USO DE EXTRATO DE CEBOLA SELVAGEM ECOLÓGICA (*URGINEA MARITIMA*) COMO INIBIDOR DE CORROSÃO PARA AÇO CARBONO EM ÁCIDO CLORÍDRICO

THE USE OF ECO-FRIENDLY WILD ONION EXTRACT (*URGINEA MARITIMA*) AS CORROSION INHIBITOR FOR CARBON STEEL IN HYDROCHLORIC ACID

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RESUMO

Embora vários compostos sintéticos tenham uma boa ação como anticorrosivo, eles não são baratos e são tóxicos para o meio ambiente e para os seres humanos. No entanto, no aço carbono (aço-C) existe uma preocupação vital que é o sério problema de corrosão devido à exposição a ambientes de acidez agressiva, ou seja, descalcificação, soluções de acidez de poços de petróleo e decapagem. Portanto, este estudo teve como objetivo avaliar o efeito de inibição do extrato de cebola selvagem (WO) como um inibidor ecológico no comportamento da corrosão do aço-C em HCl 0,5 M através da abordagem convencional de perda de peso. Várias concentrações (0%, 0,5%, 1%, 1,5%, 2% e 2,5%) de inibidor em vários períodos de imersão (2, 4 e 8 h) e em diferentes temperaturas (25 °C, 35 °C e 45 °C) foram investigados quanto à inibição da corrosão do aço-C em meios corrosivos. Na presença e ausência do inibidor, a taxa de corrosão (CR) foi investigada como afetada pela temperatura. A concentração do inibidor e a temperatura controlaram a eficiência da inibição (%E) do inibidor. Na existência de extrato de cebola selvagem, a eficiência ideal de inibição para o aço-C foi de 98,95%, 88,99% e 86,79% nas concentrações de inibidor de 2,5% nas temperaturas anteriores, respectivamente. Observou-se que a adsorção era espontânea e física, conforme comprovado pelo valor de adsorção da energia livre de ΔG°_{ads} (13,5 kJ/mol), além de também ser isotérmica de adsorção de Langmuir. Os dados de cobertura da superfície θ e da densidade de corrente de corrosão I_{corr} confirmaram o resultado anterior em que a inibição ocorre devido à adsorção da natureza física dos componentes do aditivo na superfície do aço-C.

Palavras-chave: Cebola selvagem; Aço-C; Inibição de corrosão; Inibição termodinâmica.

ABSTRACT

Even though various synthetic compounds have a well action as anticorrosive, they are not cheap and are toxic to both environment and humans. Nevertheless, in C-steel, there is a vital concern, which is serious corrosion issues happen through exposure to environments of aggressive acidity i.e., descaling, oil well solutions of acidity, and pickling. Hence, this study aimed to evaluate the inhibition effect of wild onion (WO) extract as an eco-friendly inhibitor on the behavior of corrosion for C-steel in 0.5 M HCl through the conventional weight loss approach. Various concentrations (0%, 0.5%, 1%, 1.5%, 2%, and 2.5%) of inhibitor in various times of immersion (2, 4, and 8 h) and at different temperatures (25°C, 35°C and 45°C) were investigated for their C-steel corrosion inhibition in corrosive media. In the presence and absence of the inhibitor, the corrosion rate (CR) was investigated as affected by temperature. The concentration of the inhibitor and temperature-controlled the inhibition efficiency %E of the inhibitor. At the existence of wild onion extract, the ideal efficiency of inhibition for C-steel was 98.95%, 88.99%, and 86.79% at 2.5% inhibitor concentrations at the preceding temperatures, respectively. It was noticed that adsorption was spontaneous and physical as proved through adsorption value of free energy ΔG°_{ads} (-13.5 kJ/mol) and also fitted Langmuir adsorption isotherm. The surface coverage θ and corrosion current density I_{corr} data confirmed the previous result where inhibition is due to the adsorption of physical nature for the components of the additive on the C-steel surface.

Keywords: Wild onion; C-steel; Corrosion inhibition; Inhibition thermodynamic.

1. INTRODUCTION:

In various kinds of industrial applications, carbon steel (C-steel) has a significant role due to its availability and low price (El-Lateef *et al.*, 2016). Nevertheless, in C-steel, there is a vital concern, which is serious corrosion issue happens through exposure to environments of aggressive acidity, i.e., descaling, oil well solutions of acidity, and pickling (Banerjee *et al.*, 2014). The temperature effect is of theoretical and practical significance on chemical reactions. As of most reactions, with elevated temperature, the corrosion rate of steel and Fe increases, particularly at medium where H evolution along with corrosion is accompanied, i.e., through steel corrosion in acidic solution (Hamdy and El-Gendy, 2013). As one of the most vital and significant strategies, inhibitors of corrosion have been practiced for C-steel anti-corrosion, especially in acidic solution, to prevent metal dissolution and acid consumption (Khadom *et al.*, 2009). Although various synthetic compounds have a great action as anticorrosive, they are not cheap and are toxic to both environment and humans (Garai *et al.*, 2012). Toxicity might show manifestation either through compound synthesis or through applications, which makes researchers concentrate on using substances occurring naturally to obtain inhibitors that are non-hazardous and cheap (Ahmed and Quraishi, 2013). The compound to behave as an inhibitor for corrosion, requirements should be available; as far as the behavior of chemical and its structure, an inorganic compound readily oxidizes the metal by forming on the surface a passive layer. Also, an organic molecule should possess inhibitor characters to behave as a corrosion inhibitor; the double bonds, molecules with a large structure and an active group or center, etc. These characters render these molecules cover a considerable area surface of the metal (film) with firm attachment. Plants considered as inhibitor source of many ones, which is clean and cheap (Fouda *et al.*, 2014). Plants extracts (i.e., seeds, leaves, flowers, and fruits) possess various active compounds with the ability of corrosion inhibition in aggressive media had been considered in recent studies (Khadom *et al.*, 2018; Mobin *et al.*, 2018; Haldhar *et al.*, 2018; Banu *et al.*, 2018). In this regard, different

investigators are concentrating on the growth of friendly environmentally inhibitors of corrosion, i.e., natural products, *Spirulina platensis* (Gopiraman *et al.*, 2012), *Brugmansia suaveolens* (Kamal, *et al.*, 2012), green eucalyptus leaves (Dehghani *et al.*, 2016), *Acalypha torta* (Krishnegowda *et al.*, 2013), *Luffa aegyptiaca* (Jyothi and Ravichandran, 2014), watermelon rind (Odewunmi *et al.*, 2015), *Ligularia fischeri* (Prabakaran *et al.*, 2018), *Lansea coromandelica* (Muthukrishnan *et al.*, 2017), *Tilia cordata* (Fouda *et al.*, 2017), sweet melon peels (Saeed *et al.*, 2019), *Griffonia simplicifolia* (Ituen *et al.*, 2017), *Dillenia Pentagyna* fruits (ManjunathHegde *et al.*, 2019), *Pongamia pinnata* (HouariaDerfouf *et al.*, 2019). The existence of organic plant compounds, i.e., carbohydrates, flavonoids, alkaloids, saponins, and phenols, make them well source inhibitors of corrosion. These phytochemicals possess various functional groups like hydroxyl, carbonyl, and amino (Sakunthala *et al.*, 2019). Thus, these plants behave as well as inhibitor sources of corrosion that should not be neglected.

Wild onion (WO) plant (*Urginea maritima*), a medicinal plant from the area of Mediterranean, belongs to the family Liliaceae. WO grows naturally in the wild, explicitly cultivated (Deb and Dasgupta, 1987). The parts of medicinal importance are the bulbs which harvested, transversely sliced, and grounded for being ready to used (Gentry *et al.*, 1987). Genus of *Urginea* is traditionally used in medicine because of its antiepileptic, cardiotonic, diuretic, anti-asthmatic, and dermatological characteristics (El-Seedi *et al.*, 2013).

The purpose of this study was to investigate the corrosion inhibition ability of WO extract for C-steel in a 0.5 M HCl solution using weight loss measurements.

2. MATERIALS AND METHODS:

2.1. Preparation of specimens

The coupons of C-steel (5 x 2.6 x 0.3 cm) with the composition of % V, 0.21 wt., 0.34 wt. % C, 0.51 wt. % P, , 0.035 wt. % Cr, 0.44 wt. % Mn, 0.017 wt. % Al, 0.054 wt. % Mo and Fe (balance), were applied in this paper (Figure 1). The coupon surface polished by emery paper of grit from 400 to 600 grades prepares sequentially to the polished surface. After polishing, the coupons

were washed with tap water then by distilled water and dried with clean tissue with hot air and finally in desiccators to get coupons moisture-free before storage and use.

Figure 1. Coupons of C-steel.

2.2. Wild onion (WO) extraction

WO plant was collected from lawns of Duhok governorate, Kurdistan Region of Iraq (Figure 2). The bulbs were washed and cut into small pieces and then dried under shadow at ambient temperature then ground into a fine powder. For the extraction procedure, 10 g of WO powder was weighed and mixed with 100 ml of (90% methanol) in a conical flask. The mixture was extracted during stirring for 24h at room temperature. The resultant mixture was filtered carefully, and the methanol evaporated by drying in an oven at 45°C for 2h. A yellow-brown sticky residue of dried WO extract was obtained. The dried extract was stored in a refrigerator for further use.

Figure 2. Wild onion plant (*Urginea maritima*).
Courtesy: Jarjes et al.,(2016).

2.3. Preparation of corrosive solution

The electrolyte corrosive was 0.5 M HCl solution that was prepared from HCl (37%) analytical grade and distilled water. The inhibited solutions were prepared by adding WO extract to 0.5 M HCl prepared freshly at different concentrations (0.5%,

1%, 1.5%, 2%, and 2.5%).

2.4. Loss of weight measurements

Through measurement of weight losing, each coupon specimen was weighted carefully and then immersed in the 0.5 M HCl solution with various dried extract concentrations of WO (0%, 0.5%, 1%, 1.5%, 2%, and 2.5%) at (25, 35 and 45°C), respectively. After (2, 4 and 8hr) immersion in the solution, the specimens were taken out and washed in tap water for removal all products of corrosion on the surface of the metal, washed another time with distilled water (DW), dried by clean tissue and hot air, and then weighed after they have been kept in desiccators for 3h. The same procedure was repeated for each temperature.

From the weight loss data, the CR was calculated by applying Eq. (1) (Tredder, 1984).

$$CR \text{ (mpy)} = \frac{534 W}{D A T} \quad (1)$$

Where:

CR (mpy): is the corrosion rate in mills per year.

W: is weight loss (mg).

D: density (g/cm³).

A: area of the C-steel coupon (in²).

T: time exposure (h).

By applying Eq. (2), the efficiency of inhibition (%E) was calculated (Danielson, 1982).

$$\%E = \frac{CR_o - CR_i}{CR_o} \times 100 \quad (2)$$

Where:

CR_o and CR_i: are the CRs in the presence and absence of various inhibitor concentrations, respectively.

By applying Eq. (3), the coverage of the surface (θ) was calculated (Damaskin, 1971).

$$\theta = 1 - \frac{CR_i}{CR_o} \quad (3)$$

3. RESULTS AND DISCUSSION:

3.1 Measurements of weight-losing and temperature effect

CR variation, inhibition efficiency %E and coverage of surface θ were evaluated through measurement of C-steel weight losing of in the media of corrosives with different concentrations of (WO) extract, and different temperatures are shown in the Tables 1, 2 and 3 and Figures 3, 4, 5, 6, 7 and 8. Results proved that the CR was declined as the inhibitor concentration elevated; i.e., in the solution of blank acidic, after 2h, the CR of C-steel was 41.45 (mpy), which decreased to 9.56 (mpy) with the addition of 0.5% of (WO) extract at 25°C. In the presence of 2% inhibitor, CR of C-steel was approximately 10 times lower compared to blank solution where the foregoing trials were proved. As the temperature increased, CR increased significantly from 41.45 (mpy) to 109.63 (mpy) and then to 185.7 (mpy) with an increase in temperature at the above, respectively, in blank acidic solution. Nevertheless, the effectiveness at each temperature for inhibitor extract was assumed from the significant CR decrease.

Inhibition efficiency (%E) values and surface coverage θ elevated by elevating the concentration of inhibitor because of the inhibitor adsorption on the C-steel surface which occurred and formed a layer of protective coverage on surface of the C-steel (Moore, 1994).

Maximum inhibition efficiency %E of about 98.95% was reached in 2.5% of inhibitor concentration at 25°C and after 8hr. By increasing temperature, the efficiency of inhibition %E declined; i.e. efficiency inhibition %E was decreased from 98.95% to 88.99% and then to 86.79% when temperature elevated to 35°C and to 45°C, respectively from 25°C at 2.5% of the inhibitor concentration. The CR in current density units I_{corr} can be related to the weight loss by Eq. (4) (Moore, 1994)

$$I_{corr}(\text{mA}/\text{cm}^2) = \frac{2 \cdot 1000 \cdot W \cdot F}{A_w \cdot T \cdot A} \quad (4)$$

Where:

I_{corr} = corrosion current density (mA/Cm²).

F = Faraday constant, 96500 coulombs.

A_w = Atomic weight of Fe, 55.9.

T = Exposure time (sec.).

A = Specimen external surface area (Cm²).

W = corrosion weight losing (mg.).

Table 4 shows the variation of the corrosion current density I_{corr} values for the C-steel in HCl solution of 0.5 M with different temperatures, immersion times, and inhibitor concentrations. By concentration elevating, the density of current corrosion I_{corr} decreased, and the efficiency of WO extract inhibition increased. This might occur because of the adsorption of various inhibitor compounds on the surface of C-steel. Thus, WO extract addition decreased both the partial reaction rate through declining the dissolution of anodic and lowering down the H reaction evolution and blocks C-steel attack in the acid media (Solmaz *et al.*, 2008). Table 4 shows that the values of I_{corr} were elevated with elevating temperature from 25°C to 45°C; this is due to the temperature increase, which led to the desorption of protective layer on the surface of C-steel increase (Ramesh, 2003; Shaker, 1986; Adam, 1986).

3.2. Immersion time effect

Determination of the inhibitor stability adsorption on the C-steel surface by losing weight measurement was performed with different inhibitor concentrations at different immersion times (2, 4, and 8h) and various temperatures (25°C, 35°C and 45°C). Figures 9, 10, and 11 show that as the immersion time increased, inhibition efficiency %E increased at 25°C. In comparison, at 35°C and 45°C a decrease was found in the inhibitor efficiency %E as the immersion time increased. The recent results may be due to the desorption of inhibitor on the surface of C-steel when the temperature rose. Maximum inhibition efficiency %E was lower at 45°C than at 35°C and 25°C. Based on the available literature, when temperature increased, it caused a decrease in the efficiency of inhibition. This might be attributed to the desorption of inhibitor constituents from the surface of the metal. In this situation, the adsorption is basically because of the interaction of electrostatic that declined at high temperatures. This proved that the

process of adsorption is a physisorption type (Sakunthala, 2013; Karthik and Sundaravadivelu, 2017).

3.3. Adsorption isotherm

The isotherms of adsorption are mathematical expressions that provide data about the reciprocal effect of the metal surface and adsorbing species at a fixed temperature. Generally, in acid corrosion, it is presumed that act of inhibitors by an adsorption process on the surface of the metal. The adsorption behavior is dependent on the amount of coverage surface θ , were obtained by weight loss studies. An isotherm of adsorption provides the relation between the interface coverage with the species that adsorbed and the species concentration in solution. To examine the isotherm of adsorption, a graphic relation between the concentration inhibitor C_{inh} and C_{inh}/θ at various temperatures was graphed and showed in Figure 12. In this figure, straight lines can be seen, and the correlation values of coefficient R^2 were nearly equal to one pointing this system followed Langmuir isotherm of adsorption Eq. (5) at different temperatures. The above result indicated as well that inhibitor of mono-layer should be adsorbed to the surface of C-steel without any interaction among the molecules that adsorbed (Hamdy and El-Gendy, 2013; Faustin *et al.*, 2015).

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (5)$$

Where:

C_{inh} : the concentration of inhibitor

θ : average degree of coverage on the surface metal

K_{ads} : adsorption process constant equilibrium.

The inhibitor's equilibrium adsorption constants (K_{ads}) were calculated by the linear equations intercept C_{inh}/θ versus C_{inh} [8]. To compute the free energy standard of adsorption, value of K_{ads} was used (ΔG°_{ads}) value based on the following (Bentiss *et al.*, 2009):

$$\Delta G^\circ_{ads} = -2.303 RT \log (55.5 K_{ads}) \quad (6)$$

The value of 55.5 was the

concentration of water in solution expressed in mol/L. The adsorption constants K_{ads} and free energy of standard adsorption ΔG°_{ads} values at different temperatures were grouped in Table (5).

Generally, the values of ΔG°_{ads} up to -20 kJ/mol are accepted, adsorption kinds were considered as physisorption; acts of inhibition were attributed to the interaction of electrostatic between charged metal and the charged molecules, whereas values smaller than -40 kJ/mol or around were observed as chemisorption. This is because of sharing the charge or a transfer to the surface of metal from the molecules of the inhibitor to form covalent bonding (Yurt *et al.*, 2005).

At the present investigation, the negative sign of ΔG°_{ads} proved that the inhibitor adsorption was spontaneous. The computed adsorption values of free energy ΔG°_{ads} at different temperatures were around of -13.5 kJ/mol that assists the physical mechanism of adsorption (Adejoro *et al.*, 2015; Ali *et al.*, 2017).

4. CONCLUSIONS:

The CR declined as the concentration of inhibitor elevated and the efficiency of inhibition %E raised with elevating the inhibitor of concentration but decreased with temperature raising. The decline in the efficiency of inhibition with elevating temperature was suggestive of mechanism for physical adsorption (physisorption). It might be due to solubility increase in the products of protective film precipitated on the C-steel surface, which may otherwise inhibit the process of corrosion. The overall results of the current work revealed that the eco-friendly wild onion extracts is a promising corrosion behavior of C-steel in acidic media.

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Table 1. CR, the efficiency of inhibition %E and coverage surface Θ of C-steel in corrosive media at 25°C at different immersion times.

Conc. (w/v %)	25 °C								
	Time (h)								
	2			4			6		
	CR	%E	Θ	CR	%E	Θ	CR	%E	Θ
0	41.45			68.68			94.31		
0.5	9.65	76.72	0.77	8.94	87	0.87	7.01	92.57	0.92
1	8.41	79.71	0.8	6.01	91.2	0.912	5.57	94.09	0.94
1.5	6.3	84.8	0.85	4.93	92.8	0.928	3.51	96.28	0.96
2	4.58	88.95	0.89	2.91	95.8	0.958	2.11	97.76	0.97
2.5	3.01	92.74	0.93	2.09	97	0.97	0.99	98.95	0.98

Table 2. CR, the efficiency of inhibition %E and coverage surface Θ of C-steel in corrosive media at 35°C at different immersion times.

Conc. (w/v %)	35 °C								
	Time (h)								
	2			4			6		
	CR	%E	Θ	CR	%E	Θ	CR	%E	Θ
0	109.63			146.8			196.3		
0.5	27.94	74.5	0.75	45.03	69.32	0.69	67.74	66	0.66
1	19.99	81.8	0.81	35.6	75.74	0.75	52.08	73.46	0.73
1.5	13.42	87.8	0.87	32.2	78.06	0.78	45.8	78.7	0.78
2	8.92	91.9	0.92	22.06	84.97	0.84	36.1	81.6	0.81
2.5	5.87	94.6	0.94	12.04	91.79	0.91	21.6	88.99	0.88

Table 3. CR, the efficiency of inhibition %E and coverage surface Θ of C-steel in corrosive media at 45°C at different immersion times.

Conc. (w/v %)	45 °C								
	Time (h)								
	2			4			6		
	CR	%E	Θ	CR	%E	Θ	CR	%E	Θ
0	185.7			196.9			204.6		
0.5	78.24	51.48	0.51	92.83	52.85	0.52	95.85	53.15	0.53
1	57.78	68.23	0.68	70.71	64.08	0.64	79.7	69.84	0.69
1.5	32.04	82.38	0.82	47.62	75.81	0.75	51.2	74.97	0.74
2	19.56	89.24	0.89	27.5	86.03	0.86	36.4	82.2	0.82
2.5	17.92	90.14	0.901	21.01	89.32	0.89	27.01	86.79	0.86

Table 4. Corrosion current densities (mA/cm^2) at different immersion times, different temperatures, and different inhibitor concentrations of C-steel in the corrosive media.

Conc. (w/v %)	25°C			35°C			45°C		
	2h			4h			6h		
	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}	I _{corr.}
0	90.64	150.2	206.2	239.73	321.01	429.25	406.09	430.56	447.4
0.5	21.1	19.56	15.33	61.11	98.48	148.14	171.09	203	209.27
1	18.4	13.14	12.2	43.73	77.86	113.89	126.36	154.63	174.28
1.5	13.79	10.78	7.68	29.35	70.41	100.15	70.07	104.15	111.97
2	10.02	6.37	4.62	19.52	48.24	78.94	42.77	60.13	79.61
2.5	6.59	4.58	2.18	12.85	26.33	47.25	39.19	45.95	59.06

Note: I_{corr} = corrosion current density (mA/Cm^2)

Table 5. Thermodynamic parameters obtained from Langmuir adsorption isotherm.

Temperature	R ²	K _{ads}	$\Delta G^\circ_{\text{ads}}$
25 °C	0.996	5.025	-13.9
35 °C	0.998	4.651	-14.2
45 °C	0.994	2.227	-12.7

Figure 3. Variation of CR with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (25°C).

Figure 4. Variation of CR with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (35°C).

Figure 5. Variation of CR with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (45°C).

Figure 6. Variation of inhibition efficiency %E with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (25°C).

Figure 7. Variation of inhibition efficiency %E with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (35°C).

Figure 8. Variation of inhibition efficiency %E with various concentrations of (WO) extract for C-steel coupons in 0.5 M HCl solution at (45°C).

Figure 9: Variation efficiency of inhibition %E with various times of immersion for C-steel coupons in solution of 0.5 M HCl in the presence and absence of various (WO) extract concentrations of at (25°C).

Figure 10: Variation efficiency of inhibition %E with various times of immersion for C-steel coupons in solution of 0.5 M HCl in the presence and absence of various (WO) extract concentrations of at (35 °C).

Figure 11. Variation efficiency of inhibition %E with various times of immersion for C-steel coupons in solution of 0.5 M HCl in the presence and absence of various (WO) extract concentrations of at (45°C).

Figure 12. Langmuir of isotherm adsorption model of (WO) extract on the surface of C-steel at (25°C ,35°C and 45°C).