrosion rate is calculated based on the following equation

$$CR = K1 \frac{i\ corr}{\rho} EW \tag{4}$$

CR= corrosion rate (mpy)

$K1 = 3.27 \times 10^{-3}$ (mm g/μA cm yr)

ρ= density (g/cm^3)

Potentiostat device data are calculated using the above equations and are presented in Table (1).

Table 1 Calculation of corrosion flow, corrosion potential, electrolyte resistance, flow density, cathodic and anodic slope coefficients, and corrosion rate of *Robinia pseudoacania* L fruit with a Potentiostat device

Concentration *Robinia pseudoacania* L (W/V)	$-E_{corr}$ (mv)	R_P (ohm)	B_a (v/dec)	B_c (v/dec)	*I corrosion (A)*	*i corrosion (A/cm²)*	*Corrosion rate (mpy)*
blank	243	800.5	0.0607	0.0668	$2.716*10^{-5}$	$6.497*10^{-5}$	28.381
200 ppm	211	1244	0.0612	0.0843	$1.748*10^{-5}$	$4.181*10^{-5}$	18.264
400 ppm	228	1315	0.0720	0.0791	$1.653*10^{-5}$	$3.955*10^{-5}$	17.276
600 ppm	214	1268	0.0618	0.0687	$1.714*10^{-5}$	$4.102*10^{-5}$	17.091
800 ppm	219	1508	0.0829	0.09	$1.442*10^{-5}$	$30449*10^{-5}$	15.066
1000 ppm	222	1765	0.0592	0.0720	$1.232*10^{-5}$	$2.947*10^{-5}$	12.873
1200 ppm	216	1573	0.0672	0.0959	$1.382*10^{-5}$	$3.306*10^{-5}$	14.441
1400 ppm	214	1734	0.0773	0.1087	$1.254*10^{-5}$	$2.99*10^{-5}$	13.1
1600 ppm	219	1218	0.0763	0.1044	$1.785*10^{-5}$	$4.27*10^{-5}$	18.652
1800 ppm	213	1029	0.1218	0.1171	$2.113*10^{-5}$	$5.054*10^{-5}$	22.077

Using the data derived from Potentiostat device, the inhibitory power of *Robinia pseudoacania* L solution was calculated in Table 2.

Table 2 *Robinia pseudoacania* L inhibitory percentage with different concentrations using Potentiostat device

Concentration *Robinia pseudoacania* L (W/V)	*i corrosion* (A/cm^2)	*IE %*
Blank	$6.497*10^{-5}$	-
200 ppm	$4.181*10^{-5}$	36
400 ppm	$3.955*10^{-5}$	41
600 ppm	$4.102*10^{-5}$	36.9
800 ppm	$3.449*10^{-5}$	47
1000 ppm	$2.947*10^{-5}$	55
1200 ppm	$3.306*10^{-5}$	50
1400 ppm	$2.99*10^{-5}$	54
1600 ppm	$4.24*10^{-5}$	35
1800 ppm	$5.054*10^{-5}$	23

The classic weight loss method

Weight loss method is the simplest method for studying corrosion inhibitors due to the lack of need for device (except for using the digital scale). In this method, the weight variations of the metal sample are calculated before and after exposure to the corrosive medium (in the absence and presence of inhibitor). The time for this experiment is long, but as results of this method are more real than those of the electrochemical method, it is still used [25, 26], which Wcorr is the weight loss of the sample in the presence of the inhibitor and W0 is the weight loss of the sample in the absence of the inhibitor, obtained by using equation (5).

$$IE = 1 - \frac{\Delta W\ inhibitor}{\Delta W\ blank} \times 100 \quad (5)$$

In order to perform the experiment using the classic method, the prepared electrodes were cut (Fig. 8A) with a percentage of (Cu-10Sn) as round coupons with a diameter of 0.73 cm and a thickness of 2 mm.

The coupons were polished using sandpaper with grades of 400, 800, and 2200. The coupons were then degreased in alcohol and rinsed in distilled water. The rinsed samples were heated at 80 ° C for one hour in an oven. Then, coupons were placed in a desiccator for one hour and finally the coupons were weighed to be immersed in *Robinia pseudoacania* L solution (Fig. 8B).

Fig. 8 prepared electrodes for cutting of coupons (A); Prepared coupons for immersion (B)

After one month of immersion in an inhibitory solution in the presence of a sodium-chloride 0.5 M corrosive medium, one of the coupons was removed from the control solution and *Robinia pseudoacania* L solution each week and the inhibitory power was calculated each week using equation 5. This action lasted 4 weeks on coupons. The results of the inhibitory power of the sample are presented in the tables 3 to 6 and fig. 9 to18.

Table 3Inhibitory percentage of *Robinia pseudoacania* L with different volumes in corrosive medium of Sodium chloride 0.5 M one week after immersion

Concentration *Robinia pseudoacania* L and 0.5 M NaCl (W/V)	W_0	W_{corr}	*IE %*
Blank	2.0173	2.0163	-
200 ppm	2.3110	2.3108	80
400 ppm	2.4968	2.4965	70
600 ppm	2.5848	2.5845	70
800 ppm	2.6743	2.6740	70
1000 ppm	2.2343	2.2349	90
1200 ppm	2.0018	2.0015	70
1400 ppm	2.6505	2.6500	50
1600 ppm	2.5841	2.5839	80
1800 ppm	2.0608	2.0607	90

Fig. 9 Weight loss level based on the concentration of 1800 ppm *Robinia pseudoacania* L after one week

Table 4 Inhibitory percentage of *Robinia pseudoacania* L in corrosive media of sodium chloride 0.5 M after two weeks of immersion

Concentration *Robinia pseudoacania* L and 0.5 M NaCl (W/V)	W_0	W_{corr}	*IE* %
Blank	2.0504	2.0469	-
200 ppm	2.0303	2.0297	83
400 ppm	2.5695	2.5684	69
600 ppm	2.5570	2.5557	63
800 ppm	2.5847	2.5828	46
1000 ppm	2.3056	2.3055	97
1200 ppm	2.1317	2.1314	91
1400 ppm	2.6040	2.6031	75
1600 ppm	2.8395	2.8392	91
1800 ppm	2.0985	2.0984	97

Fig. 10 Weight loss level based on 1800 ppm concentration of *Robinia pseudoacania* L after two weeks

Table 5 Inhibitory percentage of *Robinia pseudoacania* L in corrosive media of sodium chloride 0.5 M after three weeks of immersion

Concentration *Robinia pseudoacania* L and 0.5 M NaCl (W/V)	W_0	W_{corr}	*IE* %
Blank	2.0621	2.0577	-
200 ppm	2.3348	2.3333	66
400 ppm	3992.5	2.5377	50
600 ppm	2.4962	2.4943	57
800 ppm	2.6207	2.6173	23
1000 ppm	2.1186	2.1170	64
1200 ppm	2.3403	2.3390	71
1400 ppm	2.5824	2.5809	66
1600 ppm	2.6022	2.6011	75
1800 ppm	2.0587	2.0585	95

Fig. 11 Weight loss level based on 1800 ppm concentration of *Robinia pseudoacania* L after three weeks

Table 6 Inhibitory percentage of *Robinia pseudoacania* L in corrosive media of sodium chloride 0.5 M after four weeks of immersion

Concentration *Robinia pseudoacania* L and 0.5 M NaCl (W/V)	W_0	W_{corr}	*IE* %
Blank	2.1446	2.1395	-
200 ppm	2.1489	2.1465	55
400 ppm	2.5637	2.5598	27
600 ppm	2.5732	2.5690	21
800 ppm	2.5265	2.5230	34
1000 ppm	2.0030	2.0005	47
1200 ppm	2.5633	2.5618	72
1400 ppm	2.6731	2.6718	76
1600 ppm	2.4807	2.4802	90
1800 ppm	2.0847	2.0843	92

Fig. 12 Weight loss level based on 1800 ppm concentration of *Robinia pseudoacania* L after four weeks

Experiment in the humidity compartment

After preparing the coupons with a percentage of (Cu-10Sn), the coupons were completely polished using sandpaper with grades 400 to 2200 to create a completely smooth surface. Then, the coupons were rinsed with distilled water and degreased by alcohol. The samples were placed in an oven at 120 ° C for one hour. The coupons were immersed in *Robinia pseudoacania* L with concentrations of 1000 ppm for 24 and 48 hours. After removing the coupons, they were dried at room temperature for one hour and photographed to examine the change in appearance color on the coupon surfaces (Fig. 13 to 15). To accelerate the corrosion, the samples were transferred to the humidity compartment. Coupons were placed in a relative humidity of 95 ± 2 and a temperature of 25 to 30 ° C. The samples underwent sodium chloride 0.5 spray based on the standards of (ASTM, G85) and (ISO, 9227). Four weeks later, the samples were removed from the humidifier compartment and examined to evaluate the effect of the inhibitor on the coupon surfaces by using SEM-EDX device (Fig. 16 to 18).

Conclusions

Given the investigations on *Robinia pseudoacania* L fruit using potentiostat device, it was revealed that the data derived from this device showed that *Robinia pseudoacania* L fruit inhibitory power at 1000 ppm with a corrosion rate of 12.78% is 55% for bronze alloy with percentage of (Cu-10Sn) and has a mixed inhibitory effect. In the classic method of weight loss, in which the results are more real than those in electrochemical methods, the inhibitory power of *Robinia pseudoacania* L fruit was determined to be 92%. SEM images derived from the surface of coupons at the presence of *Robinia pseudoacania* L and a corrosive solution of sodium chloride 0.5 M suggest the formation of film on the samples. However, SEM images show a kind of segregation on the surface of coupons at the presence of a corrosive solution. Based on the experiments performed, it is necessary to add other natural compounds to this inhibitor for better efficiency so that appropriate and optimal conditions for this type of inhibitor can be defined.

Abbreviations

BTA: Benzotriazole

AMT: 5-ami- no-2-mercapto-1,3,4-thiadiazole

SEM-EDX: Scanning Electron Microscope-Energy Dispersive X-rays

LSV: Liner sweep voltammetry

ASTM: American Society for Testing and Materials

EDX: Energy Dispersive X-rays

EDS: Energy Dispersive Spectroscopy

PPM: Part Per Million

Declarations

Conflict of interest

All authors declare no conflict of interest exists.

Ethics approval and consent to participate

No human or animals were used in the present research.

Consent for publications

All authors read and approved the final manuscript for publication.

Availability of data and material

All the data is embedded in the manuscript.

Authors' contributions

V.P and B.F.Z.N. designed the research and wrote the paper. All authors read and approved the final manuscript.

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Figures

Figure 1

The characteristic of whole plant, Leaf and Fruit of Robinia pseudoacania L. (Photo: Vahid Pourzarghan)

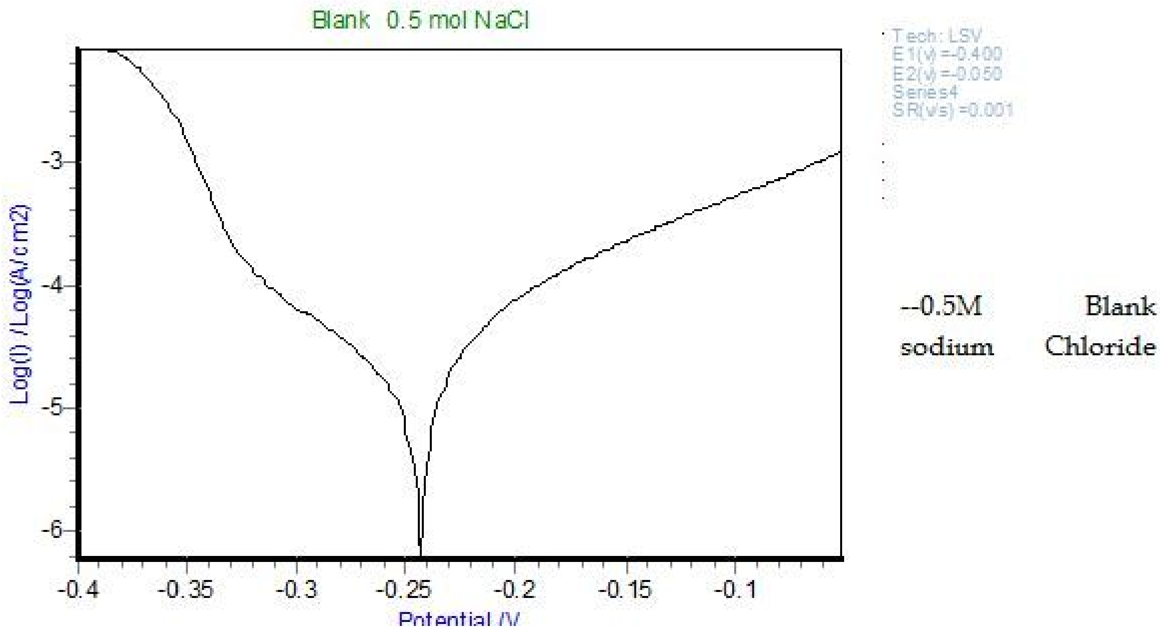


Figure 2

Tafel polarization curve of sodium chloride solution 0.5 M

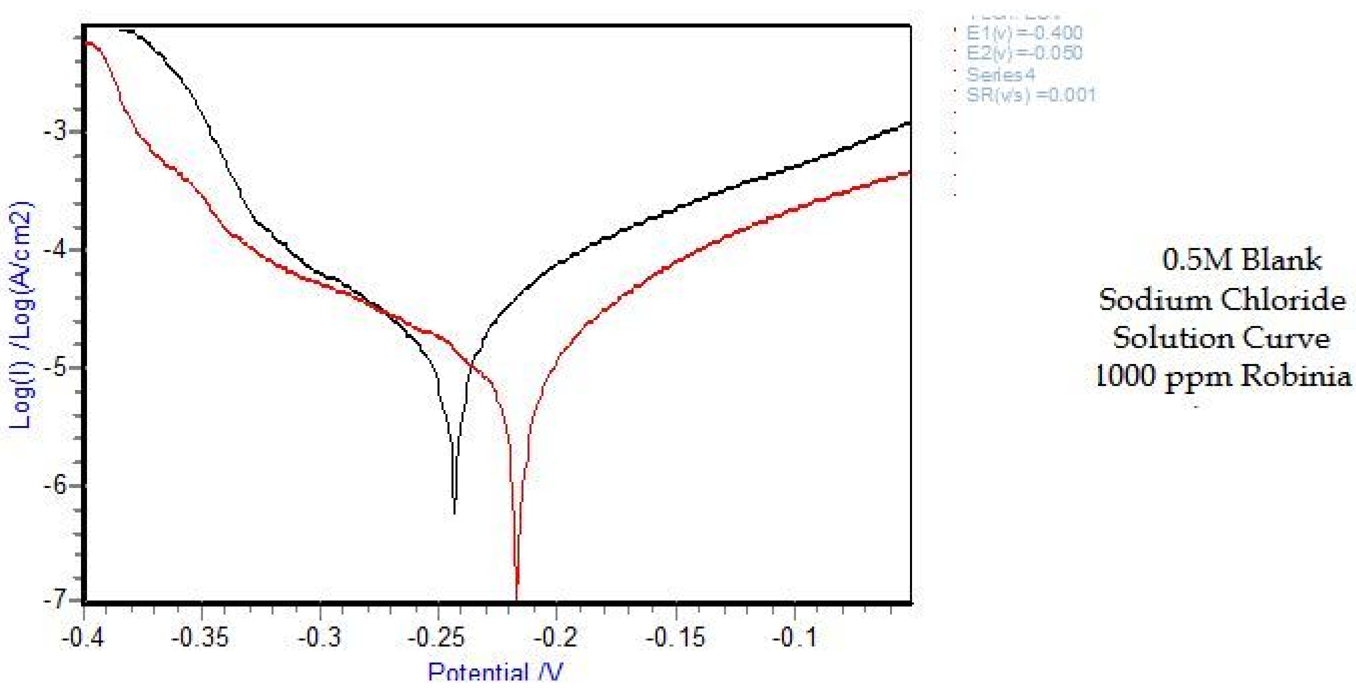


Figure 3

Tafel polarization curve of Robinia pseudoacania L solution at 1000 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

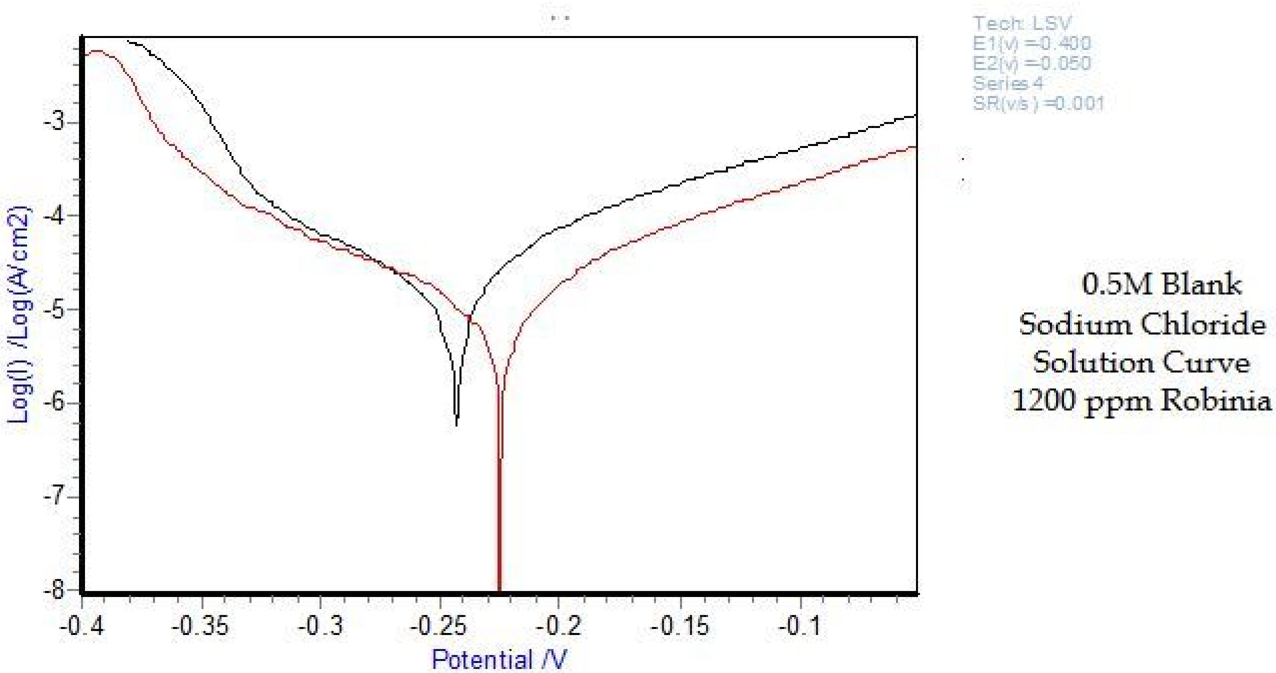


Figure 4

Tafel polarization curve of Robinia pseudoacania L solution at 1200 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

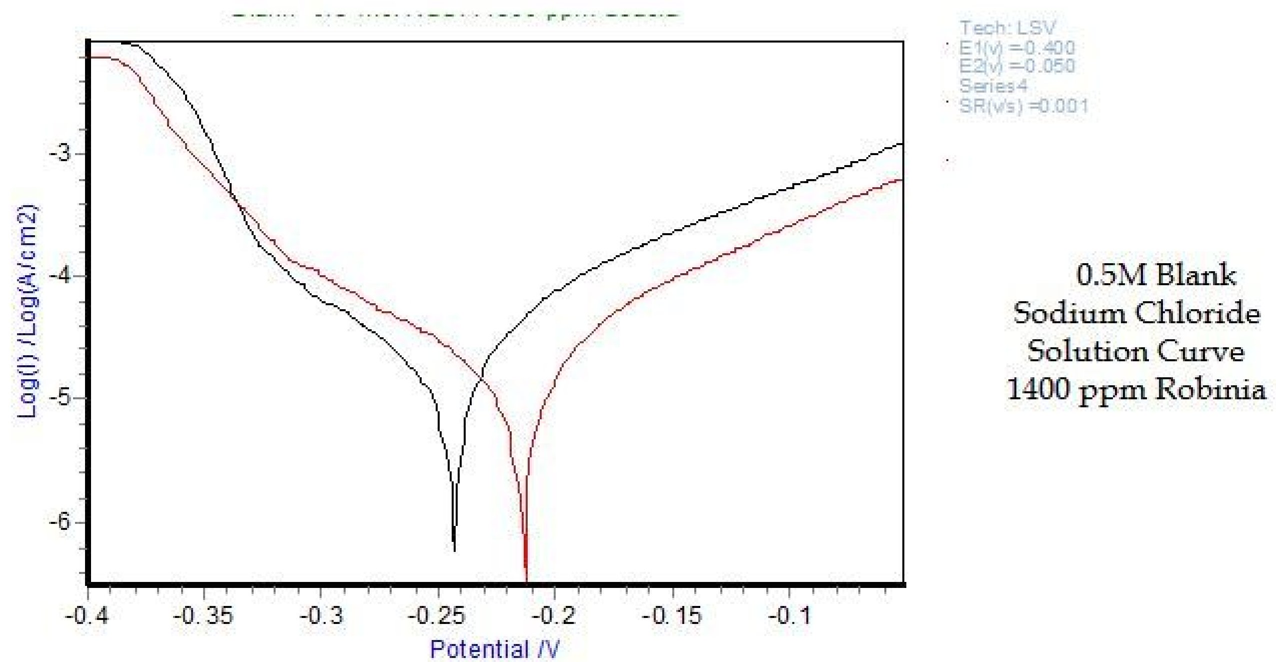


Figure 5

Tafel polarization curve of Robinia pseudoacania L solution at 1400 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

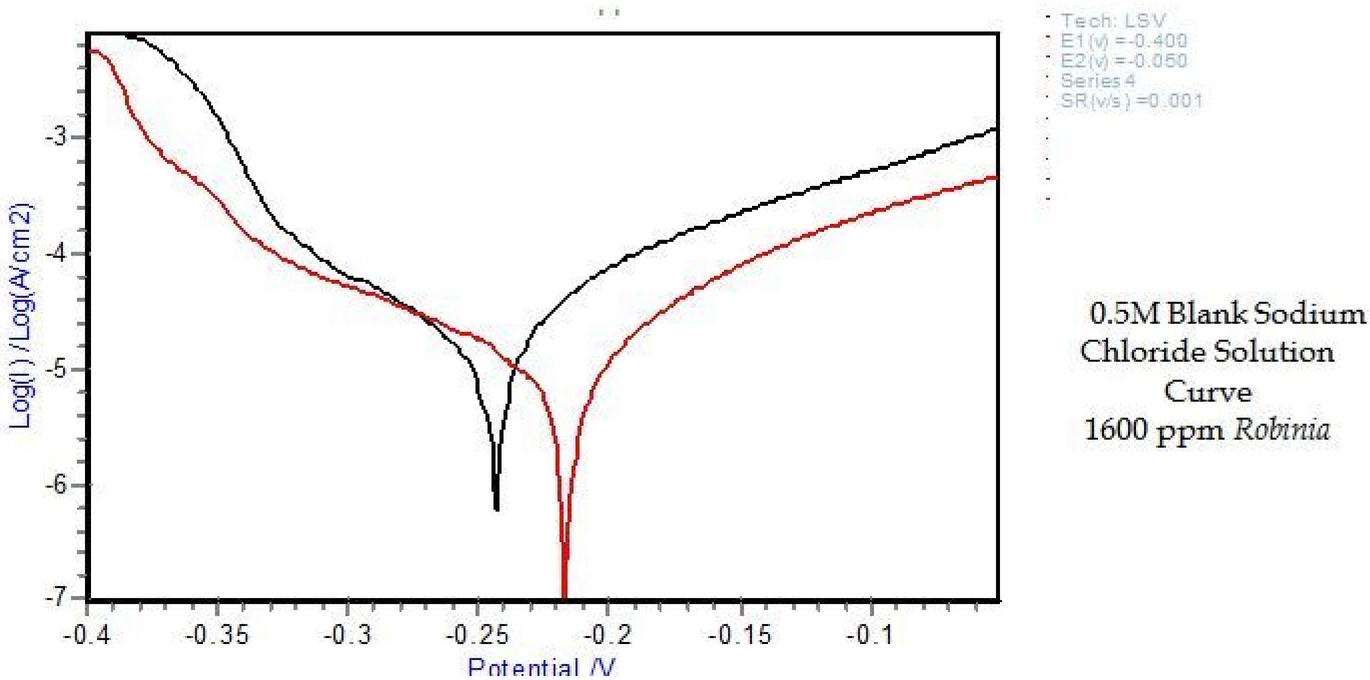


Figure 6

Tafel polarization curve of Robinia pseudoacania L solution at 1600 ppm in the presence of a corrosive solution of sodium chloride 0.5 Mm

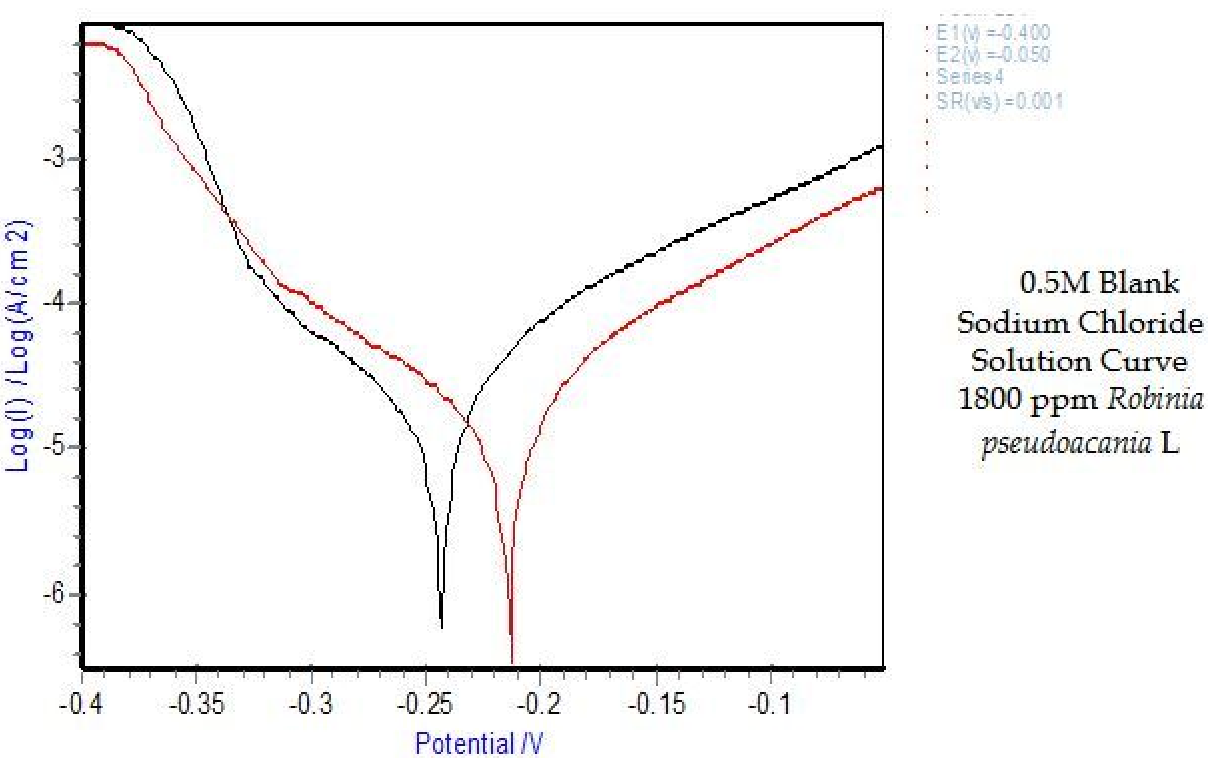


Figure 7

Tafel polarization curve of Robinia pseudoacania L solution at 1800 ppm in the presence of a corrosive solution of sodium chloride 0.5 M

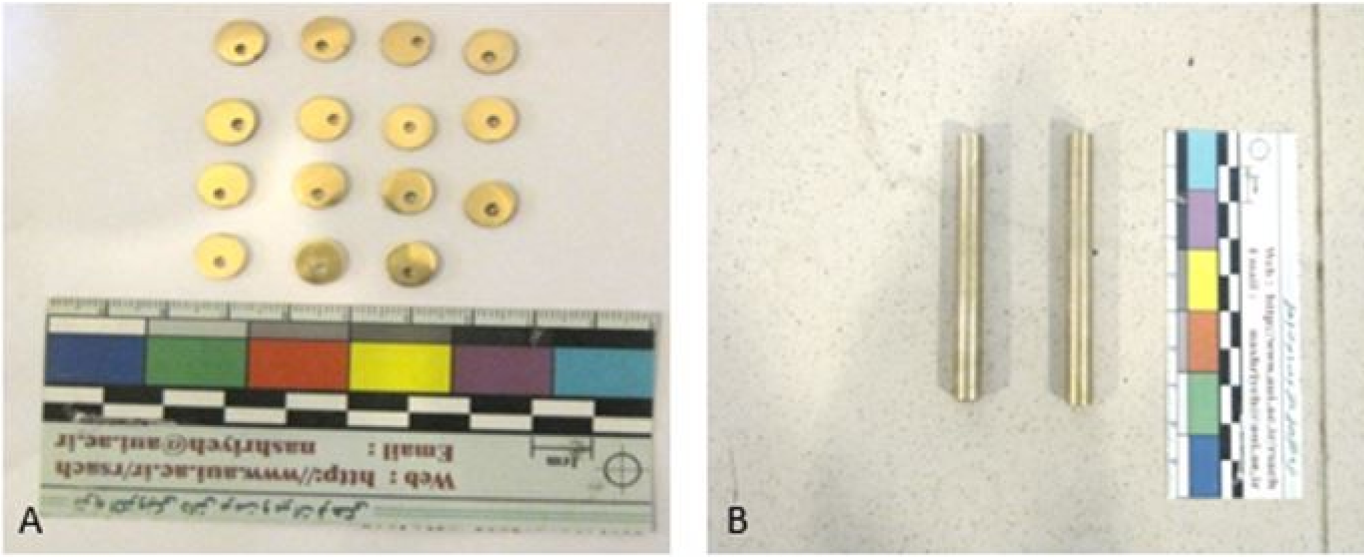


Figure 8

Prepared electrodes for cutting of coupons (A); Prepared coupons for immersion (B)

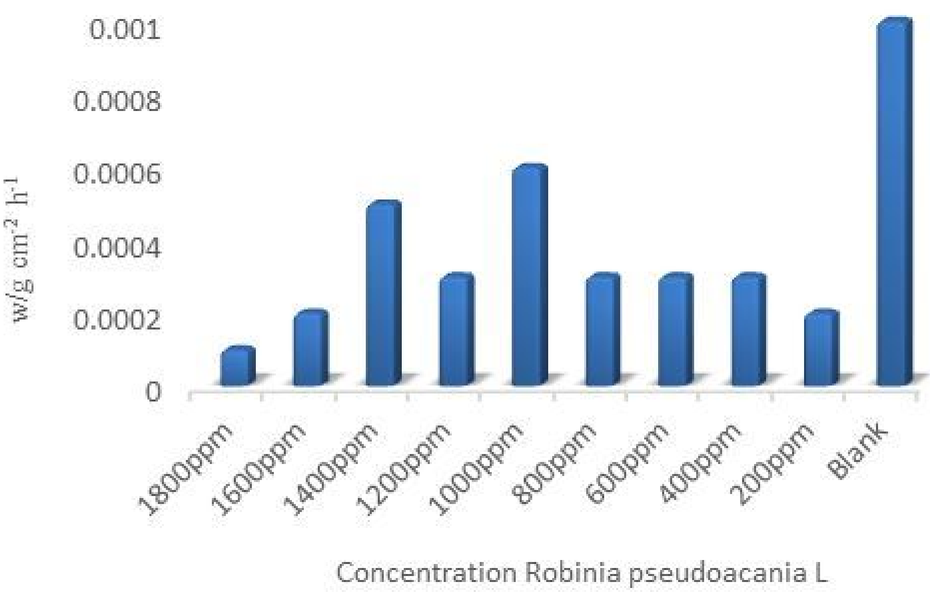


Figure 9

Weight loss level based on the concentration of 1800 ppm Robinia pseudoacania L after one week

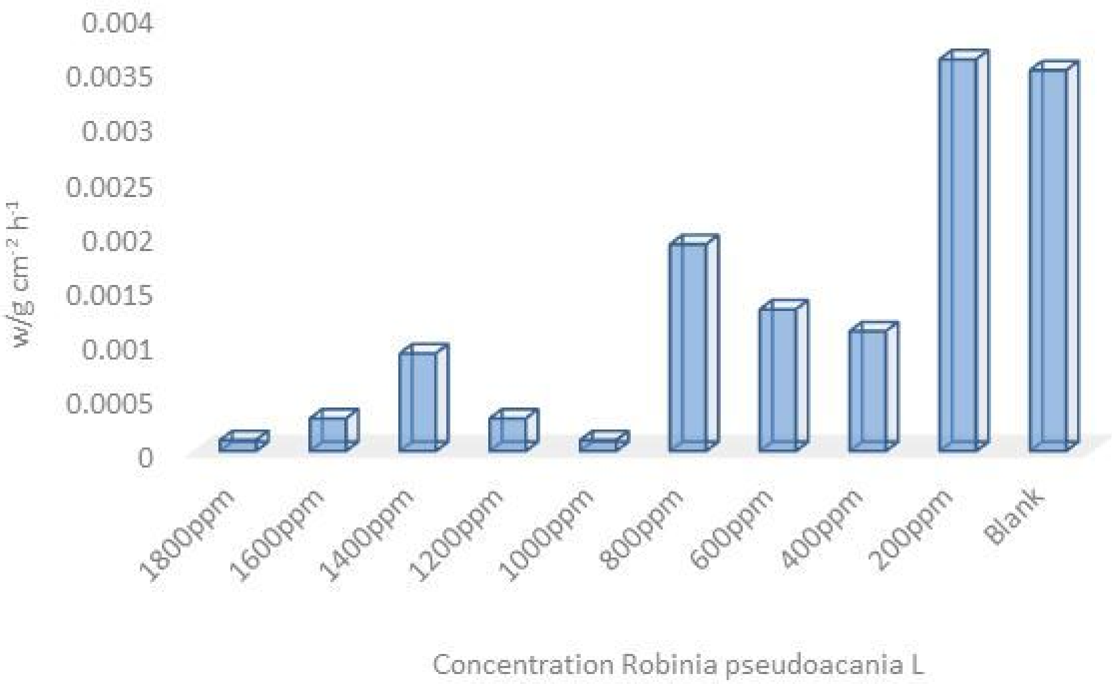


Figure 10

Weight loss level based on 1800 ppm concentration of Robinia pseudoacania L after two weeks

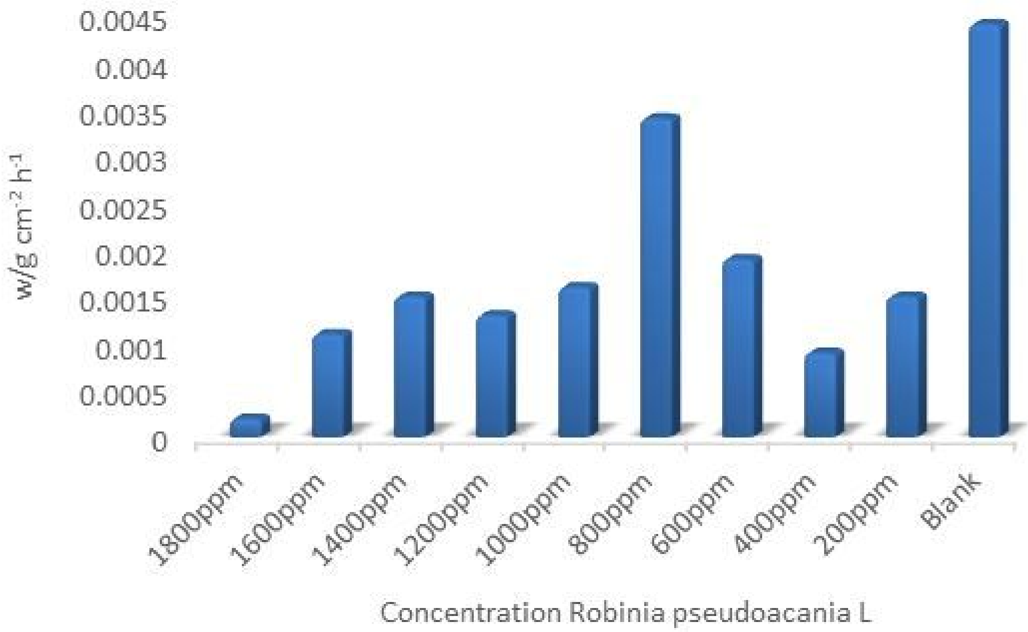


Figure 11

Weight loss level based on 1800 ppm concentration of Robinia pseudoacania L after three weeks

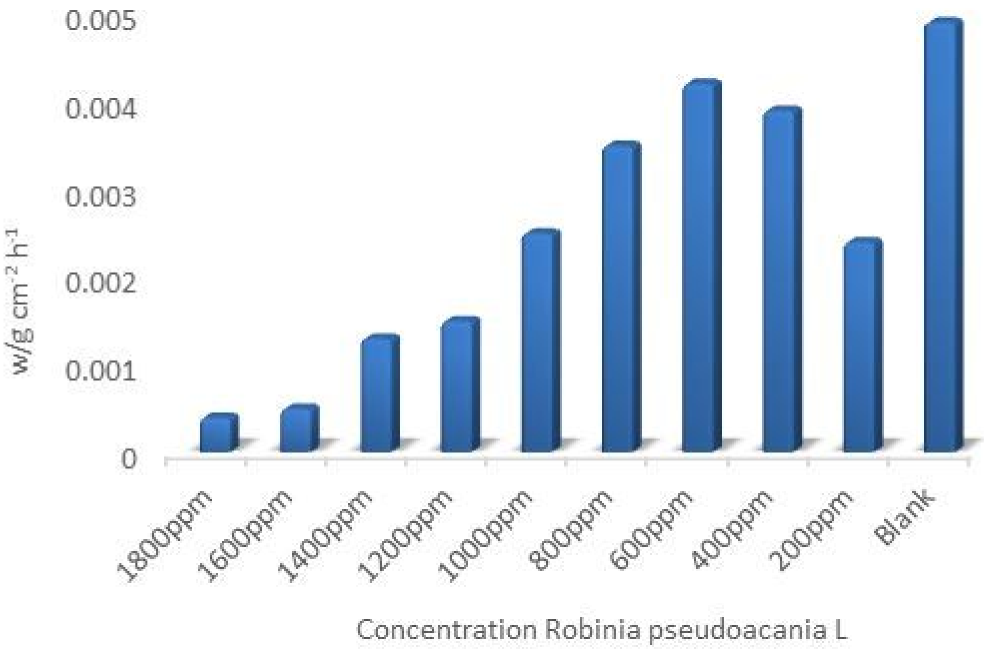


Figure 12

Weight loss level based on 1800 ppm concentration of Robinia pseudoacania L after four weeks

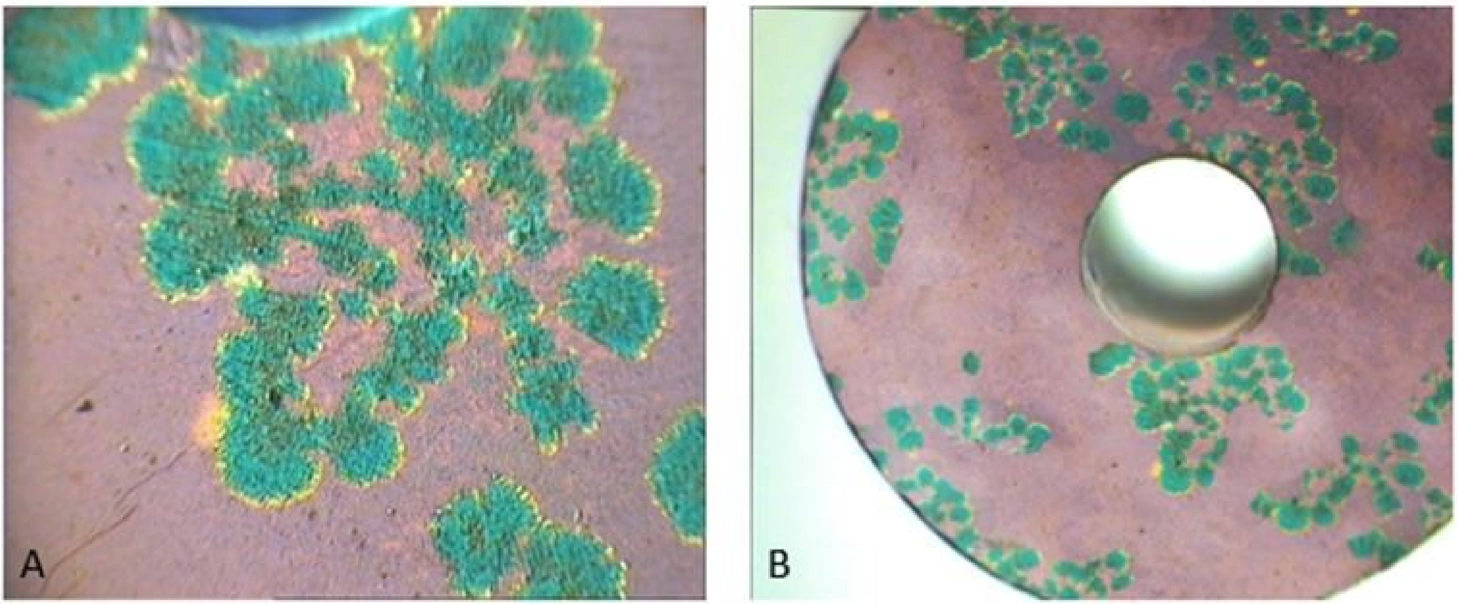


Figure 13

Coupons in a corrosive solution of sodium chloride 0.5 after 30 days of immersion. 60 x magnification (A); coupons in a corrosive solution of sodium chloride 0.5 after 30 days of immersion. 40 x magnification (B)

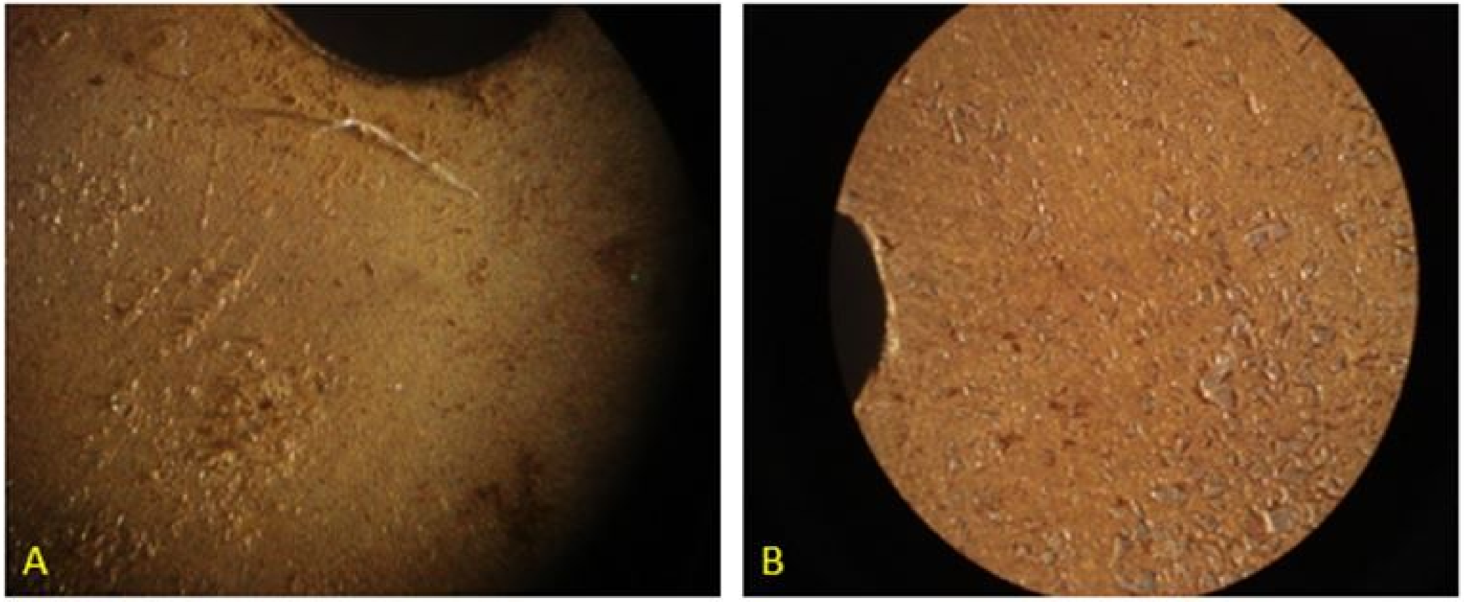


Figure 14

Coupon in the presence of Robinia pseudoacania L inhibitor with a concentration of 1000 ppm after 30 days of immersion. 60x magnification (A); coupon in the presence of Robinia pseudoacania L inhibitor with a concentration of 1000 ppm after 30 days of immersion. 40x magnification (B)

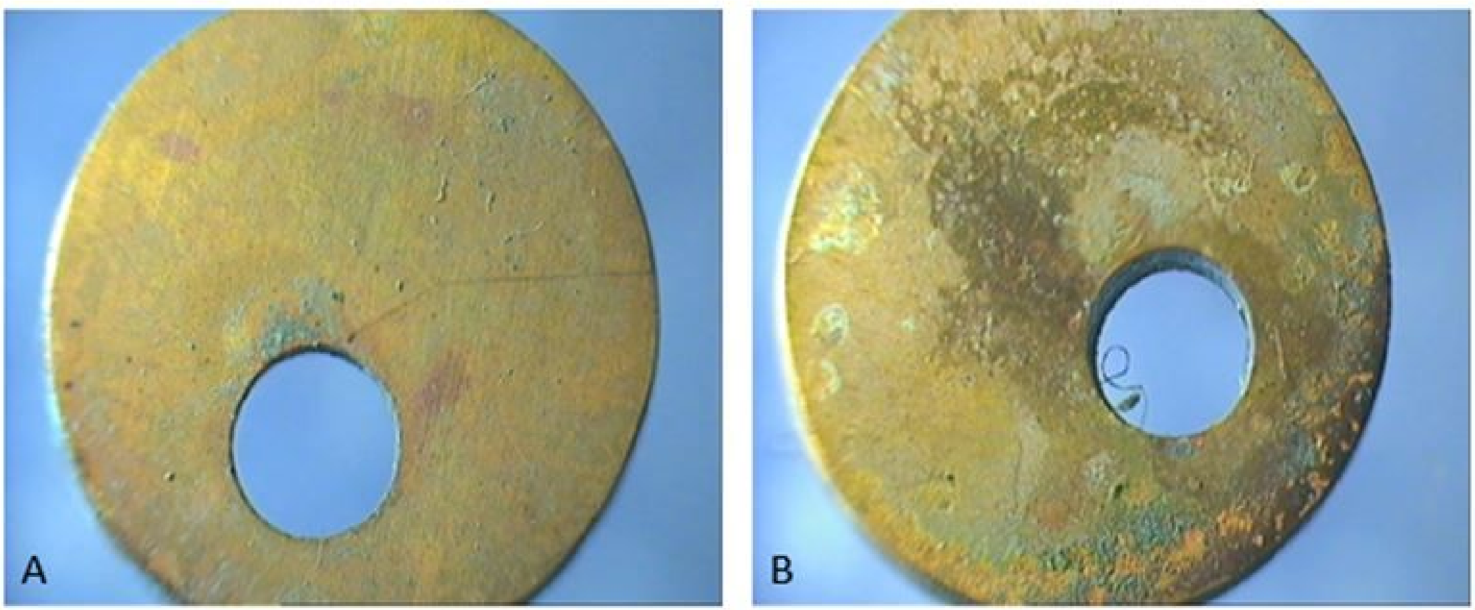


Figure 15

Coupon in the presence of Robinia pseudoacania L inhibitor with a concentration of 1000 ppm after 30 days of immersion. 20x magnification (A); coupon in the presence of Robinia pseudoacania L inhibitor with a concentration of 1000 ppm after 30 days of immersion. 20x magnification

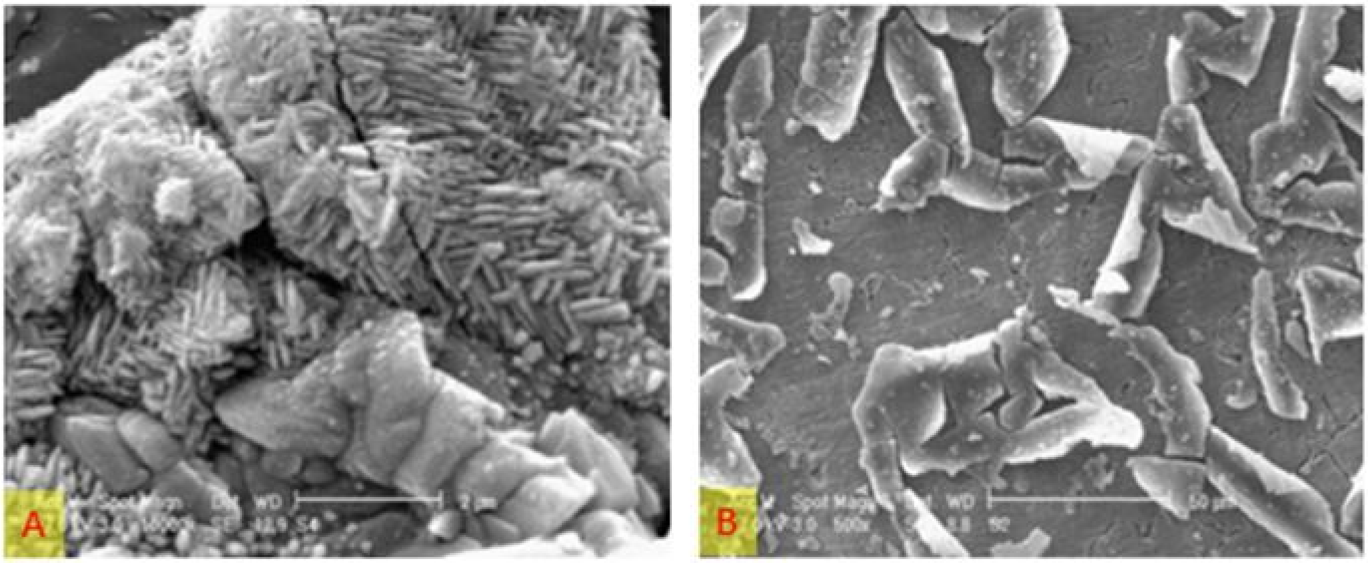
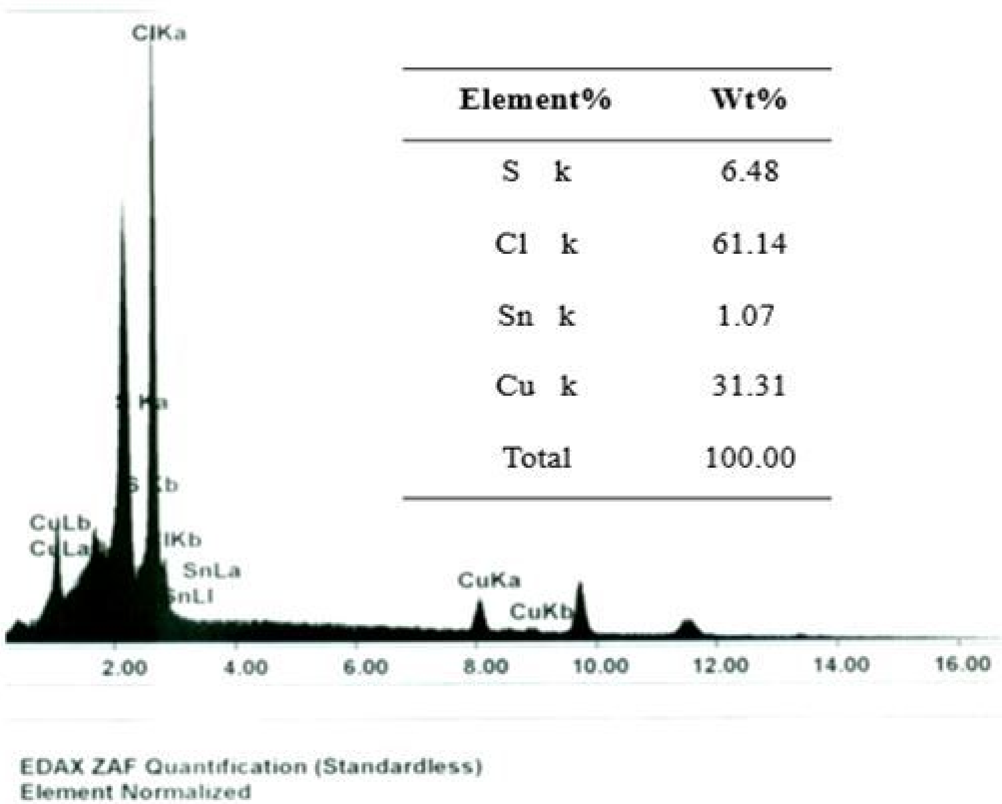


Figure 16

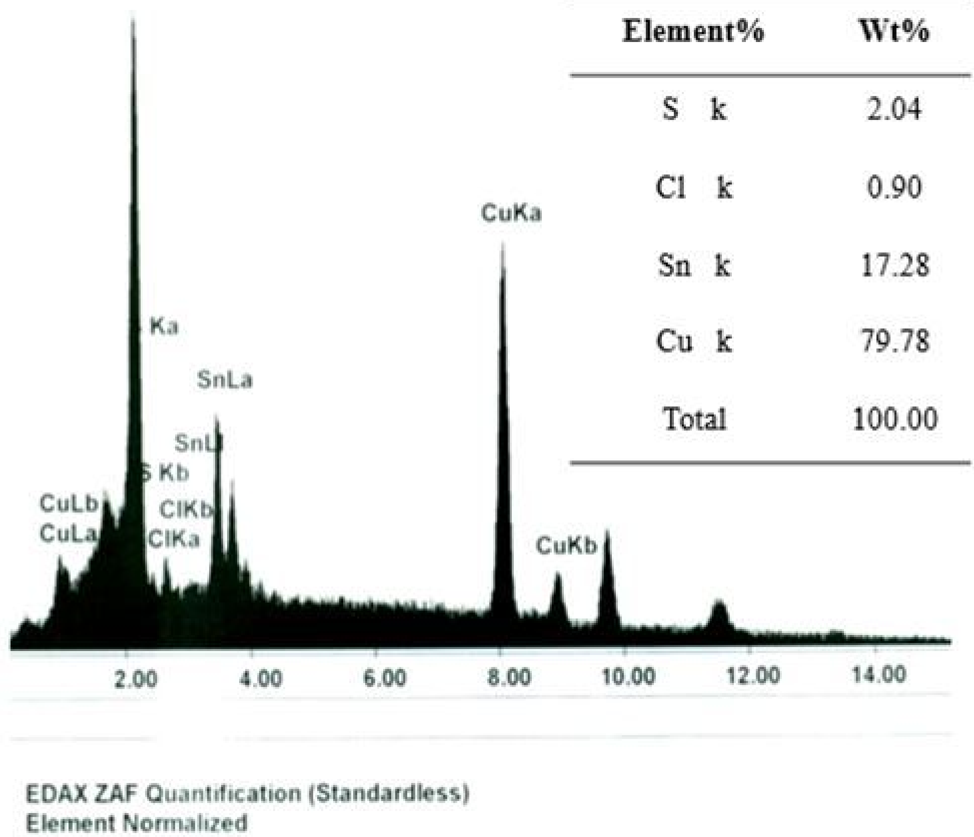
SEM analysis of control sample versus Sodium chloride 0.5 M corrosive solution (A); SEM analysis of coupon surface containing inhibitor at the presence of Sodium chloride 0.5 M corrosive solution



Element%	Wt%
S k	6.48
Cl k	61.14
Sn k	1.07
Cu k	31.31
Total	100.00

Figure 17

EDX analysis of control sample versus Sodium chloride 0.5 M corrosive solution



Element%	Wt%
S k	2.04
Cl k	0.90
Sn k	17.28
Cu k	79.78
Total	100.00

Figure 18

EDX analysis of coupon surface containing inhibitor at the presence of Sodium chloride 0.5 M corrosive solution