



## Extraction, characterization and anti-corrosion activity of *Mentha pulegium* oil: Weight loss, electrochemical, thermodynamic and surface studies



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### ABSTRACT

The corrosion inhibition of carbon steel in 1M HCl solution by *Mentha pulegium* oil was investigated using weight loss, potentiodynamic polarization and electrochemical impedance spectroscopic techniques (EIS). Surface characterization was performed using optical profiler images and AFM analysis. The effect of temperature on the corrosion behavior of carbon steel was studied in the range of 298–338 K. Inhibition efficiency of 81% was achieved with 3 mL/L of *M. pulegium* oil at 298 K. The adsorption of the inhibitor molecules on the steel surface obeys the Temkin adsorption isotherm and involves physical adsorption. The thermodynamic calculation results indicate strong interaction between inhibitor molecules and carbon steel surface. The polarization studies showed that the oil acts as mixed-type inhibitor with predominance cathodic. Double layer capacitance, ( $C_{dl}$ ) decrease indicates that a layer was formed indicating the formation of a surface film. This reflects that the inhibitor does retard the corrosion rate.

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### 1. Introduction

The study of carbon steel corrosion is a subject of both academic and industrial importance because of the increased industrial applications of acid solutions [1,2]. In recent times, corrosion prevention favors the use of environmentally friendly chemicals with low or zero toxicity. Due to the toxicity of most inorganic corrosion inhibitors, such as chromate and phosphate, the use of organic molecules and plant extracts as corrosion inhibitor is becoming increasingly popular. Recently, environmental regulations have limited the use of toxic corrosion inhibitors. Thus, natural products (e.g. vegetable oil) and other environmentally-friendly materials, have gained much attention as a possible replacement to inorganic corrosion inhibitors because they are cheap, biodegradable, and in high abundance.

The inhibition actions of these inhibitors are usually attributed to their interactions with the metal surface via physical or chemical adsorption processes [3], which takes place through the replacement of water molecules by organic inhibitor molecules from the metal surface

[5]. In general, the adsorption of an inhibitor on a metal surface depends on the nature and the surface charge of the metal, the adsorption mode, its chemical structure, and the type of the electrolyte solution [4].

The study of the relationship between adsorption and corrosion inhibition is of great importance; since the corrosion inhibition is a surface process and the degree of protection of metal is a function of adsorption [6,7]. It is generally accepted that organic compounds containing heteroatoms with high electron density such as phosphorus, nitrogen, sulfur, and oxygen as well as those containing multiple bonds are effective corrosion inhibitor [8–10]. Literature report indicates that molecules containing both nitrogen and sulfur in their molecular structure exhibited greater corrosion inhibition efficiency in comparison with those containing only one of these atoms [11–15].

Natural products have been reported as corrosion inhibitors for different metals in various environments [16–31]. The significance of this area of research is primarily due to the fact that natural products are environmentally friendly and ecologically acceptable. The yield of these natural products as well as the corrosion inhibition abilities of the plant extracts vary widely depending on the part of the plant and its location. *Mentha pulegium* used in the present investigation is a native and perennial aromatic herb of the Mediterranean region, but it is also widely grown in many parts of the world mostly in temperate

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and tropical countries. *M. pulegium* is a species of flowering plant belonging to the family of *Lamiaceae*. The leaves exhibit a very strong fragrance similar to spearmint. It is a traditional culinary herb used for folk remedy. *M. pulegium* is a source of diverse classes of natural compounds such as flavonoids, alkaloids, essential oils [32–34]. In a previous study [33], *M. pulegium* extract (MPE) was investigated as corrosion inhibitor for steel in hydrochloric acid solution under the same test conditions as used in the present work. The results obtained were encouraging (more than 82% of inhibition efficiency with 33% MPE) and that necessitated this renewed interest to further assess the possible corrosion inhibition potential of the oil of this plant.

In the present study, the effect of *M. pulegium* oil on both the corrosion and kinetics of corrosion process of carbon steel in 1 M hydrochloric acid solution has been investigated using weight loss, potentiodynamic polarization and EIS techniques. Characterization of the steel surface before and after film formation was also carried out. A probable inhibition mechanism is proposed for this work.

## 2. Experimental

### 2.1. Materials

The steel used in this study is a carbon steel with a chemical composition 0.13% C, 0.32% Si, 0.64% Mn, 0.15% Cu, 0.012% S, 0.01% P, 0.038% Al, 0.081% Cr, 0.011% Mo, 0.083% Ni, 0.01% V, and the remainder iron (Fe).

### 2.2. Preparation of specimens

The carbon steel samples were pre-treated prior to the experiments by grinding with emery paper sic until to 1500 grade; rinsed with distilled water, degreased in ethanol, washed again with bidistilled water and then dried at room temperature before use.

### 2.3. Solution preparation

The aggressive solutions of 1 M HCl were prepared by dilution of analytical grade 37% HCl with double distilled water. Inhibitor were dissolved in acid solution at the required concentrations (in mL/L), and the solution in the absence of inhibitor was taken as blank for comparison purposes. The test solutions were freshly prepared before each experiment by adding *M. pulegium* oil directly to the corrosive solution. Concentrations of *M. pulegium* oil 1, 2 and 3 mL/L. Experiments were carried out in triplicate to ensure reproducibility.

### 2.4. Plant collection and essential oil extraction

*M. pulegium* was collected in June 2009 in Ain-Defla, Northern Algeria at 280 m altitude. The aerial parts of the plant were air-dried in the laboratory at room temperature. The essential oil used was obtained by steam distillation of the aerial part (stem and leaves). The oil obtained was a colorless liquid. Essential oil was stored in sealed vials protected from the light at 4 °C before analyses. The density of the oil obtained was 0.93 g·cm<sup>-3</sup>. The oil sample was subsequently analyzed by GC and GC/MS.

#### 2.4.1. Gas chromatographic analysis

The analysis of the oil was carried out using HP5890 with FID, and a capillary column coated with 5% phenyl dimethylpolysiloxane (25 m × 0.25 mm × 0.25 μm thickness). Column temperature ranged from 40 to 240 °C at 5 °C/min. Injector temperature was 220 °C; detector temperature was 250 °C; volume injected was 0.1 μL of the oil diluted in 10% of hexane. Carrier gas was N<sub>2</sub> with 1 mL/min.

#### 2.4.2. Gas chromatography/mass spectroscopy

The oil was analyzed by GC/MS using a Perkin Elmer Clarus 500 equipped with a cross-linked 5% phenyl dimethylpolysiloxane PE Elite capillary column (30 m × 0.25 mm × 0.25 μm thickness). Operating conditions: The carrier gas flow was 1 ml He/min. The injector and detector temperatures were 250 °C and 280 °C respectively. The column temperature was held at 60 °C for 1 min, then raised from 60 °C to 150 °C at 15 °C/min and from 150 °C to 275 °C at 5 °C/min. The program was run in the split less mode with a mass range of 80–600 g, and the scan interval was 0.5 s. Detector voltage was set at 1.5 kV. The identification of the components was performed on the basis of chromatographic retention indices and by comparison of the recorded spectra with computed data libraries [35].

### 2.5. Electrochemical tests

The electrochemical study was carried out using an EG&G potentiostat/galvanostat PAR 273A piloted by CorrIII software. This potentiostat was connected to a thermostated double-wall cell with three electrodes. A saturated calomel electrode (SCE) and platinum electrode were used as reference and auxiliary electrodes, respectively. The material used for constructing the working electrode was the same used for gravimetric measurements. The surface area exposed to the electrolyte was 0.64 cm<sup>2</sup>. Potentiodynamic polarization curves were plotted at a polarization scan rate of 1 mV/s. Before all experiments, the potential was stabilized at free potential during 30 min. The polarization curves are obtained from –800 to –200 mV at 298 K. The test solution was thereafter de-aerated by bubbling with nitrogen. Gas bubbling was maintained prior and throughout each experiment. The inhibition efficiency ( $E_{pol}\%$ ) was calculated using the following equation:

$$E_{pol}\% = \frac{i_{corr} - i_{corr}^{inh}}{i_{corr}} \times 100 \quad (1)$$

where  $i_{corr}$  and  $i_{corr}^{inh}$  represent corrosion current density values without and with inhibitor, respectively.

Electrochemical impedance spectroscopy (EIS) measurements were carried out with a model 5210 analyzer controlled by Power suite software. After the determination of the steady-state current at a corrosion potential, a sine wave potential, 10 mV peak to peak at frequencies between 100 kHz and 10 MHz, was superimposed on the rest potential. Computer software automatically controlled the measurements performed at rest potentials after 30 min of exposure at 298 K. The impedance diagrams are given in the Nyquist representation. All experiments were repeated three times to ensure reproducibility.

Inhibition efficiency ( $E_{EIS}\%$ ) was obtained according to the following equation:

$$E_{EIS}\% = \frac{R_t^{inh} - R_t}{R_t^{inh}} \times 100 \quad (2)$$

where  $R_t^{inh}$  and  $R_t$  are charge transfer resistances values in the presence and absence of *M. pulegium* oil, respectively.

### 2.6. Weight loss measurements

Gravimetric experiments were performed according to the standard methods, the carbon steel specimens of dimension 2 × 1 × 0.02 cm<sup>3</sup> for weight loss measurements. Prior to all measurements, they were mechanically abraded with #180, #320, #800, #1200 and up to #1500 grades of emery papers. The specimens are washed thoroughly with bidistilled water, degreased with ethanol and dried. Gravimetric measurements were carried out in a double walled glass cell equipped with a thermostated cooling condenser. The solution volume was 60 cm<sup>3</sup>. The immersion time for the weight loss was 6 h at 298 K. The

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