Thermodynamic and Kinetic Study of Zinc bis-(Dipalmithyl Dithiophosphate) Activity as Anti-Corrosion Additive-Fatty Acid Based Through Potentiodynamic Polarization Technique

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ABSTRACT

Zinc bis-(dipalmithyl dithiophosphate) ($ZDTP_{16}$) is one product variant of zinc dialkyl dithiophosphate (ZDTP)-fatty acid based having function as corrosion inhibitor. By using 3% of effective dose for the application, its effectiveness of $ZDTP_{16}$ corrosion inhibition will achieve 97% and it will be able to decrease Cu metal corrosion rate from 0.152 to 0.004 mm per year. Thermodynamic and kinetic parameter verification indicates the decreasing of spontaneity and corrosion rate by existence of $ZDTP_{16}$ inhibitor. Gibbs free energy transition corrosion of Cu metal in electrolyte medium is measured in corrosion simulator increased from +85.22 to +91.77 kJ mol^{-1}, while its activation energy increased from +16.66 to +33.68 kJ mol^{-1}. Morphology observation of Cu metal substrate surface using SEM-EDX shows that the adsorption of $ZDTP_{16}$ at substrate surface is able to protect surface from corrosion indicated by the existence of Zn, P, S, and C constituents representing composer atoms of $ZDTP_{16}$, and the decreasing of Cl^- corrosive constituent at substrate surface.

Keywords: *$ZDTP_{16}$; adsorption; corrosion inhibitor; fatty acid*

ABSTRAK

Zink bis(dipalmitylditiofosfat)($ZDTP_{16}$) adalah salah satu varian produk dari zink dialkilditiofosfat (ZDTP) berbahan dasar asam lemak yang dapat berfungsi sebagai inhibitor korosi. Pada dosis efektif aplikasi 3%, efektivitas inhibisi korosi $ZDTP_{16}$ mencapai 97% dan mampu menurunkan laju korosi logam Cu dari 0,152 ke 0,004 mm pertahun. Verifikasi parameter termodinamika dan kinetika menunjukkan spontanitas dan laju korosi menurun dengan kehadiran inhibitor $ZDTP_{16}$. Energi bebas Gibbs transisi korosi logam Cu dalam medium elektrolit yang diukur pada simulator korosi meningkat dari +85,22 ke +91,77 kJ mol^{-1}, sedangkan energi aktivasinya meningkat dari +16,66 ke +33,68 kJ mol^{-1}. Pengamatan morfologi permukaan substrat logam Cu menggunakan SEM-EDX menunjukkan teradsorpsinya $ZDTP_{16}$ pada permukaan substrat mampu melindungi permukaan dari korosi yang ditunjukkan dari keberadaan konstituen Zn, P, S, dan C yang merupakan atom-atom penyusun $ZDTP_{16}$, dan menurunnya konstituen korosif Cl^- pada permukaan substrat.

Kata Kunci: *$ZDTP_{16}$; adsorpsi; inhibitor korosi; asam lemak*

INTRODUCTION

Corrosion is surface degradation process of a material as result of electrochemical reaction. Corrosion always become focus center and subject study because there are numerous damages resulted by such chemical process. Supposedly, industries and governments of USA, Australia and Japan had expended funds achieving 31% their gross domestic brutto respectively, in order to solve such problem [1]. Some infrastructures have oxidation process from contacting with water, air, and thermal accelerated corrosion process so enlarging destruction rate. Production machine with metal material based will suffer corrosion, and it will decrease their capability for production process.

Corrosion inhibitor application is one of easy and effective corrosion controlling techniques so that many industries had applied it. Corrosion inhibitor is chemical compound being able to hinder metal corrosion rate by its milieu. Generally, such compound applied by physical layering/*plating* at metal surface such as interface painting or layering by adsorption of inhibitor active at metal interface hence, molecular protection at metal surface/interface will be formed. However, corrosion inhibitor is surface active compound which may represent organic compound, anorganic, or

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coordination complex compound. The sample of organic compound functioned as corrosion inhibitor is ascorbic acid, polyaniline, polyamide, imidazole, dithiocarbamate, and dithiophosphate [2-3], while inorganic corrosion inhibitor among them are natrium nitrite and natrium chromate [4]. Sample of coordination complex compound functioned as corrosion inhibitor is ZDTP [3]. In the beginning, numerous ZDTPs used as lubricant additive at automotive industries [5], but, then, it had been known that this compound also may act as anti-corrosion [6].

Variously, alkyl chain at ZDTP may be produced in accordance with application necessity, the different alkyl chain will give the different physicochemical properties. The increasing of alkyl chain length at ZDTP, will increase lipophility and thermal stability. For commercial lubricant oil, the most used ZDTP has alkyl chain from C_4 to C_{10} [3]. Currently, numerous researches haven't already given report performance of ZDTP anticorrosion with oil/fatty acid based long alkyl chain. The proximate and ordered structure of ZDTP with long fattyalkyl chain will increase lipophility and solubility in base lubricant, and supposedly, will give better anti-corrosion performance than ZDTP generally applied in commercial lubricant oil currently.

Fatty alkyl application as lipophilic group on ZDTP is the best choice for downstream agro industry in Indonesia, because such group may be derived from fatty acid of vegetable oil such as palm oil representing superior commodity of Indonesia. Indonesia had given contribution around 51% for total palm oil production in the world, as world biggest palm oil producer. Indonesia and Malaysia had contributed around 87% palm oil production in the world or around 23% from total vegetable oil production in the world [7]. Indonesia palm oil export is around 75% from total national production, most of them (77%) are still in *crude palm oil* and *palm kernel oil*, and its remaining is intermediate products such as fatty *acid* and fatty alcohol [8]. Factually, it is comparative excellence in supplying raw materials for renewable vegetable oil based ZDTP agro industry development. Fatty alcohol as raw material of ZDTP may be obtained from fatty acid of palm oil conversion/reduction. By varying fatty acid/fatty alcohol type as raw material, ZDTP variants having fattyalkyl chain length difference will be obtained. Hence, the gaining of any excellent ZDTP derived from fatty acid/fatty alcohol of palm oil will become alternative for development of palm oil based downstream national superior product because it is supported by availability of abundant local raw material which will ensure supply continuity.

Performance of any anti-corrosion additive may be measured using *coupon cor*rosion by considering *coupon weight* loss [9], measure and compare change of *copperstrip corrosion* color [10] or potentiodynamic polarization by measuring corrosion current decrement by Tafel curve [11]. Polarization technique is more profitable from sides of fast work time, high sensitivity, and efficient corrosion process measurement. Additionally, corrosion current data obtained from this technique may be used to calculate change of Gibbs free energy (ΔG), enthalpy (ΔH), entropy (ΔS), and activation energy (Ea) [2,12].

In this research, $ZDTP_{16}$ is synthesized from cetyl alcohol derived from palmitic acid, a dominant fatty acid component in palm oil. Thermodynamic and kinetic parameter of anti corrosion from $ZDTP_{16}$ are calculated from corrosion current data by potentiodynamic polarization. Morphology observation of exposed substrate surface as result of corrosion monitoring by SEM-EDX. This research is aimed to synthesize $ZDTP_{16}$ from cetyl alcohol, by measuring and quantifying its inhibition performance based on parameters of activation energy, Gibbs free energy on transition state, and change of surface morphology of corrosion inhibition against Cu metal.

EXPERIMENTAL SECTION

Materials

Main materials used for $ZDTP_{16}$ synthesis are *cetyl alcohol*, ZnO (technique), *n*-heptane (AnalaR), and P_2S_5 (Merck).

Instrumentation

The main equipment used are synthesis reactor, Fourier Transform Infra red Spectrophotometer Prestige-2 (FTIR), Atomic Absorption Spectrophotometer of Shimadzu AA 6300 (AAS), *Differential Thermal Analysis (DTA)* NETZSCH TG 209F3 TGA209F3A-0257-L and *Thermal Gravimetric Analysis (TGA)*, *Scanning Electron Microscope-Electron Dispersive Xray* Zeiss Evo 50 (SEM-EDX), and Potentiostat DY2300EN.

Procedure

Synthesis and characterization of $ZDTP_{16}$ Product

$ZDTP_{16}$ synthesis is referred to Dinoiu et al. [13] by reacting *cetyl alcohol* and P_2S_5 with mol ratio 4:1 in *n*-heptane solvent (first stage reaction 1) for certain period in reactor completed with a set of H_2S absorber. Heating and stirring conducted at 90 °C at water heater. Such reaction will form dicetyl dithiophosphate acid (ADTP). As much as 1 mol ZnO is added at similar reactor to form $ZDTP_{16}$. Stirring without heating conducted for certain period in 2nd stage reaction for

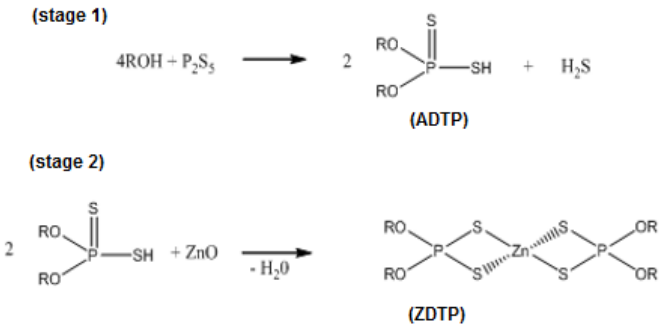

Fig. 1 ZDTP synthesis route

Table 1. Yield of $ZDTP_{16}$ product at 90 °C

Compound	Synthesis time (h)		Yield (%)
	Stage 1	Stage 2	
$ZDTP_{16}$	6	6	21.95 (n=3)
	12	12	87.17 (n=3)

n= synthesis repetition

$ZDTP_{16}$ formation. ZDTP formation reaction stage from alcohol illustrated at Fig. 1. $ZDTP_{16}$ product separation by extraction using 20 mL *n*-heptane and washed with 20 mL of water. Extraction and washing are conducted until water phase can be seen clearly. The organic phase is then vaporized and the obtained $ZDTP_{16}$ is weighed to calculate yield of product. Monitoring the success of the ZDTP16 production is conducted by functional group analysis using FTIR spectrometer, while recovery test of Zn metal in $ZDTP_{16}$ product is conducted by AAS spectrometer.

Thermal stability test of ZDTP$_{16}$ product

Thermal stability monitoring is conducted by using differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) instrument.

Performance of corrosion inhibition, kinetic and thermodynamic parameters

Work electrode of Cu with surface area of 2.19 cm^2 is prepared by sandpapering its surface and then is washed by distilled water and acetone. Subsequently, Cu electrode is put into reservoir containing 1% of NaCl solution. Reservoir is adjusted by water circulation at its mantle in order to maintain temperature stability. Consecutively, electrode of Ag/AgCl and Pt wire installed as reference and auxiliary electrodes. The test solution (NaCl 1%) is left to achieve stability with electrode for around 5 min. And then, choose linear sweep voltammetry (LSV) in DY2300EN program at computer. Blank measurement is conducted at potential range from 0 to 400 mV with *scan rate* 2 mV/s. After finishing this measurement, work electrode of Cu is cleaned by washing with 5% of HCl, distilled water, and is sandpapered again. The cleaned work electrode of Cu is then dipped in $ZDTP_{16}$ solution for 15 sec and is drained for a moment. After conditioning, sample is measured with similar potential range. Thereafter, the obtained data are processed using DY2300EN and Microsoft excel with tafel extrapolation, to get polarization curve. From that curve, potential (E_{corr}), Tafel constancy of anode (β_a) and cathode (β_c), as well as corrosion current (i_{corr}) will be obtained (14). Corrosion current data of $ZDTP_{16}$ product will be used for calculating thermodynamic parameter by using Arrhenius equation in transition condition. The exposed Cu plate is cleaned and its surface morphology is observed by using SEM. Morphology scanning is also conducted against blank Cu electrode and Cu control electrode (Cu electrode will take corrosion treatment without $ZDTP_{16}$ treatment).

RESULT AND DISCUSSION

Yield and Characterization of ZDTP$_{16}$

From repeating of reaction time variation, optimum time of $ZDTP_{16}$ synthesis obtained is 24 h. As illustrated in Table 1, synthesis time of 12 h only gives yield of 21.95%, while after 24 h gives 87.17%. Besides, synthesis yield is also influenced by synthesis temperature and 90 °C is the optimum temperature for $ZDTP_{16}$ synthesis reaction. According to Becchi et al. [5] if the reaction temperature used is more than 100 °C, ZDTP product will be decomposed, hence, the yield will be low. The obtained physical form of $ZDTP_{16}$ product is like congestion such as porridge with Zn average content of 6.06% which is higher than its theoretical calculation that is 5.35%.

Other than confirmation of Zn existence, achievement of $ZDTP_{16}$ synthesis is also verified from existence of specific spectrum band at FTIR. Fig. 2 shows confirmation of spectrum band at some wave numbers indicating existence of zinc *bis*(dipalmithyl dithiophosphate) in $ZDTP_{16}$ product. Intensity of sharp absorption at wave number of 1467.83 cm^{-1} and 1377.17 cm^{-1} indicates vibration of $–CH_2-$ and $–CH_3$ at $ZDTP_{16}$. The absence of absorption band at wave number of 3500-3200 cm^{-1} indicates that cetyl alcohol reacted completely. The existence of P-O-C groups are indicated by absorption band at wave number of 1066-894 cm^{-1}, while, absorption band at 667-543 cm^{-1} indicates the existence of P-S group [15-16], meanwhile, Zn-S group with wave number of 400-300 cm^{-1} was not measured.

Thermal Stability of ZDTP$_{16}$

Thermal stability of $ZDTP_{16}$ product is measured using DTA and TGA. TGA measures change of product

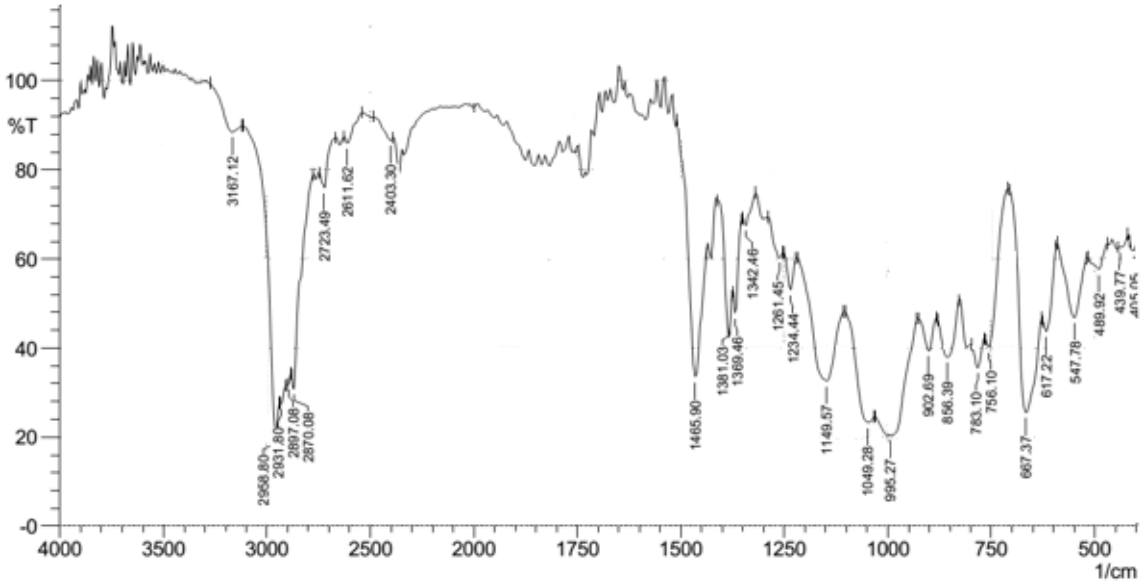

Fig 2. FTIR Spectrum of zinc bis(dipalmithyl dithiophosphate)

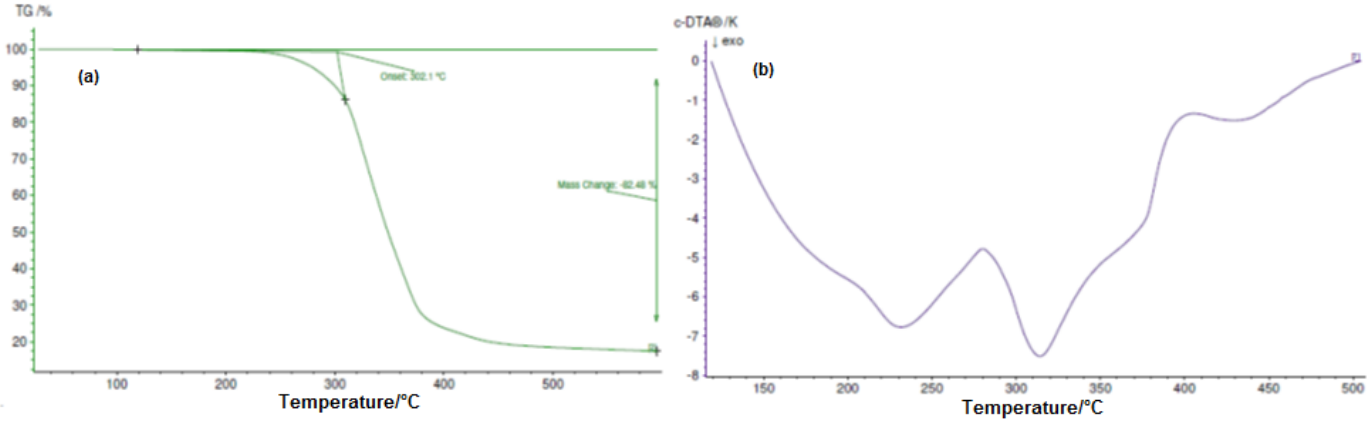

Fig. 3 DTA/TGA curve of $ZDTP_{16}$

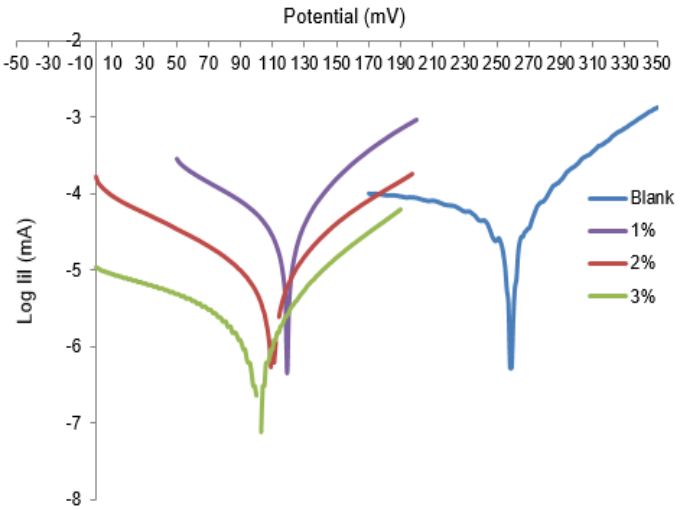

Fig 4. Potentiodynamic polarization curve of Cu metal corrosion

mass as response from temperature change. This thermal analysis is conducted to know critical temperature of $ZDTP_{16}$ stability, that is temperature when $ZDTP_{16}$ begin to be decomposed and other properties changes. Fig. 3a shows that initial decomposition temperature of $ZDTP_{16}$ is 302 °C, around this temperature mass change is occurred as result of endoterm reaction viewed from DTA curve. Continuously, decomposition occurs until 500 °C with measured mass loss of 82.48%. It indicates that $ZDTP_{16}$ variant has good thermal stability and it may be applied as corrosion inhibitor at environmental temperature achieving 300 °C without thermal degradation.

Anti-Corrosion Performance of $ZDTP_{16}$

Fig. 4 shows the result of potentiodynamic polarization of Cu metal corrosion at application dose of

Table 2. Influence of $ZDTP_{16}$ concentration against corrosion of Cu metal in 1% of NaCl at 28 °C

Concentration (%)	Potential of corrosion (mV)	Current of corrosion (mA)	% EI	θ	C_R (mmpy)
Blank	260.11	0.0225	-	-	0,1524
1.0	122.09	0.0104	53.82	0.54	0,0704
2.0	114.87	0.0035	84.33	0.84	0,0239
3.0	102.18	0.0006	97.41	0.97	0,0039

Table 3. Thermodynamic parameter and inhibitor $ZDTP_{16}$ corrosion kinetic

Test solution	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1} K^{-1})	ΔG^* (kJ mol^{-1})	E_a (kJ mol^{-1})
Blank	7.18	-257.56	85.22	16.66
Sample (3% $ZDTP_{16}$)	10.68	-267.61	91.77	33.68

$ZDTP_{16}$ 0–3%. Seemingly, curve of Tafel anode and Tafel catode cut each other and form straight line resulting current value and corrosion potential. By varying different application dose, corrosion current data will be obtained as illustrated in Table 2.

The higher $ZDTP_{16}$ concentration, the lower corrosion current value as well as increasing of inhibition effectiveness. This result indicates that the higher inhibitor concentration, the higher adsorbed inhibitor molecule of $ZDTP_{16}$ at Cu metal interface so that the corrosion process will undergo inhibition and resulting in lower corrosion current value.

Effectivity of Inhibition (% EI) is determined from difference of corrosion current sample and blank against blank corrosion current. Measurement result indicates inhibition effectiveness increment is in line with sample concentration increment which is comparable with surface closure degree (θ) increment. Because, the higher inhibitor molecule in solution, the possibility of molecule adsorption at metal surface will be higher physically and chemically. Corrosion rate (C_R) is obtained from weight loss measurement. The corrosion current decrement impact to corrosion rate value decrement indicating any corrosion delay. The same corrosion rate value at concentration of 2.0% and 3.0% indicates such concentration are optimal dose of $ZDTP_{16}$ application for protecting Cu metal from corrosion.

Thermodynamic parameter is determined based on Arrhenius equation in transition state [2]:

$$\ln\frac{i_{corr}}{T} = \ln\frac{R}{N_A h} + \frac{\Delta S^*}{R} - \frac{\Delta H^*}{RT}$$

Consecutively, parameter of ΔH^* and ΔS^* representing change of enthalpy and entropy in transition state, while $N_A h$ is Planck molar constancy (3.99 x 10^{-10}JS mol^{-1}). By varying temperature (T), then, ΔH^* and ΔS^* may be determined from ln (i_{corr}/T) *vs* 1/T curve, while change of Gibbs free energy transition (ΔG^*) is calculated according to thermodynamic equation as follows:

$$\Delta G^* = \Delta H^* - T\Delta S^*$$

The ease of any reaction occurrence is depending on required minimal energy in order to run such reaction. Activation energy may be calculated based on data plot between ln i_{corr} and 1/T according to following Arrhenius equation (Morad dan El-Dean 2006):

$$i_{corr} = Ae^{-\frac{E_a}{RT}} \quad \text{or} \quad \ln(i_{corr}) = \ln A - \frac{E_a}{RT}$$

Where A is Arrhenius constancy determined empirically, E_a is activation energy of corrosion process (kJ mol^{-1}), R is ideal gas constancy (8.314 Jmol^{-1}K^{-1}), and T is temperature (K).

Table 3 shows positive value of ΔH^* and ΔH^*_{sample} value is higher than it's blank. It indicates that a larger energy is required for corrosion occurrence with $ZDTP_{16}$ existence at Cu metal surface. Change of transition entropy clarifies the irregularity degree of system. The $ZDTP_{16}$ inhibitor existence at system will increase irregularity degree of system manifested by increasing of measured ΔS^* [12]. ΔS^*_{sample} is higher than its blank, although, such ΔS^* increment may not be made as measurement of process spontaneity for environment ΔS^* had not been determined yet. Positive value of ΔG^* identifies that electrochemical process in corrosion simulation had not taken place spontaneously; hence, it requires external energy supply by electrical power. Change of Gibbs free energy transition of sample (+91.77 kJ mol^{-1}) is more positive than blank (+85.22 kJ mol^{-1}). It indicates that corrosion spontaneity decreased by existence of $ZDTP_{16}$ as inhibitor. In other word, corrosion process had been hindered by existence of $ZDTP_{16}$ inhibitor.

Identification of kinetic corrosion manifested by activation energy parameter (E_a) obtained from curve plot of Arrhenius *ln* (i_{corr}) against 1/T. Activation energy (E_a) obtained from declivitous curve multiplied with ideal gas constancy (R). As illustrated in Table 3, seemingly, $ZDTP_{16}$ addition influences to activation energy increment from 16.66 to 33.68 kJ mol^{-1}.

Surface Protection by $ZDTP_{16}$

Fig. 5 illustrates morphology observation of Cu metal surface before and after corrosion using SEM. Seemingly, at Fig. 5c massive destruction occurs at

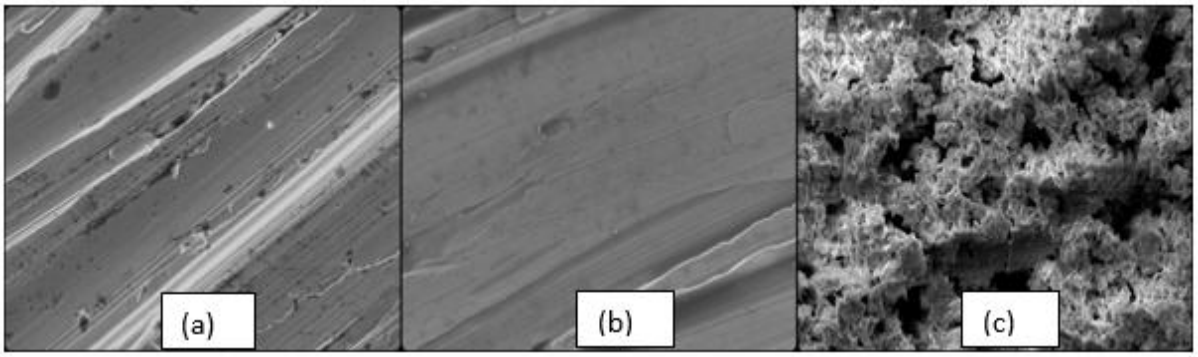

Fig 5. The SEM photograph of Cu metal surface at 1000X: prior to electrolysis/blank (a), upon electrolysis plus anti-corrosion of $ZDTP_{16}$ (b) upon electrolysis without $ZDTP_{16}$ addition/control (c)

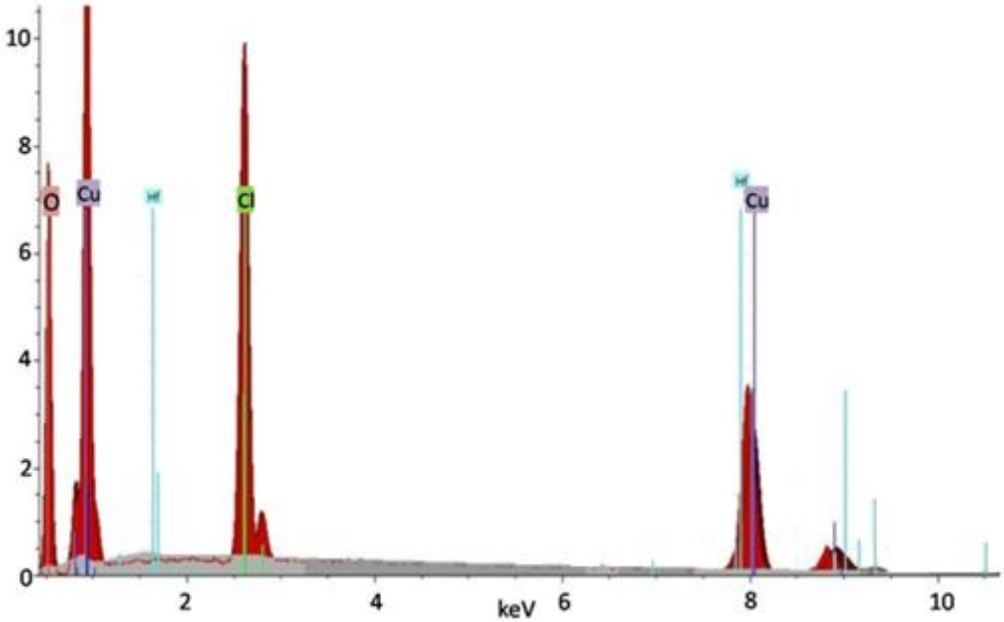

Fig 6. Analysis Result of EDX to Cu control electrode

surface of Cu electrode without $ZDTP_{16}$ treatment upon electrolysis. Corrosion at Cu electrode surface had resulted in broken, wrecked, wavy, and rough surface morphology. It also illustrates the formation of metal oxides as result of corrosion process occurred, while Fig. 5b of Cu electrode which surface layered by $ZDTP_{16}$ is not exposed, no surface destruction as result of corrosion which is almost same with Fig. 5a, electrode surface prior to electrolysis (blank).

Of course, achievement of $ZDTP_{16}$ in inhibiting corrosion will be related with the ease of such molecules adsorption at surface/interface. The adsorption of $ZDTP_{16}$ at Cu metal surface may be viewed from EDX spectrum. Fig. 6 shows EDX spectrum control of Cu metal surface (without dipping in $ZDTP_{16}$ solution) after undergoing corrosion (electrolysis). Seemingly, intensity of chlorine and oxygen atom spectrum is so high indicating that corrosion process had occurred at Cu surface with existence of chloride ion as corrosion agent derived from NaCl solution for electrolyte agent/media. The high intensity of oxygen spectrum shows corrosion process resulting oxides formation at surface. EDX result in Fig. 6 in line with SEM result at Fig. 5c showing morphology of exposed Cu electrode and oxide formation as result of corrosion.

It is different with control in Fig. 6, Fig. 7 shows analysis result of EDX against sample detecting existence of Zn, P, S, and C, other than chlorine and oxygen atom. The existence of Zn, P, S, and C atoms representing atoms composing $ZDTP_{16}$ at surface of sample electrode (by dipping at inhibitor) proven that $ZDTP_{16}$ is really adsorbed at Cu metal surface/interface as protector from corrosion attack. The decreasing of Cl atom spectrum intensity at Cu electrode surface in Fig. 7 compared to Fig. 6 shows that the corrosion is hindered by existence of $ZDTP_{16}$ at surface.

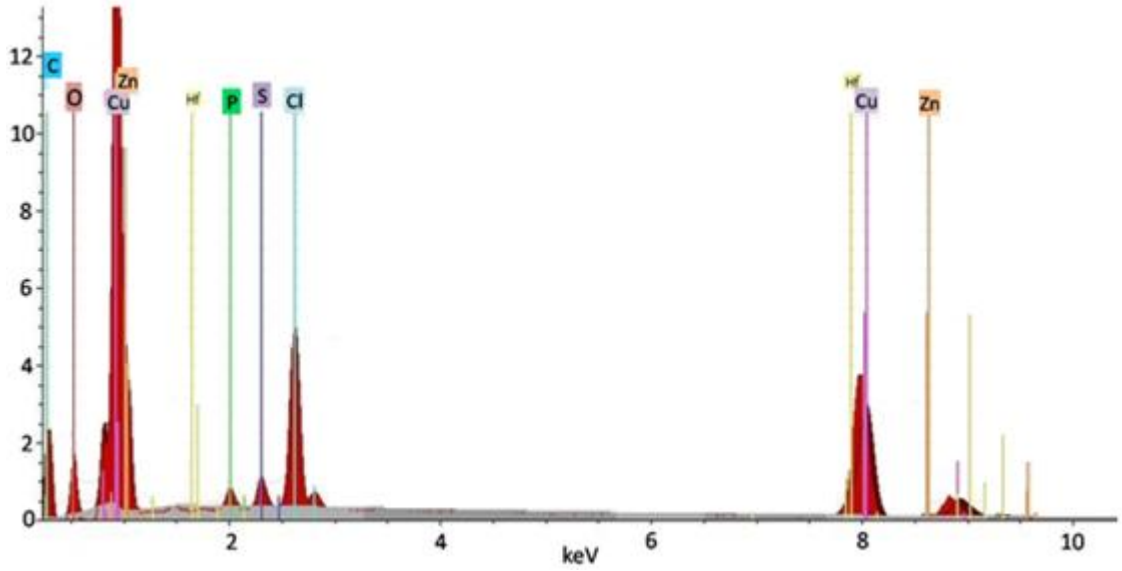

Fig 7. Analysis Result of EDX to Electrode Cu Sample

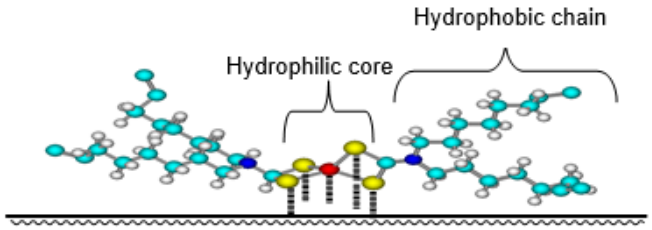

Fig 8. ZDTP adsorption model at metal surface substrate

Fig. 8 shows interaction model of hydrophilic heteroatom $ZDTP_{16}$ cluster adsorbed at surface of Cu electrode acting as layer of molecular protector.

CONCLUSION

Successfully, $ZDTP_{16}$ had been produced with yield level of 87.17% at synthesis optimum time of 24 h. $ZDTP_{16}$ has inhibition activity of 97% at application dose of 3% and is able to decrease corrosion rate of Cu metal from 0.152 to 0.004 mm per year. Such power of $ZDTP_{16}$ corrosion inhibition in line with achievement of its thermodynamic measurement parameter. By using Cu metal and NaCl electrolyte as target in simulator, Gibbs free energy transition corrosion had increased from +85.22 to +91.77 kJ mol^{-1}, while its activation energy had increased from 16.66 to 33.68 kJ mol^{-1}. Morphology observation by substrate surface of Cu metal by using SEM-EDX strengthens proof of surface protection by $ZDTP_{16}$. Existence of Zn, P, S, and C constituents representing composer atoms of $ZDTP_{16}$ and the decreasing of Cl^- as constituent corrosion at substrate surface indicates that $ZDTP_{16}$ is a molecule having surface activity and adsorbed at surface/interface hence, it may act as surface protector molecule from corrosion process.

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