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Corrosion of aluminum for beverage packaging in acidic media containing chlorides and copper ions

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Abstract

Corrosion of aluminum packaging plays an important role concerning economic and health issues. The effect of aggressive ions on the behavior of the AA3104-H19 alloy provides knowledge to the food and packaging industries to improve materials and also minimizes losses associated with the corrosion. This study evaluated the interaction of the AA3104-H19 alloy and beverage using model solutions containing chloride and copper at levels close to those found in soft drinks. The results show changes in the material at very low ion concentrations, as well as the synergistic effect of both ions in the solution.

Practical applications

Corrosion of soft drink aluminum cans is a severe industrial problem that results in huge losses of food and material, having important financial and environmental impacts. Information about the corrosion electrochemical behavior of aluminum alloys used for food and beverage is very scarce. This study evaluated the interaction of the AA3104-H19 alloy and model solutions containing chloride and copper at levels close to those found in soft drinks. The obtained results will allow researchers to better understand the interaction between beverages and packaging, also improving the packaging materials (metal and polymeric coating) to be used in contact with soft drinks. It also helps the industry to better design both packaging and products.

1 | INTRODUCTION

In aluminum alloys, corrosion occurs more easily when in contact with more electropositive elements, such as chlorine and copper. However, aluminum is a relatively corrosion-resistant metal. Consequently, the medium to which it is exposed must offer favorable conditions for the development/beginning of this process.

Acids found in foods also favor the corrosion process. Low pH favors the dissolution of the aluminum oxide layer, which acts as a natural passivation layer for the material. Moreover, anions of the acid and the Al^{3+} ions can react forming complexes. The acid most commonly used in the production of soft drinks and other beverages is citric acid (Lindsay, 2008). Various beverages have pH values equal to or less than 3.5 (Soares, Anjos, Faria, & Dantas, 2016). Under these conditions, the aluminum oxide layer dissolves (Solmaz, Kardas, Yazici, & Erbil, 2008), a process to which aqueous media with a pH lower than 4.5 is conducive (Jellesen, Rasmussen, & Hilbert, 2006). This process results in a deficient passivation layer on the internal surface of the aluminum container, thus, facilitating the development of the corrosion process when metallic exposure occurs.

Aluminum containers for foods and beverages have an internal polymer coating, with the purpose of minimizing the interaction of the metallic material with the product. However, corrosion sometimes occurs, bringing with it the necessity of evaluating the factors involved.

Chloride is an ion present in several foods and beverages and it is frequently brought up as one of the main compounds that begin the localised corrosion process in aluminum alloys (Bakos & Szabó, 2008; Blanc & Mankowski, 1997; Elola, Otero, & Porro, 1992; Fontana, 1986; Guillaumin & Mankowski, 1999; Ishii, Ozaki, Kanedo, Fukushima, & Masuda, 2007; Mayouf, Juhaiman, & Subaybani, 2008; McCafferty, 1995; Pyun & Lee, 2001; Szklarska-Smialowska, 1999; Wong & Alkire, 1990). That is why it is crucial to determine the effects of this ion on the corrosion of the aluminum alloy used in food containers, studying its influence in conditions similar to real metal exposure ones.

The beverage and packaging industries also raise the question of the influence of copper ions in the can corrosion process, as this ion is present in some beverages. The corrosion of aluminum in contact with metallic copper is easily found in the literature, which contains various studies on Al–Cu alloys. However, unlike chloride, there are few studies involving the behavior of aluminum exposed to solutions containing copper ions (Bakos & Szabó, 2008; Hack, Corbett, & Krantz, 1998). Regarding corrosion in a medium with chloride ions, studies generally assess the occurrence of galvanic corrosion in aluminum alloys containing copper (Musa, Mohamad, Kadhum, & Chee, 2011). Various studies have investigated alloys in groups 2xxx and 6xxx. However, there are no data available for the aluminum alloy AA3104-H19.

Foods and beverages are complex matrices and the role of corrosion-accelerator elements in metallic containers should be understood. The objective of this study was to determine the interaction of the aluminum alloy AA3104-H19, utilized in beverage cans, with acidic aqueous solutions containing chloride ions, copper ions, and a mixture of the two. To represent a situation similar to the real one, the ion concentrations were set equivalent to those found in beverages. To this end, polarization tests and scanning electron microscopy with X-ray microanalysis (SEM-EDS) evaluations were performed with the purpose of observing the influence of the concentration of chlorides and copper ions on the medium and changes caused to the metallic material.

2 | EXPERIMENTAL

2.1 | Preparation of model solution and sample

The model solutions were prepared with citric acid and pH = 3.0. From this solution (so-called "blank"), solutions were prepared containing chloride ions, derived from sodium chloride (Synth, Brazil), as well as solutions containing copper ions, derived from cupric sulfate pentahydrate (Synth, Brazil).

The chloride and copper ion concentrations studied are shown in Table 1 and were determined in a preliminary study carried out by the authors (Soares et al., 2016). All chemical reagents were of analytical grade.

Samples of alloy AA3104-H19 derived from the bodies of new aluminum cans (the same packages supplied to the beverage industry) were evaluated. All samples were prepared by removing the varnish with a solvent, followed by polishing them with 600 grade emery paper and rinsing them with ultrapure water.

TABLE 1 Concentration of chloride and copper ions in the model solutions (pH 3)

Ion	Concentration
Cl^-	25, 50, 100, 250, 500, and 1,000 mg kg^{-1}
Cu^{+2}	25, 50, 100, 250, 500, and 1,000 μg kg^{-1}
$Cl^- + Cu^{+2}$	50 mg kg^{-1} (Cl^-) + 50 μg kg^{-1} (Cu^{+2}) 100 mg kg^{-1} (Cl^-) + 25 μg kg^{-1} (Cu^{+2}) 250 mg kg^{-1} (Cl^-) + 25 μg kg^{-1} (Cu^{+2}) 100 mg kg^{-1} (Cl^-) + 250 μg kg^{-1} (Cu^{+2}) 250 mg kg^{-1} (Cl^-) + 250 μg kg^{-1} (Cu^{+2})

2.2 | Polarization measurements

The aluminum sample was utilized as a working electrode (WE) and the model solutions (Table 1) were used as electrolytic solutions. A system of three electrodes comprising the WE, a platinum counter electrode (CE) and a saturated calomel electrode (SCE) as a reference (RE) was utilized to obtain the polarization curves for aluminum alloy AA3104 in the solutions studied.

The tests were carried out in an air-conditioned environment at a temperature of 23 °C. The model solution was bubbled with ultrapure nitrogen gas to remove any dissolved oxygen from the medium. The assay was carried out in a potentiostat/galvanostat (PGSTAT302N, Metrohm Autolab B. V.) with nitrogen gas filling the headspace of the cell. All experiments were repeated at least three times. All reported potentials refer to SCE.

The potentiodynamic method was applied, using a scanning rate of 1 mV/s, within the potential range between −400 mV and +400 mV with respect to the E_{corr}. The response of the current as a function of the potential applied gave rise to the polarization curves obtained. The kinetic parameters of the corrosion process were determined using the software NOVA, version 1.7 (Metrohm Autolab B. V.).

2.3 | Microscopic assessment

After the polarization curve was obtained, the aluminum samples were analyzed in a scanning electron microscope (DSM 940A, Zeiss, Germany) with an energy dispersive X-ray analyser (Link Isis, Oxford, UK). All data were obtained at 25 mm (working distance) and 20 kV (high voltage).

3 | RESULTS AND DISCUSSION

3.1 | Polarization measurements for solutions containing only Cl^- or Cu^{+2}

Figure 1 shows the polarization curves for aluminum alloy AA3104-H19 in contact with the citric acid solution containing different concentrations of chloride and copper ions. The E_{corr} and j_{corr} values were plotted in Figure 2 as functions of the concentration of the ions in the solution.

The corrosion potential (E_{corr}) of the aluminum alloy decreases as the concentration of chloride ions in the medium increases. Furthermore, this potential reduction is more abrupt at the lower Cl^- concentrations (Figure 2). Given that the concentration of citric acid is not altered in the solutions studied, the lowering of the E_{corr} values is caused exclusively by adding chloride to the medium. The Cl^- ion penetrates the aluminum oxide film, causing a breakdown in the passivation and initiating the pitting process (Khireche, Boughrara, Kadri, Hamadou, & Benbrahim, 2014).

Mayouf et al. (2008) found more negative E_{corr} values (Cit: −446 mV → Cit + NaCl: −730 mV) and an increase in the j_{corr} (Cit: 0.50 μAcm^{-2} → Cit + NaCl: 26.5 μAcm^{-2}) in a medium containing NaCl (0.1 mol L^{-1}), compared to the medium without chloride, for alloy

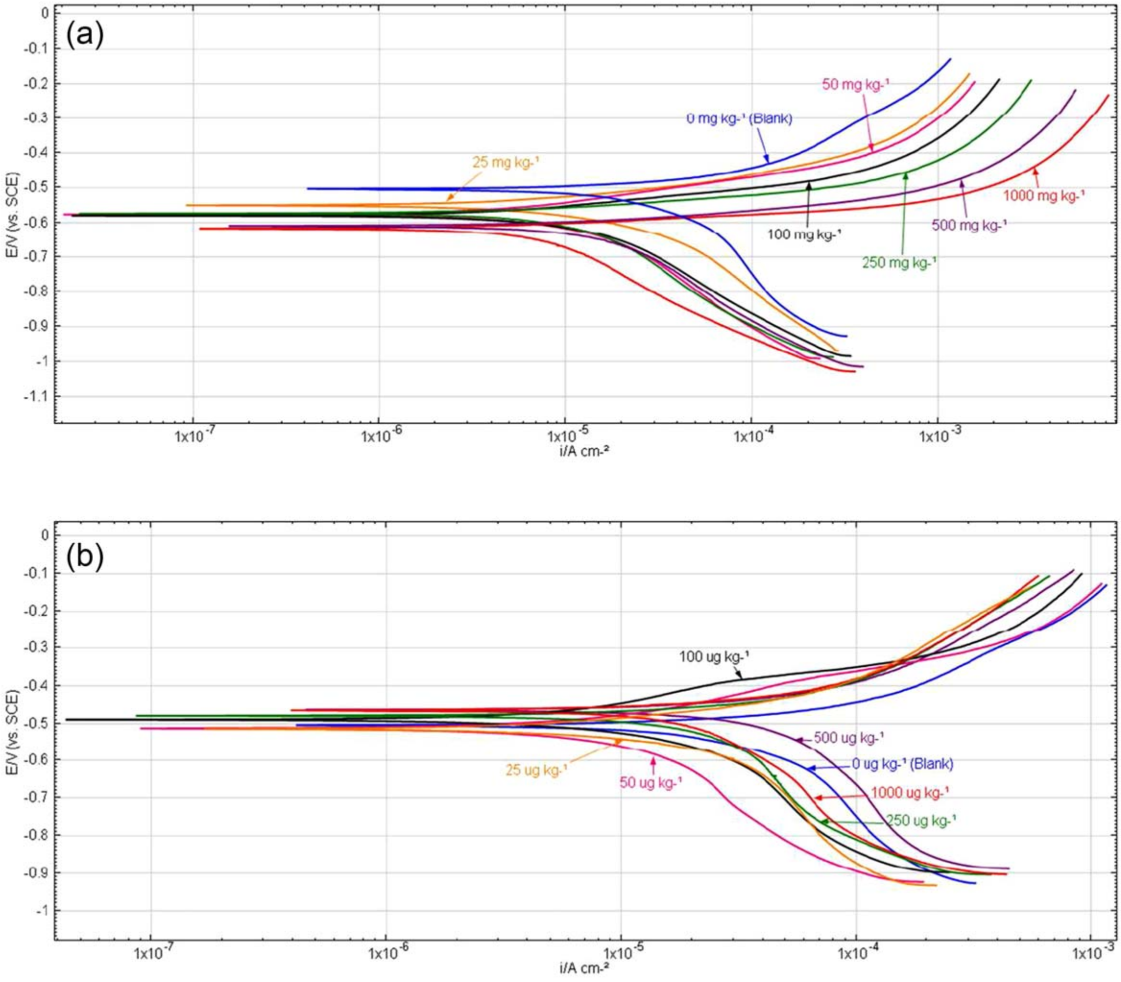


FIGURE 1 Polarization curves for different concentrations of chloride (a) and copper (b) ions in citric acid solution pH = 3.0

AA6063 in a citric acid solution in an aerated medium at 25 °C and a pH of 2.1.

The increase in the concentration of chloride ions in the solution studied here did not cause an increase in the corrosion current density (j_{corr}). However, this observation could not be incorporated into the present study, given the acidic medium studied simulating beverages, which is conducive to the dissolution of the aluminum passivation layer. Given the acidic medium studied in which partial dissociation of the citric acid occurs ($pK_{a1} = 3.15$) (Atkins & Paula, 2011) and the low concentration of chloride present in the medium, the most plausible scenario is the formation of uncharged compounds among the citrate, the Al^{+3} and the Cl^-, leaving an even lower concentration of Cl^- ions available in the solution to interact with the surface of the aluminum alloy. Such a result is important as the quantitative analysis of chloride in beverages does not consider the formation of complexes, although the citric acid is the predominant acid in beverages such as soft drinks.

The results demonstrated that the corrosion potential (E_{corr}) of the aluminum is shifted to less negative values as the concentration of copper increases (Figure 2a), which occurs because the redox potential of copper is higher than that of aluminum and because of the probable deposition of copper on the aluminum surface (Bakos & Szabó, 2008). Aluminum (Al°) undergoes an oxidation reaction, while copper (present in the solution as Cu^{+2}) undergoes a reduction reaction, which is due to the fact that aluminum is a less noble metal than copper. The product of this reaction is the dissolution of aluminum into the medium in the form of Al^{3+} and the deposition of copper in the form of metallic copper (Cu°). The corrosion current density (j_{corr}) of aluminum increases as the concentration of copper in the acidic solution increases, reflecting its conducive effect on the corrosion of aluminum.

3.2 | Measurements of polarization in solutions containing Cl^- and Cu^{+2} in association

Based on the concentrations that matched those found in beverages (Soares et al., 2016) and for which significant responses were observed in polarization assays with solutions containing only copper or chloride ions, some concentrations were selected to study the influence of chloride and copper ions present simultaneously. Figure 3 shows the polarization curves obtained for alloy AA3104-H19 with the solutions utilized.

Figure 4a shows the result of the corrosion potential of the solutions containing the relevant ions in association, compared with the results obtained from the acidic solution containing only one of the ions.

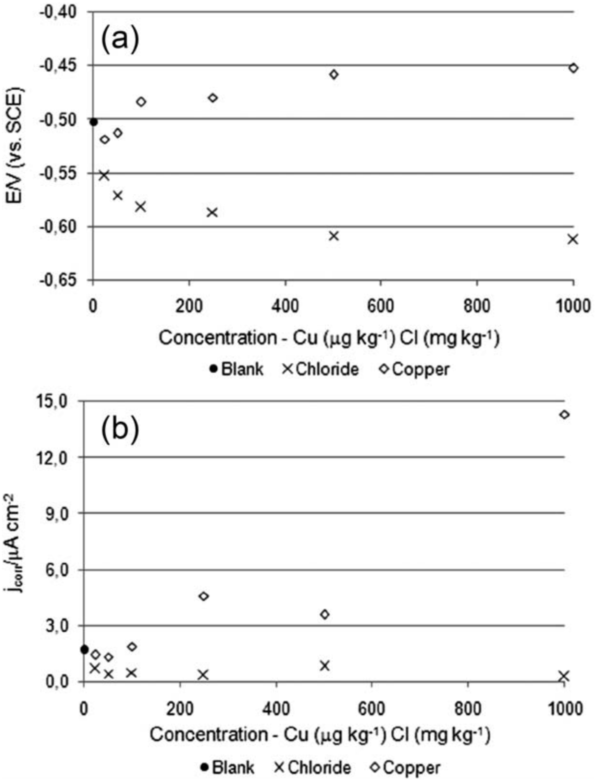


FIGURE 2 Corrosion potential (a) and corrosion current density (b) of the aluminium as a function of the concentration of chloride and copper ions

In all concentrations studied, it was observed that the corrosion potential of aluminum alloy AA3104-H19 decreased with respect to the potential observed with these ions separately. Chloride and copper ions are corrosion accelerators, and the presence of both in an aqueous acidic medium causes a synergistic effect on the observed corrosion potential.

The relationship between the observed E_{corr} and the concentrations of Cl^- and Cu^{+2} becomes more important when the increase in the concentration of copper ions in the medium is taken into account. While the increase in the concentration of copper ions in the acidic solution results in an increase in the measured E_{corr} for aluminum, the addition of this ion in a medium also containing the Cl^- ion leads to more negative values for the potential, including in comparisons between solutions containing the same concentration of Cl^-. It demonstrates that the presence of copper ions in the solution is extremely important to aluminum corrosion in chloride medium. It is important to observe that the concentrations of the copper ion studied here are those found in foods and beverages. In fact, these concentrations are lower than those generally evaluated in corrosion studies, highlighting the importance of the observed results.

Figure 4b shows the corrosion current density for the solutions containing a mixture of chloride and copper ions compared to the values of the solutions containing only one of the ions. It is observed that the corrosion current density found in the solutions containing a mixture of chloride and copper ions is lower than the values found for the solutions containing only one of these ions. Given that the corrosion current density is related to the corrosion rate of the material, the results lead to the conclusion that the corrosion rate is lower when the two elements are present in the medium.

Based on the electrochemical behavior observed, the phenomenon of corrosion probably occurs similarly to the galvanic corrosion (Bakos & Szabó, 2008), in which corrosion happens at a faster rate when aluminum is in electric contact with a more noble metal (Davis, 1999). As the concentration of copper ions in the medium rises, this metal begins to deposit over the aluminum, creating microareas of copper–aluminum contact. This microscopic region exposed to the solution containing chloride ions acts like a galvanic cell, resulting in the localised corrosion of the aluminum.

These results demonstrate that it is necessary to know the physical–chemical characteristics of foods and beverages that will be in contact with aluminum to minimize the package corrosion. Chloride is an element exhaustively found in food and beverages. For soft drinks, its great source is the water used in the production. Nevertheless, other elements, such as copper, can also be found in foods and beverages, thus justifying the knowledge of metal behavior in medium containing

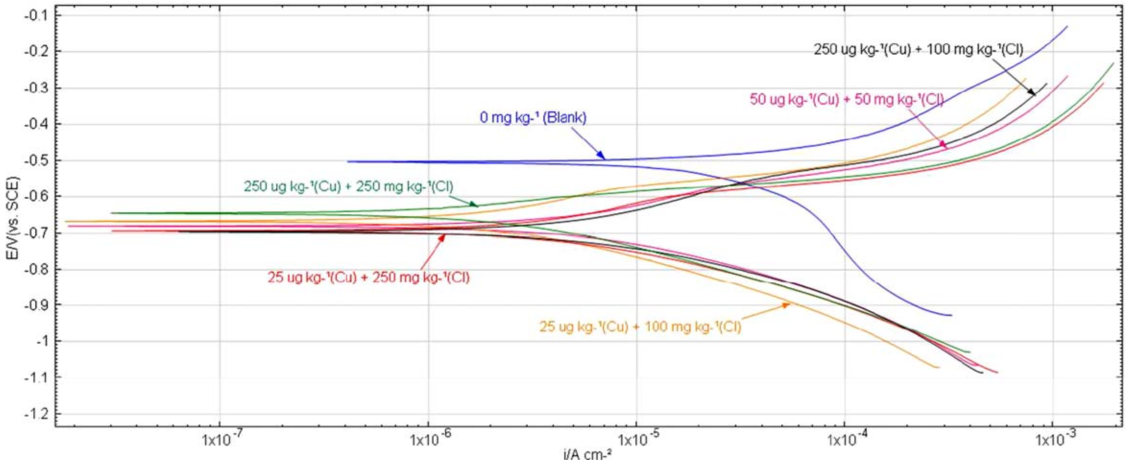


FIGURE 3 Polarization curves for citric acid solutions (pH = 3.0) with different combinations of chloride and copper in the same solution

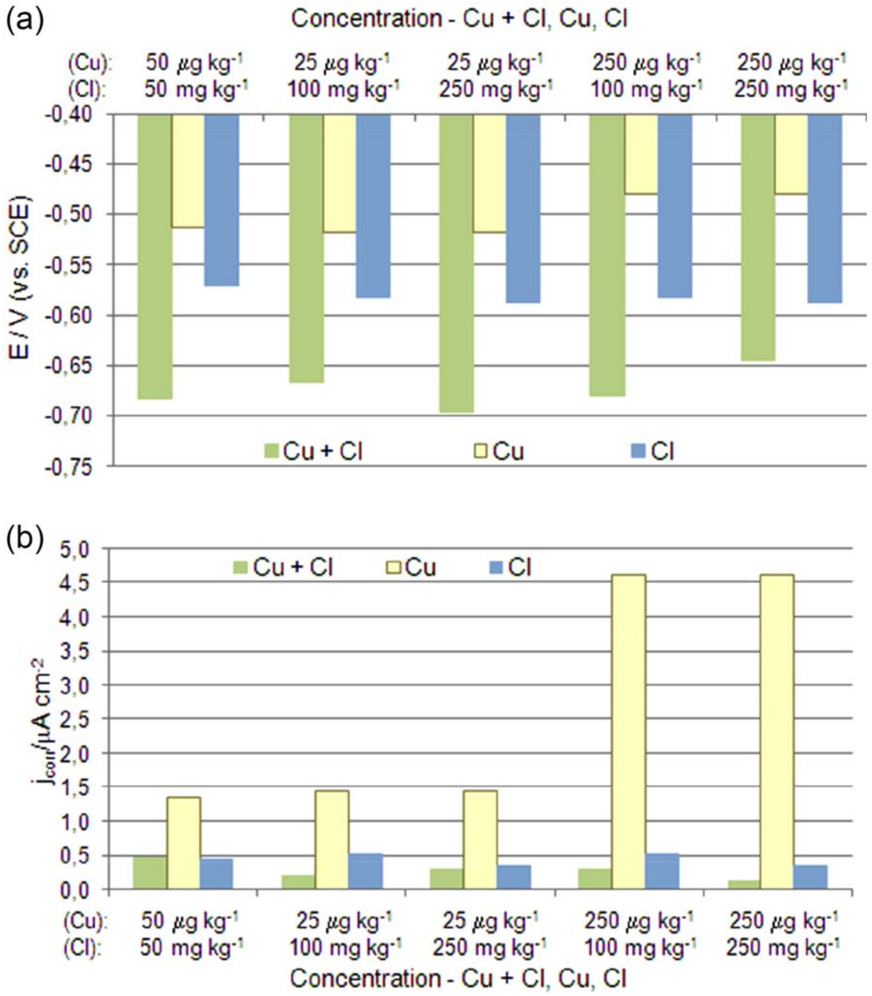


FIGURE 4 Comparison between the corrosion potential (a) and corrosion current density (b) of the aluminium, for different concentrations of chloride and copper ions and their mixture

these elements and considering the real concentration of use. This evaluation is important to minimize the occurrence of the problem and, consequently, to reduce the packaging damage and the loss of food and beverages.

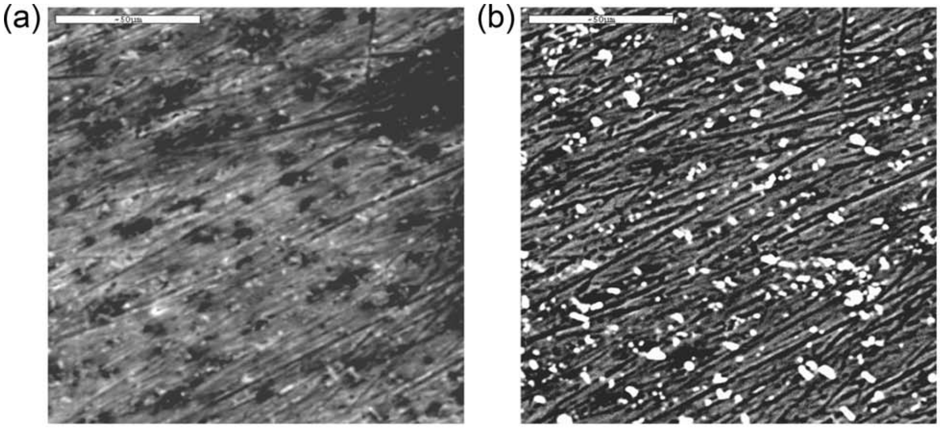
3.3 | Microscopic assessment

Figure 5 shows some representative images of the surfaces of aluminum alloy AA3104-H19 after the polarization assay with a solution containing only citric acid, with a pH of 3.0 (blank). Small dark points on the surface of the aluminum can be observed, indicating the occurrence of corrosion on the material. Furthermore, on the image generated by the Back-Scattered Electron (BSE) signal, there are various white spots. The microanalysis of which indicated a greater concentration of alloy elements, such as manganese and iron. The blank solution resulted in a small-scale surface corrosion than the other samples. This is due to the fact that the blank solution did not contain corrosion-catalyzing ions, although the acidic medium is conducive to the dissolution of the Al_2O_3 layer, which protects the surface of the aluminum.

In general, the surfaces of all samples that come into contact with chloride solutions were affected by the action of the ion present in the solution, resulting in regions with clusters of pits. This was observed even at the lowest concentration studied (25 mg $kg^{-1} = .0025\%$ Cl).

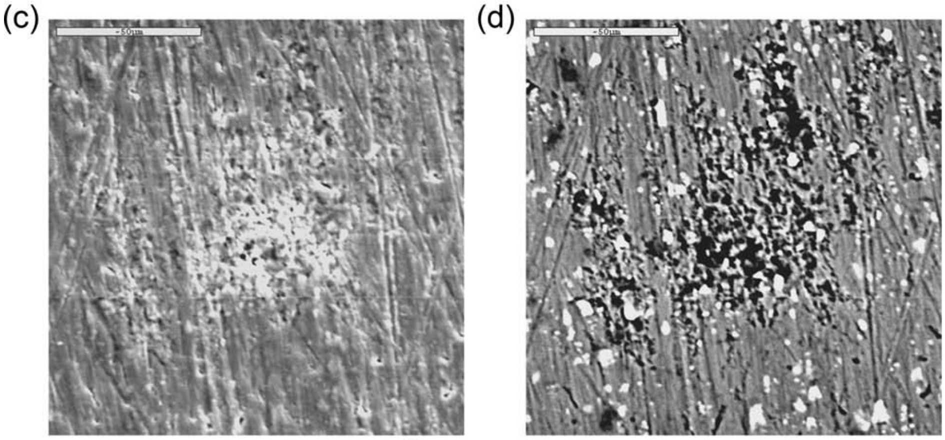
In this case, it is also worth noting the profile and extent of the surface changes, as the SEM images were obtained according to the same acquisition parameters. The solution containing the chloride ion caused greater changes to the metallic surface when compared to the solutions containing copper or containing only citric acid (blank). It results in the formation of several pits, making the appearance of the aluminum surface similar to that of generalized corrosion. The study by Mayouf et al. (2008) demonstrated that the association of chloride ions (.01 mol L^{-1}) with citrate ions (.01 mol L^{-1}) promoted greater corrosion in the aluminum alloy AA6063 than a solution containing only chloride ions. The authors attributed this greater corrosion to the addition of more anions, which may form different compounds with aluminum (Al^{3+}), acting in synergy with the chloride ions.

The microanalysis performed in regions containing pits identified the presence of chlorine and oxygen elements (Figure 6a), confirming the action of chloride on the corrosive process that developed in the



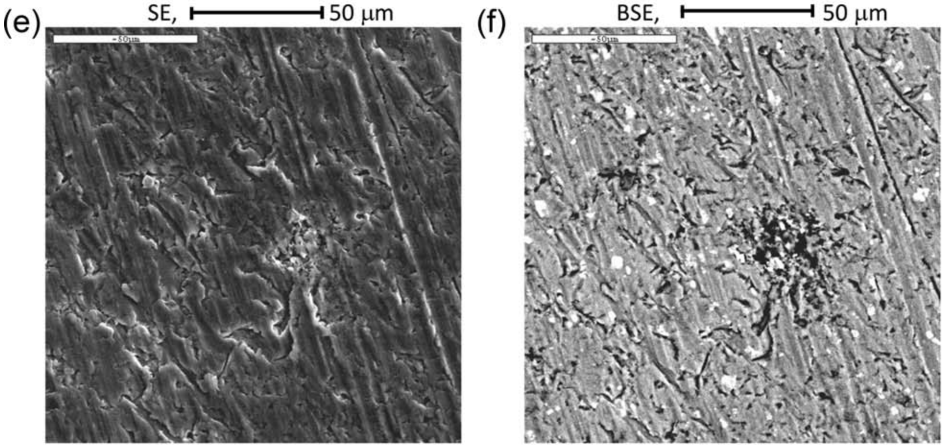
Blank, SE, 50 μm

Blank, BSE, 50 μm



Chloride solution - 25 mg kg^{-1}, SE, 50 μm

Chloride solution - 25 mg kg^{-1}, BSE, 50 μm



Copper solution 25 μg kg^{-1}, SE, 50 μm

Copper solution 25 μg kg^{-1}, BSE, 50 μm

FIGURE 5 Samples of alloy AA3104-H19 after the assay in citric acid solution without ions (a, b), with chloride (c, d), and with copper ions (e, f)

samples studied. The product of corrosion is a precipitate formed by aluminum and chloride. According to Foley and Nguyen (1982), the compound $Al(OH)_2Cl$ (aluminum oxychloride) may form in the reaction of aluminum in an aqueous medium containing chloride.

The microscopic analysis performed on the samples that underwent the polarization assay demonstrated that the changes, which occur on the surface of alloy AA3104-H19 when in contact with a copper-containing acidic solution, showed lesser attack than that caused by the chloride-containing acidic solution.

The attack of the copper-containing acidic solution causes changes throughout the surface of the aluminum. However, a more intense corrosion is observed in some points (Figure 5e,f).

In the microanalysis spectra obtained from the samples tested with a solution containing copper ions, this element's peak was not

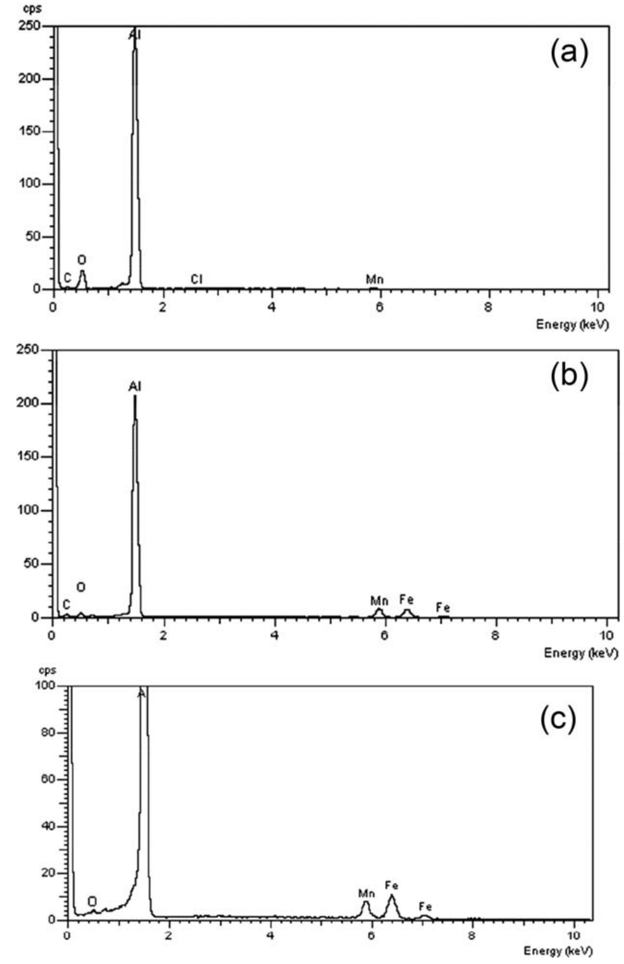


FIGURE 6 Examples of spectra obtained from the analyzed surface regions of the sample: (a) pitting region (after contact with Cl), (b) white spots, and (c) corrosion region (after contact with Cu)

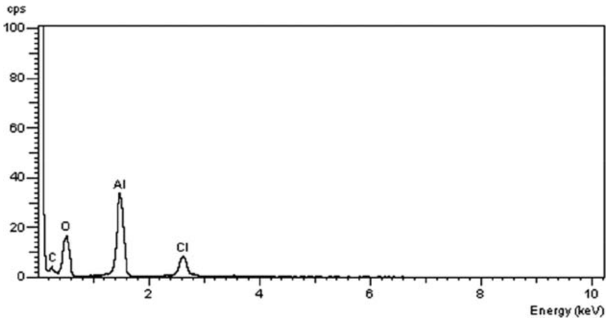


FIGURE 8 Sample of spectrum obtained from the corrosion regions after the polarization assay with both chloride (250 mg kg^{-1}) and copper (25 mg kg^{-1}) ions

observed via the microanalysis system used (EDS, which allows for the detection of chemical elements with a concentration that equals or exceeds .01% on the test surface). However, the nondetection of this element does not exclude the possibility of its presence in the material's region of corrosion.

The assessment via scanning electron microscopy, performed after determining the polarization curves, showed that the topography of the material after contact with the solutions containing chloride and copper ions had a corroded region (Figure 7), similar to that observed when the material came into contact with the solution containing only one of these ions (Figure 5).

In the analysis of the sample tested with the acidic solution containing 250 mg kg^{-1} of chloride and 25 μg kg^{-1} of copper ions, the presence of a residue on the material was observed. The microanalysis of this residue showed that it was composed of carbon, oxygen, aluminum, and chlorine (Figure 8). These results indicate that the residue analyzed is the corrosion product formed and the carbon originating from the citric acid is added to the solution. In this case, what was found was a very small point of corrosion, which developed inward into the material, not extending to the surface around the residue. This kind of topography is generally observed in aluminum cans which have undergone corrosion due to contact with the beverage.

Although the material came into contact with the solution containing copper ions, the latter was not found through the EDS system

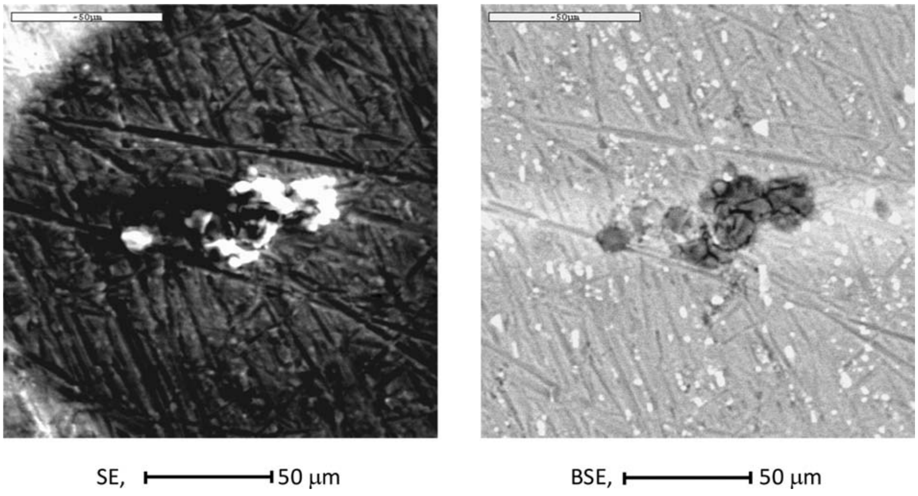


FIGURE 7 Surface of the aluminium after the polarization assay, in the presence of the acidic solution containing both chloride (250 mg kg^{-1}) and copper (25 μg kg^{-1}) ions

(Figure 8). It leads to the conclusion that the concentration of copper present in the locus of corrosion (from the aluminum alloy and the solution analyzed) is probably lower than .01% (100 mg kg^{-1}), which is the detection limit of the analytical technique used.

4 | CONCLUSIONS

The following points can be concluded:

1. The addition of Cl^- into the citric acid solution with a pH of 3.0 resulted in the reduction of the E_{corr}. The highest change was observed at the lowest concentration studied (25 mg kg^{-1}). The E_{corr} reduction becomes less significant at higher concentrations of Cl^- ion. Despite the change caused in the E_{corr}, the corrosion current density remained essentially constant in all concentrations studied.
2. The addition of cupric ions has a significant effect on the parameters studied, especially the observed change in the j_{corr}. These data are a significant contribution to future studies assessing the corrosion of aluminum cans, due to the low concentration of cupric ions in the medium, as well as in beverages.
3. It was observed that Cl^- and Cu^{+2} act synergistically on the E_{corr}, resulting in more negative values in the solution containing both ions than in the solutions containing only one of them. However, j_{corr} values remained very close to those found in the solution of only Cl^-.
4. The microscopic analysis showed that corrosion was localised, but several points were developed by the analyzed surface. The presence of Cl in the corroded region was confirmed.

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