

## **Corrosion Inhibition of Carbon Steel in 1M H<sub>2</sub>SO<sub>4</sub> by Cationic and Non Anionic Surfactants**

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### **Abstract**

The corrosion inhibition of carbon steel by two organic surfactants Di decyl benzyl tri ethyl ammonium chloride (C) and Nonyl phenol ethoxylate with about twelve ethylene oxide units (N) is investigated in 1M H<sub>2</sub>SO<sub>4</sub> using weight loss measurements and galvanostatic polarization curves. The corrosion rate decreases with the inhibitor concentration from 10 to 100 ppm and from 10 to 125 ppm of (C) and (N) respectively. It is found that the corrosion rate decreases with immersion time from 5 to 20 days and then tend to be constant. The results have shown that the lowest corrosion rate is for 75 ppm and 100 ppm for (C) and (N) respectively. The effect of temperature on corrosion inhibition has been studied and activation energies for optimum conditions of each inhibitor have been evaluated. The corrosion inhibition of carbon steel in 1M H<sub>2</sub>SO<sub>4</sub> containing (C) or (N) has been attributed to adsorption of inhibitors over the carbon steel surface. Results are correlated to the chemical structure of each inhibitor; the inhibition efficiency of (C) is higher than that of (N). Some electrochemical parameters such as  $I_{\text{corr}}$ ,  $E_{\text{corr}}$ ,  $b_a$  and  $b_c$  are evaluated from galvanostatic polarization technique, the results of inhibition efficiency evaluated from galvanostatic polarization technique are in a good agreement with those obtained from weight loss technique. Polarization curves studies showed that (C) or (N) are mixed type inhibitors in 1M H<sub>2</sub>SO<sub>4</sub> solution. The inhibition of (C) or (N) is due to their adsorbance on the metal surface. Furthermore, Scanning Electron Microscopy (SEM) is used to examine the surface morphology of the carbon steel samples both in absence and presence of each inhibitor at optimum conditions.

### **Key words:**

Corrosion inhibition, Carbon steel, Organic surfactants, Weight loss, Galvanostatic polarization, Scanning electron microscopy.

### **Introduction:**

Acids are widely used in various technological processes in industry, e.g., in pickling baths, in the extraction and processing oil and gas and in other chemical and petrochemical industries. Also, in the technical cracking of petroleum, acids appear as a result of hydrolysis of salts and may have destructive effect on the equipment. Corrosion due to acids are important and expensive problem in the petroleum refining units and it represents a significant portion of loss as a result of lost production, inefficient operation, high maintenance and the cost of corrosion control chemicals. Inhibitors are compounds that control, reduce, or prevent reactions between a metal and its surroundings when added to the medium in small quantities. Inhibitors should be effective in low concentrations for economy. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acidic media <sup>(1)</sup>.

Most of the well-known acid inhibitors are organic compounds containing nitrogen, sulphur and oxygen atoms. The influence of organic compounds containing nitrogen, such as amines and heterocyclic compounds, on the corrosion of steel in acidic solutions has been investigated by several workers <sup>(2-5)</sup>.

In previous studies, ethoxylated fatty acid <sup>(6)</sup>, ethoxylated fatty amines <sup>(7)</sup>, and propen-ethoxylated diol <sup>(8)</sup> had been used as corrosion inhibitors for steel and aluminum in acidic solutions. The existing data show that the most organic inhibitors act by adsorption on the metal surface. This phenomenon is influenced by the nature and the surface charge of metal, by the type of aggressive electrolyte and by the chemical structure of inhibitors <sup>(9)</sup>.

The inhibition efficiency (E %) depends on the parameters of the system (temperature, pH, duration, and metal composition) and on the structure of the inhibitor molecule. It is well known that heterocyclic compounds containing nitrogen atoms are good corrosion inhibitors for many metals and alloys in various aggressive media <sup>(1,2,5,9,10-16)</sup>.

In the present investigation the corrosion inhibition of carbon steel in 1M H<sub>2</sub>SO<sub>4</sub> solutions in absence and in presence of a new organic compound (Di decyl benzyl tri ethyl ammonium chloride {cationic surfactant} (C) and non anionic surfactant Nonyl phenol ethoxylate (N) have been studied using weight loss and galvanostatic polarization techniques. The effect of temperature and time of immersion on corrosion inhibition have been studied. Activation energies for the carbon steel in the acid solution and in the acid containing the best concentration of each inhibitor have been evaluated. The inhibitive effect upon using the best concentration of each inhibitor in 1M H<sub>2</sub>SO<sub>4</sub> is also examined using scanning electron microscopy.

## **Experimental:**

### **1- Chemical Composition of Carbon Steel Alloy:**

Rectangular samples of size 1x1 cm were cut from a sheet of carbon steel alloy for Weight Loss Measurements, circular sheets of 1cm<sup>2</sup> area for Scanning Electron Microscopy, and a rectangular samples of size 0.5x2 cm and 1cm<sup>2</sup> area exposed to the tested solutions for Galvanostatic Polarization Technique. The chemical composition of the carbon steel alloy is listed in Table 1.

Table 1: Chemical Composition of Carbon Steel Alloy.

| Element             | C    | Mn   | P     | Si   | S     | Fe    |
|---------------------|------|------|-------|------|-------|-------|
| Analysis (weight %) | 0.23 | 0.44 | 0.022 | 0.24 | 0.006 | 99.06 |

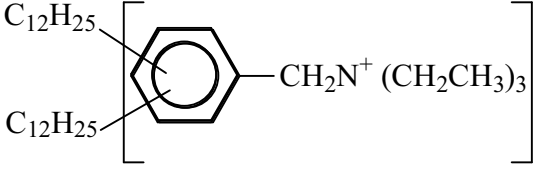
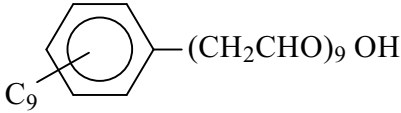
### **2- Materials and Solutions:**

#### **2-1. Inhibitors:**

Organic inhibitors (non anionic surfactant and new cationic surfactant) supplied by Omar and prepared as described previously <sup>(17)</sup> and listed in Table 2. They are added in different concentrations from 10 ppm to 100 ppm of cationic surfactant and

concentrations from 10 ppm to 125 ppm of non-anionic surfactant added each to 1 M H<sub>2</sub>SO<sub>4</sub> acid solution.

**Table 2: Structure and Name of the Inhibitors.**

| Name   | Structure  | Type of surfactant     |
|--|--|------------------------|
| <b>Di decyl benzyl tri ethyl ammonium chloride</b> |  | <b>Cationic (C)</b>    |
| <b>Nonyl phenol ethoxylate</b>                     |  | <b>Non anionic (N)</b> |

## **2-2. Solutions:**

All solutions are prepared from 96 % sulphuric acid solution with distilled water.

## **3- Procedures used for Corrosion Measurements:**

### **3-1. Weight Loss Technique:**

The specimens are polished with different grades of emery papers (300 – 800), cleaned with distilled water, and then dried with absolute ethyl alcohol.

### **3-2. Galvanostatic Polarization Technique:**

Polarization experiments are carried out in a conventional three-electrode glass cell with a capacity of 500 ml with a platinum counter electrode and a saturated calomel electrode (SCE) as reference with a fine Luggin capillary bridge to avoid ohmic polarization. All tests were performed in deaerated solutions under continuously stirred conditions. The procedure adopted for the polarization measurements was the same as described elsewhere <sup>(18)</sup>. The electrochemical studies for specimens in 1M H<sub>2</sub>SO<sub>4</sub> is compared to that in 1M H<sub>2</sub>SO<sub>4</sub> containing 75 ppm of cationic surfactant and to specimens in 1M H<sub>2</sub>SO<sub>4</sub> containing 100 ppm of non anionic surfactant at 40°C. A platinum electrode is used as an auxiliary electrode and all potential values are measured against a saturated calomel electrode (SCE).

### **3-3. Scanning Electron Microscopy:**

A JEOL (5400) Scanning Electron Microscope (Japan) is utilized to document the surface morphology of various specimens of carbon steel. All micrographs of corroded specimens are taken at a magnification of X=3500.

## **Results and Discussion:**

### **Weight Loss Technique:**

The weight loss of carbon steel in 1M sulphuric acid with the addition of various concentrations of cationic (C) and non- anionic (N) surfactants as chemical inhibitors is measured. Figure 1 and 2 show the effect of adding various concentrations [10 ppm - 100 ppm] of (C) and [10 ppm - 125 ppm] of (N) on the corrosion of carbon-steel in 1 M H<sub>2</sub>SO<sub>4</sub> solution at 30°C. From these figures we notice that the corrosion rate decreases with increasing immersion time, from 5 to 20 days then it give a constant value. The values of surface coverage ( $\theta$ ) and the percentage inhibition efficiencies % (P) increase markedly with increase of inhibitor concentration, indicating that a higher coverage of inhibitor on the surface is obtained in 1M H<sub>2</sub>SO<sub>4</sub> solution with higher concentrations of inhibitor (C) or (N). The corrosion rate decreases more markedly upon addition of (C) than (N). This behaviour is due to the dependence of inhibition efficiency on the hydrocarbon chain length where each molecule of (C) contains two-dodecyl chain (C<sub>12</sub>). The surface coverage ( $\theta$ ) is calculated using the following equation:

$$\frac{\theta}{1-\theta} = K C \quad (1)$$

where  $\theta$  is the degree of coverage, K is the equilibrium constant of the adsorption process and C is the inhibitor concentration in the bulk of solution. The percentage inhibition efficiency, (P), of (C) and (N) is calculated by applying the following equation <sup>(19)</sup>:

$$P = \frac{R'_0 - R'}{R'_0} \quad (2)$$

where  $R'_0$  and  $R'$  are, respectively, the corrosion rates in the absence and presence of a given inhibitor. The measured surface coverage ( $\theta$ ) and the percentage inhibition efficiency % (P) are represented in Table 3.

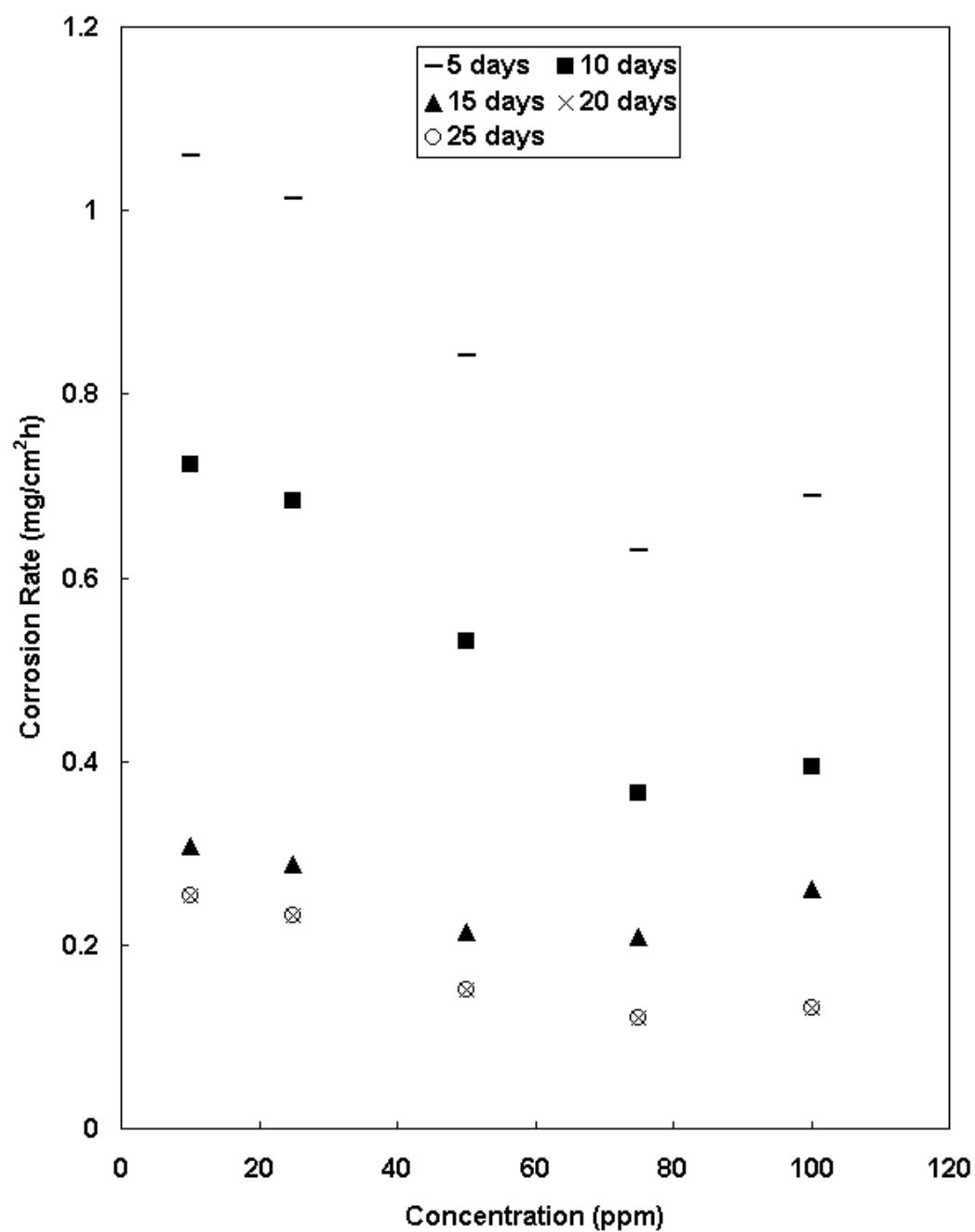


Figure 1: Corrosion Rate of carbon steel in 1M  $H_2SO_4$  solution in different concentrations of (C) at different time intervals.

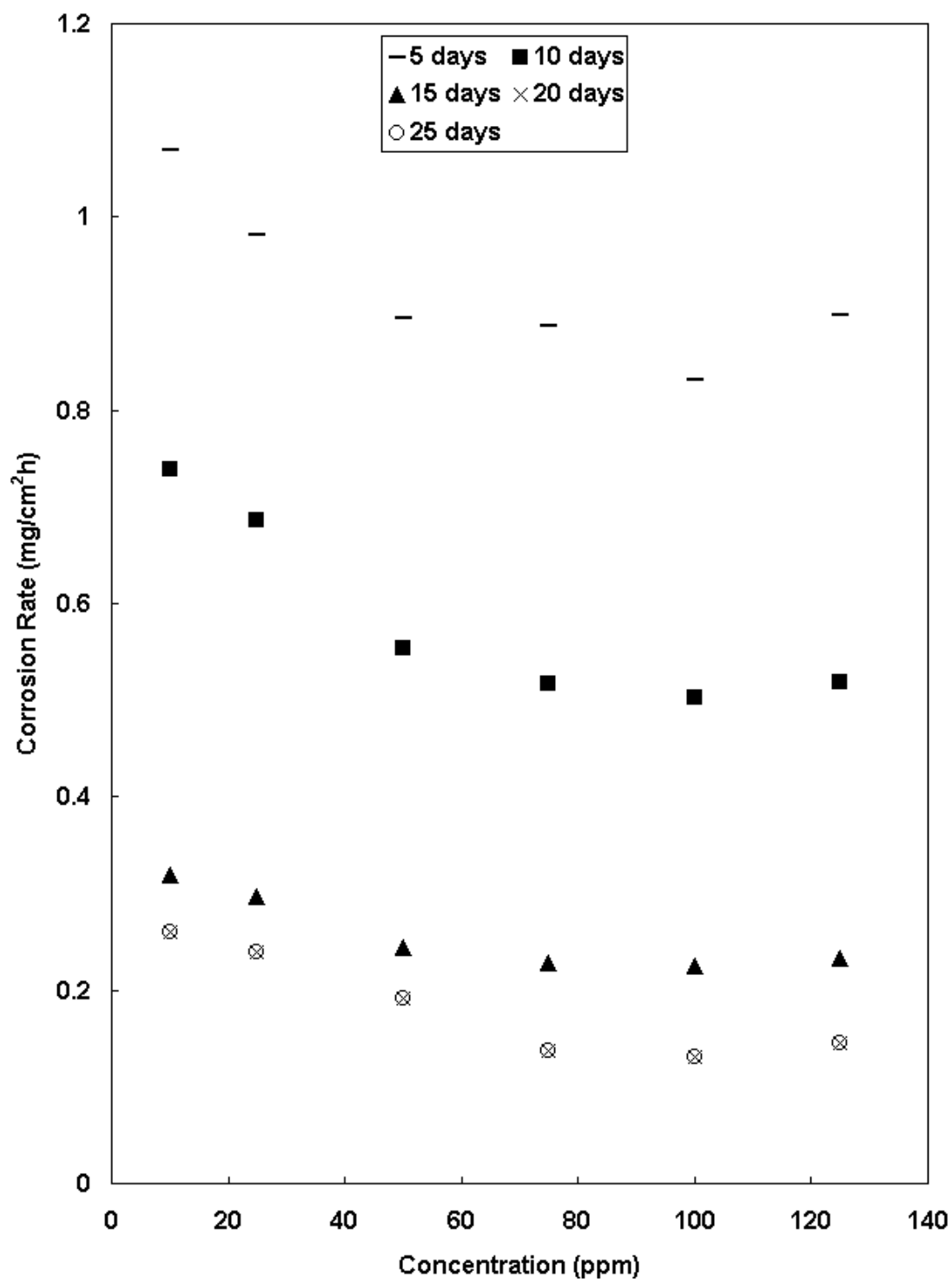


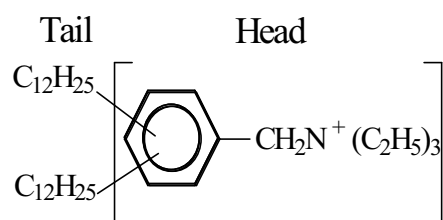
Figure 2: Corrosion Rate of carbon steel in 1M  $H_2SO_4$  solution in different concentrations of (N) at different time intervals.

**Table 3: Inhibition efficiencies of carbon steel: a) in different concentrations of (C) and b) (N) in 1M H<sub>2</sub>SO<sub>4</sub> solution at different times of immersion.**

| Time of Immersion (days) | Surface Coverage (θ) and Corrosion Inhibition Efficiency P (%) |      |       |      |       |      |       |      |   |      |       |      |       |      |       |      |       |      |
|--------------------------|--|------|-------|------|-------|------|-------|------|---|------|-------|------|-------|------|-------|------|-------|------|
|                          | Cationic Inhibitor Concentration (ppm)                         |      |       |      |       |      |       |      | Non-anionic Inhibitor Concentration (ppm) |      |       |      |       |      |       |      |       |      |
|                          | 10   |      | 25    |      | 50    |      | 75    |      | 10  |      | 25    |      | 50    |      | 75    |      | 100   |      |
|                          | θ  | P    | θ     | P    | θ     | P    | θ     | P    | θ   | P    | θ     | P    | θ     | P    | θ     | P    | θ     | P    |
| 5                        | 0.103  | 10.3 | 0.174 | 17.4 | 0.287 | 28.7 | 0.467 | 46.7 | 0.095                                     | 9.5  | 0.169 | 16.9 | 0.24  | 24   | 0.249 | 24.9 | 0.296 | 29.6 |
| 10                       | 0.126  | 12.6 | 0.172 | 17.2 | 0.359 | 35.9 | 0.559 | 55.9 | 0.106                                     | 10.6 | 0.17  | 17   | 0.331 | 33.1 | 0.375 | 37.5 | 0.393 | 39.3 |
| 15                       | 0.367  | 36.7 | 0.409 | 40.9 | 0.559 | 55.9 | 0.573 | 57.3 | 0.345                                     | 34.5 | 0.389 | 38.9 | 0.5   | 50   | 0.532 | 53.2 | 0.537 | 53.7 |
| 20                       | 0.509  | 50.9 | 0.551 | 55.1 | 0.706 | 70.6 | 0.767 | 76.7 | 0.496                                     | 49.6 | 0.536 | 53.6 | 0.629 | 62.9 | 0.735 | 73.5 | 0.747 | 74.7 |
| 25                       | 0.509  | 50.9 | 0.551 | 55.1 | 0.706 | 70.6 | 0.767 | 76.7 | 0.496                                     | 49.6 | 0.536 | 53.6 | 0.63  | 63   | 0.735 | 73.5 | 0.747 | 74.7 |

It can be suggested that, cationic or non-anionic inhibitors contact between the metal and the solution, it exists at the interface and attach or adsorb to the metal surface. The adsorption takes place by head group and tail group (hydrocarbon chain) of each molecule forming barrier film. The optimum concentration are 75 and 100 ppm for (C) and (N) respectively. These values represent the critical micelle concentration (CMC) of (C) and (N) which are 105 ppm and 80 ppm for (C) and (N) respectively.

The effect of temperature on the inhibition efficiency is determined for 1M H<sub>2</sub>SO<sub>4</sub> containing 75 ppm or 100 ppm of (C) and (N) respectively. Figure 3 and 4 show that the corrosion rate decreases slightly at 40°C, and then increases with increasing temperature. These results can be discussed according to mobility of molecule or Kraft point of these inhibitors (Kraft points are 27 and 3 for (C) and (N) respectively). Further increase of temperature the degree of adsorption of each molecule on metal surface increases since the head group of (C)



can be adsorbed through dipole - induced dipole interaction with the steel surface as a result of  $\pi$ -electron polarization or by positive charge of nitrogen atom. On the other hand, inhibitor (N) adsorbed by ethylene oxide group (OCH<sub>2</sub>CH<sub>2</sub>)<sub>12</sub>. Increasing the temperature decreases the adsorption as a result the corrosion rate increases. As the temperature increases the polarization decreases and consequently enhance the desorption process. It can be concluded that the optimum temperature is 40°C above, which the value of Kraft point. The explanation of these results may be suggested that these inhibitors are physico - chemical adsorbed on the surface of the steel. Data in Figure 5 and 6 reveal that the corrosion rate decreases with time of immersion up to 20 days then tend to be constant, this is due to the formation of stable oxide layer (Fe<sub>2</sub>O<sub>3</sub>) according to the following equation <sup>(20)</sup>:





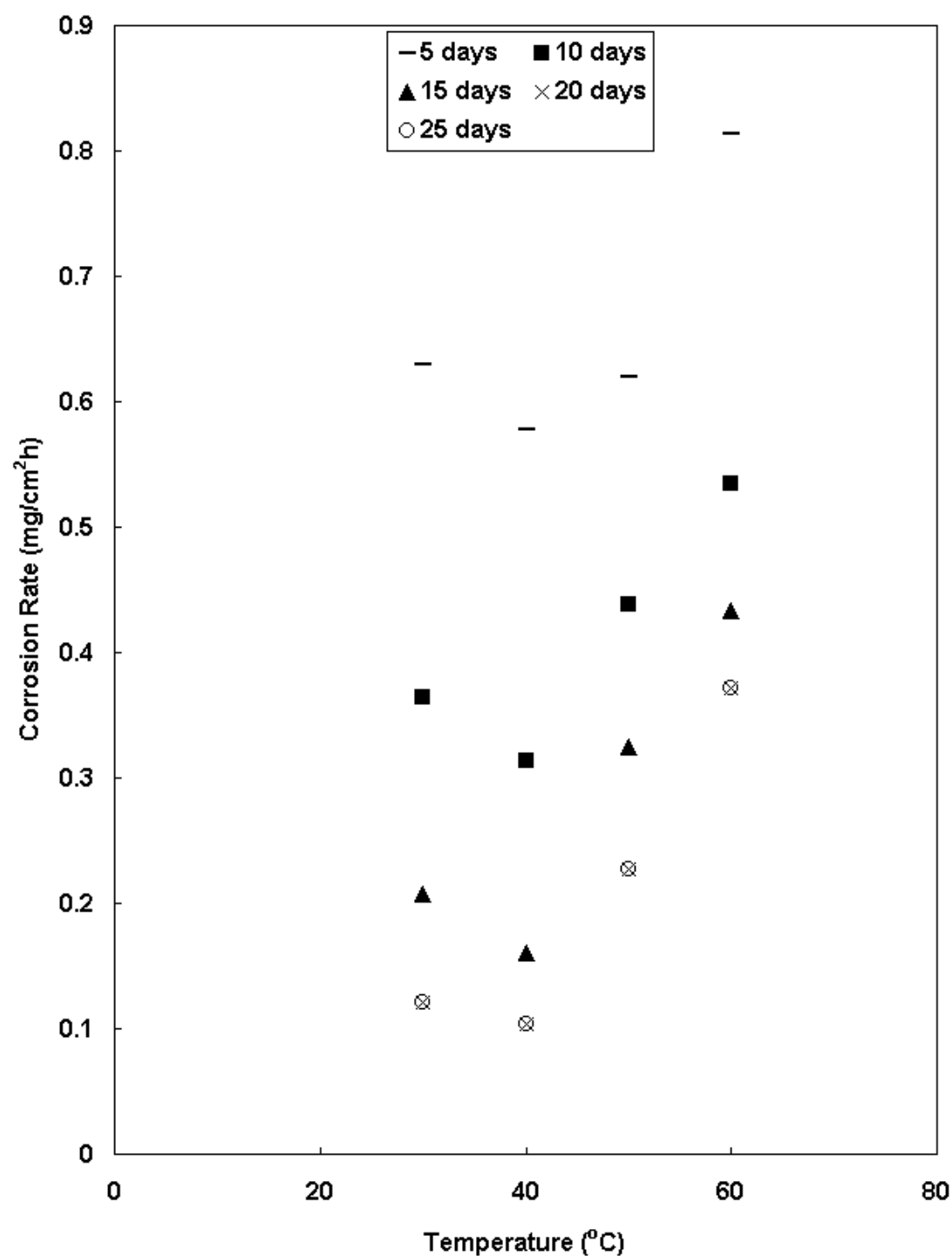


Figure 3: Effect of Temperature on the Corrosion rate of Carbon Steel in 1M  $H_2SO_4$  containing 75 ppm of (C) for different time of immersion.

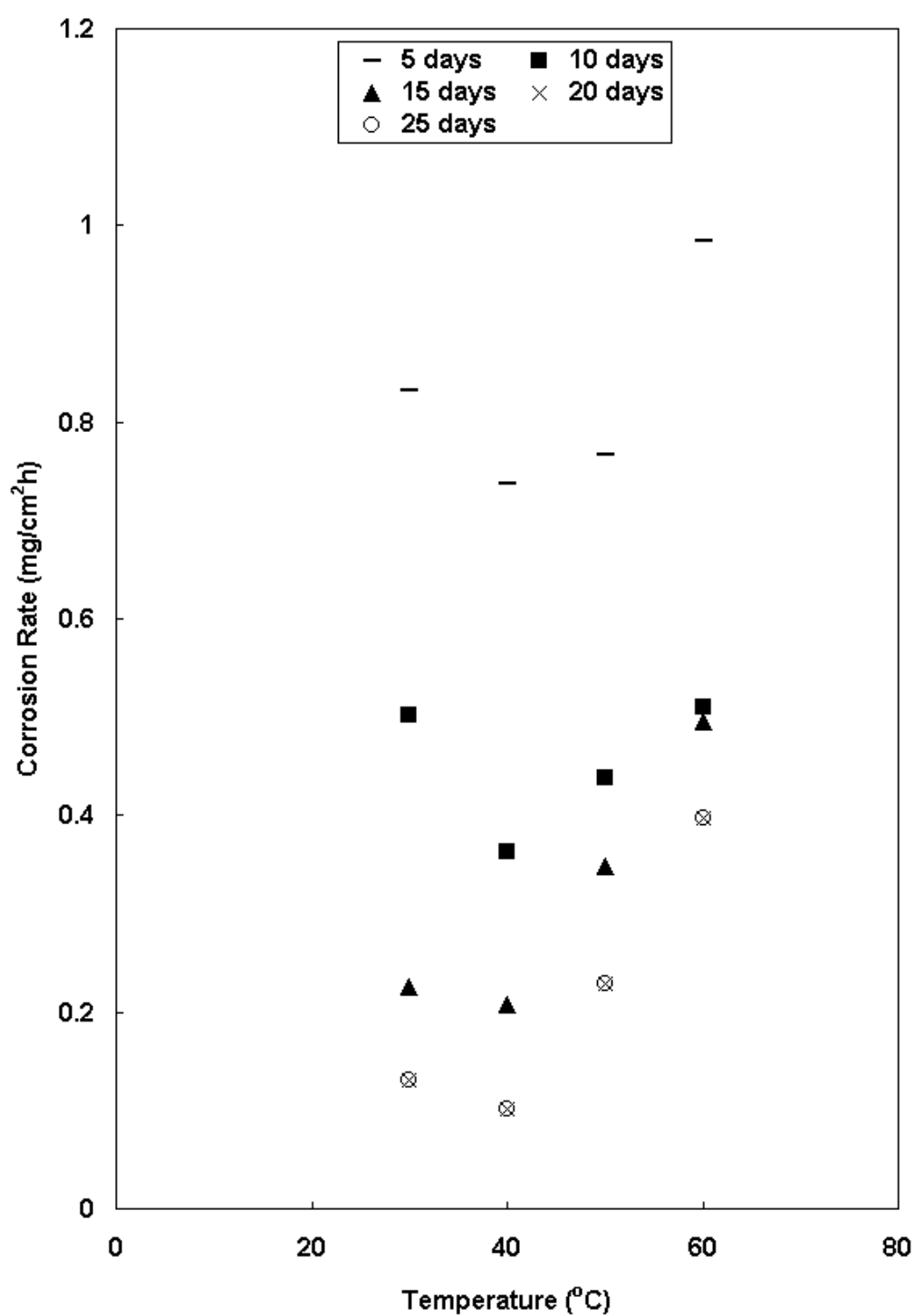


Figure 4: Effect of Temperature on the Corrosion rate of Carbon Steel in 1M  $H_2SO_4$  containing 100 ppm of (N) for different time of immersion.

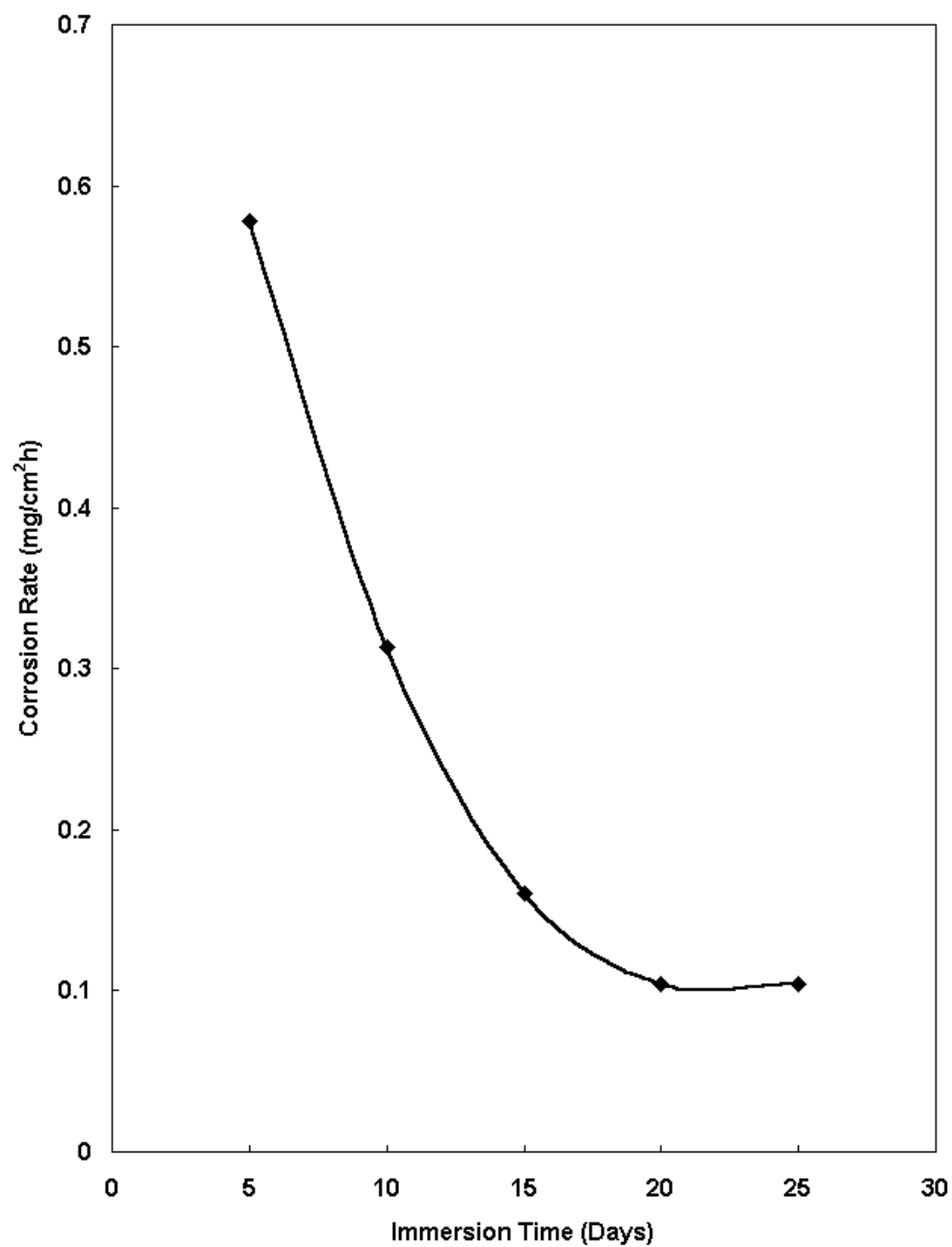


Figure 5: Effect of immersion time on the corrosion rate of carbon steel in 1M  $\text{H}_2\text{SO}_4$  solution containing 75 ppm of (C).

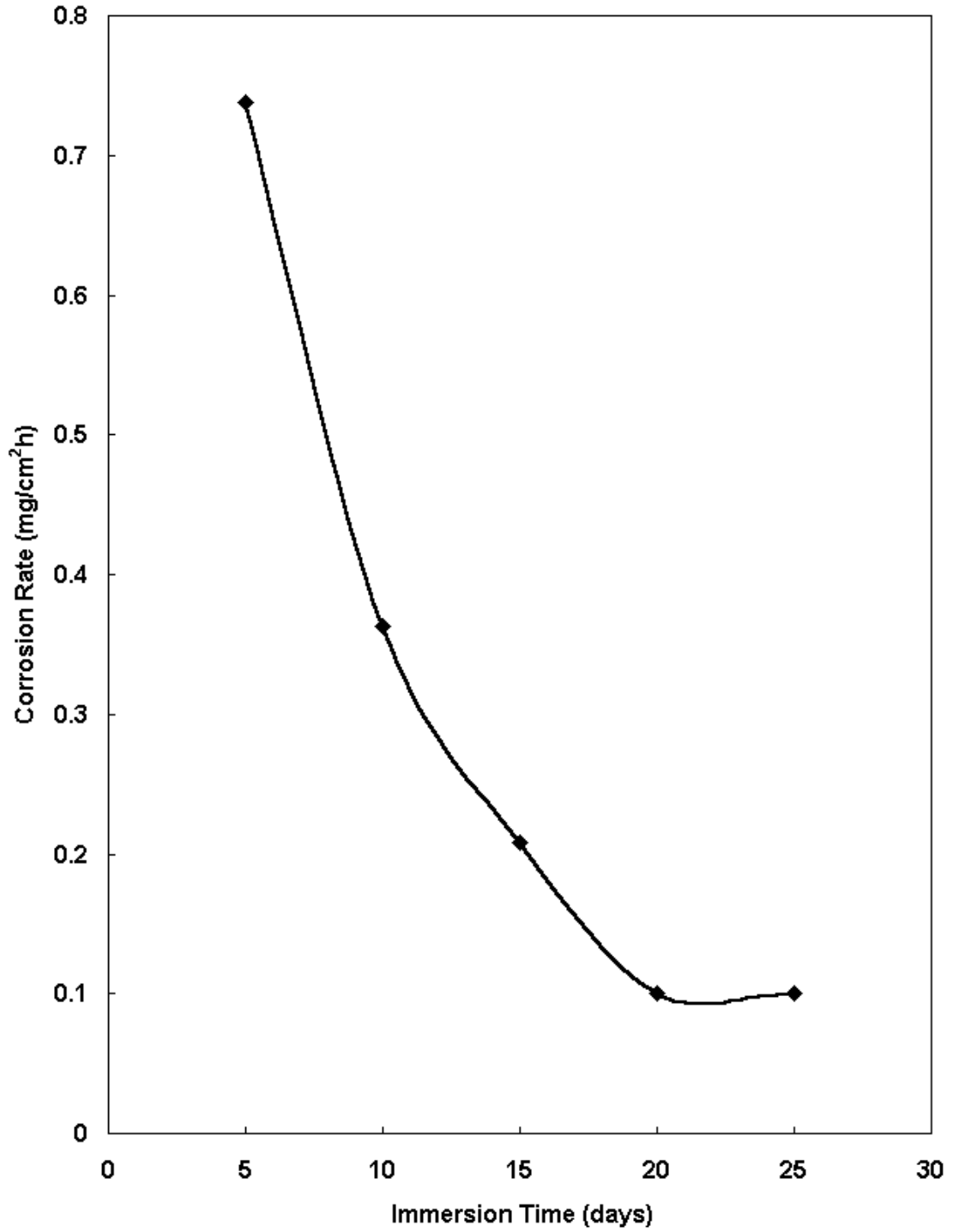
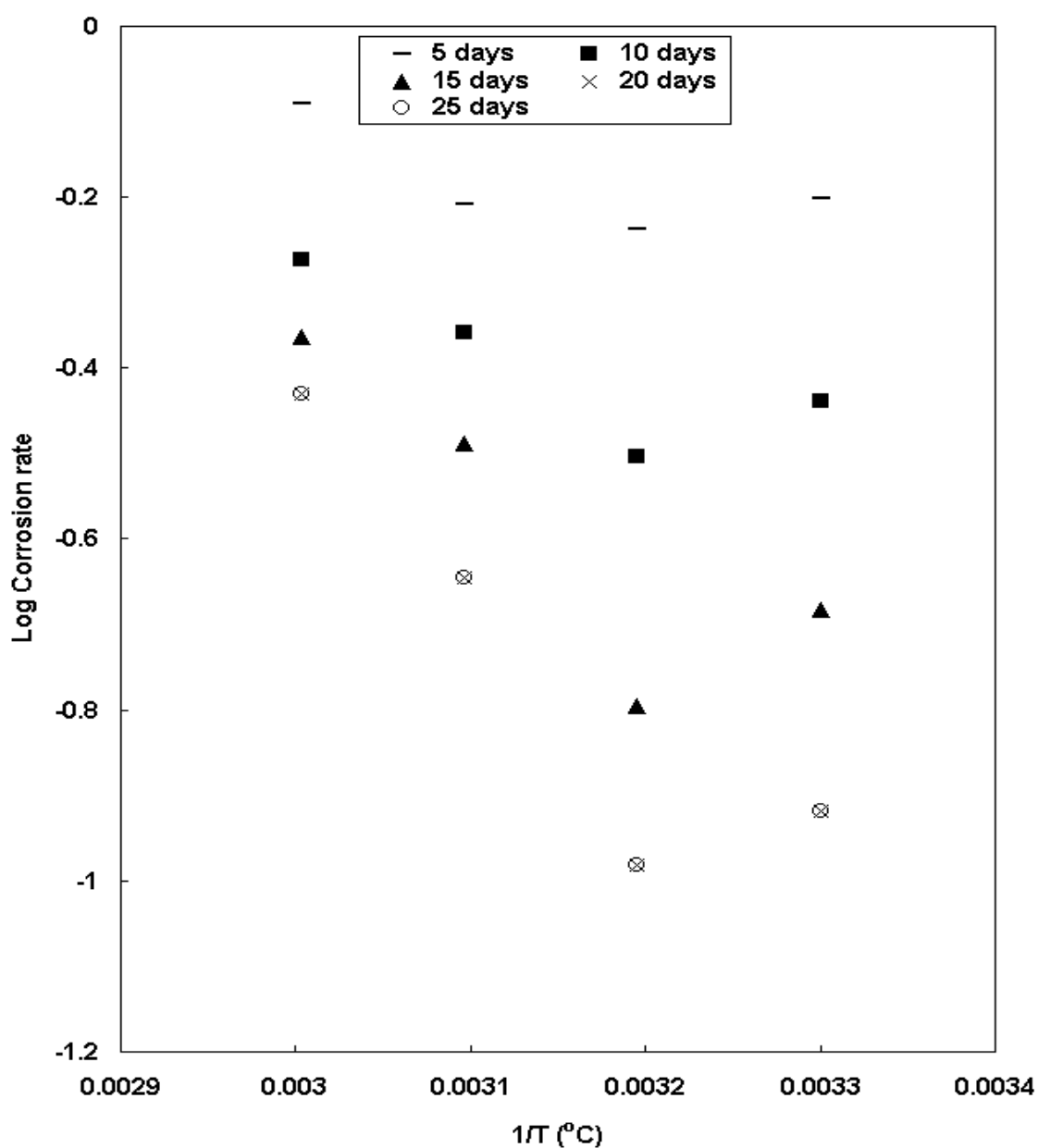


Figure 6: Effect of immersion time on the corrosion rate of carbon steel in 1M  $\text{H}_2\text{SO}_4$  solution containing 100 ppm of (N).

The activation energies of the metal dissolution reaction ( $E_a$ ) for solution of 1M  $\text{H}_2\text{SO}_4$  containing various concentrations of each inhibitor can be determined from the Arrhenius relation <sup>(21)</sup>:

$$k = A \exp\left(\frac{-E_a}{RT}\right) \quad (3)$$

that is, by determination of the slopes of the straight lines that depict the linear dependence of the logarithm of the corrosion rate,  $\ln(k)$ , on the reciprocal value of the absolute temperature. The activation energies decreases up to 20 days and then tend to be in a stable value as shown in Figure 7 and 8. The activation energies of metal dissolution reaction in presence of (C) is slightly higher than that in presence of (N), this results confirms the previous results. The obtained results of activation energies are all tabulated in Table 4.



**Figure 7: Variation of Logarithm of Corrosion Rate with the Reciprocal of absolute Temperatures for Carbon Steel in 1M  $H_2SO_4$  containing 75 ppm of (C) for different time of immersion.**

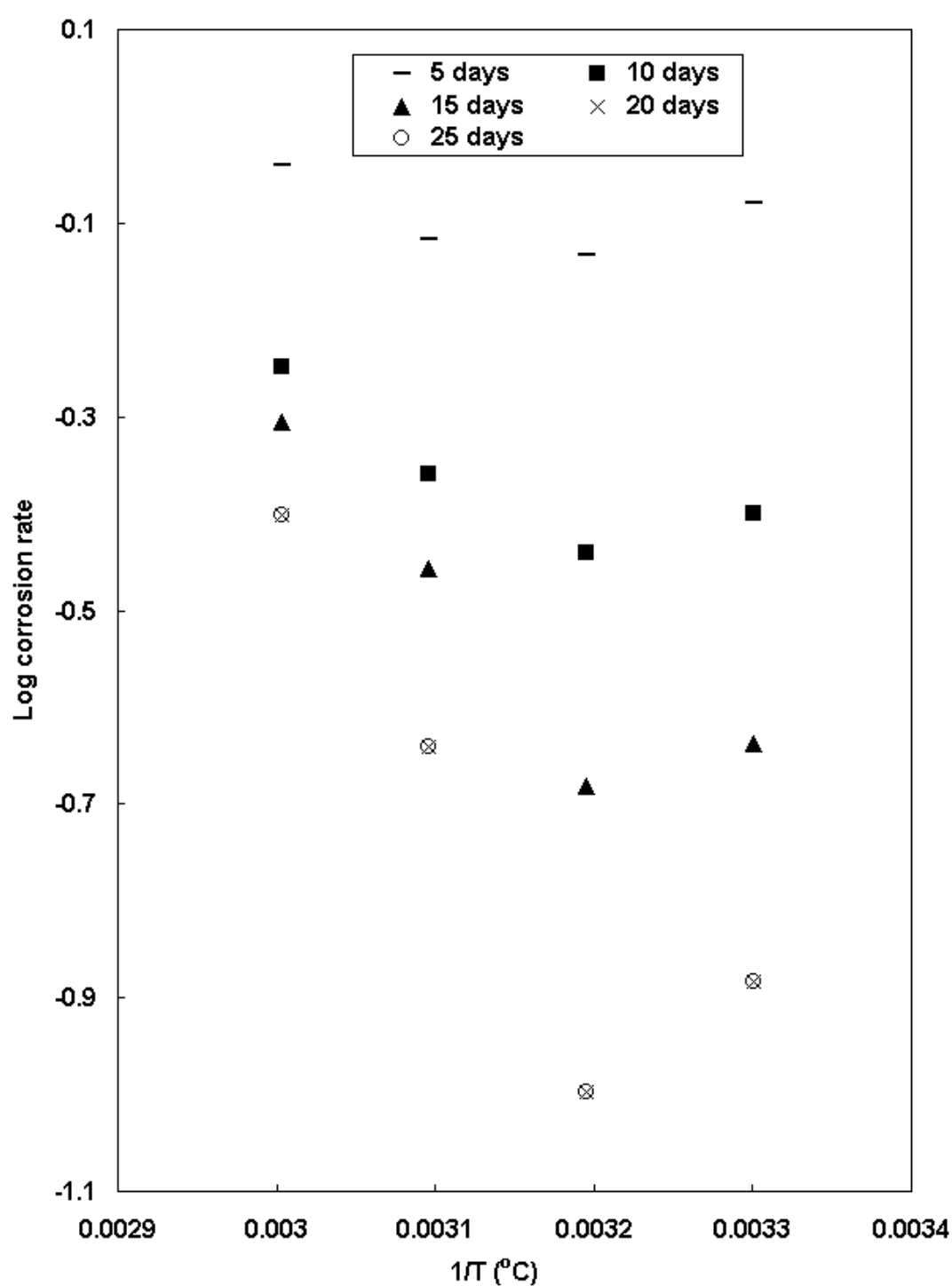


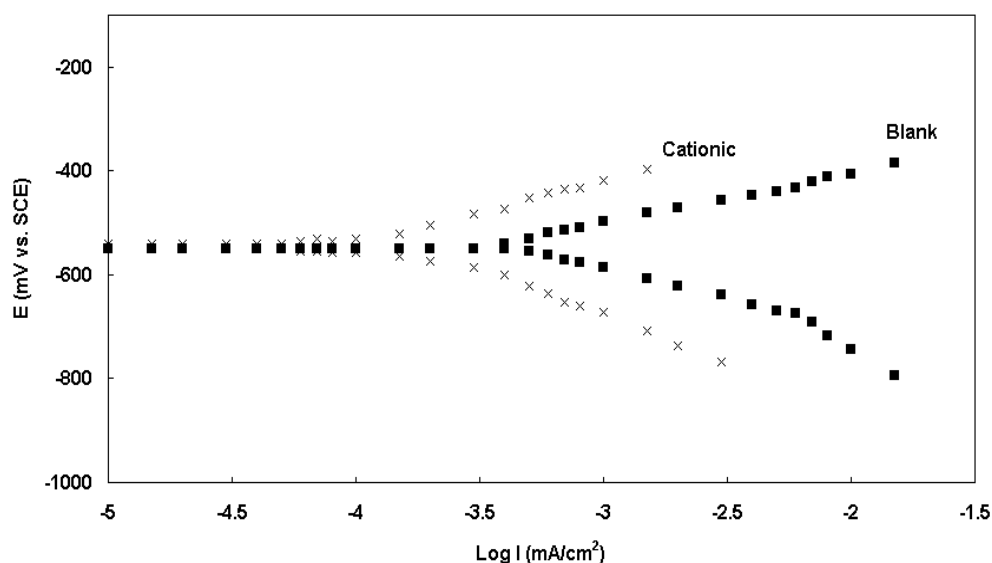
Figure 8: Variation of Logarithm of Corrosion Rate with the Reciprocal of absolute Temperatures for Carbon Steel in 1M  $\text{H}_2\text{SO}_4$  containing 100 ppm of (N) for different time of immersion.

**Table 4: Activation energies of carbon steel in optimum concentrations of (C) and (N) in 1M H<sub>2</sub>SO<sub>4</sub> solution at different times of immersion.**

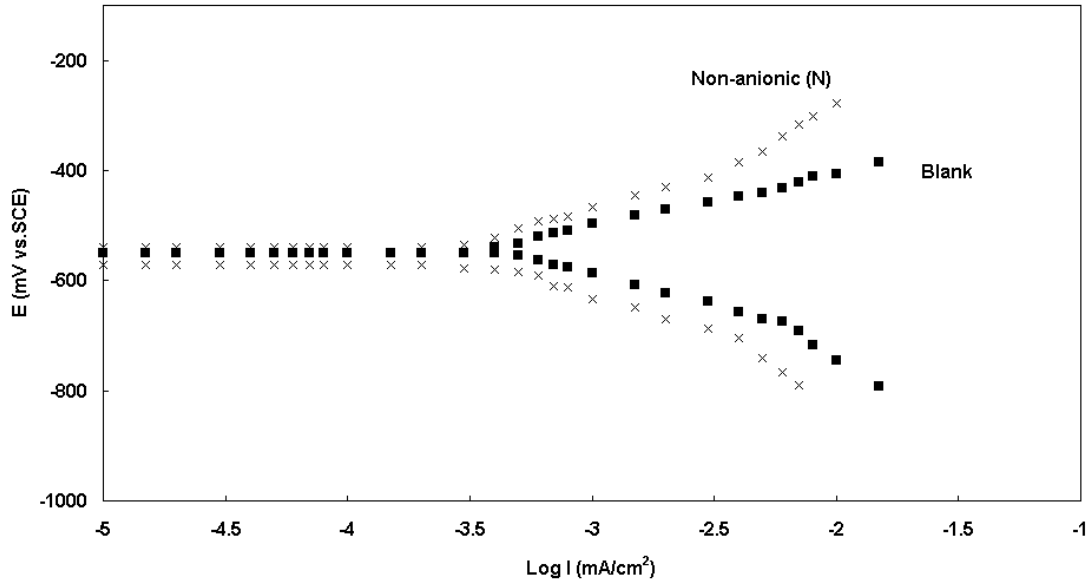
| Time (Days) | E <sub>a</sub> (k J mol <sup>-1</sup> ) |                       |
|-------------|---|-----------------------|
|             | Cationic Inhibitor                      | Non-anionic Inhibitor |
| 5           | 6.82                                    | 2.47                  |
| 10          | 12.24                                   | 10.12                 |
| 15          | 24.08                                   | 23.31                 |
| 20          | 34.44                                   | 34.27                 |
| 25          | 34.44                                   | 34.27                 |

**Galvanostatic Polarization Technique:**

At optimum conditions the polarization curves for these inhibitors are shown in Figure 9 and 10. Electrochemical parameters such as corrosion potential (E<sub>corr</sub>), corrosion current density (i<sub>corr</sub>), and Tafel slopes (i.e., cathodic [b<sub>c</sub>] and anodic [b<sub>a</sub>] Tafel slopes) are calculated from Tafel plots and are given in Table 5.



**Figure 9:** Galvanostatic Polarization Curves for Carbon Steel in 1M H<sub>2</sub>SO<sub>4</sub> (blank) and in 1M H<sub>2</sub>SO<sub>4</sub> containing 75 ppm of (C) at 40°C.



**Figure 10:** Galvanostatic Polarization Curves for Carbon Steel in 1M H<sub>2</sub>SO<sub>4</sub> (blank) and in 1M H<sub>2</sub>SO<sub>4</sub> containing 100 ppm of (N) at 40°C.

**Table 5:** Polarization parameters for the corrosion of carbon steel in 1M H<sub>2</sub>SO<sub>4</sub>.

| Inhibitor concentration (ppm)     | E <sub>corr</sub> (mV) | I <sub>corr</sub> (mA/cm <sup>2</sup> ) | b <sub>c</sub> (mV.dec <sup>-1</sup> ) | b <sub>a</sub> (mV.dec <sup>-1</sup> ) |
|-----------------------------------|------------------------|---|--|--|
| 1M H <sub>2</sub> SO <sub>4</sub> | -550                   | 0.389                                   | 110.76                                 | 74.97                                  |
| 75 ppm of (C)                     | -520                   | 0.165                                   | 109.89                                 | 72.30                                  |
| 100 ppm of (N)                    | -540                   | 0.210                                   | 105.35                                 | 71.16                                  |

The lower  $i_{corr}$  is for 1M H<sub>2</sub>SO<sub>4</sub> containing 75 ppm of (C),  $E_{corr}$  values not show any significant variations for 1M H<sub>2</sub>SO<sub>4</sub> containing 75 ppm and that containing 100 ppm of (N) and none of the inhibitors showed significant changes in  $b_a$  and  $b_c$  slopes, indicating that (C) and (N) are adsorbed onto the metal surface without affecting the anodic- and cathodic-reaction mechanisms. Corrosion inhibition efficiency  $P(\%)$  is calculated using equation <sup>(22)</sup>:

$$P = \left[ 1 - \left( \frac{I_{corr}}{I_{corr,o}} \right) \right] \times 100 \quad (4)$$

where  $I_{corr,o}$  and  $I_{corr}$  are the corrosion current densities in the absence and presence of inhibitor respectively. The comparison between the values of inhibition efficiencies by weight loss and galvanostatic polarization techniques after 5 days at 40°C are



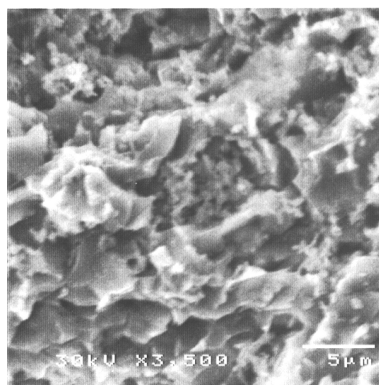
compared in Table 6. It is observed that the results of surface coverage in presence of inhibitor (C) or (N) are in a good agreement with those given by weight loss technique. This means that the inhibition efficiency calculated from Galvanostatic polarization are nearly equal to the values obtained by weight loss technique after 5 days of immersion at 40°C.

**Table 6: Inhibition efficiencies from weight loss and galvanostatic polarization techniques for carbon steel in 1M H<sub>2</sub>SO<sub>4</sub> at optimum conditions.**

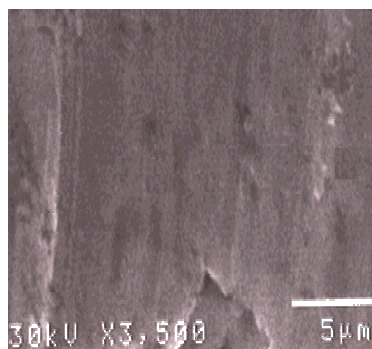
| <b>Inhibitor concentration (ppm)</b> | <b>Inhibition efficiency P (%)</b>  |                                    |
|--------------------------------------|-------------------------------------|------------------------------------|
|                                      | <b>using Polarization Technique</b> | <b>using Weight loss Technique</b> |
| <b>75 ppm of (C)</b>                 | <b>57.58</b>                        | <b>58.01</b>                       |
| <b>100 ppm of (N)</b>                | <b>46.02</b>                        | <b>46.45</b>                       |

#### **Scanning Electron Microscopy:**

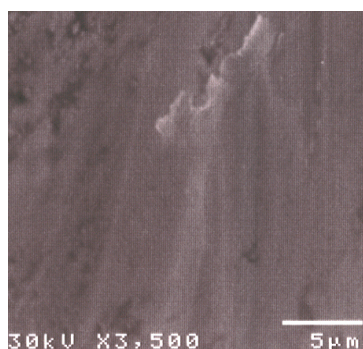
Figure 11 shows SEM image of the surface of the carbon steel specimen after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution for 5 days at 40°C. The micrograph reveals that, the surface is strongly damaged in the absence of the inhibitors (active corrosion). Figure 12 and 13 shows SEM image of the surface of another carbon steel specimen after immersion for the same time interval in 1M H<sub>2</sub>SO<sub>4</sub> solution containing 75 ppm of the inhibitor (C) and 100 ppm of the inhibitor (N) respectively. The micrograph reveals that, there is a decrease in the corrosion sites and pits over the surface of the carbon steel, in Figure 12; it appears that the surface of the specimen is covered greatly due to formation of surface film of the inhibitor. On the other hand, in Figure 13, there are greater numbers of pits in different sites on the surface compared to that in Figure 12. From these observations we can say that the cationic inhibitor give a good inhibition effect for the carbon steel in compared with the non-anionic inhibitor and this confirms the results obtained from the other techniques.



**Figure 11:** Scanning electron micrograph of carbon steel sample after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution.



**Figure 12:** Scanning Electron micrograph of carbon steel sample after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution in presence of 75 ppm of inhibitor (C).



**Figure 13:** Scanning electron micrograph of carbon steel samples after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution in presence of 100 ppm of inhibitor (N).

## **Conclusions:**

- 1- Di decyl benzyl tri ethyl ammonium chloride (C) and Nonyl phenol ethoxylate (N) organic surfactants behave as inhibitors for carbon-steel corrosion in 1M  $\text{H}_2\text{SO}_4$  from 5 days to 20 days at 30°C, it was found that, the corrosion rate decreases with time of immersion up to 20 days then tend to be constant.
- 2- The corrosion rate increases with temperature, but it was found that the lowest corrosion rate values were at 40°C.
- 3- The activation energies were determined using Arrhenius equation, it was found that the activation energies upon using (C) were lower than those upon using (N), indicating that (C) was the much better inhibitor for carbon steel in 1M  $\text{H}_2\text{SO}_4$ .
- 4- Some electrochemical parameters using galvanostatic polarization technique such as  $I_{\text{corr}}$ ,  $E_{\text{corr}}$ ,  $b_a$  and  $b_c$  were given for 75 ppm of (C) and 100 ppm of (N), from the determined  $I_{\text{corr}}$  values, the inhibition efficiencies were calculated and these values are in a good agreement with those obtained from weight loss technique.
- 5- The results of SEM indicate that, the inhibitive efficiencies of each inhibitor are high and the most effective inhibitor is the cationic one, which is in a good agreement with the studied techniques.

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