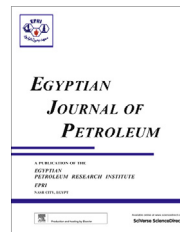




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FULL LENGTH ARTICLE

Isatin compounds as corrosion inhibitors for N80 steel in 15% HCl

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Abstract The inhibition effect of two synthesized isatin compounds namely 1-morpholinomethyl-3-(1-N-dithiooxamide)iminoisatin [MMTOI] and 1-diphenylaminomethyl-3-(1-N-dithiooxamide)iminoisatin [PAMTOI] on the corrosion inhibition of N80 steel in 15% HCl solution was studied by polarization, EIS and weight loss measurements. It was found that both the inhibitors were effective and their inhibition efficiency was significantly increased with increasing concentration. Polarization curves revealed that the used inhibitors represent mixed-type inhibitors. Adsorption of these inhibitors led to a reduction in the double layer capacitance and an increase in the charge transfer resistance, and was found also to obey Langmuir isotherm.

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1. Introduction

N80 steel is widely used as a construction material for pipe work in the oil and gas production such as down hole tubular, flow lines and transmission pipelines in petroleum industry. Mineral acids particularly hydrochloric acid are frequently used in industrial processes involving acid cleaning, acid pickling, acid descaling, and oil well acidizing [1–3]. In petroleum industry, 15% HCl is commonly used for acidizing treatment because it leaves no insoluble products after the treatment

and is found to be commercially available and cheap but adversely at the same time it severely attacks the metal casings and tubular of oil well during the acidizing process. Therefore, protective measures should be required to prevent the metal loss due to corrosion by using chemical and other means. Due to aggressiveness of acids, inhibitors are often used to reduce the rate of dissolution of metals. Most of the well-known acid inhibitors are organic compounds containing nitrogen, oxygen and/or sulfur atoms, heterocyclic compounds and pi-electrons [4–7]. The polar function is usually regarded as the reaction center for the establishment of the adsorption process [8]. It is generally accepted that organic molecules inhibit corrosion via adsorption at the metal–solution interface [9,10], resulting adsorption layer function as a barrier and isolating the metal from the corrosion [11]. Some Mannich bases have been reported as efficient corrosion inhibitors [12,13] and the literature available to date about the Mannich bases used as corrosion inhibitors is limited. Any research work has not been encountered in the literature to date about the application of

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the synthesized Mannich bases namely 1-morpholinomethyl-3-(1-N-dithiooxamide)iminoisatin [MMTOI] and 1-diphenylaminomethyl-3-(1-N-dithiooxamide)iminoisatin [PAMTOI] as corrosion inhibitors for N80 steel corrosion in 15% HCl solutions.

With a view to find out better corrosion inhibitors, isatin compounds namely 1-morpholinomethyl-3-(1-N-dithiooxamide)iminoisatin [MMTOI] and 1-diphenylaminomethyl-3-(1-N-dithiooxamide)iminoisatin [PAMTOI] have been synthesized and their inhibitive properties for oil-well tubular steel (N80) in 15% HCl solution have been studied.

2. Materials and methods

2.1. Materials

The working electrode and specimens for weight loss experiments were prepared from oil-well N80 steel sheets having the following percentage by weight (%wt) composition:

C: 0.31, Mn: 0.92, Si: 0.19, P: 0.01, S: 0.008, Cr: 0.20, Fe: Remainder

2.2. Weight loss measurements

The specimens for the weight loss experiments were of the size 3 cm × 3 cm × 0.1 cm and for electrochemical studies the size of the electrodes was 1 cm × 1 cm × 0.1 cm with a 4 cm long tag for electrochemical contact. Both sides of the specimens were exposed for both the techniques. The specimens were mechanically polished successively with 1/0, 2/0, 3/0 and 4/0 grade emery papers. After polishing with the paper of each grade, the surface was thoroughly washed with soap, running tap water, distilled water and finally degreased with acetone. The samples were dried and stored in a vacuum desiccator before immersing in the test solution. For weight loss experiments 300 mL of 15% hydrochloric acid was taken in 500 mL glass beakers with lids. The inhibition efficiencies (%IE) were evaluated after a pre-optimized time interval of 6 h using 20, 50, 100, 150 and 200 ppm of inhibitors. The specimens were removed from the electrolyte, washed thoroughly with distilled water, dried and weighed. The inhibition efficiencies were evaluated using the following formula:

$$\%IE = [(W - W_i)/W] \times 100 \quad (1)$$

where W is the weight loss in the absence of inhibitor; and W_i the weight loss in the presence of inhibitor.

2.3. Electrochemical polarization studies

The electrochemical experiments were carried out in a three necked glass assembly containing 150 mL of the electrolyte with different concentrations of inhibitors (from 10 ppm to 150 ppm by weight) dissolved in it. The potentiodynamic polarization studies were carried out with N80 steel strips having an exposed area of 1 cm². A conventional three electrode cell consisting of N80 steel as working electrode, platinum as counter electrode and a saturated calomel electrode as reference electrode were used. Polarization studies were carried

out using VoltaLab 10 electrochemical analyzer and data was analyzed using Voltamaster 4.0 software. The potential sweep rate was 10 mV s⁻¹. All experiments were performed at 25 ± 0.2 °C in an electronically controlled air thermostat. For calculating %IE by electrochemical polarization method, the following formula was used:

$$\%IE = [(I_0 - I_{inh})/I_0] \times 100 \quad (2)$$

where I_0 is the corrosion current in the absence of inhibitor and I_{inh} is the corrosion current in the presence of inhibitor.

2.4. AC Impedance studies

AC impedance studies were carried out in a three electrode cell assembly using computer controlled VoltaLab 10 electrochemical analyzer, as well as N80 steel as the working electrode, platinum as counter electrode and saturated calomel as reference electrode. The data were analyzed using Voltamaster 4.0 software. The electrochemical impedance spectra (EIS) were acquired in the frequency range from 10 kHz to 1 mHz at the rest potential by applying 5 mV sine wave AC voltage. The charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were determined from Nyquist plots. The inhibition efficiencies were calculated from charge transfer resistance values by using the following formula:

$$\%IE = [(R_{ct(inh)} - R_{ct})/R_{ct(inh)}] \times 100 \quad (3)$$

where R_{ct} is the charge transfer resistance in the absence of inhibitor; and $R_{ct(inh)}$ is the charge transfer resistance in the presence of inhibitor.

2.5. Synthesis of inhibitors

The Isatin Mannich bases namely 1-morpholinomethyl-3-(1-N-dithiooxamide)iminoisatin [MMTOI] and 1-diphenylaminomethyl-3-(1-N-dithiooxamide)iminoisatin [PAMTOI] were synthesized by the reported method [14]. Isatin and dithiooxamide in 1:1 M ratio were refluxed in ethanol for 8 h, cooled and the precipitate was filtered. This product was subsequently treated with formaldehyde and morpholine or diphenylamine to get the desired product. The name and molecular structure of studied compounds were given as Scheme 1.

3. Results

3.1. Weight loss study

Weight loss studies were performed in accordance with ASTM method. Test were conducted in 15% HCl solution for 6 h at 298 K with different concentrations of inhibitors (20–200 ppm), temperatures (298–333 K) and exposure period (6–24 h).

3.1.1. Effect of concentration

Both the inhibitors were tested for 6 h exposure period at different concentrations and their corresponding weight loss data are presented in Table 1. Both compounds inhibit the corrosion. The compounds PAMTOI and MMTOI have the maximum of 91.23% and 84.33% inhibition efficiency

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