

Research Article

Corrosion of carbon steel in flowing Sulfuric acid

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Abstract

Corrosion tests of carbon steel metal in 5% H₂SO₄ solution at different operating conditions were performed for ranges of temperature (30 to 60 °C) and rotational velocity (0 to 600 rpm) using both weight loss method and electrochemical polarization technique (Tafel extrapolation). The results revealed different trends of carbon steel against the experimental parameters. The corrosion rate was found to increase considerably with increasing temperature for the majority of results. However, increasing rotational velocity clearly enhanced the corrosion rate for carbon steel. The corrosion potential shows sharp decrease with time in first few minutes and then increase reaching an asymptotic value beyond that. In weight loss experiments, increasing temperature shifted the E_{corr} to more positive at stationary condition. Increasing velocity from 0 to 400 rpm shifted the E_{corr} to more negative at 40 °C. Then trended to positive with increasing velocity to 600 rpm. In polarization experiments, E_{corr} shifted to more positive with increasing temperature from 30 to 40 °C and small change when increasing the temperature from 40 to 60 °C. Increasing velocities shifted the corrosion potential to more positive.

Keywords: Carbon steel, Sulfuric acid, Polarization, Weight loss.

1. Introduction

Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment. Deterioration by physical causes is not called corrosion, but is described as erosion, galling, or wear. In some instances, chemical attack accompanies physical deterioration, as described by the following terms: corrosion – erosion, corrosive wear, or fretting corrosion [Revie and Uhlig, 2008].

Acidic solutions of hydrochloride acid and sulfuric acid have wide industrial applications, the most important fields being acid pickling, acid descaling, industrial cleaning and oil-well acidizing. In aqueous solutions of acids, the surface of metals and alloys are covered with highly protective oxyhydroxide passive film affecting its corrosion behavior [Singh and Ray, 2007]. The corrosion behavior of carbon steel in acidic solution is significant considering its widespread applications, namely, in the manufactures of pipe lines for petroleum industries. Acid solutions are frequently used in the removal of rust and scale-developed in industrial process [Greene *et al*, 1961].

Sameh *et al.*, 2009 were investigated the corrosion behavior of copper in deaerated and oxygenated 0.1 H₂SO₄ solutions using the rotating cylinder electrode under turbulent flow conditions. Potentiostatic polarization measurements were carried out at

different bulk temperatures of 283, 288, 293 and 298 K and various speeds of rotation 100, 200, 300 and 400 rpm.

The anodic dissolution of copper and the hydrogen evolution reaction in deaerated and oxygenated solutions, are activation controlled processes dependent on the temperature of the solution. The anodic dissolution of copper is not mass transfer controlled. Ciubotariu *et al*, 2010 investigated the electrochemical corrosion behavior of carbon steel using the EIS technique and potentiodynamic polarization methods. As test solutions sodium chloride, sodium sulphate and sulphuric acid 0.5M concentration were used.

The reading was taken for different time of immersion at room temperature and for static condition. Authors observed that the corrosion rate is higher in 0.5M H₂SO₄, and concluded that the polarization resistance decreases with time in H₂SO₄ because of the possibility of general corrosion with the dissolution of corrosion products while in 0.5M Na₂SO₄ and 0.5M NaCl solutions it increases with time. Musa *et al.*, 2011 determined the corrosion rate for mild steel in 2.5 M H₂SO₄ at 30°C for different flow velocities. Electrochemical techniques such as Tafel polarization and electrochemical impedance spectroscopy (EIS) were carried out to study the effects of flow velocity on corrosion rate of mild steel. The study revealed that the mild steel corrosion rate decreased with increases in flow velocity due to improvement of the passivation by

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an increase of the oxygen supply. Corrosion potentials were shifted toward the cathodic values and the magnitude of the impedance was rose with flow velocities.

Ajeel *et al.*, 2012 studies the corrosion behavior of low carbon steel in different concentrations of H₂SO₄ and HCL solutions. Weight loss method was used for estimation the corrosion rate in the current study. The results show that the corrosion rate increases with increasing acid concentration from (1 to 5) M. for both types of acid used. The corrosion rate of low carbon steel in acid environments indicates that the hydrochloric acid solution was more corrosive media than that of sulfuric acid solution under the same conditions. Oguike, 2014 has been studied the effect of hydrochloric acid concentration on stainless steel (Fe6956) at temperature range 303 - 333 ± 1 K using weight loss, thermometric and electrochemical polarization techniques. The corrosion rate of the stainless steel was found to be dependent on both temperature variation and acid concentration. He was found appreciable corrosion resistance of the tested Fe6956 coupons at 0.5 M HCl in all the tests, whose resistance was undermined with increase in both temperature and acid concentration. The corrosion rate was found to increase with increasing temperature and acid concentration.

To control the corrosion, good understanding of the effect of operating conditions on the corrosion behavior is required. Therefore, this work aims to study free corrosion behavior of carbon steel metal in sulfuric acid in ranges of temperatures and agitation speed. Two different technique will be used to measure the corrosion rate that are: weight loss and electrochemical polarization.

2. Experimental work

Experimental work was carried out to determine the corrosion rate of carbon steel (2×4cm² coupon specimen) under flow conditions at a rotational velocity range of 0 to 600 rpm, temperature 30, 40, 50 and 60 °C, in 5% (wt/v) sulfuric acid solution using weight loss and electrochemical polarization methods.

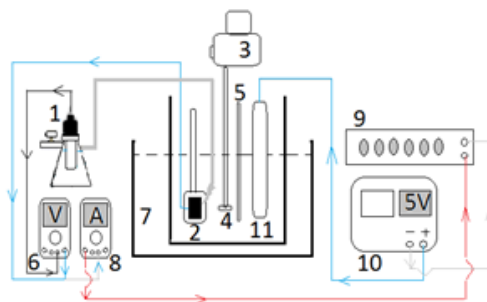
The experimental work is divided into three parts

- 1- Weight loss in order to determine the average corrosion rates under a rotational velocity of 0, 400, and 600 rpm, and temperatures 30, 40, 50 and 60 °C, which was performed to assess corrosion rate of Carbon steel specimen in a solution of 5% wt/v H₂SO₄.
- 2- Measurement of corrosion potential as a function of time against saturated calomel electrode (SCE) for all weight loss experiments.
- 3- Electrochemical polarization investigations in 5% H₂SO₄ solution to determine corrosion rate, i.e., corrosion current density using Tafel extrapolation method based on anodic and cathodic polarization

curves under different conditions of a rotational velocity of 0, 400, and 600 rpm, and temperatures 30, 40, 50 and 60 °C.

The chemical composition for carbon steel 0.62%V, 0.00% Cr, 0.22% Mn, 98.71% Fe, 0.08% Cu, and 0.08% Mo. The specimen was hole from the center in a very small area can be negligible for purpose installing into the solution by screw.

Fig.1 shows the experimental apparatus which was composed of stirrer to obtain different velocity, digital voltmeter to measure the potential, digital ammeter to measure the current, water bath having temperatures range of 10-95°C to obtain different solution temperatures, power supply to provide a constant applied voltage between the electrodes, variable resistance (rheostat) (0-10⁶ ohm) to control the current flow. Before each experiment the specimen (working electrode) was abraded with glass emery paper of grade numbers; 120, 180, 220, 400 and 2000 respectively, washed continuously with brushing by plastic brush in running tap water, followed by distilled water, dried with a clean tissue, followed by ethanol for 30 second dried with clean tissue, and then dried by using electrical oven to temperature of about 110 °C for 10 minute [Mahato *et al*, 1980]. The specimen then was stored in the desiccator over high activity silica gel until use.



1-calomel electrode (reference electrode), 2-working electrode (specimen), 3-motor, 4-stirrer, 5-thermometer, 6-voltmeter, 7-water bath, 8-ammeter, 9-resistance box, 10-power supply, 11-graphit electrode

Fig.1 Experimental apparatus

Then the specimen was weighted to the 4th decimal of gram (w₁) by using digital balance and dimensions by using ruler to the 2nd decimal of millimeter before use. After that one face of the rectangular coupon was exposed to corrosion environment for 1 h immersion in acid solutions, while the other face was completely insulated by insulating tape.

At the end of weight loss experiment, the specimen was washed by tap water with brushing to remove the corrosion products that formed on the outside surface, washed with distilled water, dried with clean tissue, rinsed in ethanol and dried by using electrical oven to a temperature about 110 °C for 10 minute. Then the specimen was kept in the desiccator to cool and then weighted (w₂). So that the corrosion rate can be determined using:

$$CR = \frac{\Delta W}{A \times t} \quad (1)$$

Where, CR=corrosion rate, ΔW =weight loss (w1-w2), A= area, and t= time.

The change in the open circuit potential of specimen as function of time was measured at the all weight loss experimental run. The electric circuit shown in Fig. 3-9 represents the half-cell potential measurement by using Saturated Calomel Electrode SCE bridged by a Luggin capillary. The test method was accomplished by placing the Luggin capillary at a distance of 1- 2 mm from the working electrode. The half-cell potential was record directly on the computerize at each 1 second for the first 10 minute and each 1 min until reach stabilize potential for 1 hour immersed in acid solution.

To calculate corrosion rate by electrochemical polarization method need to anodic and cathodic polarization curve. The electrical circuit shown in Fig.1, was connected in order to obtain the cathodic polarization curve the working electrode connected to the (-ve) terminal of power supply to serve as cathode and graphite to the (+ve) terminal to serve as anode). After the solution reached the required temperature, the specimen (cathode) and graphite (anode) were immersed in solution and the electrical circuit was switched on, where the power supply was set at 5 V (applied voltage). As the test run started, the steady state current in (mA) and potential in (V) of specimen were read. The potential was changed (5 mV) for each step and after one minute period the steady state current was recorded for each potential step, where capillary tip was always placed at a fixed distance of 1- 2 mm from cathode (specimen).

The specimen was cathodic polarized starting from maximum current pass through the cell because the resistance is very low to the corrosion potential (where $i_{app} = 0$) at a rate of 5 mV by changing the applied current using rheostat. The specimen have a clean surface in the cathodically polarized retains to cathodic protection. In the end of the cathodic polarization the anodic polarization curve was start from corrosion potential (where the current was zero) by reversing the connection of the power supply, i.e. connected the working electrode to (+) and the auxiliary electrode to the (-) of the power supply. After collecting the data by recording the current and potential, polarization curve can be drawn by using semi log scale where the x-axis represents the current density (log scale) and the y-axis represents the potential.

3. Results and Discussions

3.1 Weight loss method

In weight loss experiments, the corrosion rates of different metals in the sulfuric acid solution were investigated under ranges of temperatures and flow conditions. Fig.2 and 3 illustrates the variation of corrosion rate of carbon steel (CS) that expressed in

gmd with temperature and flow velocity for a 1 h of immersion time.

From Fig. 2 it is clear that the corrosion rate increases with increasing temperature for all investigated metals at the stationary condition. The effect of temperature on the corrosion rate for mass transfer controlled systems is governed by two changing parameters affecting the corrosion rate in conflicting ways that are the O₂ solubility and diffusivity. Increasing the temperature will increase the rate of oxygen diffusion to the metal surface by decreasing the viscosity of aqueous solutions resulting in enhanced corrosion rate. On the other hand, increasing temperature decreases the oxygen solubility the factor that restrains the corrosion [Mahato *et al*, 1980].

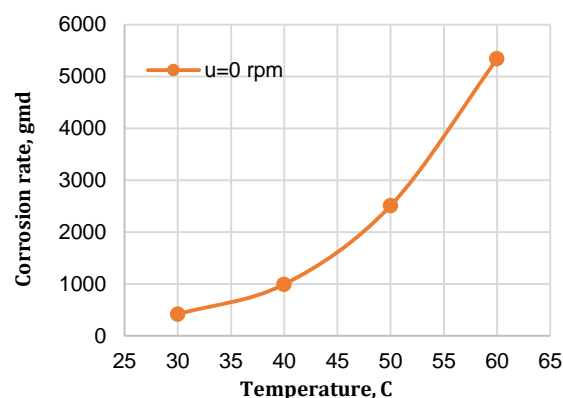


Fig.2 Variation of corrosion rate with temperature at stationary

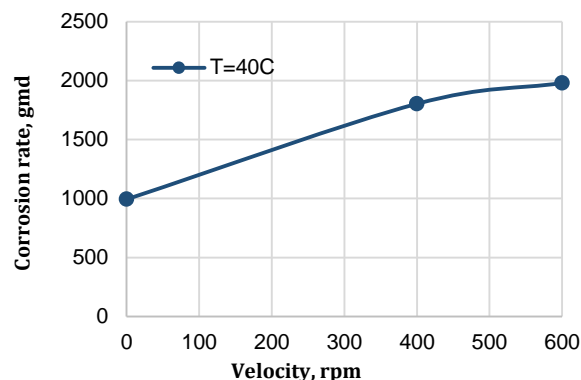


Fig.3 Variation of corrosion rate with velocity at different temperature

From Fig. 3 it is clear that increasing the flow velocity leads to an increase in the corrosion rate. This can be attributed to the increase in the concentration of oxygen close to the metal surface by eddy transport. The rate of oxygen reduction reaction is generally limited by the speed at which oxygen can reach the surface of the metal. Previous studies [Foroulis, 1979; Scheers, 1992; Shreir *et al*, 2000; Slaiman and Hasan, 2010; Hasan and Sadek, 2013] indicated that the greater turbulence due to high velocities results in more uniform O₂ concentration near the surface.

Previous studies [Alwash *et al*, 1987; Turkee, 2009; Hasan and Sadek, 2013] showed that the corrosion rate in acidic solutions increases as the flow increases. Also, according to the experimental results in Table 1, it is possible to say that the measured cathodic currents (O₂ reduction and H⁺ reduction) is affected by flow and that can be associated to the H⁺ diffusing through the corrosion products layer, where they are reduced to H₂ gas. For that reason, the H⁺ reduction is flow dependent [Ross *et al*, 1966; Poulson, 1993].

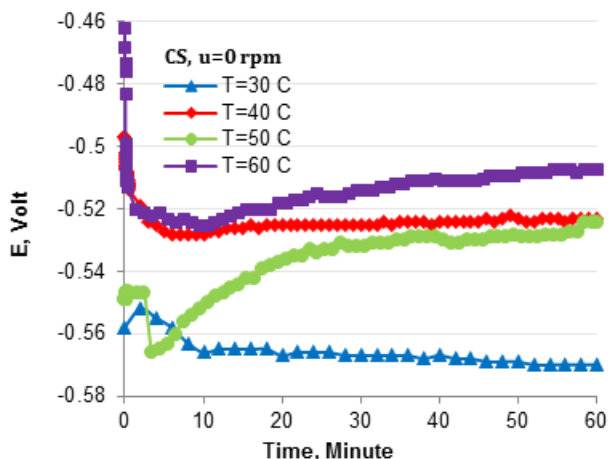


Fig.4 Corrosion potential vs. time of CS at stationary condition for 1 h immersion time at different temperature

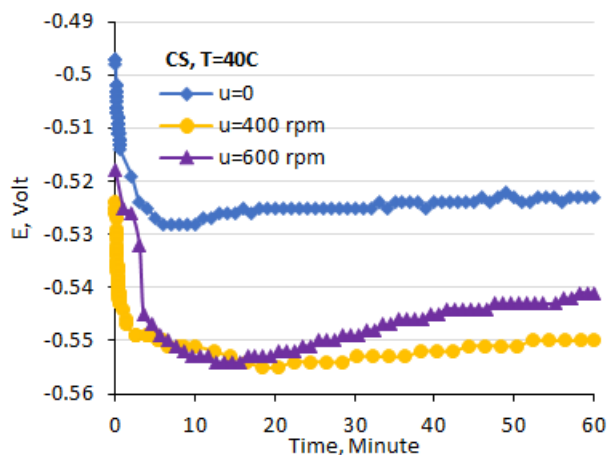


Fig. 5 Corrosion potential vs. time of CS at isothermal condition and different velocity for 1 h immersion time

Fig. 4 show the E_{corr} vs. time curves of carbon steel for 1 hour immersion time in H₂SO₄ solution. In order to analyze the E_{corr} vs. time curves for carbon steel as in fig. 4 at 30, 40, 50 and 60 °C. It is clear that the initial decrease of E_{corr} is observed before its sustained increase. This may be due to a partial dissolution of the air formed film followed by the posterior growth of a passivating film. However, CS reveals different trend at 30 °C where it exhibits potential decrease with time. The decrease of potential with time is due to the occurrence of continuous corrosion. It seems that at this condition, the formation of corrosion product layer does not affect the continuity of corrosion.

The E_{corr} at 40 °C is higher than that at 30 °C and 50 °C. This behavior of corrosion rates indicates that the combined effect of O₂ solubility and molecular diffusivity is highest at 60 °C followed by 40 °C and 50 °C and 30 °C i.e. at 60 °C and 40 °C the effect of these two parameters on the corrosion rate is maximum. These result agreement with literatures [Azambuja *et al*, 2003; Hasan and Sadeq, 2013].

Fig. 4 also reveal that initially the OCP has unstable trend with temperature, while the asymptotic values of OCP shifts to more noble values with increasing temperature. This shift can be ascribed to the formation and growth of a Fe₂O₃ film on the corroding surface.

Table 1 Corrosion rate of carbon steel in 0.52 M H₂SO₄ at isothermal condition T=40 °C for 1 h of immersions time by using weight loss method

u, rpm	ΔW, gm	A, Cm ²	CR, gmd	CR, mdd	i _{corr} , mA/cm ²
0	0.0331	8	993.4	9934	3.9835
400	0.0647	8.61	1803.4	18034	7.2320
600	0.066	8	1980	19800	7.9398

Fig. 5 shows the variation of OCP of CS with time at various agitation velocities. The figure reveals that when the velocity increases from 0 to 400 rpm, the potential is more negative. This is due to the increased arrived O₂ to the metal surface which leads to an increase in the polarization resistance to more negative values. At 600 rpm the potential shift higher than 400 rpm after 16 min.

Fig. 6 shows the E_{corr} versus temperature for carbon steel metal at stationary condition. It is clear that the E_{corr} shifts to more positive with increasing temperature. This rise in temperature may decrease the equilibrium potential of Fe and H₂ but increase the equilibrium potential of O₂ [L. Cifuentes]. The increase the diffusion rate of oxygen species by increasing the molecular diffusion coefficient and decreases the oxygen solubility. This result agrees with [Hasan *et al*, 2011] for carbon steel in 0.1N NaCl solution.

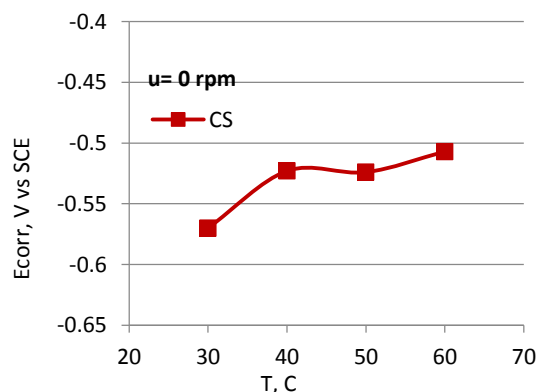


Fig.6 Variation of E_{corr} with temperature at stationary condition for 1 h immersion time (weight loss method).

Fig. 7 shows the E_{corr} versus velocity for carbon steel metal at constant temperature (40 °C). It can be seen the E_{corr} shifts to more negative with increasing velocity from 0 to 400 rpm. Then small trend to positive with increasing velocity to 600 rpm. Thus, it may be attributed this behaviour to an improvement of the passivation by an increase of the oxygen supply [Ochoa *et al*, 2005; Musa *et al*, 2011].

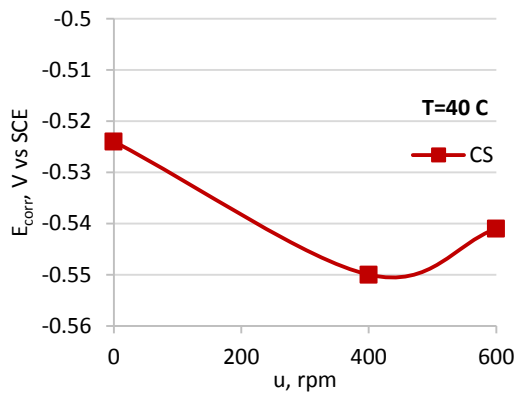


Fig.7 Variation of E_{corr} with velocity at isothermal T=40 °C for 1 h immersion time (weight loss method)

3.2 Polarization method

Polarization technique was also used to evaluate the corrosion rate of carbon steel in 0.52 M H₂SO₄ solution. Plots of cathodic and anodic polarization curves were used to determine corrosion currents density (i_{corr}), corrosion potential (E_{corr}), anodic Tafel slope (β_a) and cathodic Tafel slope (β_c) as shown in Table 2.

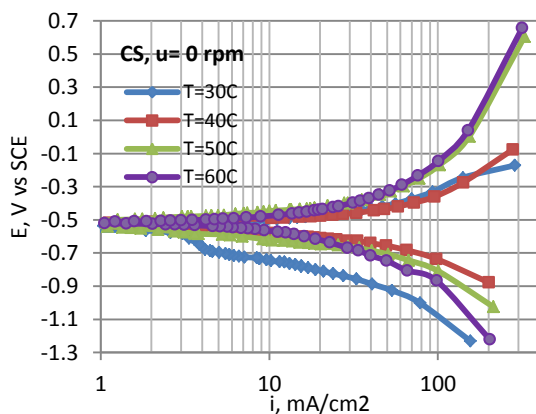


Fig.8 Anodic and cathodic polarization curves of carbon steel in stationary condition at different temperature

It is seen from the fig. 8 that the polarization curves are shifted to the high current density region as the temperature of H₂SO₄ solution is increased, indicating the increase in the corrosion rate with the increase temperature of H₂SO₄ solution as shown in Table 2.

Table 2 shows that the values of corrosion potential (E_{corr}) where slightly changed while the values of corrosion current densities (i_{corr}) of carbon steel were increased with increasing in solution temperature.

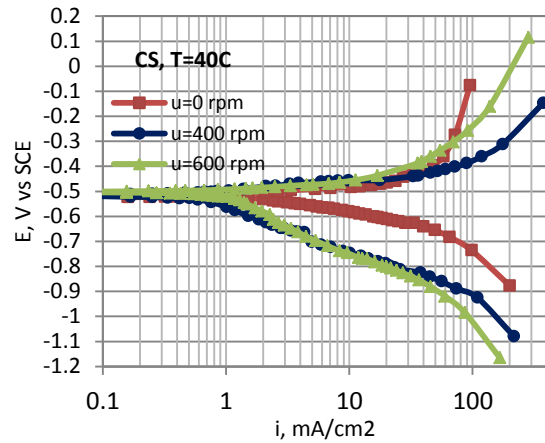


Fig.9 Anodic and cathodic polarization curves of Carbon steel at constant temperature and change agitation velocity

Fig. 9 shows the corrosion potential shift to more positive with increasing velocity due to the increased O₂ transport to the surface. This agrees with previous findings [Foroulis, 1979; Mahato *et al*, 1980; Silverman, 1984; Hasan, 2003; Hasan *et al*, 2011].

Table 2 Polarization parameters for carbon steel with different temperature and velocity in 0.52 M H₂SO₄ solution

T °C	u rpm	β_a V.cm ² /mA	β_c V.cm ² /mA	E_{corr} V	i_{corr} mA/cm ²
30	static	0.2867	-0.2679	-0.545	1.2
40	static	0.2679	-0.4040	-0.522	4
	400	0.2493	-0.7265	-0.520	6
	600	0.4663	-0.9657	-0.515	7
50	static	0.7535	-0.7535	-0.520	7
60	static	1.1917	-1.4281	-0.518	14

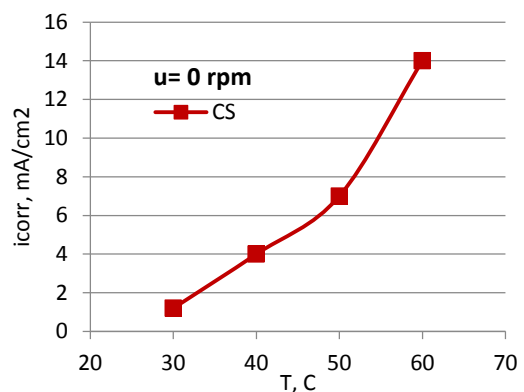


Fig.10 Variation of i_{corr} with temperature at u=0 rpm, by polarization

Fig. 10 and 11 shows the effect of temperature on i_{corr} and E_{corr} at stationary condition ($u=0$ rpm). Fig. 10 indicates a considerable increase in i_{corr} with temperature. As explained earlier, increasing the temperature will increase the rate of oxygen diffusion to the metal surface by decreasing the viscosity and enhancing the corrosion rate. The results for I_{corr} is comparable with weight loss method.

Fig.11 shows the E_{corr} shifts to more positive with increasing temperature from 30 to 40 °C and small change when increase temperature from 40 to 60 °C, due to increase oxygen supply on the surface metal with increase temperature.

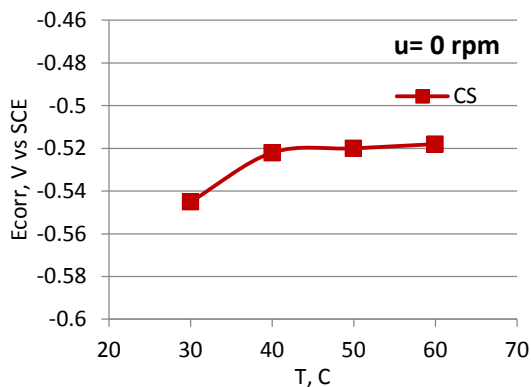


Fig.11 Variation of E_{corr} with temperature at $u=0$ rpm, by polarization

Fig. 12 and 13 shows the effect of velocity on i_{corr} and E_{corr} at isothermal temperature ($T=40$ °C), it is clear that increasing the rotational velocity leads to an increase in the exchange current density. The increase in the i_{corr} is due to the increase in the transport of oxygen towards the metal surface [Foroulis, 1979]. In fig. 13, the increment in E_{corr} with velocity is due to increase in oxygen transport to the metal surface and when the system is free from oxygen, velocity has no effect on corrosion potential (E_{corr}) [Ross *et al*, 1966].

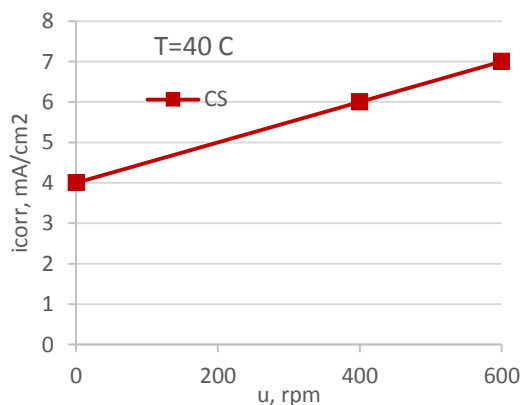


Fig.12 Variation of i_{corr} with velocity at isothermal $T=40$ °C, by polarization

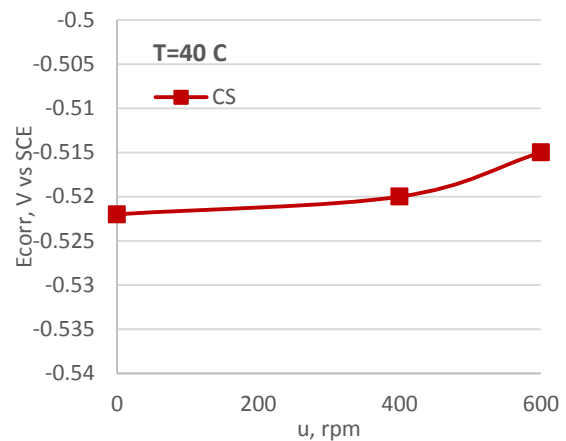


Fig.13 Variation of E_{corr} with velocity at isothermal $T=40$ °C, by polarization

Conclusions

- 1) The corrosion rate was found to increase considerably with increasing temperature for the majority of results. However, increasing rotational velocity clearly enhanced the corrosion rate for carbon steel.
- 2) The corrosion potential shows sharp decrease with time in first few minutes and then increase reaching an asymptotic value beyond that.
- 3) In weight loss experiments, increasing temperature shifts the E_{corr} to more positive at stationary condition. Increasing velocity from 0 to 400 rpm shifts the E_{corr} to more negative with at 40 °C. Then small trend to positive with increasing velocity to 600 rpm.
- 4) The polarization curves are shifted to the high current density region as the temperature and velocities of H_2SO_4 solution is increased, indicating the increase in the corrosion rate with the increase temperature and velocity.
- 5) In polarization experiments, E_{corr} shifts to more positive with increasing temperature from 30 to 40 °C and small change when increase temperature from 40 to 60 °C. Increasing velocities shifts the corrosion potential to more positive.

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