INTRODUCTION

1. Significance of the study

The pickling solution and corrosion inhibitors are commonly composed of chemicals, organic substances with synthetic-origin, they are not environmental friendly. Nitrite, chromate and other organic substances that contain aromatic rings and heterocyclic elements are common inhibitors which gave high corrosion inhibitory effeciency but due to the disadvantages such as causing cancer, hazardous to the environment, therefore, the application of these corrosion inhibitors is limited.

Other than the above-mentioned issue, the corrosion inhibitors from plant extraction, which has organic origin, have the ability to decompose or could release into the environment with no or a few risk of pollution. To be known as "green" corrosion inhibitor, these substances are usually accessible, easy to made, low cost, safe. That is why recent studies on corrosion inhibition tend to focus on green inhibitors, natural-derived inhibitors, or environmentally-friendly plant extracts in order to replace hazzardous inhibitors.

The rose myrtle (*Rhodomytus tomentosa* (Aiton) Hassk.) is an evergreen shrub growing wildly in vast of the region of Vietnam. The plant contains characteristics and a large amount of natural compounds that have potential applications for corrosion and metal protection, thus this plant is chosen to be the research subject of this thesis "Study on the ability of inhibition corrosion of rose myrtle leaves extract (*Rhodomyrtus tomentosa* (Ait.) Hassk.) aiming at application for industrial pickling".

2. Research Objectives

Contributed to the study, evaluate the corrosion inhibition ability of CT3 steel substrate of rose myrtle leaves extract, study on the kinetic and mechanism of corrosion inhibition process. Applied the research results to use rose myrtle leaves extract as a corrosion inhibitor in metal rust removal process for CT3 steel in acidic environment.

3. Scope of study

- Isolating and evaluating the corrosion inhibition ability of CT3 steel substrate of rose myrtle leaves extract and its fraction in acidic medium.
- Studying on mechanism and kinetics of corrosion inhibition process on CT3 steel substrate of the obtained extractions.
- Isolating and identifying the main ingredients that perform the corrosion inhibition ability of those extractions.
- Determining the optimal inhibitor concentration and proposing the plan to apply the extract for steel corrosion inhibition in industrial level.

4. New contributions of the thesis

1. Having successfully made rose myrtle extract and isolating 06 segments from D1 ÷D6 using Dianion column chromatography (adsorbent is Dianion HP-20) and enriching tannin content (adsorbent Sephadex LH-20).
2. Studied, compared, assessed the inhibition corrosion ability of rose myrtle extract, the extract segments, enriched tannins on CT3 steel as well as propose the adsorption model, corrosion inhibition mechanism and kinetic model of rose myrtle extract in 0.5M H_2SO_4 medium.
3. The result shown that rose myrtle extract is an inhibitor working on adsorption mechanism with impact mainly on cathode. The main component to the inhibiting corrosion impact of the plant extract is tannin present in DCS. Based on that, applying the rose myrtle extract on pickling process in practical samples, working as an environmentally friendly corrosion inhibitor in the industrial rust removal process, especially for some industrial acidic cleaning systems.

5. Thesis structure

The main content of the thesis consists of 131 pages divided into sections: Introduction: 2 pages; Chapter 1. Overview: 39 pages; Chapter 2. Experimental and research methods: 20 pages; Chapter 3. Results and discussion: 57 pages; Conclusion: 2 pages; New points of the thesis: 1 page; Published works related to the thesis: 1 page; References: 10 pages. The thesis includes 36 tables, 79 figures and 128 references.

CONTENT OF THE THESIS

CHAPTER 1. OVERVIEW

1.1. Metal corrosion in acidic medium

1.2. Corrosion inhibitor in acidic medium

1.3. Overview of rose myrtle

1.4. Necessity and research orientation

CHAPTER 2. EXPERIMENT AND RESEARCH METHOD

2.1. Research subjects

The research objects mentioned in the thesis include:

- Corrosion inhibitor from rose myrtle: extracts of rose myrtle (DCS), extraction segments (PDC) are isolated and tannins are enriched from extracts of rose myrtle.

- Tested steel sample is CT3 steel produced domestically.

2.2. Chemicals, tools and equipment

2.3. Experiment

2.3.1. Preparation of corrosion inhibitor

2.3.1.1. Rose myrtle extraction process

Boil the plant leaves with tap water, filter the residue from the extract, continue to boil the residue with water, 2nd extraction. Combining the two parts of the extract, dried to obtain DCS (from 1 kg of plant leaves obtain 1 liter of DCS). Cool and store at about 4^oC.

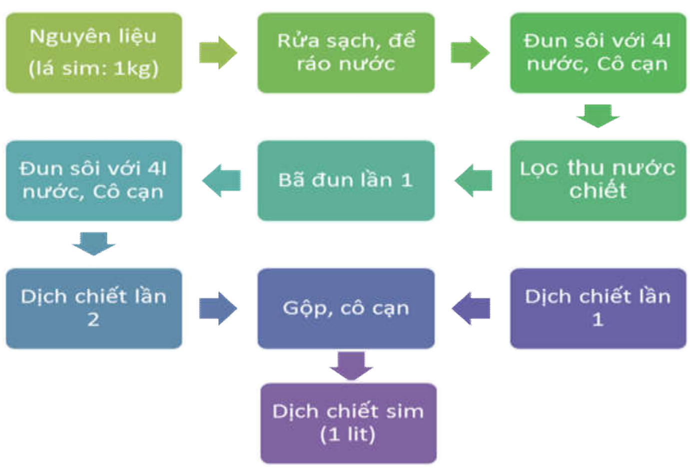


Figure 2.1. Processing of DCS

2.3.1.2. Isolation process of segments and enrichment process of tannin

The process of isolation and extraction of PDC and tannins from rose myrtle extract (DCS) is done by column chromatography methods:

- Extraction segments (D1 ÷ D6) is isolated by the Dianion HP-20 column chromatography method.

- Tannin (T) is enriched by Sephadex LH-20 chromatography.

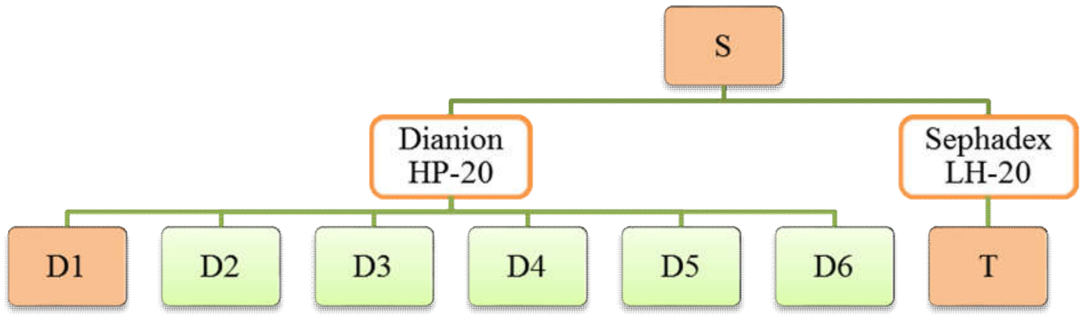


Figure 2.2. Tannin isolation and enrichment diagram

a) Segment isolation process from DCS

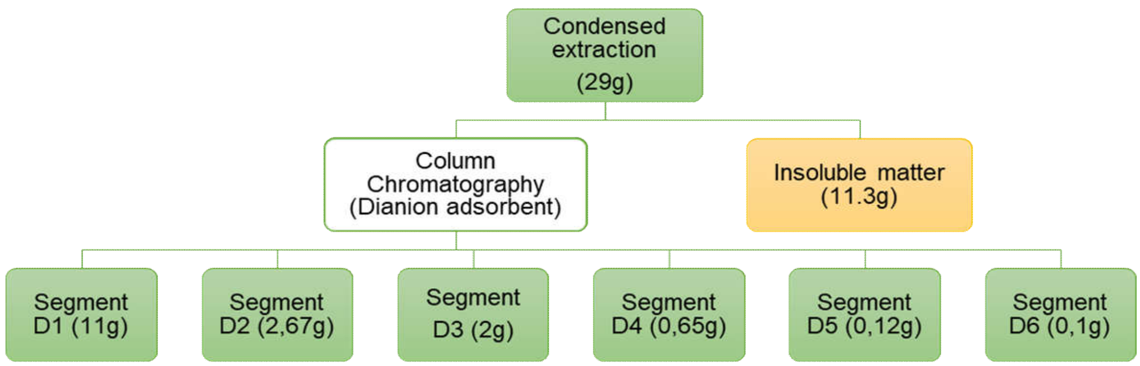


Figure 2.3. Segment isolation diagram

b) Tannin enrichment process from DCS

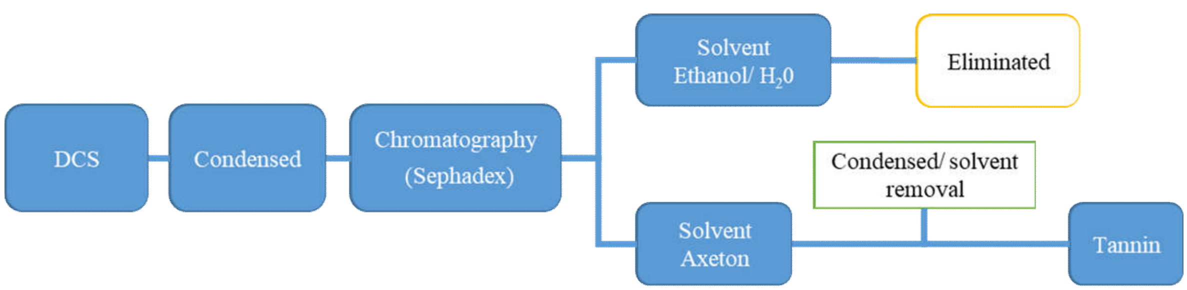


Figure 2.5. Tannin enrichment diagram

2.3.2. Evaluate the corrosion inhibition capability of rose myrtle extract

Measurements were implemented at room temperature of 25°C in 0.5 M H_2SO_4 media and 1 M HCl.

❖ *The experiment process includes the following steps:*

- Prepare samples of solutions in two 0.5 M H_2SO_4 media and 1 M HCl containing rose myrtle extract (DCS), fraction of rose myrtle extraction (PDC), and tannins with different concentrations as indicated in the table below :

Table 2.3. Sample study signboard in acidic environments

Sample	**Enviroment**	**Concentration of inhibitory use (% volume)**							
		0	**0,1**	**0,2**	**0,5**	**1**	**2**	**5**	**10**
DCS	H_2SO_4 0,5 M	A	S1A	S2A	S3A	S4A	S5A	S6A	S7A
PDC 1	H_2SO_4 0,5 M		D11A	D12A	D13A	D14A	D15A	D16A	D17A
PDC 2	H_2SO_4 0,5 M				D23A				
PDC 3	H_2SO_4 0,5 M				D33A				
PDC 4	H_2SO_4 0,5 M				D43A				
PDC 5	H_2SO_4 0,5 M				D53A				
PDC 6	H_2SO_4 0,5 M				D63A				
Tannin	H_2SO_4 0,5 M		T1A				T5A	T6A	T7A
DCS	HCl 1 M	H	S1H	S2H	S3H	S4H	S5H	S6H	S7H

2.3.2.1. Corrosion evaluation via weigh loss method

- Evaluation according to ISO 8407: 1991 standard

- Duration of testing: Total immersion time is 24h divided into monitoring points: after 2 hours, 4 hours, 8 hours, 12 hours and 24 hours.

- Preparation of steel samples: CT3 steel samples are 5 cm^2 in size. The work surface is ground with sandpaper, washed with distilled water, rinsed with acetone, dried and allowed to stabilize before use.

- After each time point, the sample is cleaned by washing, brushing, rinsing acetone, drying and drying in a desiccator, weighing the loss assessment.

2.3.2.2. *Corrosion evaluation via electrochemical methods*

Electrochemical methods for corrosion studies include: Potentiodynamic Polarization, Electrochemical impedance spectroscopy method.

❖ *Prepare a steel electrode in an electrochemical measurement*

The research steel sample is a circular CT3 steel, a 1cm^2 section is installed in a Teflon electrode mold with a fixed working electrode area in electrochemical measurements.

❖ *Testing mode*

- The polarization resistance measurement is carried out as follows: over-voltage E = E-E_{corr} is chosen as ± 25 mV, scanning speed is 0.1 mV/s, scanning is 1 cycle, i_{corr} corrosion current is calculated from the slope of the linear according to the graph.

2.4 Theoretical of research methods

CHAPTER 3. RESULTS AND DISCUSSION

3.1 Corrosion inhibition capability of rose myrtle extract in H_2SO_4 0,5M

The UCAM capability of the rose myrtle extract (DCS) for steel in the H_2SO_4 acid medium is assessed based on the corrosion rate of the steel substrate when present and not present at different concentrations.

3.1.1 E_{corr} potential of the extract samples

The potential is corrosive to E_{corr} when DCS is present in a positive direction than in an acidic environment without DCS. However, the displacement amplitude is not too large, within 25mV.

3.1.2 Potentiodynamic polarization

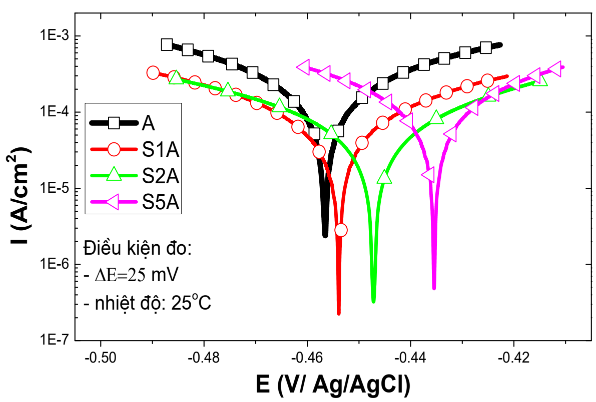


Figure 3.2. Polarization curve log|i|/E of 0,1%; 0,2%; 2% DCS (S1A, S2A, S5A) in H_2SO_4 0,5 M (A)

In 0.5M H_2SO_4 medium, corrosion current density tends to decrease when DCS content increases.

Table 3.1 Values of current density and polarization resistance of steel in H_2SO_4 0,5M by DCS concentration

Sample	**Corrosion potential E_{corr} (mV)**	**Current density J_{corr} (µA/cm²)**	**Efficiency H_{Jcorr}(%)**	**Polarization resistance R_p (Ohm)**	**Efficiency H_{Rp}(%)**
A	-456,57	739,682	0	43,80	0,00
S1A	-443,95	256,286	69,14	65,4	33,03

S2A	-445,302	228,23	71,57	92	52,39
S3A	-442,984	210,291	76,48	100	56,20
S5A	-441,073	173,588	76,22	108,6	59,67
S7A	-436,757	113,816	65,35	129	66,05

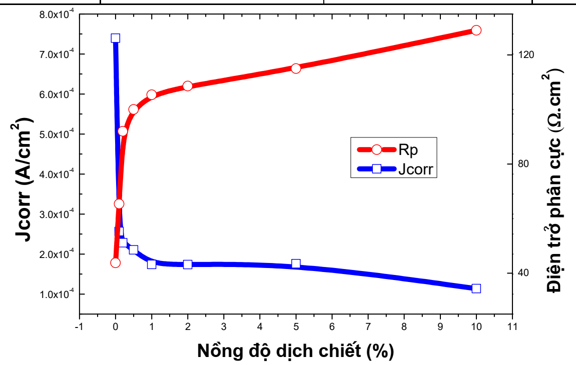


Figure 3.3. Current density and Polariazation resistance value diagram

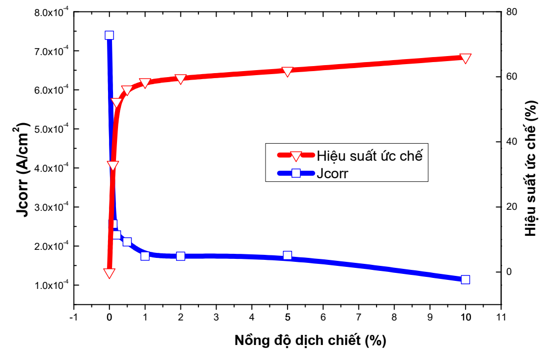


Figure 3.4. Current density J_{corr} and inhibition efficiency by DCS in H_2SO_4 0,5M

The inhibitory performance calculated according to polarization resistance is from 65-85% depending on the DCS content used. Calculated inhibitory performance shows that DCS is capable of acting as an inhibitor in 0.5M H_2SO_4 medium. At a concentration of 0.5% DCS, the inhibitory efficiency has reached the optimal level.

3.1.3 Electrochemical impedance spectroscopy method

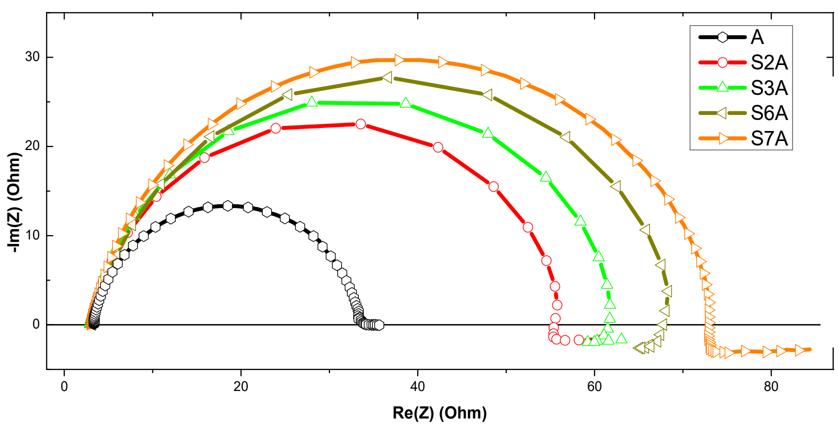


Figure 3.5. Nyquist spectroscopy plot in H_2SO_4 0,5 M (A), and in 0,2%; 0,5%; 5%; 10% DCS (S2A, S3A, S6A, S7A)

Table 3.2 Inhibition efficiency (H_{Rct}%) of steel by DCS concentration

Sample	Double layer capacitance C_{dl} (mF)	Charge-transfer resistance R_{ct} (Ωcm^2)	Frequency - Zi_{max} (Hz)	Inhibition efficiency H_{Rct}(%)
A	0,219	30,58	23,9061	0,00

S2A	0,116	52,79	21,4188	56,07
S3A	0,096	58,80	34,2654	58,00
S6A	0,069	64,617	34,2654	70,25
S7A	0,055	69,925	41,6945	70,09

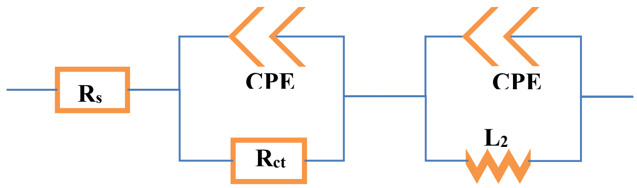


Figure 3.6. Equivalent circuit for a steel/ acid/ DCS system

Thus, in 0.5M H_2SO_4 environment, DCS shows the ability of UCAM steel due to reducing corrosion potential. At the 0.5% concentration range DCS exhibited effective inhibition, reaching 72% in 0.5M H_2SO_4 medium and the inhibitory performance did not increase significantly at larger DCS concentrations. Therefore, 0.5% DCS content was selected to carry out the evaluation, comparing the results obtained in subsequent studies.

3.1.4 Weight loss of steel in DCS/ H_2SO_4 0,5M solution

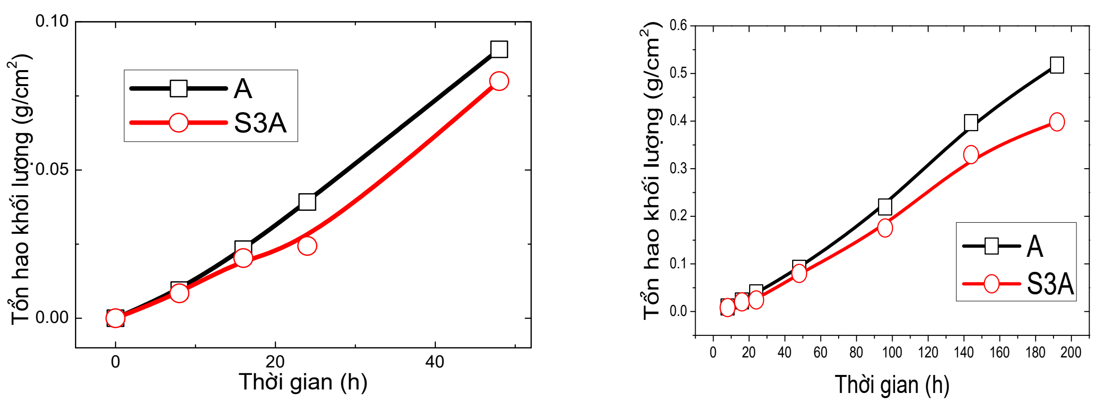


Figure 3.7. Variation of weight loss via time in H_2SO_4 0,5 M (A) and 0,5% DCS (S3A)

In the first 24 hours, the amount of loss between samples was not significantly different. However, after 24 hours of soaking, the weightloss on the samples was more pronounced and distinguishable. The results showed that the DCS concentration used is 0.5% capable of inhibiting corrosion of steel in 0.5M H_2SO_4 environment.

3.1.5 Analysing the corrosion level based on characterized surface morphology

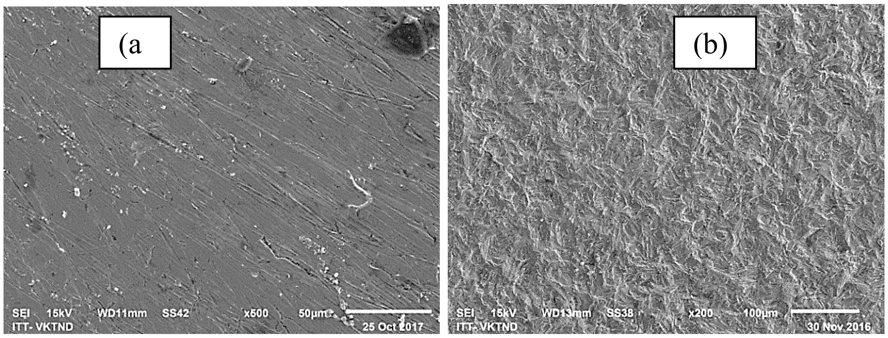


Figure 3.8. The steel surface before (a) and after (b) submersed in H_2SO_4 0,5 M

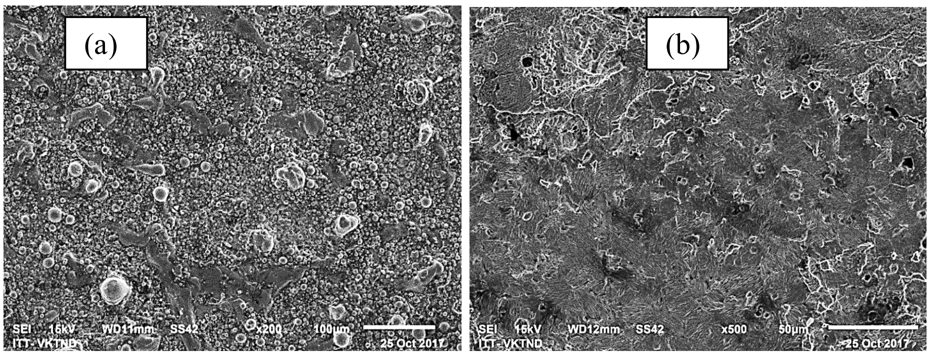


Figure 3.9. The steel surface before and after submersed in S1A (a), S3A (b)

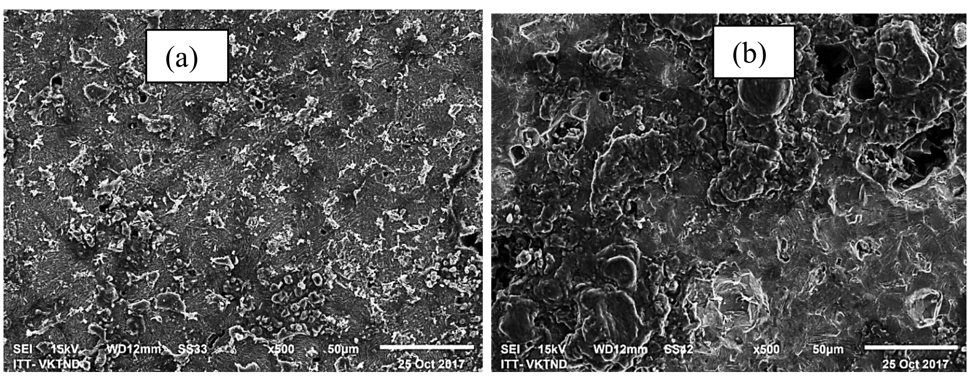


Figure 3.10. The steel surface before and after submersed in S4A (a), S5A (b)

Based on SEM images, with reference samples after acid immersion, very strong corrosion. For samples with additional DCS at different levels, the etching process causes corrosive surfaces to be of a lower degree.

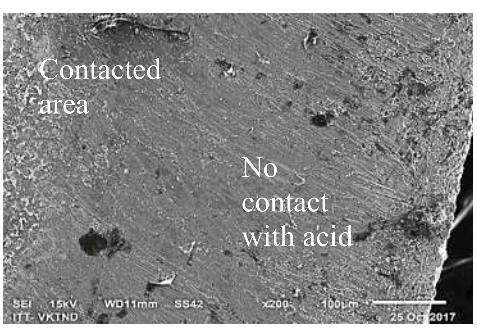


Figure 3.8. The steel surface in the boundary

The survey results and the above research shows that rose myrtle extract is capable of inhibiting corrosion of CT3 in 0.5M H_2SO_4 environment.

3.2 Isolation, evaluation the corrosion ability of main components in DCS

3.2.1 Isolation, evaluation the corrosion ability of segments

3.2.1.1 Isolation of segments

a. Phyto chemical screening of DCS and its segments

On the basis of the results obtained after conducting a preliminary survey, the classes of natural compounds in rose myrtle extract by thin-plate chromatography showed that the rose myrtle extract and segments D1 - D6 can contains multi-functional flavonoid-backbone compounds containing hydroxy -OH and carbonyl groups C=O. Rose myrtle extract and segments are indicated in blue in $FeCl_3$ reagent, pink with vanillin reagent and blue with CAM reagent.

b. IR spectrum of DCS and segments

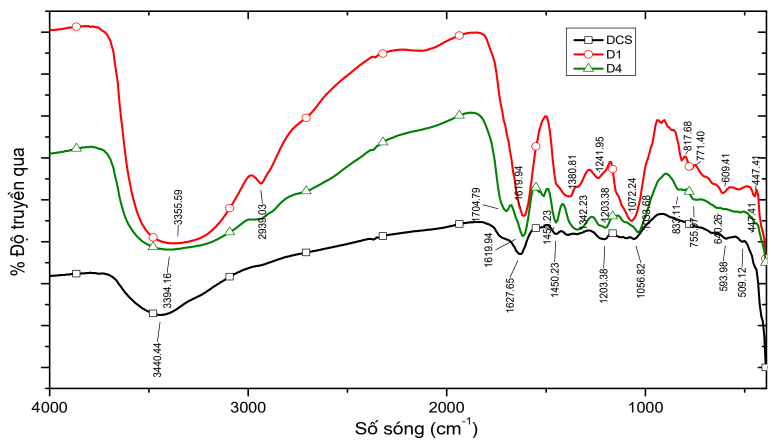


Figure 3.10. b. IR spectrum of DCS and segments D1 and D4

Infrared spectrum data shows that in D1 sample, the characteristic peaks of the functional group appear to be different and different from the samples S and D4. When comparing the homogeneity with the data in the infrared spectrum bank, the IR spectrum of D1 results close to the tannin compounds containing glycosit groups.

3.2.1.2 Evaluation the corrosion ability of segments

a. Weight loss of steel in segments/ H_2SO_4 0,5M solutions

Based on the graph, it can be seen that at the beginning of the weight loss mesurement, the difference in weight loss between the samples is not really clear.

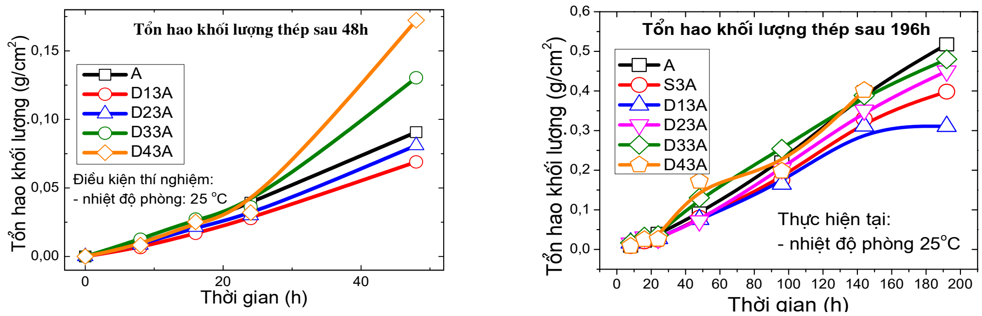


Figure 3.19. Weight loss of CT3 in H_2SO_4 0,5 M with the presence of segments 0,5%

However, after 24 hours of soaking, the weight loss on the samples was more markedly and clearly distinguishable.

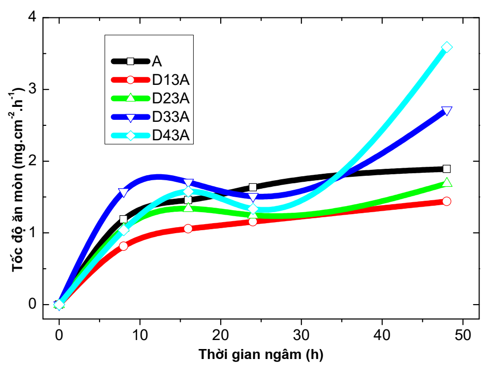


Figure 3.12. The variation of corrosion rate based on weight loss via time

Table 3.7. Inhibition efficiency based on weight loss (H_{in}) of D13A ÷ D43A

Immersion time (h)	Inhibition efficiency based on weight loss - H_{in} (%)			
	D13A	**D23A**	**D33A**	**D43A**
8	46,5	10.5	-	12.6
16	37,3	4.0	-	-
24	35,1	23.7	7.7	18.4
48	33,8	10.7	-	-

The results showed that, at a comparative concentration of 0.5%, the D1 sample showed better corrosion inhibition than the S05 sample and D2 từ D4 extraction segments. Therefore, D1 is the extraction segment selected for survey and evaluation by other electrochemical methods.

b. Equilibrium potential E_{corr} of D1 segment

In 0.5M H_2SO_4 acid medium, when present D1, E_{corr} potential is shifted to the positive. The changing tendency of corrosion potential depending on the change in concentration D1 may show a corrosion inhibitory effect in which D1 can act as a mix inhibitor acting on both anode and cathode processes.

c. Potentiodynamic polarization method

Polarized curve of steel in acid when present and not present D1 at different contents is shown in Figure 3.22 below:

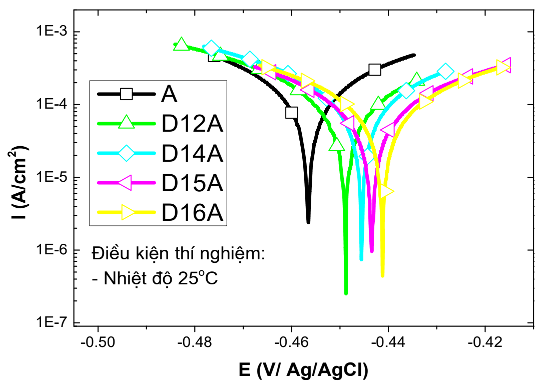


Figure 3.22. Polarization curve log|i|/E with different D1 concentration

Table 3.8 Values of current density and polarization resistance by D1

Sample	Corrosion potential E_{corr} (mV)	Current density J_{corr} ($\mu A/cm^2$)	Efficiency H_{Jcorr} (%)	Polarization resistance R_p (Ohm)
A	-456,57	739,682	0.00	43,80
D11A	-440,528	254,235	76	49,6
D12A	-451,087	217,296	66	56,8
D14A	-445,804	187,73	75	60,3
D15A	-444,35	184,912	67	75
D16A	-441,278	154,613	79	75,8

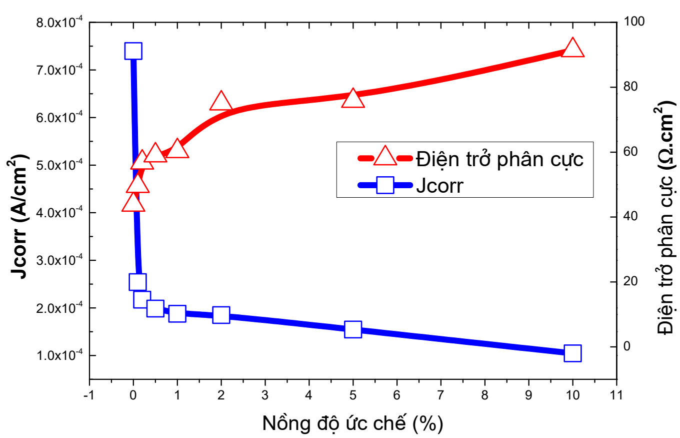


Figure 3.23. Correlation between current density and polarization resistance by D1 concentration

In 0.5M H_2SO_4 medium, corrosion current density decreases sharply when there is segmented D1 at low concentration of 0.1%. Corrosion current density tends to increase with inhibitory concentration of use.

Jcorr calculated inhibitory performance is from 66 to 86% depending on the DCS content used. The inhibitory performance is quite high, indicating that this is an effective inhibitor in 0.5M H_2SO_4 medium.

d. Electrochemical impedance spectroscopy of D1 segment

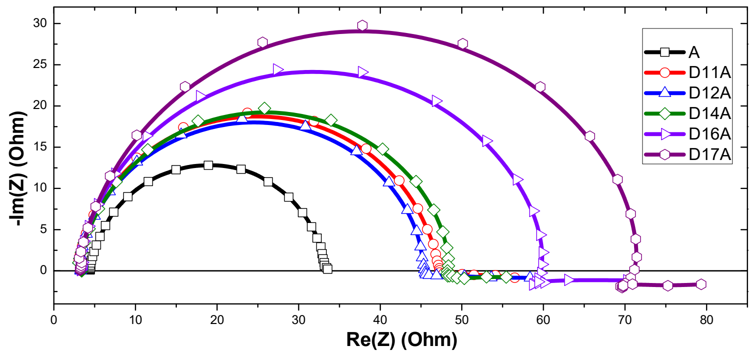


Figure 3.24. Nyquist plot of steel over D1 concentration

Table 3.9. Inhibition efficiency (H_{Rct}%) of steel based on D1 concentration

Sample	Double layer capacitance C_{dl} (mF)	Charge-transfer resistance R_{ct} (Ωcm^2)	Frequency - Zi_{max} (Hz)	Inhibition Efficiency H_{Rct} (%)
A	0,219	30,58	23,9061	0,00
D11A	0,115	43,05	31,6296	28,22
D12A	0,116	41,72	31,6296	25,93
D14A	0,091	45,55	31,6296	32,16
D15A	0,085	44,98	31,6296	31,30
D16A	0,069	57,17	50,7965	45,95

When using D1 content gradually increases, the magnitude of the charge transfer increases, the resistance converts the charge to increase with the amount of D1 used.

e. Surface morphology of steel with the presence of D1

The surface of the steel specimen with the segment D1 is corroded evenly, on the surface forming slits, shallow grooves in parallel, evenly distributed on the surface indicates that there has been an impact weakening the feeding process. worn surface. It can be said that corrosion inhibition has taken place, however, not evenly across the entire steel surface.

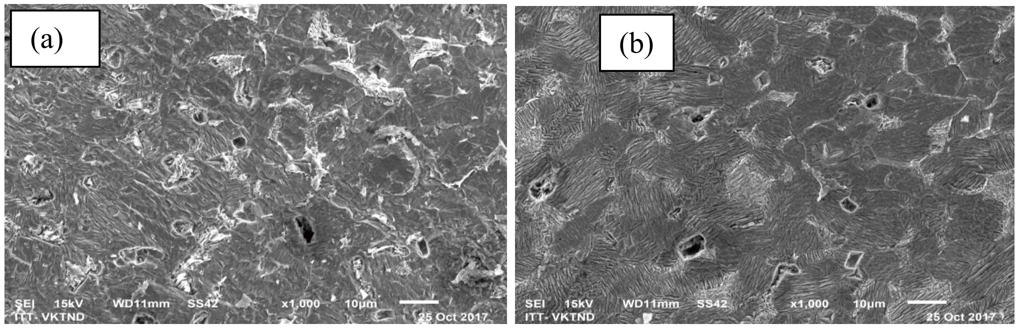


Figure 3.25. Steel surface after immersed on D11A (a) and D12A (b)

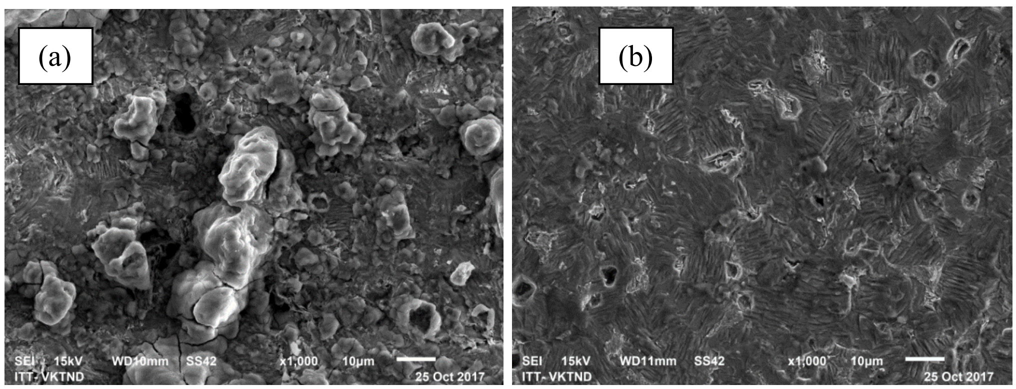


Figure 3.26. Steel surface after immersed on D13A (a) and D17A (b)

3.2.2 Enrichment and evaluation the inhibition ability of tannin

3.2.2.1 . Total polyphenol content (TPC) in DCS and segment

Total polyphenol content has been determined by Folin – Ciocalteu method. The results showed that rose myrtle extract has high TPC content than D1 segment.

Table 3.10. Total polyphenol content (TPC) value of samples

TNo	Samples	Humidity TCVN5613:2007 (%)	TPC (%)
1	Rose myrtle extract	33,14	19,15
2	D1 segment	32,84	20,99

3.2.2.2 Enrichment, qualitative and quantitative of tannin

a. *Qualitative of tanin*

From the qualitative test results, both tannin and condensed tannins were present in both samples.

b. *Quantitative method for tanin*

Tannin in rose myrtle extract was quantified based on the method of Kumazawa and colleagues using titration method with $KMnO_4$ and indigocarmin agent.

Table 3.12. Quantitative result for tannin

No	Sample	Hàm lượng (% khối lượng)
1	Rose myrtle extract - DCS	37,8
2	Enriched tannin	70,2

c. IR spectra of tannin isolated from rose myrtle extract

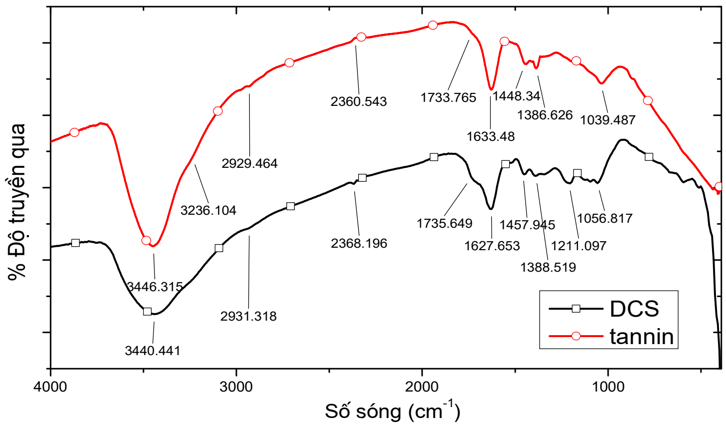


Figure 3.27. IR spectra of rose myrtle extract and tannin mixture

When compared with some published infrared spectroscopic data, the infrared spectra of the enriched tannins have a high degree of overlap with both the spectrum data of hydrolysis tannins and condensed tannins, which can be said to be made rich rose myrtle extraction is tannin.

3.2.2.3 Evaluation of inhibition ability of tannin

a. Equilibrium potential E_{corr}

The E_{corr} potential in the presence of tannin has slightly shifted into positive.

b. Potentiodynamic polarization method

Table 3.13 Electrochemical parameters and inhibition efficiency of tannin

Sample	Corrosion potential E_{corr} (mV)	Current density J_{corr} (μA/cm^2)	Efficiency H_{Jcorr}(%)	Polarization resistance R_p (Ohm)	Efficiency H_{Rp}(%)
A	-456,57	739,682	0,0	43,80	0,0
T1A	-445,448	141,614	80,9	155,1	71,8
T5A	-448,33	106,016	85,7	169,5	74,2
T6A	-447,595	97,173	86,9	180,9	75,8
T7A	-449,025	126,857	82,9	190,4	77,0

Inhibition efficiency based on current density value reachs over 80%, thus the mixture of enriched tannins has good corrosion inhibition ability in 0.5M H_2SO_4 medium.

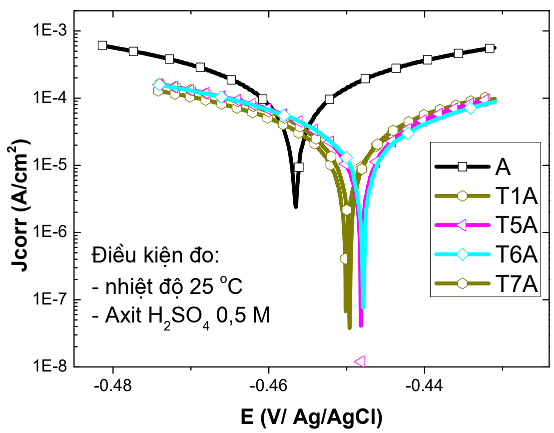


Figure 3.30. Polarization curve log|i|/E of steel by tannin concentration

c. Electrochemical impedance spectroscopy method

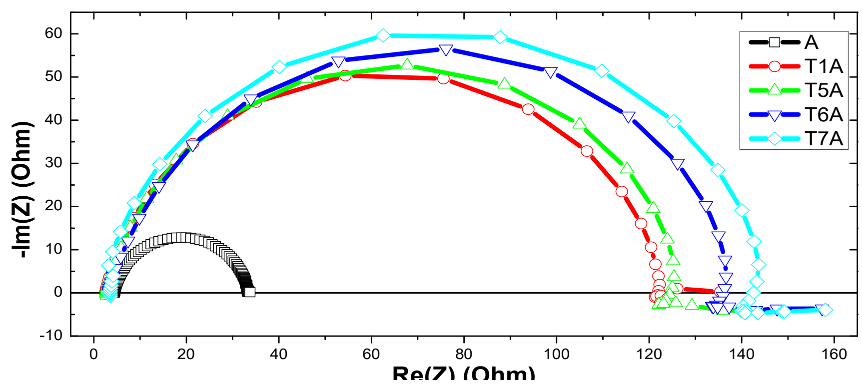


Figure 3.31. Nyquist plot over tannin concentration

The magnitude of the total arc becomes significantly increased when the tannin is present in the measuring solution, indicating an increase in the charge-transfer resistance. Charge-transfer resistance is inversely proportional to corrosion current, so it can be concluded that tannin reduces steel corrosion in 0.5M H_2SO_4 acid. The inhibition efficiency values based on the linear polarization resistance reach 74.5-78.3%, similar to other electrochemical measurement methods.

Table 3.14 Inhibition Efficiency (H_{Rct}%) by tannin concentration

Sample	Double layer capacitance C_{dl} (mF)	Charge-transfer resistance R_{ct} (Ωcm^2)	Frequency - Zi_{max} (Hz)	Inhibition efficiency H_{Rct}(%)
A	0,219	30,58	23,9061	0,00
T1A	0,1346	121,29	35,7201	74,52
T5A	0,1033	128,16	35,7201	75,89
T6A	0,08892	135,96	35,7201	77,27
T7A	0,09406	142,52	35,7201	78,32

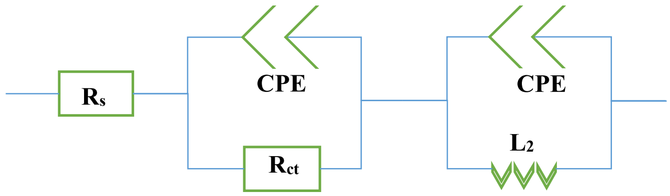


Figure 3.32. Equivalent circuit for a steel/ acid/ tannin

In the mixture of tannin, the tannin content is more than 70%. Inhibition efficiency of tannin-containing solutions reaches 86.9% based on corrosion current value, 78.3% based on charge-transfer resistance value. Thus, tannins play a major inhibition role in rose myrtle extract.

Table 3.15. Comparision between inhibition efficiency of DCS, D1 and tannin

Sample	Concentration	Inhibition Efficiency (%)		
		H_{Rct}	H_{Rp}	H_{Jcorr}
A		0,00	0,00	0,00
S5A	2%	36,20	59,67	76,53
D15A	2%	31,30	41,60	66,89
T5A	2%	75,89	74,16	85,67

3.3 Adsorption models and inhibition corrosion in H_2SO_4 0,5M

The Langmuir adsorption models is set up in the form of Parson Virial's equation 3 based on the relationship between log(C/θ) and logC. Applying Langmuir isotherm adsorption model in the case of tannin mixture which given the following results.

3.3.1 Adsorption isotherm based on J_{corr} in H_2SO_4 0,5 M

Table 3.17. Linear fitting result for the adsorption models

STT	A	B	R^2	SD	N	P
1	0,10162 ± 0,00353	1,02482 ± 0,00293	0,99996	0,00521	7	<0,0001
2	1,12643 ± 0,00618	1,02482 ± 0,00293	0,99998	0,00522	7	<0,0001

3.3.2 Adsorption isotherm based on R_{ct} in H_2SO_4 0,5M

Table 3.19. Linear fitting result

STT	A	B	R^2	SD	N	P
1	0.23778 ± 0.00771	0.94465± 0.0064	0.99989	0.01138	7	<0.0001
2	1.18243 ± 0.0135	0.94465±0.0064	0.99989	0.01138	7	<0.0001

3.3.3 Adsorption isotherm based on C_{dl} in H_2SO_4 0,5M

Table 3.20. Linear fitting result

STT	A	B	R^2	SD	N	P
1	0.14335 ± 0.024	0.83219 ± 0.01993	0.99857	0.03541	7	<0.0001
2	0.97555 ± 0.04204	0.83219 ± 0.01993	0.99857	0.03541	7	<0.0001

3.3.4 Suitability of adsorption isotherm models

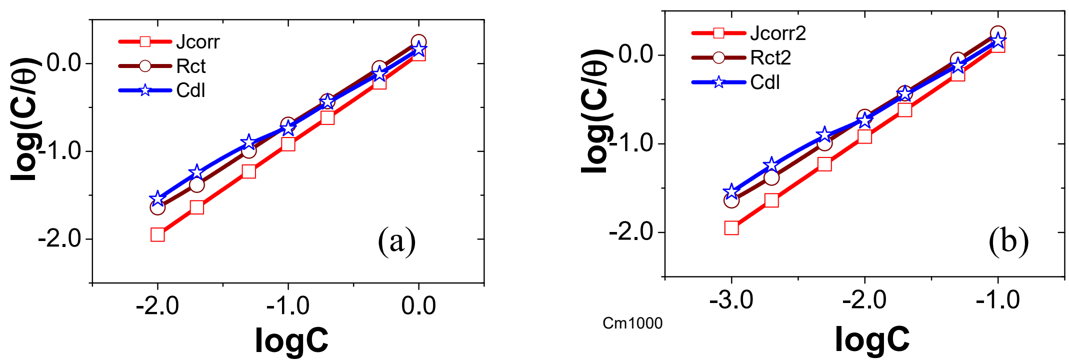


Figure 3.31 (a, b). Applying Langmuir adsorption isotherm models for rose myrtle based on J_{corr}, R_{ct} và C_{dl}

Table 3.21. Isothermal linear equation based on J_{corr}, R_{ct} và C_{dl}

Parameter		Calculated fitting equation	R^2	P
1	J_{corr}	$Log(C/\theta) = 0.10162+1.02482logC$	0.99999	<0.0001
	R_{ct}	$Log(C/\theta) = 0.238 + 0.945logC$	0.99989	<0.0001
	C_{dl}	$Log(C/\theta) = 0.143 + 0.832logC$		<0.0001
2	J_{cor}	$Log(C/\theta) = 1.12643 +1.02482logC$	0.99989	<0.0001
	R_{ct}	$Log(C/\theta) = 1.182 + 0.945logC$	0.99989	<0.0001
	C_{dl}	$Log(C/\theta) = 0.976 + 0.832logC$		<0.0001

Conclusion: According to the calculation of electrochemical parameters, the Langmuir adsorption model is the most suitable compared to other theoretical adsorption models. Thus, it can be said on the steel surface that a single layer of adsorbent is formed (in the case of using DCS as an inhibitor).

3.4 Inhibition corrosion mechanism of CT3 steel in rose myrtle extract

The adjustment of the polarization potential variation based on overpotential $\eta = \Delta E = E_{LP} - E_0$ (or E_{corr}) on the J/E plot by recalculating the efficiency on the two branches of the polarization curve at each point E_{Pi} in the entire polarization range from $-\Delta E$ to ΔE is implemented according to the formula:

$$H = 100*(1 - \frac{J_{Ci}}{J_{C0}})$$

Therei:

- J_{c0}: the polarization current density in a solution without inhibitors (C = 0),
- J_{Ci}: the polarization current density in a solution contained inhibitors with concentration Ci (Ci >0).

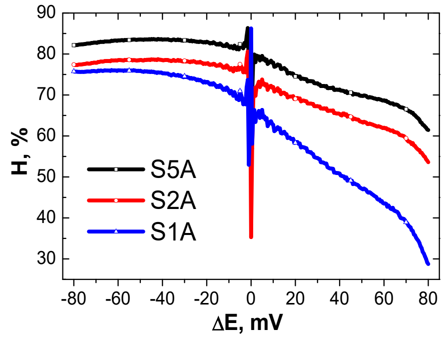


Figure 3.38. Variation of inhibition efficiency (%) as per polarization potential in both branch cathode and anod

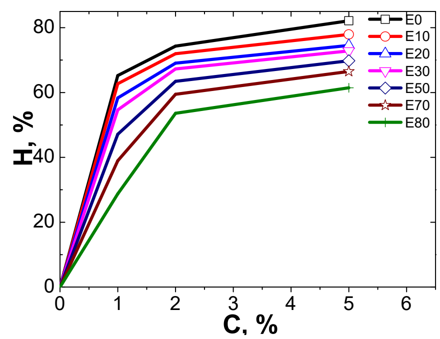


Figure 3.39. Variation of inhibition efficiency (%) over inhibitor concentration, with different polarization potential in anod (Ei, mV)

These results allow to draw conclusions:

- Inhibitors have a major impact in the cathode branch with the inhibition efficiency is stable and at high value.
- The inhibition ability to anode dissolution process is lower, and tends to decrease according to anode polarization.

3.5 Kinetic model for corrosion inhibition of DCS

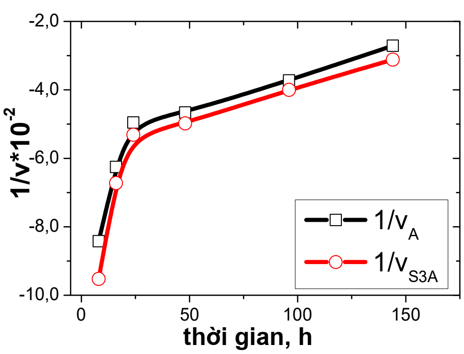


Figure 3.41. Variation of 1/v per testing time in H_2SO_4 0,5M with DCS 0,5%

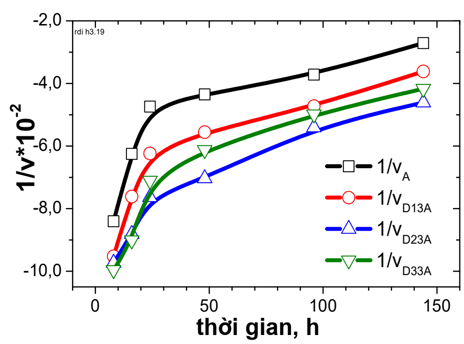


Figure 3.42. Variation of 1/v, per testing time in H_2SO_4 0,5M with different segments of 0,5%

Applied the one step reaction model with the correlation between weight loss over time as $\ln(w_f/w_0)$ per t for steel corrosion in 0.5 M H_2SO_4 acid with DCS inhibitor. It can be assumed that corrosion kinetics have the form $1/\upsilon t = kt+1/\upsilon_0$, with υ being the corrosion rate, t is the testing time. There are two defined kinetic regions: one region in the short study period of less than 25h with the variation of $1/\upsilon t$ is greater than the long study period, from 24 h to 150h.

3.6 Applying DCS in rust removal for practical steel sample

When conducting tests on actual samples, rusty steel samples are numbered and immersed in the similar period for 10 minutes in 0.5M H_2SO_4 acid with and without the presence of inhibitors.

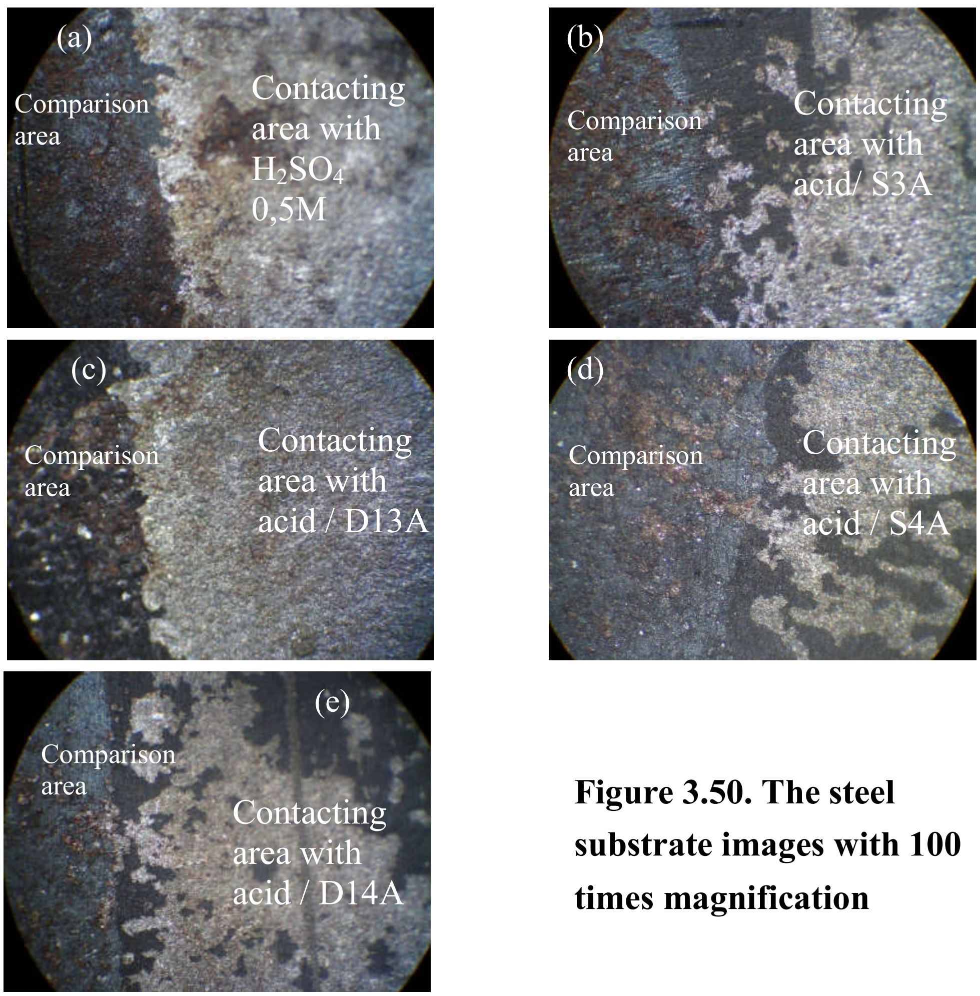
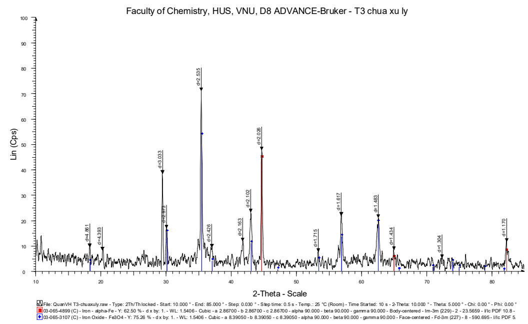


Figure 3.50. The steel substrate images with 100 times magnification

The interface between the contact area and the comparison area is obvious. The best rust removal effect is observed in the steel sample immersed in D13A solution. In other samples, although the rust layer is cleaned, there is a layer of corrosive residue appears on the surface (dark gray streaks).



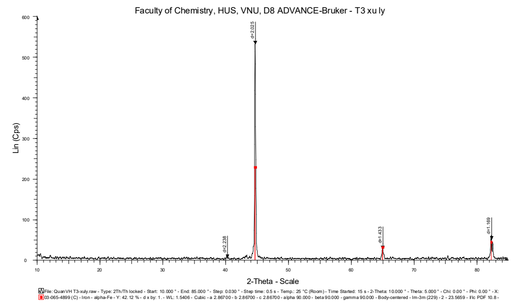


Figure 3.51 (a). XRD phase diagram of steel sample surface before pickling

Figure 3.51 (b). XRD phase diagram of steel sample surface after pickling

Conclusion: The inhibition of rose myrtle extract proved to be suitable for the rust removal process in acidic environments because it meets many major requirements such as: capable rust dissolves, protect the metal substrate, economical and environmental friendly.

3.7 DCS corrosion inhibition ability in other acidic environments

3.7.1 Equilibrium potential E_{corr} of DCS in HCl 1M

The corrosion potential shifted toward the positive because of an inhibited anode process or an enhanced cathode process. The tendency of the variation of corrosion potential depending on the change in DCS concentration may show a corrosion inhibitory effect in which DCS can act as a composite inhibitor acting on both anode and cathode processes.

3.7.2 Potentiodynamic polarization method

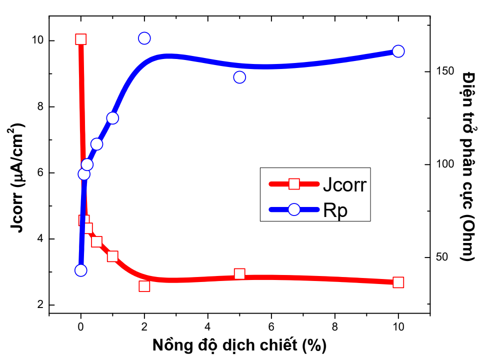


Figure 3.49. Variation of i_{corr} và R_P value over DCS concentration

According to the result, inhibition efficiency is increasing gradually when increasing the used DCS concentration and reach the maximum of 74.4% at 2% DCS concentration. Inhibition efficiency increased significantly for solutions with DCS present at low concentrations, then increased with higher concentrations and tended to stabilize in solutions with higher DCS content of 2%.

3.7.3 Electrochemical impedance spectroscopy method

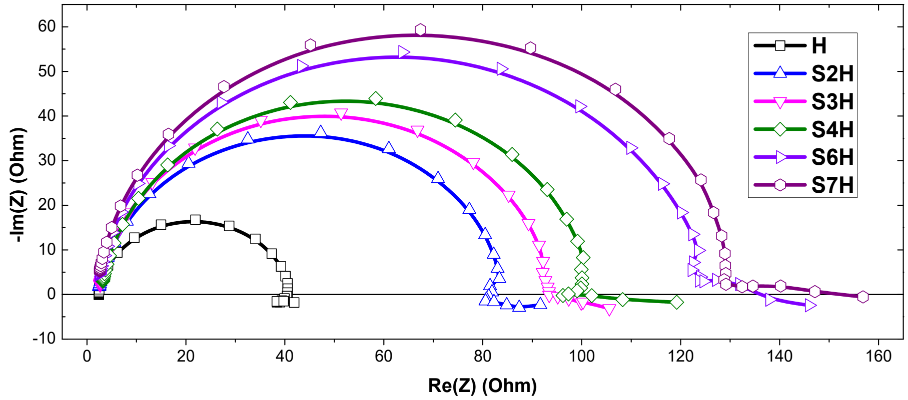


Figure 3.51. Nyquist plot over DCS concentration in HCl 1M

For steel / HCl 1M acid with presence of DCS, the equivalent circuit model is proposed as shown in Figure 3.57 below.

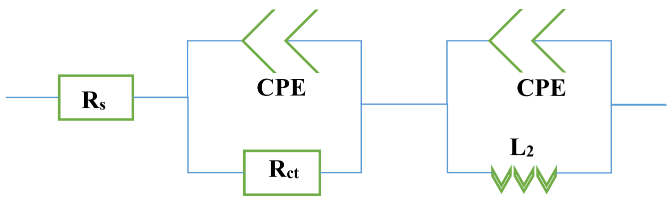


Figure 3.57. Proposed equivalent circuit for steel/ HCl 1M

When increasing the DCS concentration, the charge-transfer resistance value R_{ct} increases and the double layer capacitance C_{dl} value decreases accordingly. It could be noted that DCS has impacted the transfer of electrical charge and thereby caused a corrosion inhibitory effect on the steel substrate. The decrease in C_{dl} value indicates the formation of a layer of material on the metal surface that increases the thickness of the double charge layer. The devaluation of C_{dl} can also be caused by adsorption occurring on the surface, increasing the area of coverage on the metal surface and reducing the active centers.

3.7.4 Adsorption isotherm over R_{ct} value in HCl 1 M

3.7.4.1 Adsorption isotherm model over R_{ct}

STT	A	B	R^2	SD	N	P
1	0.10784 ± 0.00913	0.94856 ± 0.00758	0.99984	0.01346	7	<0.0001
2	1.05641 ± 0.01598	0.94856 ± 0.00758	0.99984	0.01346	7	<0.0001

1 - $\text{Log}(C/\theta) = 0.108 + 0.949\log C$

2 - $\text{Log}(C/\theta) = 1.056 + 0.949\log C$

3.7.4.2 Adsorption isotherm model over C_{dl}

STT	A	B	R^2	SD	N	P
1	0.12749 ± 0.01069	0.9201 ± 0.00887	0.99977	0.01577	7	<0.0001
2	1.0476 ± 0.01872	0.9201 ± 0.00887	0.99977	0.01577	7	<0.0001

1 - $Log(C/\theta) = 0.1275 + 0.9201 logC$

2 - $Log(C/\theta) = 1.0476 + 0.9201 logC$

3.7.4.3 Comparision between the adsorption isotherm model over R_{ct} và C_{dl}

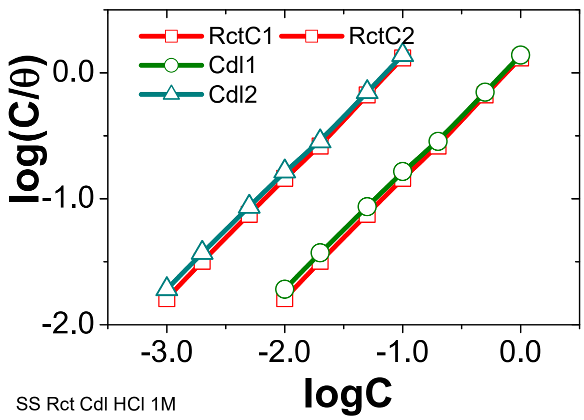


Figure 3.61. Langmuir adsorption isotherm model, HCl 1M, over R_{ct} and C_{dl}

Table 3.29. Linear fitting ressults

STT	Parameter	A	B
1	R_{ct}	0.108	0.949
	C_{dl}	0.128	0.920
2	R_{ct}	1.056	0.949
	C_{dl}	1.048	0.920

In conclusion, the rose myrtle extract shows a capable inhibition effect in 1M HCl acid medium. The values for inhibition efficiency of DCS in 1M HCl acid are similar to 0.5 M H_2SO_4 acid. In common, the rust remover solution is a mixture of the above-mentioned acids with different inhibitors.

CONCLUSION

The obtained result of this thesis is as followed:

1. Successfully producing rose myrtle extract and isolating 06 D1 ÷ D6 extraction segments by column chromatography (Dianion HP-20 adsorbent) and enriching tannin (adsorbent Sephadex LH-20). Using thin layer chromatography, infrared

spectroscopy, Folin-Ciocalteu method, A.Hagerman's method of tannin quantitative and qualitative, the results showed that the fractions and the enriched tannin mixture contained –OH, C=O groups, C=C, C–O–C, C–H bonds and aromatic rings.

2. Studied, compared, assessed the inhibition corrosion ability of rose myrtle extract, the extract segments, enriched tannins on CT3 steel in H_2SO_4 0,5 M medium. The obtained results showed that the plant extract has inhibitive ability, working on adsorption mechanism with impact mainly on cathode. The main component to the inhibiting corrosion ability is tannin.
3. Rose myrtle extract causes corrosion potential shift to the more positive area and reduces the corrosion current even at low concentrations in contact with the CT3 steel on H2SO4 0,5 M. The inhibition efficiency varies depending on the concentration and environment, reaching from 66-86% in electrochemical measurements and about 45% in weight loss method. The D1 segment showed better inhibition ability than other fractions in 0.5 M H2SO4 medium.
4. The adsorption model, corrosion inhibition mechanism and kinetics were appropriately proposed for the rose myrtle extract in H2SO4 0,5 M medium. The results showed that the plant extract inhibition property follow Langmuir adsorption model. Corrosion kinetics are in the form of 1/vt = kt+1/v0, there are two defined kinematic regions: a short period region with less than 25 hours, the variation of 1/v according to t with slope is larger and a long period region from 24 h to 150 h.
5. The inhibition corrosion ability of rose myrtle extract in 1 M HCl acid has been investigated, inhibition efficiency reach up to 80%, inhibition mechanism followed Langmuir adsorption model.
6. A preliminary solution has been proposed to apply DCS in the rust removal process for steel, the best result is in D13A solution, the steel surface after treatment are more uniform, smooth and cleaner than in other solution.