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An Oversimplified Corrosion Inhibition Experiment Suitable for the Development of the Laboratory Practice for Undergraduate Students

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An oversimplified corrosion inhibition experiment suitable for the laboratory practice for undergraduate students was developed. The different simplifications to the standard procedure of the corrosion inhibition experiment were tested. Using the examples of steel corrosion inhibition in sulfuric acid by sodium tungstate, and in hydrochloric acid by succinic acid, it was shown that the obtained results remain representative even if the experiment is performed very roughly and inaccurately. During the standard laboratory 3-hour session, both the gravimetric and electrochemical experiments in uninhibited medium with three different inhibitor concentrations could be performed by the students. The typical methodical instructions for the students and instructors were developed. The proposed laboratory experiment was tested during an academic year with the master-level students in Novosibirsk State University.

Graphical abstract



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1. Introduction

Corrosion is the degradation and deterioration of various materials (including metals, polymers, ceramics and so on) caused by the environment. The costs associated with corrosion are enormous, and the possible failure of infrastructure caused by corrosion poses a serious threat to the human safety, health and the environment. Materials degradation prevention and mitigating is the strategic task for

any industry. The education of engineers with solid knowledge of corrosion phenomena is a very important task [1].

Corrosion engineering is an inherent part of chemical engineering, however, contrary to chemical engineering education, there is a few dedicated specific programs devoted to the corrosion prevention and control [2]. Teaching corrosion engineers usually combines both the theoretical and

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experimental approaches [3]. Laboratory practice significantly improves the students' performance during the examinations [3], and the development of laboratory experiments regarding various aspects of corrosion is an essential part of the corrosion engineering education.

There are four main groups of methods for protecting metals from electrochemical corrosion. The first group includes the application of protective coatings (metallic, ceramic, polymeric, varnish, enamels, etc). The second group includes the modification (i.e. alloying) of metals intended for work in aggressive conditions with components that enhance their corrosion resistance. The third group includes an electrochemical protection, which can be cathodic or anodic in its nature. The fourth group includes an introduction of corrosion inhibitors into an aggressive environment. The inhibitor, being adsorbed on the surface of the metal or on the surface of the oxide film formed on it, improves its protective properties and slows down the oxidation processes. From all these methods, the implementation of corrosion inhibitors is probably the simplest electrochemical experiment type. Thousands of papers devoted to the topic are published worldwide annually. Usually, several experimental techniques are used to evaluate the inhibition efficiencies of different compounds [4]. Weight loss measurements allow to estimate the average corrosion rates with and without an inhibitor over the certain time periods, and gravimetric measurements are usually considered as the reference method of inhibition efficiency estimation. Potentiodynamic polarization experiments allow to calculate the Tafel slopes of polarization curves, and to estimate the effect of inhibitor on the corrosion current density and polarization resistance of the system. Electrochemical impedance spectroscopy data provide another opportunity to estimate the polarization resistance. Finally, a typical corrosion experiment involves characterization of the surface and corrosion products, and estimation of the nature and thermodynamic parameters of inhibitor adsorption on the metal surface.

However, in order to run the corrosion inhibition experiment accurately and to obtain reproducible data, one needs to prepare the sample electrodes with the constant surface. The experiment itself is time-consuming. This makes it difficult to transform the accurate corrosion inhibition experiment into the laboratory practice for the undergraduate students due to the limited time of the standard laboratory session. Therefore, the way to simplify and shorten the experiment needs to be found. Moreover, the least diligent and motivated students might work very inaccurately, and the experiment should be enough robust to allow the students to obtain representative result even if they make some experimental mistakes.

Previous attempts to develop a student laboratory experiment on corrosion involved only weight loss measurements [5, 6], visual inspection of the formation of the coloured complex of dissolved metal [7], or visual inspection of the corroded surface [8]. No student laboratory experiment involving different techniques was previously reported. Therefore, this study aims to shorten the corrosion inhibition experiment to the standard 3-hour laboratory session, to test its sensitivity to the inaccurate work, and to develop the novel combined and diverse laboratory experiment on corrosion for the students. The simplifications applied to common corrosion inhibition experiments are listed in the experimental section.

During the laboratory session, students perform gravimetric measurements and estimate the corrosion rates in presence and in absence of the chosen inhibitor. In parallel

they conduct electrochemical experiments. The open circuit potential measurements are used to identify the inhibitor type (anodic or cathodic) and to qualitatively see the influence of the inhibitor concentration in the solution on the open circuit potential shift. From the potentiodynamic polarization experiments students estimate the Tafel slopes of both anodic and cathodic branches of polarization curves, calculate the corrosion current density and polarization resistance. From the electrochemical impedance experiments students estimate the solution and charge transfer resistances. The inhibition efficiencies estimated from both gravimetric, polarization and impedance data are then compared to each other. Finally, students test the suitability of the Langmuir adsorption isotherm for the description of the inhibitor sorption of the metal surface and estimate the adsorption-desorption equilibrium constant and the Gibbs energy of adsorption.

The laboratory practice was performed during an academic year with the master-level students of Novosibirsk State University. Different common and well-known inhibitors were tested for suitability for the student laboratory practice, including the inorganic (KSCN , Na_2MoO_4 , Na_2WO_4), and organic (benzoic acid, oxalic acid, succinic acid) reagents common for any university laboratory. All these inhibitors were found suitable for the simplified laboratory corrosion experiment. Two examples are presented and discussed in this paper. The first one is the sodium tungstate, which proved itself as the very good inhibitor for the different alloys in various environments [9–17]. In this work its inhibition efficiency was estimated against the corrosion of steel in sulfuric acid with the addition of unknown impurities coming from tap water. The second one is the succinic acid, which is also a good inhibitor for steel in acidic environments [18–23]. In this work its inhibition efficiency was estimated against the corrosion of steel in hydrochloric acid.

2. Material and Methods

2.1 Reagents and Equipment

Ethanol (analytical grade), hydrochloric and sulfuric acids (pure grade), sodium tungstate dihydrate (analytical grade) and succinic acid (analytical grade) were purchased from LLC "Lenreaktiv".

Weighting of the samples was performed using the analytical balance EX224/AD (Ohaus Corp.). Electrochemical and EIS measurements were conducted using the potentiostat-galvanostat PS-50 (LLC "SmartStat"). The mercury-mercurous sulphate reference electrode by Schott Intstruments GmbH was used. The distilled water for solution preparation was produced using the aquadistiller AE-15 (LLC "Livam"). The magnetic stirrer MS-200 LT (LLC "Labtex") was used for stirring and heating the solutions. The single-channel laboratory pipettes manufactured by Thermo Fisher Scientific were used for pipetting the solutions. A laboratory glassware of 2nd grade was used.

2.2 A simplified corrosion experiment

The following simplifications were tested:

- A common steel unbent paperclip was used as the working electrode. The unused surface of the steel wire was not sealed, and the metallic surface in the contact with the solution was not held constant. The actual electrode surface was estimated after each experiment by measuring the surface of the wire

piece covered by the corrosion products. This makes the working electrodes easily replaceable in case of damage, and eliminates the necessity of time-consuming electrode preparation. The exact composition of the steel is also unknown, because it makes no sense for the laboratory practice.

- A graphite rod from the common pencil was used as the counter electrode. Its surface in the contact with the solution was also not held constant.
- A common 100 mL glass beaker was used as the electrochemical cell. No separate compartments for counter and reference electrodes were made, all three electrodes were immersed into the same corrosive medium. The electrode positions were not fixed. The electrodes were just fastened with the common laboratory stand by clamps and immersed into the solution, and consequently, the exact position and distance between the electrodes in each run might slightly differ. No Luggin capillary was used, and the IR drop was not compensated.
- The solutions were not stirred and not de-aerated.
- No water bath was used for the accurate temperature maintenance. The experiments were run at the room temperature, and the possible room temperature deviations were neglected.
- Each experiment was run only once, and without replicates. This allows to reduce the total time significantly.
- The open circuit potential was measured only over 15 minutes, even if it did not equilibrate and stabilize during this time period.
- The measurement of open circuit potential, electrochemical impedance spectrum, and polarization curve for each experiment was performed consecutively in the same medium, without replacing the solution. The possible dissolution of the sample during the open circuit and impedance measurements was therefore neglected.
- For the gravimetric measurements the small pieces of the steel wire were used instead of the rectangular plates.
- In order to simulate the possible inaccurate work by some least diligent students, the solutions were prepared using the graduated cylinders instead of the volumetric flasks.
- In order to simulate the possible inaccurate work, the solutions were prepared using the tap water of unknown composition instead of the distilled water. This introduced the unknown impurities.

2.3 Preparation of the solutions

112.26 mg of sodium tungstate dihydrate (which corresponds to 100.0 mg of anhydrous sodium tungstate) was dissolved in 100 mL of tap water of unknown composition on heating and stirring thus obtaining a 1000 ppm solution. 300 and 100 ppm solutions were prepared by diluting the 1000 ppm solution by tap water. All three sodium tungstate solutions were then equally diluted by the 1.0 M H_2SO_4 to produce a series of the acidic sodium tungstate solutions with concentrations of 50, 150, and 500 ppm in 0.5 M H_2SO_4 with the additions of the unknown dissolved substances coming from tap water.

50.0 mg of succinic acid was dissolved in 100 mL of 0.5 M HCl on heating and stirring thus obtaining a 500 ppm solution. 150 and 50 ppm solutions were prepared by diluting the 500 ppm solution by 0.5 M HCl.

All solutions were prepared using graduated cylinders instead of volumetric flasks.

2.4 Gravimetric studies

The unbent steel paperclips were cut to the small steel wire pieces, polished using the P2500 emery paper, degreased by ethanol and dried, their geometric parameters (length and diameter) were measured. The weighed pieces were immersed into the test tubes with the corrosive media for 2 h, then washed with distilled water, dried, and reweighed. From the measured weight losses, sample surface areas, and immersion times the corrosion rates were estimated. An inhibitory ability of the compound was estimated from the ratio of the corrosion rates in the absence and in the presence of the inhibitor. The corresponding equations are presented in the Supplementary Information.

2.5 Electrochemical studies

The working electrodes were made of unbent steel paperclips, polished using the P1000 emery paper and degreased by ethanol. The common wooden pencils were immersed in water and left overnight to dissolve the glue, then the wooden parts were removed, and the graphite rods were dried and used as the counter electrodes. A mercury-mercurous sulfate reference electrode was used. The electrochemical tests were carried out in a common 100 mL glass beaker, and the test solution volume was equal to 70 mL. The electrodes were fastened using the common laboratory stand by clamps, immersed into solution and connected to a potentiostat. The appearance of the electrochemical cell is shown in Figure 1.

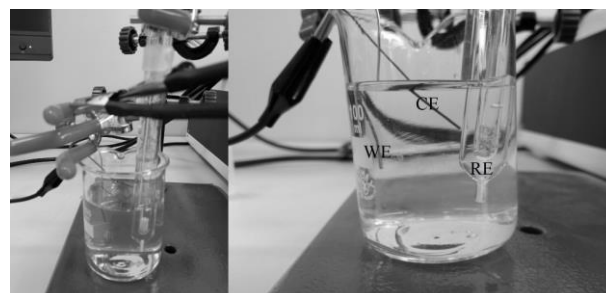


Fig. 1. The appearance of the simplified electrochemical cell for corrosion experiments.

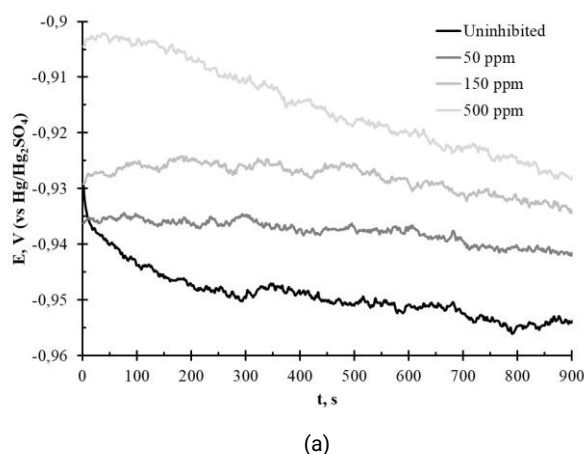
An open circuit potential (E_{corr}) was recorded during 15 min. Impedance spectra were recorded directly thereafter without replacing the solution at the open circuit potential value in the alternate current frequency interval from 100 mHz to 10 kHz with the potential amplitude of 10 mV. The obtained results were presented in the form of Bode and Nyquist plots. For the estimation of the impedance parameters, a simplified Randles equivalent electrical circuit [24], containing the solution resistance R_s , the consecutive charge transfer resistance R_{ct} of the passivation layer, and the parallel constant-phase element representing the double electric layer, was employed. The equation for the impedance of this equivalent circuit [25] is presented in the Supplementary Information.

The fitting of the equivalent circuit parameters to the experimental impedance values was performed using the free software EIS Spectrum Analyser [26]. In addition, the electric double layer capacitance was estimated. The inhibitory ability of the compounds used was estimated from the ratio of the charge transfer resistances in the presence and in the absence of the inhibitor.

Polarization curves were recorded directly after the impedance measurements without replacing the solution in the potential range from -500 to $+500$ mV relatively to the measured open circuit potential with the potential sweep rate of 10 mV/s. After the end of experiment, the corroded surface area was measured to calculate the current density. The obtained polarization curves were presented in the coordinates $E(\lg i)$, and the Tafel slopes [27] (b_a and b_c) and the corrosion current density (i_{corr}) were evaluated. Subsequently, the polarization resistances (R_p) were estimated using the Stern–Geary equation [28, 29].

The inhibitory ability of the compound was estimated both from the ratio of the corrosion current densities in the absence and in the presence of the inhibitor, and from the ratio of the calculated polarization resistances in the presence and in the absence of the inhibitor. The experiments with each corrosive medium were run only once.

3. Results and Discussion



3.1 Gravimetric studies

The dependence of the measured inhibition efficiencies at the different inhibitor concentrations (C_{inh} , ppm) is presented in Table 1.

Table 1. The results of the gravimetric measurement of the corrosion rates.

| C_{inh} , ppm | ω , mg/(cm ² · h) | η , % |
|-------------------------------------------------------------------------------------------------------------------------|-------------------------------------|------------|
| The additions of sodium tungstate and unknown dissolved substances to the 0.5 M H ₂ SO ₄ solution | | |
| 0 | 0.82 | – |
| 50 | 0.48 | 42 |
| 150 | 0.34 | 59 |
| 500 | 0.24 | 71 |
| The additions of succinic acid to the 0.5 M HCl solution | | |
| 0 | 0.74 | – |
| 50 | 0.47 | 37 |
| 150 | 0.31 | 58 |
| 500 | 0.18 | 76 |

3.2 Electrochemical studies

The results of the open circuit potential measurements are presented in Figure 2.

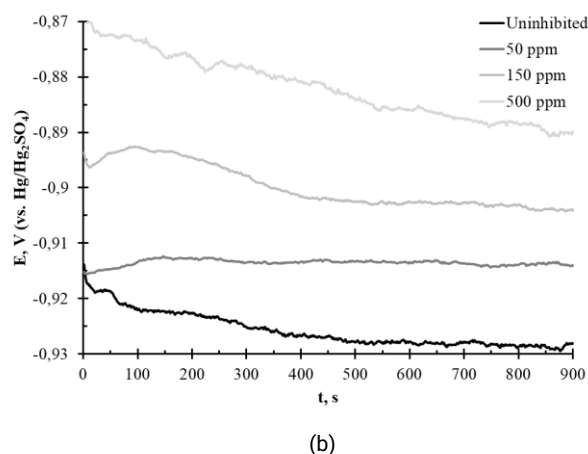


Fig. 2. The open circuit potential of steel in a) 0.5 M H₂SO₄ with the different additions of sodium tungstate and addition of the unknown dissolved substances, b) 0.5 M HCl with the different additions of succinic acid after 15 min of exposure.

The results of the electrochemical impedance measurements are presented in fig. 3 and 4, and in Table 2.

Table 2. The results of the EIS measurement of the corrosion rates.

| C_{inh} , ppm | R_s , Ohm | P , mOhm ⁻¹ ·s ⁿ | n | C_{dl} , μF | R_{ct} , Ohm | η , % |
|-------------------------------------------------------------------------------------------------------------------------|-------------|------------------------------------------|------|---------------|----------------|------------|
| The additions of sodium tungstate and unknown dissolved substances to the 0.5 M H ₂ SO ₄ solution | | | | | | |
| 0 | 0.87 | 0.12 | 0.90 | 92 | 58 | – |
| 50 | 0.81 | 0.11 | 0.90 | 64 | 84 | 31 |
| 150 | 0.32 | 0.11 | 0.89 | 40 | 118 | 51 |
| 500 | 0.38 | 0.09 | 0.91 | 61 | 157 | 63 |
| The additions of succinic acid to the 0.5 M HCl solution | | | | | | |
| 0 | 2.3 | 0.18 | 0.83 | 86 | 317 | – |
| 50 | 2.1 | 0.07 | 0.82 | 31 | 619 | 49 |
| 150 | 1.5 | 0.15 | 0.88 | 79 | 975 | 63 |
| 500 | 1.7 | 0.05 | 0.81 | 26 | 1194 | 73 |

The results of the polarization measurements are presented in Figure 5 and in Table 3.

Table 3. The results of the electrochemical measurement of the corrosion rates.

| C_{inh} , ppm | E_{corr} , mV | b_a , mV/dec | b_c , mV/dec | R_p , Ohm·cm ² | η , % | i_{corr} , μA/cm ² | η , % |
|-------------------------------------------------------------------------------------------------------------------------|-----------------|----------------|----------------|-----------------------------|------------|---------------------------------|------------|
| The additions of sodium tungstate and unknown dissolved substances to the 0.5 M H ₂ SO ₄ solution | | | | | | | |
| 0 | –954 | 53 | –150 | 168 | – | 540 | – |
| 50 | –942 | 38 | –159 | 594 | 72 | 200 | 63 |
| 150 | –934 | 30 | –162 | 1214 | 86 | 126 | 77 |
| 500 | –928 | 27 | –157 | 1369 | 88 | 119 | 78 |
| The additions of succinic acid to the 0.5 M HCl solution | | | | | | | |
| 0 | –928 | 121 | –110 | 1717 | – | 77.1 | – |
| 10 | –914 | 81 | –140 | 2581 | 33 | 45.6 | 41 |
| 30 | –904 | 72 | –121 | 5296 | 68 | 19.6 | 75 |
| 100 | –889 | 55 | –114 | 8794 | 80 | 9.7 | 87 |

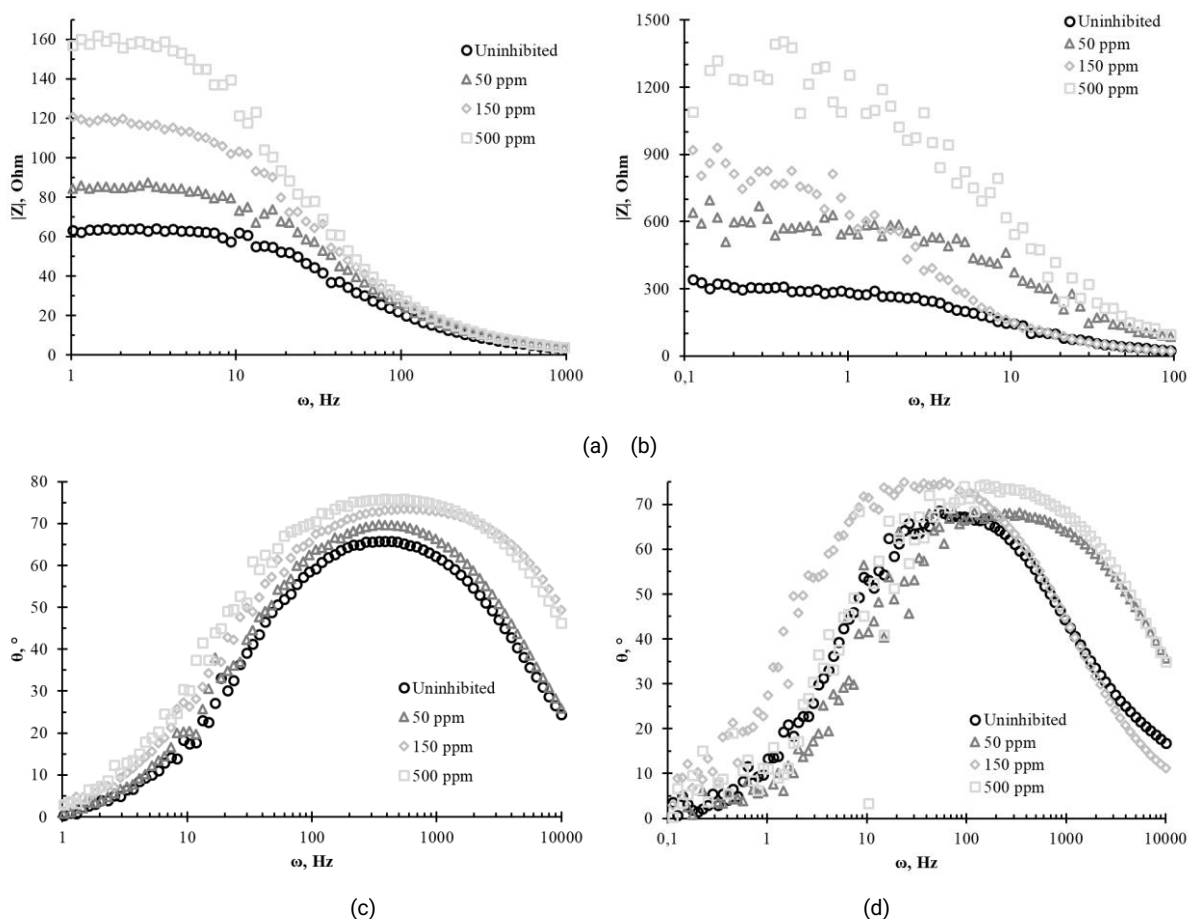


Fig. 3. The Bode plot of steel in a), c) 0.5 M H_2SO_4 with the different additions of sodium tungstate and addition of the unknown dissolved substances, b), d) 0.5 M HCl with the different additions of succinic acid after 15 min of exposure.

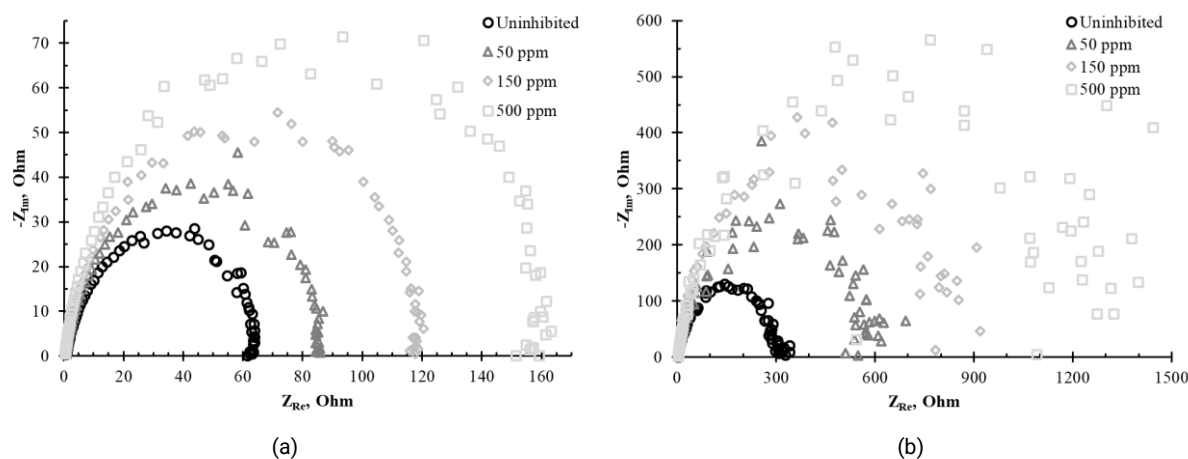


Fig. 4. The Nyquist plot of steel in a) 0.5 M H_2SO_4 with the different additions of sodium tungstate and addition of the unknown dissolved substances, b) 0.5 M HCl with the different additions of succinic acid after 15 min of exposure.

3.3 Langmuir adsorption model

The description of inhibitor adsorption on steel surface was performed in terms of the Langmuir adsorption model. The Langmuir adsorption isotherm equation [30] was linearised in the form: $\frac{c_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + c_{\text{inh}}$, where c_{inh} is concentration of the inhibitor solution, ppm, K_{ads} is the (dimensionless) adsorption-desorption equilibrium constant, and θ is the percentage of the surface covered by the inhibitor,

which assumed to be equal to the inhibition efficiency η . The dependencies of c_{inh} / θ on c_{inh} are presented in Figure 6. The data were processed using the least squares technique, and the equilibrium constants K_{ads} were estimated as the intercepts of the regression equations. The Gibbs energy changes of the sorption were also estimated.

The results are presented in Table 4.

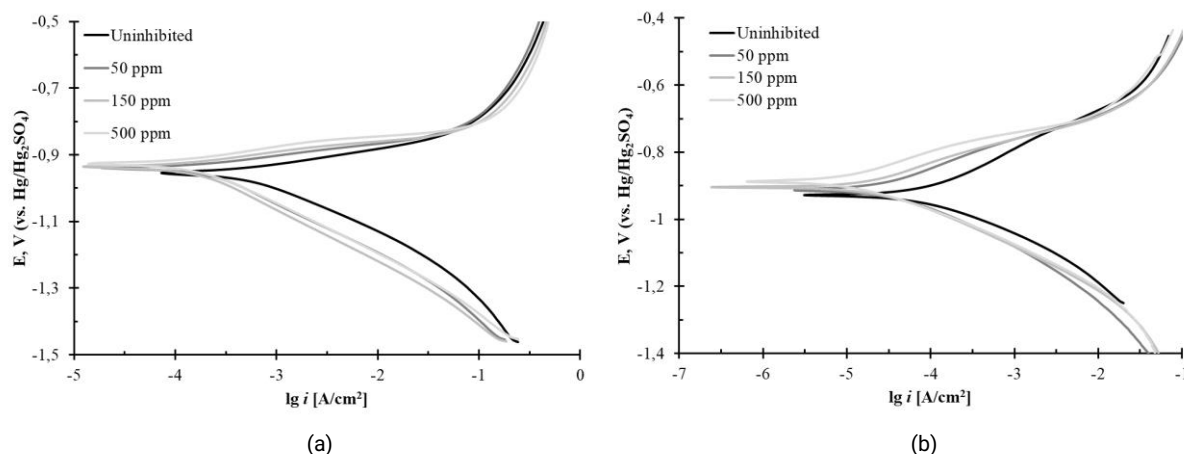


Fig. 5. The potentiodynamic polarization curves of steel in a) 0.5 M H₂SO₄ with the different additions of sodium tungstate and addition of the unknown dissolved substances, b) 0.5 M HCl with the different additions of succinic acid after 15 min of exposure.

Table 4. The parameters of the Langmuir adsorption model.

| C _{inh} , ppm | θ | C _{inh} / θ, ppm | Regression equation | K _{ads} · 10 ³ | Δ _{ads} G, kJ/mol |
|-------------------------------------------------------------------------------------------------------------------------|------|---------------------------|--------------------------------------|------------------------------------|----------------------------|
| The additions of sodium tungstate and unknown dissolved substances to the 0.5 M H ₂ SO ₄ solution | | | | | |
| 50 | 0.31 | 161 | C _{inh} / θ = (1.41 ± 0.02) | 12 ± 1 | -23 ± 1 |
| 150 | 0.51 | 294 | C _{inh} + (87 ± 5); | | |
| 500 | 0.63 | 794 | R ² = 0,999 | | |
| The additions of succinic acid to the 0.5 M HCl solution | | | | | |
| 50 | 0.49 | 102 | C _{inh} / θ = (1,29 ± 0,02) | 24 ± 3 | -25 ± 1 |
| 150 | 0.63 | 238 | C _{inh} + (41 ± 5); | | |
| 500 | 0.73 | 685 | R ² = 0,999 | | |

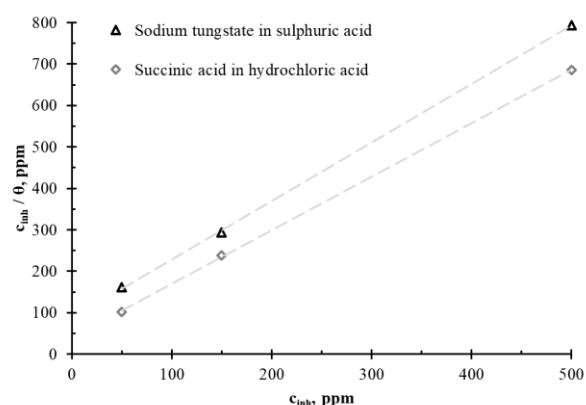


Fig. 6. The plots of C_{inh}/θ vs. C_{inh} for the adsorption of sodium tungstate and succinic acid on steel surface.

3.4 Discussion

Table 5 shows a comparison of the inhibitory properties of sodium tungstate and succinic acid against the corrosion of various alloys in different environments from the literature data and obtained in the present work.

Table 5. The inhibitory action of sodium tungstate and succinic acid on the corrosion of various alloys in different corrosive media.

| Inhibitor | Alloy | Corrosive medium | Inhibitor concentration range | Inhibition efficiency range | Δ _{ads} G, kJ/mol | Reference |
|---------------------------------|--------------------|--------------------------------------------------------------------|---------------------------------------|-----------------------------|----------------------------|-----------|
| Na ₂ WO ₄ | Mild steel | 0.5% peracetic acid | 0.3 – 4.55 mM | 15 – 95% | -31 | [9] |
| | Mild steel | Aerated distilled water | 10 ⁻⁶ – 0.1 M | Not specified | Not specified | [10] |
| | Mild steel | 0.1 M NaCl, 0.1 M Na ₂ SO ₄ | 10 ⁻⁶ – 0.1 M | 20 – 80% | Not specified | [11] |
| | Fe-Cr-Ni alloy | 10% H ₂ SO ₄ | 0.01 – 0.1 M | 80 – 95% | Not specified | [12] |
| | Al ₂ Cu | 0.5 M H ₃ PO ₄ | 10 – 200 mM | Not specified | Not specified | [13] |
| | Mild steel | 0.5 M NaCl | 100 – 800 ppm | 30 – 75% | Not specified | [14] |
| | Mild steel | 2.45 g/L NaCl + 0.45 g/L Na ₂ SO ₄ | 50 ppm | 75% | Not specified | [15] |
| | Al | 0.5 M NaCl | 10 ⁻⁴ – 0.1 M | Not specified | Not specified | [16] |
| | Al | 2M HCl | 0.15 – 0.4 M | 10 – 65% | Not specified | [17] |
| Succinic acid | Mild steel | 0.5 M H ₂ SO ₄ + unknown dissolved substance | 50 – 500 ppm | 30 – 70% | -23 | This work |
| | Carbon steel | Natural water, pH 3 | 10 – 80 ppm | 30 – 60% | -11 | [18] |
| | Mild steel | 0.01 M H ₂ SO ₄ | 10 ⁻⁷ – 10 ⁻³ M | 20 – 80% | Not specified | [19] |
| | Carbon steel | 1.0 M HCl | 10 ⁻⁴ – 0.1 M | 10 – 80% | Not specified | [20] |
| | Mild steel | 1.0 M HCl | 10 ⁻⁶ – 10 ⁻⁴ M | 60 – 90% | Not specified | [21] |
| | Carbon steel | 1.0 M HCl | 10 ⁻⁴ – 0.1 M | 10 – 80% | -25 | [22] |
| | Carbon steel | 0.6 M NaCl | 0.6 M | 55% | -33 | [23] |
| | Mild steel | 0.5 M HCl | 50 – 500 ppm | 40 – 80% | -25 | This work |

As one might see, the inhibitory properties of both sodium tungstate and succinic acid against the corrosion of different steels and alloys in both neutral and acidic environments were previously tested. The values of the inhibition efficiency obtained in this work agree reasonably fair with those reported in the literature. This implies that the corrosion inhibition experiment was found so robust, that even if it is oversimplified and performed very roughly and inaccurately, it still could give the representative results. This makes the corrosion inhibition experiment suitable for laboratory practice of undergraduate students.

If each electrochemical experiment including the recording of the open circuit potential, the electrochemical impedance spectrum, and the polarization curve is performed only once, it takes about 35 minutes to proceed. Consequently, during the standard 3-hour laboratory session four experiments could be performed, including one with the absence of the inhibitor, and three with the different inhibitor concentrations. This allows the students not only to evaluate the inhibition efficiency of the inhibitor of choice at three different points, but also to construct the Langmuir adsorption isotherm and to estimate the sorption-desorption equilibrium constant and the Gibbs energy of adsorption.

The gravimetric experiment could be run in parallel with the electrochemical studies, if the students work in pairs. This enables the students to perform a full spectrum of common quantitative measurements of inhibition activity within a single laboratory session. The students could vary the tested inhibitors and the corrosive environments according to the instructor's choice. The obtained results are representative enough to introduce the students with the typical corrosion laboratory tests.

A typical methodical instruction for the students was developed and presented in the Supplementary Information.

4. Conclusions

An oversimplified corrosion inhibition experiment suitable for the laboratory practice for undergraduate students was developed. It requires only the potentiostat and the common laboratory equipment and glassware. The common glass beaker is used as the electrochemical cell, the unbent paperclips and the graphite rods from the pencils are used as the electrodes, and each experiment is run only once. During the standard laboratory 3-hour session, both the gravimetric and electrochemical experiments in uninhibited medium and with three different inhibitor concentrations could be performed. The obtained results are enough representative to introduce the students with the common quantitative methods of study of corrosion inhibitors.

Supporting Information

A theoretical background and a detailed description of the experiment for students and instructors.

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Author Contributions

PA Nikolaychuk and NS Maltseva performed the experiments, MV Lebedeva and DV Kozlov conducted the data analysis and interpretation of the results. All authors prepared the draft and revised the manuscript.

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