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MECHANISMS OF THE MICROBIAL CORROSION OF ALUMINUM ALLOYS

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ABSTRACT

Pitting potential was used recently to assess the aggressiveness of the biological species in the microbial corrosion of aluminum alloys in hydrocarbon/water systems. This parameter is analysed here in relation to the electrolyte composition of the medium, especially with reference to chloride, nitrate, and phosphate levels. Chloride anions, usually present in hydrocarbon fluids and culture media, control the pitting process Nitrates and phosphates, used as nutrients in culture media, act as pitting inhibitors displacing the pitting potential value to high anodic regions at a low chloride/inhibitor ratio. In these conditions, microbial uptake of pitting inhibitors is the principal cause of the pitting potential decrease during microbial growth. At high chloride/inhibitor ratio or in the absence of inhibitor, metabolites production is the most important factor in the aggressiveness increase. Dodecanoate anions recently proposed as the main metabolic product leading to the passivity breakdown of aluminum alloys, show no effect on the pitting process, either in the absence or presence of chloride. The acidic metabolites produced by "Cladosporium resinae" are able to facilitate the breakdown of the passive oxide film by chloride anions decreasing the pitting potential value. The action of unidentified metabolic products or cellularlysis compounds on the corrosion process can be also envisaged.

INTRODUCTION

One of the most interesting and extensively studied cases of microbiological corrosion is that of aluminium and its alloys by microbial contaminants of kerosene fractions.

During some decades ago, a great amount of information has been collected on the microbiological aspects of the principal contaminant of jet fuels, the fungus "Cladosporium resinae" (C. resinae). Among others, taxonomic characteristics (Parbery, 1969), temperature, pH and agitation influence on growth (Parboery, 1971), survival of spores (Sheridan, 1972), and metabolic pathways of hydrocarbon degradation (Tamaka *et al.* 1968; Teh and Lee, 1973) have been specifically studied. The biodegradation of n-alkanes normally proceeds by a monoterminal attack. A primary alcohol is usually formed, followed by an aldehyde and a monocarboxilic acid. Further attack of monocarboxilic acid occurs by b-oxidation with subsequent formation of two carbon units shorter fatty acids and acetil coenzyme A, with eventual liberation of carbon dioxide (Atlas, 1981). Gas chromatography analysis of the acids derived from hydrocarbon oxidation by C. resinae reveals the presence of monocarboxilic, dicarboxilic and several tricarboxilic acids. The carboxilic acids consisted of mono C (Schiaparelli and Meybaum, 1980), di C (Lin *et al.* 1971) and C (Salvarezza *et al.* 1981) and tricarboxilic acids such as citric, isocitric, cis-aconitic, a-ketoglutaric, succinic and maleic acid Lin *et al.* 1971). Siporin and Cooney reported that acetic and dodecanoic acids are predominant in C. resinae cultures (Siporin and Cooney, 1975). The rate of hydrocarbon biodegradation is limited by the supply of nitrogen and phosphorous which are incorporated by the microorganisms into the biomass (Atlas, 1981).

The electrochemical behaviour of aluminum and its alloys in the presence of microbial contaminants of hydrocarbons has been studied using conventional polarization techniques in the last few years (de Mele *et al.* 1979; Schiaparelli and Meybaum, 1980). The results show a decrease of the pitting potential in C. resinae cultures with respect to sterile medium. The organic acid anions produced by the metabolic activity of the fungus have been considered by some authors as the only one responsible for the passivity breakdown of aluminum alloy (Schiaparelli and Meybaum, 1980). They later identified the aggressive anion as dodenanoate, neglecting the influence of the culture medium composition on the pitting process (Schiapparelli and Meybaum, 1980). On the other hand, a mechanism has been proposed involving the action of the aggressive chloride anions which are present in hydrocarbons as well as in the culture media employed in laboratory studies. In this mechanisms, the breakdown of the passive film in the presence of chlorides is facilitated by the metabolic products of C. resinae (Salvarezza *et al.* 1981). The acidic metabolites produced by the fungus are able to decrease the pitting potential value of aluminium in chloride solutions as well as the electrolyte passive film surface energy. It could be expected that the microbiological demand of nitrogen and phosphorous available in culture media as NO^-_3 and PO^{-3}_4 would change the corrosion rate due to the inhibitory properties of these anions. The present work contributes to the understanding of the microbial corrosion of aluminium alloys in hydrocarbon/water systems analyzing the importance of culture medium composition, and more specifically the role of aggressive and inhibitor anions as well as the influence of different organic acids produced by the microbial degradation of hydrocarbons.

EXPERMENTAL

The electrochemcial measurements were performed in a pyrex glass cell thermostatted at room temperature. The counter electrode was a platinum wire. A saturated calomel electrode was used as reference electrode through a luggin capillary. The working electrodes were aluminium alloy pieces of 0.4cm^3 set in Teflon and previously polished, first with a fine emery paper and then with metallographic grade alumina of 0.3m.

Polarization curves were run at 10mV/min in the anodic direction starting from –1.0V to reach the sharp current inflection corresponding to the pitting potential (E_p). In several experiments, pitting potential values were obtained from current-time plots at constant potential. In these cases, the potential was changed in steps of 20mV/20min. After the E_p was reached, microscopic observations of the test specimens were made to confirm the presence of pits on the metal surface. Microphotographs were taken using a Philips scanning electron microscope (PSEM 500).

Electrolyte solutions were prepared with analytical reagents. Dearation with pure nitrogen was carried out in the polarization experiments. Pure cultures of Cladosporium resinae which were insolated from fuel storage tanks were used. The composition of the solution employed as electrolyte and culture medium is shown in Table 1 medium a).

Table 1

Solution compositions

a Simplified Mineral Media (mol/L)	b Bushnell and Haas$^{(1)}$ (mol/L)
$CaCl_2$ 1.8 x 10^{-4}	$CaCl_2$ 1.8 x 10^{-4}
$MgSO_4$ 1.7 x 10^{-3}	$MgSO_4$ 1.7 x 10^{-3}
$(NH_4)_2SO_4$ 7.6 x 10^{-3}	NH_4NO_3 1.3 x 10^{-2}
	$(NH_4)_2SO_4$ 7.6 x 10^{-3} or
pH = 7.00 adjusted with NaOH^3	$K_2HPO_4$5.7 x 10^{-3}
	KH_2PO_4 7.3 x 10^{-3}
	$FeCl_3$ x 10^{-4}
	pH = 7.00 adjusted with NaOH^{13}

Parbery (1969), some authors (Siporin and Cooney, 1975) use Bushnell and Haas medium diluted to 1/10, with a final pH of 6.0 adjusted with H_3PO_4. No specification is made on the salt used as nitrogen source [NH_4NO_3 or $(NH_4)_2SO_4$].

The composition of the Bushnell and Haas (Bushnell and Haas, 1941)$^{)}$ medium usually employed to study the microbial corrosion of aluminum alloys in fuel/water systems is also shown (medium b).

Fungal cultures were performed in erienmeyer flasks of 500mL. The culture medium to fuel ratio was 4:1. Sterilization was by autoclaving and filtration. Static conditions were chosen for growth. The oxygen supply in this condition is ascribed to the fact that the oxygen transfer from oil to water in much faster than from air to oil. In this way, direct oxygen transfer from air to water is accomplished (Mimura*et al.* 1969). Temperature was maintained at room temperature.

RESULTS

Role of the culture medium composition

Chloride Anions Effect. The breakdown of passivating oxide film on aluminium by aggressive anions such as chloride frequently causes the failures of aluminum and its alloys in aqueous environments (Foroulis and Thubrikar, 1975). This process leads to severe pitting on the metal. It is known that water accompanying hydrocarbons contains variable levels of chloride anions. Scott and Hill (Scott and Hill, 1971) have reported chloride in the 10^{-2} to 10^{-4}m concentration range. Besides this, mineral media usually employed as electrolyte and culture medium to study the microbial corrosion of aluminum alloys in hydrocarbon/water systems, contains chloride anions in its composition.

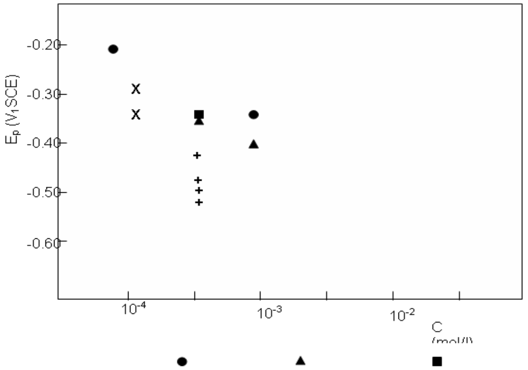

Fig. 1 E_p corresponding to 2024 aluminum alloy vs. chloride concentration plot at different scan rates: (●) v = 10mV/min; (▲) v = 20mV/20 min; (■) pitting potential in simplified mineral medium, v = 10mv/min; (+) pitting potentials of C. resinae cultures (simplified mineral medium) v = 10mV/min[14]; (x) pitting potentials of C. resinae cultures (Busihnell and Haas medium diluted 1/10) v = 10mV/min[4,8].

Figure 1 shows the pitting potential dependence of aluminum alloy with the chloride concentration in the 10^{-2} to 10^{-4}m concentration range.

As chloride concentration increases, the breakdown of passive film occurs at more cathodic potentials value. In the same figure, E_p values reported by Salvarezza, *et al.* (1980) in C. resinae cultures as a function of the chloride concentration present in the culture media are also included. In both cases, E_p values are more cathodic than those observed in the solutions containing only chloride anions. It can be seen that E_p values corresponding to sterile simplified mineral medium (-0.35 to -0.37V) are similar to those obtained in the solution containing only chloride anions at the same concentration. However, in the original Bushnell and Haas medium, as well as in the Bushnell and Haas diluted 1 to 10 medium, a more complex behaviour is expected due to the presence of phosphate and nitrate anions.

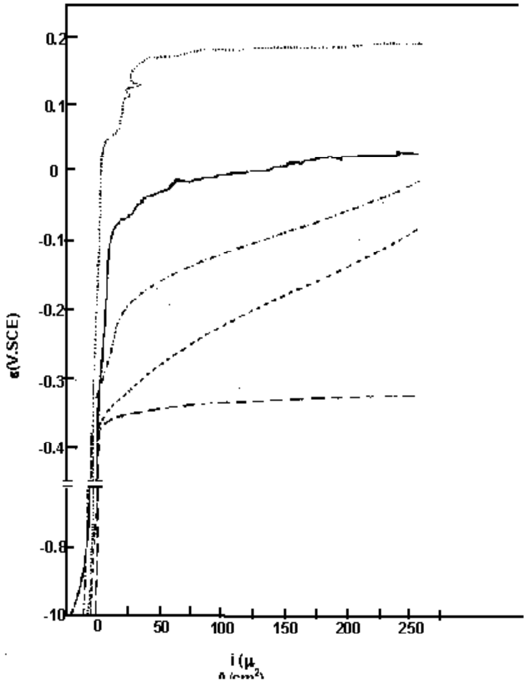

Fig. Polarization curves corresponding to 2024 aluminum alloy at a scan rate of 10mV/min in: (----) 10^{-3} M NaCl; (----) 10^{-4}M NaCl; (   ) 10^{-3} M NaCl, 5.7 x 10^{-3}M K_2HPO_4, and 7.3 x 10^{-3}M KH_2PO_4; (….) 10^{-4} NaCl, 5.7 x 10^{-4}M K_2HPO_4, and 7.3 x 10^{-4}M KH_2PO_4; (---) 3 x 10^{-3}M NaCl, 5.7 x 10^{-3}M K_2HPO_4, and 7.3 x 10^{-3}M KH_2PO_4

Phosphate and Nitrate Anions Effect. Figure 2 shows the polarization curves run at 10mV.min. in chloride 10^{-3} and 10^{-4}m (corresponding to the chloride concentration present in the original and diluted Bushnell and Haas media). When KH_2PO_4 and K_2HPO_4 are added to these chloride solutions to reach the range of concentration present in the original Bushnell and Haas and the diluted 1 to 10 media, a marked increase in the pitting potential (E_p) of the alloy can be observed.

Besides this, if chloride is increased at a constant phosphate level, the E_p decreases, showing that the E_p variation strongly depends on the chloride/phosphate ratio. Phosphate anions, by their buffering action, could prevent localized acidification which is necessary to the pitting process (Galvele, 1977).

The effect of phosphate anions on the pitting of pure aluminium in chloride solutions was recently analyzed by Rudd and Scully (Rudd and Scully, 1980) using polarization techniques at different scan rates (20mV/10min, 20mV/20min, and 10mV/30min). in phosphate solutions, the E_p determination was difficult and the results were unreliable. It was concluded that polarization techniques are not satisfactory for E_p evaluation in phosphate containing solutions. Accounting to these authors, phosphate anions act as pitting inhibitors mainly by forming insoluble precipitates with the Al^{3+} coming from the metal dissolution. The inconvenience of using phosphate anions in the culture media employed to study microbial corrosion of aluminium has also been pointed out in previous works (Salvarezza *et al.* 1981; Salvarezza*et al.* 1979).

Nitrate anions used a NH_4NO_3 in the Bushnell and Haas medium are considered the most efficient pitting inhibitors in chloride solutions. This explains the anodic values of the E_p for aluminium alloys observed in Bushnell and Haas of the order of 1.5V. In chloride solutions containing nitrate anions, the microbial uptake of nitrate is able to produce marked decrease in the E_p of aluminium alloys. Smaller effects than those produced by nitrate uptake can be expected as a consequence of phosphate anion consumption by microorganisms.

The Role of organic Acid Anions. Recently, Meybaum and Schiapparelli (Schiapparelli and Meybaum, 1980) using Bushnell and Haas medium diluted1 to 10, attributed the E_p decrease observed in C. resinae cultures to dodecanoate anions produced by the fungus oxidation of hydrocarbon chains. To study the dodecanoate action, pitting potentials (E_p) of 2024 aluminium alloys were derived from potentiostatic poarization curves in chloride solutions containing 13mg/1000mL of dodecanoic acid adjusted to 7.00 with NaOH 0.1N. The results obtained in only chloride solutions& chloride solutions plus dodecanoate are shown in Fig. 3.

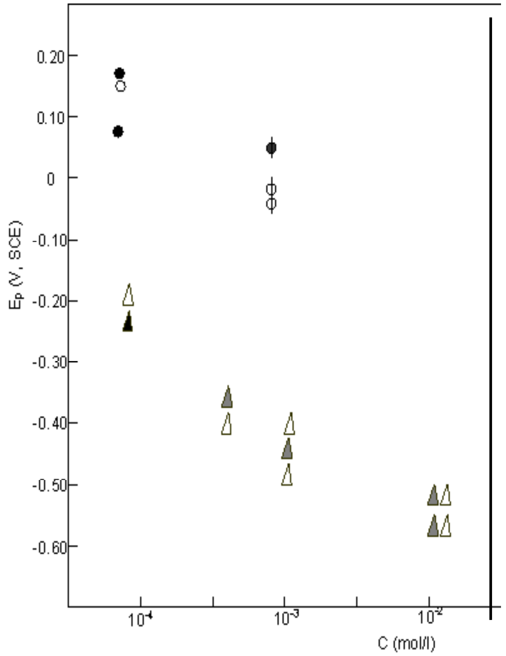

Fig. 3 E_p corresponding to 2024 aluminum alloy vs. chloride concentration plot In: (D) NaCl (v = 10mV/min); (D) NaCl (v=20mV/20 min); (▲) NaCl and dodecanoic acid (pH = 7.0 adjusted with NaOH 0.1N); v = 10mV/min; (▲) same as (▲), but v = 20mV/20 min; (f) Cl^- and phosphate concentration as in Bushnell and Haas medium; (f) same as (f) + dodecanoic acid, pH = 7.0; (O) Cl^- and phosphate as in Bushnell and Haas medium diluted to 1/10; and (●) same as (O) + dodecanoic acid (pH = 7.0).

In the experimental conditions described here, the addition of dodecanoate produces no effect on the pitting potentials. In the same Figure, pitting potentials obtained in electrolytes containing chloride and phosphate anions in the same concentration of Bushnell and Haas and Bushnell and Haas diluted 1 to 10 media, plus dodecanoic acid adjusted to pH 7.00 are observed. In spite of the difficulties to obtain pitting potential values in the presence of phosphate anions, no appreciable effect of the dodecanoate anions was noticed. Moreover, similar results were obtained in chloride solutions containing phosphate and nitrate when dodecanoate anions were present.

To study the kinetics of pit growth, test specimens of 2024 aluminium alloys were potentiostatized at 20mV above the pitting potential in NaCl 10^{-2}M, pH 7.00 and NaCl 10^{-2}M plus dodecanoic acid adjusted to pH 7.00 with NaOH 0.1N (Figure 4). In both cases, the kinetics of the potentiostatic pit.
growth approaches the Engell-Stolica (Engell and Stolica, 1959),
relation : $i = At^b$ (1)

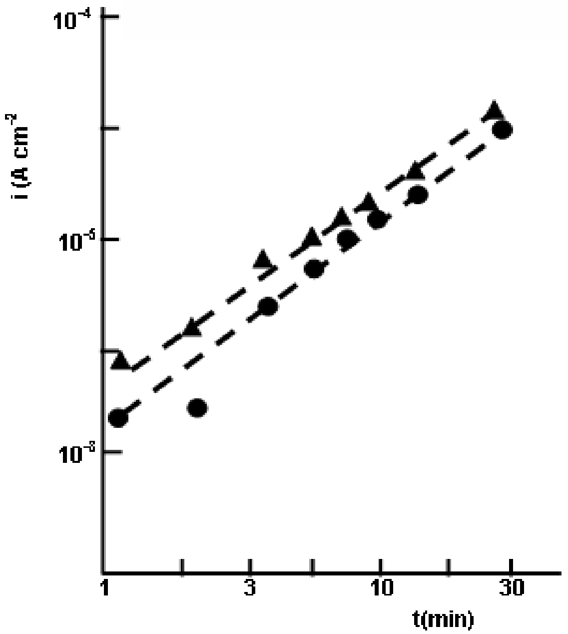

Fig. 4 Kinetics of pit growth at constant potential for 2024 aluminum alloy in (▲) 10^{-2}m NaCl and (●)10^{-2}m NaCl + dodecanoic acid (pH = 7.0 adjusted with NaOH 0.1N)

where i is the current density measured at the whole electrode, and A and b are constants. Therefore, the presence of dodecanoate anions does not change the kinetics of pit growth, showing in both cases an exponential coefficient approximately equal to 1.

Mckenzie, *et al.* showed that citric acid at the concentration found in

C.resinae cultures was able to produce the corrosion of 2024 aluminum alloy (Mckenzie *et al.* 1977). The corrosion rates were similar to those obtained in the presence of the fungus. Pitting and integranular corrosion were observed on aluminum alloy exposed to citric acid solutions.

To study the effect of citrate anions, pitting potentials were measured in solutions containing 3.6 x 10^{-4}M sodium chloride added with different concentrations of citric acid. These solutions were adjusted to pH 7.00 using NaOH 0.1N. The results are shown in Table 2.

Table 2

Pitting potentials of 2024 aluminium alloy in chloride solutions containing different organic anions

Solution	E_p (V, SCE)
NaCl 3.6 x 10^{-4}M	-0.35
NaCl 3.6 x 10^{-4}M citrate 5 x 10^{-3}M	-0.30
NaCl 3.6 x 10^{-4}M citrate 5 x 10^{-2}M	-0.24
NaCl 3.6 x 10^{-4}M acetate 1 x 10^{-2}M	-0.20
NaCl 3.6 x 10^{-4}M oxalate 1 x 10^{-2}M	-0.30

Table 3

pH values in different electrolytes during stationary growth phase

Medium	Carbon source	Temp.°C	pH	References
Bushnell and Haas	jet-fuel	25-30	4.2 – 4.8	2
Simplified Mineral	jet-fuel	28	2.9 – 5.0	9
Bushnell and Haas	jet-fuel	30	4.0	24

Data obtained in the same chloride solution containing acetic and oxalic acid adjusted to pH 7.00 is also included. At the concentrations used here, all anions acted as pitting inhibitors increasing the pitting potential value.

The Role of pH. It is well known that as a consequence of the organic acid production, low pH. values are reached during the C. resinae growth (Mckenzie, *et al.*. 1977; Siporin and Cooney, 1975). Table 3 shows pH values corresponding to the stationary growth phase reported by several authors in different electrolytes used as culture media.

In service conditions, low pH values are easily reached in the colony attachment region. Figure 5 shows polarization curves of aluminium alloy in NaCl 10^{-2}M, pH 7.00 and NaCl 10^{-2}M, pH 2.5 adjusted with NaOH 0.1N and oxalic acid respectively.

In the acid solution, a pitting potential decrease of 0.050V with respect to neutral medium can be observed. The acidity can prevent the repassivation process of the protective oxide film. Thus, pitting of the metal in acid media due to the chloride anions occurs at more cathodic potentials than in a neutral solution (Table 4).

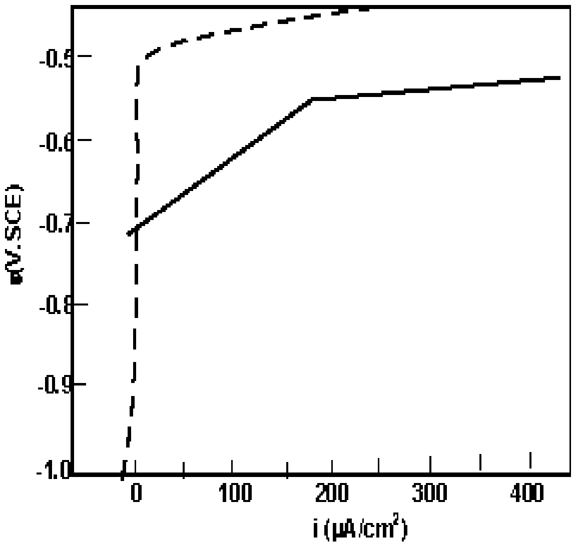

Fig. 5 Polarization curves corresponding to 2024 aluminum alloy at a scan rate v =10mV/min; (---) 10^{-2}M NaCl (pH = 7.0); (—)10^{-2}M NaCl (pH=2.5 adjusted with oxalic acid).

Table 4

Pitting potentials of Aluminium and Aluminum Alloys in Acid Media

Media	Material	E_p (V, SCE)	References
1N NaCl, pH 7.0	Aluminium	-0.80	25
1N NaCl, pH 2.0	Aluminium	-1.36	25
0.5N NaCl, Ph 6.0	Aluminium 0.1% tin	-1.39	26
0.5N NaCl, pH 3.2	Aluminium0.1% tin	-1.47	26
0.5N NaCl, pH 2.2	Aluminium 0.1% tin	-0.72	26
0.15N NaCl, pH 7.0	Aluminium	-.076	12
0.15N NaCl, pH 2.5 (acetic acid)	Aluminium	-0.78	12
0.15N NaCl, pH 2.0 (citric acid)	Aluminium	-0.50	12
0.01N NaCl, pH 7.0	2024 Aluminium alloy	-0.55	This work
0.01N NaCl, pH 2.5 (oxalic acid)	2024 Aluminium alloy	-0.77	This work

According to Schiapparelli and Meybaum (Schiapparelli and Meybaum, 1980), the E_p of 2024 aluminum alloy is not affected by the pH value of the culture medium. However, from the experimental results observed when acetic acid was added to the neutral Bushnell and Haas diluted 1 to 10 medium to reach a final pH of 3.5. On the other hand, the organic acid production counteracts the oxygen depletion due to the fungus growth, providing H^+ as cathodic reactant for the corrosion process.

DISCUSSION

Different mechanisms have been proposed to explain the microbial corrosion process of aircraft aluminum alloys. Oxygen concentration cells formed between the zones covered by the fungus respiration have been reported by

Miller (1964)) coincidently, Iverson reported the presence of tubercles on the bottom of the jet fuels tanks containing sulphate reducing bacteria, Pseudonionas sp. and C. resinae (Iverson, 1967). The fungus micelyum provided the mechanical support for the tubercular structure, but corrosion proceeds through the formation of electrochemical concentration cells and some catholic depolarization effect due to the sulphate reducers. The removal of certain metallic atoms by extra cellular enzyme activity was postulated by Hedrick, *et al.* (1968). This assumption is based on the correlation found between the degree of attack and magnesium content of different aluminum alloys and the importance of magnesium ions in the growth of microorganisms. The formation of corrosive products has been considered the most significant process in explaining the microbial corrosion of aluminum alloys in hydrocarbon-water system. In relation to this mechanism, Parbery attributed the corrosion of aluminum alloys to the action of organic acids derived from the oxidation of hydrocarbon chains by the Fungus (Parbery, 1968). Supporting this assumption, Mckenzie *et al.* 1977, verified the production of citric, cis-aconitic, isocitric, and a-ketoglutaric acid when C. resinae grows in jet fuel (Hedrick *et al.* 1968). These acids are able to produce corrosion of alluminum alloys at similar rates to those observed in Cladosporium resinae (C. resinae cultures). The acidic metabolites of the C. resinae facilitate the pitting of aluminum alloys through a decrease of the pitting potential (Salvarezza *et al.* 1979; Meybaum and Schiapparelli, 1980).

It is generally accepted that the rate of pit propagation obeys equation (1) and the exponential coefficient b is a constant dependent on pit geometry. Citrate and oxalate anions are able to form soluble complexes with the aluminum ions (Samuels *et al.* 1981). Hence, experimental results using citrate, oxalate and acetate anions show that these anions act as pitting inhibitors increasing the pitting potential of aluminum alloy in neutral chloride solutions towards more positive values.

Bohni and Uhlig also reported an increase of the pitting potential of aluminum in chloride solutions as the acetate concentration increases (Bohni and Uhlig, 1959). The inhibitory effect of acetate and oxalate anions in neutral media can be explained by the formation of a precipitation type compound (Samuels *et al.* 1981). Samuels, *et al.*, considered that stable, uncharged, slightly ionized species were formed near the metal surface hindering the dissolution process in neutral solutions. On the contrary, acetic and oxalic acids increases the corrosion rate of aluminium which depends on the pH value (Samuels *et al.* 1981). In the acid solutions, pitting occurs at more negative values than in neutral chloride solutions. The acidity can prevent the repassivation process facilitating pitting. In the acid solutions, pitting can be accomplished by the formation of soluble complexes between the organic acid anions and the aluminum cations, thus increasing the dissolution rate. When the pH is lowered from values of 7.5 to that of 2, and E_p decrease of about 0.050V is observed. Higher pitting potential decreases have been reported in simplified mineral medium contaminated with Cladosporium resinae (Salvarezza, *et al.* 1979).

There are experimental evidences that the rate of hydrocarbon biodegradation is limited by the available concentrations of nitrogen and phosphorous in the medium (Atlas, 1981). In many cases, nitrogen and phosphorous are supplied as nitrate and phosphate in the culture medium. These anions act as pitting inhibitors for aluminum in chloride solutions. During microorganism growth nitrate and phosphate are incorporated into the biomass increasing the chloride/inhibitor ratio. Consequently, the pitting potential value is displaced in the cathodic direction finally reaching values similar to those obtained in solutions containing only chloride anions. These facts could explain the marked decrease of about IV of E_p values obtained in Cladosporium resinae cultures growing inhibitor containing culture medium in respect to sterile medium.

CONCLUSION

The results of this present work show the importance of the electrolyte composition, especially the chloride/inhibitor ratio, to establish the microbial corrosion mechanism. The acidic metabolites produced during fungus growth facilitate the breakdown of passivity by the chloride anions and pitting occurs at more cathodic values than those observed in neutral chloride solutions. The acidity, however, is not the only factor that accounts for the aggressivenss increase observed in Cladosporium resinae cultures. This fact is supported on the difference between the pitting potential values measured in Cladosporium resinae cultures and the organic acid solutions. It is worth noting that more research must be done on the complex compounds derived from the fungus metabolic activity to establish the contribution of unidentified metabolites and/or cellularlysis products to the localized corrosion process.

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