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Effect of natural inhibitors on the corrosion properties of titanium and magnesium alloys

M.Faraji, L. Pezzato, A. Yazdanpanah, I. Calliari, M. Esmailzadeh

This study investigates the effects of natural inhibitors (pomegranate juice, algae extract and tomato juice) on the corrosion resistance of titanium (grade 2) and magnesium alloys (AZ31). After sample preparation, corrosion tests were conducted using a potentiostat, employing Electrochemical Impedance Spectroscopy (EIS), and Potentiodynamic Polarization (PDP) tests. Two electrolytes were tested: a solution 3.5% NaCl and a solution 0.5M NaOH. All the tests were performed with 5% of inhibitor and with the reference solution. Also, inhibition efficiency was calculated on the base of PDP tests. The study found that pomegranate juice can act as good corrosion inhibitor for titanium and magnesium alloys in aqueous solutions 0.5M NaOH. This was demonstrated by the increase in the corrosion potential and impedance modulus and decrease in the corrosion current density after the addition of pomegranate juice to the solution. However, in a 3.5% NaCl solution, the efficacy of pomegranate juice was less pronounced, probably due to the high aggressivity of the electrolyte. Tomato juice and algae extract have instead shown very low inhibition effect in all the tested conditions.

KEYWORDS: NATURAL INHIBITOR, CORROSION, POMEGRANATE JUICE, TITANIUM, MAGNESIUM

INTRODUZIONE

Corrosion is one of the most destructive phenomena that affects many industrial sectors, such as the marine construction, oil and gas and automotive. Numerous studies have been conducted via different inhibitors to prevent harmful effects of corrosive processes [1-4]. In particular the interest on green inhibitors as eco-friendly materials is currently grown to address corrosion problems [5]. In recent years, several research have been conducted on employing plant and fruit extracts as natural inhibitors, highlighting them as environmentally friendly alternatives to traditional inhibitors for preventing metal corrosion. The advantages of using these green inhibitors are manifold. Firstly, they effectively safeguard metals from corrosion while preserving ecological equilibrium. Secondly, being derived from renewable, biodegradable, and eco-friendly resources, they contribute to minimizing environmental contamination. Compared to traditional inhibitors, they are not only more economical but also several are capable of being recycled. Therefore, integrating green inhibitors into metal corrosion prevention strategies is advocated as a responsible, affordable, and eco-conscious approach [6-8].

The mature section of the pomegranate fruit is rich in

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sugars, acids, vitamins, polyphenols, polysaccharides, and crucial nutrients [9]. It is recognized for its medicinal benefits, including antioxidant, antibacterial, and inhibitory effects, which are attributed to its content of anthocyanins like pelargonidin, delphinidin, and cyaniding, along with other phenolic compounds such as hydrolysable tannins including punicalin, punicalagin, gallagic, pedunculagin, ellagic acid and organic acids [10-17]. This paper aims to investigate how the use of pomegranate juice as a natural inhibitor influence the corrosion of Titanium (grade 2) and Magnesium (AZ31) alloys, tested in two different aqueous solutions: 3.5% NaCl and 0.5M NaOH. Also, other two natural inhibitors (tomato juice and algae extract) were preliminary tested but the results were not satisfactory.

MATERIALS AND METHODS

An initial ingot of grade 2 titanium alloy and of AZ31 magnesium alloy was cut transversally with a disc cutter, under the jet of a cooling fluid, in order to avoid thermal alterations and lubricate the blade. As a result, square samples with a side of 15 mm and a thickness of no more than 3-4 mm were obtained. After cutting, the samples were grinded and polished using a series of silicon carbide (SiC) abrasive papers (160, 320, 800, 1000 and 1200 grit) to provide smooth surfaces. Then the samples were washed with ultrasound to obtain an optimal surface finish free of impurities. Both potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS) tests were conducted using a Gamry Interface 1010E potentiostat in a three-electrode configuration using a Platinum electrode as counter electrode a calomel electrode as reference and the tested sample (1 cm^2) as working electrode. PDP tests were conducted between -2V and +3V with a scan rate of 1 mV/s. EIS tests were conducted with a sinusoidal perturbation of 10 mV over the frequency range of 100 kHz to 0.1 Hz. All the electrochemical tests were conducted after 30 min of OCP stabilization in two different electrolytes 0.5 M NaOH and 3.5% NaCl aqueous solutions. The tests were firstly conducted in the solution without the inhibitor, as reference, and after the addition of 5% of corrosion inhibitor (pomegranate juice, tomato juice and algae extract). Results of the EIS were after fitted with proper equivalent circuit using Z-View software and the equivalent circuit is presented in the section of the results. Corrosion potential and corrosion current density were graphically extrapolated from PDP tests using the Tafel law and then the data were employed to calculate the inhibition efficiency.

RESULT AND DISCUSSION

Electrochemical Impedance Spectroscopy (EIS)

Fig. 1-2 show the Nyquist plots of the grade 2 titanium samples in 0.5 M NaOH and 3.5% NaCl solution with or without the natural inhibitors. In these figures, the experimental and fitting data can be observed by the scattered dots and straight line respectively.

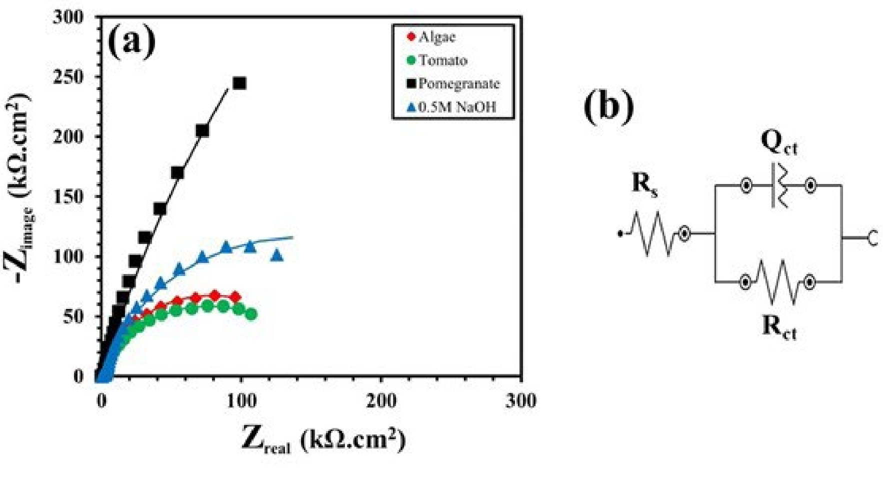

Fig.1 - a) Nyquist plots of the Ti samples in 0.5M NaOH solution with/without inhibitors b) Schematic of the equivalent electrical circuit on the electrode surface / a) Diagramma di Nyquist relativo ai test su Ti grado 2 in soluzione 0.5M NaOH b) Circuito equivalente utilizzato per il fitting dei dati sperimentali

Tab.1 - EIS parameters of the equivalent circuit for grade 2 Ti in 0.5 M NaOH with/without inhibitor / Valori ottenuti dal fitting dei dati EIS sperimentali relativi ai test su campioni in Ti grado 2 in soluzione 0.5M NaOH con e senza inibitore.

Sample	R_{sol} (Ω.cm²)	R_{ct} (Ω.cm²)	Q		$\chi^{(\%)}$
			n	Y	
0.5M NaOH	23.23	2.82×10^5	0.79	1.30×10^{-5}	1.28
Pomegranate	22.72	2.01×10^6	0.85	3.51×10^{-5}	1.93
Tomato	26.70	1.52×10^5	0.83	3.81×10^{-5}	1.18
Algae	24.23	1.56×10^5	0.85	3.53×10^{-6}	1.51

According to Fig. 1a, the impedance at lower frequencies serves as an indicative measure of both the polarization resistance and the corrosion resistance. Therefore, the corrosion resistance values of the specimens are in the order of pomegranate > 0.5 NaOH (reference) > tomato > Algae. Fig. 1b displays the equivalent electrical circuit employed to fit the experimental data obtained by EIS test. The circuit includes a solution resistance (R_s) in series with a parallel capacitive loop charge transfer resistance and constant phase element (CPE) of the oxide layer formed on the surface (R_{ct}/Q_{ct}). The CPE is inserted into the circuit to serve as an alternative to the capacitor, aiming for a closer fitting under conditions where the frequency exponent is below 1.0. [18-20]. The charge transfer resistance (R_{ct}) of the double layer is inversely proportional to the corrosion rate. According to Tab. 1, the R_{ct} value of the Ti sample in 0.5 M NaOH solution without any inhibitors is 2.82×10^5 Ω.cm^{-2}. By adding the pomegranate extract as an inhibitor, the Rct value was increased to 2.01×10^6 Ω.cm^{-2}. In other cases, it was observed that tomato juice and algae have a negative effect on the corrosion resistance of Ti samples in NaOH solution, so that the R_{ct} values is decreased to 1.52×10^5 and 1.56×10^5 Ω.cm^{-2}, respectively.

Fig. 2a displays the results of the EIS tests carried out on a grade 2 Ti sample in 3.5% NaCl solution. In this case it does not evidence a significant increase in corrosion resistance using natural inhibitors. It can be observed that the use of pomegranate juice produces only a slight inhibition effect, especially in comparison with the results obtained in 0.5 M NaOH solution. According to the equivalent electrical circuit (Fig. 2b) and EIS parameters given in Tab. 2, the R_{ct} value of the Ti sample in 3.5% NaCl solution without any inhibitors, was increased from 8.14×10^5 Ω.cm^{-2} to 1.53×10^6 Ω.cm^{-2} by adding pomegranate juice. Inversely the addition of tomato and algae decreased the corrosion resistance of the Ti alloy, so that the value of R_{ct} significantly decreased to 92413 and 39450 Ω.cm^{-2}, respectively.

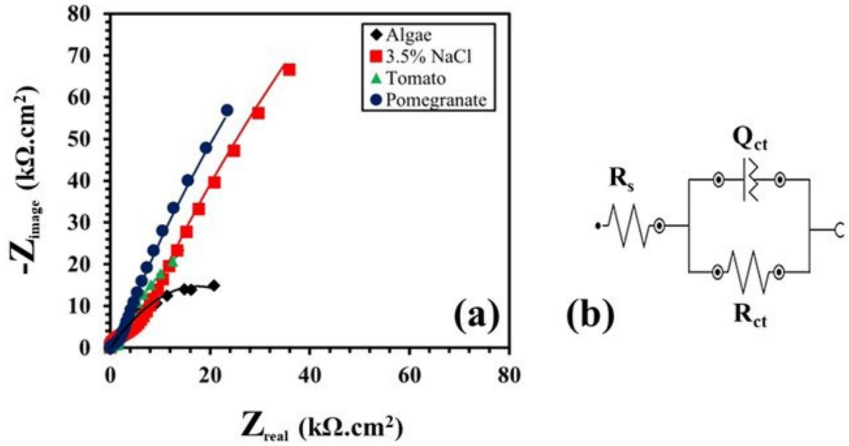

Fig.2 - a) Nyquist plots of the grade 2 Ti samples in 3.5% NaCl solution with different inhibitors. b) Schematic of the equivalent electrical circuit on the electrode surface / a) Diagramma di Nyquist relativo ai test su Ti grado 2 in soluzione 3.5% NaCl b) Circuito equivalente utilizzato per il fitting dei dati sperimentali

Tab.2 - EIS parameters of the equivalent circuit for grade 2 Ti in 3.5% NaCl solution with/without / Valori ottenuti dal fitting dei dati EIS sperimentali relativi ai test su campioni in Ti grado 2 in soluzione 3.5% NaCl con e senza inibitore inhibitor.

Sample	R_{sol} (Ω.cm²)	R_{ct} (Ω.cm²)	Q		$\chi^{(\%)}$
			n	Y	
3.5% NaCl	46.35	8.14×10^{5}	0.81	1.30×10^{-5}	1.39
Pomegranate	36.54	1.53×10^{6}	0.80	1.36×10^{-5}	1.57
Tomato	54.62	92413	0.84	2.61×10^{-5}	1.11
Algae	31.14	39450	0.83	1.53×10^{-5}	1.80

Fig. 3a reports the results of the EIS tests carried out on AZ31 samples in 0.5M NaOH solution. In the case of AZ31 alloy, it can be observed that the use of natural inhibitors does not change the impedance and corrosion resistance values; this means that the natural inhibitors studied in this work have no significant effects on the corrosion properties of magnesium in these conditions. Fig. 3b illustrates the equivalent electrical circuit employed to fit the experimental data obtained by EIS test. As shown in Tab. 3, by addition of pomegranate juice and algae into 0.5M NaOH solution, the R_{ct} value of the AZ31 alloy sample was decreased from 70506 Ω.cm^{-2} to 50825 and 43920 Ω.cm^{-2}, respectively, thus indicating negative effect of these natural compounds on the corrosion properties of AZ31 alloy.

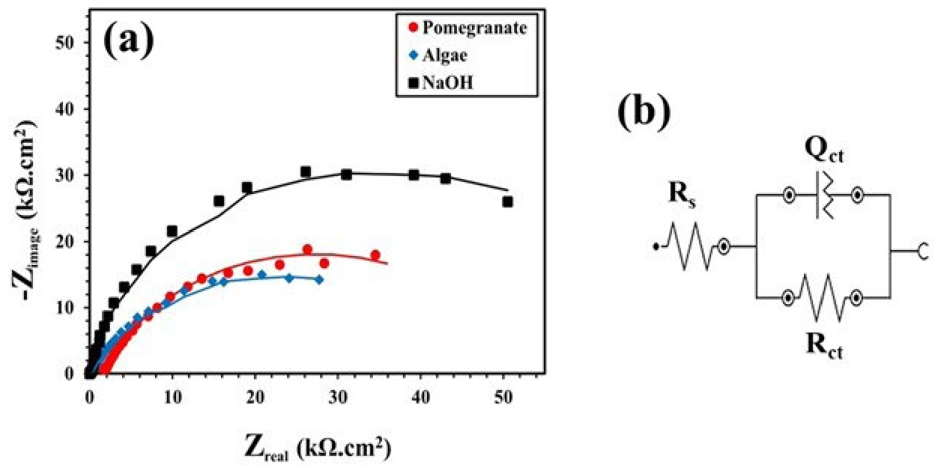

Fig.3 - a) Nyquist plots of the AZ31 samples in 0.5M NaOH solution with different inhibitors b) Schematic of the equivalent electrical circuit on the electrode surface / a) Diagramma di Nyquist relative ai test su lega di magnesio AZ31 in soluzione 0.5M NaOH b) Circuito equivalente utilizzato per il fitting dei dati sperimentali

Tab.3 - EIS parameters of the equivalent circuit for AZ31 magnesium alloy in 0.5 M NaOH with/without inhibitor / Valori ottenuti dal fitting dei dati EIS sperimentali relativi ai test su lega di magnesio AZ31 in soluzione 0.5M NaOH con e senza inibitore

Sample	R_{sol} (Ω.cm²)	R_{ct} (Ω.cm²)	Q		$\chi^{(\%)}$
			n	Y	
0.5M NaOH	30.51	70506	0.81	2.01×10^{-5}	1.06
Pomegranate	35.88	50825	0.78	3.03×10^{-5}	1.44
Algae	28.59	43920	0.85	1.57×10^{-5}	1.87

Potentiodynamic polarization (PDP)

Fig. 4 and 5 show the potentiodynamic polarization curves of grade 2 Ti and AZ31 samples in 2 different solutions (0.5 M NaOH and 3.5% NaCl). Corrosion parameters were determined by applying the Tafel extrapolation method. Linear regression was performed on the linear portions of both the anodic and cathodic branches of the polarization curves [21, 22]. For active–passive materials such as titanium, careful selection of the linear region was essential to avoid the transition region where passive film formation or breakdown occurs. In instances where a sharp increase in current density was observed (indicative of passive film breakdown or localized corrosion initiation) those data points were excluded from the linear fit.

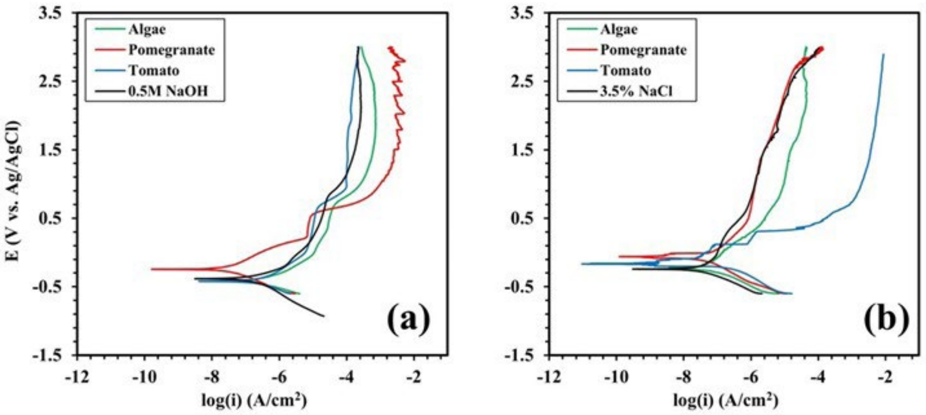

Fig.4 -PDP curves of the Ti samples in a) 0.5M NaOH and b) 3.5% NaCl solution with different inhibitors. / Risultati delle prove potenziodinamiche su Ti grado 2 in a) 0.5 M NaOH e b) 3.5% NaCl in soluzioni con diversi inibitori.

Fig. 4a shows the trends of the polarization curves in the case of titanium immersed in an aqueous solution 0.5M NaOH + natural inhibitors. By considering the graph, it can be observed that the only curve that deviates from the solution without inhibitors (reference solution) is the curve of the pomegranate juice. Tab. 6 shows the corrosion parameters of the grade 2 Ti samples graphically extrapolated from PDP curves in 0.5M NaOH, particularly the i_{corr} and E_{corr}. The presence of pomegranate juice obviously causes a shift in the E_{corr} of NaOH from -384 to -244 mV. Also, a remarkable decrease in the i_{corr} value of the sample tested in solution containing pomegranate juice can be observed, indicating high inhibition effect. According to the figure, the addition of pomegranate extract results in a pronounced increase in anodic current density at potentials above 0.5 V vs. Ag/AgCl. The anodic current for the pomegranate-treated sample is approximately one order of magnitude higher compared to the material in NaOH without inhibitors. This behavior suggests that rather than simply reducing the corrosion rate, pomegranate extract may be influencing the passivation process, potentially modifying the stability of the surface oxide layer. The increased anodic current in this range indicates that the inhibitor may be facilitating localized dissolution or altering the electrochemical properties of the protective film. In contrast, the algae and tomato extracts in NaOH do not exhibit the same extent of anodic current enhancement, indicating a different mode of interaction with the oxide layer. The fact that the anodic current density increases significantly in the presence of pomegranate extract implies that a simple Tafel extrapolation approach would be insufficient to accurately determine the corrosion rate, as it does not account for changes in passivation and trans passive behavior at higher potentials.

Fig. 4b illustrates the behavior of a grade 2 Ti sample in 3.5% NaCl solution. From the graph it can be observed that the natural inhibitors under consideration do not significantly increase the resistance of the material as comparing the anodic branch of the different curves it is evident that there is no improvement. Considering the corrosion potential there is a slight increase in this parameter in the presence of tomato juice and a more significant increase in the presence of pomegranate juice. Globally, however, it can be stated that from PDP tests the inhibitors do not produce significant positive effects to the corrosion properties 3.5% NaCl solution. Unlike the pomegranate extract in NaOH, the presence of tomato extract does not result in a significant overall improvement in corrosion resistance. The increased anodic current density suggests that the extract may be destabilizing the passive oxide film, potentially making the material more vulnerable to localized corrosion in chloride-rich environments. The algae and pomegranate-treated samples in NaCl exhibit relatively lower anodic current densities, suggesting that these extracts provide better surface protection under chloride exposure. Tab. 5 displays the corrosion parameters of the grade 2 Ti samples graphically extrapolated from PDP curves in 3.5% NaCl. The presence of pomegranate juice causes a shift in the E_{corr} of NaCl solution from -255 to -82 mV. The i_{corr} value of pomegranate juice in NaCl solution is 9.67×10^{-7} mA/mm^2, which has the best corrosion resistance among the inhibitors, but the differences are very small in comparison to the other tested samples.

Finally, in Fig. 5 the trends of the polarization curves of the AZ31 in 0.5M of NaOH solution are shown. From this graph it is first noted that the only significant improvement is at the level of corrosion potential in the case of the solution with the addition of pomegranate juice, as the corrosion potential is significantly higher than the one in other curves. Looking at the anodic branch, however, we note that the pomegranate juice does not bring particular improvements to the resistance of the material.

From the polarization curves it was possible to calculate the efficiency of the single inhibitors inserted in the different solutions. The efficiencies reported in Tables 3 to 5 have been calculated using the following formula:

$$IE\% = \frac{(i_0 - i)}{i_0} * 100 \quad (1)$$

where IE% is the inhibitors percentage efficiency, i0 is the current density in the case of the solution without inhibitors and i is the current density in the solution with the inhibitor of which the efficiency is to be calculated.

From the tables, it is possible to note that pomegranate juice has good efficiency in all three different tested solutions whereas the effect if tomato juice and algae extract is controversial and, in any case, lower than the one of pomegranate juice.

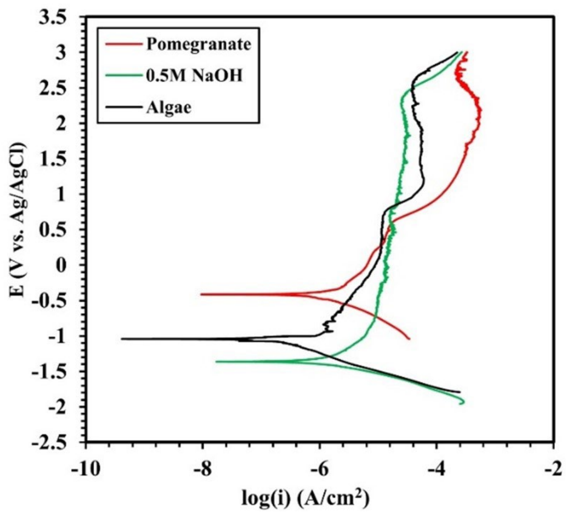

Fig.5 - PDP curves of the AZ31 alloy samples in 0.5M NaOH solution with different inhibitors / Risultati delle prove potenziodinamiche su lega AZ31 in 0.5 M NaOH con diversi inibitori.

Tab.4 - The corrosion parameters and IE% inhibitors of AZ31 alloy samples in 0.5M NaOH / Dati estrapolati dalle prove potenziodinamiche e efficienza di inibizione per i campioni di lega AZ31 in soluzione 0.5M NaO

Sample	E_0 (mV)	I_0 (mA/mm^2)	IE%
0.5M NaOH	-1057.6	7.29×10^{-5}	0
Algae	-1044.1	6.01×10^{-5}	18%
Pomegranate	-421.3	5.46×10^{-5}	25%

Tab.5 - The corrosion parameters and IE% inhibitors of Ti samples in 3.5% NaCl / Dati estrapolati dalle prove potenziodinamiche e efficienza di inibizione per i campioni di Ti grado 2 in soluzione 3.5% NaCl

Sample	E_0 (mV)	I_0 (mA/mm^2)	IE%
3.5% NaCl	-254.57	2.67×10^{-6}	0
Algae	-248.99	4.04×10^{-6}	-52%
Tomato	-167.65	1.01×10^{-6}	62%
Pomegranate	-82.40	9.67×10^{-7}	64%

Tab.6 - The corrosion parameters and IE% inhibitors of grade 2 Ti samples in 0.5M NaOH / Dati estrapolati dalle prove potenziodinamiche e efficienza di inibizione per i campioni di titanio grado 2 in soluzione 0.5M NaOH

Sample	E_0 (mV)	I_0 (mA/mm^2)	IE%
3.5% NaCl	-383.7	1.12×10^{-6}	0
Algae	-421.38	9.37×10^{-7}	16%
Tomato	-417.28	5.71×10^{-7}	49%
Pomegranate	-244.08	4.65×10^{-7}	58%

CONCLUSION

In this study the effect of 3 natural inhibitors (pomegranate and tomato juice and algae extract) on the corrosion behavior of grade 2 Ti and AZ31 Mg in two different environments (0.5M NaOH and 3.5% NaCl) was investigated. It was observed that the only substance that give significant effect in terms of corrosion inhibition is pomegranate juice. In fact, this substance has shown to produce a remarkable increase of the corrosion potential in the case of grade 2 titanium inserted in an aqueous solution with 0.5M of NaOH and a remarkable improvement of the corrosion potential also for AZ31 magnesium alloy inserted in the same solution. In the case of aqueous solution with 3.5% NaCl, however, the inhibitors were found to be less effective, even though, pomegranate juice is still the best of the three. In the EIS tests, pomegranate was found to produce a remarkable increase in the polarization resistance in the case of titanium inserted in aqueous solution with 0.5M of NaOH. Interestingly, pomegranate exhibits inhibitory effects; however, these effects are less pronounced under PD polarization. In conclusion, it is advisable to choose pomegranate juice as a possible natural inhibitor to conduct further investigations in order to clarify better the mechanism of corrosion inhibition. Algae and tomato juice instead did not give positive results in this study.

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Effetto degli inibitori naturali sulla resistenza alla corrosione delle leghe di titanio e magnesio

Il presente studio indaga gli effetti di alcuni inibitori naturali (succo di melograno e pomodoro e estratto di alghe) sulla resistenza alla corrosione del titanio (grado 2) e della lega di magnesio AZ31. Dopo la preparazione del campione,la resistenza a corrosione è stata valutata mediante spettroscopia di impedenza elettrochimica (EIS) e test di polarizzazione potenziodinamica (PDP) mendiante l'ausilio di un potenziostato. Sono stati testati due differenti elettroliti: una soluzione 3,5% NaCl e una soluzione 0,5 M NaOH. Tutti i test sono stati eseguiti con il 5% di inibitore e con la soluzione di riferimento priva di inibitore. Inoltre, è stata calcolata l'efficienza di inibizione sulla base dei risultati delle prove potenziodinamiche. Lo studio ha permesso di evidenziare che il succo di melograno può agire come un buon inibitore della corrosione per le leghe di titanio e magnesio in soluzioni acquose con 0,5 M NaOH. Ciò è stato dimostrato dall'aumento del potenziale di corrosione e del modulo di impedenza e dalla diminuzione della densità di corrente di corrosion dopo l'aggiunta di succo di melograno alla soluzione. Tuttavia, in una soluzione 3,5% NaCl, l'efficacia del succo di melograno è stata meno pronunciata, probabilmente a causa dell'elevata aggressività della soluzione. Il succo di pomodoro e l'estratto di alghe hanno invece evidenziato scarsa capacità di inibizione in tutte le condizioni testate.

PAROLE CHIAVE: INIBITORI NATURALI, CORROSIONE, SUCCO DI MELOGRANO, TITANIO, MAGNESIO

TORNA ALL'INDICE >