



## The effect of acetic acid on the CO<sub>2</sub> corrosion of grade X70 steel

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### ABSTRACT

The effect of acetic acid (HAc) on the CO<sub>2</sub> corrosion of grade X70 steel was investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), polarization tests and electrochemical impedance spectroscopy (EIS). In the absence of acetic acid, a fairly dense layer of iron carbonate (FeCO<sub>3</sub>/siderite) was formed. At 500 ppm HAc, FeCO<sub>3</sub> layer became more porous. In addition, anodic/cathodic polarization curves were activated with the more pronounced effect on the cathodic side. By adding 1000 ppm HAc, similar polarization behavior was obtained and FeCO<sub>3</sub> layer became yet more porous than previous conditions. At 2000 ppm HAc, FeCO<sub>3</sub> layer disappeared completely, while polarization behavior changed and the limiting diffusive current density was observed in the cathodic side. There were two major increases in the corrosion rate at 500 and 2000 ppm HAc. The EIS results reflected similar behavior for the specimens exposed to the solutions with 0–1000 ppm HAc. Under these conditions, a charge transfer controlled behavior due to the FeCO<sub>3</sub> layer was observed which was accelerated by increasing HAc concentration. At 2000 ppm HAc, the corrosion behavior changed considerably and the formation/adsorption of corrosion product followed by the dissolution process was observed.

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### 1. Introduction

The effect of acetic acid (HAc) on the corrosion rate of carbon steel in most of the oil and gas fields containing brine and CO<sub>2</sub> has been studied [1–5]. Acetic acid is the most common organic acid in multiphase systems containing brine. CO<sub>2</sub> corrosion in the presence of HAc is known as a major reason for premature failures in oil and gas pipelines, which are usually made of carbon steel [1,6].

In 1983, Crolet and Bonis [7] reported that the presence of HAc in brine will significantly increase the corrosion rate. He and his co-workers [8] suggested the direct reduction of HAc in the surface of steel. Hedges and McVeigh [9] reported that HAc dissociated incompletely, which could cause a reserve of H<sup>+</sup> ions more than the value defined using solution pH ( $-\log[H^+]$ ).

Oblonsky et al. [10] found solid Fe<sub>3</sub>O<sub>4</sub> and dissolved Fe<sup>2+</sup> as the corrosion products in the corrosion of Fe in the solution saturated with argon in the presence of acetate ion (in the absence of CO<sub>2</sub> corrosion). They found that acetate concentration had not any effect on the chemical analysis of corrosion product layer. Joosten et al. [11] saw an increase in corrosion rate in the presence of HAc due to decreasing of pH. Garsany and co-workers [5,12,13] emphasized that due to the fast dissociation of HAc, it is impossible to distinguish the reduction of HAc from that of H<sup>+</sup>.

Nafday and Nešić [14] reported that HAc cannot cause any localized corrosion, has not any effect on the thickness of corrosion product iron carbonate (FeCO<sub>3</sub>/siderite) layer but affects layer morphology. However, Okafor and Nešić [15] reported that acetic acid can cause localized corrosion by removing iron carbonate layer. George and Nešić [6] reported that the presence of HAc strongly affects the cathodic limiting current. The anodic reaction (iron dissolution) was unaffected or mildly retarded with increasing HAc concentration at room temperature. Gulbrandsen [3] reported undermining of corrosion product FeCO<sub>3</sub> layer in the presence of HAc.

Liu et al. [16] investigated the effect of HAc using electrochemical impedance spectroscopy (EIS). They found that the surface chemical reactions of cathodic reduction were enhanced in the presence of HAc. They also found that HAc can remove FeCO<sub>3</sub> layer. Zhang and Cheng [17,18] reported similar results. In addition, they observed an increase in the current density of anodic reactions and they saw localized corrosion on the surface of steel.

As it is seen, only Oblonsky et al. [10] investigated the chemical analysis of corrosion product layer in the presence of acetic acid and in the absence of dissolved CO<sub>2</sub> systematically. The important point is that in the CO<sub>2</sub> corrosion, under a special condition a protective corrosion product layer can form on the surface of steel. Therefore, there seems to be a gap in the investigation of chemical analysis of corrosion product layer in CO<sub>2</sub> corrosion of carbon steels in the presence of acetic acid. In addition, most of the researches have focused on the effect of acetic acid or acetate on

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the surface chemical reactions (especially in the absence of any protective layer).

There seems to be a deficiency in studying the morphology of corrosion product layer and also investigating the electrochemical behavior of corrosion in the presence of protective layer, in particular the use of EIS technique.

The main goal of this research is to investigate the effect of acetic acid on the characteristics of corrosion product layer (film/scale) and on the electrochemical behavior of the CO<sub>2</sub> corrosion of grade X70 steel in the presence and absence of corrosion product layer. The X70 steel, used for construction of pipelines, is a fairly new product in the family of carbon and low alloy steels, whose behavior against CO<sub>2</sub> corrosion in the presence of acetic acid has not been fully established. For this purpose, surface analyses such as X-ray diffraction (XRD) and scanning electron microscopy (SEM) and also electrochemical analyses such as polarization test and EIS have been conducted.

## 2. Experimental procedures

The samples were made of grade X70 steel with the nominal composition (Fe: base, C: 0.037, Si: 0.141, Mn: 1.291, P: 0.006, S: 0.013, Cr: 0.011, Ni: 0.156, Cu: 0.024, Mo: 0.207, Nb: 0.018, Co: 0.015, Al: 0.055, B: 0.006, Ti: 0.017, V: 0.039). The shape of samples was cylindrical with the exposed surface area of about 5.3 cm<sup>2</sup>. They were polished using 60–2000 grit silicon carbide papers, then degreased in acetone using an ultrasonic cleaning device and finally washed with ethanol.

The experiments were carried out in a glass cell filled with 2.5 L of deionized water and 3 wt% sodium chloride (NaCl) at atmospheric pressure in the temperature of 75 °C for 24 h. An image of the test set-up is shown in Fig. 1.

Before commencing the tests, the solution was deoxygenated by purging CO<sub>2</sub> for at least 2 h. Then the in situ pH was adjusted at 6.5 using either sodium bicarbonate (NaHCO<sub>3</sub>) or hydrochloric acid (HCl) addition. Then acetic acid was injected to the cell. Finally, samples were put in the holder, which was made of Teflon, and immersed inside the electrolyte. CO<sub>2</sub> purging was continued throughout the tests. The samples' code names are shown in Table 1. In each cell only one sample was used, while for each test a minimum of 2 experiments were carried out in order to observe reproducibility of the results.

Phase analysis of corrosion product scale was obtained via low angle XRD method using  $\varphi = 3^\circ$  with a Philips X'Pert pro instrument using Cu K $\alpha$  radiation, which is utilized for investigating thin layers. The morphology and thickness of the corrosion product layer were studied via SEM model CamScan MV 2300 OXFORD instrument.



Fig. 1. An image of the test set-up.

Table 1

The name of samples used in this research.

Concentration of HAC (ppm)	0	500	1000	2000
Name of sample	HAc0	HAc500	HAc1000	HAc2000

The polarization tests were carried out in a three-electrode cell using an EG&G potentiostat/galvanostat, model 273A. A platinum ring and an Ag/AgCl electrode were used as counter and reference electrodes, respectively. The EIS tests were undertaken using a Solartron model SI 1255 HF frequency response analyzer (FRA) coupled to the previously mentioned potentiostat/galvanostat. The EIS measurements were obtained in a frequency range of 100 kHz to 0.01 Hz with an applied AC signal of 5 mV (rms) using EIS software model 398.

## 3. Results and discussion

### 3.1. Surface analyses

#### 3.1.1. X-ray analysis

The XRD patterns of the samples are shown in Fig. 2. The peaks associated with iron carbonate and iron can be seen in the sample HAc0. The numbers and intensities of the peaks associated with FeCO<sub>3</sub> were much more than those of iron. So, it could be said that iron carbonate was the dominant phase [19]. HAc500 specimen had more iron carbonate on its surface than HAc0. Therefore, it could be suggested that the thickness of the FeCO<sub>3</sub> layer in HAc500 was more than that of HAc0. Furthermore, it was found that the amount of iron carbonate had decreased in sample HAc1000. In HAc2000 there was not any FeCO<sub>3</sub> and the only observed peak was associated with iron (Fe). Therefore, it could be deduced that HAC concentration affected the amount of precipitated FeCO<sub>3</sub> but did not have any effect on the phase analysis of the corrosion product layer.

It is known that crystalline particles can be formed of some crystallites. The mean size of iron carbonate crystallites can be measured by X-ray peak broadening of the most intense diffraction peak of FeCO<sub>3</sub> using the Scherrer formula [19–21]:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where  $D$  is the crystallite size in nanometer,  $\lambda$  is the incident X-ray wave length in nm (0.154056 for Cu K $\alpha$ ),  $\beta$  is the full width at half maximum (FWHM) of Cu diffraction peak in radians and  $\theta$  is the

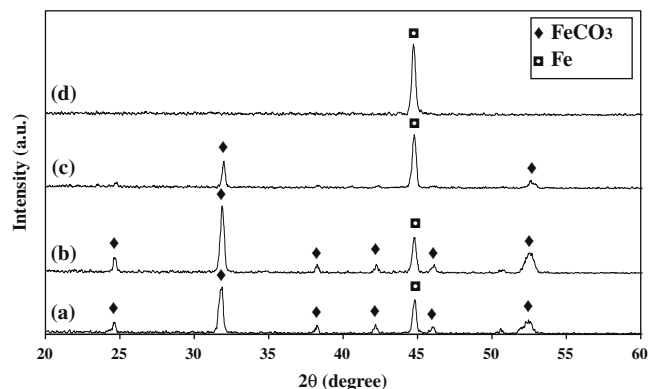


Fig. 2. The XRD patterns of the X70 steel exposed at 75 °C for 24 h in the presence of (a) 0, (b) 500, (c) 1000 and (d) 2000 ppm of acetic acid in CO<sub>2</sub>-saturated 3 wt% NaCl solution.

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