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CHEMISTRY

Effect of Citrate and Chloride Anions on the Corrosion Behavior of Aluminum in Aqueous Solutions

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Abstract. The corrosion behavior of pure aluminium in aerated aqueous solutions containing chloride and/or citrate ions was studied by means of potential - time measurements and polarization technique in the pH range 1.7 to 12. It is shown that Al corrodes in the presence of citrate ion to an extent which depends on its concentration, temperature, pH values and Cl^- ion concentration. The effect of surface treatment of the electrode surface on open-circuit potential has been studied. The present results indicate that for electropolishing, the open-circuit potential does not change significantly. On the other hand, citric acid shows some inhibitive effect due to its adsorption on the metal surface which follows the Frumkin isotherm.

Introduction

The corrosion of Al is widely studied in acid, neutral and alkaline solutions. In aqueous solutions, Al generally exhibits passive behavior and its corrosion is connected with the properties of its oxide film. Due to the amphoteric nature of the oxide film, Al dissolves easier in strong, non oxidizing acids and bases than in neutral solutions [1, p. 168]. Moreover, the corrosion rate of Al depends on the impurities in the metal. Some work has been carried out on the corrosion of Al in aqueous solutions containing organic acids [2; 3]. No work was found in the literature concerning the corrosion of Al in aqueous solutions containing both Cl^- and an organic acid, particularly citric acid. A toxic role of Al has been suggested with Alzheimer's disease [4; 6], dialysis encephalopaty/dementia [7; 8], bone disorder [9] and other disorders [10]. The estimated daily intake of Al from food sources is about 10-15 mg/day [11]. Factors affecting the Al content of food include type of Al cookware, pH values, cooking time and food additives. Al compounds are widely used in the food industry; therefore, the actual Al intake can be much greater than the figures quoted above. Moreover, it is known that complex formation increases the dissolution of a metal, and therefore, soluble complexes of Al enhance the absorption of Al [12]. Citrate forms tight complexes with Al preventing the formation of hydroxides and phosphates leading to greater solubility in the intestinal fluid. In rats, nearly one hundred times as much Al is absorbed when given the citrate form, as compared to the chloride salt [13]. This paper, which is a part of a program, is aimed to investigate the corrosion of Al and Al alloys in conditions simulating the ingredients in local cooking habits to determine the extent of Al dissolution under these conditions.

Experimental

Open-circuit potential (OCP) and polarization measurements were carried out on a corrosion system model 263 EG&G potentiostat/galvanostat. All experiments were carried out using a three electrode cell with saturated calomel electrode (SCE) with a Luggin capillary and a platinum electrode as reference and counter electrode, respectively. The aluminum working electrode (Goodfellow, 99.99%) was in the form of a rod inserted in Teflon with an exposed area of 0.282 cm^2. Al electrode was polished with grade 600 emery paper. It was then washed thoroughly with double distilled water and finally cleaned ultrasonically for 1 min. in acetone. The test solutions were prepared with double distilled water and analytical reagents. OCP measurements were recorded for 30 min. followed by polarization measurements at a scan rate of 0.3 mVs^{-1} for the Tafel plots. The scanning was always from cathodic to anodic direction. Corrosion current densities (I_{corr}) were calculated by an iterative non-linear least-squares fit of the data to the Stern-Geary equation [14] in the cathodic Tafel potential region. The iteration ceases whenever the difference between the calculated graph and the experimental graph is minimal. Temperature was 25 ± 1°C unless otherwise stated. pH of solution was adjusted to the required value using sodium citrate, citric acid and NaOH. Measurements were carried out in duplicates, if not otherwise stated.

Results and Discussion

The effects of a number of surface treatments on OCP were tested. These were wet and dry polishing, chemical etching by immersion in 5% HNO_3 for 90 s, and in 10% NaOH for 90 s and by cathodic reduction at -2.5 V for 90 s. OCP was followed in 0.02 M sodium citrate (NaCit) as a function of time till a steady-state potential values were attained, and the results are presented in Fig. 1. The first four treatments shift OCP to more negative values and reaches the steady state values (E_{ss}) after ~5 min. while for the last type of treatment, OCP does not change significantly. This may be due to the removal of the pre-immersion oxide film. The steady state potentials are almost the same, which is about -1650 mV. As a result of the above mentioned results, it has been decided to adapt wet polishing for surface treatment.

Fig. 2 shows the variation of OCP with time, at different concentrations of NaCit. It is clear that at all concentrations the potentials are drifting to more negative potentials. It is to be mentioned that the steady state potential, E_{ss}, at 1M NaCit is ~ 400 mV more negative than at 5 x 10^{-4} M NaCit. The plot of E_{ss} vs. log [NaCit] gave a straight line, which satisfies the following experimental equation:

$$E_{ss} = a - b \log [\text{NaCit}] \quad (1)$$

where "a" is a proportional constant and "b" is the order of the reaction which can be obtained from the slope of the straight line which depend on the nature of the electrolyte and surface treatment.

The variation of OCP with time at different pH values in 0.5M NaCit is shown in Fig. 3. It is obvious that a pH ≤ 7.5, the potential changes positively, while at pH ≥ 9.3, the potential changes to the negative direction. In both cases, the plot of E_{ss} against pH gave a straight line, which satisfies an equation similar to equation (1).

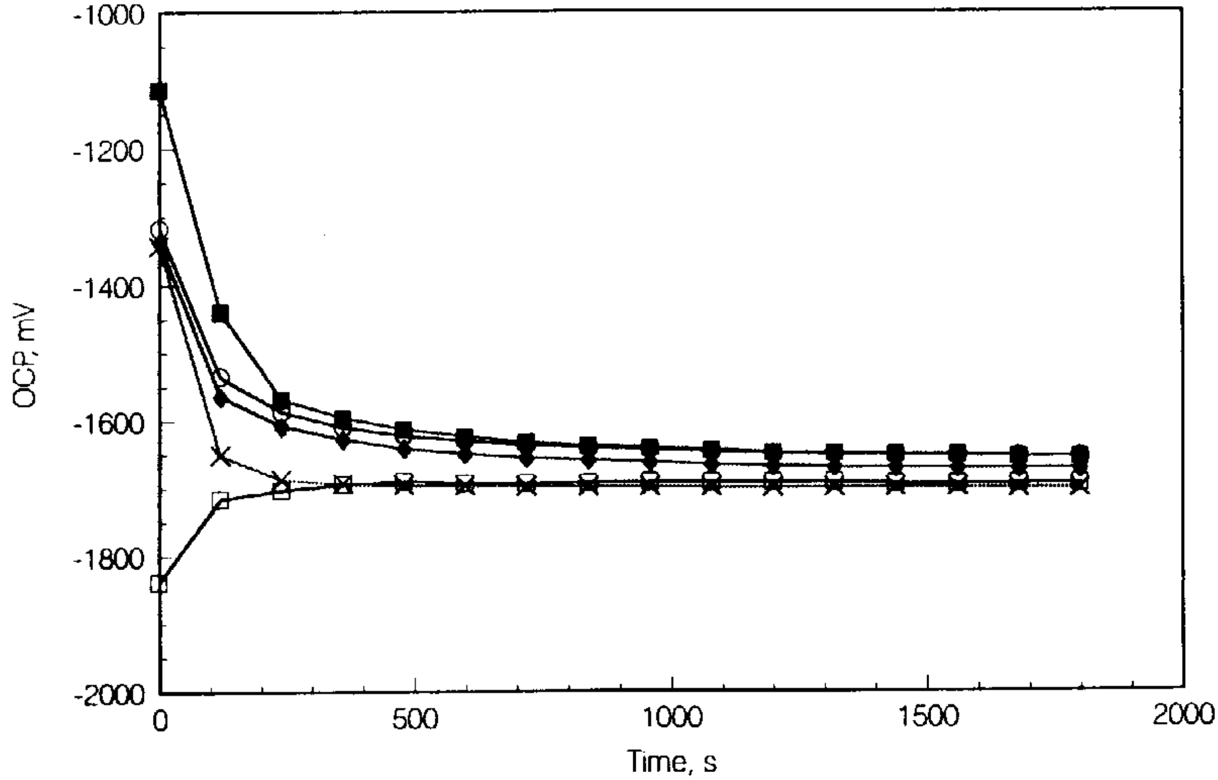

Fig. 1. Effect of surface treatment on OCP of Al electrode immersed in 0.02 M NaCit, (■) 5% HNO_3 for 90 s; (○) wet polishing; (◆) dry polishing; (x) 10% NaOH for 90 s; (□) -2.5 V for 90 s

Figure 4 represents the variation of OCP with time at various temperatures. It is clear that the potential shifts to the negative direction with increasing the temperature. Again, the plot of Ess against (t°C) gave straight line which means that an equation, like equation (1), is applicable.

It is clear that, irrespective of surface treatment, the potential of the electrode in NaCit solution attains a steady state potential shortly after immersion. This OCP is markedly dependent on the solution condition such as [NaCit], pH and temperature. Increasing of these conditions causes the potential to become more active and the tendency towards passivation is greatly reduced due to the destruction of the oxide film.

Since the variation in [NaCit] resulted in a change in pH of the solution (there is 2 pH units difference between the highest and lowest concentrations used), it was decided that it is necessary to find out whether the observed change in potential was due to the change in

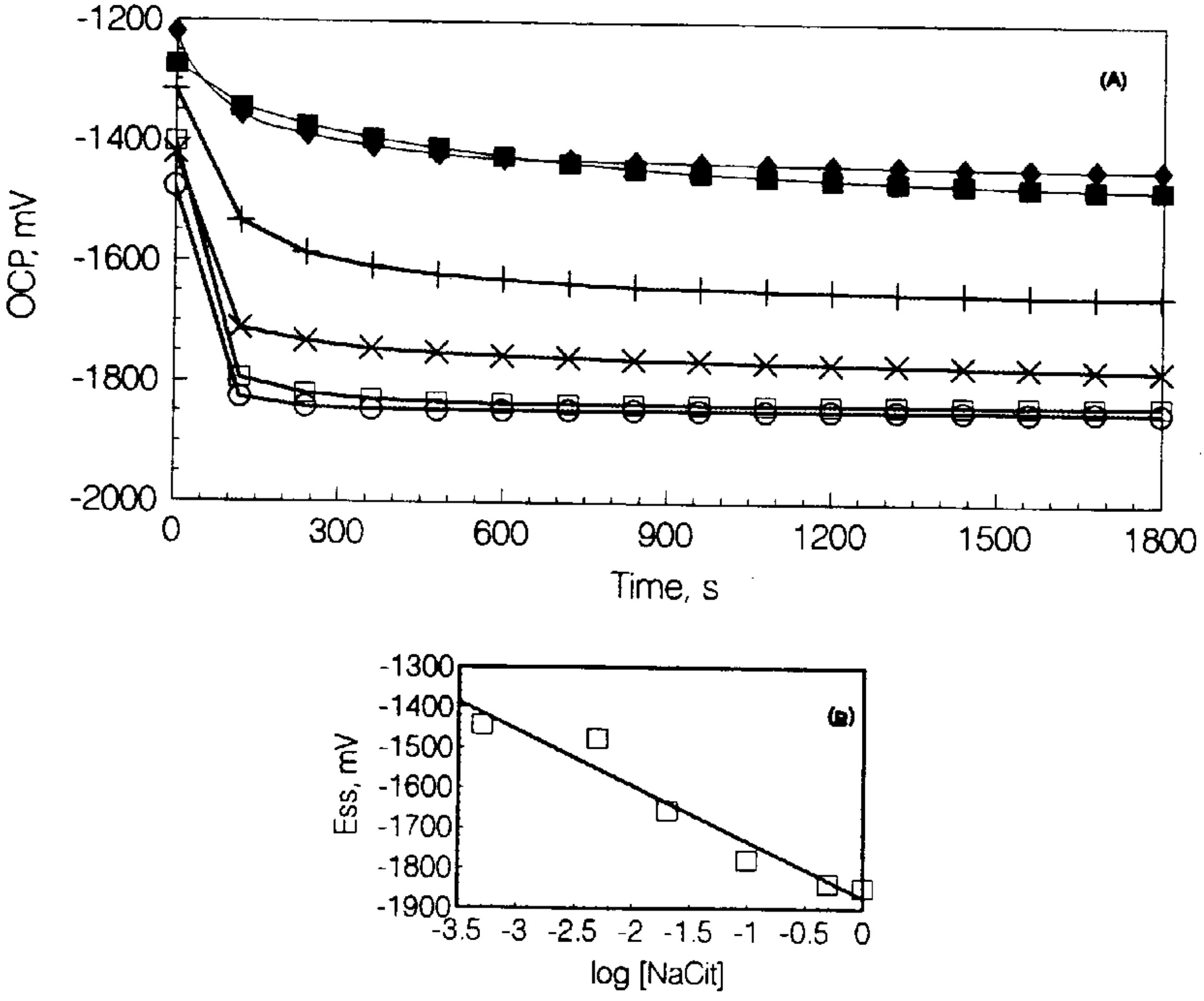

Fig. 2. (A) Variation of OCP with time at different concentration of NaCit.
(◆)5 x 10^{-4} M; (■) 5 x 10^{-3} M; (+) 0.02 M; (x) 0.1 M; (□)0.5 M; (○) 1.0 M; (B) Relationship between E_{ss} and log [NaCit].

[NaCit] or due to a change in pH values. Therefore, for nine solutions covering concentrations between 0.02-1.0 M, the pH was fixed at 4.3 by changing the concentration of sodium citrate and citric acid (CA) with the ratio [NaCit]/[CA] ≈ 1. Under these conditions it was found that OCP changes with time in the positive direction and that E_{ss} was almost constant and equals to -828 ± 37 mV. This may be attributed to a competition between two different processes, the first of which leads to the strengthening of a protective layer, while the second one leads to the breakdown of this layer. As mentioned early at low

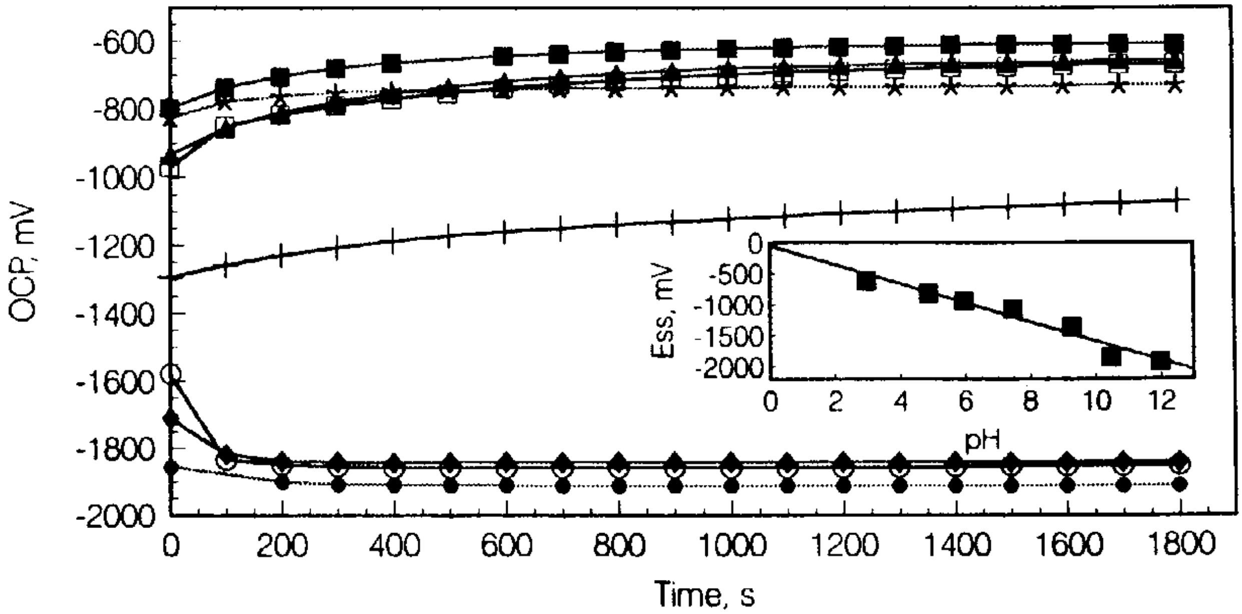

Fig. 3. Effect of pH on OCP of Al electrode in 0.5 M NaCit
(□) 1.7; (■) 3; (Δ) 4; (★) 6; (+) 7.5; (○) 9.3; (◆) 10.5; (•) 12.

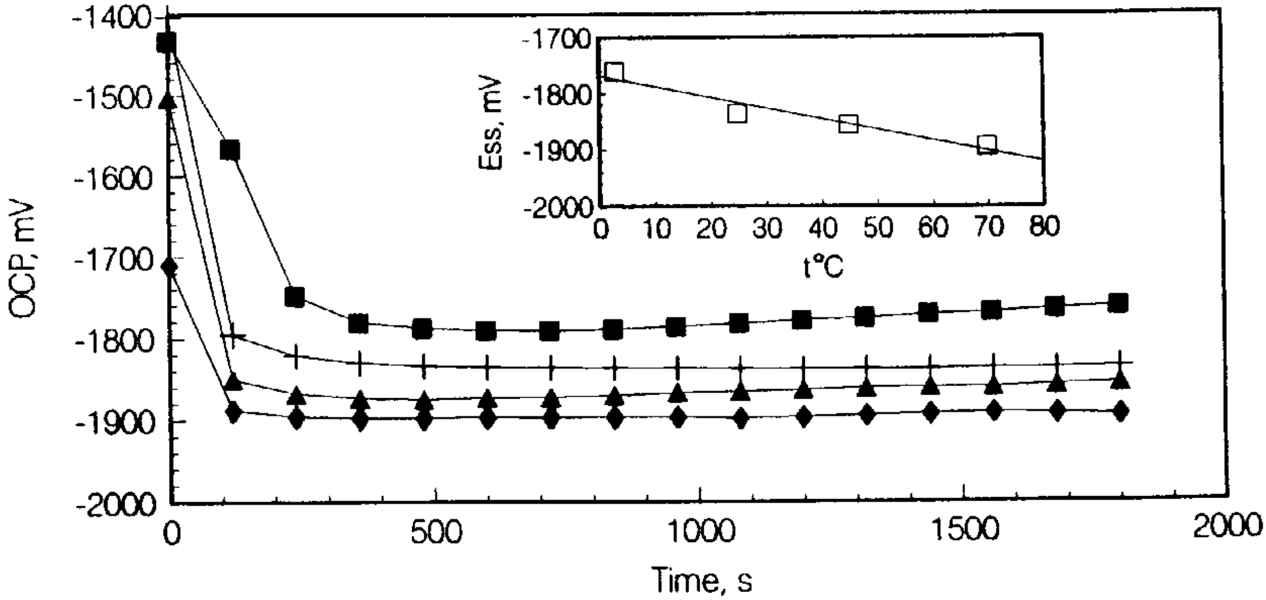

Fig. 4. Effect of temperature on OCP of Al electode in 0.5 M NaCit.
(■) 3°C; (+) 25°C; (Δ) 45°C; (◆) 70°C.

pH, where CA dominates, the potential changes positively which means the thickening of the protective layer while at high pH, where citrate ion predominates, the potential changes negatively with time, indicating the thinning of the protective layer. At low pH, H^+ and Al^{3+}

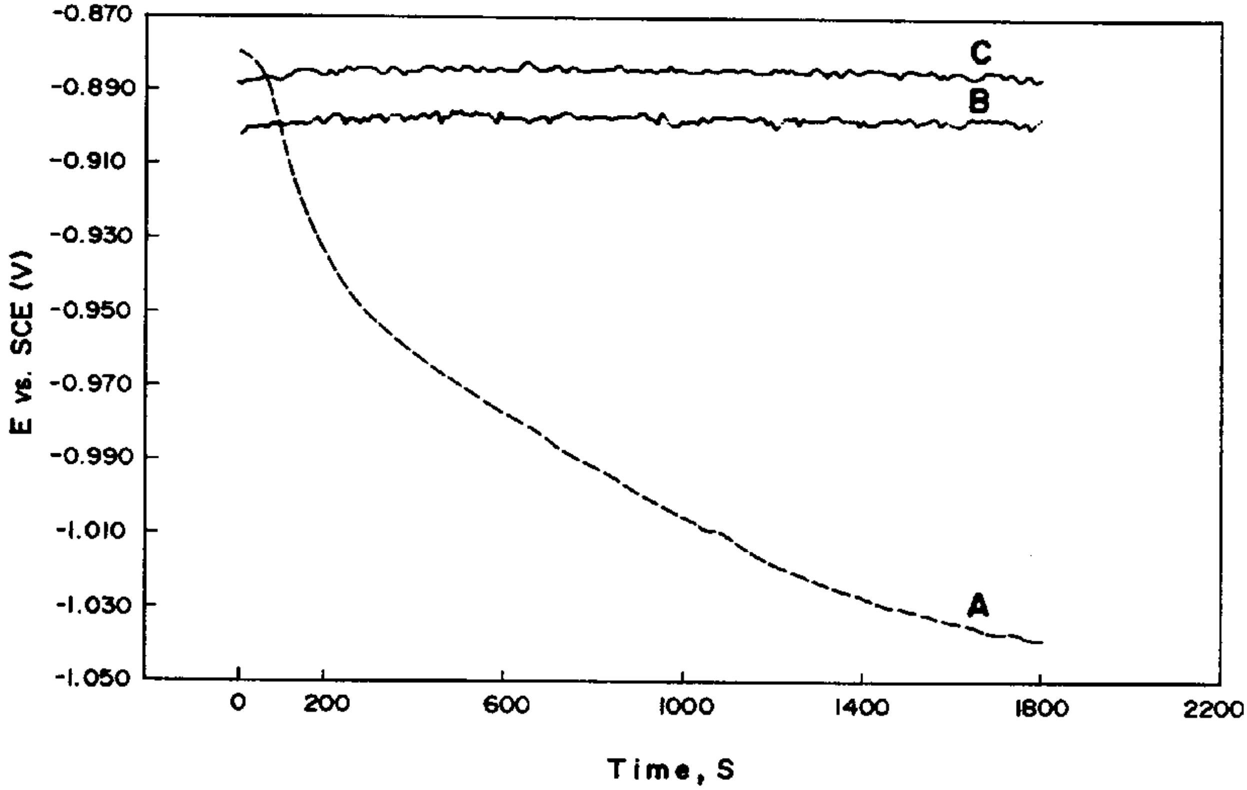

Fig. 5. Variation of OCP with time for Al electrode immersed in 0.6 M HCl and in the presence of citric acid, (A) 0.6 M HCl only; (B) 0.05 M citric acid; (C) 1.0 M citric acid.

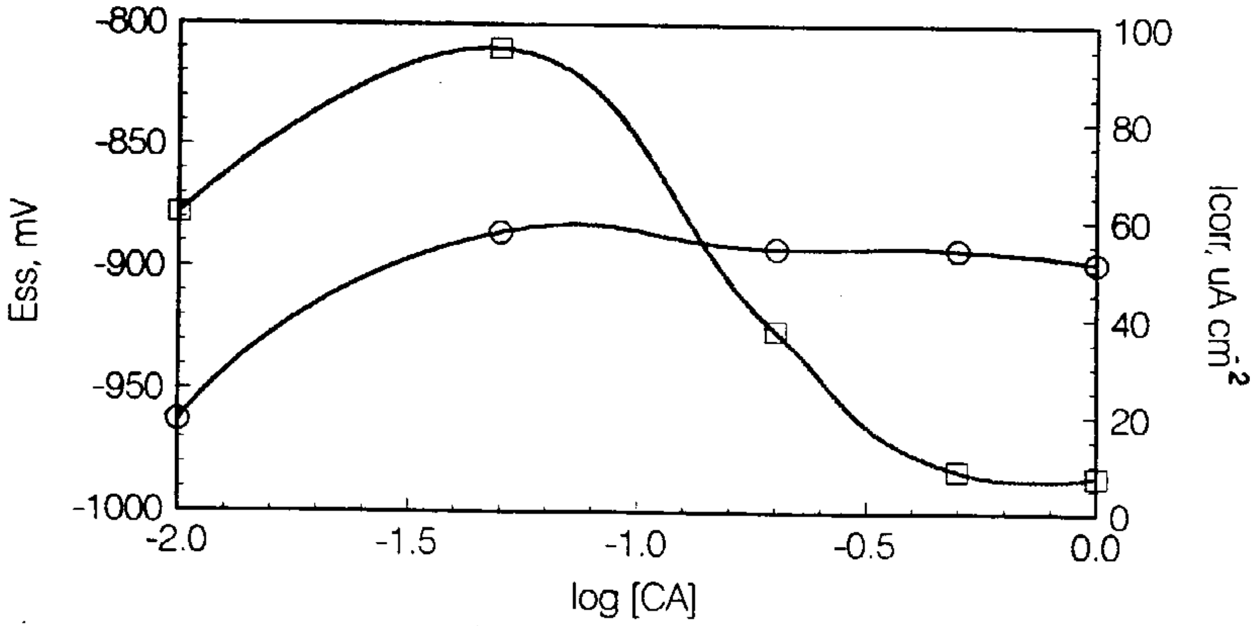

Fig. 6. Dependence of (○) E_{ss} and (□) I_{corr} of Al electrode on citric acid concentration.

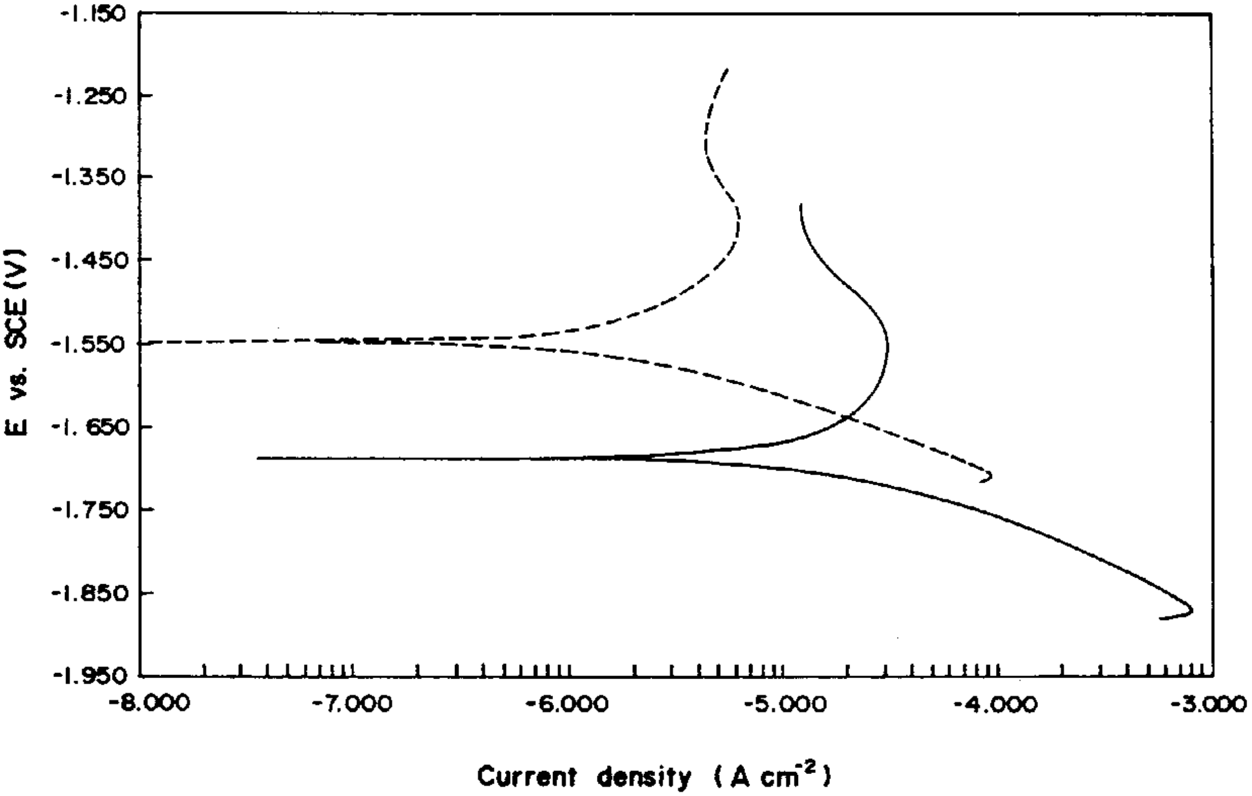

Fig. 7. Typical Tafel plot for Al electrode at (–) 0.002 M NaCit and (---) 0.02 M NaCit.

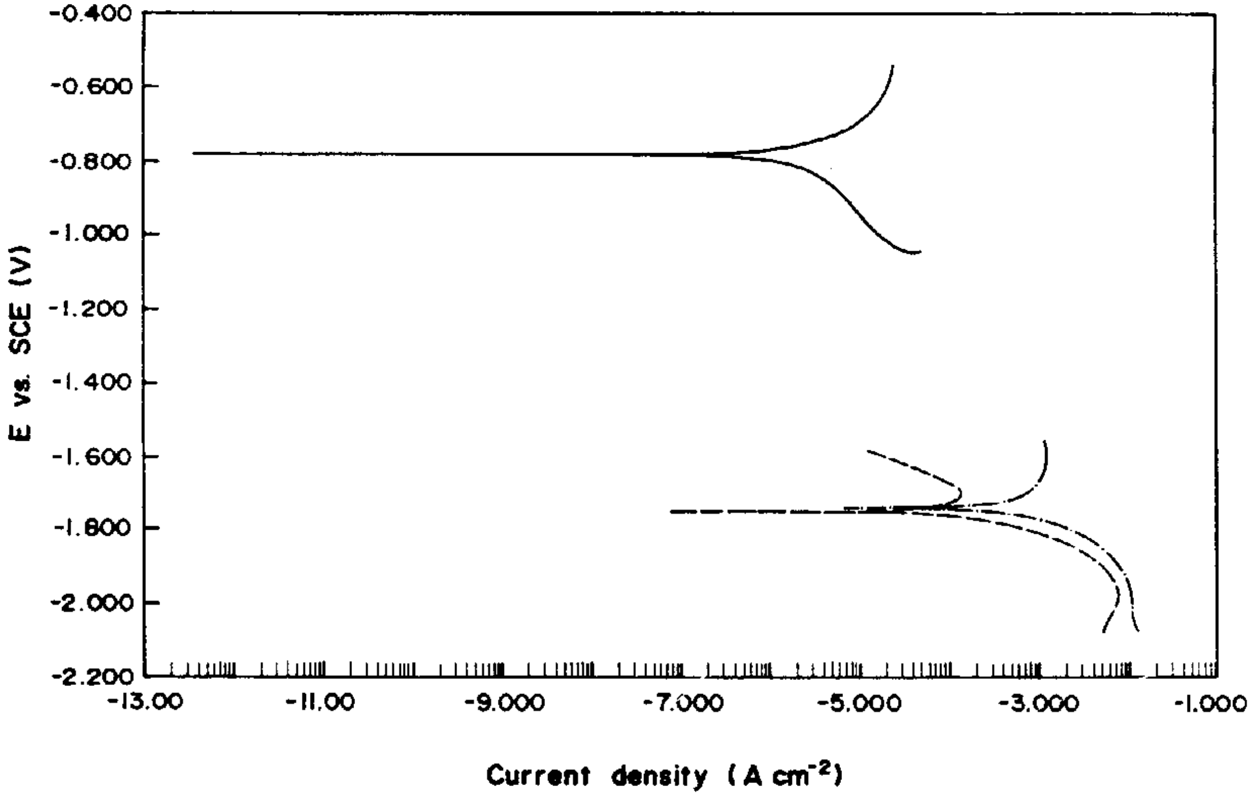

Fig. 8. Tafel plots for Al electrode in 0.5 M NaCit and(—)pH 3.5; (---) pH 9.3; (-·-·-)pH 12.5.

compete for the citrate ion [15, p. 37]. It is possible to explain that at low pH, citric acid will be adsorbed on the electrode surface leading to inhibition and thus the potential changes to the positive direction. At high pH the citrate ion forms different complexes with Al^{3+} resulting from the dissolution of Al at the anodic sites on the surface. The formation of a soluble complex between a metal ion and a ligand is known to enhance the dissolution of the metal [16]. Therefore, it may be possible that at fixed pH, the change in potential will be controlled by the competition between an adsorption and complex formation processes.

In order to test the effect of chloride ion, NaCl (0.02 - 0.50M), was added to solutions containing either 0.02M or 0.5M NaCit and OCP was followed as a function of time. In all cases the added Cl^-, makes the potentials change to the negative direction. Moreover, the E_{ss} values are more negative at higher [Cl^-] and they are reached faster at higher concentrations. This can be attributed to the adsorption of Cl^- ion on the electrode surface, where Cl^- ion enhance the anodic dissolution. In the presence of $CaCl_2$ the behavior of OCP as a function of time is similar to that observed with NaCl. It is, however, important to point out that E_{ss} is less negative at higher [$CaCl_2$]. In case of 0.02 M NaCit (pH≈8), E_{ss} was found to be independent of the salt concentration (Table 1). The more positive values of E_{ss} in the presence of $CaCl_2$ and citrate is an indication that Al is being less corrosive. This behavior may be due to the formation of a mixture of complexes of Ca^{2+} and Al^{3+} [17].

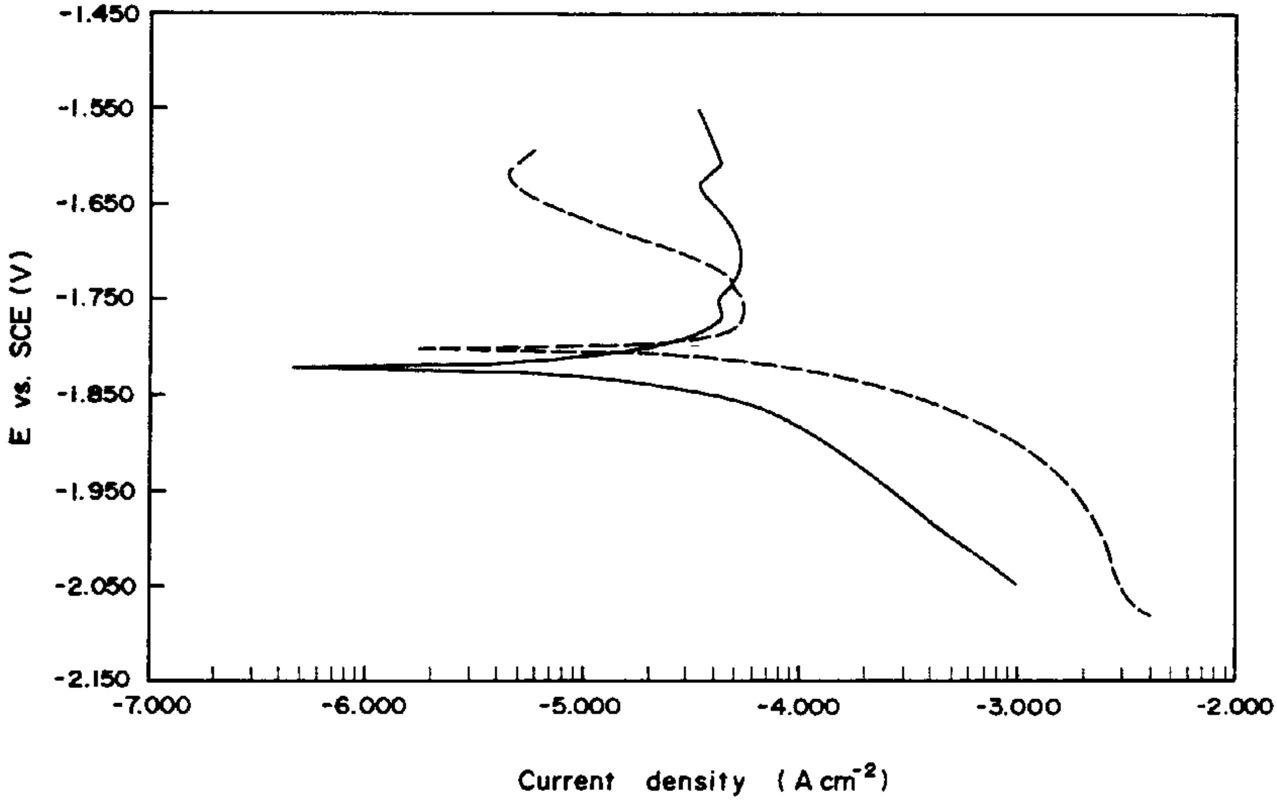

Fig. 9. Tafel plot for Al electrode in 0.5 M NaCit in presence of (---) NaCl; (---) $CaCl_2$.

Figure 5 shows that OCP for Al becomes more negative with time in the presence of 0.6 M HCl with no sign of reaching equilibrium which means that the metal is corroding but in the presence of different concentrations of citric acid, the potential is almost constant and attains equilibrium very shortly after immersion. It is to be noticed that in the presence of citric acid the potential oscillates and this was not observed in HCl solution or solutions containing citrate. This observation cannot be attributed to evolution of gases, otherwise it should be observed in HCl solution where the evolution of H_2 is considerable.

Table 1. Values of E_{SS} for Al electrode in 0.02 M NaCit in the presence of NaCl and $CaCl_2$ at pH 8 and at 25°C.

Concentration (M)	E_{ss} (mV) NaCl	 $CaCl_2$
0.00	-1749	-1749
0.02	-1756	-1596
0.05	-1769	----
0.10	-1777	-1601
0.25	----	-1559
0.50	-1777	-1574
1.00	-1748	-1523

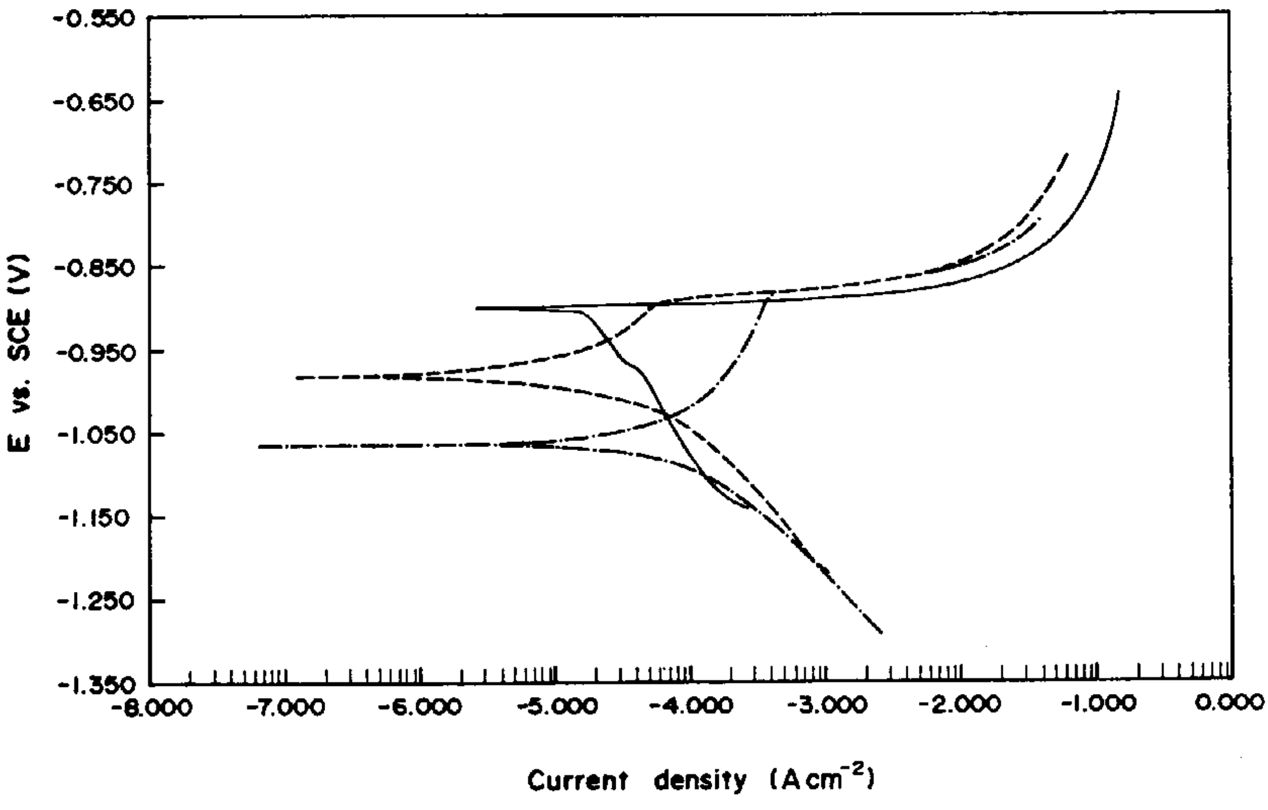

Fig. 10. Typical Tafel plot for Al in 0.6 M HCl in the absence and presence of various concentrations of CA (-·-·-)0.6 M CA; (---)0.01 M CA: (——)0.5 M CA.

As can be seen in Fig. 6 the E_{ss} becomes constant at [citric acid] $\geq$ 0.05M. In addition E_{ss} is more positive in presence of citric acid in comparison with citric acid free solutions and this is taken to indicate that the organic acid is behaving as an inhibitor.

Figure 7 shows the Tafel plots obtained for Al electrode at different NaCit concentrations. The general features of these curves are almost similar for both cathodic and anodic parts. The anodic part shows a sign of passivity in the potential range studied. The corrosion current densities change almost linearly with concentrations of citrate, as can be seen in Table 2. On the other hand, the Tafel plots at fixed citrate concentration but at different pH are not the same as can be seen in Fig. 8. These plots show that, while the cathodic parts are apparently the same, they are not so for the anodic parts. Thus at pH 9.3, the electrode is passivated and the current density decreases to about one order of magnitude of its critical value (CCd) which is about 400μA cm^{-2} with the primary passiviting potential being about -1720 mV. At the other two pH, the behavior is different as can be seen in Fig. 8. However, it is important to point out that in the very limited Tafel region the current densities increase exponentially with pH as can be seen in Table 2.

The polarization curves for solutions having fixed ratio of [citrate]/[citric acid] were found to be identical in spite of the fact that the concentrations of both components was raised fifty folds. The rate of corrosion was found to be independent to some extent of the increase of concentration of salt and acid (at constant ratio). The corrosion current density amounts to 1.5 ± 0.3μA cm^{-2} , whereas β_c equals to 0.166 ± 0.01 V/decade. This behavior which was not observed for solutions containing different [NaCit] or those

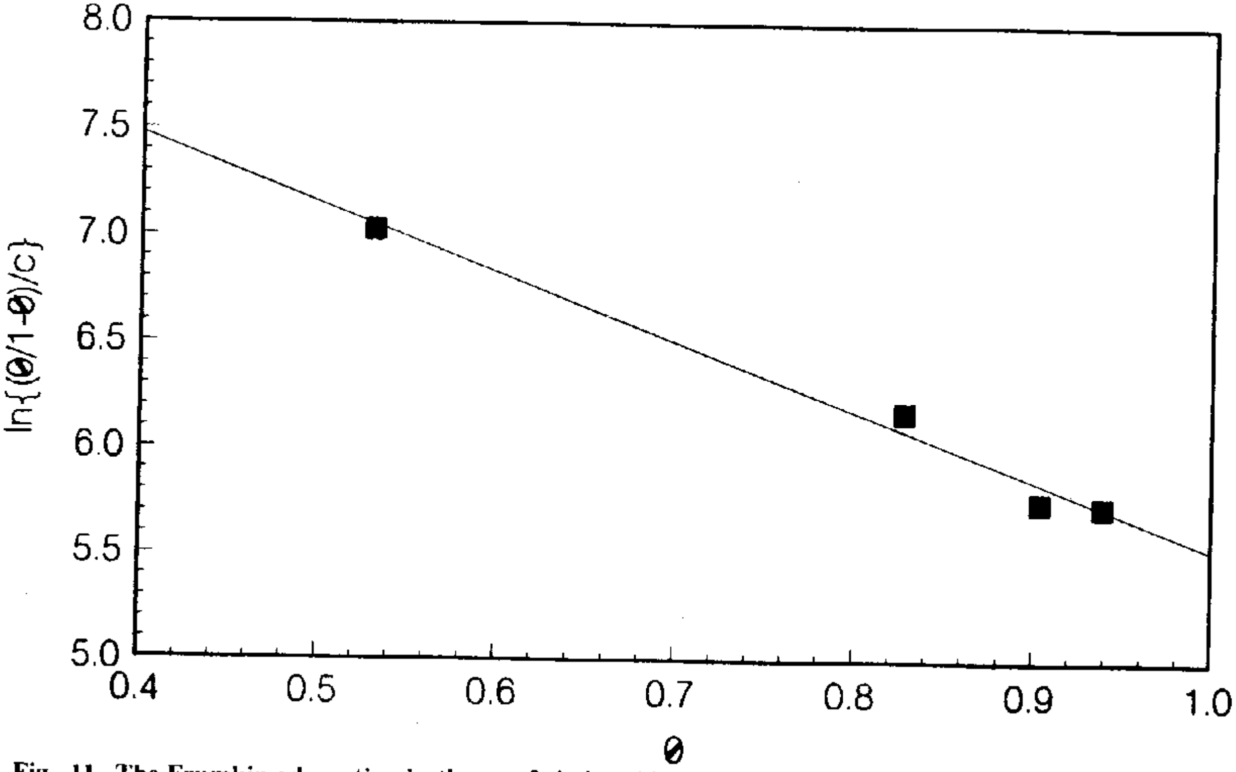

Fig. 11. The Frumkin adsorption isotherm of citric acid on Al electrode.

having the same concentration but with different pH is attributed to the presence of citric acid which seems to act as a weak inhibitor for both cathodic and anodic reactions.

For 0.5M NaCit the polarization curve obtained at 75°C was found to be very similar to that obtained at 25°C except that the primary passiviting potential is more negative than at 25°C and the CCd being one order of magnitude higher.

The addition of 0.1M of either NaCl or $CaCl_2$ to 0.5M NaCit yields the curves shown in Fig. 9. It is clear that the cathodic parts are not very different from that obtained at 0.5M NaCit at pH 9.3. The anodic curves, however, are not the same, where the current density decreases markedly in case of NaCl with a primary passivating potential of -1750 mV and a CCd of 60μA cm^{-2} . For $CaCl_2$, the anodic part of the curve shows a constant current density for a relatively wide range of potential. $CaCl_2$ shows some inhibitive action for the cathodic reaction. Table 2 summarizes the obtained parameters.

For 0.1M NaCl only it is observed that while the cathodic current density depends on potential, the anodic current density is potential dependent only for a very limited potential range (~ 50 mV relative to E_{corr}) and at more positive potentials the current density is stable. The addition of different concentrations of NaCit to 0.1M NaCl gave curves which have a common feature, particularly for the cathodic part. In case of the anodic parts, the passivity is set up very clearly at < 0.1M NaCit while at higher concentrations corrosion current density decreases to a minimum. Table 3 shows the

Table 2. Electrochemical data for Al electrode in different solutions of NaCit and CA

Conditions	E_{ss} (mV)	I_{corr} (μA cm^{-2})	βc
1- effect of NaCit:			
0.002M	-1466	1.180	0.066
0.02M	-1630	10.36	0.066
0.20M	-1841	154.2	0.077
2- effect of pH (0.5M NaCit):			
3.5	-797	1.770	0.144
9.3	-1835	190.7	0.079
12.5	-1820	741.2	0.106
3- effect of Temp. (0.5M NaCit):			
75°C	-1919	955.7	0.112
4- effect of CA:			
0.1M	-786	0.941	0.167
0.5M	-705	1.160	0.160
1.0M	-793	0.883	0.114
5- effect of Cl^- (0.5M CA):			
0.1M NaCl	-769	2.020	0.180
0.5M NaCl	-819	4.310	0.175
6- effect of Cl^- (0.5M NaCit):			
0.1M NaCl	-1833	179.4	0.107
0.1M $CaCl_2$	-1820	38.55	0.099

current density obtained for Al electrode in 0.1M NaCl with different concentrations of NaCit. It is clear that the current density attains maximum values at 0.1M NaCit.

At fixed 0.02M NaCit and in the presence of different concentrations of NaCl the polarization curves are very similar for both cathodic and anodic parts. The passivity is attained at a primary passivating potential of -1670 mV and a CCd of 20 μA cm^{-2} for all solutions of NaCit having 0.02M to 1M NaCl which is also similar to that obtained in the absence of NaCl. At this concentration of NaCit, the rates of cathodic and anodic reaction are almost constant as shown in Table 3.

It is possible that the positive drift in the potential shown in Fig. 5 is due to the protonated citric acid. Therefore, polarization curves were obtained in the same solutions to see the effect on the cathodic and anodic processes. Fig. 10 shows some typical polarization curves for Al electrode immersed in 0.6M HCl in absence and presence of different concentrations of citric acid. These figures illustrate that pitting is taking place for solutions having 0.6M HCl or those containing < 0.03M citric acid. Moreover, it is obvious that the pitting corrosion potentials are the same while the current density is higher by about one order of magnitude for 0.6M HCl. At higher concentrations of citric acid, the pitting is no longer observed.

Table 3. Electrochemical data for Al electrode in solutions containing NaCit and NaCl at 25°C

0.1M NaCl				0.02M NaCit			
NaCit (M)	E_{ss} (mV)	I_{corr}(μAcm^{-2})	β_c	NaCl (M)	E_{pit} (mV)	I_{corr}(μAcm^{-2})	β_c
0.00	-1430	0.666	0.123	0.00	-1756	10.46	0.113
0.02	-1756	15.10	0.129	0.02	---	---	---
0.05	-1822	13.17	0.06	0.05	-1769	11.68	0.105
0.10	-1817	116.1	0.106	0.10	-1777	17.04	0.105
0.50	-1580	8.5	0.10	0.50	-1777	15.90	0.105
1.0	-1534	0.195	0.127	1.0	-1748	75.59	0.104

Table 4. Electrochemical data for Al electrode in 0.6M HCl in the presence of different citric acid concentrations at 25°C

Citric Acid (M)	E_{ss} (mV)	E_{pit} (mV)	I_{corr}(μAcm^{-2})	β_c
0.0	-1066	-884	80.15	0.128
0.001	-1078	-902	37.50	0.073
0.01	-982	-890	13.70	0.058
0.03	-962	-900	7.60	0.075
0.05	-898	---	4.19	0.050
0.2	-901	---	7.97	0.087
0.5	-896	---	6.77	0.140
1.0	-907	---	9.66	0.112

Table 4 summarizes the corresponding electrochemical data. In addition, the present results indicated that citric acid acts as a mixed inhibitor and that the results obey the Frumkin adsorption isotherm which is written in the form

$$(\theta/1\text{-}\theta)\, e^{-2a\theta}=KC \tag{2}$$

where K is the binding constant of the additive to the Al surface, C is the concentration of the inhibitor, a is constant (the lateral interaction term) and θ is the surface coverage which is calculated from the relationship.

$$\theta=i_O\text{-}i\,/\,i_O \tag{3}$$

where i_O and i are the current densities in the absence and presence of the inhibitor respectively. Such a plot is shown in Fig. 11. It is clear that the isotherm is applicable and that (K) and (a) were calculated and found to be 6374 and -1.6, respectively. On the other hand, the dissolution of Al, i.e. the anodic reaction was found to be greatly enhanced in the presence of citric acid, and such results are shown in Fig. 10. The negative value of (a) though it is small, indicates that the adsorbed species have similar charges and hence a repulsion is taking place. Due to the fact that CA can easily be protonated ($K_{prot} \approx 10^5$) [15] and since the medium is highly acidic, it may be concluded that the adsorbed species is the protonated citric acid. The enhanced increase in the dissolution of the metal at such conditions may be attributed to the reaction of Al^{3+}, resulting from anodic dissolution with citric acid (and citrate) which leads to the formation of different alumino-complexes. The formation of mono and polynuclear Al-Cit complexes are well known [18] as for example $Al(HCit)^+$, Al(Cit) and $[Al_3\,(OH)_4\ Cit_3]^{4-}$.

Conclusions

On the basis of the study, the following conclusions can be drawn:

1. The steady state potential of Al in citrate solutions was found to depend on citrate ion concentration, Cl^-, pH and temperature. The increase in these factors leads to a negative change in the potential.
2. At [citrate ion]/[citric acid] ≈ 1 (pH 4.3), the OCP drifts positively but E_{ss} is almost constant.
3. From the OCP measurements, it was concluded that citrate ion accelerate the corrosion of Al while citric acid has an inhibitive effect.
4. In the presence of Cl^- (as NaCl or $CaCl_2$) and citrate, the OCP measurements indicate that the corrosion of Al is faster than in their absence.

5. The polarization curves for Al at conditions similar to those mentioned above were found to support these results obtained from the OCP measurements.
6. In the presence of citric acid, the Frumkin isotherm was found to be applicable for the adsorption of citric acid on Al surface. The equilibrium constant for adsorption and the lateral interaction term were evaluated.

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تآكل الألومنيوم في المحاليل المائية التي تحتوي على أيونات الكلور و/ أو أيونات الليمونات

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الرياض ١١٤٥١ ، المملكة العربية السعودية

(استلم في ١٢ رمضان ١٤١٥هـ؛ قبل للنشر في ٣٠ محرم ١٤١٧هـ)

ملخص البحث. تمت دراسة تآكل الألومنيوم النقي في المحاليل المائية الحاوية على أيونات الكلور و/ أو أيونات الليمونات. وقد اتضح أن الألومنيوم يتآكل في وجود أيونات الليمونات بدرجة تعتمد على تركيز هذه الأيونات، إضافة إلى درجة الحرارة والرقم الهيدروجيني وتركيز أيونات الكلور. من جهة أخرى، فقد وجد أن لحمض الليمون تأثير مانع للتآكل نتيجة امتزازه على سطح الألومنيوم الذي يتبع متساوي الدرجة (أيزوثيرم) فرومكن.