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NARROW-LEAVED PEPPERWORT (*LEPIDIUM RUDERALE L.*) FROM WESTERN KAZAKHSTAN - A NEW “GREEN” CORROSION INHIBITOR FOR CARBON STEEL

Annotation.*In present work,the inhibitor properties of Lepidiumruderale L. aqueous extract (LRAE) was investigated by weight loss measurements and electrochemical techniqueson the corrosion of carbon steel in typical acidic and saline media. It was established that the inhibitor efficiency of the LRAE reaches up to 91.05, 92.03, 93.84% in 1.0 mol/L hydrochloric, sulfuric, and phosphoric acids solutions respectively. Inhibition wasfound to increase with increasing in concentration of LRAE. In all tested media, the adsorption of the inhibitor molecules on metal surface was in accordance with Langmuir model and the high efficiency wasachieved due to the spontaneous physisorption (ΔG^0_{abs} > -20 kJ/mol). Polarization curves revealed that LRAE act as a mixed type inhibitor and the inhibition efficiency of up to 95.34% can be obtained. Electrochemical data are in great agreement with gravimetrical experiment.*

Keywords: *Lepidiumruderale L.; plant extracts; inhibition efficiency; “green” inhibitors; metal protection.*

Introduction

Corrosion is the devastating result of a chemical interaction between a metal or alloy and its environment [1]. Application of corrosion inhibitors for reducing the activity of an aggressive mediumis the most well-known method of metal protection and one of the most useful on the industry among others [2].

Corrosion inhibitors are subdivided to organic and inorganic. Most of them are not eco-friendly and "green" [3]. Practice shows that organic compounds containing N, S and O, which have a significant inhibitory effect, are not only expensive but also toxic to living beings. Therefore, it is unnecessary to stress the importance of inexpensive and safe corrosion inhibitors[4].

“Green” corrosion inhibitors can be obtained from plants. They are the rich sources of natural origin organic compounds (phenols, flavonoids, alkaloids etc.) that can protect different metals from the destructive effects of various corrosion media [5]. Whole plants or plant partscan be successfully used for this purpose[6].Nowadays, plant extracts have become indispensable eco-friendly, renewable and low-cost resources for effective anti-corrosion applications [7].

More than 1200 plant species, representing more than 700 families, are distributed in the West Kazakhstan region (WKR). The biodiversity of the WKR floraoffers unique development opportunities in the field of "green" technologies. In recent decades, this trend has become firmly established in the fields of catalysis, pharmacology as well as the protection of metals and alloys from corrosion damage.

Narrow-leaved pepperwort (*Lepidiumruderale L.*) member of *Brassicaceae* family (Fig.1) is one of the ubiquitous plants in the region and easily accessible for study and use.

Figure1 –Narrow-leaved pepperwort (*Lepidiumruderale L.*), (fam. *Brassicaceae*)

In Kazakhstan, 21 species of Brassicaceae family are described, of which four species, include *L. ruderale L.* are characterized by medicinal properties and used in folk medicine [8]. Set of phenolic compounds were identified in *L. ruderale L.* growing in Pavlodar region and it was revealed that the plant has a high biological activity [9]. Moreover,*L. ruderale L.* can grow in polluted areas [10] and therefore the phytoremediation potential of the plant for soils contaminated with heavy metals wasalso investigated[11].

In the present work, we turned our attention to another one possible application of *L. ruderale L.*, the protection of metals from corrosion in liquid media. Therefore, the possibility of using *LRAE* as a corrosion inhibitor for carbon steel in typical corrosion media was investigated.

Materials and methods

Reagents and solvents.Allreagents are analytical grade were purchase in Sigma Aldrich, Merck, Alfa Aesar and TCI and used without any further purification. Double distilled water was used to prepare the corrosion media.

Collection and preparation of plant material. Plants were collected from their natural habitat in summer 2023 in the suburbs of Uralsk, away from roads and industrial enterprises. Plant species were authenticated in the herbarium of the Faculty of Natural Sciences of M. Utemisov West Kazakhstan University and The World Flora Online database http://www.worldfloraonline.org/taxon/wfo-0000358755. Plants were washed thoroughly with tap water, double-distilled water, and kept in shaded place for 2 weeks to total removing the moisture. The dried samples were ground into a fine powder, sieved through 1.0 mm, and stored at 4^{o}C until further use.

Plant extracts preparation. To 10 g of ground plant sample in the Erlenmeyer flask, 100 ml of double-distilled water was added. Then the flask was heated in a water bath (60^{o}C) for 4 hours. Then the obtained extract was filtered using Buchner funnel, and the residue was re-extracted with another portion of solvent under the same conditions. After triple extraction, the obtained extracts were combined and evaporated from solidification. Solid residue was dried at 40^{o}C for a constant weight, and stored at 4^{o}C indark glass vials.

Sample preparation. Carbon steel coupons with exposed areas of 2.5 x 3.5 x 0.3 cm with compositions of (wt. %) 97,8 - Fe; 0,22 - C; 0,65 - Mn; 0,30 -Si; 0,04 - P; 0,05 - S; 0,30 - Cr; 0,30 - Ni; 0,30 - Cu; 0,01 - N; 0,08 - As were obtained from industry. Before experiment, coupons were polished by emery paper with 250-1200 grit numbers. Then the abraded specimens were cleaned from dust, grease and oxides by washing in running water, double distilled water, ethanol, acetone, dried on air and store in desiccator.

Weight loss (gravimetric) method. Weight loss experiments were performed to evaluate the corrosion rate (CR), inhibition efficiency (η%) and degree of surface coverage (θ). For investigation, each coupon was immersed in open-to-air 100 ml beaker containing 75 ml of corrosion medium at room temperature by 18 h. After the specified immersion time, coupons were withdrawn from the beaker and corrosion products were removed by washing of each coupon in solution containing 50% NaOH and 100 g of zinc dust [12]. Then, coupons were again washed in running water, double distilled water, rinsed in ethanol, acetone and dried in air before reweighting. All weight measurements were performed using Ohaus Adventurer Pro AV264 analytical balance with accuracy of ±0.1mg.

The corrosion rate (CR $g/m^2 \cdot h$), inhibition efficiency (η%) and degree of surface coverage (θ) were calculated according the equations 1-3 respectively:

$$CR(g/m^2 \cdot h) = \frac{\Delta m}{S \cdot \tau} \quad (1)$$

$$\eta(\%) = \frac{CR_0 - CR_i}{CR_0} \cdot 100 \quad (2)$$

$$\theta = 1 - \frac{w_1}{w_2} \quad (3)$$

where Δm – is the weight loss of mild steel (g) after the period of immersion (h), CR_0 – the corrosion rate of carbon steel without inhibitor, CR_i - the corrosion rate of carbon steel at present of inhibitor, w_1 and w_2 are the weight loss (g) for carbon steel in the presence and absence of inhibitor in solution respectively.

Adsorption and thermodynamics.To describe of inhibitor adsorption mechanism to the carbon steel surface the Langmuir [13]adsorption isothermswereused (equation 4):

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{abs}} + C_{inh} \quad (4)$$

where θ is the degree of surface coverage, C_{inh} the concentration of the inhibitor and K_{abs} is the equilibrium constant of the absorption-desorption process. The values of the absorption equilibrium constant (K_{abs}) wereobtained from the intercept of the absorption isotherms. The relationship between free Gibbs energy and absorption equilibrium constant is expressed by the equation 5.

$$\Delta G^0_{abs} = -RT\ln(55.5K_{abs}) \quad (5)$$

where ΔG^0_{abs} is the free Gibbs energy of the absorption, R is universal gas constant (8.314 J/K·mol), T – thermodynamic temperature of the system and 55.5– molar concentration of watermol/L.

Electrochemical measurement.Steel coupons after pretreatment as for the weight loss assay were used for the electrochemical experiment. The electrochemical testing was performed using the Autolab PGSTAT 101 Metrohmpotentiostat/galvanostat device equipped with NOVA 2.1.6 software. In the method of potentiodynamic polarization three-electrode setup was used: Ag/AgCl (3M KCl) as the reference electrode, the platinum as counter electrode, and steel coupon connected with a specimen alligator clamp to serve as working electrode. The electrochemical cell was of 100 ml of 1.0mol/L HCl, H_3PO_4 and $H_2SO_4$with and without inhibitor in glass beaker at room temperature. The working electrode, with 1.00 cm^2 exposed area was stabilized at open circuit potential (OCP) testing. The linear polarization measurement were performed immediately after the OCP by running a Linear Sweep Voltammetry (LSV) staircase and corrosion rate analysis. The potentiodynamic scan was carried out from -0.50 to +0.50 V at a scan rate of 0.001 V/s and a step voltage of 0.001V. The corrosion potential (E_{corr}) and corrosion current density (j_{corr}) were determined from the Tafel polarization curves.

The inhibition efficiency (η_i%) through the corrosion current was calculated using the following equation [14]:

$$\eta_i(\%) = \frac{i_{ing} - i_{corr}}{i_{ing}} \cdot 100 \qquad (6)$$

wherei_{corr} and i_{inh} are the corrosion current densities calculated from the extrapolation of Tafel slopes without and with inhibitors, A/cm^2.

The inhibition efficiency through the polarization resistance (η_R%) was calculated using the equation [15]:

$$\eta_R(\%) = \frac{R_p^{ing} - R_p^0}{R_p^{ing}} \cdot 100 \qquad (7)$$

whereR_p^{ing} and R_p^0 are the charge transfer resistance in the presence and absence of inhibitor, Ω.

Results and discussion

Weight loss assay.

Inhibition efficiency of LRAEwas examined for the 18 typical corrosion media include inorganic and organic acids (HCl, H_2SO_4, HNO_3, H_3PO_4, CH_3COOH) in different concentrations (0.1, 0.5, 1.0 mol/L), distilled water (DW), 3.0% NaCl and the model media recommended by the National Association of Corrosion Engineers of USA (NACE) contains 5 g/L NaCl and 0.25 g/L CH_3COOH [16]. Results of the screening are shown in Table 1.

Table 1 – Inhibitor efficiency (η%) of*LRAE*against corrosion of carbon steel in typical corrosion media at the inhibitor concentration of 1.0 g/L at room temperature

Entry	Corrosion media	Concentration	CR, g/m²·h		θ	η, %
			Blank	With inhibitor		
1	CH_3COOH	1,0mol/L	1,57	0,24	0,8440	84,40
2		0,5mol/L	2,91	0,62	0,7855	78,55
3		0,1mol/L	1,20	0,47	0,6104	61,04
4	H_2SO_4	1,0mol/L	36,02	2,87	0,9203	92,03±1,42
5		0,5mol/L	22,80	2,81	0,8768	87,68
6		0,1mol/L	10,35	1,72	0,8340	83,40
7	HNO_3	1,0mol/L	42,96	36,42	0,1523	15,23
8		0,5mol/L	21,99	18,27	0,1692	16,92
9		0,1mol/L	5,89	3,06	0,4806	48,06
10	H_3PO_4	1,0mol/L	18,28	1,13	0,9384	93,84±1,16
11		0,5mol/L	14,37	1,48	0,8968	89,68
12		0,1mol/L	5,91	0,93	0,8423	84,23
13	HCl	1,0mol/L	9,71	0,87	0,9105	91,05±1,45
14		0,5mol/L	8,62	0,96	0,8882	88,82
15		0,1mol/L	6,68	1,14	0,8295	82,95
16	NACE medium	NaCl 5 g/L + CH_3COOH0.25 g/L	0,66	0,22	0,6605	66,05
17	NaCl	3.0%	0,49	0,39	0,1891	18,91
18	DW	-	0,45	0,29	0,3536	35,36

The experimental data shows that the *LRAE*exhibits inhibitory properties in all 18 corrosion media and has a range of anti-corrosion efficiency from 15.23 to 93.84%. The studied extract demonstrated high efficiency in solutions of sulfuric acid (92.03 ± 1.42), phosphoric acid (93.84 ± 1.16) and hydrochloric acid (91.05±1.45)at concentrations of 1.0 mol/L (Entries 4, 10 and 13 respectively). The experiment showed good reproducibility. In addition, it was found that good anticorrosion properties LRAE was also demonstrated in acetic acid 1.0mol/L (84,40%, Entry 1), sulfuric acid 0.5 mol/L (87,68%, Entry 5), phosphoric acid 0.5 mol/L (89,68%, Entry 11)and hydrochloric acid 0.5 mol/L (88,82%, Entry 14). The least efficiency of the inhibitor was observed in nitric acid solutions (Entries 7-9) and for the neutral saline environments (DW and 3.0 NaCl) (Entries 17, 18).

Due to the very high inhibition efficiency of LRAE in 1.0 mol/L of hydrochloric, phosphoric, and sulfuric acid solutions, the next step of our investigation was focused on the determination of the kinetic and thermodynamic parameters of LRAEin these corrosive media. Further experiments were also performed at room temperature with inhibitor concentrations of 0.1, 0.5, 1.0, 1.5, and 2.0 g/L and for 2, 6, 16, 18 and 24 hours of exposure time.

Figure 2 shows the dependence of LRAE 1.0 g/L inhibitor efficiency in 1.0 mol/L hydrochloric, phosphoric and sulfuric acid solutions at room temperature ondefined exposure time.

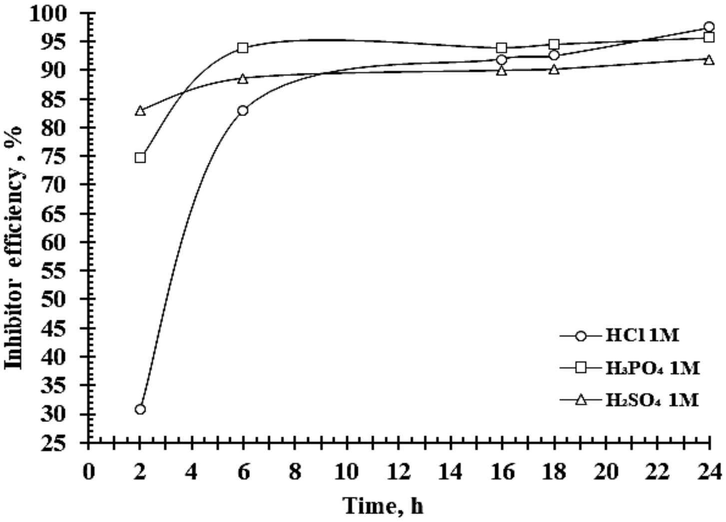

Figure 2 –Relationship between the inhibition efficiency of LRAE in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions and immersion period

Figure 2 shows that the protective efficiency of LRAEin 1.0 mol/L HCl, H_3PO_4 and H_2SO_4increases over time. The presence of an induction period indicates that in all corrosive media tested, a minimum of 6 hours is required for the inhibitor to form a maximally effective protective layer.

The dependency of the LRAE inhibition efficiency in 1.0 mol/L of hydrochloric, phosphoricand sulfuric acid solutions at various inhibitor concentrations for a 24-hour exposure period is depicted in Fig.3.

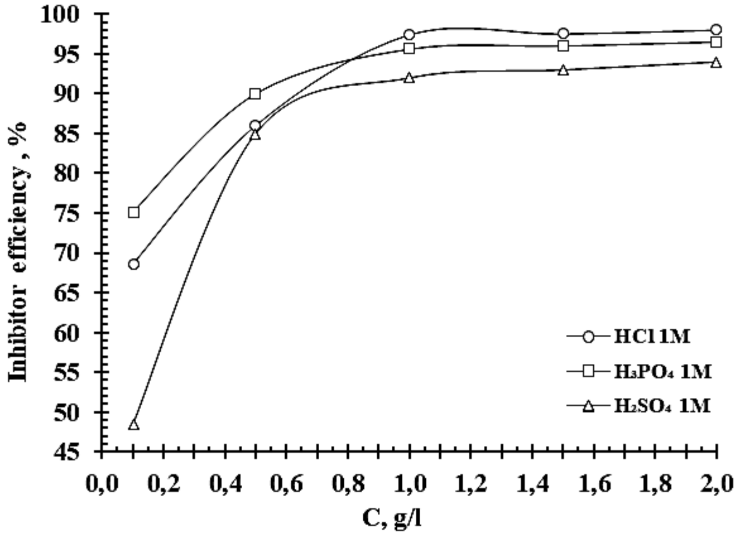

Figure 3 – Relationship between the inhibitor efficiency of LRAEin 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions and inhibitor concentrations for 24 hours immersion period

As can be seen, the addition of LRAE resulted in noticeable increasing in the inhibitor efficiency. Increasing the inhibitor concentration leads to an increase in protection properties. The most effective corrosion inhibition was observed at LRAE concentration of 2.0 g/L. These results suggest that LRAE can potentially be used as an effective corrosion inhibitor to protect metals in highly acidic environments. The reproducibility for the inhibition efficiency values were precise to ± 5%. This indicates good reproducibility.

Adsorption of inhibitor to the metal surface may have chemical, physical or mixed nature. To describe the absorption mechanism the values of free Gibbs energy ΔG^0_{abs} must be determined. The ΔG^0_{abs} values up to -20 kJ/mol indicates the electrostatic interaction between metal surface and charged inhibitor molecules (physisorption). Values around -40 kJ/mol usually accepted as a threshold value between chemisorption and physisorption, or less indicate the chemical nature of sorption [17].

For plotting the LRAE adsorption isotherms the surface coverage (θ) values for different concentrations of the inhibitors have been evaluated from the weight loss data in accordance with equation 4. Langmuir isotherms of LRAEadsorption in hydrochloric, phosphoricand sulfuric acid solutions at room temperature was shown in Fig.4.

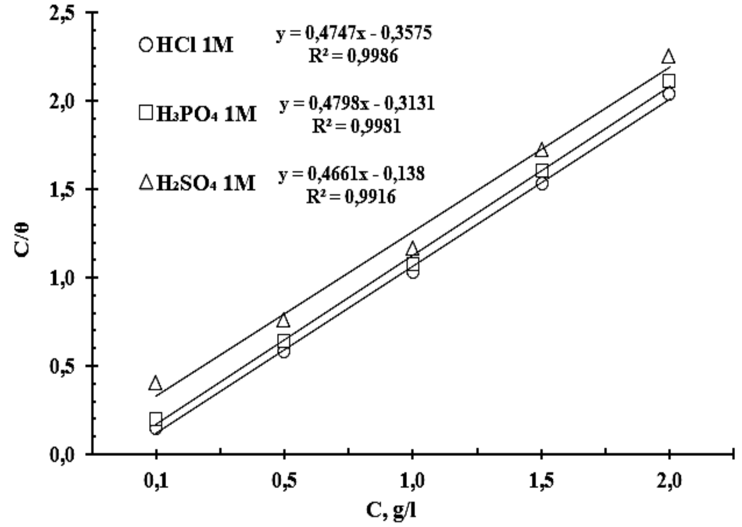

Figure 4 – Langmuir adsorption isotherms for LRAEin 1.0 mol/L HCl, H_3PO_4 and H_2SO_4 solutions for 24 immersion period at room temperature

Plots of C/θ against C (Fig. 4) show straight lines. Linear plots and closed to one values of the correlation coefficient (R^2> 0.99) indicate that the Langmuir model is suitable for the description of the LRAE adsorption on metal surface. It confirms that the inhibition is due to the adsorption of the active organic compounds onto metal surface. This also clearly indicates that the adsorption of LRAEoccurs as a substitution between the organic molecules in the aqueous solution and the water molecules on the metal surface [18].

In accordance with the slopes and intercepts of the straight lines, free Gibbs energy (ΔG^0_{ads}) and K_{abs} values for LRAEwere calculated and are given in Table 3.

Table 3 – Thermodynamic parameters ofLRAE adsorption on carbon steel surface in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions

Corrosion media	Slope	Intercept	R^2	K_{abs}	ΔG^0_{abs}, kJ/mol
HCl	0,4747	0,3575	0,9986	2,7972	-12,29
H_3PO_4	0,4798	0,3131	0,9981	3,1939	-12,61
H_2SO_4	0,4661	0,1380	0,9916	7,2464	-14,61

Negative values of ΔG^0_{ads} indicate the absorption of LRAE on metal surface is a spontaneousprocess [19]. The value of ΔG^0_{ads} > -20 kJ/molclearly indicates that adsorption is physical (physisorption) and for all investigated media occurs as a result of electrostatic interaction between charged inhibitor molecules and the metal surface [20].

Metal surface analysis.

The protective efficiency of LRAE was also studied by the micro images of steel surface. The polished specimen and the test specimens that are immersed in the blank and in the inhibitor LRAEwere observed under an optical microscope. Microscope image of carbon steel specimen before and after of the immersion in the absence and presence of LRAE are depicted in Fig.5.

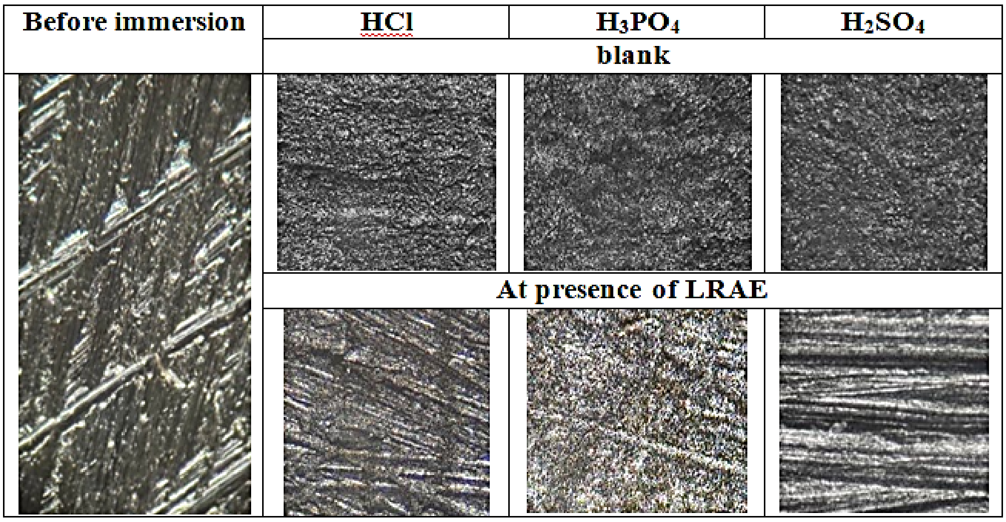

Figure 5 – Micro images(^{x}50) of the mild steel surface after 24h immersion in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions in the absence and presence ofLRAE (1.0 g/L)

Before immersion in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4without the addition of LRAE, the surface of the steel specimens was homogeneous with ametallic luster and well-defined structure. After immersion time without an inhibitor, a drastic change in metal surface structure is observed with the formation of a uniform dark, friable layer with noticeable pits and cracks.

In the presence of LRAE, the metal surface retains its structure during exposure, but with the loss of the original luster due to the formation of protective film. At the same time, visible corrosion damage on the surface is significantly reduced. The surface analysis study confirms the corrosion inhibition of carbon steel by the LRAE.

Electrochemical experiment.

Figure 6 shows the potentiodynamic polarization curves for carbon steelin 1.0 mol/L HCl, H_3PO_4 and $H_2SO_4$with and without LRAE.

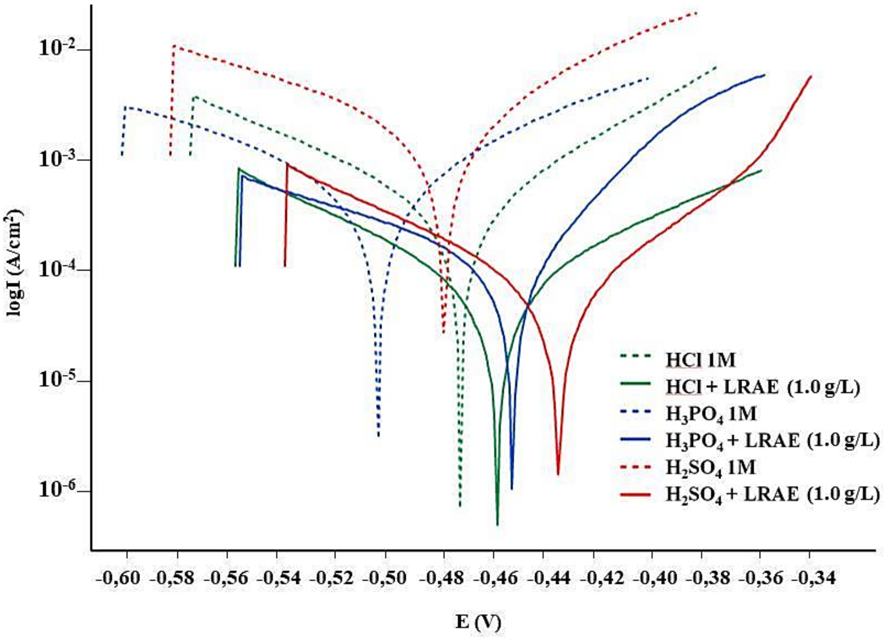

Figure 6 –Polarization curvesfor carbon steel in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions in the absence and presence of1.0 g/L LRAEat room temperature

Electrochemical data of the corrosion process calculated from polarization curves are presentin Table 4.

Table 4 – Electrochemical parameters for carbon steel corrosion in 1.0 mol/L HCl, H_3PO_4 and H_2SO_4solutions at presence and absence of 1.0 g/L LRAE at room temperature

Corrosion medium	$-E_{корр}$, mV	J_{corr}, A/cm^2	η_i, %	$\lvert b_a \rvert$, mV/dec	$\lvert b_c \rvert$, mV/dec	R_p, Ω	η_R, %
HCl 1.0 M	472,75	$3{,}15\cdot10^{-4}$	-	71,13	91,09	60,37	-
HCl 1.0 M + LRAE	458,56	$6{,}93\cdot10^{-5}$	78,04	92,90	101,19	316,80	80,94
H_3PO_4 1.0 M	503,95	$5{,}09\cdot10^{-4}$	-	92,19	111,73	47,34	-
H_3PO_4 1.0 M + LRAE	452,90	$1{,}19\cdot10^{-4}$	76,48	45,27	138,44	135,96	65,18
H_2SO_4 1.0 M	478,84	$1{,}59\cdot10^{-3}$	-	74,33	119,49	13,66	-
H_2SO_4 1.0 M + LRAE	435,42	$5{,}89\cdot10^{-5}$	96,31	59,98	91,31	293,25	95,34

The presence of LRAEleads to a significant decrease in the carbon steel corrosion ratein 1.0 mol/L HCl, H_3PO_4 and H_2SO_4. The values of the corrosion current density (J_{corr}) are significantly lower at presence of the inhibitor, which indicates LRAE effectiveness in protecting of metal against corrosion [21]. The values of polarization resistance (R_p) increase with the addition ofLRAE, which confirms the formation of a protective layer on the metal surface thereby blocking the subsequent corrosion damage [22].

The presence of an inhibitor leads to a noticeable shift in both the cathodic and anodic parts of the polarization curves. Therefore, the LRAEbehaved as a mixed type inhibitor and slowing down both anodic and cathodic reactions [23]. Nevertheless, for all media $|b_c|$ values are greater than $|b_a|$ suggesting that the effect of the inhibitor on the cathodic polarization is more pronounced than on the anodic polarization.

In the presence of LRAE, the inhibition efficiency values calculated from electrochemical data (polarization resistance (R_p, Ω)and current density (J_{corr}, A/cm^2)) for all corrosive environments increase significantly. This also indicates that the investigated inhibitor has a high ability to protect the metal surface from corrosion. Results of electrochemical measurement are in great agreement with the gravimetrical data.

Conclusion

In this work, the anticorrosion potential of the *L. ruderale L.* aqueous extract (LRAE) was investigated. It was found that LRAE has an excellent protective properties on carbon steel in highly acidic media include 1.0 mol/L hydrochloric, sulfuric and phosphoric acid solutions giving up to 93.84% efficiency. The adsorption of LRAE on the metal surface obeys the Langmuir model that indicating the formation of a monolayer stable protective film of the inhibitor due to spontaneous physisorption.The results of our investigation suggest that LRAE is one of the most promising candidates for the development of "green" corrosion inhibitors for various industrial and technical applications.

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КЛОПОВНИК СОРНЫЙ (LEPIDIUM RUDERALE L.) ИЗ ЗАПАДНОГО КАЗАХСТАНА - НОВЫЙ "ЗЕЛЕНЫЙ" ИНГИБИТОР КОРРОЗИИ ДЛЯ УГЛЕРОДИСТОЙ СТАЛИ

Аннотация. *В данной работе исследована ингибирующая способность водного экстракта Lepidiumruderale L. (LRAE)относительно коррозии углеродистой стали в типичных кислых и солевых средах с использованием метода потери массы и электрохимических измерений. Установлено, что эффективность ингибитора LRAE достигает 91.05%, 92.03% и 93.84% в растворах 1.0 моль/л соляной, серной и фосфорной кислот соответственно. Обнаружено, что ингибирующая способностьLRAE увеличивается с ростом концентрации. Во всех исследованных средах адсорбция ингибитора на поверхности металла соответствует модели Ленгмюра, а высокая эффективность достигается за счет самопроизвольной физической адсорбции ($\Delta G^0_{абс}$ > -20 кДж/моль). Поляризационные кривые показывают, что LRAE действует как ингибитор смешанного типа и эффективность ингибирования может достигать 95.34%. Электрохимические данные отлично согласуются с результатами гравиметрического эксперимента.*

Ключевые слова: *Lepidiumruderale L.; растительные экстракты; эффективность ингибиторов;«зеленые» ингибиторы; защита металла.*

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БАТЫС ҚАЗАҚСТАННЫҢ АРАМ СЫТЫРМАҒЫ (LEPIDIUM RUDERALE L.)– КӨМІРТАНДЫ БОЛАТ ҮШІН ЖАҢА «ЖАСЫЛ» КОРРОЗИЯНЫҢ ИНГИБИТОРЫ

Аңдатпа. *Бұл жұмыста Lepidium ruderale L. (LRAE) сулы сығындысының типтік қышқылды және тұзды орталарда көміртекті болаттың коррозиясына қарсы ингибиторлық тиімділігі масса жоғалту әдісі және электрохимиялық өлшеулер арқылы зерттелді. LRAE ингибиторының тиімділігі тұз, күкірт және фосфор қышқылдарының 1,0 моль/л ерітінділерінде сәйкесінше 91,05%, 92,03% және 93,84% жететіні анықталды.LRAE концентрацияның көтерілуімен ингибирлеу жоғарылайтының анықталды.Барлық зерттелетін орталарда металл бетіндегі ингибиторлық адсорбция Ленгмюр моделіне сәйкес келеді және өздігінен физикалық адсорбция ($\Delta G^0_{абс}$ > -20 кДж/моль) есебінен жоғары тиімділікке қол жеткізіледі. Поляризация қисықтары LRAE аралас типті ингибитор ретінде әрекет ететінін көрсетеді және тежеу тиімділігі 95,34% дейін жоғары болуы мүмкін. Электрохимиялық деректер гравиметриялық эксперимент нәтижелерімен сәйкес келеді.*

Кілт сөздер: *Lepidium ruderale L.; өсімдік сығындылары; ингибиторлардың тиімділігі; жасыл ингибиторлар; металды қорғау.*