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Coating of Carbon Nanotubes Using Chemical Method to Enhance the Corrosion Protection of Copper and Aluminum Metals in Seawater Medium

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Abstract

In this study, carbon nanotubes were prepared using a pure chemical method modified similar to the Hummers method with simple changes in the work steps. The carbon nanotubes were then coated and reduced on copper and aluminum metals using the electrodeposition method (EDP) for corrosion protection application in seawater medium (NaCl 3.5%) at four different temperatures: 20, 30, 40, and 50 °C, which were studied using three electrode potentiostats. All corrosion measurements, thermodynamics, and kinetics parameters were nominated from Tafel plots. The films deposited by the carbon nanotubes were examined by the SEM technique, and this technique showed the formation of carbon nanotubes.

Keywords: A pure chemical method modified, Corrosion protection, Copper and Aluminum, Carbon nanotubes.

طلاء الأنابيب النانوية الكربونية باستخدام الطريقة الكيميائية لتعزيز الحماية من تآكل معادن النحاس والألمنيوم في وسط مياه البحر

مازن حسن رحيمة

قسم طب الاسنان, كلية التراث الجامعة, بغداد, العراق

الخلاصة

في هذه الدراسة، تم تحضير الأنابيب النانوية الكربونية باستخدام طريقة كيميائية نقية معدلة على غرار طريقة هامرز مع تغييرات بسيطة في خطوات العمل. ثم تم طلاء الأنابيب النانوية الكربونية واختزالها على معادن النحاس والألمنيوم بطريقة الترسيب الكهربائي (EDP) لتطبيق الحماية من التآكل في وسط ماء البحر (NaCl 3.5٪) عند أربع درجات حرارية مختلفة: 20 ، 30 ، 40 ، 50 درجة مئوية ، و التي تم دراستها باستخدام ثلاثة أقطاب كهربائية. جميع قياسات التآكل والديناميكا الحرارية والمعلمات الحركية تم حسابها من منحنيات تافل. تم فحص الأفلام المترسبة من الأنابيب النانوية الكربونية بتقنية المجهر الماسح الالكتروني SEM ، وأظهرت هذه التقنية تكوين الأنابيب النانوية الكربونية.

1. Introduction

Nanoparticles have unique optical, chemical, and electronic properties that are very different from those found in large particles [1]. Since Iijima's initial discovery of carbon

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nanotubes in 1991 [2], a lot of researchers have been interested in finding new, highly productive, and inexpensive ways to make them. Carbon nanotubes are a type of carbon material (also known as an allotrope), including carbon nanotubes, diamond, graphite, grapheme, and fullerenes [3]. Carbon nanotubes are used in many applications; this is because the gap between single-walled carbon nanotubes can differ from zero to about 2 volts, so their electrical conductivity can be one of a metal or semiconductor [4]. Carbon nanotubes can be prepared by different routes, such as arc discharge (AD) [2], laser ablation [5,6], and chemical vapor deposition (CVD) [7-9]. Most of the methods that were mentioned to prepare carbon nanotubes require harsh conditions of high pressure and high temperature with low productivity, so we resorted to inventing a new pure chemical method that does not require harsh conditions and operates at room temperature, low cost, and with high productivity. The hexagonal graphene sheet can theoretically be wound to create carbon nanotubes, as seen in Figure 1 [9].

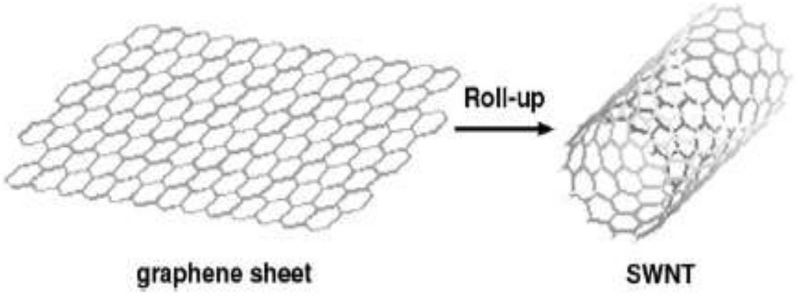


Figure 1: Show create a single wall carbon nanotube by rolling-up agraphene sheet [9].

It is possible to turn this theoretical principle into practice by devising a modified chemical method at room temperature similar to the Hummers method [10] with a small change in some of the work steps. This is mentioned in some research [11]. The use of the electrodeposition method (EDP) for coating and reducing graphene oxide on metals has been presented and recorded in many types of research [12-17]. So we can use this method to coat and reduce carbon nanotubes on metals to enhance corrosion protection for those metals. Corrosion is the result of the electrochemical reactions that occur between the metal and the environment, which lead to the electrochemical destruction of metals. In order to solve this issue, various types of research have been conducted, the most significant of which is the coating of metals with nanomaterials that prevent corrosion [18-22]. The use of carbon nanotubes in studies to enhance the corrosion protection of metals is numerous and recorded in many types of research [23-26].

2. Materials and Methods

2.1. Preparation of carbon nanotube

Graphite (2 g) was added to concentrated H_2SO_4 (46 mL, specific gravity 1.84) with stirring at room temperature. Sodium nitrate ($NaNO_3$) (1 g) was then added while the reaction mixture was at 0 °C using ice. Following that, $KMnO_4$ (6 g) was added slowly before being heated to 35 °C in a water bath with stirring for 30 minutes. Distilled water (100 mL, electrical conductivity 1µs.cm) was then added and the solution was stirred for 15 minutes at 90 °C. Another amount of distilled water (332 mL) was added. The mixture was then aged in the air for one week at room temperature. At the end of the aging period, the color of the solution has changed from yellow to dark brown. This is an indication of the conversion of graphene oxide to carbon nanotubes and carbon nanofibers. H_2O_2 (10 mL, 30%) was added to stop the oxidation. The mixture was filtered and rinsed with aqueous hydrochloric solution (170 mL, 4%) before washing with deionized water (130 mL) to remove the remaining acid. The desired product was washed with distilled water until the pH reached 6, then filtered and dried. The above-mentioned route for preparing carbon nanotubes is similar to the Hammers method [10] which was used for the fabrication of graphene oxide.

2.2. Carbon nanotube deposition and reduction

Carbon nanotubes were coated and reduced on copper and aluminum using electrophoresis deposition (EPD) [12,13]. First, a suspension of carbon nanotubes was prepared by the above method. This was done *via* adding carbon nanotubes (1 g) to deionized water (200 mL) and mixing the mixture well using an ultrasonic probe homogenizer for 15 minutes. The solution of carbon nanotubes was transferred into a two-electrode glass cell body (25 mL), and a well-cleaned and polished piece of the metal under investigation (copper and aluminum) served as the cathode, while a piece of pure platinum acted as the anode, and the two electrodes were connected to a DC power supply. The carbon nanotubes were supposed to be coated and reduced on the two metals under study after shedding several voltages for 5 minutes. The coated carbon nanotube layers were characterized using atomic force microscopy (AFM) and scanning electron microscopy (SEM).

2.3. Corrosion measurement

The corrosion tests were conducted with the use of an advanced potentiostat (wenking MLab-200, Bank Elektronik-Intelligent Controls GmbH, Germany) with all the accessories of the cell body, electrodes (reference electrode, auxiliary electrode, working electrode), and working electrode holder. The corrosive medium was artificial seawater (3.5% NaCl) prepared by dissolving sodium chloride (35 g) in deionized water (1000 mL). The polarization curves (Tafel plots) were scanned between -200 and + 200 mv from the open circuit potential and the corrosion currents (i_{coor}) plus the corrosion potentials (E_{coor}) of the metals under study (before and after coating), which were evaluated by extrapolating the cathodic and anodic Tafel lines at four different temperatures of 20, 30, 40, and 50 °C.

3. Results and Discussion

3.1. The prepared carbon nanotube characterization

The scanning electron microscopy (SEM) method was used to characterize the produced carbon nanotubes. The SEM images in Figure 2 show the morphology and topographic structure of the produced carbon nanotubes; they represent nanotubes and nanofibers. Using the software image J, the SEM images show that the prepared carbon nanotubes have an average maximum length of 10 µm and a minimum diameter (aspect ratio) ranging from 4 to 16 nm. This indicates that the prepared carbon nanotubes are of the type MCNTs (multi-walled carbon nanotubes), as shown in Table 1. The graphene oxide is converted into carbon nanotubes and carbon nanofiber in the SEM image that is displayed.

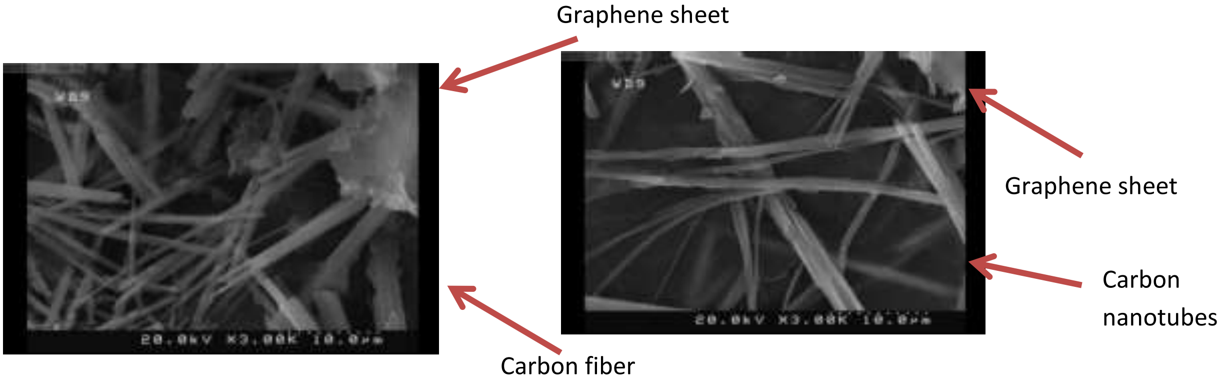


Figure 2: SEM images of carbon nanotube and carbon nanofiber produced by a new route from graphite flakes.

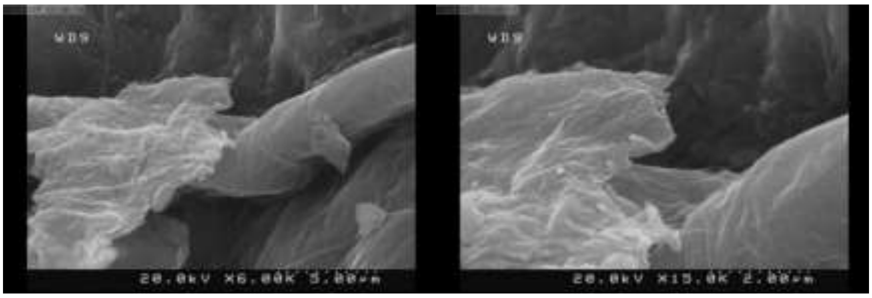


Figure 3: SEM images of graphene sheets before conversion into carbon nanotubes.

Table 1: Shows the lengths, widths, and aspect ratios resulting from the analysis of SEM images of carbon nanotubes prepared using the program image J.

No.	Length	Width	Aspect ratio
1	279.635	28.653	**9.759**
2	208.195	34.928	**5.961**
3	152.643	23.77	**6.421**
4	151.921	21.378	**7.106**
5	196.163	21.932	**8.944**
6	155.168	22.561	**6.877**
7	91.761	21.378	**4.292**
8	310.203	20	**15.510**
9	133.33	16.971	**7.856**
10	288.295	27.857	**10.349**
11	365.79	52.01	**7.0333**
12	352.892	35.44	**9.957**
13	258.017	32.558	**7.924**
14	238.948	27.857	**8.577**
15	132.004	29.682	**4.447**
16	189.73	29.682	**6.392**
17	266.092	21.587	**12.326**
18	149.833	18.028	**8.311**
19	178.443	27.166	**6.568**
20	302.002	27.659	**10.918**
21	507.001	45.804	**11.068**
22	413.807	22.136	**18.693**
23	646.696	40.05	**16.147**

3.2. Corrosion Parameters Measurement

Figures 3, 4, 5, and 6 illustrate Tafel polarization curves (Tafel plots) for two metals, copper and aluminum, both uncoated and coated with carbon nanotubes, in artificial seawater at temperatures ranging from 20 to 50 °C. These curves were obtained by measuring the corrosion potentials (Ecorr) in mV versus SCE *vs.* current density. The corrosion current density (i_{corr} in $\mu A \cdot cm^{-2}$) and βa, and βc in $mv \cdot dec^{-1}$ were determined from Tafel plots and weight loss (WL) in $g \cdot m^{-2} \cdot day^{-1}$ and penetration loss in $mm \cdot y^{-1}$ were calculated by faradic conversions, while Rp in $\Omega \cdot cm^2$ was calculated by Stern-Geary equation [27]:

$$Rp = babc / 2.303(ba + bc)ic \qquad --- (1)$$

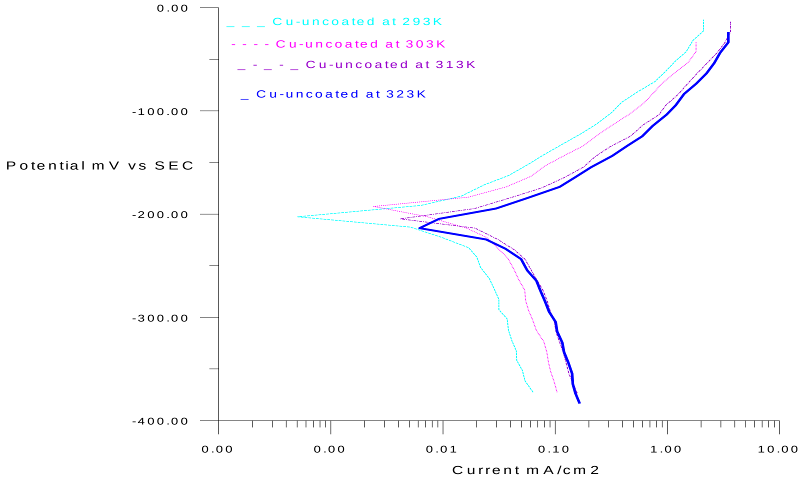


Figure 4: Tafel plots of uncoated copper in NaCl solution (3.5%) with different temperatures

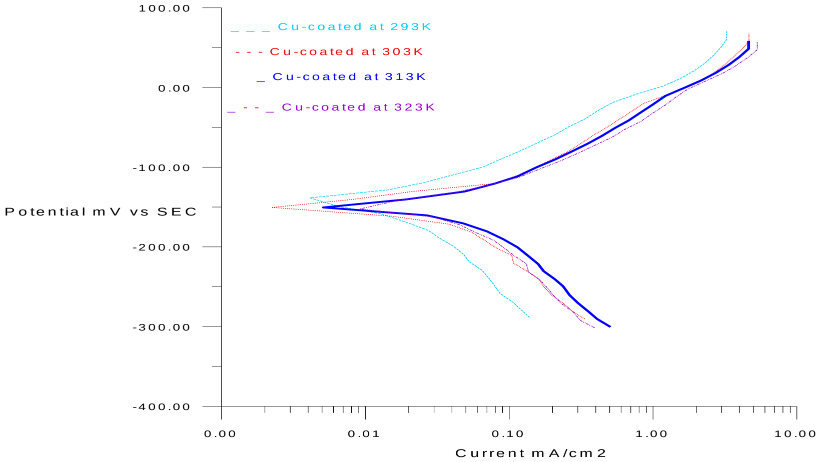


Figure 5: Tafel plots of the coated copper in NaCl solution (3.5%) with different temperatures

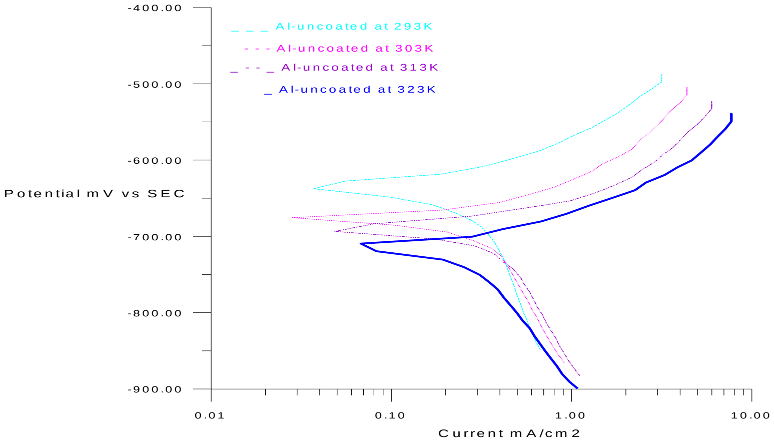


Figure 6: Tafel plots of the uncoated aluminum in NaCl solution (3.5%) with different temperatures.

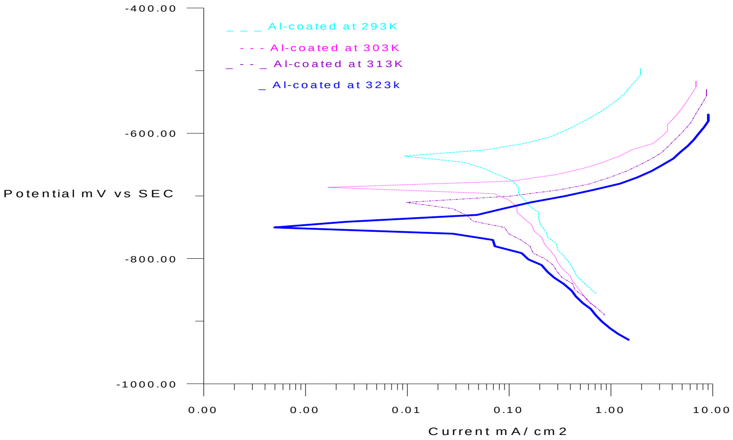


Figure 7: Tafel plot of coated aluminum in NaCl solution (3.5%) with different temperatures.

Surface coverage (θ) and protection efficiencies (% PE) were calculated from equations 2 and 3, respectively, and were defined as [27]:

$$\%PE = \frac{(icorr)uncoated - (icorr)coated}{(icorr)uncoated} * 100 \qquad --- (2)$$

$$\Theta = \frac{(icorr)uncoated - (icorr)coated}{(icorr)uncoated} \qquad --- (3)$$

Where $(i_{corr})_{uncoated}$ and $(i_{coor})_{coated}$ the uncoated and coated corrosion current density values, respectively, are determined by extrapolation of Tafel lines. The thermodynamic parameters of the corrosion reaction are represented by the activation energy (Ea), the entropy ($\Delta S*$), and the enthalpy (ΔH) which were calculated using Arrhenius plot 4 and its derivative formulation 5, called the transition state [28].

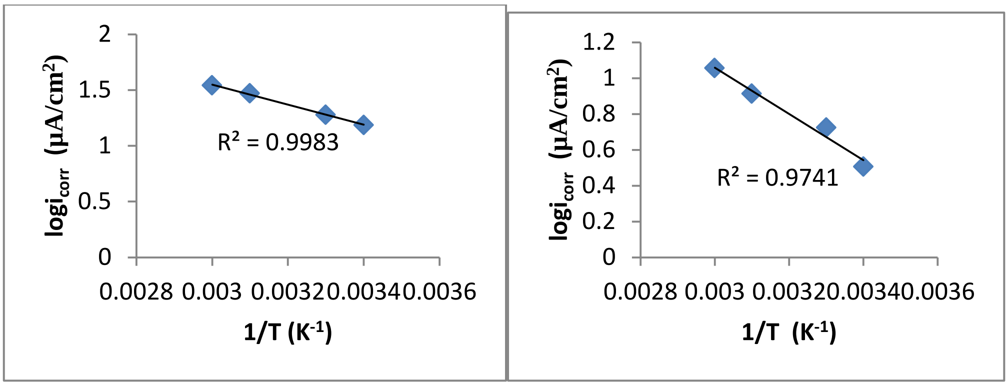
$$\log icorr = -\frac{Ea}{2.303 * R}\left[\frac{1}{T}\right] + \log A \qquad --- (4)$$

$$\log\left(\frac{icorr}{T}\right) = \log\left(\frac{R}{Nh}\right) + \frac{\Delta S *}{2.303R} - \frac{\Delta H *}{2.303R}\left(\frac{1}{T}\right) \qquad --- (5)$$

While Gibbs free energy ($\Delta G*$) was calculated using the equation 6:

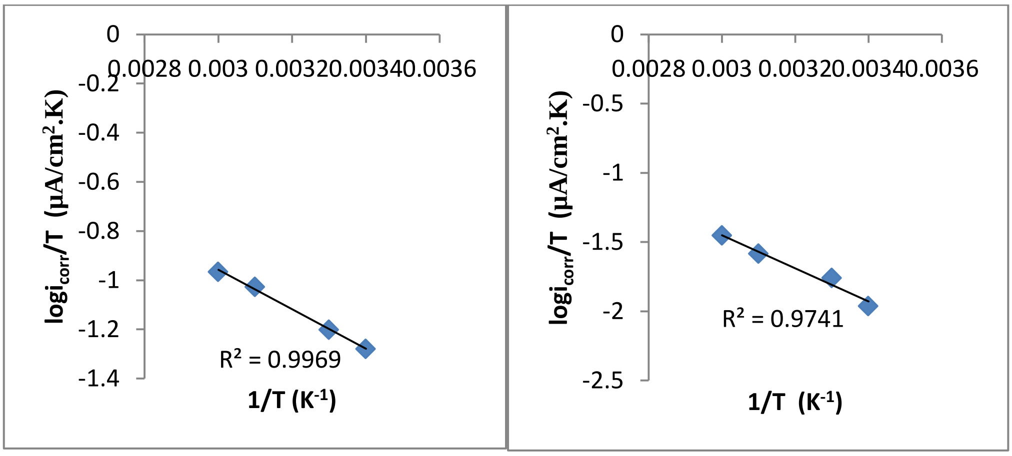
$$\Delta G^* = \Delta H^* - T\Delta S^* \qquad --- (6)$$

Where (i_{corr}) is the corrosion current density, h is the Planck's constant (6.626 × 10^{-34} J.S), N is Avogadro number (6.022 × 10^{23}), R is the universal gas constant (8.315 $JK^{-1}mol^{-1}$), ΔH* is the enthalpy of activation, ΔS* is the entropy of activation, and ΔG* is the Gibbs free energy. A plot of log (i_{corr}) *vs.* 1/T of equation 4 provides straight lines with a slope of (-Ea / 2.303R) and an intercept of (log A) for uncoated and coated with carbon nanotubes for two metals, copper and aluminum, as shown in Figures 7 and 9, respectively. A plot of log (i_{corr} / T) Vs 1/T of equation 5 provides straight lines with a slope of (-ΔH*/2.303R) and an intercept of (log R/Nh) + ΔS*/ 2.303R) for uncoated and coated with carbon nanotubes for two metals, copper and aluminum, as shown in Figures 8 and 10, respectively.



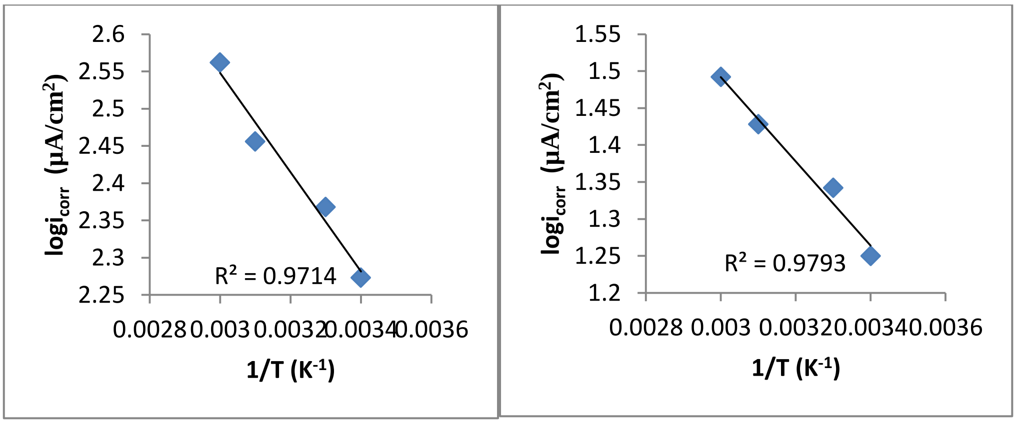
(A) (B)

Figure 8: log i_{corr} *vs*. 1/T for A (uncoated) and B (reduced carbon nanotube-coated copper) in seawater at different temperatures.



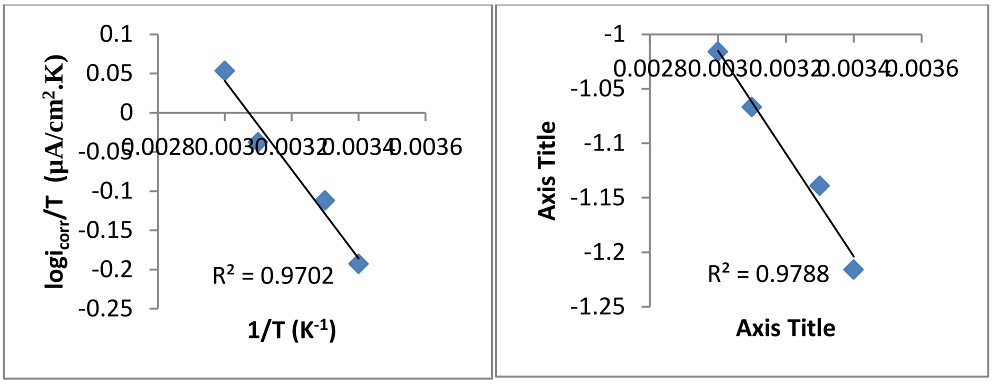
(A) (B)

Figure 9: Plot of $logi_{corr}$/T *vs*. 1/T for A (uncoated) and B (reduced carbon nanotube-coated copper) in NaCl solution (3.5%) to determine entropy (ΔS) and enthalpy (ΔH).



(A) (B)

Figure 10: log icorr *vs*. 1/T for A (uncoated) and B (reduced carbon nanotube with coated aluminum) in seawater at different temperatures.



(A) (B)

Figure 11: Plot of logicorr/T *vs*. 1/T for A (uncoated) and B (reduced carbon nanotube with coated aluminum) in NaCl solution (3.5%) to determine entropy (ΔS) and enthalpy (ΔH).

Table 2: Corrosion measurement parameters and the values of thermodynamics for uncoated and coated copper with carbon nanotubes in one medium at different temperatures ranging from 20 to50 °C.

System		T °C	E_{corr} mV	I_{corr} µA/cm^2	B_a (mV/Dec)	B_c (mV/Dec)	R_p Ω.cm^2	WL g/(m^2 d)	PL mm/y	PE %	E_a kJ/mol	ΔH_a kJ/mol	ΔS_a J/mol.K	ΔG_a kJ/mol	Θ In 20 °C
3.5% NaCl	Uncoated Cu	20	-202.6	15.36	77	258.7	1677.5	4.37	0.179	-				65.1	
		30	-193.5	19	62	173	1043.1	5.41	0.221		17.27	-15.4	-169.7	66.8	4.43
		40	-204.6	29.4	66.3	160	692.3	8.37	0.342					68.5	
		50	-213.8	34.9	73.7	186.5	657.2	9.93	0.406					70.2	
	Coated Cu	20	-139.7	3.2	64.3	134.2	5898.7	4.02	0.165	79.2	24.72	-22.87	-156.6	68.8	
		30	-151.2	5.3	79.6	134.7	4099.1	9.83	0.402	72.1				70.3	
		40	-150.3	8.2	83.5	116.6	2633.7	11.4	0.468	72.1				71.9	
		50	-153	11.4	73.5	115.1	1708.5	9.69	0.397	67.3				73.5	

Table 3: Corrosion measurement parameters and the values of thermodynamics for uncoated and coated aluminum with carbon nanotubes in one medium at different temperatures ranging from 20 to 50 C°.

System		T °C	E_{corr} mV	I_{corr} µA/cm²	B_a	B_c (mV/Dec)	R_p	WL	PL	PE %	E_a	ΔH_a	ΔS_a	ΔG_a	Θ%
					(mV/Dec)		**Ω.cm²**	**g/(m² d)**	**mm/y**		**kJ/mol**	**kJ/mol**	**J/mol.K**	**kJ/mol**	**In 20 °C**
3.5% NaCl	**Uncoated Al**	20	-638.6	187.86	94.6	226.8	154.3	15.1	2.04	-	12.75	10.8-4	-164.14	58.93	11.9
		30	-677	233.68	80.7	230.8	111.11	18.8	2.54					60.57	
		40	-694.6	286.15	82	234	92.14	23	3.11					62.22	
		50	-710.7	365.2	65.4	237.9	60.99	17.3	2.34					63.89	
	Coated Al	20	-637.2	17.8	64.4	254.6	1253.83	6.16	0.833	90.52	10.92	9.04-	-189.76	64.64	
		30	-687.6	22	39.6	170	633.92	5.79	0.782	90.03				66.54	
		40	-711	26.8	30.9	156.2	417.96	4.73	0.639	90.63				68.43	
		50	-751.3	31.1	57.5	119	541.27	4.93	0.666	91.48				70.33	

These results reflect the following important facts: all samples coated with carbon nanotubes showed a good improvement in reducing the corrosion effect of the studied samples compared to the uncoated samples; and corrosion protection values for aluminum are almost constant with increased temperature (*circa* 90%). It is noted that there is no effect of increasing temperature on corrosion protection values as shown in Table 2. This is because the value of the activation energy of coated metal is lower than that of uncoated metal. While copper shows the effect of increasing temperature on the protection values, as shown in Table 1, in the range between 67 and 79%, because the value of the activation energy of a coated metal is higher than that of an uncoated metal. Thermodynamic and kinetics data showed that the activation energy (*E*a) values for copper coated with carbon nanotubes are higher than the uncoated ones, which supports good protection efficiency of 67-79% for copper, while we noticed a decrease in the value of the activation energy for aluminum coated with carbon nanotubes; the reason is that there was no corrosion occurring on the aluminum surface. The number of corrosion sites can be represented by the pre-exponential factor *A* in the Arrhenius equation [28]:

$$R = A \exp(-Ea / RT) \qquad \text{----------- (7)}$$

A is a term that includes factors such as the frequency of collisions and their orientation.

The corrosion potentials of the coated carbon nanotubes on copper decreased as compared to the uncoated ones, whereas the corrosion potentials of the coated nanotubes on aluminum were the reverse. The reason is that the activation energy of uncoated aluminum is higher than that of coated one. The polarization resistance values of coated metals (Cu and Al) are significantly lower than those of uncoated metals, indicating that the carbon nanotube layer improved the high protection of metals. Chemical adsorption requires activation energy values of more than 80 KJ/mol and since the activation energy values for the metals under study (physical adsorption of carbon nanotube particles on the metal surface) showed less than 80 KJ/mol (from 10 to 24 KJ/mol), this indicates that it is a physical adsorption [29]. There is further evidence that it is physical adsorption when the enthalpy ($\Delta H*$) values are less than 40 KJ/mol for the studied metals, because when the enthalpy values are 100 KJ/mol, the adsorption is chemical. Enthalpy values in Tables 1 and 2 have a negative sign, indicating that the corrosion process is exothermic [30]. The values of $\Delta S*$ for the uncoated and coated carbon nanotubes are negative. This means that the activated complex, which is the rate-limiting step of the reaction, represents an association rather than dissociation (this confirms a decrease in the number of degrees of kinetics through the formation of the activated complex). This indicates a decrease in disorder taking place by going from the reactants to the activated complex (the overall degree of freedom throughout the formation of the activated complex for the reaction of copper with negative species leading to the formation of a corrosion product) [31].

4. Conclusion

A simple and inexpensive chemical method produces carbon nanotubes with high quality and high productivity. The EPD method of carbon nanotubes on the surface of metals gives highly sticky layers. These layers indicated good corrosion inhibition properties (particularly for aluminum, which demonstrated the highest protection of nearly 90%) against artificial seawater at four different temperatures (20-50 °C).

Conflicts of Interest

The author declares that they have no conflicts of interest.

Acknowledgments

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