

ELECTROCHEMICAL STUDIES OF PITTING CORROSION OF CU-FE ALLOY IN SODIUM CHLORIDE SOLUTIONS

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ABSTRACT

The electrochemical studies of pitting corrosion of Cu- Fe alloy (70% Cu- 30%Fe) were carried out in sodium chloride solutions. The techniques of measurements were: open circuit potential, potentiodynamic polarization and electrochemical impedance spectroscopy. The effect of different concentrations of the inorganic additives (WO_4^{2-} , PO_4^{3-} , NO_2^- , $\text{B}_4\text{O}_7^{2-}$, CrO_4^{2-}) on the pitting corrosion of the alloy in 0.5 M NaCl was also studied. It was found that the alloy underwent pitting corrosion in sodium chloride solution at definite pitting potential depending on the concentration of NaCl according to the relation: $E_{\text{pit}} = q_1 - b_1 \log C_{\text{Cl}^-}$. The investigated inorganic additives had inhibiting effect on the pitting corrosion of the alloy in 0.5 M NaCl and their efficiency decreases according to the order $\text{WO}_4^{2-} > \text{PO}_4^{3-} > \text{NO}_2^- > \text{B}_4\text{O}_7^{2-} > \text{CrO}_4^{2-}$.

Key Words: Pitting corrosion, Potentiodynamic Impedance spectroscopy, Inhibiting effect of inorganic additives.

INTRODUCTION

Copper and its alloys are widely used as preferred materials for transport of water in domestic and industrial water utilities, in plumbing within buildings and heat exchangers and also used in the petrochemical, marine, food, paper, and textile industries. This related to their corrosion resistance (mechanical workability, excellent electrical and thermal conductivity and resistance to macrofouling^[1]). However, under some exceptional conditions, pitting corrosion of copper and its alloys may occur, especially in the presence of certain anions such as chloride, bicarbonate, sulphate, nitrate and others. Several authors ^[2-7] studied the pitting corrosion of copper and its alloys in chloride solutions. Each of them examined the issue from different viewpoints and with different experimental treatments, in the presence of various anions.

In the present work the electrochemical studies of pitting corrosion of Cu- Fe alloy in sodium chloride solutions were carried out using different electrochemical techniques of measurements. Also, the effect of different inorganic additives on the pitting corrosion of the alloy was studied.

EXPERIMENTAL WORK

The investigated copper – iron alloy (70% Cu- 30% Fe) was prepared in non-ferrous laboratory at (MRDI), Egypt. The materials were melted using crucible furnace at temperature range (100 – 1550°C and then casted into cylindrical rods 10cm length and 1cm diameter. For the electrochemical measurements, the electrodes were prepared from the casted rods. A stout copper wire lead was fixed at one end of the cylindrical electrode by mechanical jamming. The electrode was then fitted into a Pyrex glass tubing of appropriate internal diameter and fixed with araldite. Just before use the electrodes were mechanically polished by emery papers of different grades up to 4/0. The polished electrode were then washed thoroughly with distilled water and then degreased by acetone.

In this work the electrochemical behaviour of copper –iron alloy was studied in NaCl solutions with different concentrations. The effect of different concentrations of the inorganic additives WO_4^{2-} , PO_4^{3-} , NO_2^- , $\text{B}_4\text{O}_7^{2-}$, and CrO_4^{2-} in 0.5 M HCl on the pitting corrosion investigation the techniques of measurements were (i) open circuit potential, (ii) potentiodynamic polarization, (iii) electrochemical impedance spectroscopy. In open- circuit measurements the potential was measured by digital multimeter (model 1008, Kyoritsu, Japan). The potentiodynamic polarization was performed at scanning rate of 1mv/ sec, using a Wenking potentiostan Type POS 73. The current density – potential curves were recorded on X-Y recorder Type PL-3. The potentials were measured relative to a saturated calomel electrode (SCE).

The impedance measurements were carried out using impedance measurement system (1 M6 Zahner elektrik Meßtechnik, Germany).

RESULTS AND DISCUSSION

Figure (1) shows the corrosion potential – time plots of the investigated electrode immersed in different concentrations of sodium chloride solution (1×10^{-4} – 5×10^{-1} M) under open- circuit conditions. It is clear from these plots that on immersion of the electrode in sodium chloride solutions the corrosion potential shifts to more negative (active) direction till reaching its steady state. The values of steady state potential for the electrode and time required to reach it are listed in Table (1). These data indicate that with increasing sodium concentration the more negative values of steady state potential are obtained and longer time to reach it is required.

Figure (2) represents the variation of the steady state potential, E_{ss} , with the logarithm of NaCl concentration, $\log C_{\text{Cl}^-}$. The plot of this figure indicate that E_{ss} slightly shifts to the more negative direction with the increase of NaCl concentration from 10^{-4} to 10^{-3} M and greatly with the increase of NaCl concentration up to 5×10^{-1} M. The obtained curves contains two segments, the first one has a slope of about 20

mV/decade and the second one with slope of about 68 mV/decade. This indicates that there is a relationship between the steady state potential, E_{ss} , and logarithm of sodium chloride concentration in the form:

$$E_{ss} = a - b \log C_{Cl^-} \quad \dots\dots(1)$$

where a and b are constants, their values depends on the concentration of sodium chloride solution.

The curves of Fig.(3) show the potentiodynamic anodic polarization behaviour of the alloy in NaCl solution of different concentrations from 10^{-4} to 5×10^{-1} M at a sweep rate of 1 mV/sec. It is clear from these curves that the current suddenly increases at a definite potential in the passive region of alloy. The value of this potential depends on the molar concentration of sodium chloride, where it shifts to the more negative (less positive) direction with the increase of concentration, C . The sudden increase in current is due to the breakdown of passivity and the initiation of pitting corrosion. For this reason, the potential at which the sudden increase of the current occurs is called the pitting potential, E_{pit} . From the curves of Fig. (3), the values of E_{pit} are deduced for the different NaCl concentrations and listed in Table (2).

Fig.(4) represents the dependence of the pitting potential, E_{pit} , on the logarithm chloride ions concentration for the investigated alloy.

The previous studies carried out on the pitting corrosion in NaCl media reveal that there is a straight line relationship between E_{pit} and $\log C_{Cl^-}$ in the following form ^[8-10]

$$E_{pit} = a_1 - b_1 \log C_{Cl^-} \quad \dots\dots(2)$$

where a_1 and b_1 are constants, which depend on the nature of the metal and type of the aggressive anion. The obtained curve in Fig. (4) has a sigmoidal nature. The first segment represents a slight shift of E_{pit} in a negative direction with a slope mV/decade when the concentration of chloride ions changed from 10^{-4} to 10^{-3} M. While, the second segment represents a great shift of E_{pit} in a negative direction with a slope of 123 mV/decade when the concentration of chloride ions changed from 10^{-3} to 5×10^{-1} M. It can be concluded that the low concentrations of chloride ions are not sufficient to destroy completely the passivating film formed on the electrode surface, or that the pits formed are not completely active and may undergo repassivation ^[10-12]. At higher, concentrations of chloride ions, it can be assumed that these concentration are sufficient to destroy the passivating film formed on the alloy surface, or that the pits formed are active and non passivable.

The effect of inorganic anions, WO_4^{2-} , PO_4^{3-} , NO_2^- , $B_4O_7^{2-}$, CrO_4^{2-} , on the pitting behaviour of the investigated alloy in 0.5 M NaCl has been studied using the potentiodynamic and electrochemical impedance spectroscopy techniques.

Figure (5) shows the potentiodynamic anodic behaviour of the alloy in 0.5 M NaCl in presence of different concentrations of Na₂CrO₄ additive. Similar plots are obtained for the other additives, WO₄²⁻, PO₄³⁻, Na₂⁻, B₄O₇²⁻ and not shown. The different values of pitting potential for the alloy in presence of different concentrations of the inorganic additives are deduced from the plots of Fig.(5) and similar ones and listed in Table (3). The results of this table indicate that the pitting potential, E_{pit}, of the electrode shifts in the noble direction in presence of increasing concentration of the inorganic additive. This reveals that these anions have an inhibiting effect on the pitting corrosion of the alloy in sodium chloride solutions. The dependence of E_{pit}, in 0.5M NaCl, on the concentration of the inhibiting anions is depicted in Fig.(6). Pitting potential, E_{pit}, varies linearly with the logarithm of the molar concentration of the inhibitor according to the following equation:

$$E_{\text{pit}} = a_2 + b_2 \log C_{\text{inh}} \quad \dots\dots(3)$$

where, a₂ and b₂ are constant, their values depend on the type of inhibiting anions. The different values of a₂ and b₂ are deduced from the intercepts and slopes of the straight lines of Fig.(6), respectively. The values of a₂ are: 285, 275, 260, 215, 200 mV for WO₄²⁻, PO₄³⁻, NO₂⁻, B₄O₇²⁻, CrO₄²⁻, respectively. While the values of b₂ are: 200, 171.67, 141.67, 116.67, 83.33 mV/decade for the anions, respectively. The values of constants a₂ and b₂ indicate that the efficiency of pitting corrosion inhibitors decreases according the order: WO₄²⁻ > PO₄³⁻ > NO₂⁻ > B₄O₇²⁻ > CrO₄²⁻.

Figure (7) represents the impedance diagrams (Nyquist plots) for the working electrode immersed in 0.5 M NaCl solution in absence and in presence of increasing molar concentration of Na₂CrO₄. Similar impedance diagrams are obtained in the presence of other inorganic additives and not shown. The impedance diagrams of Fig.(7) and similar ones are not perfect semicircles and this difference has been attributed to the frequency dispersion.^[13,14] The charge transfer resistance (R_t) values are calculated from the difference in impedance at lower and higher frequencies as previously suggested^[15-17]. To obtain the double layer capacitance (c_{dl}), the frequency at which the imaginary component of the impedance is maximum, (-Z_{i,max}), is found and C_{dl} values are obtained from the equation:

$$F(-Z_{i,\text{max}}) = (2\pi C_{\text{dl}} R_t)^{-1} \quad \dots\dots(4)$$

The inhibition efficiency of pitting corrosion of the investigated alloy in 0.5 M NaCl solution in presence of inorganic additives can be calculated from the charge transfer resistance (R_t), using the following equation:

$$I\% = \frac{R_{t\text{corr}}^{-1} - R_{t\text{corr}(\text{inh})}^{-1}}{R_{t\text{corr}}^{-1}} \quad \dots\dots(5)$$

where R_{tcorr} and R_{tcorr(inh)} are the charge transfer resistance values in absence and in presence of inorganic additives, respectively. The impedance parameters derived from

these investigations are listed in Tables (4-8) for the anions WO_4^{2-} , PO_4^{3-} , NO_2^- , $\text{B}_4\text{O}_7^{2-}$, CrO_4^{2-} , respectively. These results indicate that as the concentration of the additive increases the values of R_t and $I\%$ increases, but the values of C_{dl} decreases. This indicates the occurrence of adsorption of the additives on the alloy surface. The inhibition efficiency of the inorganic additives decreases according to the order: $\text{WO}_4^{2-} > \text{PO}_4^{3-} > \text{NO}_2^- > \text{B}_4\text{O}_7^{2-} > \text{CrO}_4^{2-}$. This order is the same as that obtained by potentiodynamic polarization measurements.

The dependence of R_t in 0.5 M NaCl solution, on the concentration of the inhibiting anions is depicted in Fig.(8). R_t varies linearly with the logarithm of the molar inhibitor concentration according to the equation:

$$R_t = a_3 + b_3 \log C_{inh} \quad \dots\dots(6)$$

where a_3 and b_3 are constants found to be depended on the type of the inhibiting anions. The values of a_3 and b_3 are deduced from the plots of Fig. (8). The values of a_3 are found to be: 475, 275, 175, 140, 125 Ohm. cm^2 for the anions: WO_4^{2-} , PO_4^{3-} , NO_2^- , $\text{B}_4\text{O}_7^{2-}$, CrO_4^{2-} , respectively.

The values of b_3 are found to be: 373.33, 300, 208.33, 146.67, 116.67 Ohm. cm^2 /decade for the anions: WO_4^{2-} , PO_4^{3-} , NO_2^- , $\text{B}_4\text{O}_7^{2-}$, CrO_4^{2-} , respectively. This means that the efficiency of the inorganic additives decreases according to the order: $\text{WO}_4^{2-} > \text{PO}_4^{3-} > \text{NO}_2^- > \text{B}_4\text{O}_7^{2-} > \text{CrO}_4^{2-}$. This order is the same as that above mentioned.

Figure (9) shows the plots of C_{dl} versus the logarithm of the concentration of the inorganic additives. These plots indicate that, in presence of less effective anions, there is a sharp decrease in C_{dl} values with the increases of concentration followed by a gradual decrease. While in presence of the more effective anions there is a gradual decrease in the C_{dl} values with the increase of inhibitor concentration.

The change in the R_t and C_{dl} can be explained by the reversible competitive adsorption between the inhibitive anions and aggressive Cl^- ions. If inhibiting anions adsorption predominates on the open surface, the protective passive film formation occurs. However, if adsorption of Cl^- ions predominates, pitting corrosion will occur on the surface.

The inhibiting effect of these anions can be explains by adsorption these anions which reduces the free energy of the system and impedes the passage of the metallic ions from the lattice into the solution. It must, however, kept in mind that the inhibiting properties of these additives depend greatly on the presence of activating ions (e.g. Cl^-) in solution and the ratio between these ions. Also, such behaviour of the electrode is undoubtedly associated with concurrent adsorption. The amount of adsorbed Cl^- decreased when these additives are introduced into the electrolyte. For higher concentrations of inhibiting ions the adsorption of activating ions (Cl^- ions) can be excluded completely certain definite ratios of the concentration of activating and

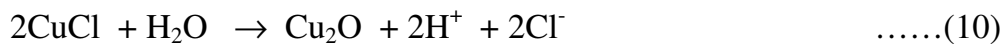
inhibiting ions are necessary for the passivation and protection of the alloy electrode from the effect of chloride ions.

It is clear from the results of tables (1,2) that with increasing NaCl concentration the steady state potential, E_{ss} , and pitting potential shift to the more negative direction (less positive). This shift is significant at higher concentration of NaCl ($C > 10^{-3}$ M). At lower concentrations the polarization curves show anodic reactions which are essentially by the applied, potential. At such concentrations the chloride ions have a little significant effect.

The anodic reactions reveal that:

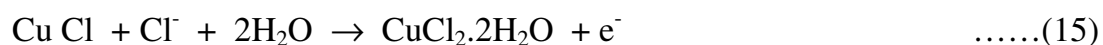


In such conditions, Cu_2O and $\text{Fe}(\text{OH})_2$ are probably formed as^[18]



The small variation in E_{ss} and E_{pit} recorded at 10^{-4} and 10^{-3} M NaCl indicate that healing and formation of the oxide and hydroxide probably take place simultaneously.

As the concentration of NaCl was further increased ($C > 10^{-3}$ M), E_{ss} and E_{pit} greatly shifted to the more negative direction. The alloy tends to lose its passivity in the presence of these high sodium chloride concentration, which may be one or more of the reactions.^[19,20]

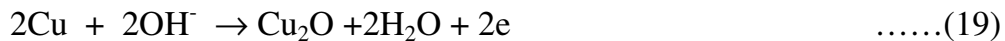
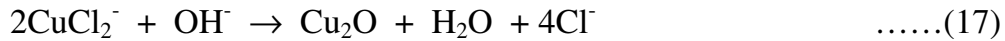


Or the occurrence of the autocatalytic reaction as^[21,22]



this means that the presence of high Cl^- ions concentration not only prevents the formation of the passive region but it acts in the dissolution of the alloy.

In the presence of inhibiting anions the decrease in corrosion rates of the alloy may be due to the formation of cuprous oxide, Cu_2O , which was further oxidized to a less protective hydroxy chloride (paratanamite) according to the following equations [1,22-24]



Compounds such as Na_2PO_4 , $\text{Na}_2\text{B}_4\text{O}_7$ which are non oxidizing agents and Na_2WO_4 and Na_2CrO_4 of type MO_4^{2-} can protect metals against corrosion. Upon hydrolysis of these compounds, hydroxyl ions appear which are capable of forming a passivating oxide as $\text{Fe} + \text{OH}^- \rightarrow \text{FeO} + \text{H}_2\text{O}$ with out external polarization^[25]. As Cu_2O and $\text{Cu}_2(\text{OH})_2\text{Cl}$ may be formed by the above mentioned reactions (17-19).

It should be mentioned that, the effect of the inhibiting anion may be also due to the specific action of the anions themselves. For example in the presence of Na_2PO_4 the protective film may consist of $\gamma\text{-Fe}_2\text{O}_3$ with $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ imbedded in it. Also, in the presence of CrO_4^{2-} , the protective film formed on the surface of the alloy may involve Cr_2O_3 and $\text{Cr}(\text{OH})_3$ ^[26]

CONCLUSIONS

- 1- The Cu-Fe alloy undergoes pitting corrosion in high sodium chloride solutions.
- 2- The pitting potential, E_{pit} shifts to more negative values with the increase of the molar concentration of NaCl.
- 3- The addition of inhibiting anions shifts the corrosion potential, E_{pit} to the more positive direction.
- 4- The values of R_t increases with the increase of inhibiting anions concentration, but C_{dl} decreases.
- 5- The inhibition efficiency of the inhibiting anions decreases according to the order: $\text{WO}_4^{2-} > \text{PO}_4^{3-} > \text{NO}_2^- > \text{B}_4\text{O}_7^{2-} > \text{CrO}_4^{2-}$.

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Table (1) Data of open circuit measurements for the alloy in different concentrations of NaCl

Concentration (M)	Steady- state E_{ss} / mV	Time of steady state, T_s / min
1×10^{-4}	-205	35
1×10^{-3}	-225	45
1×10^{-2}	-300	60
1×10^{-1}	-350	70
5×10^{-1}	-385	85

Table (2) Data of potentiodynamic anodic polarization measurements for the alloy in different concentrations of NaCl

Concentration (M)	Pitting corrosion E_{pit} (mV)
1×10^{-4}	+550
1×10^{-3}	+525
1×10^{-2}	+350
1×10^{-1}	+280
5×10^{-1}	+200

Table (3) Data of potentiodynamic anodic polarization measurements for the alloy in 0.5 M NaCl in presence of different concentrations of inorganic additives

Concentration (M)	E_{pit} (mV)				
	WO_4^{-2}	PO_4^{-3}	NO_2^-	$B_2O_7^{-2}$	CrO_4^{-2}
0.00	200	200	200	200	200
1×10^{-4}	330	315	300	250	225
1×10^{-3}	500	475	450	370	300
1×10^{-2}	680	650	580	475	400
1×10^{-1}	870	815	725	600	475
5×10^{-1}	925	875	775	685	530

Table (4) Data of impedance measurements for the alloy in 0.5M NaCl in absence and presence of different concentration of WO_4^{2-}

Concentration (M)	R_t $\Omega \cdot \text{cm}^2$	C_{dl}	I%
0.00	50	1273.88	-
1×10^{-4}	550	13.16	90.9
1×10^{-3}	850	8.13	94.12
1×10^{-2}	1200	2.76	95.83
1×10^{-1}	1550	1.66	96.77
5×10^{-1}	1670	1.43	97.00

Table (5) Data of impedance measurements for the alloy in 0.5M NaCl in absence and presence of different concentration of PO_4^{3-}

Concentration (M)	R_t $\Omega \cdot \text{cm}^2$	C_{dl}	I%
0.00	50	1273.88	-
1×10^{-4}	350	32.50	85.71
1×10^{-3}	640	9.72	92.18
1×10^{-2}	900	4.92	94.44
1×10^{-1}	1150	3.01	95.65
5×10^{-1}	1250	2.55	96.00

Table (6) Data of impedance measurements for the alloy in 0.5M NaCl in absence and presence of different concentration of NO_2^-

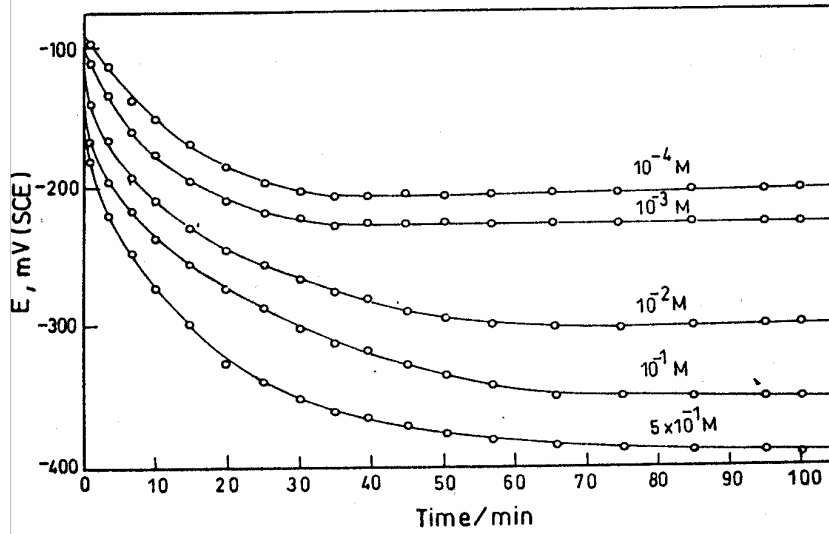
Concentration (M)	R_t $\Omega \cdot \text{cm}^2$	C_{dl}	I%
0.00	50	1273.88	-
1×10^{-4}	225	78.63	77.78
1×10^{-3}	400	24.88	87.5
1×10^{-2}	600	11.06	91.67
1×10^{-1}	800	6.22	93.75
5×10^{-1}	850	5.51	94.12

Table (7) Data of impedance measurements for the alloy in 0.5M NaCl in absence and presence of different concentration of $B_4O_7^{-2}$

Concentration (M)	R_t $\Omega \cdot cm^2$	C_{dl}	I%
0.00	50	1273.88	-
1×10^{-4}	175	130	71.43
1×10^{-3}	300	44.23	83.33
1×10^{-2}	450	19.66	88.89
1×10^{-1}	580	11.83	91.38
5×10^{-1}	615	10.53	91.87

Table (8) Data of impedance measurements for the alloy in 0.5M NaCl in absence and presence of different concentration of CrO_4^{-2}

Concentration (M)	R_t $\Omega \cdot cm^2$	C_{dl}	I%
0.00	50	1273.88	-
1×10^{-4}	150	212.32	66.67
1×10^{-3}	250	63.69	80.00
1×10^{-2}	350	30.33	85.71
1×10^{-1}	470	17.31	89.36
5×10^{-1}	500	14.15	90.00



Fig(1): Potential- time curves of the alloy in different concentrations of NaCl, under open circuit conditions.

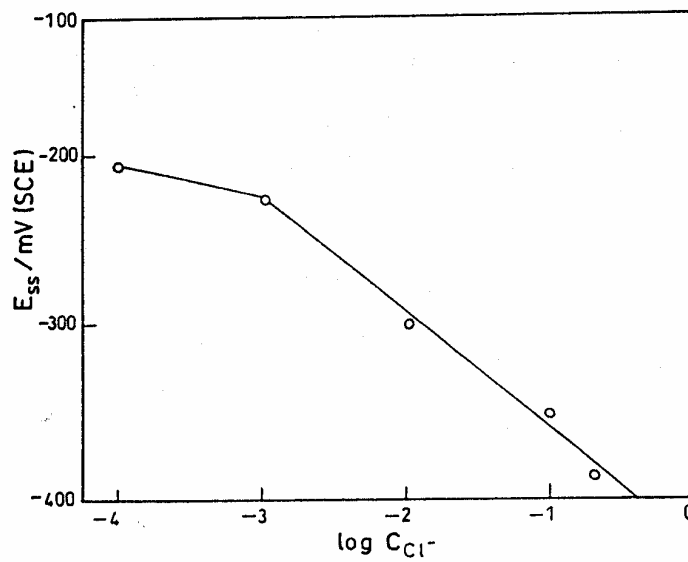


Fig.(2): Variation of the steady state potential of the alloy with the logarithm of NaCl concentration.

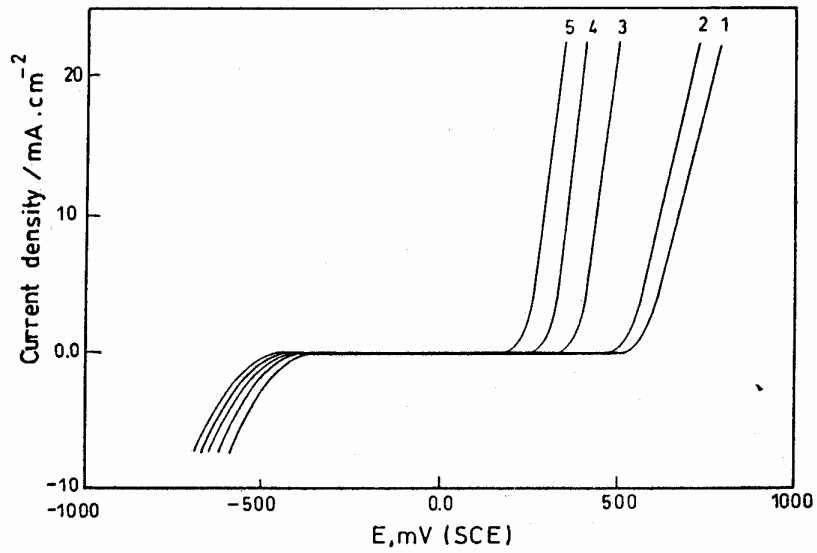


Fig.(3): Potentiodynamic anodic polarization curves of the alloy in NaCl solutions of different concentrations: 1- 1×10^{-4} , 2- 1×10^{-3} , 3- 1×10^{-2} , 4- 1×10^{-1} , 5- 5×10^{-1} M NaCl.

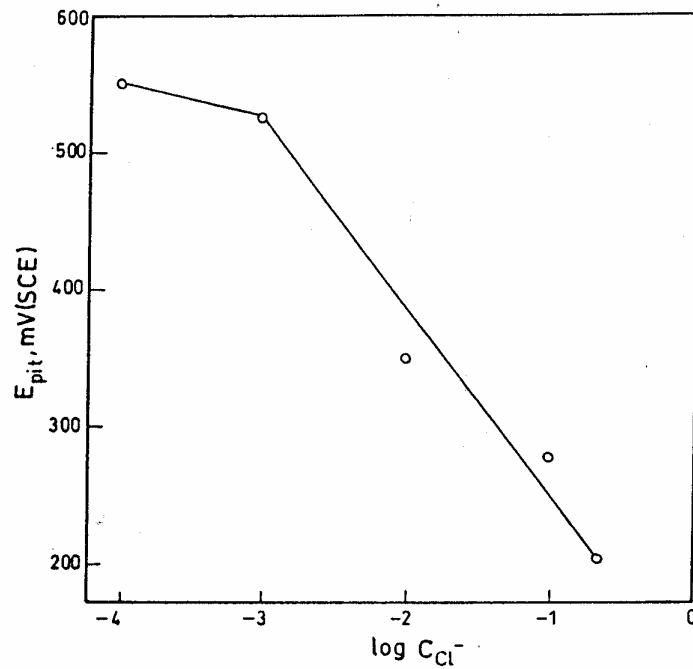


Fig.(4): Variation of the pitting potential, E_{pit} , with the logarithm of sodium chloride concentration.

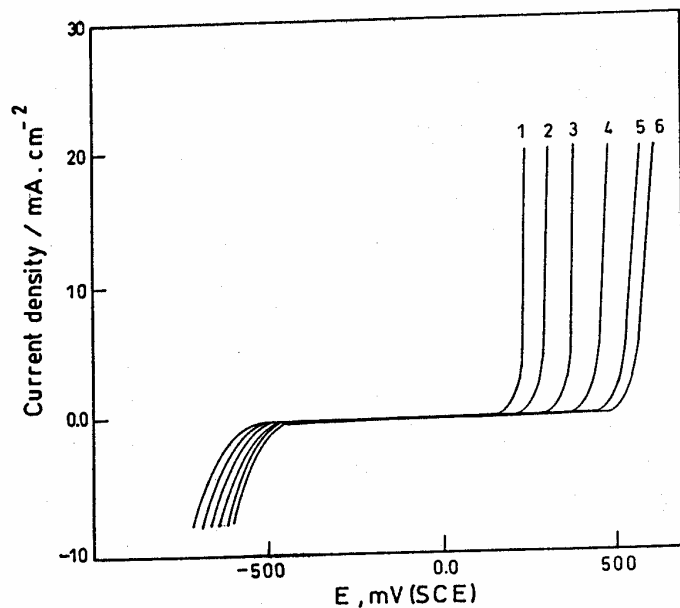


Fig.(5): Potentiodynamic anodic polarization curves of the alloy in 0.5 M NaCl containing different concentrations of Na_2CrO_4 , 1-blank, 2- 1×10^{-4} , 3- 1×10^{-3} , 4- 1×10^{-2} , 5- 1×10^{-1} , 6- 5×10^{-1} M Na_2CrO_4 .

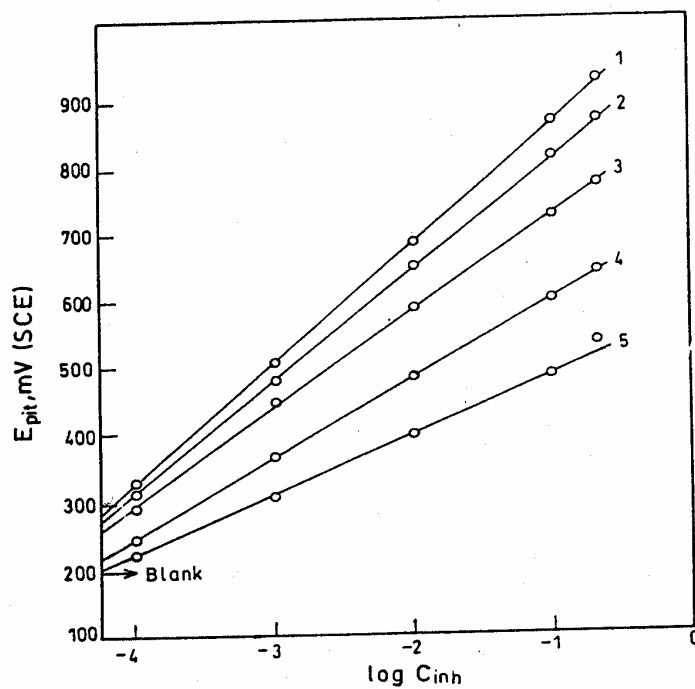


Fig.(6): Variation of the pitting potential, E_{pit} , with the logarithm of inhibitors concentrations 1- WO_4^{2-} , 2- PO_4^{3-} , 3- NO_2^- , 4- $\text{B}_4\text{O}_7^{2-}$, 5- CrO_4^{2-} .

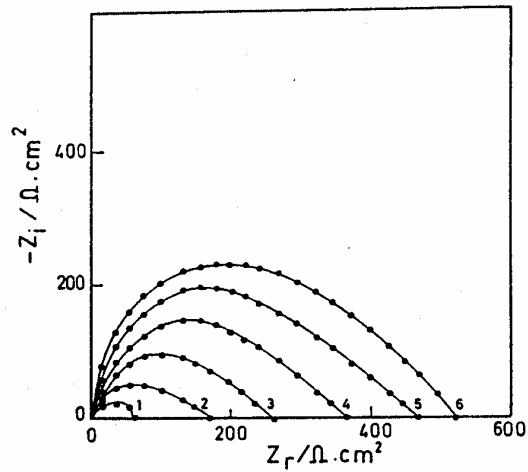


Fig.(7): Impedance diagrams (Nyquist plots) for the alloy in 0.5 NaCl solution containing molar concentration of Na_2CrO_4 . 1-blank 2- 1×10^{-4} , 3- 1×10^{-3} , 4- 1×10^{-2} , 5- 1×10^{-1} , 6- 5×10^{-1} M.

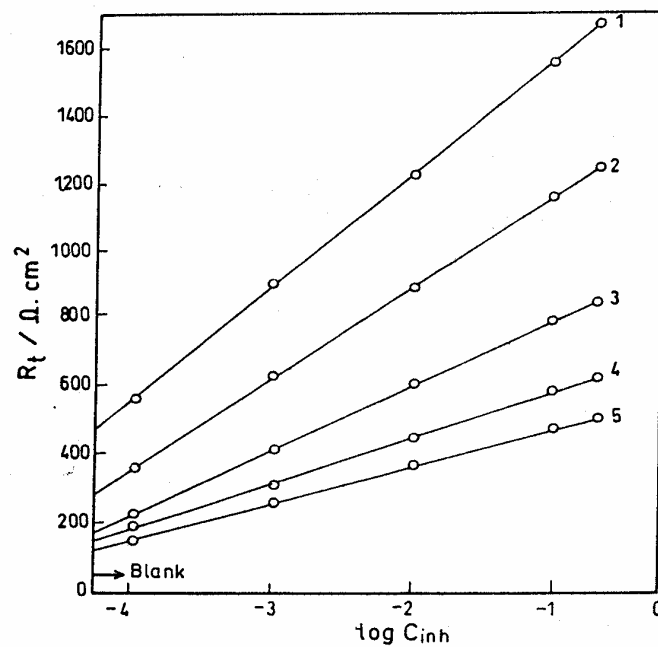


Fig.(8): Variation of R_t of the alloy with the logarithm of molar concentrations of inhibitors: 1- WO_4^{2-} , 2- PO_4^{3-} , 3- NO_2^- , 4- $\text{B}_4\text{O}_7^{2-}$, 5- CrO_4^{2-} .

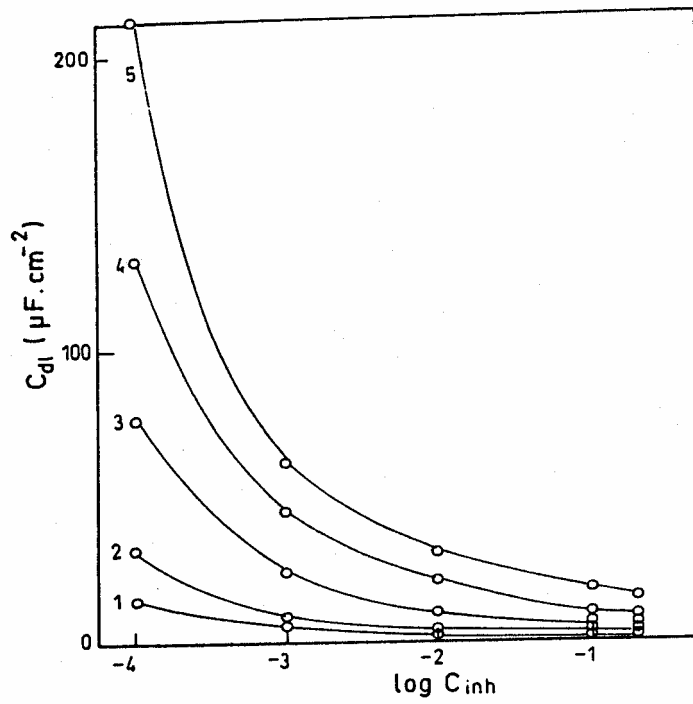


Fig.(9): Variation of C_{dl} with logarithm of molar concentrations of inhibitors. 1- WO_4^{2-} , 2- PO_4^{3-} , 3- NO_2^- , 4- $\text{B}_4\text{O}_7^{2-}$, 5- CrO_4^{2-} .