

# Manufacturing of Corrosion Inhibitors and Flow Improvers from Recycling of Waste at Hail City, Saudi Arabia: Physicochemical and Electrochemical Studies on Steel in Petroleum Industries

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Cite This: *ACS Omega* 2023, 8, 24279–24290



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**ABSTRACT:** The aim of the present research paper is to convert daily waste materials generated by human activity at Hail city into useful petroleum additives. In this respect, novel multifunctional corrosion inhibitors working as inhibitors and flow improvers for crude oil were prepared. Polyethylene terephthalate (PET) plastic waste is used in the production of corrosion inhibitors and flow improvers for petroleum crude oil. A multifunctional corrosion inhibitor for the SABIC carbon steel in corrosive seawater for application in the petroleum industry was manufactured using PET waste that was gathered, cleaned, and used as starting materials. The PET green recycling method takes place via the Abdel-Hameed green recycling reported method. In the first step, PET waste was reacted without a solvent with a diamine to form the diamino derivative of phthalic acid amide (PETAA), which was characterized by FT-IR,  $^1\text{H}$ NMR, and elemental analysis. In the presence of a catalyst, the used recycling method is a solvent-free green recycling process that is environmentally friendly. Chemical and electrochemical measurements were performed, and the effects of concentration and temperature were studied. The inhibition efficiency was found to increase with concentration. A maximum inhibition of 97% was obtained using 4000 ppm from the prepared PETAA inhibitor, while the efficiency decreased with temperature. Potentiodynamic polarization (PDP) data indicates the mixed-type nature of the used inhibitor. According to potentiodynamic polarization data, the inhibitor boosts polarization resistance and inhibition performance by adsorbing on the metal/electrolyte interface. The data from electrochemical impedance spectroscopy (EIS) show that the charge-transfer mechanism is the primary governing factor in the steel dissolution process. The size of the semicircle grows in direct proportion to the concentration of the inhibitor. The prepared additive acts as a flow improver (viscosity improvers and pour point depressants) for waxy crude oil, indicating that it can be used in the manufacturing of multifunctional inhibitors in the petroleum industry. The depression in the pour point temperatures depended on the concentration and composition of the additive prepared from the plastic waste collected from Hail city.



## 1. INTRODUCTION

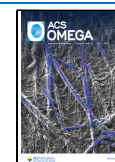
Hail city is a civilized city with a long history and an important strategic location in the Kingdom of Saudi Arabia's north-western center. It benefits from urbanization and progress at all levels of the environment, industry, commerce, and services. It is regarded as one of the most significant geographical locations because it is the center of commerce, movement, and transportation and industrial axes. Sustainable development of the environment and availability of local products based on the local and national industries are one of the greatest competitive features of a country and a region. With the increase of civil civilization, the daily production of plastic materials increases, as well as the daily consumption of plastic

materials, which may result in the accumulation of plastic waste on a daily basis. Plastic bottle production in the world exceeds 200 million tons per year, accounting for approximately 19% of total solid waste accumulated in the environment.<sup>1–5</sup> Many researchers have worked hard to convert solid and plastic waste into useful chemicals for

**Received:** February 28, 2023

**Accepted:** June 14, 2023

**Published:** June 27, 2023



industrial purposes.<sup>1–12</sup> Plastic waste was recycled, and its products were used as flow improvers for local petroleum raw materials and as metal corrosion inhibitors in the petroleum industry. Metal corrosion in the petroleum industry is a constant problem that occurs in oil depots, pipelines, boilers, and tankers due to the fact that the raw materials and petroleum products contain corrosive ions such as sulfates, chlorides, and nitrates in addition to containing a percentage of water that is often mixed with petroleum crude. There are many methods used to protect metals from corrosion, such as paints, coatings, galvanizing, anodic and cathodic protection, as well as corrosion inhibitors. Corrosion inhibitors are substances that are added in very small quantities for limiting the corrosion process by either inhibiting the anode or cathode reactions or both.<sup>10–16</sup> Corrosion inhibitors contain effective functional groups that enable them to be adsorbed on the metal surface such as hydroxyl and amine groups, as well as oxygen, nitrogen, sulfur, and heterocyclic materials. Organic and inorganic materials have been used as metal corrosion inhibitors in acidic, alkaline, and saline corrosive media and marine environments.<sup>15–30</sup> Environmental waste is recycled, and the results of recycling are used as metal corrosion inhibitors, whereas the waste of PET and PEAA plastics is recycled, and the recycled products are used as corrosion inhibitors for carbon steel used in the petroleum industry in acidic media and marine environments.<sup>1–12</sup> The surfactant materials prepared from plastic waste had an effective activity in adsorption on the surface of metals and protection of metals from corrosion,<sup>1–10</sup> as well as expired drugs were used as inhibitors.<sup>16–30</sup> Polymeric materials and heterocyclic compounds<sup>31–37</sup> were used to protect metals from corrosion. Corrosion of metals is a pervasive process related to the industry and to the daily use of metals in the environment. The corrosion protection costs for industrialized and wealthy countries are roughly 7% of their overall income. Multifunctional corrosion inhibitors in the petroleum industry are considered an important target from the viewpoint of the industry and the environment, whereby petroleum additives are provided that act as inhibitors and scavengers of hydrogen sulfide gas and petroleum pesticides, as well as improvers for the flow of petroleum crude oil. Hence, these functions are combined in one additive. Multifunctional inhibitors have been used on a small scale as indicated by previous literature studies. This work aims at several collective goals represented in recycling the daily plastic waste collected in the Hail region and converting it into petroleum additives that act as metal corrosion inhibitors and improvers for the flow of petroleum ores.

The use of plastic waste and its modified derivatives as corrosion inhibitors can be traced back to 2006 when Reda Abdel-Hameed and colleagues created a water-soluble surfactant derived from plastic waste.<sup>1–3,38–40</sup> This surfactant was evaluated as a corrosion inhibitor for C-steel in 1 M hydrochloric acid corrosive media,<sup>38–40</sup> and Abdel-Hameed's works have attracted the attention of other scientists to this crucial research area. In the previous work, the corrosion inhibition of carbon steel (SABIC steel) used in the petroleum industry was studied using a silver nanomaterial and an expired medicinal drug inhibitor system.<sup>48</sup> Chemical, analytical, and electrochemical techniques were used for the evaluation of the synthesized nanoparticle–expired drug inhibitor system as an inhibitor for carbon steel (SABIC steel). Recently, polymeric surfactant materials have been used on a large scale for the

protection of metals and alloys in industrial fields;<sup>51–55</sup> the nonionic surfactants derived from oxadiazole and thiadiazol<sup>52</sup> were prepared, characterized, and evaluated as inhibitors for carbon steel in a hydrochloric acid medium. Chemical (weight loss) and electrochemical [pour point depressant (PPD) and EIS] techniques were utilized for testing the inhibition efficiency and studying the inhibition mechanism.<sup>52</sup> In the work reported by Abdallah et al.,<sup>52</sup> the inhibition of the nonionic surfactant derived from oxadiazole and thiadiazol reached 89%. Abdallah and co-workers in 2022 studied the effect of some polymeric surfactant materials on the protection of SABIC iron in the hydrochloric acid corrosive medium by experimental and theoretical quantum chemical studies.<sup>53</sup> Protection of carbon steel materials in the acidization of oil and gas wells was studied by Zarrouk and Abdallah et al. in 2022<sup>54</sup> using cationic gemini surfactant materials, and experimental and computational theoretical studies were performed for the interpretation of corrosion inhibition.<sup>54</sup> SAPIC iron protection was performed recently, 2022,<sup>55</sup> using tryptophan and histidine acting as a mixed inhibitor due to their adsorption on the iron surface, obeying the Langmuir isotherm model.<sup>55</sup> A series of reported works aimed to reduce environmental pollution by converting plastic waste into useful products, as well as to assess the inhibition action of plastic waste on the corrosion of some steel alloys in various aqueous media.<sup>1–11</sup> The use of plastic waste as corrosion inhibitors includes the synthesis of new compounds with new structural properties from PET in order to improve their efficiencies in the field of corrosion protection of steel. A surfactant inhibitor derived from waste has numerous advantages, including high inhibition efficiency, low cost, low toxicity, availability, renewability, and ease of production.<sup>1–11</sup> R. S. Abdel-Hameed reviewed the scientific efforts in the field of using plastic waste as green corrosion inhibitors toward metals in 2016 when he collected and summarized his studies and other scientist's studies in a review article published in the Indian Journal of Materials Science.<sup>3</sup> Atta et al., 2007,<sup>57</sup> studied the corrosion inhibition effect of two oligomers derived from the glycolysis products of poly(ethylene terephthalate) (PET) with diethanol and triethanol amines, which was measured from weight loss and electrochemical polarization techniques. synthesized a new water-soluble thiol derivative (GT-SH) from PET. In 2017, Reda. S. Abdel-Hameed introduced the Reda Abdel-Hameed green method,<sup>39</sup> which is a novel idea that involved green recycling of plastic waste via a solvent-free green method and using the modified products obtained from the green synthesis process as corrosion inhibitors for carbon steel in an acidic medium using chemical, electrochemical, adsorption, and thermodynamic studies.<sup>39</sup> In 2021, Reda Abdel-Hameed and co-workers studied the green synthesis and characterization of nonionic surfactants derived from PET and calculated their thermodynamic parameters for micellization and adsorption at different temperatures in order to use the higher adsorption-prepared surfactants as corrosion inhibitors for metals and alloys in the future research projects.<sup>5</sup> In 2020, Tuama and co-workers studied the corrosion inhibition of C-steel material in acidic media using modified PET waste;<sup>56</sup> From the literature, the area of using of the recycling product of PET plastic waste still promises as an attractive field for many researchers due to the variety of the products, which depends on the different methods used for the recycling process. In addition, plastic waste is considered an inexpensive and safe corrosion inhibitor for metals and alloys in different corrosive media in the

**Table 1. Gravimetric Composition of the Steels (SABIC Carbon Steel in Saudi Arabia) Employed in the Present Study**

element	Mn	V	Si	Ni	Cr	Al	Zn	S	P	C	Fe
composition: weight (%)	1.27	0.046	0.035	0.011	0.011	0.046	0.315	0.008	0.202	0.09	the rest

scientific and applied fields as well. This work is concerned with the recycling of used PET waste bottles and the use of recycling products in the preparation of surface-active materials that act as corrosion inhibitors for carbon steel (SABIC steel) used in the petroleum industry and as flow improvers for some local petroleum crude oil. Chemical, physicochemical, and electrochemical techniques were used to evaluate the prepared materials as multifunctional inhibitors. The effect of concentration and temperature on the efficiency of inhibitors was studied, as well as the adsorption curves.

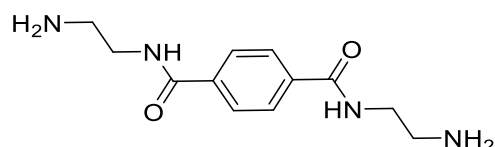
## 2. EXPERIMENTAL SECTION

**2.1. Materials and Test Solutions.** Plastic waste of the type PET, as used in water bottles, was collected from beverage bottles in Hail city, Saudi Arabia. The bottle label and other contaminants were removed, and the bottles were washed with detergent and water, dried, and cut into small pieces (approximately 1 cm<sup>2</sup> in size) to be used as starting raw materials in the green recycling process. Sigma-Aldrich Chemicals Co. provided all of the chemicals used in this experiment. The corrosive solution used in this study is 3.5% sodium chloride (NaCl Shinyo Pure Chemicals, 99.99%); this solution is referred to as artificial seawater. The concentration 3.5% NaCl is similar to the concentration of NaCl in seawater. The carbon steel samples are the same steel samples studied in previous works.<sup>12,48</sup> In a previous study, it was used as a reference sample and acted as a working electrode, and its composition is represented in Table 1. Table 1 shows the gravimetric composition of the steel materials used in this study [type-X60 carbon steel rods (CSX60 and SABIC), Kingdom of Saudi Arabia] with a composition similar to the steel used in the petroleum industry for the SABIC carbon steel in Saudi Arabia. It has a comparable composition to the carbon steel used in automobiles, boilers, and water tanks.

The test solution from the prepared inhibitor PETAA was prepared by dissolving the PETAA compound in artificial aqueous ethanol at a ratio of 3:1 v/v %; this dissolving ratio is constant for all the tested solutions. The constant percent of the used ethanol for the dissolution process of the inhibitor indicated that the inhibition action is due to the inhibitor PETAA compound as the inhibition, which may happen due to presence of ethanol as a solvent, is neglected because of its constant ratio. This solution is used as a standard solution for all the experiments from which calculated amounts (1000–4000 ppm) were added to a 100 mL beaker containing 3.5% sodium chloride solution as a corrosive medium. Sodium chloride of 3.5% concentration is considered as artificial seawater.

**2.2. Recycling Process of PET Plastic Waste.** The PET bottles collected from Hail city, Saudi Arabia, are subjected to an eco-friendly recycling process using the solvent-free green method in the presence of 1.0% of the total mass of sodium acetate as a catalyst. PET waste was depolymerized with ethylene diamine using 1.0% (W %) sodium acetate as a catalyst and 1.0% (W %) acetic acid as a co-catalyst (by weight based on the weight of PET). The reaction mixtures were heated for 4 h at a temperature of about 170–190 °C and for 3 h at 200 °C while being vigorously stirred in a nitrogen

environment. The reaction temperature was subsequently decreased to 100 °C for 1 h. It took some time for the combination to cool to room temperature. Phase separation was performed once the reaction was complete, and the product (bis-(2-amino-ethyl)-terephthalamide) (PETAA) was obtained in the organic layer as a yellowish product with the chemical structure depicted in Figure 1. Figure 2 represents the general diagram of the green synthesis method known as the Abdel-Hameed green method.<sup>38–40</sup>



*N1,N4-bis(2-aminoethyl)terephthalamide*  
**Chemical Formula:** C<sub>12</sub>H<sub>18</sub>N<sub>4</sub>O<sub>2</sub>  
*Exact Mass:* 250.14, *Molecular Weight:* 250.30  
*Elemental Analysis:* C, 57.58; H, 7.25; N, 22.38; O, 12.78

**Figure 1.** Chemical structure of the PETAA inhibitor molecule (Bis-(2-amino-ethyl)-terephthalamide) derived from plastic waste.

**2.3. Elemental and Spectroscopic Analyses.** Spectroscopic and elemental analyses were carried out at the Cairo University Micro Analytical Center. The fine chemicals were sourced from Aldrich Co. As the reactions were being monitored using TLC, the resultant compounds were crystallized and then further purified using a column chromatography procedure.

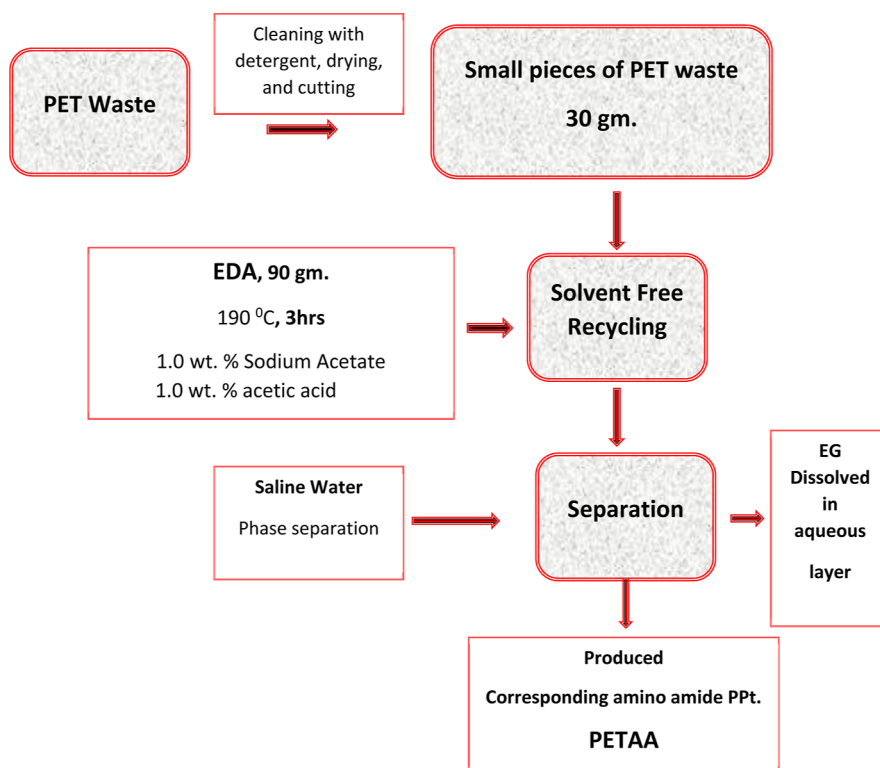
**2.4. Infrared Spectroscopic Analysis.** The pure produced chemicals were evaluated using an ATI Mattson Genesis Series FT-IR spectrophotometer. The materials' thin films were examined between two potassium bromide (KBr) discs.

**2.5. Analysis Using Nuclear Magnetic Resonance Spectroscopy.** The produced surfactants were dissolved in DMSO and spectroscopically analyzed using a Jeol NMR spectrometer model JNM-EX to determine the chemical structure and HLB by comparing the integral traces of various peaks (270 MHz).

**2.6. Pour Point Measurement.** The tested crude oils (50 mL) were heated up to 60 °C, and the pour point (PPD) additives were added at different concentrations. The solution was transferred to a bottle test tube in a water bath cooled at 48 °C.<sup>35</sup> The tube was transferred to another cooling bath cooled down to 24 °C. The tube was transferred to a cooling bath cooled down to 0 °C; then, the pour point temperature (PPD) was measured at a temperature of 48 °C, ASTM D 97–93.

**2.7. Rheological Measurements.** A Haake viscometer model Rotovisco RV12 was utilized to measure the dynamic viscosity of untreated and treated crude oils with some selected PPDs at different concentrations (from 500 to 3000 ppm) and at different temperatures above and below the pour point (PP) of crude oils ranging from 36 to 15 °C. The yield point and the apparent viscosity values were determined.<sup>35</sup>

**2.8. Open-Circuit Potential Measurement.** The potential of a carbon steel electrode in artificial seawater made of a



**Figure 2.** Flow diagram of the solvent-free green synthesis process known as the Reda Abdel-Hameed green recycling flow diagram (solvent-free green recycling process).

3.5% NaCl solution was measured at 30 °C in both the absence and the presence of different amounts of the used inhibitor. The multimeter was utilized for all measurements until the steady-state potentials were attained.

### 2.9. Potentiodynamic Polarization Measurements.

For all electrochemical measurements, an analytical radiometer with a Volta master is used in the tests (PGZ301, DYNAMIC ELS VOLTAMMETRY). The counter electrode is made of a platinum wire. Calomel is used as a reference electrode because it is uniform and is the electrode to which all potential is attributed. A steel cylindrical electrode with 1 cm<sup>2</sup> area is used as another working electrode. Silicon carbide abrasive sheets of grades 600, 1000, 1200, 1500, and 2000 are used to abrade and clean the electrode. After cleaning with water, the electrode was degreased with ethyl alcohol, rinsed with distilled water, dried, and kept ready for use as an electrode. Each experiment was carried out in accordance with this methodology. The working electrode was immersed in the corrosive solutions for 50 min. Electrochemical scanning is carried out at a scan rate of 1 mV/s. The inhibitory efficiency percent (% I.E.) was calculated using the mathematical formula shown below.<sup>41–44</sup>

$$\% \text{ I. E.} = \left( \frac{I - I_0}{I} \right) \times 100 \quad (1)$$

where  $I$  and  $I_0$  are, respectively, the densities of the corrosion currents in the presence and absence of inhibitors. It was possible to calculate and tabulate the corrosion potential ( $E_{\text{corr}}$ ), corrosion current ( $I_{\text{corr}}$ ), and Tafel constants ( $\beta_a$  and  $\beta_c$ ) using the values of the cathodic and anodic Tafel slopes that were discovered from the Tafel plot.<sup>41–44</sup>

**2.10. Electrochemical Impedance Measurements.** A three-electrode assembly with carbon steel specimens with an exposed surface area of 1 cm<sup>2</sup> as the working electrode,

platinum wire as the counter electrode, and calomel as the reference electrode was employed in the electrochemical tests. Measurements of impedance responses were made at an open-circuit potential (OCP) (frequency range: 100 kHz to 10 mHz, 5mV amplitude). All experiments and measurements were carried out at room temperature in an agitated or still environment. Charge-transfer resistance, denoted as  $R_p$  in the Nyquist plot impedance data, was used to assess the inhibitory efficiency.<sup>25–30</sup>

Charge-transfer resistance, denoted as  $R_{ct}$  in the impedance data of the Nyquist plots, was used to compute the inhibitory efficiency as follows<sup>41–44</sup>

$$\text{I. E. } \% = \left[ \frac{R_{ct} - R_{ct0}}{R_{ct}} \right] \times 100 \quad (2)$$

where  $R_{ct}$  is the charge-transfer resistance with an inhibitor, while  $R_{ct0}$  is the charge-transfer resistance without an inhibitor.

**2.11. Weight Loss Measurement of the Corrosion Rate and Corrosion Inhibition.** The procedure is based on estimating the weight loss (WL) of a sample of the surface ( $S$ ) that has been immersed in the abrasive solution for a while ( $t$ ). The experiments are conducted at room temperature in 100 mL glass vials with a non-aerated medium. Before and following the 6 day immersion, the iron samples are cleaned with distilled water, degreased with acetone, and then dried. The following relationship<sup>41–44</sup> was used to calculate the corrosion rate  $W$

$$W = \frac{m_i - m_f}{S_t} \quad (3)$$

where  $W$  (mg cm<sup>-2</sup> h<sup>-1</sup>) is the corrosion rate,  $m_i$  (mg) and  $m_f$  (mg) are the masses before and after exposure to the test solution, respectively,  $S$  (cm<sup>2</sup>) is the surface area of the specimen, and  $t$  (h) is the immersion time. The I.E. % and the

surface coverage ( $\theta$ ), which represents the part of the metal surface covered by the inhibitor molecules, were calculated according to the following equations<sup>41–44</sup>

$$\% \text{ I. E} = \left( \frac{[W^0 - W]}{W^0} \right) \times 100 \quad (4)$$

$$\theta = ([W^0 - W]/W^0) \quad (5)$$

where  $W^0$  and  $W$  represent the corrosion rates in the absence and presence of the inhibitors, respectively.

### 3. RESULTS AND DISCUSSION

**3.1. Green Recycling: Synthesis of Bis-(2-Hydroxy-Propyl)-Terephthalate, PETAA.** Chemical recycling of plastic waste is a common practice; however, because it involves use a lot of organic solvents and other chemicals to process PET, it is not acceptable from an environmental or green perspective. Reda Abdel-Hameed became interested in finding a green and acceptable method for recycling plastic waste because chemical recycling is not environmentally acceptable. He was able to do this in 2017 when he used a green, solvent-free method to recycle plastic waste and published the findings of his research in 2017. In order to offer a focused effort and a dual solution to the two problems of plastic waste accumulation and metal corrosion, Abdel-Hameed's method did not focus just on the green recycling process.<sup>34,35</sup> He also looked at the application of green recycling products as corrosion inhibitors for steel. Figure 2 shows the environmentally friendly recycling process developed by Reda Abdel-Hameed for PET waste. In the current study, PET (1% w/w) and a trimolecular ratio of 1,2-diamino ethane (3% w/w) were refluxed for 6 h at a temperature of 190–200 °C as indicated in the **Recycling Process of PET Plastic Waste** section. The used catalyst and a co-catalyst were made up of 1.0% sodium acetate and 1.0% acetic acid (from the total weight of the reactants).<sup>38–40</sup> Saline water was added to the reaction mixture after it had been cooled to 100 °C and brought to room temperature in order to remove the glycol. The result was a yellowish, viscous residue, which was further purified using column chromatography. The product compound was characterized by FT-IR, <sup>1</sup>HNMR, and elemental analysis (listed in Table 2). Figure 1 depicts the product's

**Table 2. Resulting Compound's Elemental Composition for the Prepared Additive PETAA Derived from Recycling of PET Plastic Waste**

analysis	molecular formula (mol-f)	molecular weight (mol-wt.)	C %	H %	N %	O %
calculated	C <sub>12</sub> H <sub>18</sub> N <sub>4</sub> O <sub>2</sub>	250.4	54.6	7.2	22.4	22.2
Found		294.6	53.8	6.9	20.8	21.6

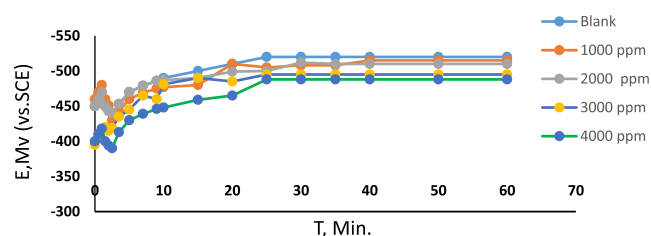
chemical makeup and the synthesis method in a diagram form.<sup>38–40</sup> The elemental analysis listed in Table 2 agrees well with the suggested structure drawn in Figure 1.

The resulting compound's IR spectra were used to confirm the structure of PETAA. Strong bands can be seen in the spectra of PETAA at  $\nu$  cm<sup>-1</sup>, including 3421–3274 (NH<sub>2</sub> and NH), 2830–2950 (C–H methylene groups), and 1665 (C=O) cm<sup>-1</sup>. The products' end with amino groups is indicated by the appearance of a prominent band at 3421 cm<sup>-1</sup> in the spectra. On the other hand, the band seen at 810 cm<sup>-1</sup> for all

depolymerized PET is attributed to p-substituted phenyl's –CH out-of-plane bending. This band verifies that the depolymerized materials include phenyl rings. Strong peaks at 1745 cm<sup>-1</sup>, which were attributed to amide group C=O stretching, show that all depolymerized PET products contain the amide functional group.

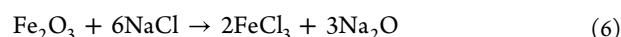
<sup>1</sup>HNMR provides additional support to the products of PET with 1,2-diamino ethane (ED). In this regard, the PETAA spectra display signals at chemical shifts of 8 ppm and 4.5 ppm, respectively, which stand in for the p-substituted phenyl group and COOCH<sub>2</sub>CH<sub>2</sub>O–H of the generated PET. The 6.93 signal was seen (s, NH). The product compound made from PET waste contains active amino amide groups, according to the results of FT-IR and <sup>1</sup>HNMR analysis.

**3.2. OCP Measurements.** Figure 3 shows the potential for corrosion of carbon steel electrodes in simulated seawater with



**Figure 3.** Potential–time curves of SABIC carbon steel corrosion in 3.5% NaCl with and without varying dosages of the green inhibitor prepared from plastic waste (PETAA) at 303 K.

a concentration of 3.5% NaCl as a function of immersion time in both the absence and presence of a green corrosion inhibitor manufactured from recovered PET plastic waste (PETAA). For a steel electrode immersed in a 3.5% solution of sodium chloride to simulate a marine environment, the potential obviously increases to a higher negative potential initially, resulting in a brief step.<sup>41–44</sup> The following chemical equation states that



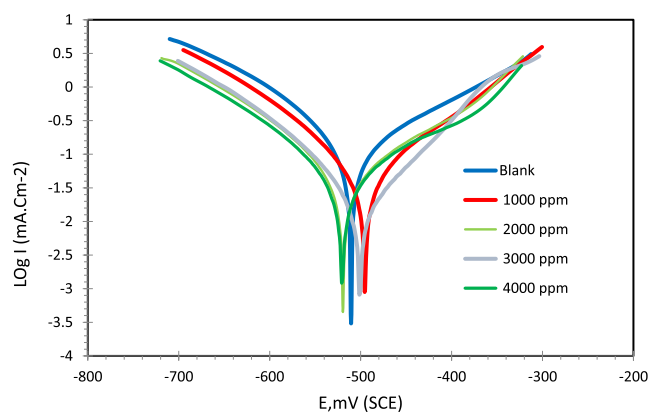
A fresh oxide layer then develops within the solution, causing the potential to change towards a noble direction until a steady-state potential is attained. The cathodic process is reduced when inhibitor molecules are added to the aggressive medium, which causes a negative shift in the OCP.<sup>41–44</sup>

### 3.3. Potentiodynamic Polarization Measurements.

Using potentiodynamic polarization (PDP) curves, the corrosion inhibition process factors that alter the anodic/cathodic process are analyzed. The estimated parameters are presented in Table 3. It has been demonstrated that the utilized green inhibitors are extremely biocompatible, nontoxic, and capable of forming a surface coating on steel. By slowing down the inhibitors, they can affect the progression of corrosion. At the intersection of the anodic and cathodic curves, the corrosion current density and potential are determined. When compared to blank,  $E_{\text{corr}}$  moves toward the value in the presence of an inhibitor, but no observable difference has been observed (Figure 4). As a result, the inhibitor made from PET plastic waste (PETAA) mostly exerts anodic effects and serves as an anodic inhibitor. As the inhibitor concentrations increased, the  $I_{\text{corr}}$  values decreased, proving that the presence of inhibitors results in the formation of a shield on the carbon steel surface that impedes

**Table 3. Potentiodynamic Properties of SABIC Carbon Steel Corrosion at 303 K in 3% NaCl with and without Varying Dosages of the Green Inhibitor Prepared from Plastic Waste (PETAA) at 303 K**

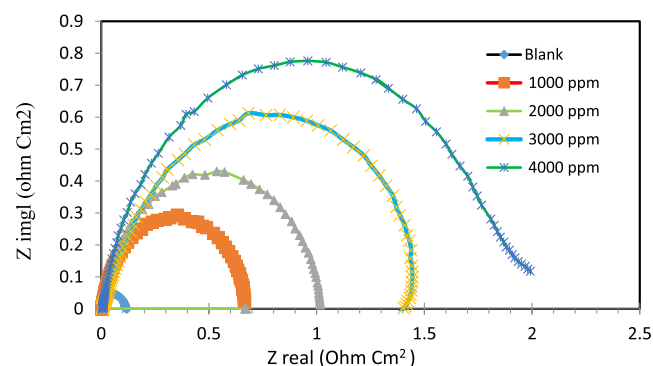
solution type	inhibitor prepared from plastic waste (PETAA) concentration ppm	$I_{\text{corr.}}$ mA cm <sup>-2</sup>	$-E_{\text{corr.}}$ (mV) (SCE)	$(\beta_a)$ (mV dec <sup>-1</sup> )	$(\beta_c)$ (mV dec <sup>-1</sup> )	$\theta$	% I.E.
blank solution	free	2.9	515	113	134		
green inhibitor prepared from plastic waste (PETAA)	1000	0.24	488	132	151	0.917	91.7
	2000	0.16	495	153	168	0.945	94.5
	3000	0.13	510	160	171	0.955	95.5
	4000	0.10	515	167	181	0.966	96.6

**Figure 4.** PDP curves of SABIC carbon steel corrosion in 3.5% NaCl with and without varying dosages of the green inhibitor prepared from plastic waste (PETAA) at 303 K.

electrochemical processes.<sup>41–44</sup> The examination of the corrosion functions in Table 3 revealed that the values of  $\beta_a$  and  $\beta_c$  remain relatively stable with increasing inhibitor concentrations. For  $\beta_a$  and  $\beta_c$ , the shift is approximately 54 and 47 mV, respectively. This shows that the inhibitor being employed appears to affect both the cathodic H<sub>2</sub> evolution and the anodic steel corrosion reactions and that there is also no difference in the value of  $E_{\text{corr.}}$ , changing by only about 94 mV. These findings support the hypothesis that the inhibitor acted as a mixed inhibitor, primarily cathodic [ $\beta_c > \beta_a$ ].<sup>51</sup> The % I.E. values rise as the  $I_{\text{corr.}}$  values decline. At 4000 ppm of the used PETAA inhibitor, the I.E. % increased to 96.6%. This reveals that the tested PETAA inhibitor was effective at preventing corrosion of carbon steel in solutions containing 3.5% NaCl as an artificial seawater.

**3.4. Electrochemical Impedance Spectroscopy.** Electrochemical impedance spectroscopy (EIS) is an extremely precise method for investigating corrosion and inhibitory mechanisms. EIS with and without an inhibitor was used to examine corrosion inhibition at varying concentrations of amino amide derived from PET plastic waste (PETAA), a

green inhibitor. Table 4 presents the findings for the impedance diagram characteristic parameters. The values of protection efficacy (I.E. %) listed in Table 4 were obtained using the equation 2 mentioned in the experimental part (Electrochemical Impedance Measurements). It is discovered that the correlation between corrosion rate and  $R_{\text{ct}}$ , a measure of electron transmission across a contact, is inverse. The electrochemical impedance characteristics were calculated using a semicircle fitting technique. In Nyquist plots, a single time constant is represented as a perfect semicircle. Impedance tests reveal details regarding the properties of the inhibition process, surface resistance, capacitance, and a substance's capacity to prevent corrosion. Deviations from completely circular forms are attributed to frequency dispersion of interfacial impedance, which is associated with non-homogeneity of the surface and metal roughness.<sup>41–44</sup> A pattern emerged from our investigation, although it was not a whole semicircle. This could be because there is an inhibitor completely deflecting focus away from the semicircle. The Nyquist plot's single semicircles move in lockstep with the impedance of the x-actual axis (Figure 5). The impedances

**Figure 5.** Shows the Nyquist plots for SABIC carbon steel corrosion in 3.5% NaCl with and without varying dosages of the green inhibitor prepared from plastic waste (PETAA) at 303 K.**Table 4. Data of the EIS Properties of the Corrosion SABIC Carbon Steel Corrosion at 303 K in 3.5% NaCl with and without Varying Dosages of the Green Inhibitor Prepared from Plastic Waste (PETAA) as Green Inhibitor at 303 K**

solution type	inhibitor prepared from plastic waste (PETAA). Concentration ppm	$(R_s)$ ( $\Omega/\text{cm}^2$ )	$(R_{\text{ct}})$ ( $\Omega/\text{cm}^2$ )	$(C_{\text{dl}})$ ( $\mu\text{F cm}^{-2}$ )	$\theta$	% I.E.
blank solution	free	0.015	0.1	31.847		
inhibitor Green inhibitor Prepared from plastic waste (PETAA)	1000	0.017	0.7	0.909	0.857	85.71
	2000	0.02	1	0.398	0.900	90.0
	3000	0.021	1.4	0.189	0.926	92.85
	4000	0.021	2.2	0.090	0.955	95.45

**Table 5. Effect of Concentration on the Corrosion Parameters of Corrosion SABIC Carbon Steel Corrosion in 3.5% NaCl with and without Varying Dosages of the Green Inhibitor Prepared from Plastic Waste (PETAA) as a Green Inhibitor Data Obtained From Weight Loss Measurements at Different Temperatures**

inhibitor Concentrations (ppm)	333 K		323 K		313 K		303 K	
	I.E. %	$\theta$	I.E. %	$\theta$	I.E. %	$\theta$	I.E. %	$\theta$
blank								
1000	87.6	0.876	89.6	0.896	90.7	0.907	93.8	0.938
2000	89.8	0.898	91.3	0.913	92.7	0.927	94.6	0.946
3000	90.9	0.909	92.2	0.922	93.6	0.936	96.0	96.0
4000	91.7	0.917	92.9	0.929	94.8	0.948	97.1	97.1

increased as the inhibitor concentrations increased. The impedance profiles also remained the same at all concentrations. Inhibitors can change the course of corrosion by slowing it down. Furthermore, the rate-controlling mechanism can be identified as a reaction-controlled mechanism as a result of the inhibitor corrosion.<sup>41–44</sup> As the inhibitor concentrations grow, the  $R_{ct}$  values go up. As a result, the majority of the corrosion reaction is managed by the charge-transfer mechanism. Due to the inhibitor's enhanced surface coverage, a rise in inhibitor concentration also causes a decrease in  $C_{dl}$  values. An improvement in inhibitory effectiveness is to be blamed for this. A lower local dielectric constant and/or a thicker electric double layer on the surface of carbon steel may be responsible for the drop in  $C_{dl}$  values.<sup>41–44</sup> The decline in  $C_{dl}$  values could be explained by a lower local dielectric constant and/or a thicker electric double layer on the carbon steel surface.<sup>41–44</sup> Figure 5 shows that the size of the semicircle rises as the additive amount is increased. The shape of the resulting spectra always provides a clue as to the process, which is driven by charge-transfer resistance and is unaffected by the additive amount. The values of  $R_{ct}$  in Table 4 increase as the doses of the PETAA inhibitor are increased, and this increase may be caused by the development of an adhering coating on the surface of the steel. This is due to a decrease in charge transfer between the steel surface and the corrosive medium caused by the addition of PETAA inhibitor molecules, which promotes the adsorption of PETAA's amido amide functional groups across the carbon steel surface.<sup>51–55</sup> In addition, the  $C_{dl}$  values drop with increasing PETA inhibitor dosages, which typically happens when the dielectric constant falls. This is explained by the inhibitor's elements adhering to the steel/electrolyte interface and shielding the steel from the hostile medium. The  $C_{dl}$  values are determined from the following relation

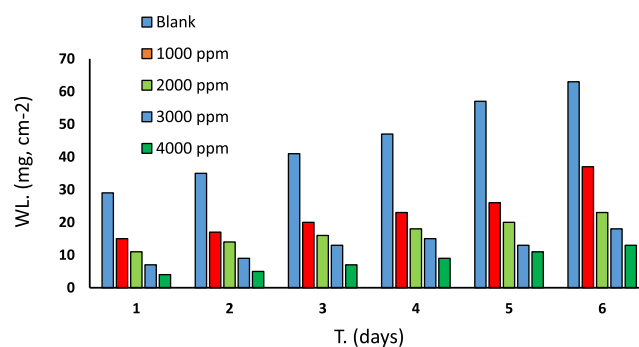
$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (7)$$

where  $f_{max}$  is the maximum frequency. The corrosion results in Table 4 demonstrate that the protection effectiveness increases with increasing inhibitor concentration (PETAA). These outcomes demonstrate the researched PETAA inhibitor's potential as a steel inhibitor in 3.5% NaCl as an artificial seawater.

**3.5. Weight Loss Measurements. 3.5.1. Concentrations' Effect.** After 6 days of immersion in a corrosive environment of 3.5% sodium chloride, the corrosion rate and corrosion inhibition efficiency percent (% I.E.) of the prepared inhibitor for various amounts of PETAA inhibitor were determined using gravimetric techniques. The corrosion rate (W) and corrosion inhibition efficiency (I.E. %) values are shown in

Table 5. According to gravimetric results, adding a prepared green inhibitor (PETAA) reduces weight loss and, as a result, corrosion rate and inhibition efficiency. This behavior was also made to occur more frequently by raising the PETAA inhibitor concentrations. It is evident that the effectiveness of the inhibition rises with the inhibitor concentration, reaching 97% at 4000 ppm. According to equations 3 and 4 described in the Experimental Section part, the inhibitory efficiency percentage I.E. and the surface coverage ( $\theta$ ), which reflects the portion of the metal surface covered by the inhibitor molecules, were computed. Table 5 shows that the values of inhibition efficiency rise with increasing inhibitor concentrations.

Figure 6 shows that the weight loss of carbon steel samples is decreased by increasing the amounts of the prepared PETAA

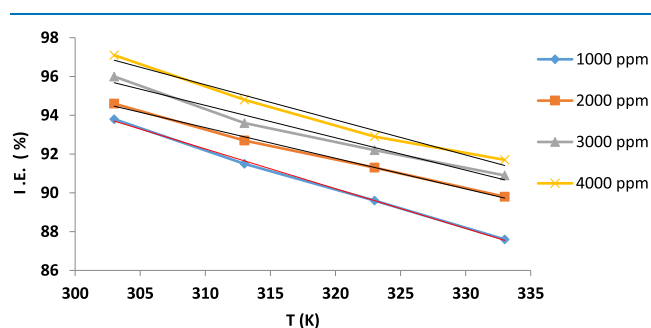


**Figure 6.** Weight loss of steel over time in 3.5% NaCl in both the presence and absence of a prepared PETAA inhibitor acting as a green SAPIC steel corrosion inhibitor.

inhibitors. This demonstrated that the presence of these compounds reduced the dissolution of iron in 3.5% NaCl solution, proving that these compounds function as inhibitors. In both unconstrained and inhibited 3% NaCl, the rate of weight loss over time is linear. This shows how there are no insoluble surface coatings present during corrosion. The inhibitors in this instance are first adsorbed into the metal surface and then prevent corrosion by either simply blocking the anodic and cathodic reaction sites or by changing the mechanism of the anodic and cathodic partial processes.<sup>36–44</sup> The maximal inhibitory efficiency utilizing 4000 ppm of the produced inhibitor is 97%, which is larger than the efficiency obtained in the cited references (previous research).<sup>51–58</sup> This observation may be the result of a chemical bond created between the amide linkage and the surface of the steel in a chloride environment. Hence, chemical adsorption is possible.

**3.5.2. Effect of Temperature.** Gravimetric (weight loss) measurements were made at various temperatures, such as 303, 313, and 323, at 333 K to clarify the mechanism of inhibition and identify the kinetic parameters of the corrosion process.

Figure 7 shows graphically how temperature affects the percentage of steel that is inhibited in the presence of the



**Figure 7.** Data from weight loss and the effect of temperature on the percent I.E. for SAPIC carbon steel in 3.5% NaCl in the presence of various concentrations of the prepared PETAA inhibitor acting as the green inhibitor.

inhibitor. It is clear that the percent I.E. values decreased as the temperature rose, indicating that the protective film of these compounds formed on the surface of the steel is less stable at higher temperatures. This may be because higher temperatures cause some molecules that have been adsorbed to desorb from the steel's surface, exposing more of the metal to the acidic environment.<sup>41–45</sup> Each experiment was carried out three times, and the recorded data was averaged with a ( $\pm 0.1$ ) error.

**3.6. Adsorption Isotherm Model.** The primary factors influencing the adsorption process are the charge and type of the metal surface, its electronic properties, the adsorption of solvent and other ionic species, the temperature at which corrosion occurs, and the electrochemical potential at the solution interface, also the derivatives' inclusion of electro-repelling or electrodonating groups. To determine the values of surface coverage ( $\theta$ ) at various drug concentrations and to explain the best-fit isotherm for the adsorption process, the adsorption isotherm was found to follow the Langmuir isotherm on the bases of fitting different isotherms and studying the best fit. The slope of the isotherm in the case of Langmuir is nearly equal unity, and the adsorption of the inhibitor is a monolayer. In addition, a physicochemical adsorption mechanism was suggested.<sup>51–58</sup>

Weight loss data was employed. According to the following equation,<sup>41–44</sup> the Langmuir adsorption isotherm best fits the results

$$C_i/\theta = 1/K_{\text{ads}} + C_i \quad (8)$$

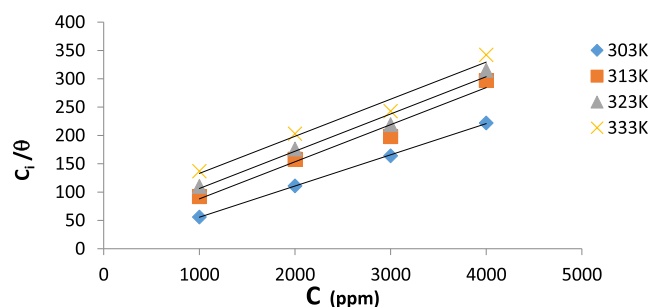
where  $K$  and  $C$  are the equilibrium constant of the adsorption process and the drug concentration, respectively.<sup>48–55</sup> The large values of the calculated ( $K_{\text{ads}}$ ) indicate the stronger adsorption of the inhibitor on the metal surface.<sup>51–55</sup>

Plotting  $C/\theta$  versus  $C$  gave a straight line, as shown in Figure 8. The straight line with approximately a unit slope value has an intercept of  $1/K$ . The standard free energy of adsorption  $\Delta G_{\text{ads}}^{\circ}$  is calculated using the equation

$$K_{\text{ads}} = 1/55.5 \exp(-\Delta G_{\text{ads}}/RT) \quad (9)$$

where  $K_{\text{ads}}$  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  $\Delta G_{\text{ads}}$  is  $-34.25$  kJ/mol.

The absence of a positive result for  $\Delta G_{\text{ads}}^{\circ}$  denotes spontaneous inhibitor molecule adsorption on the metal



**Figure 8.** Langmuir adsorption isotherm model for SAPIC carbon steel in 3.5% NaCl in the presence of various concentrations of the prepared PETAA inhibitor acting as the green inhibitor at different temperatures.

surface.<sup>41–44</sup> The obtained value, on the other hand, is lower than the  $-40$  kJ/mol threshold value needed for chemical adsorption, which suggests that the process of adsorption is physical. The absence of a positive result for  $\Delta G_{\text{ads}}^{\circ}$  denotes spontaneous drug molecule adsorption on the metal surface.<sup>48–50</sup> It is widely acknowledged that the investigated compound PETAA inhibitor derived from PET plastic waste inhibits corrosion by adsorption and adhering at the metal/solution contact. Additionally, it has thought that the development of a solid organic molecule complex with the metal atom has drawn a lot of interest.<sup>48–50</sup>

**3.7. Pour Point Measurement.** **3.7.1. Effect of Additives on Pour Point Temperatures of the Tested Crude Oils.** Nucleation, adsorption, co-crystallization, and improved waxy solubility are a few of the proposed methods by which additives produce smaller, more uniformly shaped wax crystals.<sup>46,47</sup> The tested crude oils' untreated and treated pour point temperatures (PPT) with additive concentrations were computed and are shown in Table 6. This table shows

**Table 6.** Pour Point Measurements (PPD) for the Green Additive Prepared from Plastic Waste (PETAA) via Green Recycling of PET

additive type	additive prepared from plastic waste (PETAA) concentration (ppm)	pour point temperature (PPT) °C
blank solution	free	27
green inhibitor prepared from plastic waste (PETAA)	1000	23
	2000	15
	3000	15
	4000	9

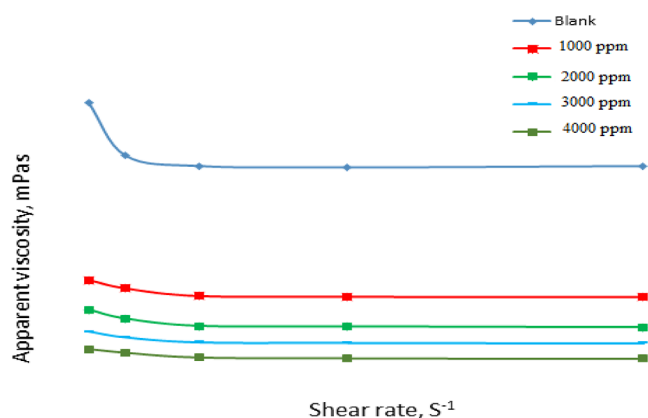
that for several crude oils, the PPT values declined when the additive concentrations rose from 1000 ppm to 4000 ppm. On the other hand, the data in Table 6 shows that depending on the makeup of crude oils, the produced amido amide made from PET plastic waste can be utilized as a pure point depressant. Pour point value reduction higher than  $21$  °C ( $\Delta P$  values) was achieved by adding 3000 and 4000 ppm of PETAA additive to the crude oils. This means that, in this concentration range, the additive co-crystallizes with paraffin, modifying their crystals. The results in Table 6 indicate that the prepared PETAA compound derived from PET plastic waste acts as a good PPD at concentrations ranging from 1000 to 4000 ppm. The same compound acts as a corrosion inhibitor of SABIC carbon steel used in the manufacturing of



petroleum pipelines at the same concentration range. This observation is a good result, proving the possibility of applying the prepared materials as a multifunctional corrosion inhibitor as it acts as a good corrosion inhibitor and a PPD as well. This means that the additive function as a flow improver and a corrosion inhibitor (multifunctional), this process saves money, chemical, energy, and waste at the same time.

**3.8. Rheological Measurements.** The rheology and its effects can be seen everywhere: Rheology is involved whenever a material flows or is deformed. Rheology is the study of the flow and deformation properties of materials. When matter flows or is deformed, three factors must be considered: the internal structure, external forces acting on the substance, and environmental conditions such as temperature. Rheometers are used to gauge a material's rheological characteristics. They gauge the measuring bob's torque and deflection angle. This indicates that the rheometer prepares a specific current that corresponds to a specified torque during a viscosity measurement.

**3.8.1. Evaluation of PEAA Grafts as a Flow Improver.** Through rheological studies at concentrations ranging from 1000 to 4000 ppm, PETAA polymer-derived PET waste was assessed for its efficacy as a flow improver for the tested crude oils. A range of temperatures, from 15 °C to 36 °C, were used to measure the viscosity–shear rate connection as a flow improver at various concentrations; the apparent viscosity–shear rate connections for the treated and untreated crude oils are presented in Figure 9 as an example. Similar behaviors



**Figure 9.** Relation between the shear rate and apparent viscosity of untreated crude oil and treated with PETAA polymers at different concentrations (1000–4000 ppm) and at 15 °C.

(curves) were observed at other examined temperatures. It was discovered that PETAA polymers with alkyl side chains ( $\text{CH}_2\text{—CH}_2$ ) linked to the amide group are effective flow-improving additives. However, their yield stress and viscosity values were lower compared to untreated crude oils even at low temperatures. On the other hand, the apparent viscosity–shear rate relationships exhibit non-Newtonian pseudo-plastic behaviors at various low concentrations and temperatures (equal to or below their pour point). However, when the shear rate rises, the dynamic viscosity falls until it reaches a limiting value. The apparent viscosity is the term used to describe this infinite shear rate viscosity.<sup>46,47</sup>

These findings suggest that PETAA polymeric additives can improve the flow characteristics of the tested crude oils by dispersing asphaltene particles around the wax crystals. Thus,

their chemical structure can be used to explain how rheological parameters behaved to decrease when the synthesized substance was added. Wax crystals in crude oil did not aggregate because of the strong polarity of nitrogen in the amino and amide groups along the additive suture.<sup>46,47</sup>

**3.9. Corrosion Inhibition Mechanism.** Adsorption of the inhibitor molecules at the metal/solution interface is the first step in the inhibitory mechanism, which controls corrosion.<sup>41–44</sup> When organic molecules are inhibited, four different forms of adsorption may occur at the metal/solution contact:<sup>48–50</sup>

- 1 The attraction between charged molecules and metals through electrostatic forces
- 2 The interaction of the metal with the unshared electron pairs in the molecule.
- 3 Interactions of Pi electrons with metal, and
- 4 A combination of the first three possibilities.

A few factors that affect an inhibitor's ability to inhibit include the number of adsorption sites and their charge density, molecule size, the heat of hydrogenation, the mechanism of interaction with the metal surface, and the production of metallic complexes.<sup>48</sup> A metal with a vacant low-energy electron orbital, an inhibitor with molecules that are very loosely linked to electrons, or a heteroatom with lone pair electrons must be present along with charged species in the majority of the solution in order for physical adsorption to occur. In addition to the electrical connection, chemical interaction also exists. Non-bonding oxygen, nitrogen, and benzene ring electrons were responsible for initiating the chemical interaction.<sup>41–44</sup> In the present work, a succinct explanation of the amido amide inhibitor compound's interaction with steel will be provided by means of an electrical interaction between the unshared electron pairs and the d-orbital from the steel material. The heteroatoms of the amide functional group are adsorbed on the surface. Moreover, through the  $\pi$  orbitals of the unsaturation from the aromatic nucleus, the alkyl chain is adsorbed on the material's surface. The molecule is therefore located in steel. The alkyl chain is positioned closer to the surface in contrast to the amido amide made from plastic waste in order to keep the medium from coming into contact with the steel. The combination of these two forms enhances the amido amide made from plastic waste's ability to stop corrosion.

## 4. CONCLUSIONS

The plastic waste bottles gathered from (Hail city) were cleaned with detergent, dried, and used as a raw material for the production of multifunctional corrosion inhibitors, which act as flow improvers for crude oil and as corrosion inhibitors. The Abdel-Hameed green synthesis reported method was used for the green synthesis of the product (*N,N'*-bis-(2-amino-propyl)-terephthalamide), PETAA, prepared from PET waste via reaction with ethylene diamine and sodium acetate as catalysts. The used technique is solvent-free effective and environmentally friendly. The prepared compound was characterized using FT-IR, <sup>1</sup>HNMR, and elemental analysis. The product PETAA was evaluated as a corrosion inhibitor for SABIC carbon steel used in the petroleum industry in 3.5% NaCl as artificial seawater. The inhibition efficiency of the PETAA inhibitor was found to increase by increasing the concentration but decreased by rising the temperature. According to the PDP data, the corrosion potential ( $E_{\text{corr}}$ )

values are continuously declining (negative values), indicating that the utilized PETAA inhibitor is a mixed-type inhibitor. The addition of inhibitor molecules to the marine environment produces a negative shift in the OCP due to the retardation of the cathodic reaction. The EIS data demonstrate that the charge-transfer mechanism is the primary governing factor in the carbon steel dissolution process. The size of the semicircle increases proportionally to the inhibitor concentration. It was found that SABIC steel corrosion prevention was caused by the adsorption and adherence of inhibitor particles (PETAA, prepared from PET waste) to the carbon steel surface, where they created an inert protective layer. The prepared PETAA compound derived from PET plastic waste acts as a good PPD at concentrations ranging from 1000–4000 ppm. The same compound acts as a corrosion inhibitor of SABIC carbon steel used in the manufacturing of petroleum pipelines at the same concentration range. These findings suggest that PETAA polymeric additives can improve the flow characteristics of the tested crude oils by dispersing asphaltene particles around the wax crystals. This means that the used additive acts as a multifunctional additive for petroleum worked as a corrosion inhibitor and as a flow improver at the same time.

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### Notes

The authors declare no competing financial interest.

## ■ ACKNOWLEDGMENTS

“This research has been funded by the Scientific Research Deanship at University of Ha'il—Saudi Arabia—through project number RD-21030”.

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