

Corrosion Inhibition of 13Cr Stainless Steel in HCl/HAc/HF Acid Solution

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The corrosion and inhibition of 13Cr stainless steel in conventional HCl/HAc/HF acid solution were investigated by electrochemical measurements and soaking experiments. Results manifested that the HAc/HF acid solution as a low-corrosion acid can replace the conventional HCl/HF acid in oil well acidification construction process. EIS studies indicated that the mixed inhibitor formed by regular inhibitor and oxalic acid can effectively inhibit the corrosion reaction by forming an adsorption layer function as a barrier. Polarization curves indicated that the mixed inhibitor can reduce anodic dissolution and cathodic hydrogen evolution reactions simultaneously. In addition, the corrosion morphology of the corrosion product was examined by scanning electron microscopy. The results of the study can provide a theoretical basis for the search of low-corrosive acid to replace the conventional acid in oil well acidification construction process.

Keywords: 13Cr stainless steel, HCl/HAc/HF acid, corrosion inhibitor, oxalic acid, weight loss method, electrochemical measurement

1. INTRODUCTION

In recent years, the development of gas wells increased with the growing demand for energy in the world. As 13Cr stainless steel has good corrosion inhibition in gas wells environment, it is widely used in the tube and casing of gas wells [1-4]. Acidification is a conventional and necessary method for gas wells to stimulate and stabilize production [5-7]. The main acid solution system used for acidification is mud acid, which consists of HCl and HF [8-10]. However, 13Cr stainless steel has poor acid corrosion resistance [11, 12], and the tubing failure statistics in gas wells show that the common characteristic of tubing failure wells within one year of production is caused by acidification. Therefore, it is necessary

to pay attention to the corrosion mechanism and anticorrosion method of 13Cr stainless steel in acid environment.

It has been reported that the corrosion of 13Cr stainless steel in acid environment is mainly pitting corrosion [13], especially in the presence of Cl [14-16]. The experts found that the corrosion of 13Cr stainless steel during acidification can be effectively solved by adding corrosion inhibitor containing iminazole [17], alkynol [18], aldehyde [19], furfurylalcohol [20] or diethylenediamine [21]. Cr inhibits metal corrosion by forming a passivation film to isolate the metal from the corrosion solution [22]. However, the passivation film will be dissolved rapidly in acid solution [23], so the tube and casing of 13Cr stainless steel is easy to be corroded during acidification [11, 12]. In order to reduce the corrosion of steel tube and casing of 13Cr stainless steel caused by acidification, the corrosion inhibitor should form an effective barrier on the 13Cr stainless steel surface to reduce the dissolution of the passivation film by acid solution [24].

In this paper, the corrosion inhibition effect of a mixture obtained by corrosion inhibitor and oxalic acid according to the mass ratio on 13Cr stainless steel was studied. The corrosion inhibitor can form a physical film and the oxalic acid can combine with Cr to form a chemical layer on the steel surface, which lessens the corrosion of acid solution on the steel, and reduces pitting corrosion of 13Cr stainless steel by preventing acid solution to Cr passivation film [25]. The corrosion behavior and corrosion inhibition performance of the inhibitor on 13Cr stainless steel in acid solution were studied by weightlessness and electrochemical method. It is hoped that the corrosion inhibitor can reduce the corrosion of 13Cr stainless steel during acidification.

2. EXPERIMENT

2.1 Materials

Hydrochloric acid, hydrofluoric acid, acetic acid, oxalic acid, imidazoline, butylene glycol, polyvinylpyrrolidone, ethanol and acetone were obtained from Chengdu Kelon Chemical Reagent Company of China, and of analytical reagent grade. The oleic-based imidazoline was purchased from Chengdu Acidizing Petroleum Technology Development Co., Ltd.. The 13Cr stainless steel specimens (50mm×10mm×3mm) used in this work were investigated in the as-received condition. Its chemical composition is shown in Tab. 1. The corrosion solution was prepared by diluting the HCl, HF and HAc with distilled water. The corrosion inhibitor was composed of 85% oleic-based imidazoline (C₂₂H₄₃N₃), 10% butylene glycol (C₄H₁₀O₂) and 5% polyvinylpyrrolidone ((C₆H₉NO)_n).

Table 1. Chemical composition of 13Cr stainless steel

Alloy	C	Si	S	Cr	Ni	Fe
Wt%	0.02	0.3	0.5	13.8	6.2	Bal.

2.2 Instruments

The following instruments were used: DZKW-4 constant temperature water-bath ($\pm 0.1^\circ\text{C}$, Zhongxingweiye, Beijing, China); FA2204 electronic analytical balance ($\pm 0.1\text{mg}$, Shanghai Anting, China); CHI600 electrochemical workstation (Chenhua Instrument Company, Shanghai, China); scanning electron microscope (SEM, Apreo S produced by Prisma E) with energy dispersive spectroscopy (EDS).

2.3 Experimental method

2.3.1 Corrosion experiment with the weight loss method

The 13Cr stainless steel was washed with acetone and ethyl alcohol, then dried and weighed. The 13Cr stainless steel weight loss experiments were performed by immersing the steel in the Acid 1 to Acid 10 solution for different temperatures. The steel was immersed in acid solution using a constant temperature water bath. Then, after four hours, the 13Cr stainless steel was removed, cleaned, dried and reweighed. The corrosion rate (C_R) was calculated using equation (1):

$$C_R = \frac{10^6 \Delta m}{St} \quad (1)$$

where C_R ($\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$) is the corrosion rate, S (mm^2) is the surface area of the steel coupon, t (h) is the time of the corrosion reaction, and Δm (g) is the weight loss of the steel specimen. The inhibition efficiency (IE) was calculated using equation (2):

$$IE = \frac{C_R - C_{Ri}}{C_R} \times 100\% \quad (2)$$

where C_R and C_{Ri} in equation (2) are the corrosion rates without and with inhibitor, respectively.

2.3.2 Electrochemical measurements

Polarization and electrochemical impedance spectroscopy (EIS) were used to investigate the corrosion behavior of 13Cr stainless steel in acid solution at 333 K. The reference electrode, auxiliary electrode and working electrode used the standard chlorinated silver, platinum electrode and 13Cr stainless steel of 1 cm^2 ($10\text{ mm} \times 10\text{ mm}$), respectively. All electrochemical measurements were obtained in acid solution, after reaching a steady open circuit potential. All potentials were measured versus SCE. Potentiodynamic polarization curves were obtained at the scan rate of $1.0\text{ mV}\cdot\text{s}^{-1}$ in the potential range from -300 to $+300\text{ mV}$. EIS measurements were carried out using AC signals of amplitude 5 mV peak-to-peak at the open circuit potential (E_{oc}) in the frequency range 10^5 Hz to 10^{-2} Hz .

2.3.3 Surface analysis of 13Cr stainless steel

The microstructure of the 13Cr stainless steel surface immersed in acid solution was assessed by SEM. The elemental composition and content of corrosion products on the 13Cr stainless steel surface

were obtained using EDS analysis. The microstructure and elemental composition analysis were used to study the corrosion mechanism of 13Cr stainless steel in the organic retarded mud acid solution and the anti-corrosion mechanism of the corrosion inhibitor based on emulsion and Cr coordination on 13Cr stainless steel.

3. RESULTS AND DISCUSSION

3.1 Weight loss measurements

3.1.1 Effect of different acid solution on corrosion of 13Cr stainless steel

The corrosion rates of 13Cr stainless steel in different acid solutions at different temperatures were obtained by weight loss method and graphically showed by Fig. 1.

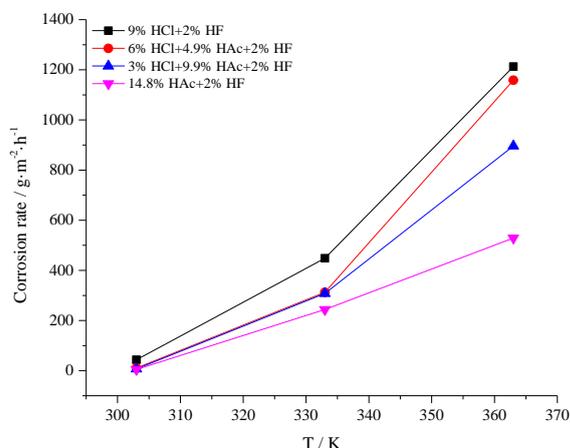


Figure 1. Corrosion rate of 13Cr stainless steel in different acid solutions at different temperatures obtained by weight loss method for 4h

The results showed in Fig. 1 indicated that the corrosion rate decreased with the decreasing concentration of HCl, although the concentration of organic acids increased. The corrosion effect of HAc/HF acid solution on 13Cr stainless steel was weaker than that of HCl/HF acid which has the same acidification capacity. The results provide a theoretical basis for the search of a weaker corrosive acid to replace the conventional HCl/HF acid in oil well acidification construction process.

3.1.2 Effect of inhibitor on corrosion rate

There are many effective measurements to reduce and control corrosion of steel caused by acid solution [26]. Among which, adding corrosion inhibitor is the most simple, effective and commonly used measure in the oil industry [27]. In this paper, the corrosion inhibitor was used to inhibit the corrosion of acid solution on 13Cr stainless steel in order to get a low-corrosive acidification solution. The

corrosion rate of 13Cr stainless steel in different acid solution with different inhibitor at different temperature was obtained by weight loss method and summarized in Tab. 2.

Table 2. Corrosion rate of 13Cr stainless steel and inhibition efficiency of different inhibitor in different solution at 333 K and 363 K obtained by weight loss method for 4h

Temperature	Solution	C_R (g/m ² ·h ⁻¹)	IE (%)
333 K	9% HCl + 2% HF + 1% inhibitor	10.7	97.6
	9% HCl + 2% HF + 1% H ₂ C ₂ O ₄	208.8	53.4
	9% HCl + 2% HF + 1% inhibitor + 1% H ₂ C ₂ O ₄	7.4	98.4
	14.8% HAc + 2% HF + 1% inhibitor	2.9	98.8
	14.8% HAc + 2% HF + 1% H ₂ C ₂ O ₄	12.0	95.0
	14.8% HAc + 2% HF + 1% inhibitor + 1% H ₂ C ₂ O ₄	0.6	99.8
363 K	9% HCl + 2% HF + 1% inhibitor	84.7	93.0
	9% HCl + 2% HF + 1% H ₂ C ₂ O ₄	1175.0	3.1
	9% HCl + 2% HF + 1% inhibitor + 1% H ₂ C ₂ O ₄	83.1	93.2
	14.8% HAc + 2% HF + 1% inhibitor	7.5	98.6
	14.8% HAc + 2% HF + 1% H ₂ C ₂ O ₄	27.0	94.8
	14.8% HAc + 2% HF + 1% inhibitor + 1% H ₂ C ₂ O ₄	3.5	99.3

The results showed in Tab. 2 indicated that the corrosion inhibitor can significantly reduce the corrosion rate of acid solution, and its corrosion inhibition efficiency is more than 97% when used alone in different solutions at 333K and 363K. However, the corrosion rate of 13Cr stainless steel is still higher than the first standard in the evaluation index of acidizing corrosion inhibitor. The addition of H₂C₂O₄ can effectively reduce the corrosion rate of 14.8% HAc + 2% HF solution and the inhibition efficiency can reach to 94% both at 333 K and 363 K. However, its corrosion inhibition ability to HCl/HF solution is very weak. The corrosion rate of 13Cr stainless steel is much less than the first standard in the evaluation index of acidizing corrosion inhibitor in 14.8% HAc + 2% HF solution with mixture of inhibitor and H₂C₂O₄, the inhibition efficiency of inhibitive mixture can reach to 99.8% and 99.3% at 333 K and 363 K, respectively.

3.2 Electrochemical measurements

3.2.1 Open circuit potential measurements

The change of open circuit potential of 13Cr stainless steel in different solutions with time at 333 K is shown in Fig. 2.

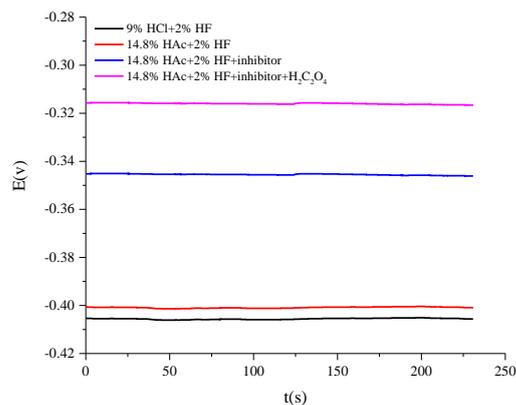


Figure 2. Open circuit potential of 13Cr stainless steel in different acid solution with and without inhibitor at 333 K

It can be seen from Fig. 2 that the open circuit potential of 13Cr stainless steel in 9% HCl + 2% HF and 14.8% HAc + 2% HF solutions was very slow and the corrosion reaction was easy to occur [28]. However, the open circuit potential of 13Cr stainless steel moved to the positive direction after adding different inhibitors compared to that of free 14.8% HAc + 2% HF acid solution, the difficulty of corrosion reaction increased with the increasing of open circuit potential. It took about 240 s to reach the steady state for the 13Cr stainless steel in these solutions, so the working electrode was immersed into solution for at least 5 min before each electrochemical measurement.

3.2.2 Polarization curve analysis

Polarization curves of 13Cr stainless steel different acid solution at 333 K are shown in Fig. 3. The Polarization parameters of 13Cr stainless steel specimens are summarized in Tab. 3.

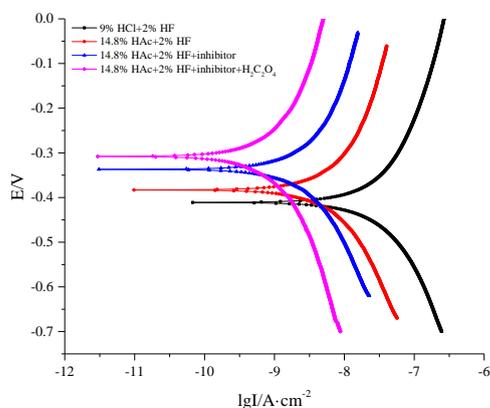


Figure 3. Polarization curves for 13Cr stainless steel in different acid solution with and without inhibitor at 333 K

It can be found that the corrosion current density of 13Cr stainless steel in 14.8 % HAc + 2% HF solution is almost half of that in 9% HCl + 2% HF solution, which proved that even with the same acidity, the corrosion effect of HAc/HF acid solution on 13Cr stainless steel is far less than that of conventional HCl/HF acid. Therefore, it is feasible to choose HAc/HF acid with the same acidizing capacity rather than conventional HCl/HF acid to reduce the corrosion of pipe string in the process of acidizing construction.

Table 3. Polarization parameters of 13Cr stainless steel specimens in different acid solution with and without inhibitor at 333 K

system	E_{corr} (mV)	I_{corr} (mA/cm ²)	IE_p
9% HCl + 2% HF	-434	429.3	-
14.8% HAc + 2% HF	-405	233.6	-
14.8% HAc + 2% HF + inhibitor	-348	2.8	98.9%
14.8% HAc + 2% HF + inhibitor + H ₂ C ₂ O ₄	-314	0.5	99.8%

The results of polarization curve analysis also demonstrated that the corrosion current density of 13Cr stainless steel in the system with inhibitor reduced significantly and the inhibition efficiency was 98.9%, and the inhibition efficiency was further increased to 99.8% by H₂C₂O₄. The corrosion current density of 13Cr stainless steel is only 0.5 mA/cm² in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution at 333 K. According to the literatures [29, 30], it has been reported that if the displacement in E_{corr} in the presence of inhibitor is more than 85 mV, the inhibitor can be classified as a cathode or anode type, on the contrary, if the displacement in E_{corr} in the presence of inhibitor is less than 85 mV, the inhibitor can be classified as a mixed type. Based on this result, it can be speculated that the inhibitor is a mixed inhibitor which can inhibit the cathode and anode reaction of corrosion, but the presence of H₂C₂O₄ enhanced the inhibitive effect of cathode corrosion reaction, and the mixture of inhibitor and H₂C₂O₄ acted as a cathode corrosion inhibitor.

3.2.2 AC Impedance analysis

Fig. 4 and Fig. 5 show that all of the electrochemical impedance spectrums and Bode curves are in the same shapes, which indicated that 13Cr stainless steel exhibits the same electrochemical behavior in these solutions when the corrosion system reaches equilibrium [31, 32]. The impedance parameters and inhibition efficiency obtained by AC Impedance analysis were calculated and listed in Tab. 4.

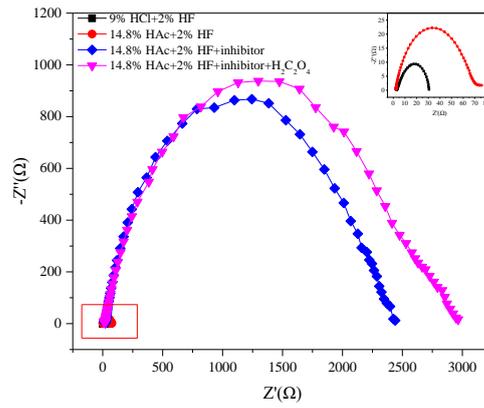


Figure 4. Nyquist curves for 13Cr stainless steel immersed in different acid solution with and without inhibitor at 333 K

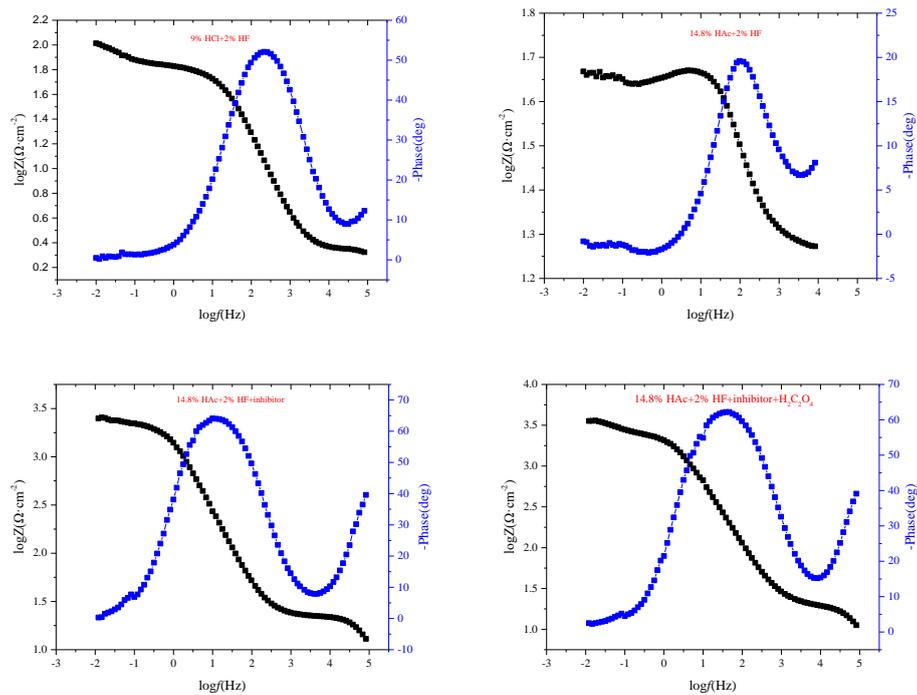


Figure 5. Bode curves for 13Cr stainless steel immersed in different acid solution with and without inhibitor at 333 K

Table 4. EIS parameters of 13Cr stainless steel specimens in different acid solution with and without inhibitor at 333 K

system	R_p ($\Omega \cdot \text{cm}^2$)	f_{max} (Hz)	C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)	IE_p
9% HCl + 2% HF	31	12.3	129.7	-
14.8% HAC + 2% HF	76	17.7	98.7	-
14.8% HAC + 2% HF + inhibitor	2440	33.9	38.1	96.9%
14.8% HAC + 2% HF + inhibitor + $\text{H}_2\text{C}_2\text{O}_4$	2965	39.1	29.4	97.4%

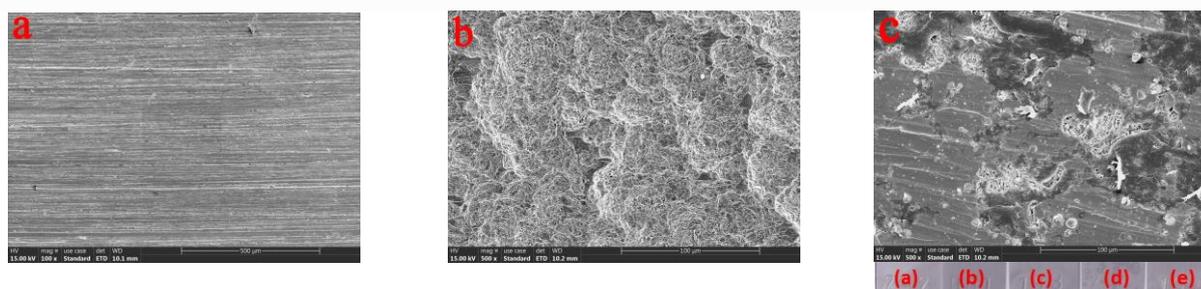
The results of AC Impedance analysis indicated that the corrosion ability of 14.8% HAc + 2% HF solution to 13Cr stainless steel is weaker than that of conventional HCl/HF acid, and the addition of inhibitor and H₂C₂O₄ significantly increased the corrosion resistance of 13Cr stainless steel in 14.8% HAc + 2% HF solution. It also can be seen that these impedance loops are not perfect semicircles, which can be termed as frequency dispersion effect as a result of the roughness and inhomogeneity of the metal electrode surface [33]. It can be seen that the R_p value increases while C_{dl} value decreases with addition of inhibitor. These results suggest that the inhibitor and H₂C₂O₄ can be adsorbed on the surface of the steel to form a protective film, which improves the impedance value and prevents the acid solution from corroding the steel [34, 35].

3.3 Analysis of the 13Cr stainless steel surface and corrosion products

3.3.1 Corrosion morphologies

The microscopic morphologies of the 13Cr stainless steel after immersed in HCl/HF acid solution and HAc/HF acid solution, were revealed by SEM and the results are shown in Fig. 6. As shown in Fig. 6(b) and Fig. 6(c), the 13Cr stainless steel surface appeared very rough due to the formation of uniform corrosion products after immersed in 9% HCl + 2% HF solution, and the formation of uneven dot corrosion products after immersed in 14.8% HAc + 2% HF solution. Fig. 6(d) shows that the 13Cr stainless steel surface appeared many small pitting corrosion after immersed in 14.8% HAc + 2% HF + inhibitor solution, and Fig. 6(e) shows a good corrosion inhibition effect after immersed in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution, the result is attributed to that the inhibitor without H₂C₂O₄ cannot inhibit pitting corrosion of 13Cr stainless by acid solution, this may be because of that the conventional corrosion inhibitor has good adsorption performance on the surface of Fe, but cannot adsorb on the surface of Cr [36].

The HF/HAc acid can be used instead of the HCl/HF acid to reduce the corrosion of 13Cr stainless steel during acidizing of gas wells, and a corrosion inhibitor containing H₂C₂O₄ is also used. Compared with the HCl/HF acid, the HAc/HF acid reduces the effective H⁺ concentration of acid solution. The conventional corrosion inhibitor reduces the corrosion of acid solution to Fe, while the H₂C₂O₄ with Cr form coordination compound to reduce pitting corrosion of 13Cr stainless steel [37].



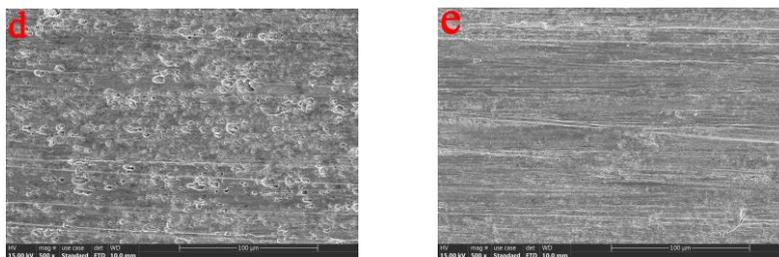


Figure 6. Scanning electron micrographs of 13Cr stainless steel surface after immersing in different acid solution with and without inhibitor at 333 K after 4 h (a: Polished 13Cr stainless steel surface, b: after immersing in 9% HCl + 2% HF solution, c: after immersing in 14.8% HAc + 2% HF solution, d: after immersing in 14.8% HAc + 2% HF + inhibitor solution, e: after immersing in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution)

3.3.2 Component of corrosion products

To further investigate the elements in the layer covering the steel surface, the element distribution of the steel immersed in HCl/HF acid solution and HAc/HF acid solution were determined by EDS, as shown in Fig. 7 and Tab. 5. The surface of the 13Cr stainless steel after immersed in 9% HCl + 2% HF and 14.8% HAc + 2% HF solution has F element, which indicates that steel has been corroded by HF. The O element on surface of the 13Cr stainless steel after immersed in 9% HCl + 2% HF solution (14.53 Wt%) is higher than that immersed in 14.8% HAc + 2% HF solution (7.29 Wt%), and the Cr element on surface of the 13Cr stainless steel after immersed in 14.8% HAc + 2% HF solution (30.15 Wt%) is higher than that immersed in 9% HCl + 2% HF solution (12.64 Wt%), which shows that HCl is more corrosive to 13Cr stainless steel than HAc at the same concentration of H⁺.

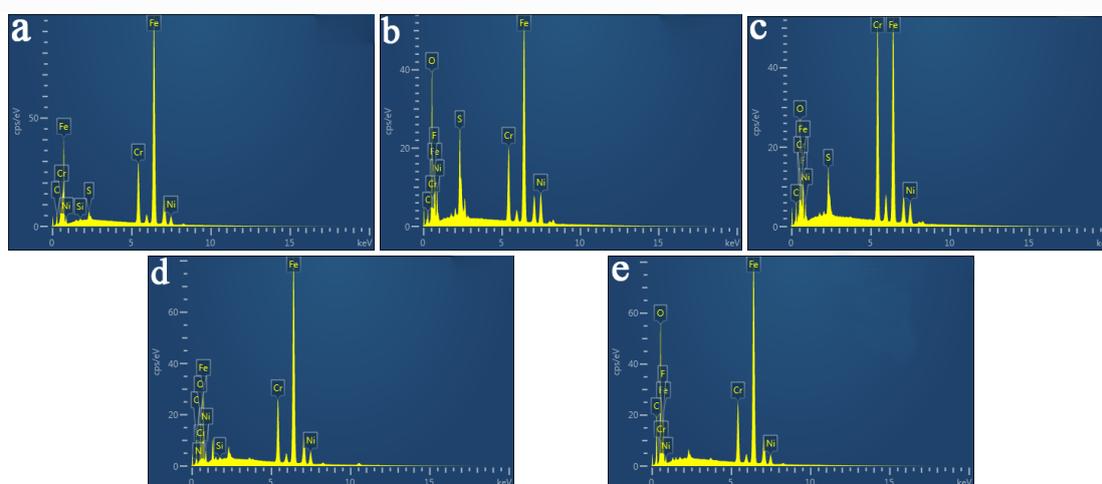


Figure 7. EDS Elemental Analysis of 13Cr Steel Surface after immersing in different acid solution with and without inhibitor at 333 K after 4 h (a: Polished 13Cr stainless steel surface, b: after immersing in 9% HCl + 2% HF solution, c: after immersing in 14.8% HAc + 2% HF solution, d: after immersing in 14.8% HAc + 2% HF + inhibitor solution, e: after immersing in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution)

The N element on surface of the 13Cr stainless steel after immersed in 14.8% HAc + 2% HF + inhibitor solution was observed, indicating that the inhibitor molecules were absorbed on the steel surface to form the protective film.

Table 5. Elemental content of 13Cr stainless steel surface after immersing in different acid solution with and without inhibitor at 333 K after 4 h

		C	N	O	F	Si	S	Cr	Ni	Fe
a	Wt%	0.02	-	-	-	0.18	0.57	14.02	5.23	79.97
	At%	0.11	-	-	-	0.36	0.98	14.84	4.91	78.80
b	Wt%	0.02	-	14.53	0.76	-	4.07	12.64	12.73	55.24
	At%	0.06	-	35.96	1.57	-	5.03	9.63	8.58	39.16
c	Wt%	0.03	-	7.29	2.24	-	1.93	30.15	7.87	50.49
	At%	0.10	-	20.20	5.24	-	2.67	25.73	5.95	40.11
d	Wt%	0.05	0.10	5.19	-	0.19	-	13.77	7.01	73.69
	At%	0.21	0.34	15.84	-	0.32	-	12.95	5.84	64.50
e	Wt%	0.08	-	14.66	1.27	-	-	11.75	4.77	67.46
	At%	0.26	-	36.59	2.67	-	-	9.02	3.24	48.22

Notes: (a) blank of 13Cr stainless steel; (b) after immersing 9% HCl + 2% HF solution; (c) after immersing in 14.8% HAc + 2% HF solution; (d) after immersing in 14.8% HAc + 2% HF + inhibitor solution; (e) after immersing in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution.

Wt%: the relative mass percentage of the elements; At%: the relative atomic number percentage of the elements.

The O element on surface of the 13Cr stainless steel after immersed in 14.8% HAc + 2% HF + inhibitor + H₂C₂O₄ solution (14.66 Wt%) is higher than that immersed in 14.8% HAc + 2% HF + inhibitor solution (5.19 Wt%), indicating that a protective film on the surface of 13Cr stainless steel was formed by the H₂C₂O₄ with Cr to form a coordination compound. The EDS results show that the corrosion of Fe and Cr could be controlled simultaneously for 13Cr stainless steel corrosion prevention.

4. CONCLUSION

(1) The corrosion effect of HAc/HF acid solution on 13Cr stainless steel was weaker than that of HCl/HF acid which has the same acidification capacity, can replace the conventional HCl/HF acid in oil well acidification construction process.

(2) The mixture composed by inhibitor and oxalic acid can effectively reduce the corrosion rate of 13Cr stainless steel in different acid solutions, especially in HAc/HF acid solution, in which the corrosion rate can be reduced to 0.55 g/m²·h and 3.49 g/m²·h at 333 K and 363 K, respectively.

(3) The mixture can inhibit the cathode and anode reaction of corrosion by forming a protective film on the surface to isolate the 13Cr stainless steel surface from the acid solution.

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