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Synthesis And Corrosion Inhibition Property Of Sodium Diphenyltin (IV) Dodecanedioate On Mild Steel In 0.5 M H_2SO_4 Solution

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Abstract:
Sodium diphenyltin (IV) dodecanedioate (SDD), ($Ph_2Sn[OCO(CH_2)_{10}COONa]_2$) was synthesized and studied for corrosion inhibition potential on mild steel (MS) in 0.5 M H_2SO_4. The synthesized SSD was obtained as white crystalline salt with actual yield of 1.2118 g and a percentage yield of 90.30 %. The corrosion inhibition test was carried out using weight loss measurements by varying concentration of the complex and the temperature respectively. The inhibition efficiency increased with concentration but decreased with rise in temperature, suggesting physisorption mechanism. The adsorption of the inhibitor on the mild steel surface was spontaneous and found to align with Langmuir and Freundlich adsorption isotherm models. Kinetic treatment of the data followed a pseudo-first order reaction. The values of activation energy (Ea) obtained further corroborated physical adsorption mechanism. The values of ΔH and ΔS obtained points to an endothermic nature of the adsorption as well as follow associative mechanism. The obtained results clearly revealed that SDD is as an effective inhibitor of the corrosion of MS in 0.5 M H_2SO_4 solution.

Key Word*: Synthesis, Diphenyyltin (IV) dodecaredioate, Corrosion inhibition, Mild Steel, Kinetic parameters*

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I. Introduction

Almost all the environments are potentially harmful to metals in contact with them, causing frequent irreversible interfacial reaction when they come in contact. This interfacial reaction results in drastic degradation of the metal, thus, affecting its useful life span [1]. The high cost of production and potentially harmful effects of cobalt compounds used as corrosion inhibitors on the ecosystem necessitated the search for a low-cost, efficient, and environmentally friendly alternative [2].

The chemistry of tin compounds continue to be of interest to chemist and chemical engineers due to their wide range of application as agricultural biocides, antitumor agents, wood preservatives, antioxidants for poly propylene, stabilizers for polyvinyl chloride, marine antifouling coating, flame retardants, anti-wear agents and recycling agents [3]. Organotin chemistry is part of the wider field of organometallic chemistry. This area remains rich with many applications in industry and continuing activity in the research laboratory [4].

Organotin (IV) compounds are characterized by the presence of at least one covalent Sn-C bonds. These compounds contain tetravalent Sn centres and can be classified as mono, di, tri and tetraorganotin (IV), depending on the number of alkyl or aryl moieties attached to the tin metal [5].The synthesis of organotin (IV) complex with new ligand in this study is an attempt to further explore their properties and develop corrosion inhibitors for mild steel.

Mild steel is the major item of construction in industries. It is a material of choice because it is cheap and easily obtained when compared to stainless steel. Mild steel have good strength, it is hard and can be bent, worked or can be welded into an endless variety of shapes for use from vehicles to building materials. However, they are prone to the damaging effects of corrosion attack overtime as they continuously interact with the environment. Over the years, the chemical used to reduce corrosion effect are toxic and pose threat to the environment. Therefore, the question is how can we substitute the toxic chemicals and obtain sustainable means of arresting corrosion of mild steel [6,7]? The study of corrosion is of paramount importance because of the direct and indirect losses caused by this scourge. One of the methods to slow down the corrosion process is by applying inhibitors. An inhibitor is any substance, which when added to a corrosive environment in small quantity reduce the corrosion rate of the metal. Corrosion inhibition is a surface process which involves

adsorption of the organic compounds on a metal surface. It is an electrochemical reaction, which occurs naturally and proceeds by itself; as such, corrosion cannot be totally stopped [8,9] but reduced.

II. Material And Methods

Diphenyltin(IV) oxide, $(C_6H_5)_2SnO$, dodecanedioic acid, NaOH, H_2SO_4, Ammonium acetate, $C_2H_7NO_2$, methanol, n-propanol Acetone, were Zayo sigma Aldrich products and were used without further purification.

Experimental Procedure

Experimental procedure used for this research work was adopted from [10-12]

Preparation of $\mathbf{HOOC(CH_2)_{10}COONa}$

The ligand was prepared by reacting sodium hydroxide (0.05mol, 1.0204 g) with dodecanedioic acid (0.05mol, 5.8674 g). The mixture was completely dissolved in 200 mL of distilled water in 250 mL flat bottom flask containing a magnetic stirrer bar and reflux for 1hour giving a clear solution. The solution was cooled in an ice-bath and white crystals of sodium hydrogen dodecanedioate precipitated out of the solution. The crystals were filtered using Buchner funnel and allowed to dry for five days in a desiccator to a constant weight.

Synthesis of $\mathbf{Ph_2Sn[OCO(CH_2)_{10}COONa]_2}$

Diphenyltin (IV) oxide (0.0017mol, 0.5000 g) was refluxed in a mixture of methanol and n-propanol of ratio 4:1 in a 250 mL flat bottom flask for 40 minutes using Dean and stark apparatus at 60^0C. A 0.8732 g of intermediate product called propoxide was obtained. Sodium hydrogen dodecanedioate in methanol was added while being stirred vigorously on a hot plate magnetic stirrer. The product was obtained after methanol was distilled off at 67^0C. The resulting mixture was then heated in an oven for period of 72 hours at 40^0C, and a white crystal of sodium diphenyltin (IV) dodecanedioate was obtained.

Corrosion Inhibition Mechanism

Pre-weighed MS coupons were submerged in 200 mL of 0.5 M H_2SO_4 solution. The SDD concentrations utilized in this study ranged from 0.1, 0.3 and 0.5 g/L. Tests were run for 305, 309 and 313 K respectively for 7 hours of immersion duration. The MS coupons were taken out of the acid-inhibitor system once the immersion period had passed, cleaned, dried, and weighed. The weight loss was taken to be the difference between the coupon's initial and final weight at a particular period. Every test was carried out in duplicate. The inhibition efficiency (%IE) and corrosion rate of mild steel were calculated from the weight loss results, according to equations 1 and 2 [14],

$$IE_{\exp} = \left(1 - \frac{W_{(1)}}{W_{(0)}}\right) x100 \qquad (1)$$

where, $W_{(0)}$ is the weight loss of the mild steel without inhibitor and $W_{(1)}$ is the weight loss of mild steel with inhibitor.

$$CR\left(gh^{-1}cm^{-2}\right) = \frac{\Delta W}{At} \qquad (2)$$

where ΔW is the weight loss, A is the area of the coupon and t is the immersion time.

III. Result/Discussion

Synthesis of SDD

SDD was synthesis following the mechanistic steps shown as equation 1-3 below. A white crystalline salt with actual yield of 1.2118 g and a percentage yield of 90.30% was obtained.

$$NaOH + HOCO(CH_2)_{10}COOH \xrightarrow{reflux} HOOC(CH_2)_{10}COONa + H_2O \qquad (3)$$

$$Ph_2SnO + CH_3OH : CH_3CH_2OH \xrightarrow{reflux} Ph_2Sn[OC_3H_7]_2 + CH_3OH + H_2O \qquad (4)$$

$$Ph_2Sn[OC_3H_7]_2 + 2HOOC(CH_2)_{10}COONa \xrightarrow{reflux} Ph_2Sn[OCO(CH_2)_{10}COONa]_2 + 2H_2O \qquad (5)$$

Corrosion Inhibition Test

Effect of Temperature

Variation in weight loss and inhibition efficiency of MS in 0.5 M H_2SO_4 solution in the absence and presence of SDD are presented in Figures 1and 2 respectively.

The weight loss increased with temperature but decreases as the concentration of the inhibitor increases. There is a progressive increase in weight loss as the temperature is increased from 305 to 313K. This signifies that the dissolution of the metals increased at higher temperatures. The observation is attributed to general rule guiding the rate of chemical reaction, which says that chemical reaction increase with increasing temperatures. Also an increased temperature favors the formation of activated molecules, which may be doubled in number, with 10^{o}C rise in temperature thereby increasing the reaction rate these is because the reactant molecules gain more energy are able to overcome the energy barrier more rapidly. An increase in temperature may also increase the solubility of the protective films on the metals, thus increasing the susceptibility of the metal to corrosion [15-16].

The variation of inhibition efficiency with temperature for 7 hours immersion period in the presence or the absence of SDD are shown in **Figure 2**. The inhibition efficiency decreased with increasing temperature, a trend similar to the corrosion rate. This may be as a result of increasing solubility of the adsorbed protective inhibitor films on the mild steel coupons, thereby increasing the susceptibility of these coupons to dissolution in the acid media [17]. This result suggests a physical interaction (physisorption) of the inhibitor on the metal surface, as physisorption is characterized with a decrease in inhibition efficiency with temperature as opposed to chemical adsorption mechanism, where inhibition efficiency is expected to increase with increase in temperature [18].

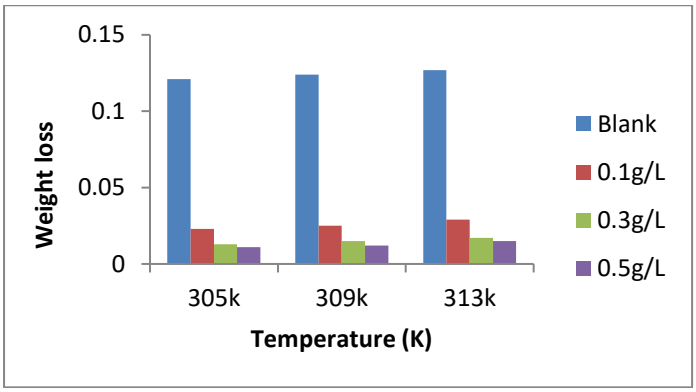

Figure 1: Variation in weight loss for the effect of temperature on the corrosion of mild steel in 0.5 M H_2SO_4 solution in the absence and presence of S

Table 1: Inhibition Efficiency obtained from Weight Loss Measurements of MS. Inhibition Efficiency (%)

System (g/L)	305 K	309 K	313 K
0.1	80.99	79.84	77.17
0.3	89.26	87.90	86.61
0.5	90.91	90.32	88.19

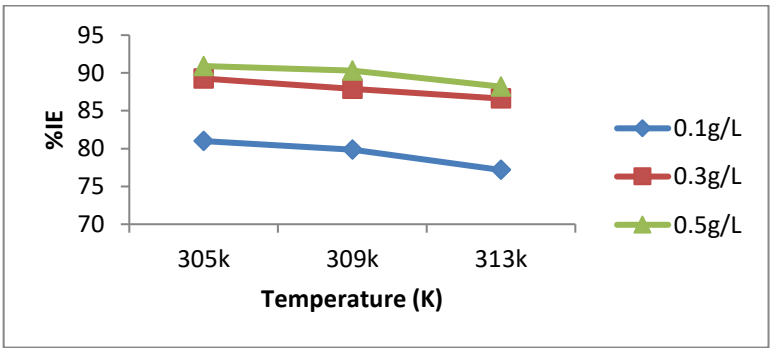

Figure 2: Effect of temperature on corrosion inhibition efficiency of mild steel in 0.5 M H_2SO_4 solution in the absence and presence of SDD

Kinetics/Thermodynamics of the Corrosion
Rate constant and half-life

The corrosion reaction is a heterogeneous reaction which is composed of anodic and cathodic reactions at the same or different rate [19]. It is on this basis that kinetic analysis of the data is considered necessary. The half-life was obtained from equation (7), [20].

The effect of temperature on the corrosion rate of MS in 0.5 M H_2SO_4 solution in the absence and presence of SDD (**Fig 3**). It is evident that the corrosion rate of MS with or without the inhibitor increased with increase in temperature. This is because as the temperature increased the rate of corrosion of the MS coupons also increased due to increasing average kinetic energy of the reacting molecules [21].

Figure 4 shows the plot of –log (weight loss) against temperature (K) in the absence and presence of SDD. The rate constant parameters; rate constant and half-life are also recorded in **Table 2**. The plots showed a linear variation and slop, k, which confirms a pseudo-first order reaction kinetics with respect to the corrosion of MS in 0.5 M H_2SO_4 solution in the absence and presence of SDD. It is evidenced from the result that the half-life decreased with temperature. This further supports the fact that the interaction between the mild steel and the inhibitors is physisorptive. This result is in line with that reported by Ijuo *et al.,* [22].

The plot of log (CR/T) vs. 1/T (Fig 4) and the thermodynamics parameter (Table 3) were obtained using equations 6 and 7 below.

$$log\frac{CR_2}{CR_1} = \frac{Ea}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad (6)$$

$$CR = \frac{RT}{Nh}\exp\left(\frac{\Delta S_{ads}}{R}\right)\exp\left(\frac{-\Delta H_{ads}}{RT}\right) \quad (7)$$

Where CR_1 and CR_2 are the corrosion rates of mild steel at the temperature T_1 and T_2, E_a is the activation energy, R is the gas constant, N is the Avogadro's number, h is the plank's constant, T is temperature, ΔS_{ads} and ΔH_{ads} are the entropy and enthalpy of adsorption of the inhibitor on a metal respectively.

The results showed that Ea values in the presence of SDD were higher than that of the blank, (Table 3). This showed that the adsorbed inhibitor has provided a physical barrier to the change and mass transfer, leading to reduction in corrosion rate (Eddy *et al*., 2010 and 2011). It has been reported that the value of Ea greater than 80 $kJmol^{-1}$ indicates chemical adsorption, whereas, Ea less than 80 $KJmol^{-1}$ infers physical adsorption [23]. On the basis of the experimentally determined Ea values that are all less than 80 $KJmol^{-1}$, it is evidenced that the additives were physically adsorbed on the coupons. Therefore, it is plausible that a multilayer protective coverage on the entire mild steel surface was obtained [24].

The results showed that all the enthalpy of activation for the inhibitors are positive, reflecting the endothermic nature of the MS dissolution process. Also, the entropies of activation energy were positive for the SDD, indicating that the activation complex represents association steps and that the reaction was spontaneous and feasible. These results were in excellent agreement with the reports of previous work [25].

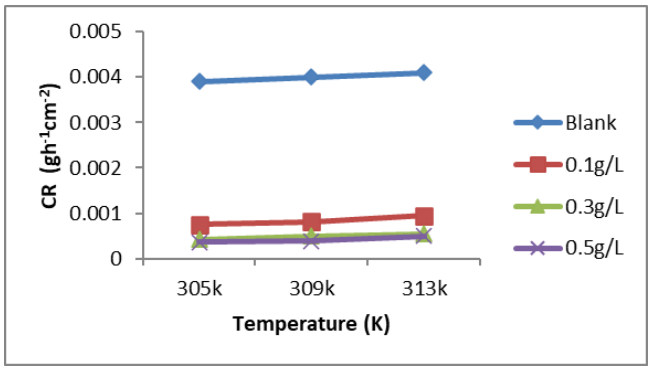

Figure 3: Effect of temperature on corrosion rate of mild steel in 0.5 M H_2SO_4 solution in the absence and presence of SDD.

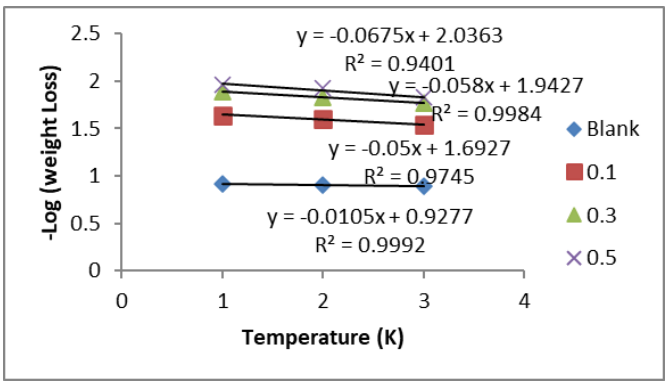

Figure 4: Variation of -log (weight loss) with temperature (K) for mild steel in 0.5 M H_2SO_4 solution in the absence and presence of SDD.

Table 2: Rate Constant and Half-life parameter at different Concentration

System	Rate Constant k	Half Life
Blank	0.010	69.30
0.1g/L	0.050	13.86
0.3g/L	0.058	11.95
0.5g/L	0.067	10.34

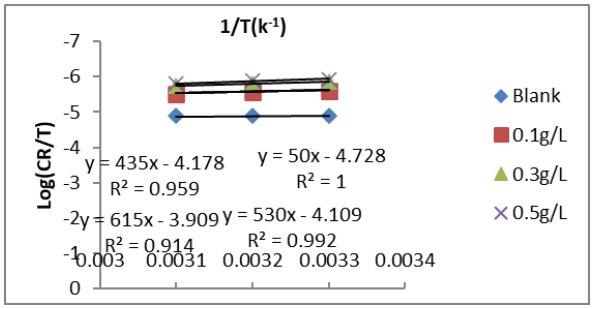

Figure 4: Transition state plots for the corrosion of mild steel in 0.5 M H_2SO_4 solution in the absence and presence of SDD.

Table 3: Thermodynamic activation Parameters for the Dissolution of MS.

System	Ea (kJmol^{-1})	ΔH (jmol^{-1})	ΔS(kJmol^{-1})
Blank	4.96	957.36	0.101
0.1g/L	22.41	8329.01	0.090
0.3g/L	26.76	10147.96	0.089
0.5g/L	30.59	11775.49	0.085

Adsorption Isotherm

Different adsorption isotherms were tested in order to obtain more information about the interaction between the inhibitor and the MS surface. The various isotherms tested includes Temkin, Frumkin, Freundlich and Langmuir adsorption isotherms and linear regression coefficients ($r2$) were used to determine the best fit. Langmuir and Freundlich adsorption isotherms were found to be best fit in which case all the linear regression coefficients ($r2$) were close to unity. Langmuir adsorption isotherm assumes that the solid surface contains a fixed number of adsorption sites and each site holds one adsorbed species [26]. Langmuir isotherm gives a straight line between $C\theta$ *vs* C [27] as shown in **Figure 5**, where C is the concentration of the inhibitors and θ the surface coverage. The results obtained for ΔG and Kads are shown in **Table 4**. The values obtained for ΔG were negative indicating that the adsorption process proceeded spontaneously.

Freundlich adsorption isotherm is based on the assumption that adsorption sites are distributed exponentially with respect to energy of adsorption and that the surface sites are subdivided into several types, each possessing a characteristic heat of adsorption [28]. The Freundlich equation was applied indiscriminately to isotherms, all of which were limited to adsorption from dilute solutions, showing that adsorption increased indefinitely with increasing concentration. A plot of log θ against log C are shown in **Figures 6**. The linearity shows that the adsorption of the inhibitors on mild steel surface in aqueous medium follows Feundlich isotherm [29].

Generally, the value of ΔG°ads ≤ −20 kJ mol^{-1} signify physisorption and values more negative than −40 kJ mol^{-1} signify chemisorption (Eddy and Ekop, 2007; Ihebrodike *et al*., 2010, Saratha *et al;* 2011). The results are presented in Table 7. The values of ΔGads are negative and less than -40 kJmol^{-1}. This implies that the adsorption of the inhibitor on metal surface is spontaneous and confirms physical adsorption mechanism [30-31].

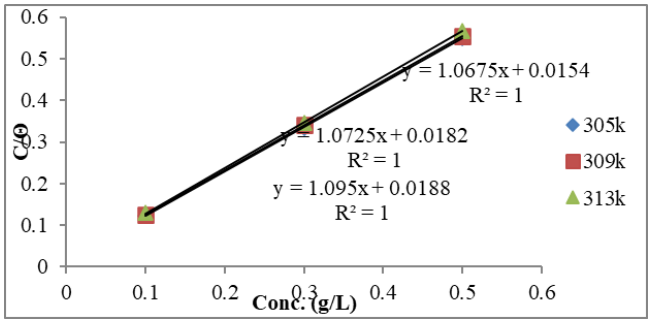

Figure 5: Langmuir isotherm for the adsorption of SDD on MS surface in 0.5 M H_2SO_4

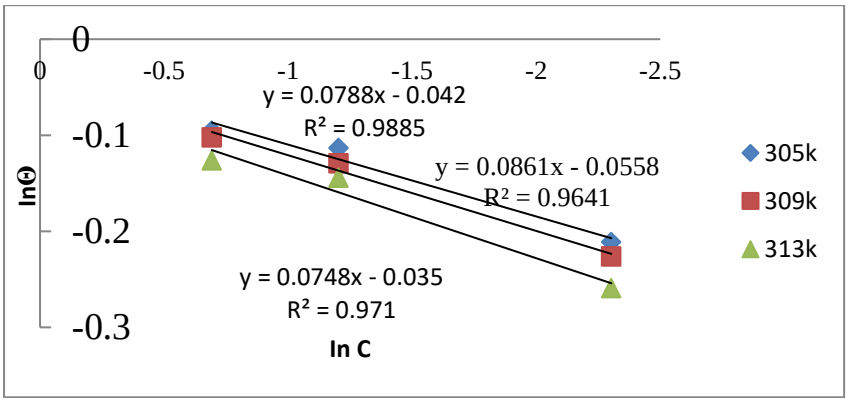

Figure 6: Freundlich isotherm for the adsorption of SDD on MS surface in 0.5 M H_2SO_4

Table 4: Langmuir and Freundlich adsorption isotherm parameters obtained from the corrosion data for mild steel in 0.5 M H_2SO_4 containing SDD.

Isotherm	Intercept	Slope	k_{ads}	R^2
Langmuir				
305K	0.015	1.067	19.98	1.000
309K	0.018	1.072	18.62	1.000
313K	0.018	1.095	14.95	1.000
Freundlich				
305K	0.035	0.074	1.084	0.971
309K	0.042	0.078	1.102	0.988
313K	0.055	0.086	1.135	0.964

IV. Conclusion

The organotin (IV) derivative of dodecanedioic acid was synthesized and tested for corrosion inhibition property. The synthesized complex was only soluble in organic solvent but insoluble in water and has the melting point at 116-120°C. The SDD has shown the ability to effectively inhibit the corrosion of MS in 0.5 M H_2SO_4, giving up to 90.91%1E at the concentration of 0.5 g/L and temperature of 305 K. The adsorption of SSD on the MS surface obeys Langmuir adsorption isotherms. The inhibition efficiency increased with increase in temperature and the activation energy increases with increase in concentration. SDD can therefore be used as inhibitor of corrosion of steel.

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