

Ethanol extract of Camphor tree bark as green corrosion inhibitor of LCS in 0.5 M H₂SO₄ with and without synergistic effect

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Abstract

This work tested the ethanol extract of Camphor tree *Cinnamomum camphora* bark (CTBEE) as a green corrosion inhibitor of low carbon steel (LCS) in 0.5 M H₂SO₄ with and without the synergistic effect of 0.1 M NaCl or 800 ppm of KI. The corrosion protection of LCS was significantly increased in the presence of CTBEE-KI and CBWE-NaCl blends compared to CTBEE alone, according to data performed by electrochemical impedance spectroscopy (EIS) and electrochemical polarization (EP) analyses. The formation of a film by the blends blocks the LCS surface area preventing corrosive gradients to reach its surface. Inhibition efficiency (IE%) of 81 and 89% were obtained in the presence of 800 ppm CTBEE-0.1 M NaCl and CTBEE-800 ppm KI, respectively, compared to IE of 65% in the presence of 800 ppm CTBEE only. A maximum IE% of 89 and 97% were reached after 72 hours of immersion in the presence of CTBEE-NaCl and CTBEE-KI, respectively. Potentiodynamic experiments indicate that the blends act as mixed-type inhibitors. Inhibition efficiency of CTBEE, CTBEE-KI and CTBEE-NaCl remained effectively high with testing period of immersion time which proved the good stability of CTBEE in the corrosive acidic media.

1. Introduction

Ocean-related companies and activities contribute significantly to global economic growth and jobs. As a vital provider of a number of societal requirements such as transportation, recreation, food, electricity, and minerals, they are vital for humanity's future welfare. Global megatrends such as population growth, rising urbanisation rates, and climate change adaptation are driving growth in ocean economic activity through expansion of existing (transportation, fishing, natural oil and gas, coastal tourism) and creation of new (offshore renewable energy production, deep-sea oil and natural gas, marine fish farming, seabed mining) sectors. Water pollution due to oil production activities is a significant problem worldwide [1–5]. One of these activities is the use of different types of corrosion inhibitors to control the degradation of metals used in production equipment and many of these inhibitors are harmful to the aquatic environment [6–10]. Down-hole tubules, flow lines, and transmission pipelines are all made of carbon steel (CS) [11–15]. In corrosive conditions such as those found in the oil industry, however, CS has poor corrosion resistance [16–20]. Some of the most frequent techniques of regulating corrosion rate in acid solutions in oil operations include pickling, industrial acid cleaning, oil and gas well acidizing, and the removal of rust, scale, and corrosion elements [21–24], is to use corrosion inhibitors. The majority of inhibitor defense is based on the adsorption of inhibitor molecules on metal surfaces, followed by the creation of a protective layer [25, 26]. Quantitative structure activity relationship have been studied and the relation between chemistry structure of compounds and their inhibition efficiencies toward corrosion of different metal surfaces were reported [27–33]. The large number of the corrosion inhibitors used in operation were natural products derived from plant parts such as roots, bark, seeds, stems, leaves, flowers, and fruits [34–37]. Literature has shown that different plant extracts such as *Azadirachta indica* leaves [38], *Cassia fistula* L. pods [39], *Murraya koenigii* leaves [40], *Ervatamia coronaria* leaves [41], *Hyalomma tick* extract [42], *Cynara scolymus* L extract [43], *Gloriosa superba* seeds extract [44], *Hymenaea stigonocarpa* fruit shell extract [45], *Sargassum muticum* extract [46], *Ficus racemosa* leaf

extract [47], *Ficus tikoua* leaves extract [48], *Calendula officinalis* flower heads extract [49], ginger extract [50], bee pollen extract [51], *Idesia polycarpa* Maxim fruits extract [52], and *Bbauhinia purpurea* leaves extract were pronounced as green corrosion inhibitors of different metals in various corrosive media [53].

The camphor tree is a Laurel family evergreen tree native to Asia, where it is commonly produced commercially for the extraction of camphor oil. They have lustrous, elliptical leaves that start off light-bright green and turn a deep green as they mature [54]. Tree of camphor *Cinnamomum camphora* is a huge, attractive tree that is commonly used as a decorative street or shade tree in warm climates around the world. The twigs, leaves, and wood are used to make camphor and camphor oil, and the twigs, leaves, and wood are used to make furniture. *Cinnamomum camphora* is a camphor-scented plant native to China and Japan. The shiny green leaves give off a heavy camphor scent when crushed. Camphor trees have a high rate of growth and the potential to produce large quantities of shiny black berries, which are easily eaten by birds, allowing the seed to spread. [55, 56]. It has little greenish white to pale yellow blooms that bloom on three-inch-long panicles. Birds love the small, dark-blue to black berries that bloom in the spring. Although most camphor trees grow to be between 25 and 50 feet tall, a few exceptional examples have grown to be over 100 feet tall [57, 58].

The increasing concerns about the health risks and environmental effects of various types of inhibitors has led researchers to develop eco friendly materials can be used as corrosion inhibitor and are inexpensive, safe, and have little or no negative impact on the environment [34–37]. In this regard, the aim of this study is to see if camphor tree bark ethanol extract can be used as a green corrosion inhibitor for low carbon steel in a corrosive environment of 0.5 M H₂SO₄, and to test the performance and mechanism of corrosion inhibition using various electrochemical methods.

2. Experimental

2.1. Extract, solutions, and chemicals

Camphor tree bark ethanol extract (CTBEE) was used as eco-friendly corrosion inhibitor. The inhibitor was extracted from Camphor tree *Cinnamomum camphora* bark by boiling of 100 g of dry fine powder of Camphor tree bark (CTB) in ethanol (500 mL) for two hours and then cooled to room temperature and filtered. The filtrate was evaporated at low pressure to give a dry red powder followed by complete drying in an oven at 50°C for 24 hours to give 3.1 g of the dried red powder which was stored at room temperature until it was used. The inhibitor extract was prepared by dissolving the required weight of the dried red powder of CTB in 5 mL of ethanol and then added to the corrosion solution to form the desired final concentration. The corrosive media (electrolyte) 0.5 M H₂SO₄ is made by diluting concentrated H₂SO₄ (98% from Sigma-Aldrich, USA) with deionized water to the appropriate concentration. Weight of 200, 400, 600, 800 and 1000 mg of solid inhibitor extract and dissolved in 5 mL of ethanol then added to 0.5 M H₂SO₄ to prepare 1 L of the testing solutions. In addition, NaCl and KI salts were added in separate to the electrolyte for synergistic study.

2.2. CS Alloy, electrochemical, corrosion cell, and equipment

The working electrode was made of a rod of low carbon steel (LCS) alloy with the following composition (wt %): C (0.068), Si (0.249), Mn (0.662), P (0.015), S (0.022), Ni (0.027), Cu (0.031), N (0.122), Fe (98.62), and traces of other elements. Teflon coat and epoxy resin were used to seal the LCS rod, leaving a circle exposed surface area of 0.64 cm^2 . The counter and reference electrodes were a graphite tubular rod (1 cm diameter $\times$ 15 cm length) and a saturated calomel electrode (SCE), respectively. A standard three-electrode Gamry Potentiostat/Galvanostat (model Series-G 300) with Gamry Fram work version 6.12 software was used to perform the electrochemical measurements. AC signals were used to test impedance in the frequency range of 100 kHz–0.1 Hz with a peak-to-peak amplitude of 10 mV under open circuit potential (OCP) conditions on the LCS rod. The equivalent circuit model and Echem Analyst software were used to interpret the obtained impedance spectra. The sample surface corrosion and composition is evaluated using scanning electron microscopy and EDX (FEI-SEM QUANTA 250). FTIR of CTBE was performed using Bruker VERTEX 70 FT-IR spectrometer in a spectral range of 4000–400 cm^{-1} coupled with attenuated total reflection (ATR) Platinum 100 for direct sample analysis. Analysis of the CTBEE was performed using coupled Agilent gas chromatography–mass spectrometry (GC–MS) in full scan mode.

Helium was used as the makeup gas and carrier gas at a rate of 1.22 mL min^{-1} on a gas chromatograph (7890A GC system) fitted with an HP-5MS 5% Phenyl Methyl Silox capillary column (30 m $\times$ 250 μm $\times$ 0.25 μm) in a splitless injection mode heated at 300°C, pressure of 22.231 psi. The column temperature was increased from 90°C (held for 1 minute) to 205°C at an 8°C min^{-1} rate, then from 205°C (held for 1 minute) to 240°C at a 5°C min^{-1} rate, then from 240 (held for 1 minute) to 300°C at an 8°C rate, and finally held isothermally at 300°C for 30 min, for a total run time of 61.875 min. The mass spectrometry (5975C inert MSD with triple-Axis Detector) was set to a complete scan mode between 10 and 550 Da per second, with an electron energy of 70 eV and an electron impact ionisation source of 70 eV (EI). The data was collected in full scan mode with a mass range of 40–600 m/z and a scan time of 500 ms. The MS fragmentation of CTBEE GC-MS peaks was obtained using ChemStation software to monitor the GC-MS instrument and mass spectra from the NIST library mounted in the GC-MS computer.

2.3. Methodology of electrochemical experiments

The working electrode is polished before each test on wet silicon carbide sheets with successively fine grades, then washed with distilled water, degreased with ethanol using an ultrasonic bath, dried, and immersed in an acidic corrosion solution. The corrosive solution consisted of 0.5 M H_2SO_4 with different concentrations of CTBEE in the absence and presence of 0.1 M NaCl and 800 ppm KI. Distilled water and analytical chemicals were used to make the electrolyte. All of the data in this analysis (current, impedance, and resistance) is expressed in terms of the working electrode's geometric surface area. The electrochemical tests were carried out at room temperature (25°C). After reaching a constant value of free corrosion potential, this is done by immersing the working electrode in the test medium for approximately

30 minutes before starting the electrode measurement process, then EIS and polarization measurements are performed. The polarization curves are dynamically determined effectively at a scan rate of 5 mV/s, starting with OCP and moving towards the cathodic direction (-250 mV versus OCP), and then inverted to a final voltage of ca. $+250$ mV for OCP.

3. Results And Discussion

3.1. FTIR and GC-MS analyses of CTBEE

The FTIR of CTBEE is shown in Fig. 1. The O-H bond stretch vibration is assigned to an explicit broad peak at 3272.4 cm^{-1} . The vibration of C-H (CH_2) bond stretch is described by the peaks at 2921.45 and 2851.77 cm^{-1} . The COOH or COOR bond is ascribed to the peak at 1698.76 cm^{-1} , demonstrating the existence of carboxylic groups in the CTBEE. The C = C bond vibration is assigned to different compounds in the CBEE by the peaks at 1611.70 and 1509.73 cm^{-1} . The rocking bending vibration of C-H is responsible for the peaks at 1447.15 and 1335.51 cm^{-1} . The vibration of C-C bond stretch showed the peaks at 1219.58 and 1196.99 cm^{-1} . The vibration of aromatic C-H on the C-H aliphatic group is responsible for the peaks at 1108.44 and 1047.16 cm^{-1} . The CTBEE had a lot of O atom functional groups and bonds, according to the FT-IR review. As a result, CTBEE's ability as a highly efficient corrosion inhibitor has been demonstrated.

Figure 2 shows the profile of semi-volatile, volatile, and other important chemical compounds extracted from camphor tree bark using ethanol. By using GC-MS to profile semi-volatile compounds extracted from camphor tree bark by ethanol (Table 1), 65 major compounds were reported, with many more minor peaks remaining unidentified [59–61]. The compound groups explored included alkenes, fatty alcohols, fatty acids, glycerolipids, phenols and derivatives, steroids, and triterpenes. In the literature, the majority of these chemicals have been recognized as key components of camphor tree extract [59–61]. Several aldehydes, esters, ethers, ketones, carboxylic acids, and azo compounds were among the compounds studied, all of which are promising corrosion inhibitors.

3.2. EIS measurements

The corrosion inhibition efficiency of Camphor tree bark ethanol extract and its improvement by the synergistic effect of 0.1 M NaCl and 800 ppm of KI for the LCS surface in $0.5\text{ M H}_2\text{SO}_4$ solution were evaluated using Electrochemical Impedance Spectroscopy (EIS). At OCP, EIS measurements were taken over a broad frequency range, from 100 kHz to 0.10 Hz , with an AC voltage amplitude of 10 mV . Before taking measurements at room temperature, the electrode was stabilized at OCP for 30 min . Figure 3 shows EIS data obtained on an LCS electrode in corrosive media in the absence and presence of 200 , 400 , 600 , 800 , and 1000 ppm Camphor bark ethanol extract, while Figs. 4 and 5 show the results in the same conditions with the addition of 0.1 M NaCl and 800 ppm KI , respectively. The figures show that as the inhibitor concentration in the electrolyte increases, the diameter of the semicircle grows, suggesting an improvement in LCS corrosion resistance.

EIS data were simulated and equivalent resistance measured by using equivalent electrical circuit (EEC) as shown in Fig. 6, and the relevant efficiencies were calculated and presented in Table 2 using Eq. (1) [62, 63]. R_{el} is the ohmic resistance between the reference and working electrodes; R_1 is the polarization resistance; CPE_1 is the capacitance of the electrode double layer at the electrode/electrolyte interface (constant phase element).

$$IE\% = (1 - \frac{R_1^{(b)}}{R_1^{(in)}}) \times 100 \quad (1)$$

Where $R_1^{(b)}$ is the polarization resistances of the blank and $R_1^{(in)}$ is the polarization resistances in the presence of the inhibitor.

Table 2
The equivalent circuit and calculated IE% data of LCS in 0.5 M H₂SO₄ having different concentrations of CTBEE, CTBEE-0.1 M NaCl, and CTBEE-800 ppm KI.

Electrolyte composition	$R_1 (\Omega \text{ cm}^2)$	$\text{CPE} \times 10^6 (\Omega^{-1} \text{ cm}^{-2} \text{ S}^\alpha)$	α	IE %
Blank (0.5 M H ₂ SO ₄) (A)	15.25	372	0.863	-
A + 200 ppm CTBEE	28.27	345	0.804	46.06
A + 400 ppm CTBEE	31.73	229	0.831	51.94
A + 600 ppm CTBEE	39.57	355	0.810	61.46
A + 800 ppm CTBEE	43.83	241	0.842	65.21
A + 1000 ppm CTBEE	38.68	311	0.801	60.57
Blank + 0.1 M NaCl (B)	35.56	374	0.814	57.11
B + 200 ppm CTBEE	44.39	424	0.767	65.65
B + 400 ppm CTBEE	48.63	375	0.792	68.64
B + 600 ppm CTBEE	59.5	335	0.797	74.37
B + 800 ppm CTBEE	78.44	299	0.797	80.56
B + 1000 ppm CTBEE	51.28	393	0.775	70.26
Blank + 800 ppm KI (C)	52.44	402	0.763	70.92
C + 200 ppm CTBEE	60.62	219	0.816	74.84
C + 400 ppm CTBEE	109.9	461	0.674	86.12
C + 600 ppm CTBEE	83.24	308	0.759	81.68
C + 800 ppm CTBEE	136.6	297	0.742	88.84
C + 1000 ppm CTBEE	115.1	300	0.742	86.75

3.2. Potentiodynamic measurements

To validate the data calculated from EIS measurements, potentiodynamic experiments were carried out. Figures 7–9 show potentiodynamic curves obtained on a low carbon steel electrode in 0.5 M H₂SO₄ solution in the absence and presence of 0.1 M NaCl and 800 ppm of KI, respectively, at different concentrations of Camphor tree bark ethanol extract in the electrolyte. The anodic and cathodic currents also decrease as the inhibitor concentration in the corrosive solution increases, as shown by the curves in Figs. 8 and 9. This revealed that the CTBEE-NaCl and CTBEE-KI blends are mixed-type corrosion

inhibitors, while the curves in Fig. 7 showed that CTBEE alone is a cathodic inhibitor. Induction of the anodic and cathodic curves to the related OCP yielded the corresponding corrosion current densities i which were presented in Table 3 along with the calculated Inhibition Performance. In the absence of a synergistic effect between NaCl and KI, the findings are presented in Table 3. Eq. (2) was used to measure the IE % [50, 63].

$$IE\% = \left[1 - \frac{i_{in}}{i_b}\right] \times 100 \quad (2)$$

Where i_{in} (Acm^{-2}) is the corrosion current at a specific inhibitor extract concentration, while i_b is the corrosion current in the absence of inhibitor in the blank solution.

Table 3
Corrosion current density (j) and inhibition efficiency values for LCS in a 0.5 M H_2SO_4 media containing different concentrations of CTBEE, CTBEE-0.1 M NaCl, and CTBEE-800 ppm KI.

Electrolyte composition	Corrosion current density (j) ($mAcm^{-2}$)	Inhibition Efficiency (%)
Blank (0.5M H_2SO_4) (A)	0.91	-
A + 200 ppm CTBEE	0.505	44.51
A + 400 ppm CTBEE	0.421	53.74
A + 600 ppm CTBEE	0.383	57.91
A + 800 ppm CTBEE	0.278	69.45
A + 1000 ppm CTBEE	0.375	58.79
A + 0.1 M NaCl + 200 ppm CTBEE	0.359	60.55
A + 0.1 M NaCl + 400 ppm CTBEE	0.281	69.12
A + 0.1 M NaCl + 600 ppm CTBEE	0.226	75.16
A + 0.1 M NaCl + 800 ppm CTBEE	0.158	82.64
A + 0.1 M NaCl + 1000 ppm CTBEE	0.308	66.15
A + 800 ppm KI + 200 ppm CTBEE	0.249	72.64
A + 800 ppm KI + 400 ppm CTBEE	0.145	84.07
A + 800 ppm KI + 600 ppm CTBEE	0.189	79.23
A + 800 ppm KI + 800 ppm CTBEE	0.12	86.81
A + 800 ppm KI + 1000 ppm CTBEE	0.168	81.54

3.3 Adsorption isotherm

Temkin, Freundlich, and Langmuir adsorption isotherms models were used to characterize the inhibitor adsorption on the LCS surface. The data better suit the Langmuir adsorption isotherm. The mean corrosion inhibition efficiency values obtained using the two experimental techniques (Fig. 9) are provided in Fig. 10 in the form of the linearized Langmuir isotherm by the Eq. (3) assuming that the inhibition efficiency (IE%) is proportional to the inhibitor surface coverage (θ) [64–66].

$$\frac{C}{\theta} = C + \frac{1}{K_{ads}}$$

3

Where C represents the inhibitor extract concentration, S represents the surface coverage, and K_{ads} represents the adsorption equilibrium constant. If the plot of C/θ vs C is linear and the slope of the line is similar to unity, an inhibitor is said to obey Langmuir. Similarly, the plot of θ vs $\log C$ for Temkin and the plot of $\log \theta$ vs $\log C$ for Freundlich should be linear. According to the correlation coefficient (R^2) obtained from plots in Table 4, the Langmuir isotherm suits the experiment data better, with R^2 close to unity, suggesting monolayer adsorption of the inhibitor on the surface of LCS.

Table 4
Data obtained from isotherm models at 298K.

inhibitor	Langmuir		Temkin		Freundlich	
	R^2	Slope	R^2	Slope	R^2	Slope
CTBEE	0.978	1.44	0.822	0.274	0.849	0.220
CTBEE + 0.1 M NaCl	0.964	1.35	0.835	0.164	0.406	0.101
CTBEE + 800 ppm KI	0.994	1.140	0.58	0.149	0.59	0.081

The adsorption equilibrium constants $K_{ads} = 0.007, 0.055$, and $0.029 \text{ dm}^3 \text{ mg}^{-1} (\text{L mg}^{-1})$ were determined from the intercept of the line in Fig. 10, and the corresponding normal Gibbs free energy of adsorption (kJ mol^{-1}) was calculated from Eq. (4) [67, 68].

$$K_{ads} = \frac{1}{C_{solvent}} \exp\left(\frac{-\Delta G_{ads}^0}{RT}\right) \quad (4)$$

T is the temperature (K), $C_{solvent}$ is the concentration of the solvent, which in this case is water ($C_{H_2O} = 10^6 \text{ mg dm}^{-3}$), and R is the gas constant ($\text{J mol}^{-1} \text{K}^{-1}$). At 298 K, the typical Gibbs free energies of adsorption of CTBEE alone, extract blend with 0.1 M NaCl, and 800 ppm KI on the LCS surface were -22 , -27 and -25 kJ mol^{-1} . Normal Gibbs free energy of adsorption in aqueous solution magnitudes around

or higher (more negative) than -40 kJ mol^{-1} indicate charge sharing between the molecules and the metal (chemisorption), whereas around -20 kJ mol^{-1} or lower indicate adsorption with electrostatic interaction between the adsorbent and the adsorbate (physisorption), according to the literature [63]. Thus, the measured negative values of standard Gibbs energy of adsorption around -20 kJ mol^{-1} show that CTBEE physisorption occurs, and the negative values of $-\Delta G_{ads}^0$ proved the spontaneity of the CTBEE adsorption process on the metal surface and ensure the stability of the CTBEE layer adsorbed on the surface of LCS.

3.4. The influence of time on the effectiveness of inhibition

EIS experiments were carried out in the absence and presence of 800 ppm CTBEE with 0.1 M NaCl and 800 ppm KI separately in 0.5 M H_2SO_4 solution at different time intervals to measure the time necessary for the CTBEE to achieve optimum IE% and its stability under the conditions of testing. Tables 5–6 and Fig. 11 display the associated inhibition efficiency and corrosion resistance values. The data show that the CTBEE inhibition efficiency was as high as 81.67 percent 30 minutes after immersion of the working electrode in the electrolyte, and reached a limit of 90.26 percent after 5 hours with the synergistic effect of 0.1 M NaCl. On the other hand, with the synergistic effect of 800 ppm of KI, the CTBEE shows corrosion inhibition efficiency of 90% after 30 min and IE% remained as high as the maximum efficiency (96%) after 6 h of testing time.

The weight loss experiments and the scanning electron microscopy (SEM) analysis results of the LCS samples also demonstrate CTBEE's long-term corrosion inhibition efficiency (Fig. 12). Weight loss data reported in Table 7 showed that during 24 h of immersion time of the samples in 0.5 M H_2SO_4 in the absence and present of 800 ppm of CTBEE and with additive of 0.1 M NaCl and 800 ppm of KI, the IE% reached 70, 85 and 90%, respectively.

The sample surface scan results in the absence and presence of CTBEE with the additives of NaCl and KI are shown in Fig. 12 after 24 hours of immersion in the corrosive environment. The SEM result shows that in the absence of the inhibitor in the testing media, the LCS surface corrosion is relatively higher, and the LCS surface is clearly damaged as compared to the sample surfaces in Fig. 12c, which show the free corrosion sample surface and the slightly corroded surface of the samples immersed in the corrosive solution in the presence of CTBEE-NaCl.

Table 5
IE% and R_p obtained on time from EIS measurements recorded on LCS
in the presence of CTBEE-0.1 M NaCl in a 0.5 M H₂SO₄ solution.

Time	0 ppm CTBEE	800 ppm CTBEE + 0.1M NaCl	IE%
	^(b) R_p (ohmcm ⁻²)	⁽ⁱⁿ⁾ R_p (ohmcm ⁻²)	
0.5 h	15.25	83.19	81.67
1.0 h	13.6	88.01	84.55
2.0 h	11.05	78.23	85.87
4.0 h	7.31	61.51	88.12
5.0 h	5.25	53.91	90.26
6.0 h	5.25	48.89	89.26

Table 6
IE% and R_p obtained on time from EIS measurements recorded on LCS
in the presence of 800 ppm CTBEE/800 ppm KI in a 0.5 M H₂SO₄
solution.

Time	0 ppm CTBEE	800 ppm CTBEE + 800 ppm KI	IE%
	^(b) R_p (ohmcm ⁻²)	⁽ⁱⁿ⁾ R_p (ohmcm ⁻²)	
0.5 h	15.25	161.20	90.54
1.0 h	13.6	177.6	92.34
2.0 h	11.05	182.1	92.53
4.0 h	7.31	185.8	94.05
5.0 h	5.25	190.6	96.16
6.0 h	5.25	160.3	96.72

Table 7

Weight loss, corrosion rate and IE% obtained from weight-loss measurements for LCS immersed 24 h in : (A) 0.5 M H₂SO₄, (B) 0.5 M H₂SO₄ + 800 ppm CTBEE, (C) 0.5 M H₂SO₄ + 800 ppm CTBEE + 0.1 M NaCl, (D) 0.5 M H₂SO₄ + 800 ppm CTBEE + 800 ppm KI.

Media	Inhibitor concentration	Weight lost W_i (gcm ⁻²)	CR (mm/y)	IE%
A	Blank (0.5 M H ₂ SO ₄)	0.0650	34.65	-
B	Blank + 800 ppm CTBEE	0.0190	11.37	70
C	Blank + 800 ppm CTBEE + 0.1M NaCl	0.0096	5.622	85
D	Blank + 800 ppm CTBEE + 800 ppm KI	0.0064	3.378	90

3.6 Mechanism of corrosion inhibition and EDX analysis

CTBEE is an important inhibitor of LCS corrosion in a 0.5 M H₂SO₄ solution, according to the results of all experimental techniques. The inhibitor blends behave as mixed-type inhibitors, eliminating all partial corrosion reactions, as shown by the potentiodynamic results plots in Figs. 8 and 9. The inhibitor blend's inhibition function is mediated by the formation of a surface adsorbed monolayer, which creates a strong hydrophobic barrier on the surface of low carbon steel due to the presence of many types of long chain hydrocarbon (–CH₂). The interaction between the Fe²⁺ ions on the surface of the LCS and the donor–acceptor lone pairs of the heteroatoms in the inhibitor molecule that causes adsorption on the anodic sites. In an acidic medium, the amine group occurs in protonated form and is adsorbed on cathodic sites, slowing hydrogen evolution. Because of the presence of lone pairs of electrons on nitrogen and oxygen atoms, the CTBEE effectively prevents LCS corrosion. The surface coverage, and thus the adsorbed inhibitor molecules concentration on the LCS surface, are proportional to the corrosion inhibition effectiveness. EDX measurements were taken in order to predict the tightness of the inhibitor layer on the LCS surface. The weight percentage of carbon, oxygen, and nitrogen elements (which are the most content of the CTBE molecule) on the surface of LCS sample immersed for 24 h in 0.5 M H₂SO₄ solution in presence of 800 ppm CTBEE with 0.1 M NaCl and 800 ppm KI separately shows a substantial increase in the weight percentage of carbon, oxygen, and nitrogen elements (which are the most content of the organic inhibitor molecule) (Table 8). These increasing of element percentages on the sample of LCS are proportional with amount of inhibitor adsorbed on the surface of LCS and then with the LCS surface coverage.

Table 8
EDX analysis results of the LCS surface sample obtained and after immersion in 0.5 M H₂SO₄ containing 800 ppm CTBEE and 0.1 M NaCl and 800 ppm KI for 24 h.

Element	wt.%		
	LCS	LCS + CTBEE-KI	LCS + CTBEE-NaCl
Carbon	1.15	2.39	2.81
Oxygen	1.66	2.03	3.33
Nitrogen	0.122	0.79	0.66

3.7 The compounds responsible for corrosion inhibition

According to the literature review, we can expect the natural organic compounds mentioned in Table 9 to have the greatest impact as corrosion inhibitors in the camphor tree bark ethanol extract.

Cinnamaldehyde and Vanillin were investigated as corrosion inhibitors in acid solution to protect the surface of copper and mild steel. The inhibitors are environmentally friendly and can cover up to 98.36% of the metal's surface, greatly reducing corrosion [69–71]. The inhibiting activity of guanidine derivatives on copper and iron corrosion inhibition in acid and 3 percent NaCl solutions was investigated [72–74]. In a 0.05 wt percent NaCl solution, the inhibition activity of acetophenone derivatives as an environmentally friendly corrosion inhibitor for AZ91D magnesium alloy was investigated [75, 76]. The corrosion inhibition of steel in 1 M HCl in the presence of benzoic acid derivatives was investigated using various electrochemical techniques, with inhibition efficiency reaching 97.0 percent at 1.0 mM inhibitor concentration [77]. Ethanolic solutions of long-chain carboxylic acids with the general formula CH₃(CH₂)_n-2COOH (12 ≤ n ≤ 16; HC₁₂, HC₁₄, and HC₁₆) are used to coat iron heritage objects [78, 79]. Weight loss measurements and different electrochemical techniques were used to investigate the inhibiting effect and adsorption actions of acetamide derivatives on mild steel in hydrochloric acid solution, and showed greater than 90% inhibition efficiency at 0.01 M [80, 81]. In a sulfuric acid medium, the inhibitory effect of benzoic acid and salicylic acid derivatives on steel with known composition was investigated [82, 83]. For the inhibitive performance of methanolic extract of microalgae *Chlorococcum* sp., effectively contributed by levoglucosenone and hexadecanoic acid for corrosion resistance on mild steel in acidic atmosphere, a green and holistic approach is proposed [84–87]. The effect of combining CO with a common acid corrosion inhibitor, *trans*-cinnamaldehyde (TCA), at high pressure and temperature was studied on a high-Ni ferrous alloy (Incoloy 825) and two high-Fe alloys (13Cr-L80 (Uniloy-420) and N80 steels) in very aggressive conditions (15 percent, w/w) HCl solution [88–92]. Corrosion inhibition of mild steel in acidic medium with acetohydrazide derivatives [93]. The chelating agent citric acid, which is a sustainable raw material and completely non-toxic, can prevent corrosion of aluminum, mild steel, and carbon steel [94–96]. Metal corrosion inhibitors made up of organic compounds with –CN and –NO₂ substituents are widely used. –CN and –NO₂ substituents, on the other

hand, improve the inhibition potency of organic inhibitors, whether they are too large or too small. These substituents frequently reduce the solubility of their base compounds in polar fluids. The inhibitory efficacy of macromolecules, oligomers, and polymers is expected to increase in the presence of $-\text{CN}$ and $-\text{NO}_2$ substituents [97].

4. Conclusions

Corrosion inhibitor (CTBEE) extracted from camphor tree as a natural plants found in the fields and gardens acts as an inhibitor of LCS corrosion of the mixed-type inhibitor with NaCl and KI, according to potential dynamic measurements. The damping process depends on the formation of a surface layer on the surface of the metal by absorbing the retarder particles and their physical attachment to the metal surface, which represents a hydrophobic barrier and prevents corrosive media to attack the surface of LCS due to the presence of a long hydrocarbon chain which is available in the plant extract as explained in the chromatographic analysis of the extract. It also showed the presence of many compounds that have properties that make them can be used as inhibitors to metal corrosion. The high inhibition efficiency (IE%) of the CTBEE-NaCl and CTBEE-KI inhibitor against the corrosion of low carbon steels in sulfuric acid solution was stably detected by EIS and the potential dynamic results. During prolonged 24 hours continuous immersion periods of LCS in 0.5 M H_2SO_4 solution, at low concentrations of CTBEE, EIS and weight loss measurements, revealed that the inhibitor blends gave good long-term (800 ppm) effective anti-corrosion performance. The Langmuir isothermal, which indicates self-assembling monolayer adsorption, and the free energy values associated with Gibbs free energy indicating spontaneous adsorption and desorption, which were found to be in very good agreement with the CTBEE adsorption processes.

Table 9

Some compounds found in the camphor ethanol extract which expected to be responsible for the above observed corrosion inhibition as found in literature.

No.	Scan (min)	Compound found in Camphor ethanol extract	Similar or related compound found in literature and investigated for metal corrosion inhibition	Metal tested	Ref.
1	12.195	Vanillin	Cinnamaldehyde, vanillin	Copper, mild steel	[69–71]
2	14.383	1-(3-(Cyclohexylamino)propyl) guanidine	Guanidine derivative	Copper, Armco iron	[72–74]
3	14.845	3',5'-Dimethoxyacetophenone	2-Hydroxy-4-methoxy-acetophenone	AZ91D magnesium alloy	[75, 76]
4	15.294	4-(methylthio)-benzoic acid	Amino benzoic acid, 4-aminoantipyrine derived	Mild steel	[77]
5	17.962	Tetradecanoic acid	Long-chain monocarboxylic acids, <i>n</i> -tetradecanoic acid	Iron, copper	[78, 79]
6	18.933	N-methyl-N-[4-(3-hydroxypyrrolidinyl)-2-butynyl]-Acetamide	Acetamide derivative	Mild steel	[80, 81]
7	19.555	4-hydroxy-3,5-dimethoxy benzoic acid	Benzoic acid, salicylic acid, benzoic acid derivative	Steel, mild steel	[82, 83]
8	20.815	<i>n</i> -Hexadecanoic acid, methyl ester	<i>n</i> -Hexadecanoic acid (plant extract)	Copper	[84]
9	21.602	<i>n</i> -Hexadecanoic acid	<i>n</i> -Hexadecanoic acid (plant extract)	Mild steel	[85, 86]
10	22.039	<i>n</i> -Hexadecanoic acid, ethyl ester	Levoglucosenone and hexadecanoic acid (microalgae extract)	Mild steel	[87]
11	22.365	3,5-Dimethoxy-4-hydroxycinnamaldehyde	<i>trans</i> -Cinnamaldehyde,	I825, L80-13Cr and N80 alloys, steel, Carbon Steel	[88–92]
12	22.561	2-(2-benzothiazolylthio)-N2-(3-fluorobenzylideno)-Acethydrazide	Acetohydrazide derivatives	Mild steel	[93]

No.	Scan (min)	Compound found in Camphor ethanol extract	Similar or related compound found in literature and investigated for metal corrosion inhibition	Metal tested	Ref.
13	27.038	Tributyl acetylcitrate	Citric acid, N-doped citric acid, Functionalization of citric acid	Aluminum pigment, mild steel, carbon steel	[94–96]
14	45.852	6-Amino-5-cyano-4-(5-cyano-2,4-dimethyl-1H-pyrrol-3-yl)-2-methyl-4H-pyran-3-carboxylic acid ethyl ester	–CN and –NO ₂ substituents in different compounds	Different metals	[97]

Declarations

Compliance with Ethical Standards

Declarations

Consent to participate is not applicable

Competing interests

The authors declare that they have no conflict of interest.

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Data availability

All data generated or analyzed during this study are included in this published article. If someone wants to request data from this study, they should contact Professor Dr. A. El Nemr.

Conflict of interest

The authors declare there is no conflict of interest regarding the publication of this article.

Author contributions

A. Elhebshi and N. Barghout carried out the main research work activities. A. El-Nemr conceptualized the main research protocol. S. Ragab conducted analyses and discussions of the novel ideas of the work. I. Ashour and M.S. El-Deab supervised the research work. A. Elhebshi and N. Barghout wrote the original manuscript and reviewed the whole manuscript with a particular attention to the electrochemical and

corrosion studies. A. El-Nemr supervised the experimental work and measurements step by step, reviewed the whole manuscript and prepared the final form of manuscript.

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Table

Table 1 is available in the Supplementary Files section.

Figures

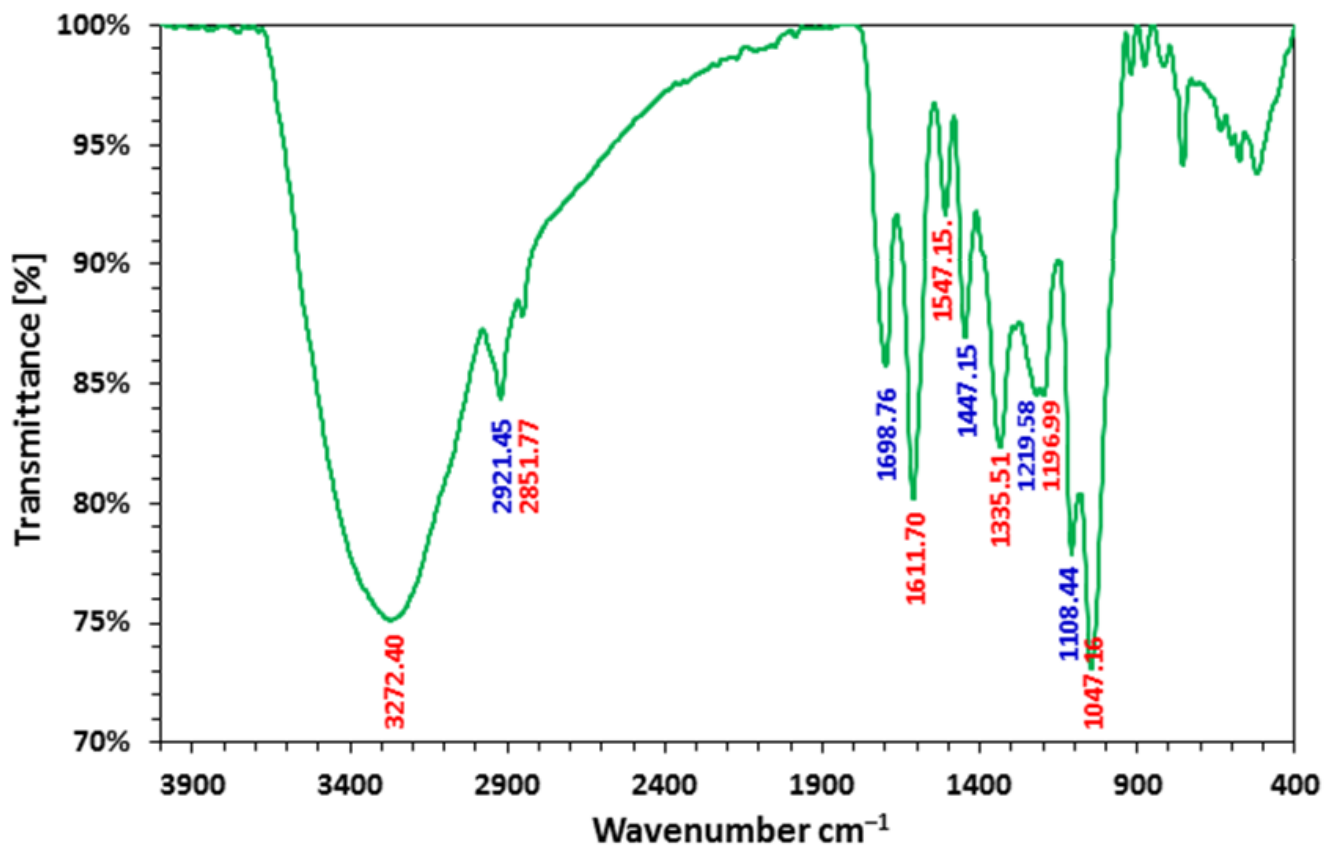


Figure 1

FTIR analysis of camphor tree bark ethanol extract (CTBEE).

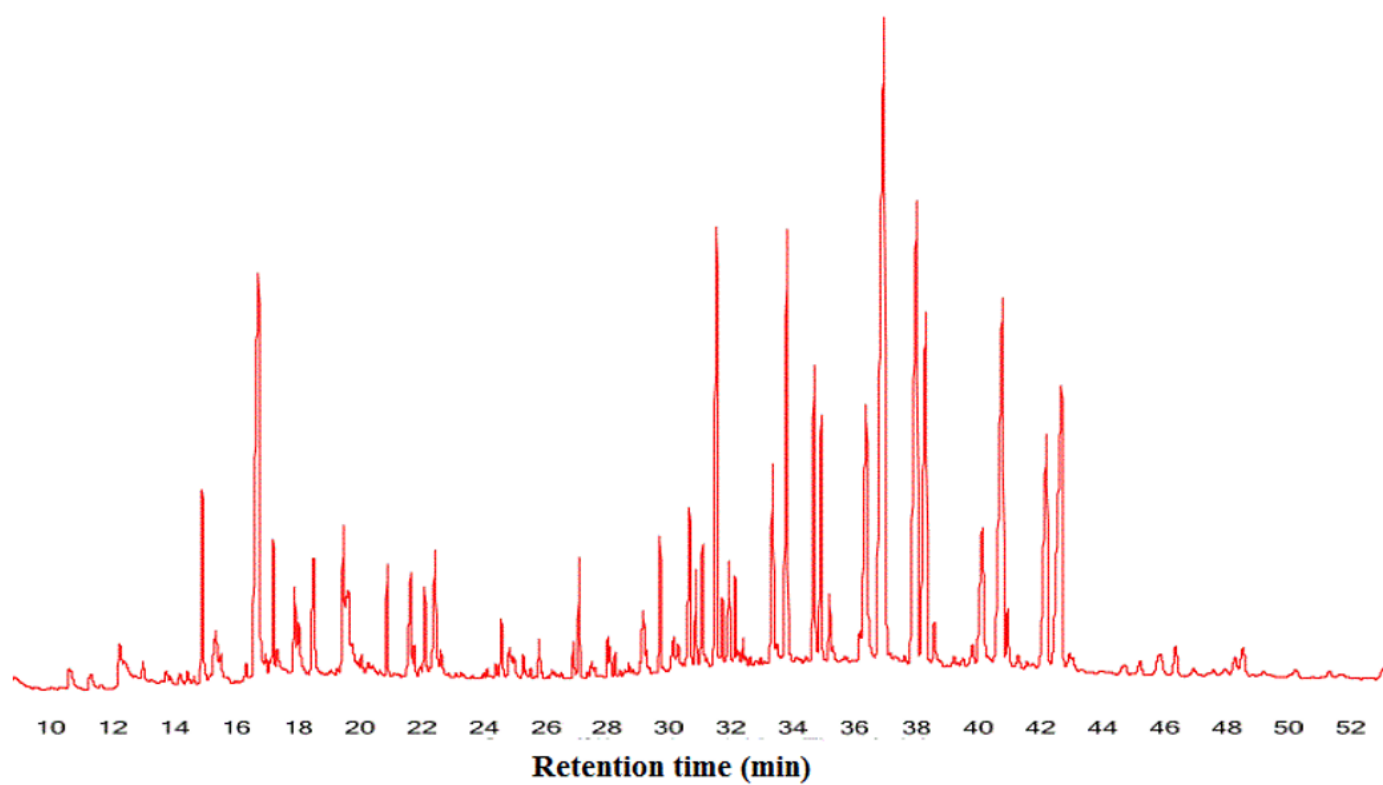


Figure 2

GC-MS analysis of camphor tree bark ethanol extract (CTBEE).

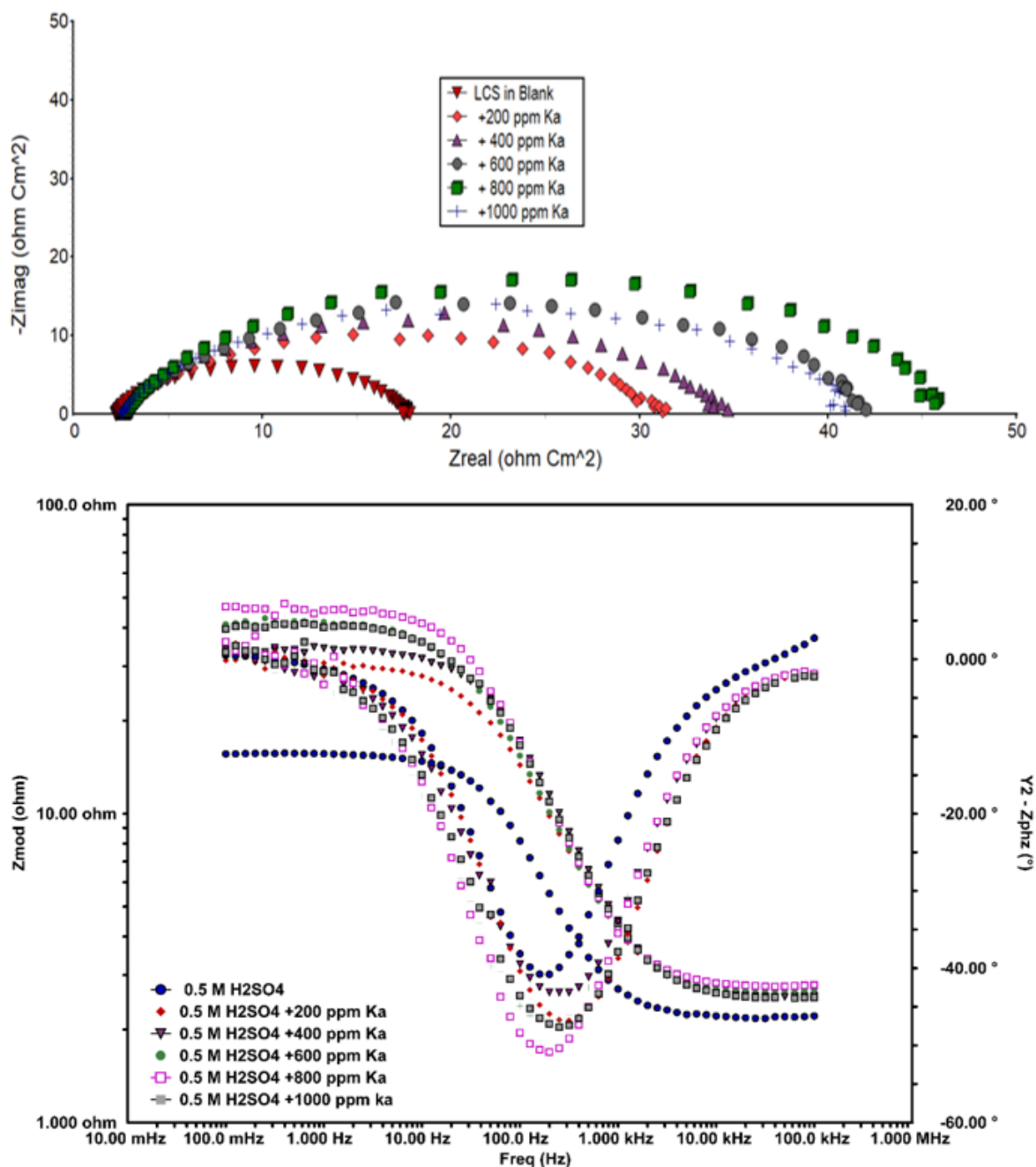


Figure 3

(a) Nyquist plot of LCS obtained at different concentrations of Camphor bark ethanol extract in 0.5 M H₂SO₄ at 298 K. (b) Bode plot of LCS obtained at different concentrations of Camphor bark ethanol extract in 0.5 M H₂SO₄ at 298 K.

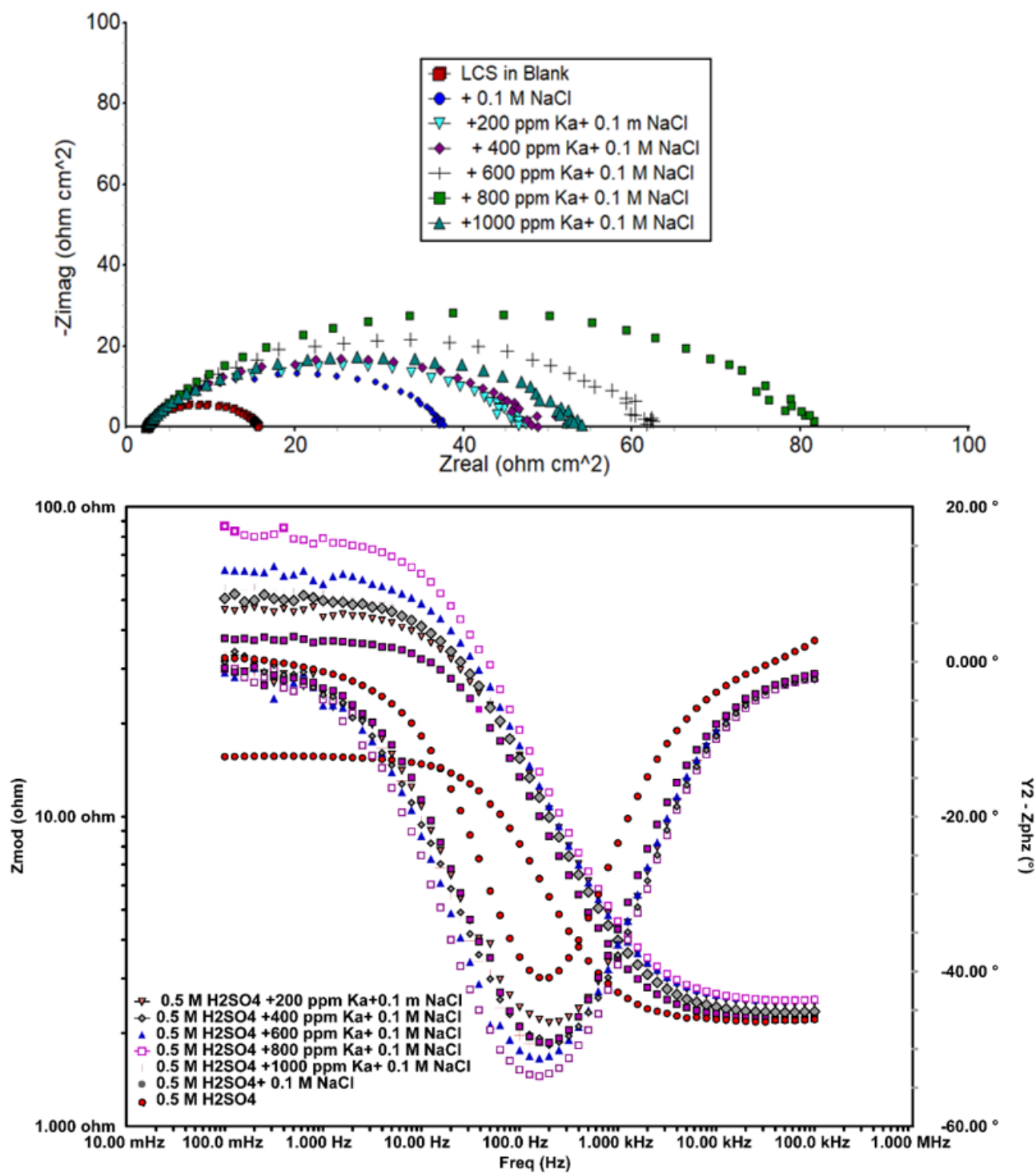


Figure 4

(a) Nyquist plot of LCS obtained at different concentrations of Camphor bark ethanol extract in 0.5 M H_2SO_4 and 0.1 M of NaCl at 298 K. (b) Bode plot of LCS obtained at different concentrations of Camphor bark ethanol extract in 0.5 M H_2SO_4 and 0.1 M of NaCl at 298 K.

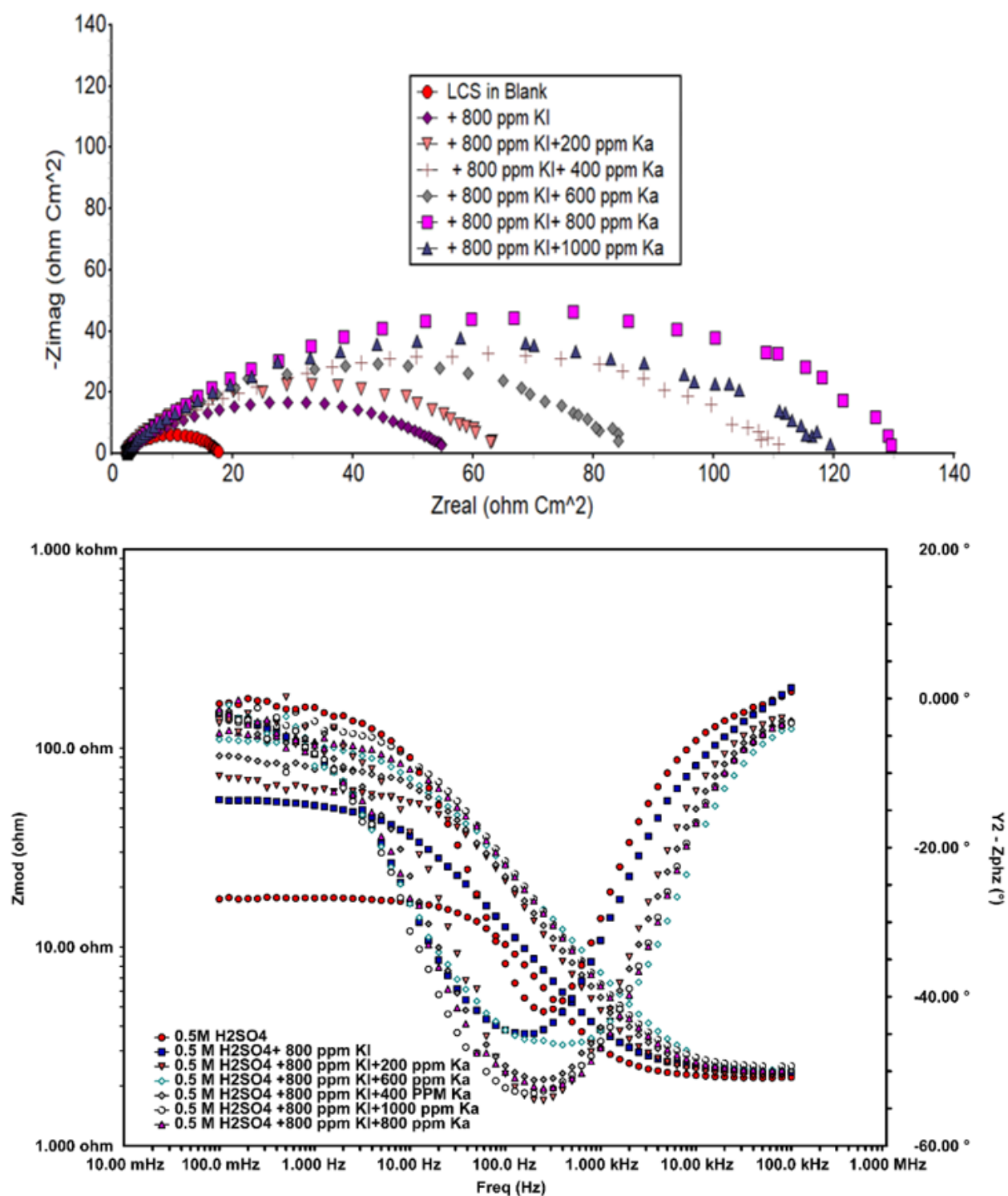


Figure 5

(a) Nyquist plot of LCS performed at different concentrations of Camphor bark ethanol extract in 0.5 M H_2SO_4 and 800 ppm of KI at 298 K. (b) Bode plot of LCS performed at different concentrations of Camphor bark ethanol extract in 0.5 M H_2SO_4 and 800 ppm of KI at 298 K.

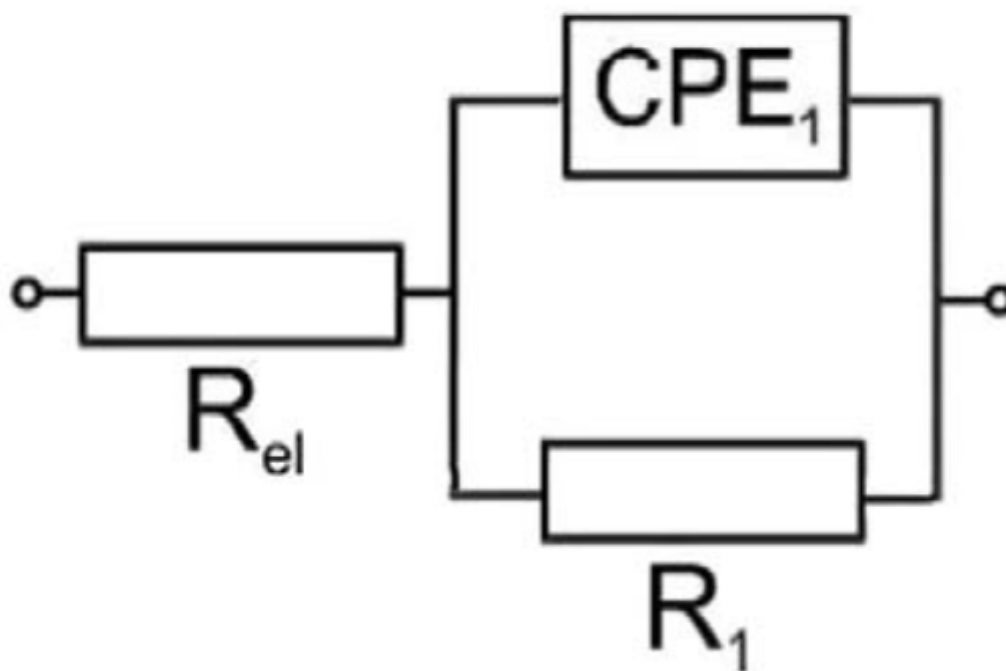


Figure 6

Proposed model of EEC that applied to simulate EIS data obtained on LCS electrode immersed in the corrosive media containing 0 and different concentration of the camphor bark ethanol extract. Where R_{el} , R_1 , and CPE_1 , are respectively, represent resistance of electrolyte, resistance of polarization and the capacitance of double-layer.

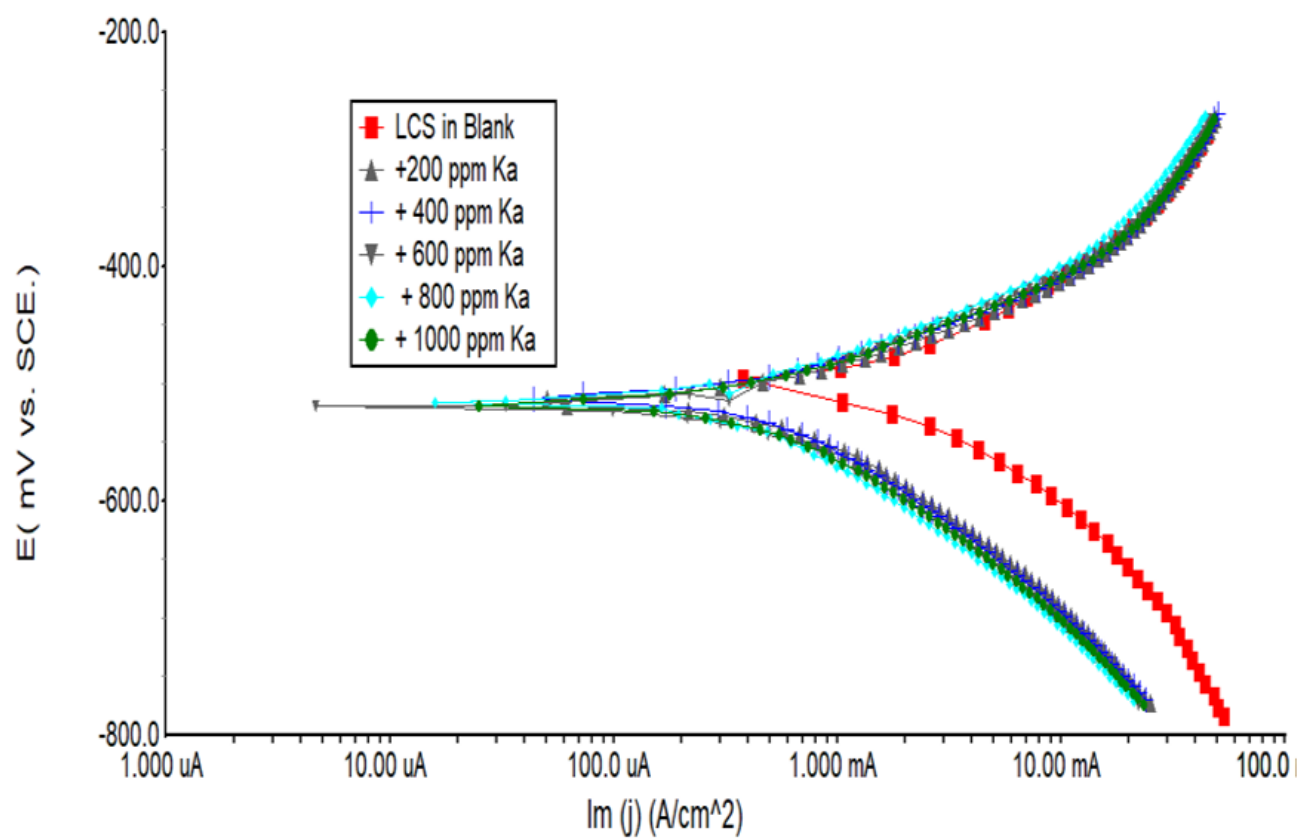


Figure 7

LCS electrode Potentiodynamic plots recorded at different concentrations of CTBEE in a 0.5 M H₂SO₄ solution. Scan rate = 5 mV/s.

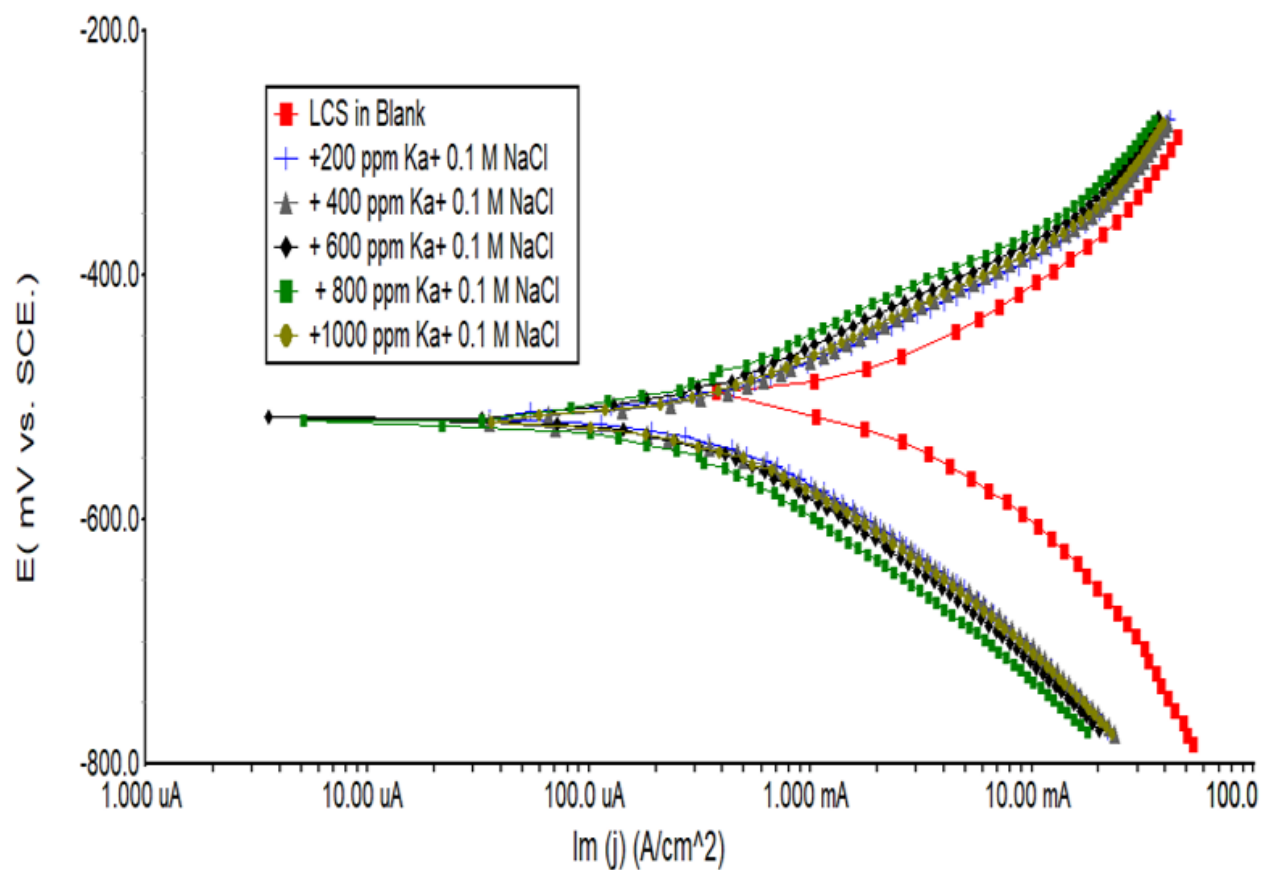


Figure 8

LCS electrode Potentiodynamic plots obtained at different concentrations of CTBEE in a 0.5 M H_2SO_4 solution in the present of 0.1 M NaCl. Scan rate = 5 mV/s.

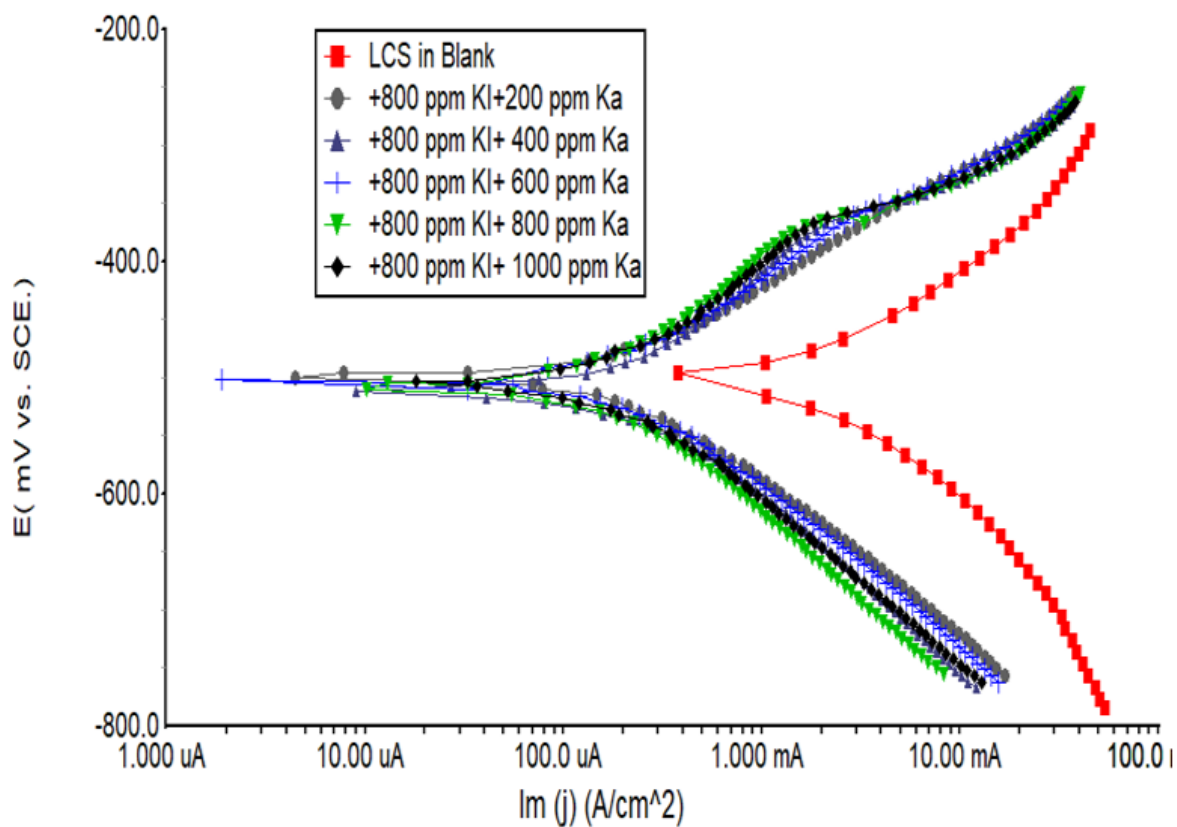


Figure 9

LCS electrode Potentiodynamic plots performed at different concentrations of CTBEE in presence of 800 ppm KI in a 0.5 M H_2SO_4 solution. Scan rate = 5 mV/s.

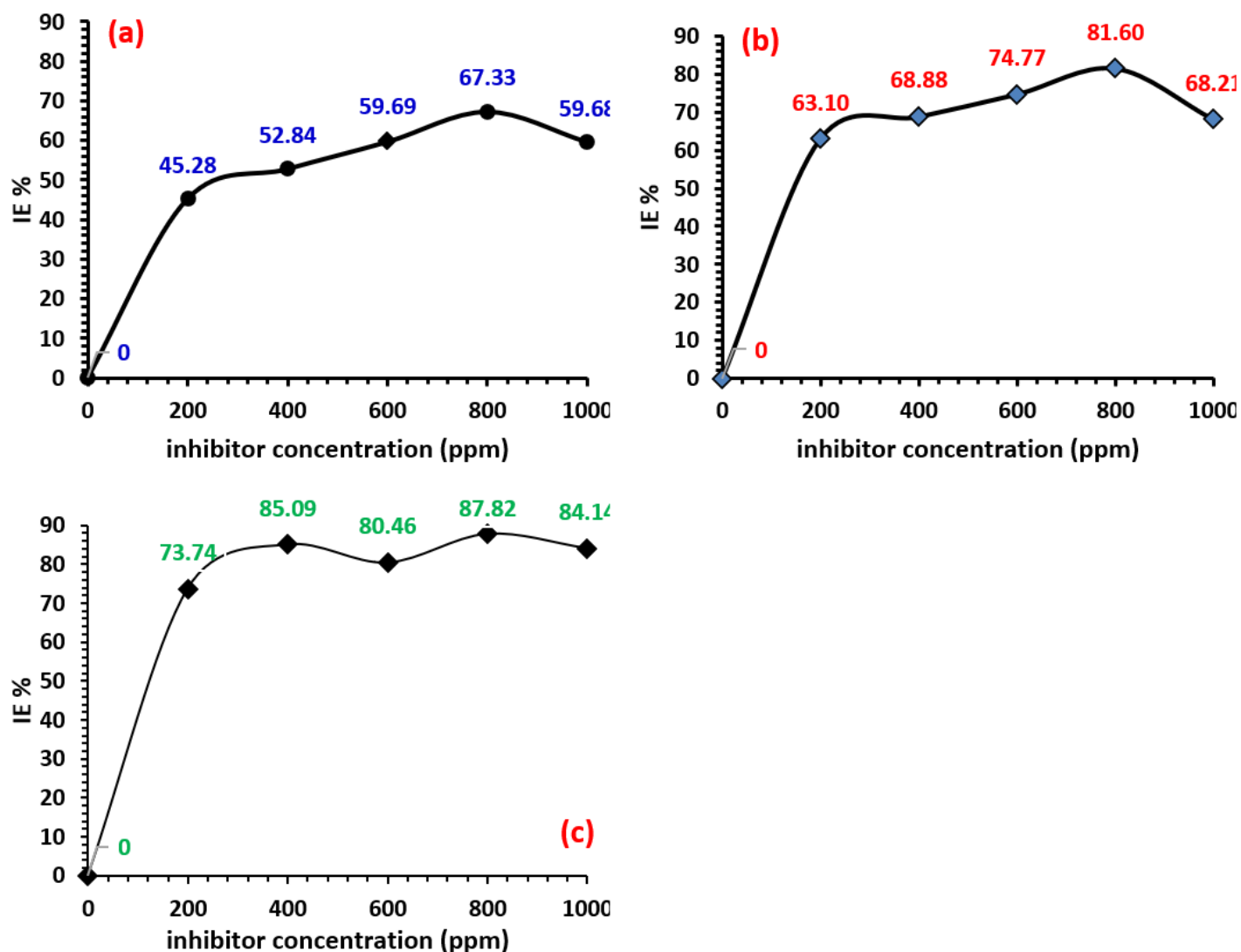


Figure 10

Fig. 9. The average corrosion IE% of LCS in 0.5 M H_2SO_4 solution obtained by EIS, and potentiodynamic measurements using (a) CTBEE (200-1000 ppm); (b) CTBEE (200-1000 ppm) in the presence of 0.1 M NaCl; (c) CTBEE (200-1000 ppm) in the presence of 800 ppm KI.

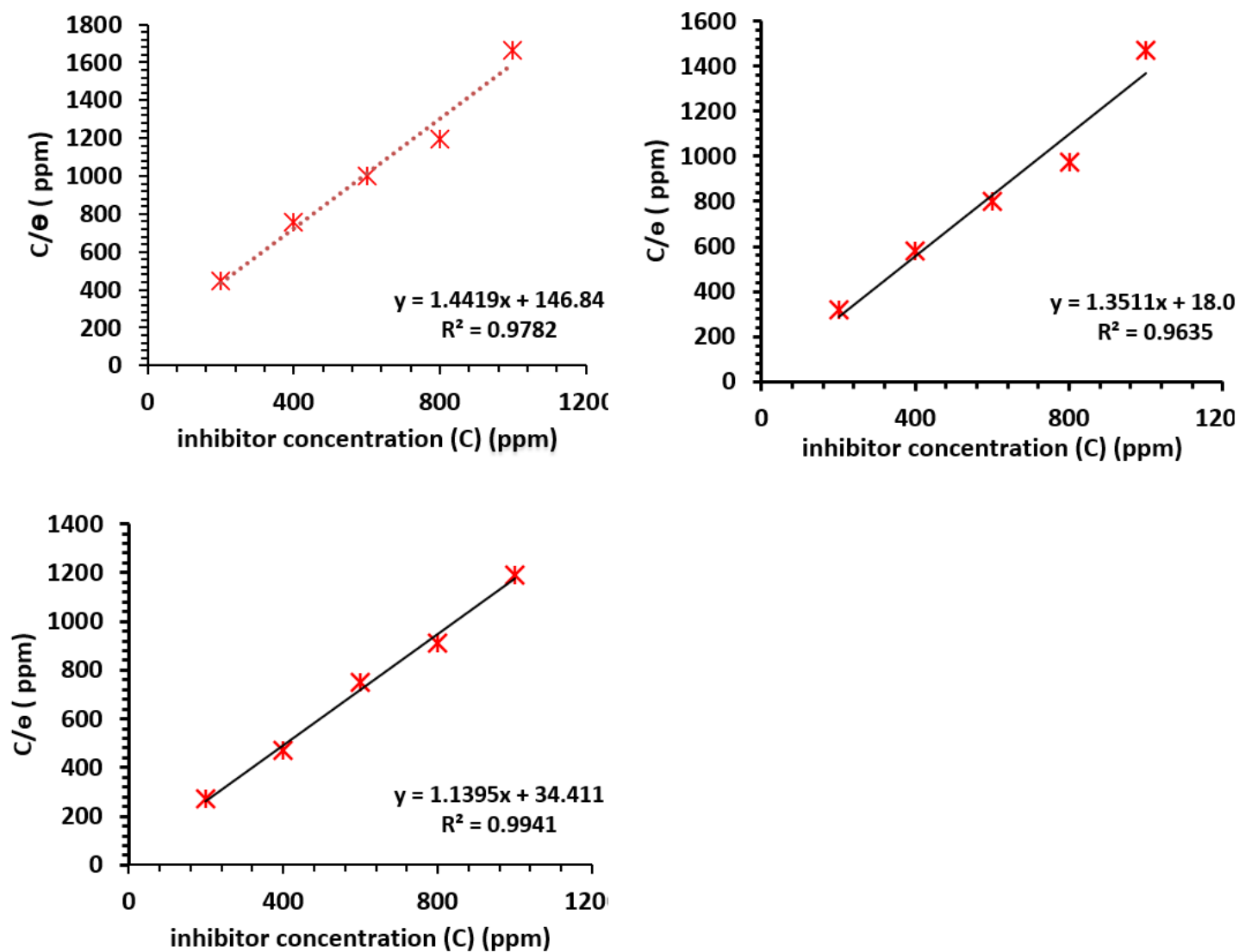


Figure 11

Fig. 10. Langmuir isotherm model plot (a) CTBEE alone on the surface of LCS; (b) CTBEE-0.1 M NaCl; (c) CTBEE-KI.

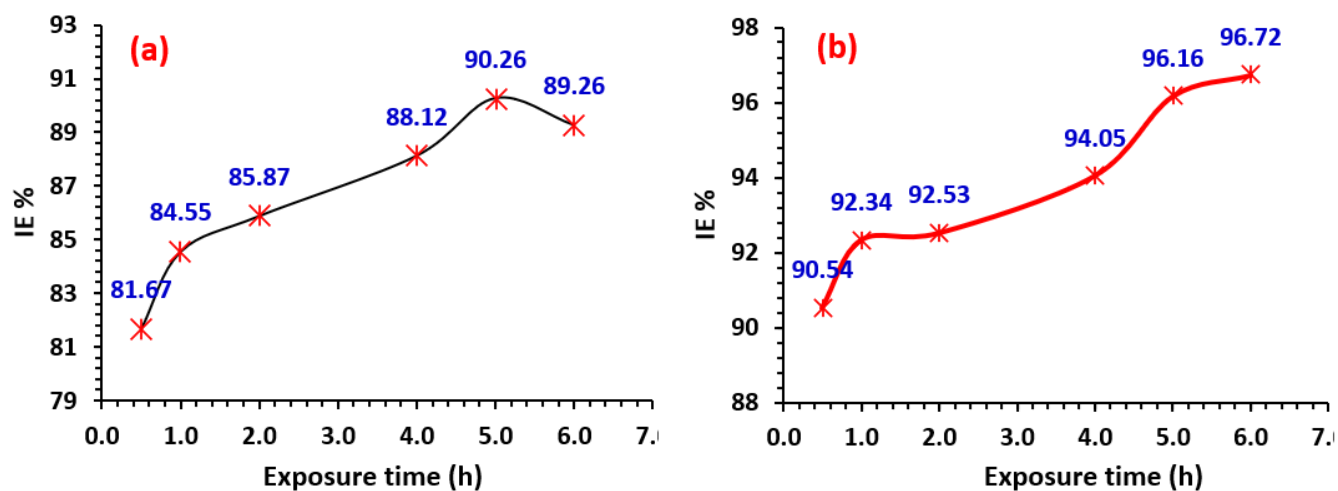


Figure 12

Fig. 11. The inhibition efficiency at different time intervals obtained from EIS experiments on LCS (a) in the presence of 800 ppm CTBEE and 0.1M NaCl; (b) in the presence of 800 ppm CTBEE and 800 ppm KI in a 0.5 M H_2SO_4 solution.

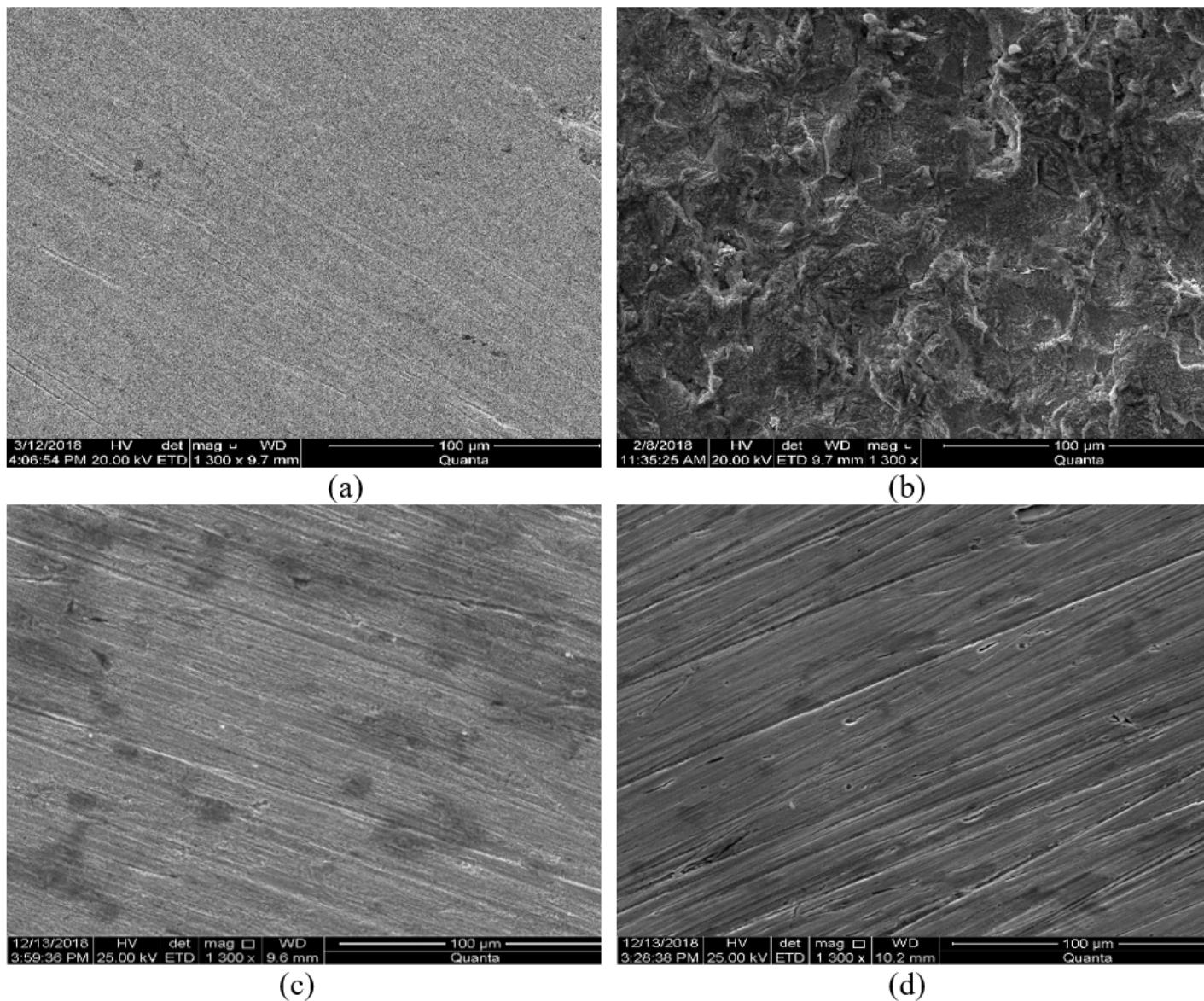


Figure 13

Fig. 12. SEM micrographs of a low carbon steel surface (a) is for the fresh sample before immersion; SEM micrographs of LCS surface taken after one day of immersion in 0.5 M H_2SO_4 solution (b) in the absence of inhibitors, (c) in the presence of 800 ppm Ka + 0.1M NaCl, (d) in the presence of 800 ppm Ka + 800 ppm KI.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Table1.docx](#)