UNIVERSITI PUTRA MALAYSIA

COCONUT PULP WASTE EXTRACT (Cocos nucifera L.) AS CORROSION INHIBITOR FOR MILD STEEL IN 1.0 M ACID CORROSION

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FK 2017 58

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

April 2017

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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April 2017

Chairman : Shafreeza Binti Sobri, PhD
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Corrosion control method, especially the use of inhibitors has gained monumental importance nowadays. The demand for green inhibitor to replace toxic one has increased tremendously. The use of local wastes which are organic in nature for the production of green corrosion inhibitor is no doubt the trend of the day. To arrive at an inexpensive, non-toxic and eco-friendly inhibitor formulation, the present study on the use of coconut pulp waste extract of *Cocos nucifera L*. has been carried out. Coconut pulp waste were chosen to be extracted and investigated in terms of their corrosion inhibition efficiencies for mild steel in 1.0 M HCl using weight loss analysis at different inhibitor concentrations and immersion time, tafel polarization curve and impedance curve at different inhibitor concentrations. The interaction between inhibitor and specimen surface was study using Langmuir, Freundlich and Temkin adsorption isotherms. Weight loss measurement were conducted at different of immersion time which is at 6, 12, 18, 24 and 48 hours. Result from weight loss study showed that as the concentration of the inhibitor increases, the rate of corrosion decreases. It also showed that as the concentration of the inhibitor increases, the inhibition efficiency also increases up to 94.52 %. Further examination study using scanning electron microscopy (SEM) showed the deposition of coconut pulp extract on the metal surface. In potentiodynamic polarization method and electrochemical impedance spectroscopy the electrochemical measurement were carried out using Autolab Potentiostat/Galvonastat. The experiments were conducted using 250 ml of electrolyte (1 M HCl) without and with different concentrations of the extracts of 2, 4, 6, 8, and 10 % v/v. The obtained inhibition efficiencies of extracts from polarization measurements was 89.38 % and 83.47 % from the impedance measurement. The impedance measurement revealed that when the concentration of the coconut pulp increased the charge transfer resistance increased and the double layer capacitance decreased. In polarization measurement the value from corrosion current E_{corr} suggested that coconut pulp extract is a mixed type nature of the inhibitor molecules. The adsorption interaction between the corrosion inhibitor with mild steel surface obey the Langmuir adsorption isotherm as the value of the correlation coefficient (R^2) is very near to unity. From the value of free energy of adsorption ($\Delta G°_{ads}$) showed a spontaneous adsorption and physisorption process is occurred.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

EKSTRAK HAMPAS KELAPA (*Cocos nucifera* L.) SEBAGAI PERENCAT KAKISAN KEPADA KELULI LEMBUT DALAM 1.0 M LARUTAN ASID

Oleh

NORIN HAFIZAH BINTI RAHIM

April 2017

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Kaedah kawalan hakisan, terutamanya penggunaan perencat telah meraih perhatian penting pada masa kini untuk menggantikan perencat yang bertoksik kepada mesra alam. Penggunaan bahan buangan yang organik secara semulajadi untuk menghasilkan perencat mesra alam adalah tidak lagi meragukan. Untuk mencapai satu formulasi perencat yang murah, tidak mengandungi toksik dan mesra alam, kajian menggunakan ekstrak hampas kelapa telah di jalankan. Kelapa sisa pulpa telah dipilih untuk diekstrak dan disiasat dari segi kecekapan perencatan kakisan untuk keluli lembut dalam 1.0 M HCl menggunakan kaedah penurunan berat pada masa rendaman dan berbeza kepekatan perencat, polarisasi tafel dan impedans pada berbeza kepekatan perencat. Interaksi antara permukaan keluli lembut dan perencat juga di siasat menggunakan Langmuir, Freundlich dan Temkin penjerapan isoterma. Pengukuran berat badan telah dijalankan di masa yang berbeza rendaman iaitu pada 6, 12, 18, 24 dan 48 jam. Dalam kaedah penurunan berat, spesimen direndam dalam 100 ml elektrolit (1.0 M HCl) tanpa dan dengan kepekatan yang berbeza daripada ekstrak 2, 4, 6, 8, dan 10 v/v%. Keputusan daripada kajian penurunan berat menunjukkan bahawa bertambah kepekatan perencat, kadar kakisan berkurangan. Ia juga menunjukkan bahawa bertambah kepekatan perencat, kecekapan perencatan juga meningkat sehingga 94.52%. Kajian pemeriksaan lanjut dengan menggunakan mikroskop elektron pengimbas (SEM) menunjukkan bahawa pemendapan ekstrak pulpa kelapa pada permukaan logam. Autolab Potentiostat / Galvonastat di gunakan dalam kaedah polarisasi potentiodynamic dan elektrokimia impedans spektroskopi. Kajian ini telah dijalankan dengan menggunakan 250 ml elektrolit (1 M HCl) tanpa dan dengan kepekatan yang berbeza daripada ekstrak 2, 4, 6, 8, dan 10 v/v%. Kecekapan perencatan diperolehi daripada ekstrak dari ukuran polarisasi adalah 89.38% dan 83.47% dari pengukuran galangan. Pengukuran impedance menunjukan kepekatan pulpa kelapa meningkat rintangan pemindahan caj meningkat dan lapisan kemuatan double menurun. Dalam pengukuran polarisasi nilai daripada E_{corr} semasa kakisan mencadangkan bahawa ekstrak pulpa kelapa adalah sejenis sifat campuran molekul inhibitor. Interaksi penjerapan antara perencat dan permukaan keluli lembut adalah mematuhi penjerapan Langmuir di mana nilai pekali korelasi adalah sangat hampir dengan satu. Daripada nilai tenaga bebas penjerapan ($\Delta G°_{ads}$) menunjukkan penjerapan dan physisorption proses spontan yang berlaku.

ACKNOWLEDGEMENTS

Firstly, I would like to express my sincere gratitude to my supervisor Dr Shafreeza binti Sobri for the supervision of my master for her patience, motivation, and immense knowledge. Her guidance helped me in all the time of research and writing of this thesis. Furthermore, I would like to thank the rest co-supervisors, Assoc. Prof. Dr Zurina Binti Zainal Abidin and Assoc. Prof. Dr Janet Lim Hong Ngee for their generous support, encouragement and opinion.

I would like also to thank to University Putra Malaysia (UPM) for the Fundamental Research Grant Scheme (FRGS), The Ministry of Higher Education and Universiti Teknologi Mara (UiTM) for a scholarship for the financial support for my graduates study.

Finally, I would like to thank my family, to my parents, siblings and my spouse for providing me with unfailing support and continuous encouragement throughout my years of study and through the process of researching and writing this thesis. This accomplishment would not have been possible without them.

Thank you.

I certify that a Thesis Examination Committee has met on April 27, 2017 to conduct the final examination of Norin Hafizah Binti Rahim on her thesis entitled "Coconut Pulp Waste Extract (*Cocos nucifera L.*) As Corrosion Inhibitor For Mild Steel In 1.0 M Acid Corrosion" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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TABLE OF CONTENTS

LIST OF ABBREVIATIONS

AC	Alternating-current
C_{inh}	Corrosion Inhibitor
CPE	Constant phase element
CR	Corrosion rate
E_a	Activation Energy
IE	Inhibition efficiency
IE %	Percentage of inhibition efficiency
OCP	Open circuit potential
Pt	Platinum electrode
R	Gas constant
v/v %	Volume per volume percent

LIST OF SYMBOLS

°C	Degree celsius
A	Ampere
Ag/AgCl	Silver/Silver chloride
β_a	Anodic tafel slope
βc	Cathodic tafel slope
C	Concentration
C_{dl}	Double layer capacitance
cm	Centimeter
cm^2	Centimeter square
E_{corr}	Corrosion potential
f_{max}	The frequency at the apex of the Nyquist plot
g	Gram
g cm^{-2} h^{-1}	Gram per square centimeter hour
g cm^{-3}	Gram per cubic centimeter
HCl	Hydrochloric acid
I^0corr	Corrosion current density value without extract
I^icorr	Corrosion current density value with extract
K^{-1}	Kelvin^{-1}
kJ/mol	Kilojoules per mole

m^2	Meter square
mm/year	Millimeter per year
mV	Millivolt
mV dec^{-1}	Millivolt per decade
mV/s	Millivolt per second
R	Gas constant
R'ct	Charge transfer resistance in the absence of extract
R^0ct	Charge transfer resistance in the presence of extract
R^2	Correlation coefficient
R_{ct}	Resistance of charge transfer
R_s	Resistance of solution
V	Volt
W_0	The weight loss values in the absence of inhibitor
Wi	The weight loss values in the presence of inhibitor
ΔG°ads	Free energy of adsorption
θ	Degree of surface coverage
μA	Microampere
μA cm^{-2}	Microampere per square centimeter
μF/cm^2	Micro farad centimeter square
Ω cm^2	Ohm centimeter square

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background of study

In recent years, mild steel is widely used in the engineering material of construction industry because of its excellent in mechanical properties, easy fabrication and also very economical compared to other metallic materials. Often, mild steel is used in the fabrication of reaction vessel and storage tanks (Kamal & Sethuraman, 2012). Meanwhile hydrochloric acid (HCl) acid is broadly utilized in several industries as pickling of steel, production of inorganic and organic compound, oil well acidification, descaling and cleaning agents to remove the unwanted oxide films and corrosion products (Afia et al., 2012; Chen et al., 2013). Compared to other mineral acids, HCl is generally used because of its benefits which are low cost, productive and straightforward (Gopiraman et al., 2012).

In the presence of acid, mild steel will corrode easily especially in petrochemical industries which is in cleaning process, the reaction between the hydrochloric acid and the mild steel will form a corrosion (Anupama et al., 2015). Another example is the removal of harmful rust in some industrial processes which is carried out by mainly acid pickling using hydrochloric acid solution. Thus, corrosion caused by mild steel in the presence of acidic solution need to be solved and overcome as it is the main concern nowadays (Li et al., 2009).

The effects of the corrosion on the environment is important and need to be addressed because the corrosion problem have become worldwide. Corrosion can cause adverse impact on the industry and effect economic losses due to cost needed for replacement of affected equipment and structure for repairing purpose (Al-amiery et al., 2015). These effects of corrosion affect the economy as well as can give some negative implications to people working in the industry and the area nearby. The example of an industry that is affected by the corrosion is the petroleum industry and it also is estimated that the corrosion costs 2.5 billion dollar/annum loss (Afia et al., 2014).

To overcome the effects of corrosion, many methods have been used to protect the metals, but among the method the use of inhibitor is very famous because of it is easy to apply (Raja & Sethuraman, 2008). Inhibitors are substances that decelerate the rate of corrosion (Rajalakshmi & Safina, 2012). Good inhibitors contain heterocyclic compounds containing O, S and N as heteroatoms (Kamal & Sethuraman, 2012). The utilization of inhibitors in slowing down the rate of corrosion has turned out to be proficient and generally practicable. Nonetheless, most of the compounds that build this inhibitor are toxic to the environments and human (Bhawsar, 2015). Due to the toxicity of these inhibitor, researchers have discovered other alternative to replace the toxic inhibitor. This is called green inhibitor which are non- toxic and not harmful to the environment (Sangeetha et al., 2011).In addition, due to the high operating cost of

synthetic inhibitor and its environmental effects, the research findings show the synthetic corrosion inhibitor cannot be utilized (Kamal & Sethuraman, 2012). As result of that, there are research being carried out recently to find the potential use of green corrosion inhibitor (Ji et al., 2015). The utilization of natural product to protect metal against corrosion has become popular and there are many reports on the effectiveness of the natural product in the inhibition of the metal in various acid media. The source of the natural product comes from fruits, plants and their peels which are a very good source for active organic compound (Muthukrishnan et al., 2015). Therefore, the utilization of natural product is not only low cost but also is environment friendly (Kamal & Sethuraman, 2012).

The research on the natural products as green inhibitor for corrosion inhibitor is an important field to study due to eco-friendly and the source of the natural products are cheap, readily available, and ecologically acceptable (Abdel-Gaber et al., 2006). Recent literature study shows that green inhibitor are found in natural product and proved to be effective against corrosion. These product among them are spirulina plantesis (Kamal & Sethuraman, 2012), litchi (Ramananda Singh et al., 2015), watermelon rind (Odewunmi et al., 2014), mangrove tannin (Shah et al., 2013), turmeric and ginger (Aziz & Sirat, 2015), apricot juice (Yaro et al., 2013). As a result, researchers have come up with a good number of naturally occurring substances which can be used as inhibitors for metals corrosion in different aggressive mediums.

In the present study, the selected natural product which is a waste from coconut pulp of *Cocos nucifera L* is examined for its potential to inhibit corrosion of mild steel in acid media. Furthermore there are no recent study about this natural product on the reported literature. *Cocos nucifera L* is an unbranched monoecious plant of the Palmae family. Coconut is one of the key plantation crops of the tropics which is a member of the monocotyledonous family Aracaceae (Yong et al., 2009).

1.2 Problem statement

In recent years, industrial sectors are concerned with the corrosion of metal and alloys and they also received huge attention from researcher to overcome these problems (Al-amiery et al., 2015). To decelerate the rate of corrosion or to stop the corrosion to occur, it has become an interesting and comprehensive field to be explored and studied. One of the common methods to deal with this problems is using the chemical inhibitor. The chemical inhibitor derived from the compound of synthetic origins has their own limitation which is not environmental friendly (Afia et al., 2014). Furthermore, due to the increasing of ecological awareness and the rigid environmental regulation, researchers begin to find other alternative methods to substitute the inhibitor from the hazardous to the eco-friendly inhibitor (Kamal & Sethuraman, 2012). Natural products are cheap and environmental safe. Hence, the benefits of using natural product extracts as corrosion inhibitors is welcome as they are environmental friendly. The extracts of the plant products are heteroatoms groups, molecular and electronic structures which show close similarity to those of conventional organic inhibitor molecules that serve as corrosion inhibitor (Özcan et al., 2008).

1.3 Significance of the research

As mentioned above, industrial sectors are affected due to corrosion. Therefore, the use of natural inhibitor rather than synthetic inhibitor is the best solution to overcome or minimizing the corrosion. In this research, the waste from coconut pulp was used as corrosion inhibitor for mild steel in acid solution. The coconut pulp extract which is from waste is very valuable and the cost of materials is very low. It is shows environmentally safe besides materials are eco-friendly (Yaro et al., 2013). Here trash is being used to create wealth. Therefore, the findings of this research will give beneficial for all industries that use mild steel under corrosive media. As a consequence, this research brings benefits which is environmentally safe and it is applicable to all industries to decelerate the corrosion.

1.4 Objectives

The objectives of the research are:

1 To evaluate the inhibition efficiency of coconut pulp waste extract as corrosion inhibitor in 1.0 M HCl using weight loss analysis (at different inhibitor concentrations and immersion time), tafel polarization curve and impedance curve (at different inhibitor concentrations).
2 To study the interaction between inhibitor and specimen surface using Langmuir, Freundlich and Temkin adsorption isotherms.

1.5 Scope of study

The scope of this study focuses on the inhibitive properties of waste product of coconut pulp (*Cocos nucifera L.*) in acid media on mild steel in 1.0 M HCl. In this research, there are divided and focused into 5 parts. Firstly is the preparation and characterization of active compound of inhibitor in coconut pulp waste extract. The coconut pulp waste extract was prepared from the waste material of coconut pulp to a powder form before reflux process. The functional group contained in the coconut pulp waste was identified using FTIR analysis. Secondly is the weight loss study to know the inhibition efficiency of the mild steel in 1.0M HCl at different concentration of inhibitor and time of immersion. Different concentration of 2, 4, 6, 8, and 10 % v/v of coconut pulp extract were used at different time immersion at 6, 12, 18, 24, and 48 hours. Results from the weight loss analysis were used to study the interaction between inhibitor and mild steel surface. There are three types of different isotherms were studied namely langmuir, freundlich and temkin isotherms. Electrochemical measurement which is tafel polarization curve and impedance curve were recorded using a three-electrode cell by Autolab potentiostat. Lastly it is to study and compare the morphology of the surface with and without the presence of inhibitor using SEM and EDX analysis.

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