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Advances in Applied Science Research, 2016, 7(2):92-102



Recycling of the used cooking oils as corrosion inhibitors for carbon steel in hydrochloric acid corrosive medium

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ABSTRACT

Alleviation of environmental pollution caused by accumulation of used cooking oils (UCO) and corrosion of steel alloys. In this respect, the used cooking oil (Triolein) was collected, Purified, then reacted with ethanol amine (EA) in the presence of sodium acetate as a catalyst, the obtained Octadec-9-enoic acid (2-hydroxy-ethyl)-amide, (Octa Dec Hydroxy Amide ,ODHA) as nonionic surfactant which separated and characterized by FT-IR, ¹HNMR was evaluated as corrosion inhibitors for steel in 1 M HCl aqueous corrosive medium by electrochemical methods(open circuit potential, potentiodynamic polarization), and chemical methods(weight loss) at different concentration and temperatures It was found that the inhibition efficiency increased with inhibitor concentration and decrease with temperature. Adsorption was found to follow the Langmuir isotherm. The obtained polarization curves indicate that these compounds act as mixed-type inhibitors. Inhibition efficiency obtained from both polarization and weight loss measurements are in a good agreement.

Keywords: Corrosion; Inhibition; Steel; surfactant, polarization, oil recycling

INTRODUCTION

The highly corrosive nature of aqueous acid media on most metal results on highly economical loose (i.e., it affects the operation of equipment and results on highly risk on the safety condition), therefore the inhibition of the corrosion of steel in aqueous solutions gains tremendous technological importance due to the increased industrial applications of this material. Use of inhibitor is one of the most practical methods for protection of steel against corrosion in acidic media. Recently, the inhibition of the corrosion of steel in acidic aqueous solutions by different organic compounds (called adsorption inhibitors) have been widely studied [1-5]. On the other hand, ionic surfactants [6-10] were also reported as good inhibitors for steel corrosion in acidic media. However, very few studies have been devoted to nonionic surfactants as corrosion inhibitors for metals [10-12]. Surfactant inhibitors show many advantages such as high inhibition efficiency, low price, low toxicity and easy production [10-13]. Adsorption of the surfactant on the metal surface can markedly change the corrosion-resisting property of the metal , therefore the study of the relationship between the adsorption and corrosion inhibition is of great importance. Surfactant adsorption could be attributed to electrostatic interaction, van der Waals interaction, hydrogen bonding and/or salvation and desolvation of adsorbate and adsorbent species [14, 15].

Oil recycling is increasingly being carried out to produce Oil fuel. In the UK, waste cooking oil collection is governed by the environment agency. All waste cooking oil collections need to be carried out by a company

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registered as a waste carrier by the environment agency [16]. Used cooking oil (UCO) is a domestic waste generated daily by food industries, restaurants, and households. It is estimated that in Europe 5 kg of UCO are generated per inhabitant, totaling 2.5 million metric tons per year. Recovering UCO for the production of biodiesel offers away of minimizing and avoiding this waste and related pollution [17]. there is no reported works on the use of expired or used cooking oils as corrosion inhibitors, so that the present work is the first trial to use the modified cooking oils as corrosion inhibitor for steel in hydrochloric acid corrosive medium. The aim of the present work is to evaluate the inhibition action of one of the nonionic surfactant prepared from the used cooking oil (Triolein) after recovery via reacting the purified oil with ethanol amine in the presence of sodium acetate as catalyst, the obtained Octadec-9-enoic acid (2-hydroxy-ethyl)-amide, (Octa Dec Hydroxy amide, ODHA) were evaluated as corrosion inhibitor for steel alloy by weight loss and electrochemical techniques (open circuit potential and potentiodynamic polarization measurements) at different concentration and temperature.

MATERIALS AND METHODS

2.1. Materials

Used cooking oil waste (**UCO**) was collected from the fried food restaurants. Ethanolamine (EA) and manganese acetate were purchased from Aldrich Chemical Co., Ltd. (UK), were used as catalysts for recycling of **UCO**. Steel sample with the following composition (wt. %): 0.18C, 0.6Mn, 0.05 S, 0.04 P, 0.1 Si and balance Fe. Test materials were abraded with different emery papers up to 1000 grade, cleaned with acetone, washed with double distilled water and properly dried prior to exposure. The aggressive solution (1 M HCl) was prepared by appropriate dilution of analytical grade 36% HCl with double distilled water.

2.1.1. Purification of the used cooking oil (UCO).

The collected UCO was left for a while to allow the fine particles to settle out, and then pour off the oil, mixing the oil with equal volume of aqueous saturated sodium chloride solution, boiling for 15 minutes, left for 1 hour in separating funnel, after separation the oil layer was dried by heating for 20 minutes, left to cool, and dissolved in chloroform, the reaction mixture was refluxed for 30 minutes at 80 $^{\circ}$ C, the hot mixture was filtered off and the chloroform was evaporated using evaporator to leave the purified cooking oil.

2.1.2. Synthesis

Elemental and spectroscopic analyses were carried out in the Micro Analytical Center of Cairo University. The fine chemicals were purchased from Aldrich Co. and the organic solvents were of spectroscopic grades. The reactions were monitored using TLC and the resultant compounds were crystallized and then extra purified using a column chromatography technique.

Synthesis of Octadec-9-enoic acid (2-hydroxy-ethyl)-amide, (Octa Dec Hydroxyl amide, ODHA)

Triolein (I) (0.1 mol) and an equimolar ratio of ethanol amine were refluxed in ethanolic solution of sodium ethoxide for 3 hours at 100 C°. The reaction mixture was cooled, filtered off to remove glycerol and unreacted materials the product residue is a white yellow oily viscous product, (III). This compound was extra purified using column chromatography, silica gel, eluting with ethyl acetate/hexanes (20/80).

III: (Y: 69%); Mol. F.: C20H39NO₂; Mol. Wt.: 325.53; m/z (FABMS): 326.3; Elemental analysis: (Calculated %) C:

73.79, H: 12.08, N: 4.30, O: 9.83 (*Found %*) C: 73.83, H: 13.00, N: 4.25, O: 9.81; ¹*HNMR* (400 MHz, DMSO-d6):0.89 (t, 3H-CH₃), 1.11-1.37 (m, 20H-10CH₂), 1.54 (p, 2H-CH₂), 1.91 (q, 4H-2CH₂), 2.08 (t, 2H-CH₂-CO), 3.29 (t, 2H-CH₂-NH), 3.76 (t, 2H-CH₂-CO), 4.16 (s, OH), 5.42 (q, 2H-CH=), 6.93 (s, NH). ¹³*CNMR* (400 MHz, DMSO): 11.90 (CH₃), 22.19 (CH₂), 25.63 (CH₂), 28.40 (CH₂), 29.45 (CH₂), 30.14 (6CH₂), 31.80 (CH₂), 33.70 (2CH₂-CH=), 36.44 (CH₂-CO), 41.32 (CH₂-NH), 61.14 (CH₂OH), 130.50 (2CH=), 172.66 (C=O).



Fig. 1. Chemical structure of (ODHA) compound (III) and the Scheme of the synthesis

2.2. Weight loss technique

Coupons of steel of $2 \times 2 \times 0.1$ cm dimensions were used as test specimens, the specimens were polished by 410 and 610 emery papers, respectively, degreased with acetone, washed with distilled water and finally dried using two filter papers. The described treatment was carried out immediately before each measurement. The specimen of the given metal was immersed in 100 ml of the test solution (i.e., 1 M HCl) with different inhibitor concentration (**ODHA**), samples maintained in test solution up to 10 hours. Experiments were carried out under different temperature 30, 40, 50, and 60° C.

2.3. Open circuit potential

The potential of steel electrode was measured against saturated calomel electrode (SCE) in 1 M HCl solution in absence and presence of different concentrations of the inhibitor at 30 °C. All measurements were carried out using Multi-tester until the steady-state potentials are reached. Temperature effect was also studied using concentration of 400 ppm of inhibitor.

2. 4. Polarization measurements

Platinum electrode and a saturated calomel (SCE) electrode were used as auxiliary and reference electrodes, respectively. The samples of steel, 1 cm², were first immersed into the solution for 30 minutes to establish a steady state open circuit potential. The effect of inhibitor on steel corrosion was determined by measuring corrosion rate in different concentration of ODHA. For the evaluation of inhibitor concentration effects on inhibition efficiency (the protection of corrosion) experiments were carried out in 1 M HCl in the absence and presence of various concentrations of inhibitors. The cell was left open to air at room temperature (30 $^{\circ}$ C). All potential values were reported in millivolt (SCE). The electrochemical polarization was also done after 30 min. immersion in inhibitor solution.

RESULTS AND DISCUSSION

3.1. Weight loss measurements

3.1.1. Effect of inhibitor concentration

The weight loss results of mild steel in 1M HCl in the absence and presence of various concentrations of the used (Octa Dec Hydroxy Amide ,ODHA) inhibitor are summarized in Table (1). The I.E (%) was calculated using the following equation:

I.E (%) =
$$(W2 - W1/W1) \times 100$$

(1)

Where W1 and W2 are weight loss of mild steel in the presence and absence of the inhibitor respectively. The inhibition efficiency increased with increase in the concentration of the inhibitor.

Table 1. Degree of surface coverage (θ) and percentage inhibition efficiency (%IE) of the used inhibitor in 1 M HCl at 303 K, obtained from weight loss measurements

Conc.(ppm)	Wt. loss mg.cm ⁻²	Corrosion rate mg.cm ² hr ⁻¹	θ	%IE
BLANK	63.46	9.06	0	0
100	12.5	1.7	0.81	81
200	9.5	1.3	0.856	85.6
300	8.1	1.1	0.878	87.8
400	6.3	0.9	0.90	90

3.1.2. Effect of temperature

Weight loss measurements were performed at 303,313,323,333K, to elucidate the mechanism of inhibition and to determine the kinetic parameters of the corrosion process, the effect of temperature on the corrosion inhibition efficiency of mild steel in the presence of the inhibitor is graphically represented in Figure (2).



Fig.2. Effect of temperature on I.E for mild steel in 1M HCl in the presence of different concentrations of the used inhibitor (ODHA)

The inhibition efficiency decreased with increase in temperature, leading to the conclusion that the protective film of these compounds formed on the mild steel surface is less stable at higher temperature; which may be due to the desorption of some adsorbed molecules from the surface of the mild steel at higher temperature due to which greater area of the metal is exposed to the acidic environment.

The apparent activation energy (E_a) of metal corrosion in acid media can be calculated from the Arrhenius equation [18]:

$$\ln C R = (E_a/RT) + A$$

Where E_a is the apparent activation energy for the corrosion of mild steel, R is the universal gas constant, A is Arrhenius pre exponential factor and T is the absolute temperature. Figure(3) depicts the plot of log CR vs. 1/T and the values of E_a obtained from the slope of the plot are given in Table(2).

(2)

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Table. 2. Values of activation parameters for mild steel in 1M HCl in the absence and presence of different concentrations of the inhibitor (ODHA)

Inhibitor concentration	E _a	ΔH_a	$-\Delta S_a$
(ppm)	(kJmol ⁻¹)	(kJmol ⁻¹)	(J k ⁻¹ mol ⁻¹)
blank	41.4	41.1	154.3
100	52.7	49.4	155.8
200	56.2	53.1	152.8
300	57.4	57.5	147.7
400	58.4	59.5	140.4

The higher value of activation energy (E_a) in the presence of inhibitor than in its absence is attributed to its physical adsorption, its chemisorption is pronounced in the opposite case [19].



Fig.3. Arrhenius plot for mild steel in 1M HCl solution in the absence and presence of different concentrations of the inhibitor (ODHA)

In the present study the higher value of E_a for mild steel in presence of (ODHA) inhibitor compared to that in its absence is attributed to its physical adsorption. The increase in E_a can be attributed to an appreciable decrease in the adsorption of the inhibitor on the mild steel surface with increase in temperature and a corresponding increase in corrosion rates occurs due to the fact that greater area of metal is exposed to acid environment [20]. An alternative form of Arrhenius equation is the transition state equation [18]:

$$CR = (RT/Nh) \exp (\Delta S_a/R) \exp (-\Delta H_a/RT)$$

where, h is the Plank's constant, N is the Avogadro's number, ΔS_a is the entropy of activation, and ΔH_a is the enthalpy of activation. A plot of log (CR/T) vs. 1/T gave a straight line as shown in Figure(4) with a slope of (- ΔH /2.303R) and an intercept of [log(R/Nh) + (ΔS /R)], from which the values of ΔH_a and ΔS_a were calculated and listed in Table 2.

(3)



Fig.4. Transition state plot for mild steel in 1M HCl solution in the absence and presence of different concentrations of the inhibitor (ODHA)

The positive values of ΔH_a for corrosion of mild steel in the presence and absence of the inhibitor reflect the endothermic nature of metal dissolution process. The increase in ΔH_a with increase in the concentration of the inhibitor for mild steel corrosion reveals that decrease in mild steel corrosion rate is mainly controlled by kinetic parameters of activation [19]. The entropy of activation values are less negative for inhibited solutions than that for the uninhibited solutions. This suggests that an increase in randomness occurred while moving from reactants to the activated complex [19].

3.1.3. Adsorption Isotherm and thermodynamic parameters

In order to understand the mechanism of corrosion inhibition, the adsorption behavior of the organic adsorbate on the metal surface has to be known. The degree of surface coverage (Θ) for different concentration of the inhibitor (C_i) has been evaluated. The data were tested graphically by fitting to various isotherms. A straight line with correlation coefficient nearly equal to 1.0 was obtained on plotting C_i/Θ against C_i as shown in Figure(5) suggesting, Adsorption of the compound on the mild steel surface followed Langmuir adsorption isotherm model at all studied temperatures.



Fig.5. Langmuir adsorption isotherm for mild steel in 1M HCl solution at various temperatures

According to this isotherm, the surface coverage (Θ) is related [21] to inhibitor concentration C_{inhi} by equation (4)

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(5)

$$C_{inhi} / \Theta = 1 / K_{ads} + C$$
(4)

where, K_{ads} is equilibrium constant of the equilibrium adsorption process. This isotherm assumes that adsorbed molecule occupies only one site and it does not interact with other adsorbed species.

The K_{ads} values can be calculated from the intercept lines on the C_{inhi} / Θ axis. This is related to the standard free energy of adsorption (ΔG_{ads}) by equation (5):

$$\Delta G_{ads} = -RT \ln(55.5 \text{ K}_{ads})$$

where, R is the gas constant and T is the absolute temperature. The constant value of 55.5 is the concentration of water in solution in mol/L. The values of ΔG_a for the inhibitor on the surface of mild steel are given in Table 3.

The negative value of ΔG_a indicated spontaneous adsorption of the inhibitor on the mild steel surface. Generally, the magnitude of ΔG_a around -20kJ/mol or less negative indicates electrostatic interactions between inhibitor and the charged metal surface (i.e., physisorption). Those around -40kJ/mol or more negative are indicative of charge sharing or transferring from organic species to the metal surface to form a coordinate type of metal bond (i.e., chemisorptions). In the present work, the calculated values ΔG° at 303K for mild steel is -41.5 kJ/mol, which indicate that adsorption of the inhibitor on the mild steel surface involves both physical and chemical process [22]. But the inhibition efficiency decreased with increasing temperature indicating inhibitor adsorbed predominantly physically on the surface of mild steel. The enthalpy of adsorption (ΔH_{ads}) can be calculated from the rearranged Gibbs-Helmholtz equation:

$$\Delta G_{ads}/T = (\Delta H_{ads}/T) + K \tag{6}$$

The variation of $\Delta G_{ads}/T$ with 1/T gave a straight line with a slope of ΔH_{ads} as shown in Figure(6). The entropy of adsorption ΔS_{ads} was calculated using the following thermodynamic equation:

$$\Delta S_{ads} = (\Delta H_{ads} - \Delta G_{ads})/T$$
(7)

The calculated values of heat of adsorption and entropy of adsorption are listed in Table 3.



Fig.6. ΔG_{ads} /T versus 1/T plot for mild steel in 1M HCl solution

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Temperature	ΔG_{ads}	ΔH_{ads}	ΔS_{ads}
(K)	(kJmol ⁻¹)	(kJmol ⁻¹)	(J k ⁻¹ mol ⁻¹)
303	-35.7	- 9.3	86.3
313	-36.5	- 9.3	85.9
323	-37.4	- 9.3	86.3
333	-38.3	- 9.3	86.2

Table. 3. Values of thermodynamic parameters for the adsorption of the inhibitor in 1M HCl on the mild steel at different temperatures

It is well known fact that adsorption is an exothermic phenomenon accompanied by a decrease in entropy [22, 23]. In aqueous solutions the adsorption of the organic molecule is generally accompanied by desorption of water molecules [23].

Inhibitor $_{(sol)}$ + x H₂O (ads) Inhibitor $_{(ads)}$ + x H₂O $_{(sol)}$

The negative sign of ΔH_{ads} indicated the exothermic process of adsorption of the inhibitor on mild steel surface in HCl. The positive value of ΔS_{ads} in the presence of inhibitor can be attributed to the increase in the solvent entropy and more positive desorption entropy. It is also interpreted that the increase of disorderness is due to more water molecules which can be desorbed from the metal surface by one inhibitor molecule. Therefore it is revealed that decrease in the enthalpy is the driving force for the adsorption of the inhibitor on the surface of mild steel [24].

3.1.4. Corrosion Inhibition Mechanism

In hydrochloric acid medium, the metal surface is negatively charged due to the specifically adsorbed chloride ions on the metal surface.

 $Fe + Cl^{-} \rightarrow (FeCl^{-})_{ads}$

In acidic solution, the oxygen, nitrogen atom, and amide group of the ODAH inhibitor can be protonated easily, due to high electron density on it, leading to positively charged inhibitor species. The adsorption can occur via electrostatic interaction between positively charged inhibitor molecule and negatively charged metal surface **[25,26]** leading to physisorpton of the inhibitor molecules. Further, co-ordinate bond may be formed between unshared epairs of unprotonated nitrogen atom of the inhibitor and vacant d-orbitals of metal surface atoms. Additionally inhibitor molecule may be chemically adsorbed due to interaction of amide groups of the inhibitor molecules with vacant d-orbitals of metal **[26]**.

3.2. Open circuit potential measurements

The potential of mild steel electrodes immersed in 1 M HCl solution was measured as a function of immersion time in the absence and presence of ODHA inhibitors derived from used cooking oil,UCO, as shown in Figure(7) It is clear that the potential of mild steel electrode immersed in 1MHCl solution (blank curve) tends towards more negative potential firstly, giving rise to short step. This behavior was reported by another investigators **[25-29]** which represents the breakdown of the pre-immersion air formed oxide film presents on the surface according to the following equation:

 $Fe_2O_3 \ + \ 6HCl \quad \rightarrow \ 2FeCl_3 \ + \ 3H_2O$

This is followed by the growth of a new oxide film inside the solution, so that the potential was shifted again to more noble direction until steady state potential is established. Addition of inhibitor molecules to the aggressive medium produces a negative shift in the open circuit potential due to the retardation of the cathodic reaction.



Fig. 7. Potential-time curves for carbon steel immersed in 1M HCl solution in the absence and presence of (Octa Dec Hydroxy Amide, ODHA)

3.3. Potentiodynamic Polarization measurements

Polarization curves for mild steel in 1M hydrochloric acid without and with addition of different concentration of the inhibitor are shown in Figure(8). The values of electrochemical parameters associated with polarization measurements, such as corrosion potential (E_{corr}), corrosion current densities (i_{corr}), tafel slopes ($\beta a, \beta c$) and calculated inhibition efficiency (IE %) are listed in Table (4). The inhibition efficiency is given by the following equation [23-30]:

IE (%) =
$$1 - (I_{inh}/I_{uninh}) \ge 100$$

(8)

Where: I_{uninh} and I_{inh} are the corrosion current densities in the absence and presence of inhibitor respectively.

Table .4. Electrochemical parameters of mild steel in 1M HCl solution at 303K in absence and presence (Octa Dec Hydroxy Amide, ODHA) inhibitor

inhibitor concentration (ppm)	<i>i</i> _{corr} mA/cm ²	E _{corr} - mV	Ba mV/decade	Bc mV/decade	I.E(%)	Θ
Blank	282	549	87	126	-	-
100	71	479	82	118	74.8	0.748
200	60	465	80	116	78.7	0.787
300	34	458	81	114	87.9	0.879
400	30	449	78	111	89.4	0.894



l = *blank*, *2* = *100 ppm*, *3* = *200 ppm*, *4* = *300 ppm*, *5* = *400 ppm*

Fig.8. Potentiodynamic polarisation curves of mild steel in 1M HCl solution at 30 mC containing various concentrations of ODHA inhibitor

Corrosion current density decreased noticeably with increase in inhibitor concentration indicating, the increased inhibition efficiency with the increase in the concentration of the inhibitor. Generally, in acidic solution the anodic process of corrosion is the passage of metal ions from the solid metal to the solution, and the principal cathodic process is the discharge of hydrogen ions to produce hydrogen molecules or reduction of oxygen [25-30]. In the present study, the corrosion potential values slightly shifted to more positive value indicating addition of inhibitor molecule reduces both anodic dissolution and cathodic reduction; therefore, (Octa Dec Hydroxy Amide ,ODHA) could be classified as a mixed type inhibitor, but the anodic effect is more pronounced. The anodic and cathodic Tafel constants did not change significantly with increase in inhibitor concentration suggested that presence of inhibitor does not alter the reaction mechanism and inhibition effect has occurred due to simple blocking of the active sites [29,30], thereby reducing available surface area of the corroding metal.

CONCLUSION

1- Results obtained from the experimental data shows that the used cooking oils, UCO, can be recycled after purification via reaction with ethanol amine in the presence of catalyst to give Octadec-9-enoic acid (2-hydroxy-ethyl)-amide, (Octa Dec Hydroxyl Amide ,ODHA).

2- The product of the recycling process (ODHA) acts as a good inhibitor for the corrosion of mild steel in 1 M HCl.

3- The inhibition efficiency of ODHA decreased with temperature, which leads to an increase in activation energy of corrosion process.

4- The adsorption of ODHA inhibitor follows Langmuir's adsorption isotherm and The adsorption process is spontaneous and exothermic, accompanied by an increase of entropy.

5- Addition of inhibitor molecules to the aggressive medium produces a negative shift in the open circuit potential due to the retardation of the cathodic reaction.

6- Potentiodynamic polarization curves reveals that ODHA, inhibitor which prepared from waste cooking oil is a mixed-type but predominantly anodic inhibitor.

7- The results obtained from different experimental studies are in good agreement

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