Research Article

Electrodeposited Reduced Graphene Oxide Films on Stainless Steel, Copper, and Aluminum for Corrosion Protection Enhancement

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The enhancement of corrosion protection of metals and alloys by coating with simple, low cost, and highly adhered layer is still a main goal of many workers. In this research graphite flakes converted into graphene oxide using modified Hammers method and then reduced graphene oxide was electrodeposited on stainless steel 316, copper, and aluminum for corrosion protection application in seawater at four temperatures, namely, 20, 30, 40, and 50°C. All corrosion measurements, kinetics, and thermodynamics parameters were established from Tafel plots using three-electrode potentiostat. The deposited films were examined by FTIR, Raman, XRD, SEM, and AFM techniques; they revealed high percentages of conversion to the few layers of graphene with confirmed defects.

1. Introduction

Graphene has attracted extreme interest in the industrial and scientific community since the experimental realization of graphene in 2004 [1], for many reasons; graphene has the highest electronic conductivity at room temperature; and it is only one monoatomic layer thick and almost transparent; it is durable and at the same time flexible; it is chemically inert and impermeable to most gasses. The chemical inertness and impermeability towards most gasses in combination with its strength and single atomic layer thickness make it a fascinating candidate for coating purposes, particularly for anticorrosion coatings; a new and better protecting material has been sought intensively [2, 3]. Graphene as nanosheets and nanoplatelets and functionalized graphene can be prepared by various methods such as chemical vapor deposition, chemical or mechanical exfoliation, and cleavage and annealing a single-crystal SiC [4–8]. Most of these methods need high energy requirement with low production yield. Among the preparation methods, chemical process has become a promising route to produce graphene sheets; it is simple, inexpensive, and suitable for large-scale or mass production. The process involves significant steps: graphite

oxidation, exfoliation of graphite oxide, and reduction of graphene oxide sheets [9].

Graphene-polymers composites based materials have found wide applications in corrosion protective coatings applications [10–14], and little work has been done to aid in the corrosion protection of metal and alloys using graphene layer alone due to weak adhesively on metal surfaces. This study focused on the chemical synthesis and deposit of well-adhered graphene coating to shield carbon steel for enhancing the corrosion protection. The effectiveness of the graphene-metal coating is estimated via electrochemical procedure in 3.5 wt% NaCl aqueous solution. The test serves to deduce the corrosion inhibition ability of the graphene.

2. Materials and Methods

All chemicals and materials used in this work were used as received.

2.1. Preparation of Graphene Oxide. Graphene oxide was synthesized from graphite flakes by Hummers Jr. and Offeman method [15], graphite (1.0 g) (Mesh100, Sigma-Aldrich, USA)

was added to 23 mL of concentrated sulphuric acid (BDH, "AnalaR" sp. gr. 1.84) under stirring at room temperature, then 0.5 g of sodium nitrate (BDH, "AnalaR") was added, and the mixture was cooled to 0°C. Under powerful agitation, 3.0 g of potassium permanganate (BDH, GPR) was added slowly while the temperature of the suspension was kept near 2°C. The reaction mixture was transferred to a water bath at a temperature of 35°C and stirred for 30 min. Then, 50 mL of deionized water DI ($\sim 1 \mu\text{S}\cdot\text{cm}$) was added, and the solution was stirred for 15 min at 90°C. Additional 166 mL of water was added and followed by a slow addition of 5 mL of hydrogen peroxide (30%, Seema International), turning the color of the solution from yellow to dark brown. The mixture was filtered and rinsed with 85 mL of 4% HCl aqueous solution followed by washing with 65 mL of distilled water to remove the acid, and then oxidation product was washed until the pH reached 6 and then filtered, dried, and examined by FTIR, Raman, AFM, and SEM techniques.

2.2. Deposition of Reduced Graphene Oxide. Suspension of the produced graphene, as described in step 1, was prepared by adding 0.5 g of graphene oxide powder to 100 mL of distilled water; an ultrasonic probe homogenizer (MTI, USA, 300 W) was used to mix the solution for 10 min. The GO solution was transferred into two-electrode glass cell body (25 mL), and a well cleaned and polished piece of the metal under investigation (SS316, Cu, and Al) serves as cathode, while a piece of pure platinum acts as anode, and the two electrodes were connected to a DC power supply. The deposition starts on applying several DC volts for several minutes. Each metal deposited at ranges of potentials and times and the layers were visually examined

It seemed that a mixed function of the electroreduction process, first reduction of the graphene oxide, and the second one is electrophoretic deposition (EPD) of the graphene layers on the metal surfaces. The adherence power was tested with simple plaster tape procedure according to ASTM D823-95(2012) e1.

The deposited graphene layers were characterized using XRD, FTIR, Raman, AFM, and SEM.

2.3. Corrosion Measurement. The corrosion tests were conducted with the use of advanced potentiostat (Wenking MLab-200, Bank Elektronik-Intelligent Controls GmbH, Germany) with all accessories, cell, electrode, and working electrode holder. The corrosive medium was artificial sea water made by dissolving 35 g of NaCl in 1-liter distilled water, the polarization curves were scanned between -200 and +200 mV from the open circuit potential and the corrosion currents i_{corr} , and corrosion potentials E_{corr} were evaluated by extrapolation of the cathodic and anodic Tafel lines from the polarization curves at four different temperatures, namely, 20, 30, 40, and 50°C.

3. Results and Discussion

3.1. Graphene Oxide Characterization. The FTIR spectrum of the produced graphene oxide, GO (brown in color),

FIGURE 1: The FTIR transmission spectrum of GO produced by Hammers method.

FIGURE 2: Raman spectrum of the graphene oxide sheets produced by modified Hammers method from graphite flakes.

showed transmission peaks at 3400, 1700, 1630, 1400, and 1080 cm^{-1} , belonging to the stretching and bending of the vibrations of the O-H and C=O from carbonyl groups, C=C configurable vibrations from the aromatics, and C-OO stretching vibrations [16], as assigned in Figure 1.

Figure 2 shows the Raman spectrum of the produced graphene oxide characterized by the appearance of broad peak at wavelength at 1355 cm^{-1} , which belonged to the band D, with the peak of band G which belonged to graphite [17].

The SEM images in Figure 3 show the morphology and topographic structure of the produced GO; they represent nanoplates with thicknesses varying between 15 and 65 nm.

Similar estimation of the GO nanoplates thicknesses was deduced from section line AFM analysis as shown in Figure 4.

Figure 5 shows the XRD spectrum for the started graphite flakes and the produced GO which proved the success of the conducted chemical exfoliation process; the main peaks at 2θ of 26.5° belonging to the plane (200) [18] became broader which means much less numbers of graphene layers as estimated using Scherer equation [19].

3.2. Electrodeposited Reduced GO Characterization. The FTIR spectrum of the reduced GO revealed the absence of the C-O and C-OH absorption bands; the only peaks belonged to C=C and C-H vibrations bands, as shown in Figure 6.

FIGURE 3: SEM images of graphene oxide sheets produced by modified Hammers method from graphite flakes.

FIGURE 4: AFM images show the 3D and section line analysis of GO produced by modified Hammers method from graphite flakes.

FIGURE 5: The XRD spectrums for the started graphite flakes (a) and the produced GO (b).

FIGURE 6: The FTIR transmission spectrum of electroreduced GO produced by Hammers method.

FIGURE 7: Raman spectrum of the electroreduced GO produced by Hammers method.

Figure 7 shows the Raman spectrum of the reduced graphene oxide; it recorded the appearance of the peak 2733 cm^{-1} which belongs to the 2D band, in addition to the peaks (1366 and 1660 cm^{-1}) for D band and G band, respectively, indicating that the produced graphene sheets got some degree of defection; the 2D/G intensity ratio was 1.51 which means that the reduced GO was composed of multiple layers of defected graphene nanosheets [20].

The SEM images for the reduced graphene oxide in Figure 8 revealed larger areas of nanosheets with smaller thicknesses which attributed to the loss of the oxygen bonds (carbonyl, epoxides, etc.) due to the reduction process [9, 21].

The cross-sectional analysis of the AFM scans shows a thicknesses ($\sim 15\text{ nm}$) smaller than that of the GO before electroreduction step, as shown in Figure 9.

3.3. Corrosion Parameters Measurement. Figures 10, 11, and 12 show Tafel polarization curves of the uncoated and reduced graphene oxide coated SS316, copper, and aluminum in seawater, respectively, in the temperature range ($20\text{--}50^\circ\text{C}$).

The corrosion potentials (E_{corr}) in mV versus SCE, the corrosion current density (i_{corr} in $\mu\text{A}\cdot\text{cm}^{-2}$) and β_a , and β_c in $\text{mV}\cdot\text{dec}^{-1}$ were established from Tafel plots (Figure 10), and weight loss (WL) in $\text{g}\cdot\text{m}^{-2}\cdot\text{day}^{-1}$ and penetration loss in $\text{mm}\cdot\text{y}^{-1}$ were calculated by faradic conversions, while R_p in $\Omega\cdot\text{cm}^2$ was calculated using the following equation [22]:

$$R_p = 2.303 \left[\frac{\beta_a \cdot \beta_c}{(\beta_a + \beta_c)} \right] * \frac{1}{i_{\text{corr}}} \quad (1)$$

Surface coverage (θ) and protection efficiencies (% PE) were calculated from i_{corr} as reported in [22, 23] and listed in Table 1.

The thermodynamic parameters of the corrosion process, namely, the activation energy (E_a), the entropy (ΔS^*), the enthalpy (ΔH^*), and the Gibbs free energy (ΔG^*) of activation, were calculated using Arrhenius plot and its derivative formulation (transition state) [24, 25]; all values are listed in Table 2.

All reduced graphene coated samples showed good enhancing for corrosion inhibition action in comparison with the bare samples; the best protection efficiencies were for SS316 and their values reached 95% at 20°C and then decreased with increasing temperature. While the copper and aluminum samples showed nearly constant corrosion efficiencies values (around 65%), no effect of rising temperature was noticed in these values (Table 1).

The thermodynamic and kinetics data show that the E_a values for the graphene coated SS are higher than the

FIGURE 8: SEM images of electroreduced GO sheets produced by modified Hammers method from graphite flakes.

FIGURE 9: AFM images show the 3D and section line analysis of electroreduced GO produced by modified Hammers method from graphite flakes.

TABLE 1: Corrosion parameters for uncoated and graphene coated specimens in seawater at different temperature.

Material	T °C	E_{corr} mV versus SCE	i_{corr} $\text{mA}\cdot\text{cm}^{-2}$	β_a $\text{mV}\cdot\text{dec}^{-1}$	β_c $\text{mV}\cdot\text{dec}^{-1}$	Rp $\Omega\cdot\text{cm}^2$	WL $\text{Mg}\cdot\text{m}^{-2}\cdot\text{d}^{-1}$	PL $\text{mm}\cdot\text{y}^{-1}$	θ	PE%
Uncoated SS	20	-179.2	27.00	113.8	101.6	801.5	6.75	0.3024		
	30	-194.7	31.23	125.7	106.5	581.1	7.80	0.3497		
	40	-202.2	44.34	141.3	102.3	446.5	11.08	0.4966		
	50	-215.6	59.23	137.3	109.5	257.8	14.80	0.6633		
Reduced GO coated SS	20	-65.4	1.22	116	192.1	3881.7	0.30	0.0136	0.954	95.4
	30	-172.2	6.51	111.7	121.5	1262.3	1.62	0.0729	0.791	79.1
	40	-171.6	13.46	76.5	80.1	1719.9	3.36	0.1507	0.696	69.6
	50	-167.8	16.04	98.5	179	501.8	4.01	0.1796	0.729	72.9
Uncoated Cu	20	-206.6	25.61	251	80.3	1031.4	7.27	0.2970		
	30	-204.9	30.95	338.7	90.7	1003.7	8.78	0.3590		
	40	-211.3	47.41	322.6	90.8	648.9	13.46	0.5499		
	50	-222.3	56.4	278.7	88.8	518.4	16.01	0.6542		
Reduced GO coated Cu	20	-145.1	6.01	72.3	58.3	2331.8	1.70	0.0697	0.765	76.5
	30	-140.8	10.66	53.5	47	1019.1	3.02	0.1236	0.655	65.5
	40	-155.3	15.95	59.2	58.5	801.0	4.52	0.1850	0.663	66.3
	50	-173.1	18.76	59.2	59.4	686.2	5.32	0.2176	0.667	66.7
Uncoated Al	20	-635.7	239.38	378.7	105.7	149.8	67.98	2.7768		
	30	-713.5	267.93	318.8	79.2	102.8	76.09	3.1079		
	40	-676.5	317.33	424.4	103.7	114.0	90.12	3.6810		
	50	-693.2	375.11	416.6	90.8	86.2	106.53	4.3512		
Reduced GO coated Al	20	-647.9	88.22	75.0	72.1	180.9	25.05	1.0233	0.631	63.1
	30	-670.1	94.54	81.0	73.4	176.8	26.84	1.0966	0.647	64.7
	40	-644.2	115.01	101.9	74	161.8	32.66	1.3341	0.637	63.7
	50	-735.0	118.7	98.4	95.5	177.2	33.71	1.3769	0.683	68.3

TABLE 2: The calculated thermodynamic functions, E_a , ΔH^* , ΔS^* , and ΔG^* , for the corrosion process of the uncoated and reduced GO coated SS, Cu, and Al in seawater.

System in 3.5% NaCl	E_a $\text{kJ}\cdot\text{mol}^{-1}$	ΔH^* $\text{kJ}\cdot\text{mol}^{-1}$	ΔS^* $\text{J}\cdot\text{mol}^{-1}$	ΔG^* ($\text{kJ}\cdot\text{mol}^{-1}$)			
				20	30	40	50
Uncoated SS	21.217	-18.66	-154.16	26.500	28.002	29.604	31.146
G-coated SS	67.264	-64.708	-19.35	59.03	58.844	58.65	58.45
Uncoated Cu	30.236	-19.430	-151.75	25.032	26.550	28.067	29.585
G-coated Cu	19.405	-27.679	-134.71	11.791	13.138	14.485	15.832
Uncoated Al	11.901	-9.343	-167.52	58.426	60.101	61.776	63.451
G-coated Al	8.561	-6.004	-187.12	60.830	62.701	64.572	66.443

uncoated ones which support the good protection efficiencies (70–95%) for SS, while the decrease in apparent activation energy for the graphene coated Cu and Al leads to less protection efficiencies, around 65% in this case; the number of corrosion sites can be represented by preexponential factor A in the Arrhenius equation:

$$R = Ae^{-E_a/RT}. \quad (2)$$

A is a term which includes factors like the frequency of collisions and their orientation. It varies slightly with temperature

but highly with the type of surface and is responsible for the corrosion protection abilities rather than E_a as in the case of Al and Cu [26].

On the other hand, the corrosion potentials or the open circuit potentials values of the reduced graphene coated specimens went to less negative values as compared to the uncoated ones.

The polarization resistances of the uncoated samples of SS316 and copper were always less than the coated samples, which can be attributed to the existence of some nonconductive graphene oxide [27], except for Al which shows the

FIGURE 10: Tafel plots of SS316 specimens in seawater system without and with reduced GO coating at different temperature.

FIGURE 12: Tafel plots of aluminum specimens in seawater system without and with reduced GO coating at different temperature.

FIGURE 11: Tafel plots of copper specimens in seawater system without and with reduced GO coating at different temperature.

reverse of this behavior; the reason for such manner is the ability of Al to liberate hydrogen gas in the aqueous solution [28].

The result suggests that corrosion inhibition by the reduced graphene layer is brought about by increasing its activation energy. There is an increase in E_a as a nature of the surface of the SS changed making a barrier for mass and

charge transfer. The range of E_a values (from 21 KJ/mol to 67 KJ/mol) is lower than the threshold value of 80 KJ/mol required for chemisorption. This means that adsorption is physical adsorption [29]. Hence, the slightly higher energy of activation values suggests strong physisorption.

Generally, the values of ΔH^* lower than 40 KJ/mol which attributed the physical adsorption and the value 100 KJ/mol indicate chemical adsorption. From Table 2; it was found that the negative sign and values (6.004 KJ/mol to 64.708 KJ/mol) of activation of enthalpy (ΔH^*) reflect the exothermic corrosion process [30]. The increase in ΔH^* for the graphene coated specimens reveals that corrosion rate is mainly controlled by kinetic parameters of activation [31]. The values of ΔS^* for the uncoated and graphene coated are negative. This implies that the activation complex is the rate determining step representing association rather than dissociation, indicating that a decrease in disorder takes place going from reactants to the activated complex [32].

4. Conclusions

Good adhered continuous layers of reduced graphene oxide were deposited electrochemically from solution of graphene oxide synthesized via Hammers procedure, on surfaces of stainless steel 316, copper, and aluminum; these layers exhibited good corrosion inhibition properties against artificial seawater in the temperature range of 20 to 50°C.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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