

Experimental Investigation of Inhibition Efficiency of Crocin for Chloride-Induced Corrosion of Aluminum Alloys

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Abstract

The use of Crocin, derived from the flowers of *Crocus sativus*, is investigated as corrosion inhibitor for the AA1050, AA5083, AA5754 and AA6082 aluminum alloys in chloride ions environment. Aluminum and aluminum alloys are subjected to corrosion in the aggressive environment of chlorides, so several green corrosion inhibitors, mostly of plant origin, with minimum impact on health and the environment have been examined. In this study, the inhibition efficiency of 1.25 mM Crocin in a 0.01 M NaCl corrosive solution was assessed via electrochemical corrosion techniques and gravimetric mass loss measurements of the aluminum alloys. The surface of the specimens was examined using Scanning Electron Microscopy, Energy Dispersive Spectroscopy, Stereomicroscopy and Glossiness measurements. Experimental results reveal the protective anticorrosive action of Crocin for all aluminum alloys in the sodium chloride medium.

Keywords

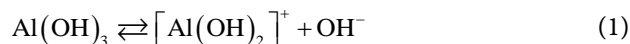
Crocin, Aluminum Corrosion, Eco-Friendly Inhibitors, Sodium Chloride Environment, Electrochemical and Gravimetric Methods

1. Introduction

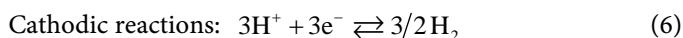
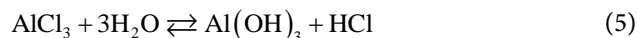
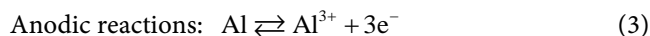
Aluminum alloys are commonly employed in aerospace and automotive industry, in construction, and in marine, packaging, electrical, and electronic applications. The 1xxx series, commercially pure aluminum, is more corrosion resistant than any of the aluminum alloys. The 5xxx series alloys contain magnesium, resulting in high strength and moderate resistance against uniform and localized

corrosion in chloride-containing solutions [1]. The Al-Si-Mg alloys (6xxx) have a good balance of corrosion resistance and strength [2] [3] [4].

Aluminum is very reactive, as newly created aluminum surface exposed to air or water at room temperature forms immediately a very thin, impermeable and adherent oxide film, which generally protects the metal from corrosion [5]. This layer consists of Al_2O_3 , $\text{Al}(\text{OH})_3$, $\text{AlO}\cdot\text{OH}$, and their hydrous forms, with certain oxyhydroxides present in the bulk of the aluminum oxide. The hydration of the surface is followed by dissociation and ionization through two different ways as follows [6]:



When this protective film is damaged, it reforms instantaneously in non-aggressive environments, but once this film is removed or destroyed in acidic, alkaline, or chloride-containing solutions, self-repair is not possible and corrosion initiates [7] [8]. In chloride solutions, Cl^- ions are attracted to the surface, adsorb and penetrate the passive oxide layer in several sites, breaking it and causing pitting corrosion [9]. Anodic and cathodic reactions on the aluminum surface in the aqueous environment of chloride ions are described as follows [2] [7] [8] [10]:



In addition, repassivation of the surface is delayed due to the oxygen reduction, which causes a local increase in pH and formation of aluminate ions ($\text{AlO}\cdot\text{O}^-$) [11].

Corrosion protection of aluminum and its alloys is very important for financial and environmental reasons and conservation of natural resources, energy and materials. Corrosion inhibitors have been extensively investigated [9] [12]. Organic compounds, especially those containing polar groups with phosphorus, nitrogen, sulphur, and oxygen atoms in conjugated molecular systems, presented inhibition efficiency determined by their electron density and their contribution to electron acceptance or donation processes [8] [9] [11] [12] [13] [14]. Thus, their action is attributed to their affinity to interact and be adsorbed on the aluminum surface acting as nucleophiles, while their available electron pairs can be shared with the metal surface acting as electrophiles. Their activity occurs through the formation of protective layers on the aluminum surface by physisorption, chemisorption or by synergistic mechanisms [9] [12].

However, many organic compounds have been questioned due to their toxic-

ty and negative effects on the environment and ecosystems. In this direction, substances from natural products have gained significant attention as eco-friendly corrosion inhibitors for aluminum and other metallic materials [15] [16] [17] [18]. Saccharides, polysaccharides and derivatives from reducing sugars, such as glucose, fructose and maltose [19], natural glucomannan [20], chitosan and its derivatives [21] [22], carboxylic acids [6], tannins and other polyphenolic compounds [23], as well as plant extracts [24] and seeds [25] have been reported to interact and protect metals in corrosive media.

In this study Crocin, a natural organic substance from the flowers of *Crocus sativus* (saffron), is assessed as green anticorrosive inhibitor for AA1050, AA5083, AA5754 and AA6082 alloys in sodium chloride solution. Crocin is a water-soluble carotenoid, used in nutritional supplements due to its antioxidant and neuro-protective action [26] [27] [28] [29]. It has been also tested previously for the corrosion protection of the AA1050 aluminum alloy, and found to present corrosion inhibiting action [30]. The antioxidant behavior of Crocin has been attributed to the extended π -electron conjugated system of its polyene chain, while the sugar moiety of its molecule is assumed to have significant contribution to its action, too [31].

Crocin is tested for its effectiveness against corrosion of AA1050, AA5083, AA5754 and AA6082 aluminum alloys in 0.01 M NaCl solution at room temperature. Crocin was added in the corrosive solution at a concentration of 1.25 mM, which has already proven to be an optimum one for the protection of the AA1050 alloy in similar testing conditions [30]. Experimental methods used were Linear and Tafel polarization, mass loss measurements, Scanning Electron Microscopy, Energy Dispersive Spectroscopy, Stereomicroscopy and Glossiness measurements.

2. Materials and Methods

Test specimens were coupons of AA1050, AA5083, AA5754 and AA6082 aluminum alloys. The %wt composition of the aluminum alloys specimens is presented in **Table 1**. All samples were mechanically polished, degreased and cleaned with acetone and deionized water.

The corrosive solution (CS) was 0.01 M NaCl p.a., whereas the inhibiting solution (IS) was 0.01M NaCl with 1.25 mM Crocin, supplied from Sigma-Aldrich (**Figure 1**).

Table 1. Composition (%wt) of aluminum and aluminum alloys.

| Type | Si | Fe | Mn | Mg | Cu | Ti | Cr | Zn | Al |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|----------|
| AA1050 | 0.168 | 0.245 | 0.003 | 0.001 | 0.001 | 0.006 | 0.002 | 0.013 | balanced |
| AA5083 | 0.133 | 0.318 | 0.523 | 4.698 | 0.061 | 0.015 | 0.061 | 0.117 | balanced |
| AA5754 | 0.125 | 0.273 | 0.142 | 2.939 | 0.017 | 0.014 | 0.052 | 0.008 | balanced |
| AA6082 | 0.900 | 0.430 | 0.460 | 0.800 | 0.080 | 0.030 | 0.020 | 0.050 | balanced |

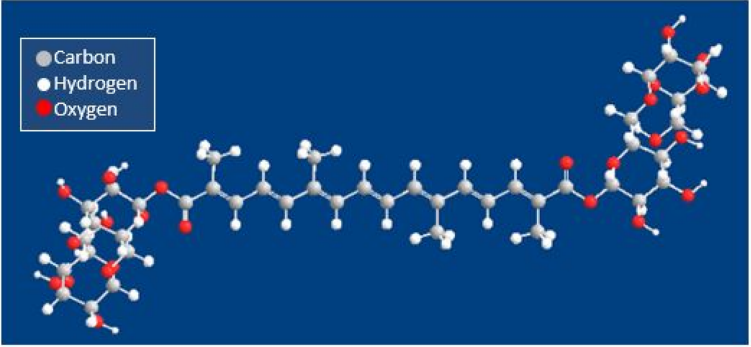
| Name | Chemical structure |
|------------|---|
| Crocin |  |
| IUPAC name | bis[(2S,3R,4S,5S,6R)-3,4,5-trihydroxy-6-[[[(2R,3R,4S,5S,6R)-3,4,5-trihydroxy-6-(hydroxymethyl)oxan-2-yl]oxymethyl]oxan-2-yl]] (2E,4E,6E,8E,10E,12E,14E)-2,6,11,15-tetramethylhexadeca-2,4,6,8,10,12,14-heptaenedioate |

Figure 1. Chemical structure of Crocin (8,8-diapo-8,8-carotenoid acid, $C_{44}H_{64}O_{24}$).

For the study of the corrosion behavior of the aluminum alloys, the electrochemical methods of Linear and Tafel Polarization were employed, using the Gamry Interface 1000 and the DC105 Corrosion Software. The three-electrode setup consisted of Ag/AgCl reference electrode (RE), Pt counter electrode (CE) and aluminum alloy specimen working electrode (WE) with an active surface area of 1 cm^2 . Open Circuit Potential (OCP) was recorded for one hour until an almost constant value was attained. Linear Polarization scan was performed at scan rate of 0.125 mV/s for $\pm 0.02 \text{ V}$ vs. OCP and Tafel scan was carried out at scan rate of 0.166 mV/s from -0.250 V to $+0.400 \text{ V}$ vs. OCP.

Inhibition efficiency (IE_e) of Crocin from corrosion currents obtained from Tafel curves was determined by the equation:

$$IE_e (\%) = \left[\frac{(I_{\text{corr,CS}} - I_{\text{corr,IS}})}{I_{\text{corr,CS}}} \right] \times 100 \quad (8)$$

where $I_{\text{corr,CS}}$ and $I_{\text{corr,IS}}$ are the corrosion currents in the corrosive and inhibiting solutions, respectively.

Mass loss measurements were performed with aluminum alloys samples immersed for 13 weeks in the corrosive and the inhibiting solutions at room temperature. Their mass was measured weekly after being descaled in HNO_3 according to ISO 8407 standard. Three independent experiments were performed for each alloy in order to ensure reproducibility of the results and an average value of the mass was calculated.

Inhibition efficiency (IE_m) of Crocin from mass loss measurements was assessed using the equation:

$$IE_m (\%) = \left[\frac{(\Delta m_{\text{CS}} - \Delta m_{\text{IS}})}{\Delta m_{\text{CS}}} \right] \times 100 \quad (9)$$

where Δm_{CS} and Δm_{IS} are the mass loss values in the corrosive and inhibiting solutions, respectively.

Stereomicroscopy via an Olympus SZ61 Stereo Microscope equipped with a camera (Image Pro Plus—Infinity Capture) and Scanning Electron Microscopy (SEM) equipped with Energy Dispersive Spectroscopy (EDS) (JEOL JSM-6510

LV—EDAX/Oxford Instruments) were employed for observation and analysis of the specimens' surface.

Moreover, surface gloss of specimens was measured using a TG 60/268 Lovibond gloss meter and GQC6 Quality Control Software, according to ISO 7668 standard. The gloss meter determines the intensity of light reflected from the aluminum alloy surface at three measurement angles of 20°, 60° and 85°, giving information on the status of the surface [32].

3. Results and Discussion

Tafel potentiodynamic polarization curves for all aluminum alloys specimens in contact with corrosive and inhibiting solutions are presented in **Figure 2**. Overpotential (η) is calculated as the difference between the potential (E) and the corrosion potential (E_{corr}):

$$\eta = E - E_{\text{corr}} \quad (10)$$

In the corrosive solution, no sign of passivation is observed in the anodic branch of the Tafel curves for the AA5083, AA5754 and AA6082 alloys, indicating an extended dissolution of them. Instead, the AA1050 alloy presents a typical Tafelian shape, where differentiation between the pitting potential (E_{pit}) and the

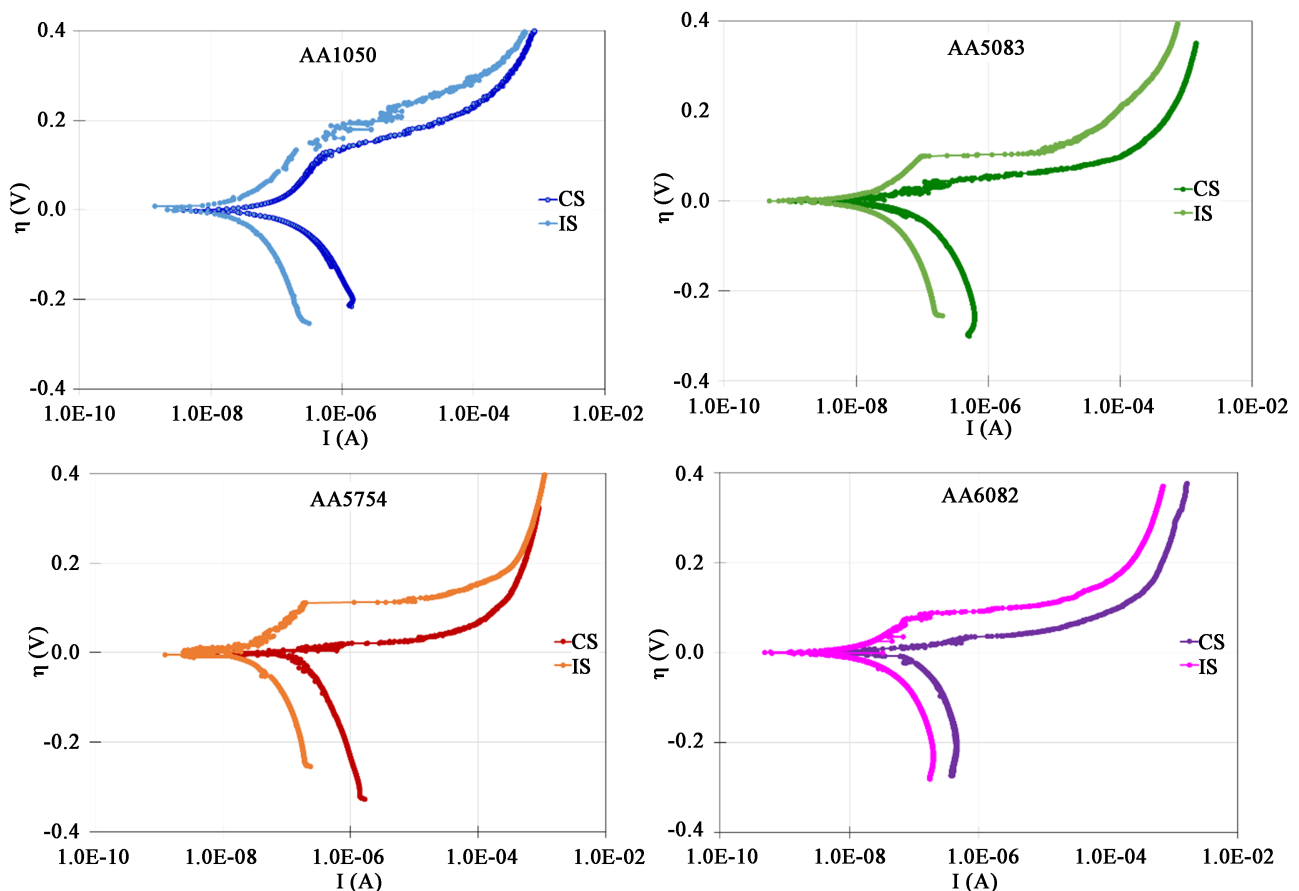


Figure 2. Tafel Polarization curves for AA1050, AA5083, AA5754 and AA6082 aluminum alloys test specimens immersed in the corrosive (CS) and the inhibiting (IS) solutions.

corrosion potential (E_{corr}) is observed, confirming the better corrosion resistance of the commercially pure aluminum alloy [17] [22].

In the inhibiting solution a distinction between the values of E_{pit} and E_{corr} is visible in all cases, which shows the protective action of Crocin. Moreover, the current has lower values both in cathodic and anodic branches of the Tafel curves than in the corrosive solution for all the aluminum alloys, suggesting that Crocin acts as a mixed type inhibitor. The above results indicate the formation of a protective film on the surface of the alloys via adsorption of Crocin.

Inhibition efficiency ($IE_e(\%)$) of Crocin evaluated from corrosion currents is displayed in **Figure 3**. All values are high, fluctuating from 80% for AA5754 to 92% for AA5083 and AA6082 specimens. These results confirm the high protective capacity of Crocin, which covers a large part of the aluminum alloys surface available for corrosion reactions and blocks the access of chloride ions.

Mass loss measurements performed weekly for 13 weeks of specimens' immersion in the NaCl corrosive environment, in the absence or presence of Crocin, attested the inhibiting action of Crocin, as noticed by the values of mass loss given in **Figure 4**. The corresponding ($IE_m(\%)$) inhibition efficiencies vary between 80 and 95%, values that are similar to those obtained from the electrochemical measurements, and high enough to verify again the inhibitive character of Crocin. The apparent exquisite protection of Crocin in the case of the AA5083 specimens compared to the other aluminum alloys in the corrosive solution, is accredited to the stronger tendency of this alloy for corrosion, due to its higher Mg-content.

Photographs of aluminum alloys specimens after immersion for 13 weeks in the test solutions and cleaning are presented in **Figure 5**. Differences in surface appearance of all alloys are obvious: specimens in corrosive solution are blurry, dark, with corrosion products accumulated on the surfaces, whereas specimens

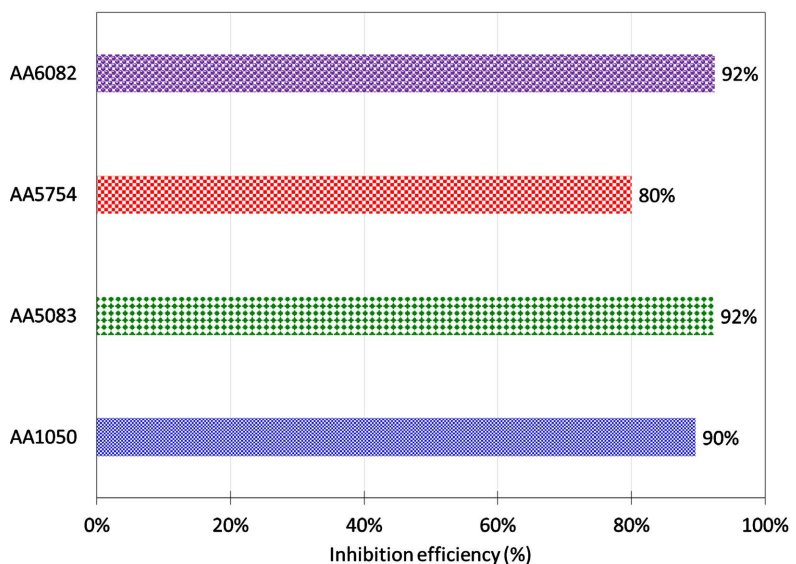


Figure 3. Inhibition efficiency (%) of Crocin for AA1050, AA5083, AA5754 and AA6082 aluminum alloys specimens calculated from corrosion currents.

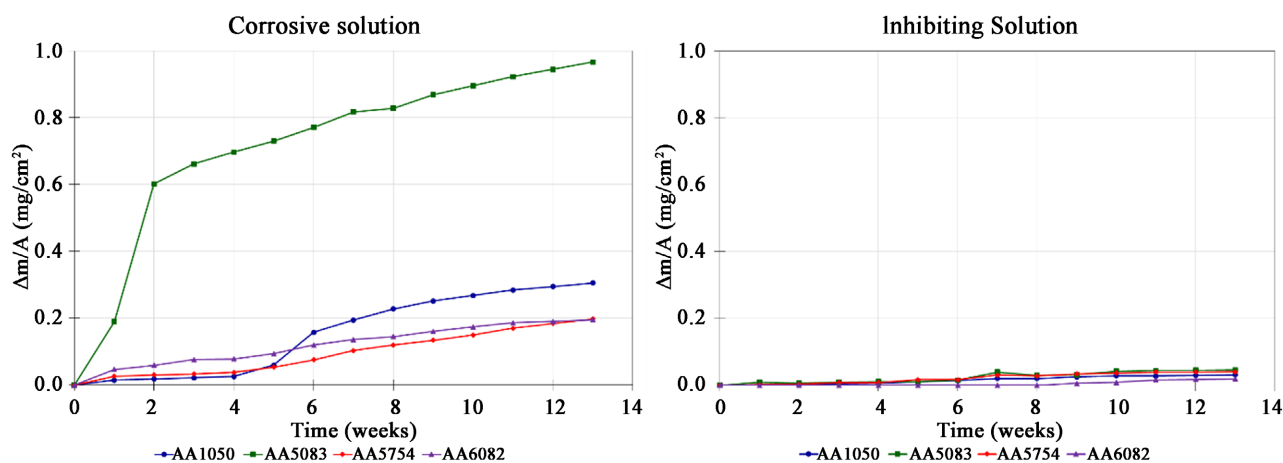


Figure 4. Mass loss of the AA1050, AA5083, AA5754 and AA6082 aluminum alloys specimens vs. immersion time in the corrosive and inhibiting solutions.

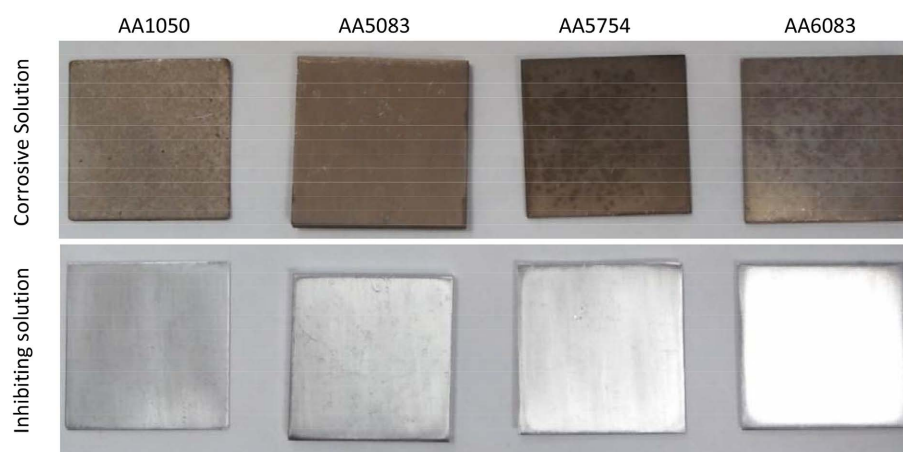


Figure 5. AA1050, AA5083, AA5754 and AA6082 aluminum alloys specimens after immersion in the corrosive and inhibiting solutions for 13 weeks and cleaning.

in the inhibiting solution of Crocin are light, shiny as new, with no visible corrosion signs on them.

The above visual qualitative assessment of the surface morphology of the aluminum alloys specimens was quantified via glossiness measurements (**Figure 6**). Important differences in Glossiness Units (GU) values are detected between the specimens immersed in Crocin solution and the ones in the corrosive solution. Values in the inhibiting solution are always higher than in the corrosive solution, from up to about 50 times in the 20°-angle for the AA5083 and AA5754 alloys to less than twice in the 85°-angle for the AA1050 alloy. Specimens exposed to the corrosive solution have lower GU values and a matt appearance, attributable to augmented scattering of light on their surfaces, indicating the presence of corrosion products (oxides and hydroxides) and defects on them. On the contrary, specimens' surface in the presence of Crocin reflects the major part of the incident light, resulting in shiny appearance.

Scanning Electron Microscopy images at 200× magnification, obtained from

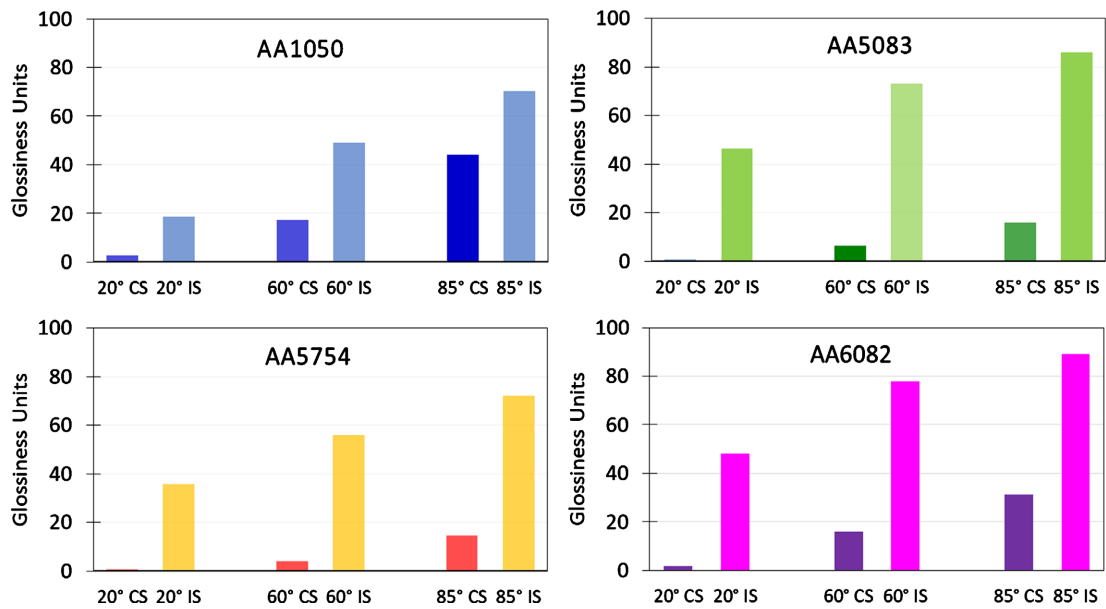


Figure 6. Glossiness of AA1050, AA5083, AA5754 and AA6082 aluminum alloys specimens after immersion in the corrosive and inhibiting solutions for 13 weeks.

the above specimens, are presented in **Figure 7**. Visualization of surface morphology reveal minimum local modifications, without any traces of localized pitting corrosion in the presence of Crocin, in comparison to the material topology with evident signs of corrosion in the absence of Crocin.

Elemental analysis by EDS showed that the surfaces of all samples immersed in the corrosive solution had Al/O weight ratios lower than those before their exposure to the testing solution, indicating the existence of corrosion products rich in O on them. On the contrary, the surfaces of the samples immersed in the Crocin containing solution had similar chemical composition with the initial uncorroded surfaces of the alloys. For example, in the case of AA1050 specimens, the Al/O ratio determined on the surface exposed in the corrosive solution is reduced by 30% compared to that on the initial surface, whereas this ratio is reduced by only 5% on specimens in the inhibiting solution. Moreover, a depletion of the alloying elements (Fe, Mg, and Mn) in the case of the AA5083, AA5754 and AA6082 alloys was found in the corrosive solution, whilst this phenomenon is limited in the inhibiting solution. For example, in the case of AA5754 specimens, the Al/Mg ratio determined on the surface exposed in the corrosive solution is increased by 90% compared to that on the initial surface while by only 3.5% on specimens in the inhibiting solution [33] [34] [35] [36]. These results demonstrate the influence of Crocin against dealloying and selective removal of alloying elements.

All the above results validate the protective action of Crocin against chloride-induced corrosion of the examined AA1050, AA5083, AA5754 and AA6082 aluminum alloys. Considering the chemical structure of Crocin (**Figure 1**), it could be assumed that its inhibiting action is attributed to its chemical bonding with the alloys surface, through the hydroxyl groups of the extreme glucose rings

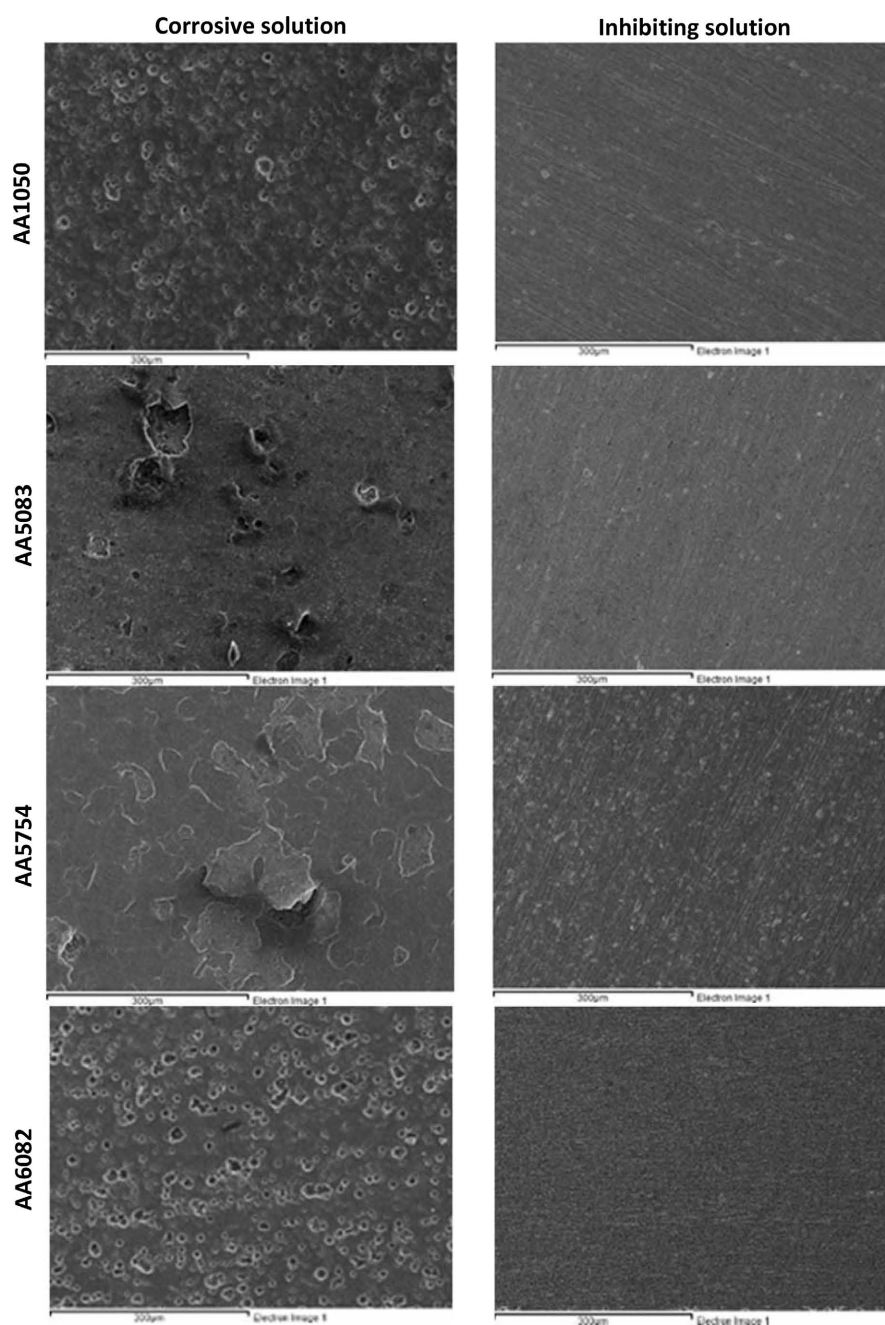


Figure 7. SEM micrographs (200×) of AA1050, AA5083, AA5754 and AA6082 specimens' surfaces after immersion in the corrosive and inhibiting solutions for 13 weeks.

of its molecules. This process is assisted by the electron donating character of Crocin, due to the π -conjugated system of its polyene chain, which is extended to the carbonyls of the esterified groups of its molecules. Thus, a well-attached on the surfaces Crocin layer, which covers their largest part, is formed, resulting in the aluminum alloys protection against corrosion. The above findings are in accordance with the antioxidant function of Crocin against free radicals, mentioned by Akhtari *et al.* [31].

Moreover, the increased concentration of Crocin detected on points where

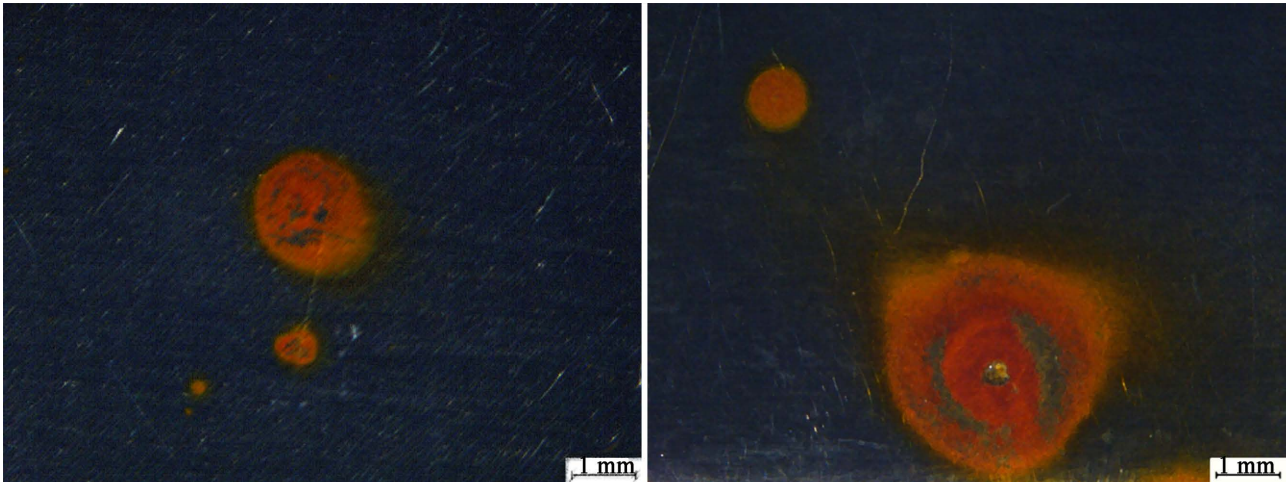


Figure 8. Crocin adsorbed on surface defects of AA1050 (left) and AA6082 (right) specimens after immersion in the inhibiting solution for 13 weeks, as observed by Stereomicroscopy.

pitting corrosion has initiated (**Figure 8**), indicates that Crocin adsorbs more intensely on these points. This could be attributed to the interactions of Crocin and its cation with aluminate ions ($\text{AlO}\cdot\text{O}^-$), which are the main corrosion products formed on the aluminum surface in aqueous environment of chloride ions [6]. Thus, even in the case where pitting corrosion has initiated, Crocin impedes the further progress of corrosion, by blocking the access of the chloride ions to the surface.

4. Conclusions

This study evidences the effectiveness of Crocin as an eco-friendly corrosion inhibitor for the AA1050, AA5083, AA5754 and AA6082 aluminum alloys in the NaCl corroding environment. Crocin was found to retard the dissolution of the natural protective aluminum oxide layer, obstruct pitting corrosion and reduce the formation of corrosion products. Particularly:

- Electrochemical measurements showed that Crocin acts as a mixed type inhibitor for the corrosion of all the examined aluminum alloys in the 0.01 M NaCl solution.
- The inhibition efficiency of Crocin calculated from corrosion currents and mass loss measurements is high, varying from 80% to 95%.
- Observation and analysis of the alloys surfaces after immersion in the corrosive and inhibiting solutions for 13 weeks revealed less corroded surfaces in the presence of Crocin, without any signs of localized pitting corrosion and with a limited depletion of the alloying elements in the case of the AA5083, AA5754 and AA6082 alloys.
- Inhibiting action of Crocin is attributed to the formation of a protective film on the specimens via chemisorption of its molecules on the alloys surface. Moreover if pitting corrosion initiates, Crocin is adsorbed preferentially on the defects of the specimens' surface, blocking the access of the chloride ions and impeding the further progress of corrosion.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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