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Research Article

Inhibition of Mild Steel Corrosion in 1M Hydrochloric Medium by the Methanolic Extract of *Ammi visnaga* L. Lam Seeds

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The chemical composition of the methanolic extract of *Ammi visnaga* (Khella) seeds from the Sidi Slimane region is determined for the first time by Gas Chromatography coupled with Mass Spectrometry (GC/MS). Ten compounds representing 99.638% of the total extract were identified. Khellin (49.011%), Visnagin (26.537%) and Dimethylethylamine (15.108%) are the major components. Moreover, the inhibitory effect of the Methanolic extract of the seeds of *Ammi visnaga* on the corrosion of mild steel in a solution of 1M HCl is determined using weight loss measurements, the potentiodynamic technique as well as the technique of electrochemical impedance spectroscopy (EIS). It is found that the extract reduces the corrosion rate of the steel in the acid solution. Inhibition efficiency increases as the concentration of the extract increases. The tested compound has an inhibition efficiency of 84% for a concentration equal to 1.0 g/L. The polarization measurements indicate that the examined extract acts as a mixed inhibitor with predominant anodic efficacy. The data obtained from EIS studies are analyzed to model this process using appropriate equivalent circuit models. The adsorption of the extract on the surface of the mild steel obeys the Langmuir adsorption isotherm in acidic medium and the activation is determined and discussed.

1. Introduction

In the last few decades, mild steel (MS) is widely applied as a constructional material in a large number of industries due to its excellent mechanical properties and it's exceptionally low cost [1, 2]. Although, mild steel finds a wide range of technological applications, its poor corrosion resistance in the acid solution, restrains its utility. In addition, mild steel is low natural stability and is profoundly degraded in the mineral acid environment such as HCl, H_2SO_4, HNO_3, etc., [3, 4]. For instance, hydrochloric acid solutions are commonly used for pickling, industrial acid cleaning, acid de-scaling, and oil well-acidifying processes [5–7]. Because of the aggressiveness of acid solutions, mild steel corrodes severely during these processes, particularly with the use of hydrochloric acid, which results in a terrible waste of both resources and money [8, 9]. Therefore, the use of inhibitors is one of the usual methods of protecting metallic materials against corrosion in acidic environments, i.e., inhibitors compound is often added in the acid solutions to minimize the corrosion of mild steel in these processes [10–16]. Moreover, in the corrosion studies, the selection of a good corrosion inhibitor is controlled by its economic availability, its efficiency to inhibit the substrate material and its environmental side effects. The majority of synthetic compounds have good anticorrosion action, but most of them are highly toxic to humans and the environment [17]. Therefore, due to environmental concerns, different inhibitors extracted from natural plants (vegetable oils or plant tannins) have allowed researchers to achieve high inhibitory efficiency values. In recent years, plant extracts as corrosion inhibitors attracted great attention due to their properties like low cost, environment-suitability, renewability and also due to the cost effectiveness and simplicity of the methods utilized during the extraction of these plants [18]. In this context, the literature

Table 1: Chemical composition of mild steel.

Elements	C	Si	Mn	S	Cr	Ni	P	Fe
Composition (w%)	0.14–0.20	0.24	0.5	0.05	0.1	0.01	0.05	Rest

review reveals that exclusive and extensive works have been done in the research areas related to plant leaves, bark, and stem as corrosion inhibitors for steel in HCl medium like Ylang-ylang [19], Kimbiolongo [20], Carvi [21], Nypa fruticans Wurmb [22], *Osmanthus fragran* [23], *Phyllanthus amarus* [24], *Tabernaemontana divaricata* [25], *Pimenta dioica* [24], *Bryophyllum Pinnatum* [26]. Furthermore, the existing literature discovered that adsorption of corrosion inhibitors takes place through adsorptive interactions between concerned compounds and metal surface. Generally, organic compounds containing nitrogen, sulfur and/or oxygen atoms and polar functional groups are considered to be good corrosion inhibitors in wet corrosion environments [9, 27, 28]. In the present work, we chose *Ammi visnaga* L. Lam as a corrosion inhibitor, an annual plant of the family Apiaceae, grown in many parts of the world such as Europe, Asia, and Africa. The aqueous extraction of its seeds is used for the treatment of several diseases without effects [29]. The aim of this study is to evaluate the inhibitory action of the methanolic extract of Khella seeds as well as its mode of action on mild steel in a solution of 1.0 M HCl using several methods including weight loss measurements, adsorption isotherms, potentiodynamic polarization (PDP), and electrochemical impedance spectroscopy (EIS). As mentioned above, the choice of this metal is justified by its conductive properties and its lower cost in comparison with other more conductive metals such as gold or silver.

2. Material and Methods

2.1. Origin of the Plant. The plant is collected in October 2015, in the region of Sidi Slimane, which is a predominantly rural subdivision of the Rabat-Salé-Kenitra region in Morocco.

2.2. Preparation of the Extract. The seeds of *Ammi visnaga* L. Lam. are milled in a blender and kept until use. The powders (20 g) are Soxhlet extracted with 99.8% methanol (300 ml) for 4 hours. The solution obtained is removed from the filtrate by evaporation under reduced pressure in a rotavapor. A crude extract characterized by a dark brown color is recovered.

2.3. Chemical Composition. The device used is a Bruker 456 GC Triple Quadrupole EVOQ equipped with an 8400 series auto-injector (Bruker, Germany). The system is equipped with a capillary column type RXI-5SIL MS (30 m × 0.25 mm × 0.25 μm film thickness, Bruker, Germany). The temperature is set from 35 to 300°C at 5°C min^{-1} and then held at 300°C for 10 minutes. Helium gas is used as a carrier gas with a constant flow rate of 1.5 ml min^{-1}. A sample of 1.0 μl is automatically injected into the nondivided mode. The temperature of the MS interface is 280°C. For CG mass detection, an electron ionization system with ionization energy of 70 eV is used and the scanning range is 10^{-600} amu. The identification and percentage composition of the compounds are performed using the MS library and the NIST 2014, 11th edition and Wiley 5th edition spectrometer data bank.

2.4. Corrosion Tests

2.4.1. Preparation of Mild Steel. The material used as working electrode in this study is mild steel whose chemical and mass composition is given in Table 1.

To have a good reproducibility of measurements, it is necessary to have a clean surface state. The surface of the samples undergoes mechanical polishing on abrasive papers (carbon, silica) of increasing particle size ranging from 80 to 1200 mm followed by rinsing with distilled water and drying.

2.4.2. Preparation of the Corrosive Solution. The corrosive solution consists of a molar solution of 1M hydrochloric acid (1 mol L^{-1}) prepared from a commercial solution of hydrochloric acid (37%) using bidistilled water.

2.4.3. Gravimetric Measurements. The principle of this method is based on the measurement of the weight loss ΔP experienced by a surface sample S, during the time t of immersion in the corrosive solution, in the absence and in the presence of the inhibitor, maintained at a constant temperature. The gravimetric tests are carried out in a double-walled cell equipped with a condenser and a thermometer. However, a circulating water thermostat keeps the electrolyte at the desired temperature. As well as the electrolyte volume is 100 ml, and the samples are in rectangular form of dimensions 2 cm × 2 cm × 0.2 cm. Before any measurement, the samples are polished with sandpaper of decreasing grain size up to 1200 followed by washing with distilled water and acetone and drying in air. After weighing accurately, the samples are immersed in beakers containing 100 ml of acid solutions without and with different concentrations of the extract at a temperature of 303 K for 12 hours immersion time. At last, each value of the gravimetric tests is the average of three trials.

2.4.4. Electrochemical Studies. Potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS) tests are carried out by a Volta lab potentiostat (Tacussel-Radiometer PGZ 100) controlled by the Voltamaster 4 software. This assembly has three electrodes: mild steel as working electrode (ET), platinum as auxiliary electrode (CE) and Ag/AgCl electrode as reference electrode. The working electrode is immersed in the test solution for 30 min until a steady-state open circuit potential (E_{ocp}) is established. The intensity-potential curves or polarization curves of the metal/solution interface are obtained in potentiodynamic mode. The potential applied to the sample varies continuously from −800 to −200 mV vs. ECS, with a sweep rate of 30 mVmn^{-1}. The intensity of the

Table 2: Chemical composition of the methanolic extract of the seeds of *Ammi visnaga*.

No.	Name	Retention time	% of Total
1	*Dimethylethylamine*	5.302	15.108
2	2,4-Dimethyl-2-pentene	5.364	0.070
3	3-oxo-3-(2,2,2-trimethyldiazan-2-iumyl)-1-propanolate	7.439	1.493
4	*Visnagin*	77.696	26.537
5	5-Acetyl-2-amino-3-cyano-4-(5'-H-2'-furyl)-6-methyl-4H-pyran	80.937	0.147
6	Lavandulyl acetate	84.614	0.672
7	*Khellin*	86.217	49.011
8	2-[(3-Cyano-6,7-dihydro-5H-cyclopenta[b]pyridin-2-yl) sulfanyl]-N-phenylacetamide	92.468	1.732
9	Edulisin III	103.465	2.763
10	Binapacryl	106.568	2.105
	Total		99.638

current is measured between the working electrode and the platinum counter-electrode. Before drawing these curves, the working electrode is maintained at its abandonment potential for 10 minutes. For impedance measurements, the amplitude of the sinusoidal disturbance applied to the dropout potential is chosen to satisfy the linearity conditions (10 mV peak-to-peak). The frequencies scanned during these impedance measurements go from 100 kHz to 10^{-1} Hz at the rate of 5 points per decade. The direct current (DC) voltage was taken from the E_{corr} to the reference electrode obtained from the OCP.

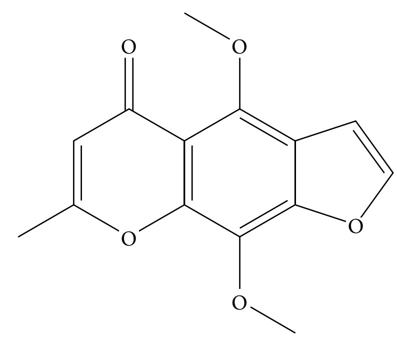


Figure 1: Chemical structure of Khellin.

3. Results and Discussion

3.1. Identification of Compounds. The analysis of the methanolic extract of the Khella seeds by GC/MS made it possible to identify 10 compounds representing 99.638% of the total extract dominated by three compounds whose proportion is greater than 15%: Khellin (49.011%), Visnagin (26.537%) and Dimethylethylamine (15.108%). The other constituents are present in small quantities (<3%). We also note some compounds detected for the first time in our species such as dimethylethylamine as major compounds (Table 2). This variation can be attributed to ecological and/or genetic factors, which influence the plant biosynthetic pathways and consequently the relative proportion of the main common compounds [30].

The common feature of the chemical composition of khella seeds is its high prevalence of furanochromones (Khellin and Visnagin) (Figures 1, 2), which has been the subject of several research studies [31, 32].

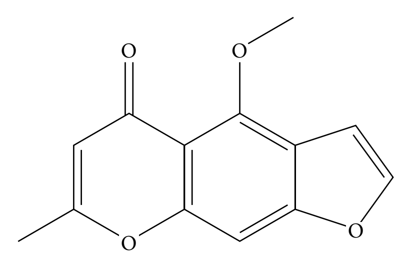


Figure 2: Chemical structure of Visnagin.

3.2. Gravimetric Study. The concentration effect is determined by the immersion of the substrates in the corrosive solution, without and with the addition of the methanolic extract of Khella seeds at different concentrations (0.2, 0.4, 0.6, and 1 g/l). The inhibitory efficacy is determined after 6 h at 303 K. The corrosion rate (W) and the inhibition efficiency are determined using Equations (1) and (2) given below:

$$W : \frac{W_b - W_a}{At}, \quad (1)$$

$$EI(\%) = \left(\frac{(W - W_{\text{inh}})}{W}\right) \times 100, \quad (2)$$

where W_b and W_a are the weights of the samples before and after immersion in the tested solution,

W, W_{inh}, respectively, represent the corrosion rates of the steel after immersion in the absence and in the presence of the inhibitor.

On the surface of the mild steel specimen (cm^2) and t is the exposure time (h).

The values of the corrosion rate (W) and the inhibitory efficiency (EI%) obtained by the weight loss method at different concentrations of the extract are summarized in Table 3.

Table 3: Corrosion parameters obtained from weight loss measurements for carbon steel in 1.0 M HCl containing various concentration of EMG at 303 K.

Inhibitor	Concentration (g/L)	Corrosion rate (mg/cm^2 h)	Inhibition efficiency (%)	Surface average (θ)
Blank	-	1.135	-	-
EMG	0.2	0.4995	56	0.56
	0.4	0.3519	69	0.69
	0.6	0.2611	77	0.77
	1	0.1929	83	0.83

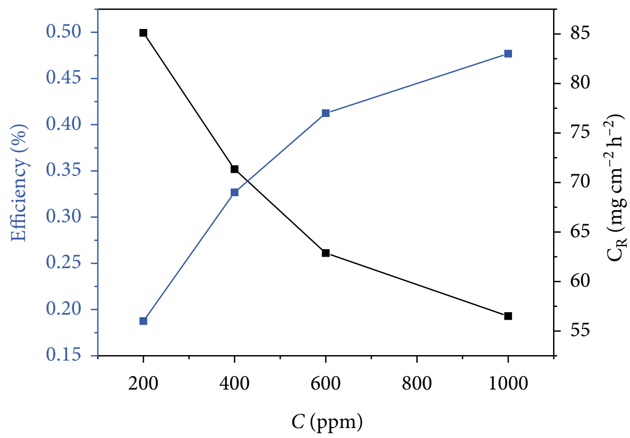


Figure 3: Variation in corrosion rate and inhibition efficiency of mild steel in 1.0 M HCl with different concentrations of EMG after 6 hours of immersion.

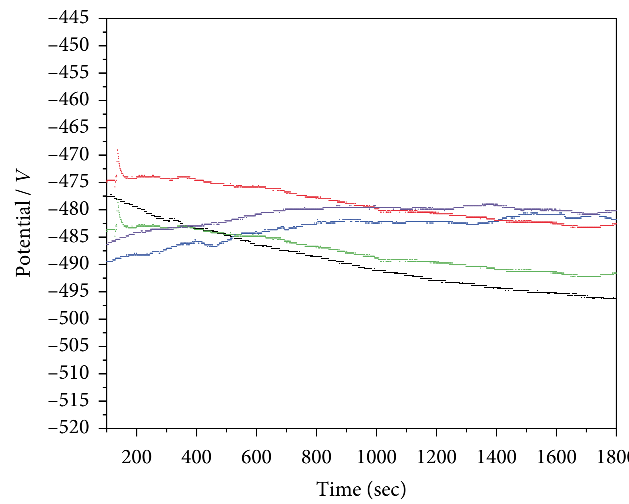


Figure 4: OCP of mild steel electrode in 1.0 M HCl at 303 K without and with different concentrations of inhibitors.

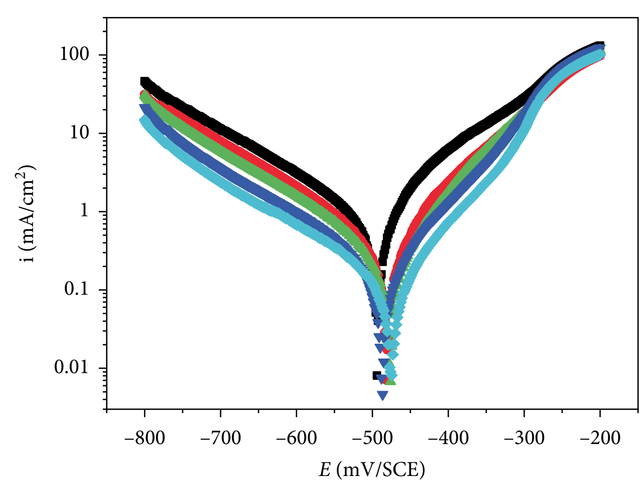


Figure 5: Polarization curve of mild steel in 1M HCl with and without addition of different concentrations of EMG at 303 K.

The methanolic extract of khella seeds inhibits the corrosion of steel in all concentrations tested. Moreover, the rate of corrosion decreases constantly when the concentration of inhibitor increases, while the inhibitory efficiency increases when the concentration increases to 84%, at a concentration of 1 g/l (Figure 3). This behavior can be attributed to the increase in the area covered by adsorbed molecules on the steel surface, which reduces the direct contact between steel and the corrosive environment. However, the mechanism of the inhibitory effect of khilah seed extract was studied by comparing the complexing capacity of kellin and visnagin compounds. Which conductimetric titrations have shown the possible formation of Fe-khellin or Fe-visnagin complex, which is generally attributed to chemisorption or chemical bonding between iron and inhibitory molecules [33].

3.3. Polarization Curve. Prior to the electrochemical analysis, the mild steel electrode was immersed in the corrosion solution for 1800 s in order to establish a steady state Open Circuit Potential (OCP) (Figure 4). For PDP measurements, the cathodic and anodic polarization curves of mild steel in 1M HCl medium in the absence and in the presence of different concentrations of the methanolic extract of the seeds of *Ammi visnaga* L. are presented in Figure 5 below. These are obtained after 6 hours of immersion in Ecorr and at a temperature of 303 K.

Table 3 groups together the values of the electrochemical parameters determined from the previously obtained polarization curves, namely the corrosion current density icorr, the corrosion potential Ecorr, the slopes of cathode tafel bc and anodic ba as well as the inhibitory efficiency of corrosion E% defined as follows:

Table 4: Electrochemical parameters and corrosion inhibitory efficacy of steel in 1M HCl without and with addition of EMG at 303 K.

Inhibitor	Conc. (g/L)	$-E_{corr}$ (mV/SCE)	$-\beta c$ (mV dec^{-1})	βa (mV dec^{-1})	i_{corr} (μA cm^{-2})	η_{Tafel} (%)
Blank	-	496.0	87.5	139.7	576.0	-
EMG	0.2	481.2	68.4	139.5	276.36	51
	0.4	482.3	79.1	146.9	186.12	67
	0.6	491.6	81.7	147.2	141	75
	1	480.6	78.1	142.4	101.52	82

$$EI\% = \frac{\mathring{i}_{corr} - i_{corr}}{\mathring{i}_{corr}} \times 100. \qquad (3)$$

$\mathring{i}_{corr}$ and i_{corr} respectively correspond to the corrosion current densities in the absence and in the presence of the inhibitor at different concentrations. These densities are determined by extrapolation of the Tafel straight lines to the corrosion potential. The values of the cathodic slopes bc vary delicately with the addition of the green inhibitor, unlike the anode slopes ba which fluctuate a little more considerably, which declares our extract as a mixed inhibitor, with an anodic predominance. As well as, this inhibitor reduces the dissolution of mild steel and slows down the evolution reaction of hydrogen. In fact, the inhibitory efficacy increases with the inhibitor concentration and reaches 82% at 1 g/l (Table 4). This indicates that the methanolic extract of *Ammi visnaga* probably acts by blocking the active sites of the metal surface by forming a barrier for the transfer of charge and mass which leads to the decrease of the interaction of the metal with the corrosive environment by delaying the rate of corrosion.

3.4. Electrochemical Impedance Spectroscopy. The electrochemical impedance diagrams are read at the corrosion potential, in different concentrations and temperatures with an immersion time. The measurements are made in the frequency range 100 kHz–100 mHz. The Nyquist curves are presented in Figure 5. The corrosion behavior of steel in the 1M HCl corrosive solution in the absence and in the presence of different concentrations of the methanolic extract of *A. visnaga* seeds is studied by the EIS method at 303 K after 6 hours of immersion (Figure 6). Nyquist curves include a depressed capacitive semicircle indicative of a double layer capacitance, this depression may be the result of a heterogeneous surface that results from the surface roughness, the distribution of the inhibitor [34, 35], or the formation of porous layers [36, 37]. Indeed, a remarkable increase in the diameter of the semicircle is observed with the presence of the inhibitor which causes the adsorption of the extract on the surface of the steel [38]. This can be attributed to the presence of furanochromones (Khellin and Visagin) that could cover the surface of the steel and trap the antioxidant molecules chelating the Fe^{2+} cations. The extract of *Ammi visnaga* also has an effect on the corrosion of SX 316 steel in an acidic solution by formation of kellin iron complex. The metal/solution interface is represented by the equivalent circuit (Figure 7), the polarization resistance values R_p and the double layer capacitance shown in Table 5. The CPE in Figure 7 represents the constant phase element to be used in place of the double layer capacitance (C_{dl}). The inhibition efficiency is calculated by the equation below (4):

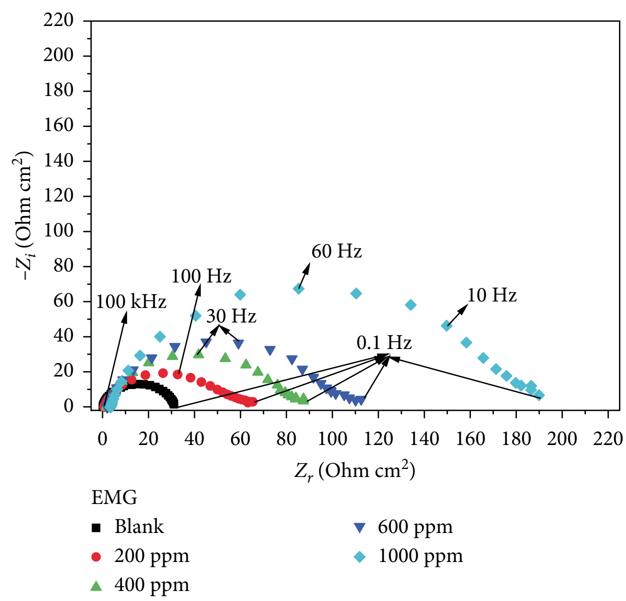


Figure 6: Nyquist diagrams of steel in 1 M HCl without and in the presence of different concentrations of EMG.

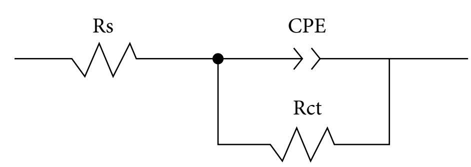


Figure 7: Equivalent electrical circuit corresponding to the corrosion process on the carbon steel in hydrochloric acid.

$$IE_{eis}\% = \frac{R'_p - R_p}{R'_p} \times 100, \qquad (4)$$

where R'_p and R_p are the polarization resistances in presence and absence of inhibitor respectively.

In addition, to examine the corrosion behavior of mild steel, Bode plots have been recorded for mild steel in 1M HCl in the absence and presence of each concentration of our studied compound. The phase angle plots and Bode impedance magnitude are shown in Figure 8. Generally, Bode plot gives the general idea for the anticorrosion activity of tested inhibitor. As we all know, when inhibitors possess a high resistance, the current mostly passes through a capacitor, therefore, phase angle would be near 90° [39]. In contrast, when the inhibitor resistance is low, i.e., current mostly passes through the resistor and hence, phase angle would be near 0° [39]. The Bode spectra at different concentrations of the

Table 5: Impedance parameters for corrosion of carbon steel in 1.0 M HCl in the absence and presence of different concentrations of EMG at 303 K.

Inhibitor	Conc (g/L)	R_p (Ω cm^2)	n	Q ($\mu s^n \Omega^{-1}$ cm^{-2})	C_{dl} (μF cm^{-2})	η_{EIS} %
Blank	-	29.35	0.91	176.10	91.63	-
EMG	0.2	62.46	0.88	152.94	81.11	53
	0.4	83.87	0.87	146.45	75.89	65
	0.6	104.83	0.85	129.65	60.72	72
	1	183.45	0.87	66.95	34.69	84

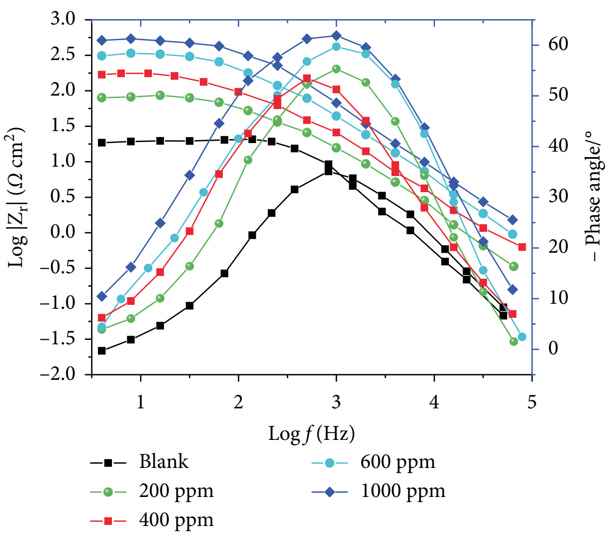


Figure 8: Bode [log (Freq/Hz) vs. log (| Zr | /ohm.cm2)] and phase angle [log (Freq/Hz) vs.–Phase/deg] plots of impendence spectra for mild steel in 1 M HCl in presence and absence of EMG compound at 303 K.

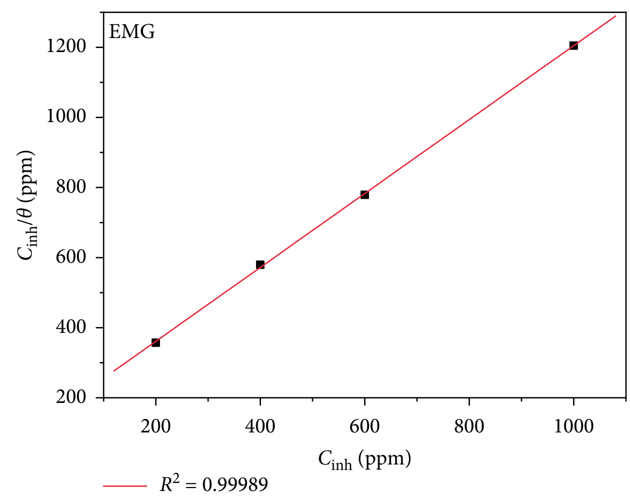


Figure 9: Langmuir adsorption of EMG on the mild steel surface in 1.0 M HCl solution at 303 K. EMG is the methanolic extract of the seeds.

investigated inhibitor showed that when the concentration of the inhibitor increases, phase angle increases meaning the formation of a protective film over the mild steel surface [40]. Moreover, in the present study, the phase angle values are less than 90°, which signified the non-ideal behavior of the capacitor. It is also evident from Figure 6 that the phase angle in the absence and presence of inhibitor is 35° and 62° (at 1000 ppm) respectively. Finally, the findings obtained from Bode spectra also indicate the higher performance of the methanolic extract of *A. visnaga* seeds to protect the metal surface (direct relation between phase angle and inhibitor performance), which is attributed to the formation of a protective layer on mild steel surface leading to a successful retardation of the corrosion phenomenon [41].

3.5. Adsorption Isotherm. The values of the recovery rate (θ) with different concentrations of EMG obtained from the gravimetric study are used to determine the isotherm corresponding to the adsorption process of the inhibitor. The recovery rate (θ) of the surface by the molecules of the inhibitor, are determined by the Equation (5):

$$\theta = \frac{W_0 - W_{inh}}{W_0}, \quad (5)$$

where W_0 and W_{inh} respectively represent the values of the corrosion rate of the steel after immersion in the absence and in the presence of the inhibitor.

According to the Langmuir isotherm, θ is related to the inhibitor concentration C_{inh} by the following equation (6):

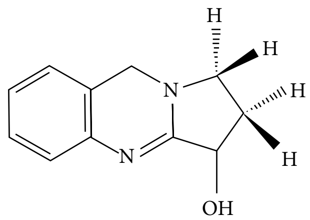
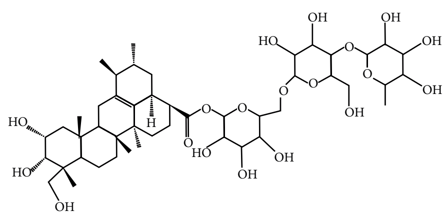
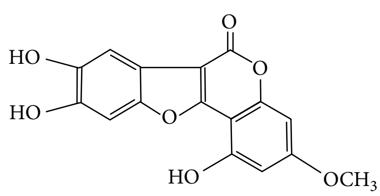
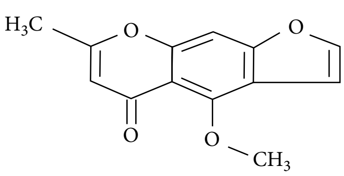
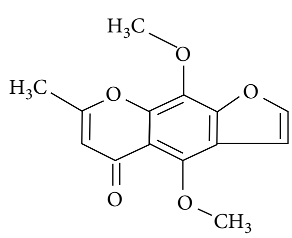
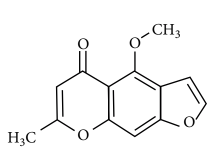
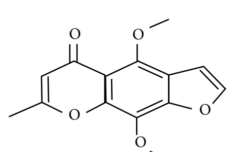
$$\frac{C_{inb}}{\theta} = \frac{1}{K_{ads} + C_{inh}}, \quad (6)$$

where K_{ads}: equilibrium constant of the adsorption process.

The variation of the ratio C_{inh}/θ as a function of the inhibitor concentration is illustrated in Figure 9. The variation C_{inh}/θ is linear, which clearly indicates that the adsorption mechanism follows the langmuir isotherm. This indicates the adsorption of the inhibitory molecules on the metal surface as a film insulating the metal from the aggressive environment.

3.6. Comparison with Similar Extracts and the Proposed Corrosion Inhibition Mechanism. In recent years, great efforts have been devoted to studying the inhibition performance of inhibitors to find efficient and environmentally safe corrosion inhibitors for mild steel corrosion. In this context, several studies have been developed by many organic compounds extracted from natural plants in order to stop the dissolution of the metal surface generated by corrosion process. Table 6 compares the inhibition performance of the extract of *Ammi visnaga* seeds investigated with similar molecules extracted from natural plants used as corrosion inhibitors for steels in

Table 6: Comparison of the inhibition efficiency of the seeds of *Ammi visnaga* with similar molecules studied previously.

Inhibitor	Metal/medium	Inhibition efficiency (%)/reference
	Mild steel/1.0 N HCl	97.2 [42]
	Mild steel/1.0 N HCl	98.3 [42]
	Mild steel/1.0 N HCl	85.7 [42]
Emilia sonchifilia leaves extract	Mild steel/1.0 M HCl	65.69 [43]
Leaf extract of *Khaya senegalensis* (Mahogany)	C-steel/1.0 M HCl	87.41 [44]
Extract of *Pterocarpus soyauxii Taub* leaves	Mild steel/2.5 M Na_2CO_3	70.05 [45]
Nerium Oleander Extract	Carbon Steel/1.0 M Na_2CO_3	89.4 [46]
	SX 316 steel/2.0 M HCl	95.83 [29]
	SX 316 steel/ 2.0 M HCl	98.27 [29]
 	Mild steel/1.0 M HCl	84.0 [Present work]

HCl, H_2SO_4, and Na_2CO_3 solutions. It can be noted that the plants extract show good inhibitive performances for steels corrosion, which explains the recent interest in developing and evaluating new plant extracts for corrosion inhibition studies.

The methanolic extract of the seeds of *Ammi visnaga* reveals the presence of heteroatoms that come from Visnagin, Khellin, hydrocarbons and aromatic rings from other phytoconstituents of the extracts. The adsorption of inhibitor molecules present in the extracts blocks the surface of the mild steel against charge and mass transfer and thereby hindering corrosion. In HCl medium, the anions, which are chloride ions, in this case, were adsorbed into the electrode's surface by partial charge [47]. Bearing in mind that the mild steel surface is positively charged in HCl solution, the protonated molecules can adsorb on the surface of the mild steel by the anions as a linking bridge [48], i.e., electrostatic attraction. It is then clear that physisorption is the first adsorption mechanism, and then the chemisorption mechanism can take place through the sharing of electrons between heteroatoms, pi-electrons of the benzene rings and vacant d-orbital of iron.

4. Conclusion

(i) The major constituents of the methanolic extract of khella seeds are: Khellin (49.011%), Visnagin (26.537%), and Dimethylethylamine (15.108%).

(ii) The methanolic extract of the seeds of *Ammi visnaga* is a good inhibitor of mild steel in hydrochloric medium.

(iii) The percentage inhibition efficiency reaches 84% for a concentration equal to 1.0 g/L.

(iv) The studied extract acts as a mixed inhibitor with predominant anodic efficacy.

(v) The inhibition action of the alcoholic extract is attributed to the majority identified compounds such as furanochromones (Khellin and Visnagin).

(vi) The inhibition effect is achieved by the adsorption of furanochromones on the surface of the steel.

(vii) The adsorption of the extract on the steel in 1M HCl obeys the Langmuir adsorption isotherm model.

Data availability

No data were used to support this study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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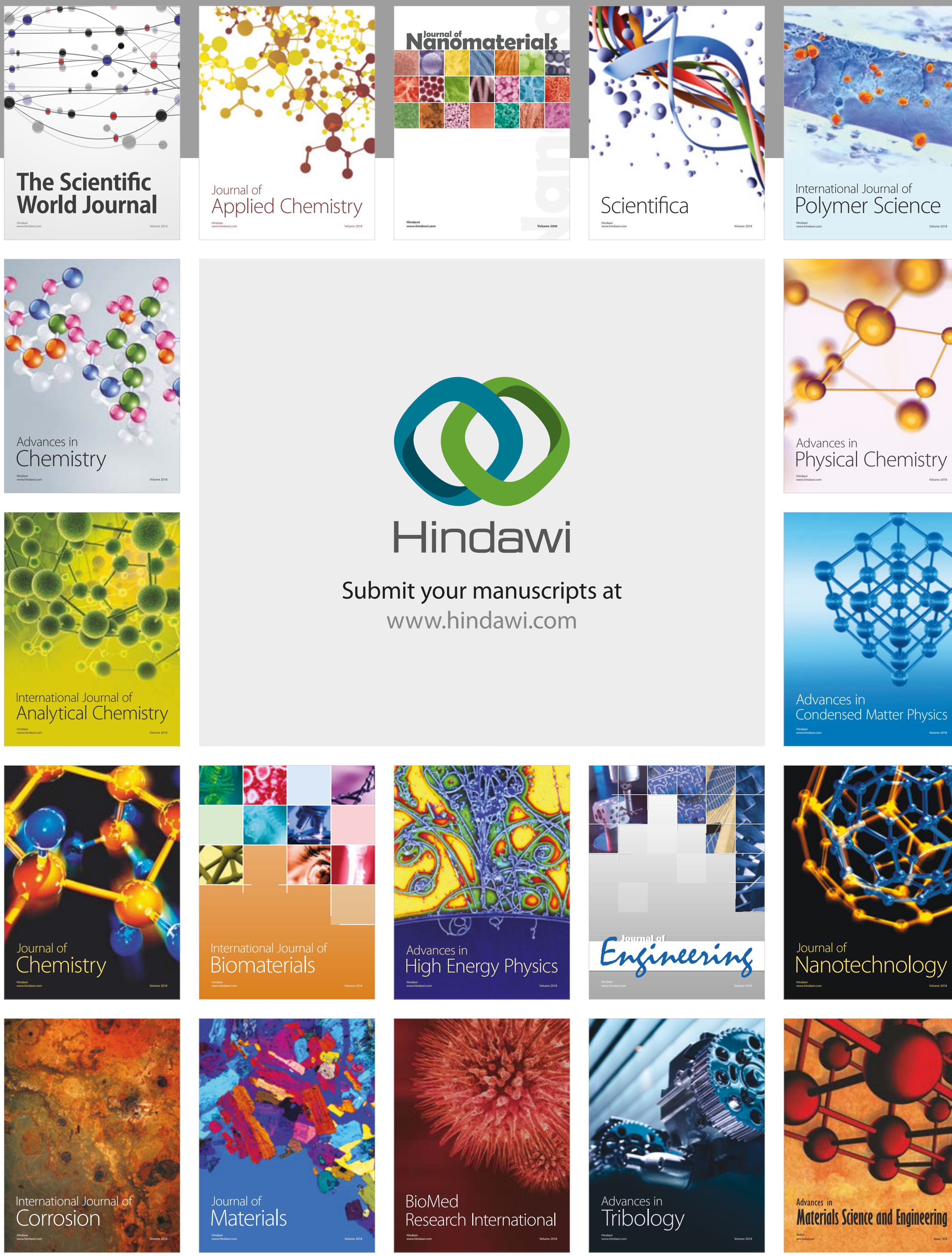
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