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Inhibitory Effect of Essential Oil from Rond Left Mint (*Mentha Rotundifolia*) on the Corrosion of C35 Mild Steel in an Acidic Environment

Amal Sbai[1], Fatima Z. Talbi[2]*, Hajar El Omari[3,4], Abdelazize Chaouche[1], Ouakki Moussa[5], Karima EL Kabous[1], Mariam Barrahi[1], Fouad El-akhal[6], Meryem M. Alaoui[7,8], Fatiha El Khayyat[9], Mohammed Ouhssine[1]

[1]*Laboratoire d'Agrophysiologie, Biotechnologie, Environnement et Qualité, Faculté des Sciences, Université Ibn Tofail, B.P.133, 14000, Kénitra, Maroc*
[2]*Sidi Mohamed Ben Abdellah University, Faculty of Sciences Dhar El Mahraz, Laboratory of Biotechnology, Conservation and Valorization of Natural Resources (LBCVNR), 30000 Fez,Morocco*
[3]*Natural Resources Management and Development Team, Laboratory of Health and Environment, Faculty of Sciences, Moulay Ismail University, 50000 Meknes, Morocco*
[4]*Ministry of Health and Social Protection, Higher Institute of Nursing Professions and Health Techniques of Fez, Annex Meknes, Morocco*
[5]*Laboratoire d'Ingénierie des Matériaux et d'Environnement, Modélisation et Application, Faculté des Sciences, Université Ibn Tofail, BP 133, Kénitra 14 000, Maroc*
[6]*Higher Institute of Nursing Professions and Health Techniques of Tetouan (annex Al Hoceima), Regional Health Directorate, Mohamed V Hospital, 32000 Al Hoceima, Morocco*
[7]*Laboratory of Biochemistry, Environment and Agrifood, URAC36, Faculty of Sciences and Technologies, Hassan II University Casablanca, BP 146, Mohammedia 20650, Morocco*
[8]*Higher Institute of Nursing Professions and Health Techniques of Fez, Regional Health Directorate, EL Ghassani Hospital, Fez 30000, Morocco*
[9]*Natural Resources and Sustainable Development Laboratory, Faculty of Sciences, Ibn Tofail University, BP133, Kenitra 14000, Morocco*

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ABSTRACT

Metals such as iron are a target of different oxidative agents in acidic environments inducing significant economic losses. Ecological inhibitors of corrosion constitute an excellent alternative to chemical agents. Therefore, the scientific community proceeded to the use of plant extracts, including essential oils as inhibitors, presenting a cost-effective way to reduce steel losses. The present study was conducted to investigate the anticorrosive effect of *Mentha rotundifolia* essential oil in an acidic environment. *Mentha rotundifolia* essential oil was recovered using Clevenger apparatus. Different concentrations of essential oil were prepared (0.5, 1, 1.5, and 2g/L) to examine their anticorrosive impact on C35 steel in an acidic environment using 1M HCl by calculating electrochemical impedance, spectroscopic polarization curves, and weight loss measurement.
The results revealed that the essential oil yield was 1.58%. The EO showed an interesting anticorrosive effect at a concentration of 1g/L. Furthermore, the application of the EO at a concentration of 0.5g/L exhibited a higher anticorrosive effect by increasing double-layer capacitance to Cdl (77.41 μF/cm^2). The findings of the study revealed that *Mentha rotundifolia* essential oil, at varying concentrations, effectively inhibited steel corrosion in an acidic environment.

Keywords: Corrosion, Essential oil, Electrochemical impedance, *Mentha rotundifolia,* Mild steel C35, Spectroscopy polarization

Introduction

Corrosion constitutes a serious challenge for industries that cause the heaviest economic losses.[1] Acidic environments easily accelerate the corrosion process of metals, including iron and alloys. They are the most widely utilized materials in construction, industrial, and other engineering applications. unfortunately, these metals are susceptible to deterioration under acidic conditions.[2] Therefore, metal protection is coming to the forefront to minimize economic losses using different strategies such as chemical and natural agents.[3–9] Corrosion inhibitors could play a pivotal role in preventing and protecting steel corrosion.
Heteroatoms are components widely used as inhibitors in industry, including nitrogen, sulfur, and so on. The mechanism of action of these compounds is based on their ability to adsorb molecules that induce metal corrosion.[6] Nowadays, natural anticorrosive agents have undeniable effectiveness against corrosion.[10] This strategy has gained an important interest because of its beneficial properties such as being environmentally friendly and ecologically acceptable.[11] Plants are an exhaustible source of phytochemicals with multifaceted purposes. Several studies have documented an interesting anticorrosive effect of bioactive compounds isolated from natural products, including fenugreek, henna, olive, jojoba, black pepper, *Optimum viridis*, *Andrographis paniculata* and so on. [3,7–9,12,13]
Within this framework, the present study highlighted the anticorrosive effect of *Mentha rotudifolia* essential oil at different concentrations of mild steel C35 in an acidic environment.

*Corresponding author. E mail: fatimazahra.talbi@uhp.ac.ma; ouhssineunivit@gmail.com
Tel: +212671543020

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Materiel and Methods

Essential oil extraction
The essential oil of *Mentha rotundifolia* collected from Kenitra city during April-September 2019 was recovered using the hydrodistillation method in a Clevenger apparatus. The extraction procedure consists of boiling 100g of dried leaves in ½ L of distilled water for three hours at 350°C. Then, the essential oil was stored in favorable conditions until experimentation.

Preparation of mild steel
The electrochemical investigations were carried out on metal samples of a specific size (surface 1*2 cm). as shown in Table 1, the samples were mechanically polished with various grades of emery paper to achieve a very smooth surface (grit sandpaper between P40 and P2000) as shown in Table 1.

Preparation of acidic solutions
The preparation of acidic solutions consists of blending Merck 37% analytical grade HCl and water to prepare the 1 M HCl. Before each experiment, the solutions were freshly produced by mixing the essential oil directly at different concentration (0.5, 1.5, and 2g/L) (Table 2) or to the corrosive solution.

Weight loss measurement (Gravimetry)
In a solution of the four concentrations, 30 mL of 1 M HCl was each added at room temperature. The five substrates were added to these solutions after weighing (FH-100 and FH-200 include a glass weighing chamber) and the 5th served as a control (the solution did not contain essential oil). After 6 h, the substrate was reweighed to calculate the gravimetric speed and the effectiveness of the essential oil.[14,15]

Speed = % u0394M (Mi -Mf)/ S* T
Efficiency = high speed % u2013l lowspeed/ control speed

Electrochemical measurements and tests
The electrochemical measurements were carried out in a cell of Pyrex glass in three electrodes. The work electrode (work electrod; WE) in the shape of a steel disc shared on 1cm2 surface and embedded in polytetrafluoroethylene (PTEF). A 30 min stabilization period is required before electrochemical measurements in order to achieve a stable corrosion potential value (Ecorr).
The electrochemical impedance measurements were performed using the 5210 function analyzer of the transfer model, driven by the power suite software. After determining the permanent current, the sinusoidal voltage (10mv) at frequencies varies between 10 and 100MkH which is superimposed on the potential of the rest. Computer programs have the ability to automatically check the measurements made of the resting potential after the 30 min stabilization period and at 298K. The impedance diagrams are provided as «Nyquist diagram» and the tests are done three times to ensure reproducibility.

Table 1: The composition of C35 mild steel.

Element other than iron	Carbon	Silicon	Magnesium	Sulfur	Chromium	Titanium	Nickel	Cobalt	Copper
Content Wt (%)	0 .37	0.23	0.68	0.016	0.077	0.011	0.059	0.009	0.160

Table 2: The concentrations of the essential oil used

Concentration in HE (g/L)	Ecor(mv)	Icor (µA cm^{-2})	Bc(mv)	Ba(mv)	IE%
Witness	-498	983	-140	150	-
0.5	-455	212	-120	134	78.
1	-439	93	-105	136	90.5
1.5	-450	108	-115	128	89.0
2	-449	148	-122	123	84.9

Ecorr: Corrosion Potential; Icorr: Current Densities; Bc and Ba: are the exponents of Tafel's laws ; IE%: Inhibition Efficiency ; HE : huile essentielle ; essential oil

Results and Discussion

Gravimetry results
Table 3 presents results of theinhibition efficiency (%IE) and corrosion rate (Wcorr) values obtained from gravimetric measurements of mild steel C35 in the presence or not of the essential oil at different concentrations in an acid solution (HCl 1M) and temperature of 303 K after 6 h of immersion.
The treatment of the obtained results showed a negative correlation between corrosion current (Icorr) and essential oil concentration. The concentration decreased from 983 µAcm^{-2} at 0.5 g/L to 148 µAcm^{-2} at 2 g/L. A similar observation was made for the corrosion potential (Ecorr), whose values increased from -498 mv at 0.5 g/L to 449 mv at 2 g/L. Meanwhile, the %IE increased from 78.4% at 0.5 g/L to a maximum value of 90.5% at 1g/L. Thereafter, this value experienced a decline (84.9% at 2 g/L).
The obtained results from the present study are concordance with those reported by A. khadraoui et al.[16,17] The essential oil under study exhibited the highest inhibition efficiency (87%) at a highest concentration examined (2mL/L). The efficiency of inhibition decreased slightly and eventually reached steady state values. This effect could be ascribed to the chemically dense composition of the essential oil under study, which act as natural adsorbent molecules. Phytochemicals react with different oxidants to create a mass and load transfer barrier that protects the metal surface against corrosion.

The inhibition efficiency E (%), is defined by the following equation:

$$E\% = \frac{(1 - \text{Icorr}) * 100}{\text{Icorr}}$$

Polarization curves
Figure 1 depicts the polarization curves of the cathode and anode of steel C35 in an acidic solution of 1M HCl with or without essential oil at different concentrations. The addition of a natural inhibitor to C53 steel in 1M HCl solution affects the anodic and cathodic reactions. This decreases in the anodic partial current, which corresponds to metal breakdown.
It has been found that the decreasing of Icorr is highly correlated with phytochemicals of *Mentha rotundifolia* that adsorb onto the carbon steel surface. Both the cathodic and anodic branches are affected by the natural oil, while the inhibitor's stronger anodic action ends around 300 mV. This potential, which is in the adsorption rate domain, is known as the desorption potential. These findings suggest that the inhibitor is functioning as a combination that resembles an inhibitor. Furthermore, geometric blocking is thought to cause adsorption when

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Ecorr differs slightly between inhibited and uncontrolled solutions. It was also discovered that the cathodic current-potential curves produce Tafel lines, indicating that the hydrogen evolution reaction inactivated. Tafel cathodic slope values alrer somewhat when oil activated show a slight change with the addition of oil. This result indicates that the mechanism at the electrode reaction level is not affected.

Table 3: Electrochemical parameters and corrosion-inhibiting efficiency of C35 steel in HCl (1 M) with and without the addition of *Mentha rotundifolia* essential oil.

xx	**C (g/L)**	**Ltr (Ω cm^2)**	**Dlc (µF cm^{-2})**	***η*imp/effectiveness %**
HCl	--	35.0	121	-
	0.5	157.4	77.41	77.7
inh	1.0	331.3	33.79	89.4
	1.5	313.5	41.32	88.8
	2.0	228.7	31.56	84.7

C: Concentration; Ltr: Load transfer resistance; Dlc: Capacity of double layer

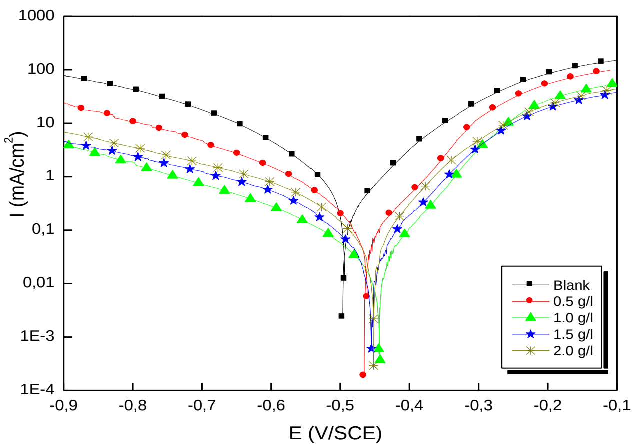

Figure 1: Polarization curves of C35 steel in 1 M HCl medium at different concentrations of *Mentha rotundifolia* essential oil.

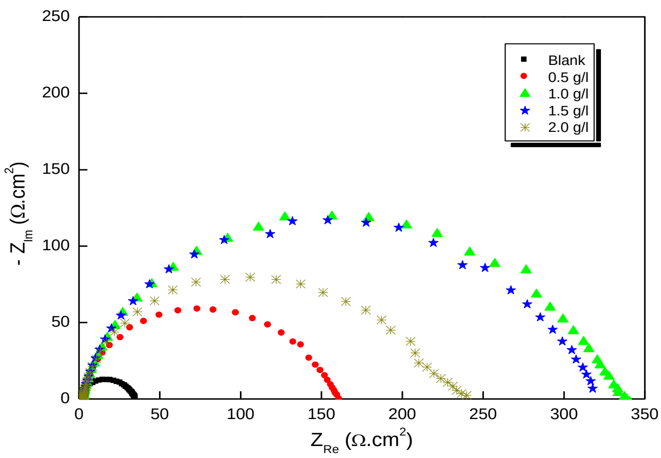

Figure 2: Nyquist diagram of C35 mild steel in 1 M HCl at different concentrations of *Mentha rotundifolia* essential oil.
Dlc: Double layer capacity ; Ltr: Load transfer resistance
Impedance curves

Figure 2 displays the results of the determination of the capacitive half-loop diameter. The analysis of findings revealed that diameter values increased with increasing of inhibitor concentration. The graphic demonstrates the mechanism charge transfer on a solid electrode with heterogeneous and irregular surface that controls the corrosion response. On the basis of values of charge transfer resistance, the inhibitory efficiency was deducted using the following formula:

ERt (%) = (Rt – Rt) Rt×100
Rt and R°t are respectively, load transfer resistance values with and without inhibitor addition.
The values of double layer diameter were exploited to determine the load transfer resistance as follows:

$$Cdl = 12\pi \int \max x\, R_t$$

Cdl : capasity of double layer (µF.cm^{-2})
$\int$ max : the maximum frequency (Hz)
Rt :the load transfer resistance (kΩ.cm^2).

Table 3 represents the results of the impact of essential oil concentration on double layer Cdl. The analysis of the obtained results demonstrated that essential oil has a positive effect on the capacity of the double layer. It is clearly seen that the elevation of essential oil concentration correlated negatively with the capacity of the double layer Cdl (Table 3). The Cdl decreased from 77.41 Uf/cm^2 at a concentration of 0.5 g/L to 31.56 Uf/cm^2 at a concentration of 2g/L. However, a significant increase of the load transfer resistance was observed (157.4 Ohm/cm^2 at 0.5 g/L to 228.7 Ohm.cm^2 at 2 g/L of the inhibitor). This results could be explained by the adsorption effect acted by bioactive compounds contained in the essential oil, which are implicated in the iron surface protection. It has been documented that adsorption effect of the inhibitor is involved in the Cdl decrease process throughout the formation of an iron surface complex preventing the oxidant attack.[18,19] Furthermore, the efficiency of corrosion inhibition is highly related to the inhibitor concentration. It is worthy to be noted that the weight loss measurements yielded results that were consistent with the electrochemical study. The most common type is the constant phase element (CPE), which has a noninteger power dependence on frequency. As a result, the equivalent circuit depicted in Figure 2 is utilized to evaluate the impedance spectra, where Rs represents the solution resistance and Rt denotes the resistance.

The effect of temperature on the inhibitory efficiency of Mentha rotundifolia essential oil
The results of the temperature effect on the inhibitory efficiency of essential oil under study are depicted in Figure 3. It is clearly seen that temperature increase (25°C to 55°C) induced an elevation of the current densities. Furthermore, a significant decrease of corrosion potential was observed that indicating the beneficial impact of *Mentha rotundifolia* essential oil as an effective corrosion natural inhibitor. Table 4 shows the obtained results of corrosion current density, corrosion potential and inhibitory efficiency.

Thermodynamic parameters
Table 5 displays the results of the thermodynamic parameters, including Ea, Sa, and Ha for mild steel dissolution reaction in 1 M HCl. The application of essential oil increased significantly all studied parameters (Table 5) Whereas, the obtained results from the current study are disaccord with those reported.[16,20–25] According to the literature, the shift from physical adsorption to chemisorption is responsible for the fluctuation of Ea with concentration, which varies by a higher value less than a solution.

Conclusion

The present study was undertaken to examine the anticorrosive effect of *Mentha rotundifolia* essential oil at different doses under acidic conditions. The results revealed that the essential oil under study exhibited excellent inhibition activities against the corrosion of C35 steel in an HCl solution. The cathode curves in the form of Tafel, indicate that the hydrogen fixed on the surface of the metal in this acid solution can present an active mechanism. As the inhibitor concentration increases, the corrosion current density decreases. The effectiveness of this inhibitor is due to the adsorption of CL- on the metal surface. *M. rotundifolia* essential oil could be useful as an alternative natural anticorrosive agent.

Table 4: Inhibitory efficiencies and electrochemical parameters of steel35 in 1 M HCl in the absence and presence of the inhibitor (2 g/L) at different temperatures

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Electrochemical parameters	Temp K	Ecorr (mV/ECS)	Icorr (µA/cm²)	Bc mv	Ba mv	E%
Blank	298	-498	983	-140	150	-
	308	-491	1200	-184	112	-
	318	-475	1450	-171	124	-
	328	-465	2200	-161	118	-
	298	-439	93	-105	136	90.5
	308	-436	139	-186	105	88.4
Inhibitor	318	-435	213	-168	113	85.3
	328	-444	388	-178	149	82.3

Temp: Temperature ; Bc, Ba: the exponents of Tafel's laws; Ecorr: Corrosion Potential; E%: Effeciency; Icorr: Current Densities.

Table 5: Thermodynamic parameters

Medium	Concentration	Ea(KJ/mol)	ΔHa(KJ/mol)	ΔSa(KJ/mol.K)
	Blank	21	18.5	-126
1 M HCl	1.0 g/L of inh	38	35.5	-88.3

Ea: Activation energy; ΔHa: The enthalpy of activation; ΔSa: The entropy of activation.

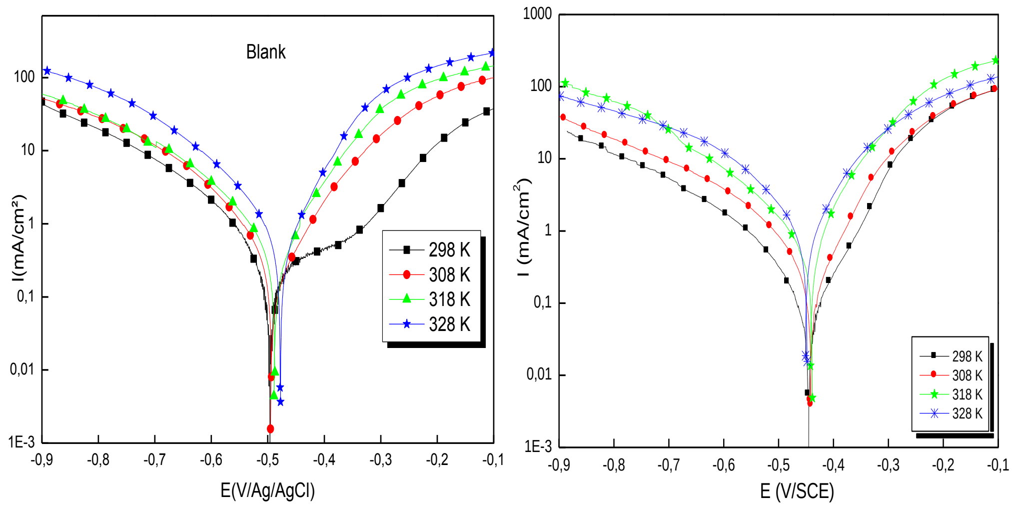

Figure 3: Polarization covers made for C35 in the 1 M HCl solution and at 2 g/L of the essential oil of *Mentha Rotundifolia* at different temperatures.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.\

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