

Inhibition of Aluminum Corrosion in Hydrochloric Acid by Cellulose and Chitosan

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Abstract: The inhibitive effects of the natural polymer namely, carboxy methyl chitosan (CMCH) and carboxy methyl Cellulose (CMC) for the corrosion of aluminum in 0.5 M HCl solution was investigated using weight loss measurements, galvanostatic, and potentiodynamic anodic polarization techniques. The results drawn from these techniques indicated that the inhibition efficiency increased with the concentration of inhibitor but decreased with temperature. Some activated thermodynamic parameters were computed. The inhibition efficiency of the CMCH is more than CMC depends on the chemical structure of the inhibitor.. Inhibition was explained in term of the adsorption of these compounds on the aluminum surface. The adsorption process is fitting a Temkin adsorption isotherm. Polarization data proves that these inhibitors are mixed inhibitors. It was found that CMCH and CMC compounds protect the aluminum surface from pitting attack in chloride containing solution by shifting the pitting corrosion potential to more noble direction.

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1. Introduction

Aluminum (Al) and its alloys are widely used in many industries such as reaction vessels, pipes, machinery and chemical batteries because of their low density and good resistance to corrosion.. Hydrochloric acid solution is used for pickling, chemical and electrochemical etching of Al. Aluminum generally exhibits passive behavior in aqueous solution due to the formation of strong and compact adherent passive oxide film on its surface which influences its corrosion susceptibility. The adhesive passivating surface oxide film is amphoteric and consequently the metal dissolves readily when exposed to aggressive acidic solution. In fact, the corrosion of metallic materials in acidic solution causes considerable costs [1]. One of the most practical methods for protection of metal against corrosion in acidic solution is use of organic compounds [2-10]. The inhibition action of these compounds is due to the adsorption on the metal surface. The adsorption process depends mainly on certain physico-chemical properties of the molecule such as functional groups, steric factors, aromaticity, electron density at the donor atoms, π -orbital's character of donating electrons [11,12] and also on the electronic structure of the molecules. The inhibition efficiency increase with increasing the number of aromatic rings[13,14]. Numerous organic compounds including polymers have been proved to

be effective corrosion inhibitors for metals in acidic medium [1].

The aim of the present work is to study the inhibition action of the natural polymers namely, carboxy methyl chitosan (hydrogel polymer) (CMCH) and carboxy methyl Cellulose (polysaccharide (CMC)) for aluminum in 0.5 M HCl solution using different techniques, e.g., weight loss at different temperatures, galvanostatic and potentiodynamic anodic polarization.

2. Experimental

Aluminum metal with purity 99.95% provided by the "Aluminum Company of Egypt, Nagh Ammady" was studied in the present work . For weight loss measurements, corrosion inhibition tests were performed using coupons measuring 1 x 2 x 0.1 cm³ prepared from pure aluminum. The aluminum coupons were polished with emery papers, then degreased with acetone and washed with distilled water. The coupons were dried and kept in a desiccator. The weight loss (g cm⁻²) was determined at different immersion times at 30°C by weighing the cleaned samples before and after hanging the coupon into 50 cm³ of the corrosive solution. For electrochemical measurements, the investigated materials were cut as cylindrical rods, welded with Cu-wire for electrical connection and mounted into glass tubes of appropriate diameter using Araldite to offer an active flat disc shaped surface of(0.785 cm²)

geometric area, to contact the test solution. Prior to each experiment, the surface pretreatment of the working electrode was performed by mechanical polishing (using a polishing machine model POLIMENT I, BUEHLER POLISHER) of the electrode surface with successive grades of emery papers down to 1200 grit up to a mirror finish. The electrode was then, rinsed with acetone, distilled water, and finally dipped in the electrolytic cell. A conventional electrochemical cell of capacity 100 cm³ was used containing three compartments for working, platinum spiral counter and reference electrodes. A Luggin–Haber capillary was also included in the design. The reference electrode was a normal calomel one used directly in contact with the working solution. The experiments were carried out in aerated stagnant and containing various concentrations of the three polymers. All solutions were prepared from analytical grade chemical reagents using doubly distilled water and were used without further purification. The potentiodynamic current–potential curves were recorded by changing the electrode potential automatically from -2.0 to 2.0 V at a scan rate of 1 mV sec⁻¹. Both cathodic and anodic polarization curves were recorded galvanosttically using EG & G model 363 potentiostat/galvanostat corrosion measurement system and digital millimeters (Fluke-73) were used for accurate measurements of potential and current density. Solutions were not decreased to make the conditions identical to weight loss measurements. All the experiments were carried out at 25±1°C by using ultra circulating thermostat.

The chemicals and solutions were provided by Sigma Aldrich, Germany. Hydrochloric acid (HCl), carboxy methyl chitosan ,carboxy methyl cellulose and sodium chloride were provided by El Gomhoria Co., Egypt. Used water was Deionized Double distilled water (DDI)with a resistivity of 18MΩcm. All purchased Chemicals were used without additional purification. The natural polymers used as inhibitors are carboxy methyl chitosan(CMCH), compound I and carboxy methyl cellulose(CMC), compound II.

3. Results and Discussion

3.1. Weight loss measurements

The corrosion behavior of (Al)in is characterized by the extent to which it dissolves in the solution. The degree of dissolution, of course, dependent on the surface area of the metal exposed and the time of exposure; hence the amount of corrosion is given with respect to area and time. The resulting quantity, corrosion rate, is thus a fundamental measurement in corrosion science. Corrosion rates can be evaluated by measuring either

the concentration of the dissolved metal in solution by chemical analysis or by measuring is directly related to the extent of corrosion and does not rely on any assumptions about reactions occurring during corrosion the weight losses given by:

$$\Delta W = W_1 - W_2 \quad (1)$$

where W₁ and W₂ are the weights of specimen before and after reaction, respectively. The inhibition or acceleration efficiency %I was computed from the equation:

$$\%I = \frac{\Delta W - \Delta W_i}{\Delta W} \times 100 \quad (2)$$

where ΔW and ΔW_i are the weight losses per unit area in absence and presence of the additive, respectively.

Fig. 1 shows the weight loss –time curves for aluminum coupons in 0.5 M HCl solution in the absence and presence of different concentrations of compound CMCH at 30°C as an example. Similar curves was obtained for other compound CMC (not shown).It is obvious that the weight loss of Al in presence of CMCH and CMC varies linearly with time, and is much lower than that obtained in the blank solution. The linearity obtained indicated the absence of insoluble surface film during corrosion and the inhibitor were first adsorbed onto the metal surface and thereafter, impede the corrosion process[15]

The percentage of inhibition efficiency (%I.E) of polymer compounds was determined by using the equation.

$$\%IE = (1 - I_{add}/I_{free})100 \quad (4)$$

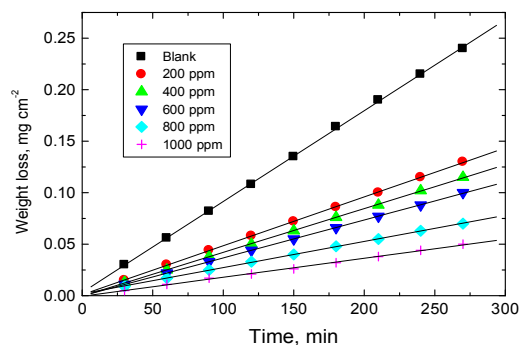


Fig. 1: Weight loss-time curves for aluminum in 0.5 M HCl solution in the absence and presence of different concentrations of compound CMCH at 30°C

The calculated values of %I.E are given in Table 1. It is clear from this table, the order of the inhibition efficiency of polymer compounds decreases in the following order:

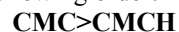


Table 1: Percentages of Inhibition of aluminum dissolution at 210 min. immersion in 0.5 M HCl in the presence of different concentrations of different polymeric compounds at 30 °C

% Inhibition (I.E %)		
Concentration, ppm	CMCH	CMC
200	39.4	48.5
400	45.8	57.2
600	54.2	64.1
800	63.02	73.6
1000	69.8	81.3

3.2. Effect of temperature and activation parameters

The effect of temperature on the corrosion rate of Al in 0.5 M HCl solution containing 1000ppm of two polymer compounds was studied using weight loss measurements over temperature range from 30-60° Similar figures were obtained to figure 1 (not shown).

The effect of rising temperature on the values of the inhibition efficiency is listed in Table 2. As the temperature increases the weight loss increase and hence the inhibition efficiency of the two polymer compounds decreases. This is due to the desorption is aided by increasing the temperature This means that the adsorption of polymeric compounds on the aluminum surface is physically.

Table 2: Percentage of Inhibition efficiency of aluminum dissolution in 0.5 M HCl in the presence of 1000 ppm concentrations of the different polymer compounds at different temperatures and at 210 min. immersion.

Temperature, K	% Inhibition (I.E %)	
	CMCH	CMC
303	69.8	81.3
313	58.7	65.6
323	47.9	58.4
333	40.6	49.8

The results of table (2) show that the percentage of inhibition is decreased with increasing the temperature, this behavior can be explained on the basis that, the increase of the temperature leads to desorption of the adsorbed molecules of the inhibitors from the metal surface.

The apparent activation energy E_a^* , the enthalpy of activation ΔH^* and the entropy of activation ΔS^* for the corrosion of aluminum samples in 0.5M HCl solutions in the absence and presence of 1000 ppm of

polymer compounds were calculated from Arrhenius-type equation[16]:

$$\text{Rate} = A \exp(-E_a^*/RT) \quad (5)$$

and transition – state equation:

$$\text{Rate} = RT/Nh \exp(\Delta S^*/R) \exp(-\Delta H^*/RT) \quad (6)$$

where A is the frequency factor h is the plank's constant, N is Avogadro's number and R is the universal gas constant. A plot of log rate vs. 1/T (Fig.2) give straight lines with slope of $-E_a^*/2.303R$

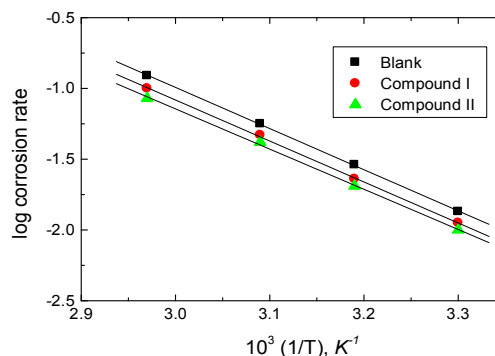


Fig. 2: Log corrosion rate-1/T curves for aluminum dissolution in 0.5 M HCl in absence and/or presence of 1000ppm of polymer compounds

Figure (3) represent plots of the log rate \log_{rat}/T vs. 1/T are obtained straight lines with slope of $(-\Delta H^*/2.303R)$ and an intercept of $\log[(RT/Nh) - (\Delta S^*/2.303R)]$.

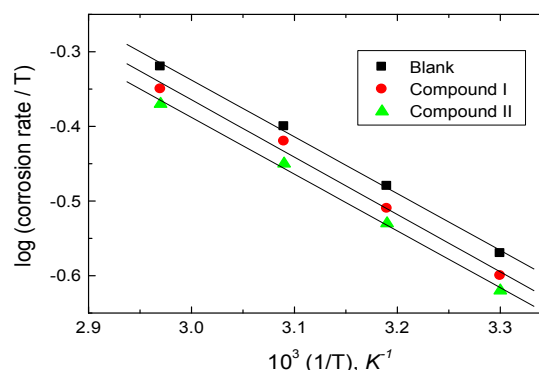


Fig.3: Log(corrosion rate/T) – (1/T) curves for aluminum dissolution in 0.5 M HCl in absence and/or presence of 1000 ppm of polymer compounds

The calculated values of the apparent activation energy, E_a^* , activation entropies, ΔS^* and activation enthalpies, ΔH^* are given in table (3).

Table 3: Activation parameters of aluminum dissolution in 0.5 HCl solution in the absence and presence of 1000 ppm polymer compounds.

Inhibitors	Activation parameters		
	Ea*,K J mol ⁻¹	ΔH*,KJ mol ⁻¹	- ΔS*, J mol ⁻¹
Free acid	52.32	51.36	90.56
CMCH	61.72	57.85	75.6
CMC	63.95	59.54	74.9

From the results of Table (3), it is clear that the presence of the tested compounds increase the activation energy values and consequently decreased the corrosion rate of the aluminum. These results indicate that these tested compounds act as inhibitors through increasing activation energy of aluminum dissolution by making a barrier to mass and charge transfer by their adsorption on aluminum surface. The positive signs of ΔH* reflect the endothermic nature of the aluminum dissolution process. The values of ΔH* are different for studied compounds which means that their structure affect the strength of its adsorption on the metal surface.

The negative values of ΔS* in the absence and presence of the inhibitors implies that, the activated complex is the rate determining step and represents association rather than dissociation. It also reveals that an increase in the order takes place in going from reactants to the activated complex[17]. The order of the inhibition efficiency of polymeric compounds as gathered from the increase in Ea* and ΔH* values and decrease in ΔS* values are:

$$\text{CMCH} > \text{CMC}$$

3.3. Adsorption isotherm

The efficiency of natural polymer as corrosion inhibitor mainly depends on its adsorption ability at the metal / solution interface. The adsorption depends on the structure of the inhibitor, the type of the metal and the nature of its surface, the nature of the corrosion medium and its pH value, the temperature and the electrochemical potential of the metal-solution interface. Also, the adsorption provides information about the interaction among the adsorbed molecules themselves as well as their interaction with the metal surface.

The surface coverage(θ) which represents the part of the metal surface covered by the inhibitor molecules was calculated using the following equation:

$$\theta = [1 - (W/W^0)] \times 100 \quad (7)$$

where, W⁰ and W are the values of the average weight loss without and with addition of the inhibitor, respectively.

Adsorption isotherm equations are generally of the form:

$$f(\theta, x) \exp(-a, \theta) = KC \quad (8)$$

where f(θ, x) is the configurationally factor that depends essentially on the physical model and assumptions underlying the derivation of the isotherm a is a molecular interaction parameter depending upon molecule interactions in the adsorption layer and the degree of heterogeneity of the surface. All adsorption expressions include the equilibrium constant of the adsorption process, K, which is related to the standard free energy of adsorption (ΔG⁰_{ads}) by:

$$K = 1/55.5 \exp - \Delta G_{\text{ads}} / RT \quad (9)$$

where R is the universal gas constant and T is the absolute temperature. A number of mathematical relationships for the adsorption isotherms have been suggested to fit the experiment data of the present work. The simplest equation is that due to Temkin and is given by the general equation:

$$\ln KC = a \theta \quad (10)$$

where K is the equilibrium constant of the adsorption reaction, C is the inhibitor concentration in the bulk of the solution, a is the interaction parameter and θ is the surface coverage. The surface coverage, i.e., the fraction of the surface covered by the inhibitor molecules.

Plots of θ vs. log C (Temkin adsorption plots) for adsorption of polymeric compounds on the surface of aluminum in 0.5 M HCl acid at 30°C is shown in Fig. (4).

The data gave straight lines indicating that Temkin's isotherm is valid for these systems. Temkin's isotherm is applied for ideal case of physical and chemical adsorption on a smooth surface with no interaction between the adsorbed molecules.

On the other hand, it is found that the kinetic-thermodynamic model of El Awady *et al.* [18]

$$\log \theta / (1 - \theta) = \log K + y \log C \quad (11)$$

is valid to operate the present adsorption data. The equilibrium constant of adsorption K = K (1/y), where 1/y is the number of the surface active sites occupied by one polymeric compounds molecule and C is the bulk concentration of the inhibitor. The relation between log θ / (1 - θ) against log C is given in Fig(5).

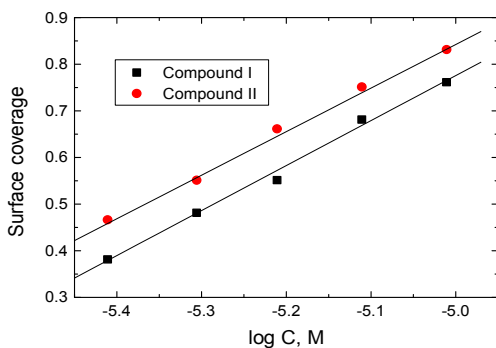


Fig. 4: Curve fitting of corrosion data for aluminum in 0.5 M HCl in presence of different concentration of polymer compounds to the Temkin isotherm at 30°C

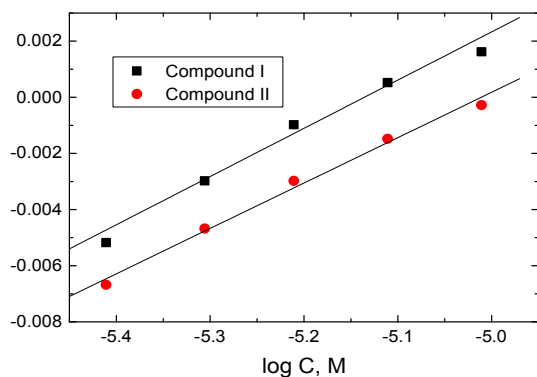


Fig. 5: Curve fitting of corrosion data for aluminum in 0.5 M HCl in presence of different concentration of polymer compounds to the kinetic model at 30°C. The calculated values of $1/y$, K and ΔG_{ads}^0 are given in table (4).

Table 4: Inhibitor binding (K), free energy of binding (ΔG_{ads}^0), number of active sites ($1/y$) and later interaction (a) for polymeric compounds at 30°C.

Inhibitor	Kinetic model			Temkin		
	$1/y$	$K \times 10^{-5}$	$-\Delta G_{ads}^0$ KJ mol $^{-1}$	a	$K \times 10^{-6}$	$-\Delta G_{ads}^0$ KJ mol $^{-1}$
I	0.56	25.87	46.45	3.8	31.36	47.78
II	0.77	29.97	43.15	4.5	37.72	48.25

Inspection of the data of these tables shows that the large values of ΔG_{ads}^0 and its negative sign, indicate that the adsorption of polymeric compounds on the aluminum surface is proceeding spontaneously and is accompanied by a highly-efficient adsorption. It is worth noting that the value of $1/y$ is more than unity. This means that the given inhibitor molecules will occupy more than one active site. In general, the values of ΔG_{ads}^0 obtained from El-Awady *et al.*,

model are comparable with those obtained from Temkin's isotherm.

From these results it may be generalized that the more efficient inhibitor has more negative ΔG_{ads}^0 . Value so that from the tabulated values of ΔG_{ads}^0 the order of the inhibition efficiency of polymer compounds decreases in the following order:



3.4. Galvanostatic Polarization Technique.

Fig.6 represents the anodic and cathodic polarization curves of aluminum electrode in 0.5M HCl solutions containing different concentrations of CMC compound as an example. Similar curves were obtained for the CMCH compound (not shown)

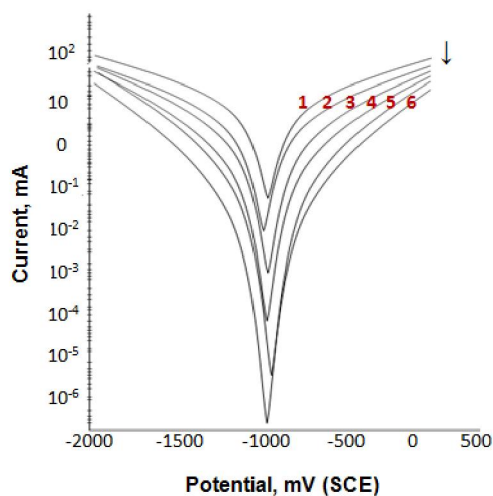


Fig. 6: Galvanostatic polarization curves of aluminum in 0.5M HCl containing different concentrations of CMC compound. (1) 0.00 (2) 200 (3) 400 (4) 600 (5) 800 (6) 1000 ppm.

Inspection of Fig.6 reveals that, both anodic and cathodic polarization curves are shifted to less current density values in presence of polymer compound. This behavior suggests the inhibitive action of carboxy methyl chitosan and carboxy methyl cellulose compounds

The values of corrosion current density (I_{corr}) were determined by the intersection of the extrapolated cathodic and anodic Tafel lines (linear part) with the stationary corrosion potential (E_{corr}).

The percentage inhibition efficiency (% I.E) imparted by the added inhibitor, which is defined as the percentage of the relative decrease in corrosion rate brought about by the presence of a certain concentration of the inhibitor is given by:

$$\% \text{ I.E} = (1 - I_{add} / I_{free}) \times 100 \quad (12)$$

where, I_{free} and I_{add} are the corrosion current densities in the absence and presence of the inhibitors, respectively.

Tables (5) show the effect of different concentrations of polyethylene glycol compounds on the corrosion parameters such as: cathodic Tafel slope (b_c), anodic Tafel slope (b_a), corrosion potential (E_{corr}), corrosion current density (I_{corr}), percentage inhibition efficiency (%I.E) and surface coverage (θ). An inspection of this table, it is clear that the corrosion potentials (E_{corr}) is shifted to more negative values and Tafel lines are shifted to more positive and negative

potential for anodic and cathodic process, respectively, relative to the blank curve. This means that these compounds influence both anodic and cathodic process. However, the data suggested that these compounds act mainly as mixed type inhibitors. The values of anodic and cathodic Tafel slopes (b_a & b_c) are approximately constant which suggest the simple blocking of the available surface area of the metal by the inhibitor molecules.

Table 5: The effect of concentration of compound (I) on the free corrosion potential (E_{corr}), Corrosion current density (I_{corr}), Tafel slopes (β_a & β_c), inhibition efficiency (%I.E) and Degree of surface coverage (Θ) in presence of 0.5 HCl at 30° C.

Inhibitors	Concentration (ppm)	$-E_{corr.}$ mV. (SCE)	$I_{corr.}$ mAcm ⁻²	β_a , m V dec ⁻¹	β_c , m V dec ⁻¹	Θ	%I.E
CMCH	0	799	155	217	89	----	----
	200	767	82	168	120	0.35	35.5
	400	758	74	140	122	0.38	38.2
	600	753	60	135	125	0.47	47.6
	800	724	52	138	129	0.54	54.3
	1000	936	30	122	132	0.70	70.9
CMC	0	799	155	217	89	-----	-----
	200	774	77	228	135	0.40	40.2
	400	767	70	250	140	0.44	44.7
	600	758	52	262	146	0.49	49.9
	800	753	48	290	165	0.52	52.6
	1000	724	26	297	152	0.74	74.9

In other words, the adsorbed inhibitor molecule decreases the surface area available for the both metal dissolution and hydrogen evolution reactions without affecting the reaction mechanism [19]. The reaction between the metal covered with inhibitor molecule may take place by diffusion H^+ and Cl^- ions through the pores of the protective layer on the metal surface.

From these Fig. (6) and table (5) it is clear that, the increase of additive concentrations decreases the corrosion current density (I_{corr}), and hence increases the inhibition efficiency of these compounds. The values of inhibition efficiency of polymers compounds increases in the following order:

$$CMC > CMCH$$

3.5. Inhibition of Pitting Corrosion of aluminum

Fig.7 represents the of potentiodynamic anodic polarization curves of aluminum electrode in 0.5M HCl+0.5 M NaCl (as a pitting corrosion agent) in absence and present of different concentrations of CMC compound at a scanning rate of 1mVsec⁻¹ as an example of the additives compounds. Similar curves were obtained for the other CMCH compounds (not shown). The potential was swept from negative potential toward anodic direction up to the pitting potential ($E_{pitt.}$). The pitting potential was taken as the potential at which the current flowing, along the passive film increases suddenly to higher values,

denoting the destruction of passive film and initiation of visible pits. It was found that the pitting potential of the aluminum electrode is shifted to more positive (noble) values with increasing the concentration of these additives. This indicates that increased resistance to pitting attack [20].

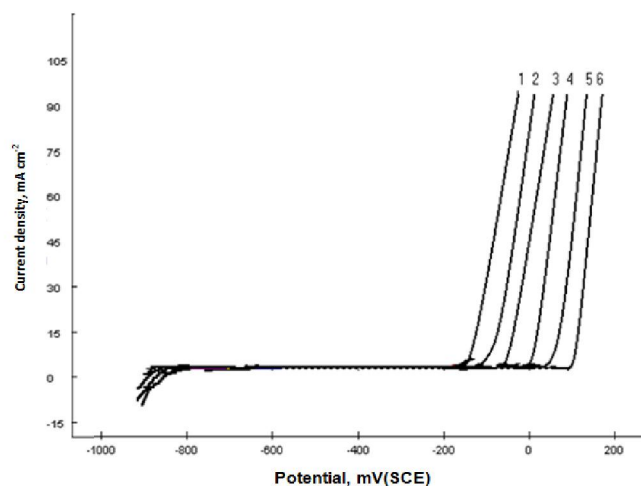


Fig. 7: Potentiodynamic anodic polarization curves of aluminum in 0.5M HCl solution + 0.5M NaCl containing different concentrations of compound CMC at a scan rate 1mVsec⁻¹. (1) 0.00 (2) 200 (3) 400 (4) 600 (5) 800 (6) 1000 ppm.

Fig. (8): represents the relationship between pitting potential and the logarithmic of the molar concentration of the added compounds. Straight lines were obtained and the following conclusion can be drawn:

a. The increase of inhibitor concentration causes the shift of the pitting potential into more positive values in accordance with the following equation:

$$E_{\text{pitt}} = a_1 + b_1 \log C_{\text{inh}} \quad (13)$$

where, a_1 and b_1 are constants which depend on both the composition of additives and the nature of the electrode.

b. Inhibition afforded by these compounds using the same different concentrations of the additives decreases in the following order:

$$\text{CMC} > \text{CMCH}$$

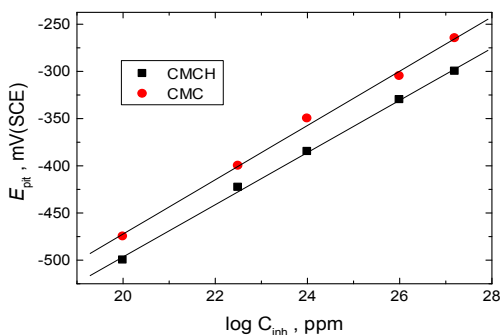


Fig. 8: The relationship between the pitting potential of aluminum and logarithm the concentration compounds in presence 0.5 M NaCl solution. compound (1) is CMCH compound (2) is CMC.

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