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CORROSION INHIBITION OF STEEL IN MARINE ENVIRONMENT USING AMINO AMIDE DERIVED FROM PET PLASTIC WASTE

R. S. ABDEL HAMEED [a,b,*], M. ALELAIMI [a], M. T. QURESHI [a,c],
S. R. AL-MHYAWI [d], A. M. AL-BONAYAN[e], M. F. H. ABD EL-KADER [a, f]
[a]*Basic Science Department, Preparatory Year, University of Ha'il, 1560, Hail, KSA*
[b]*Faculty of Science, Department of Chemistry, Al-Azhar University,11884, Cairo, Egypt*
[c]*Department of Physics, Hazara University, Mansehra Pakistan*
[d]*Chemistry department, Faculty of Science, Univeresity of Jeddah, Jeddah, KSA*
[e]*Chemistry Department, Faculty of Science, Umm Al-Qura University, Makkah Mukaramha, KSA*
[f]*Biophysics Department, Faculty of science, Cairo University, Egypt*

Inhibition of steel from corrosion process in artificial marine environment of 2.0 M sodium chloride solution using amino amide compound derived from Poly (ethylene terephthalate) plastic waste, PET. In this respect solvent free efficient green recycling of PET waste via aminolysis with 1,3-diaminopropane in the presence of (sodium acetate/acetic acid) catalyst, the product is (N, N'-Bis-(3-amino-propyl)-terephthalamide) compound as nonionic surfactant was separated in good yield, characterized by FT-IR and ^{1}HNMR, and evaluated as green corrosion inhibitor for steel alloys used in manufacturer of petroleum pipe lines using electrochemical techniques and atomic absorption spectroscopy(AAS). Effect of inhibitor concentration and temperature were studied. The corrosion inhibition efficiency found to increases with increasing of the inhibitor concentration and decreased by rising the temperature. Potentiodynamic polarization curves indicate that the used system act as mixed inhibitor. The data of AAS show that the ferric ion Fe^{+3} concentrations were decreased by increasing inhibitor concentration. The inhibition of amino amide compound derived from waste is due to adsorption and adhesion of its molecules on the steel surface which obeys Langmuir desorption isotherm model.

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1. Introduction

The overall world production of polyester was about 30 milion tons in 2000, this value increased to 55 million in 2012 and most consisted of PET. As a result of the diversity of its applications in a high volume of consumer products, large amount of PET waste is also generated, which includes polymer manufacturing waste as well as the products after the end of their useful life. PET is semi-crystalline, thermoplastic polyester of characteristic high strength, transparency, and not biodegradability [1]. PET is not a hazardous product, but its waste quantity increases drastically. With the increasing pressure of keeping the environmental clean. Recycling of PET waste is an ecofriendly manner is the only solution. PET waste can be recycled by different methods like physical recycling and chemical recycling. Polyethylene Terephthalate (PET) is widely used thermoplastic that generates tones of wastes that may otherwise disrupt the balance of ecosystem due to its non-biodegradation in the environment. The chemical recycling of PET to useful materials is an accepted technique among recycling methods (mechanical, thermal, and chemical) that follows the rules of sustainable developments [1-7]. There are different chemical depolymerization methods used for PET chain scission, such as methanolysis, glycolysis, hydrolysis, aminolysis, aminolysis, and hydrogenation, which are based on the types of chemical

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[*] Corresponding author: mredars2@yahoo.com

reagents [1-7]. During chemical recycling, PET waste can be depolymerized to base monomers or oligomers. With the use of solvent of depolymerization, generally called solvolysis of polymer, methanolysis and glycolysis [5-7] are the main possible routes. The PET bottle was patented in 1973 by Nathaniel Wyeth and began to be used popularly for the production of disposable soft drink bottles in the 1980s. in 1987, more than 700 million pounds of PET were consumed in their production [7]. Steel is aback boon of heavy industries, Corrosion control of steel is of technical, economical, and environmental importance, corrosion inhibitors are commonly used to reduce the corrosion attack on the steel surface [7]. The presence of the inhibitors leads to decline in the rate of corrosion of steel and hence the lifetime increases [7]. Most of the effective acid inhibitors are organic compounds containing nitrogen, oxygen and /or sulfur and these compounds adsorp at the steel surface and hence slow down the rate of corrosion [8-12]. The strength of the adsorption depends on the several factors such as the chemical structure of the inhibitors, the presence of electro donating or electro repelling group, molecular weight of the inhibitor, temperature and electrochemical potential at the metal / solution interface [13,14]. surfactant inhibitor has many advantages such as high inhibition efficiency, low price, low toxicity and easy production [7,15]. The adsorption of the surfactant on the metal surface can markedly change the corrosion resisting property of the metal [16], and so the study of the relationship between the adsorption and corrosion inhibition is of great importance. In the previous works [16-24] depolymerization by aminolysis of PET, with monoethanolamine, and ethylene di amine the products nonionic surfactants act as good corrosion inhibitors for steel in HCl. This work is one from the series aimed to alleviate the environmental pollution by accumulation of plastic waste by converting waste into modified products, and to evaluate the modified products as corrosion inhibitors for metals and alloys in different aqueous media [16-24]. In the present work 1,3 –di amino propane was used to convert PET waste into (N, N'-Bis-(3-amino-propyl)-terephthalamide) BAPTA, via solvent free green recycling, the product was evaluated as corrosion inhibitor for carbon steel in 2.0 M sodium chloride solution as artificial sea water by electrochemical, and atomic absorption spectroscopy (AAS) techniques. Effect of concentration and adsorption isotherms were studied.

2. Experimental

2.1. Materials

All chemicals used in this work were purchased from Sigma Aldrich chemicals Co. except polyethylene terephthalate (PET) waste that was collected from beverage drinking water bottles. The bottle label and other polymers contaminate were removed and PET wastes were cut into small pieces 1 mm^2, washed with water and soap, dried, and subjected to green recycling process. The corrosive sodium chloride (NaCl Shinyo Pure Chemicals, 99.99%), The gravimetric composition of iron materials employed in this study is given in Table 1.

Table 1. Composition of the steel specimens used in the present study.

Element	**C**	**Mn**	**P**	**Mo**	**Al**	**Sn**	**V**	**Nb**	**Ni**	**Cu**	**Fe**
Wight %	0.17	0.057	0.011	0.022	0.011	0.005	0.004	0.007	0.027	0.043	balance

2.2. Recycling process

PET waste was depolymerized with 1,3—diamino propane, at weight ratio of PET to Glycol (PG) 1:3 (wt% of PET: wt% of PG) using 1.0% of acetate and 1.0 % acetic acid as a co catalyst (by weight based on weight of PET). The reaction mixtures were heated under vigorous stirring in nitrogen atmosphere at temperature about 170–190 ^{o}C for 4 h and at 200 ^{o}C for 3 h. The temperature of the reaction was then lowered to 100 ^{o}C for 1 h. The mixture was allowed to cool to room temperature. at the end of the reaction, saline water was added to the reaction mixture with vigorous agitation followed by phase separation [7,24] the product, (N, N'-Bis-(3-amino-propyl)-terephthalamide) (BAPTA) was obtained in organic layer as yellowish viscous product, which have the chemical structure as showed in Fig. 1). The general diagram for the green synthesis procedure as presented in Fig. 2.

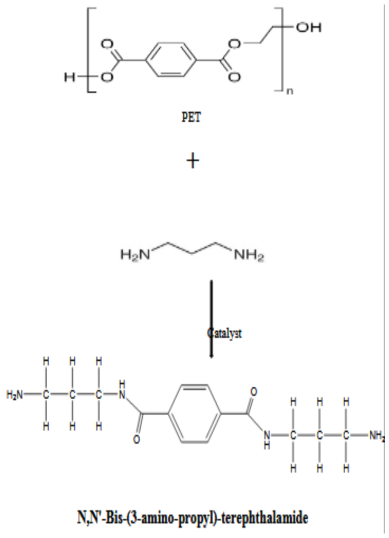

Fig. 1. the chemical scheme of the green synthesis process for the (N, N'-Bis-(3-amino-propyl)-terephthalamide), BAPTA inhibitor compound.

2.3. Elemental and Spectroscopic Analysis

Elemental and spectroscopic analyses were carried out in the Micro Analytical Center of Cairo University. The fine chemicals were purchased from Aldrich Co. The reactions were monitored using TLC and the resultant compounds were crystallized and then extra purified using a column chromatography technique.

2.4. Infrared Spectroscopic Analysis

The purified synthesized compounds were analyzed using ATI Mattson Genesis Series FTIR spectrophotometer. The samples were investigated as thin film between two KBr discs.

2.5. Nuclear Magnetic Resonance Spectroscopic Analysis

The prepared surfactants were dissolved in DMSO and analyzed using Jeol NMR spectrometer model JNM-EX (270 MHz) as another spectroscopic technique for determining the chemical structure and the HLB by comparing integral trace of different peaks.

2.6. Atomic absorption Spectroscopy (AAS)

Quantitative determinations of iron ions contents in corrosive solutions in the absence and presence of the used inhibitor was determined by atomic absorption spectroscopy(AAS). Concentration of the ferric ions passed into solution has been performed by using Varian Spectra AA 220 atomic absorption spectroscopy. In order to determine the concentrations of iron ions within corrosive solution both when the inhibitor was absent and present, we dissolved the corrosive medium by aqua regia [26].

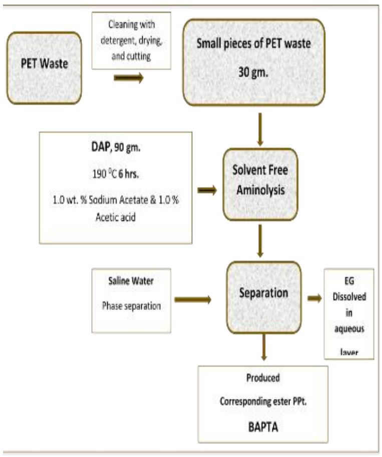

Fig. 2. The Flow Diagram of the solvent free green synthesis Processes.

2.7. Open circuit potential

The potential of steel electrode was measured against saturated calomel electrode (SCE) in 2.0 M NaCl solution in absence and presence of different concentrations of used inhibitor at 30°C. All measurements were carried out using Multi-tester until the steady-state potentials are reached.

2.8. Potentiodynamic polarization measurement

The working electrode was made from carbon steel rod that has the same composition as mentioned in point 2.1. The rod was axially embedded in araldite holder to offer an active flat disc shaped surface of an area 1 cm^{-2}. Prior to each experiment, the working electrode was polished successively with fine emery paper, rinsed with acetone, washed with double distilled water and finally dried before dipping into the electrolytic cell. A platinum wire was used as a counter electrode and a saturated calomel electrode (SCE) as the reference electrode to which all potentials are referred. The electrochemical experiments are performed using radiometer analytical, Volta master (PGZ301, DYNAMIC ELS VOLTAMMETRY). Under stirring with scan rate 1 mV/s and potential was scanned in the range of -1800 to 0 mV. Three compartment cell with a saturated calomel reference electrode (SCE) and a platinum foil auxiliary electrode was used.

3. Results and discussion

3.1. Chemistry: Synthesis of Bis-(3-hydroxy-propyl)-terephthalate, BAPTA

PET (1%w/w) and an tri molecular ratio of 1,3-di amino propane (3 %w/w) were refluxed in the presence of co catalyst consists of 1.0% sodium acetate and 1.0 % acetic acid (from the total weight of the reactants) for 6 hours at temperature (190-200 C^{o}) as mentioned before Part 2.2. [7,24]. The reaction mixture was cooled to 100 ^{O}C, then to room temperature, saline water was added to remove glycol the product residue is a yellowish viscous product, this compound was extra purified using column chromatography. Fig. 1 show the chemical structure of the product and scheme of the synthesis process, the obtained compound has the elemental analysis as given in Table 2.

Table 2. Elemental analysis of the Prepared Compound from PET waste.

Analysis	***Molecular Formula (Mol.F)***	***Molecular Weight (Mol.wt.)***	***C %***	***H %***	***N %***	***O %***
Calculated	$C_{14}H_{22}N_4O_2$	*278.5*	*60.03*	*7.89*	*20.10*	*11.5*
Found		*277.4*	*59.6*	*7.3*	*19.2*	*11.2*

The structure of the obtained compound, BAPTA was verified form their IR spectra. The spectra of BAPTA shows the presence of strong bands at ν cm^{-1}: 3421–3274 (NH2, NH), 2830-2950 (C-H methylene groups), and 1665 (C=O) $cm^{-1}$the presence of strong band at 3421cm^{-1}, in the spectra, indicates the termination of the products with amino groups. On the other hand, the band observed at 810 cm^{-1} for all depolymerized PET is assigned to –CH out-of-plane bending of p-substituted phenyl. This band confirms the presence of phenyl rings in depolymerized products. The presence of strong peaks at 1745 cm^{-1} which were assigned for C=O stretching of amide groups, indicates the incorporation of amide functional group in all depolymerized PET products. A further confirmation for the products of PET with (1,3-di di amino propane) is given by ^{1}HNMR. In this respect the spectra of BAPTA show the signals at chemical shifts 8 ppm, and 4.5 ppm, represent p-substituted phenyl group, and $COOCH_2CH_2CH_2O$-H of produced PET respectively, were observed in the spectra. The signal observed at 6.93 (s, NH). The result of investigation of both FT-IR and ^{1}HNMR indicates the presence of active amino amide groups in the product compound derived from PET waste.

3.2. Atomic Absorption Spectroscopy(AAS)

Steel corrosion is a complex process that occurs when iron is exposed to oxygen and humidity and is exacerbated by the presence of chloride ions. The deterioration of iron structures or other components can be costly to society and can be evaluated by following the properties of the corroding material [26,27]. The iron ions released into solution due to corrosion were detected directly by atomic absorption spectroscopy and their concentration was determined using a calibration curve. The corrosion of the iron samples in solution was accelerated by high salinity, lowering pH, the presence of chloride ions, and temperatures [26,27]. In the present study the ferric ions Fe^{+3} concentrations result due to corrosion of iron by the corrosive environment after 7 days, were determined by atomic absorption spectroscopy(AAS). The concentrations of the ions passed into solution has been performed by using AAS and in order to determine the concentrations of ferric ions within corrosive solution both when the green inhibitor was absent and present. The data of AAS are listed in table 3. Which show that the ferric ion Fe^{+3} concentrations in the corrosive medium were decreased by increasing inhibitor concentration. Here ferric ions concentration taken as a function of corrosion rate consequently as the ferric ions in the solution increases the corrosion rate increases and vice versa. All process is influenced by adding inhibitor. The data in table 3 indicated that, the addition of BAPTA compound inhibit the corrosion of iron in artificial marine environment and decrease the iron dissolution process (ferric ion concentration Fe^{+3}) in this environment.

Table 3. Effect inhibitor concentrations on ferric ions concentrations as result of AAS technique.

Sample	**Inhibitor Concentration**	**Ferric ions concentrations [Fe^{+3}], ppm**
Blank	Free	127
BAPTA	50 ppm	41
	100 ppm	29
	150 ppm	23
	200 ppm	19
	250 ppm	18
	300 ppm	18

3.3. Open circuit Potential Measurements

The potential time curves of the carbon steel electrode in the absence and presence of different concentration of the used inhibitor at 30 ^{0}C was measured against SCE reference electrode for 60 min the obtained data are presented in Fig. 3. As seen from Fig. 3 in uninhibited

2.0M NaCl solution, the *E* value of steel tends towards more negative potentials then slightly shifted to positive direction. After that, it was kept at a nearly stable value. The same behavior appears at addition of the inhibitors but a steady state potential, which produced from addition of inhibitors shifted to more noble values with inhibitors. This shift increase with increasing inhibitor concentrations. The inhibitors raise the free corrosion potential of steel (shifted it to more noble values) compared to the blank solution. This initially indicates that the studied inhibitors act as anodic inhibitor [29-33]. In all curves the steady-state values are always more negative than the immersion potential suggesting that before the steady state condition is achieved the steel oxide film has to dissolve [29-33].

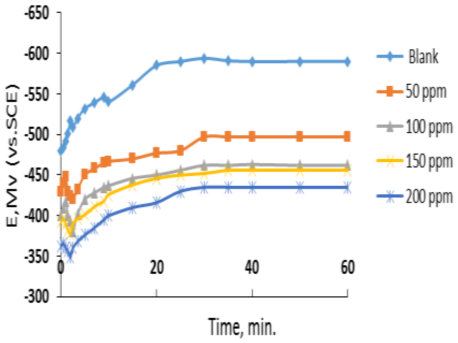

Fig. 3. Potential–time curves for carbon steel immersed in 2.0 M NaCl solution in the absence and presence of the BAPTA inhibitor compound derived from PET waste at room temperature.

3.4. Potentiodynamic Polarization Measurements

Potentiodynamic Polarization studies have been used to evaluate the corrosion inhibition efficiency of the used inhibitors by study the formation of protective film on the metal surface. The potentiodynamic polarization curves of carbon steel immersed in the test solutions are shown in figure 4. The Tafel corrosion parameters and inhibition efficiency are given in table 4. As shown from the table the inhibitors shifts the corrosion potential to less negative values versus SCE. This indicates that the anodic current reaction is controlled predominantly. The corrosion current potential values for carbon steel in 2.0 M NaCl solution decreases in the presence of inhibitors and the corrosion resistant values has increased . this indicates that a protective film formed on the metal surface. From the table 4 we can concluded that the addition of inhibitors enhance the corrosion inhibition efficiency. The shift in anodic Tafel slope (β_a) is greater than the shift in cathodic Tafel slope (β_c) in case of inhibitors system indicating that the inhibitors act as mixed inhibitors mainly anodic [30]. The inhibition efficiency and surface coverage is given by the following equations [34-38]

$$\%I.E = 1- (I_{inh} / I_{uninh}) \times 100 \qquad (1)$$

$$\theta = 1- (I_{inh} / I_{uninh}) \qquad (2)$$

where: I_{uninh} and I_{inh} are the corrosion current densities in the absence and presence of inhibitor respectively.

Table 4. Polarization data for steel electrode in 2.0 M NaCl solution with various concentrations of the BAPTA inhibitor compound derived from PET waste. at room temperature.

Inhibitors	**Conc., ppm**	**-E_{corr} mV (SCE)**	**I_{corr} mA cm^{-2}**	**β_c $mVdec^{-1}$**	**β_a $mVdec^{-1}$**	**% I.E**	**θ**
Blank	-----	481	1.2	103	99	------	-----
	50	523	0.20	127	101	83.3	0.833
	100	531	0.15	132	117	87.5	0.875
	150	539	0.10	139	123	91.7	0.917
	200	541	0.089	143	129	92.6	92.6

3.4. Adsorption Isotherm

The adsorption process depends mainly on the charge and the nature of the metal surface, electronic characteristics of the metal surface, adsorption of solvent and other ionic species, temperature of corrosion reaction, the electrochemical potential at solution-interface. The presence of the electro repelling or electro donating groups in the derivatives. Tafel polarization is used to find out the values of surface coverage (θ) at different drug concentrations to explain the best fit isotherm for the adsorption process. The results are best fitted by Langmuir adsorption isotherm according to the following equation[32]:

$$C_i/\theta = 1/K_{ads} + C_i \qquad (3)$$

where, K and C are the equilibrium constant of adsorption process and the drug concentration, respectively [32].

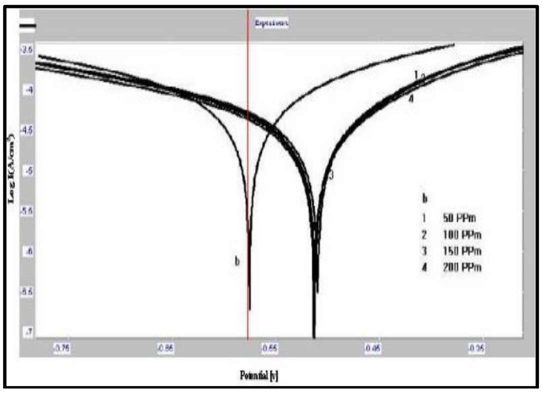

Fig. 4. Potentidynamic polarization curves of iron in 2.0 M NaCl solution containing different concentrations of the BAPTA inhibitor compound derived from PET waste at room temperature.

Plotting C/θ versus C gave straight line, as shown in Fig. 5. The straight line with approximately unit slope value has an intercept of 1/K. The standard free energy of adsorption $\Delta G°_{ads}$ is calculated using the equation:

$$K_{ads} = 1/55.5 \exp(-\Delta G_{ads} / RT) \qquad (4)$$

where K_{ads} is the adsorption equilibrium constant, 55.5 is the dose of water in the bulk of solution in mole/liter, T is the absolute temperature and R is the gas constant. The calculated value of ΔG_{ads} is -34.25 kJ/mol. The negative value of $\Delta G°_{ads}$ indicates that the adsorption process of drug molecules on the metal surface is spontaneous [32]. On the other hand, the obtained value is less than the threshold value of -40 kJ/mol required for chemical adsorption which indicates that the mechanism of adsorption is a physical [30-32]. It is generally accepted that the studied expired drug, compound inhibit the corrosion process by adsorbing at the metal/solution interface. In addition, it is believed that the formation of a solid organic molecule complex with the metal atom has received considerable attention [31,32].

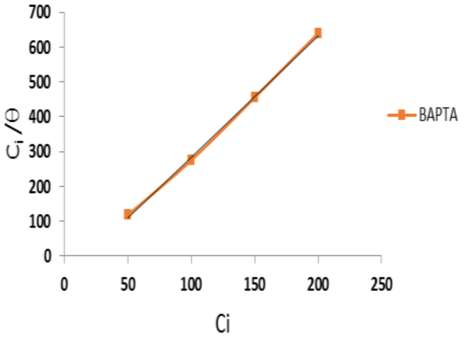

Fig. 5. Langmuir adsorption isotherm for carbon steel in 2.0 M NaCl inhibited by the used inhibitor derived from PET plastic waste.

4. Conclusions

From the obtained results we can conclude the following points:

PET waste could be depolymerized using propylene glycol in the presence of manganese dioxide catalyst to give N,N'-Bis-(3-amino-propyl)-terephthalamide, BAPTA, the process is solvent free efficient green process. BAPTA compound act as mixed inhibitor mainly anodic for carbon steel in 2.0 M NaCl medium.

The corrosion inhibition efficiency increase with inhibitor concentration and the inhibition due to adsorption of inhibitors molecules on the steel surface which obeys Langmuir adsorption isotherm model. The corrosion inhibition efficiency increase by increasing BAPTA inhibitor concentrations reach to 92.6 % at 200 ppm. As soon as the Fe+3 ion decreases.

The data of AAS show that the ferric ion Fe+3 concentrations were decreased by increasing inhibitor concentrations. Potentodynamic measurements results indicate that the values of Ecorr. change slowly to negative values indicating that used inhibitors are of mixed type inhibitors mainly anodic.

All the used techniques are in good agreement to each other (±2 %) and shows that the BAPTA inhibitor compound derived from PET waste acts as green corrosion inhibitors for steel in artificial marine environment.

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