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Synthesis of Carbon Nanotubes Using Modified Hummers Method for Cathode Electrodes in Dye-Sensitized Solar Cell

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Abstract:
In this research, carbon nanotubes (CNTs) is prepared through the Hummers method with a slight change in some of the work steps, thus, a new method has been created for preparing carbon nanotubes which is similar to the original Hummers method that is used to prepare graphene oxide. Then, the suspension carbon nanotubes is transferred to a simple electrode position platform consisting of two electrodes and the cell body for the coating and reduction of the carbon nanotubes on ITO glass which represents the cathode electrode while platinum represents the anode electrode. The deposited layer of carbon nanotubes is examined through the scanning electron microscope technique (SEM), and the images throughout the research show the formation of carbon nanotubes. In the second part of the research, a dye-sensitized solar cell (DSSC) is prepared to utilize carbon nanotube as the cathode electrode, titanium oxide nanoparticles as the anode electrode by using a natural dye (pomegranate dye). The assembled dye-sensitized solar cell (DSSC) is tested by two electrodes potentiostat using Xenon light source and all parameters are established; V_{ocp}, I_{max}, V_{max}, I_{sc} and calculated full factor and conversion efficiency.

Keywords: Carbon nanotubes, Dye-sensitized solar cell, Electrophoretic deposition method, Modified method, Natural dye.

Introduction:
Nanoparticles have new physicochemical properties such as the optical, chemical, and electronic properties of nanoparticles which may differ significantly from those of each material in the bulk[1]. Carbon nanotubes are first spotted by Sumio Iijima in 1991[2]. CNTs are of great interest in current research as well as future industrial applications; including their use as a cathode electrode in dye-sensitized solar cells (DSSC) because they have a high electrical conductivity. Carbon nanotubes can be synthesized in several routes such as arc discharge (AD) [2], laser ablation, and chemical vapor deposition CVD [3-7]. These routes are the most common methods used to prepare it. Most of these methods which have been already mentioned above need special requirements of high pressure and energy in addition to the high cost with low production yield. So, it is possible to find out suitable methods for preparing that are simple and low cost with high production yield. Among all these methods, the chemical method is the most appropriate one. Theoretically, it is possible to synthesize carbon nanotubes by rolling graphene sheets as shown in Fig.1 below:

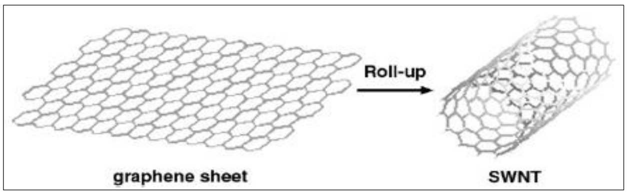

Figure 1. It is possible to build carbon nanotubes by rolling graphene sheet[8].

Moreover, this can be also transformed in practice by using chemical methods such as the Staudenmaier method [9] by making a slight change in the procedure. This has been recorded in some researches, in this process, a multi-walled carbon nanotube (MWCNT) is prepared by utilizing the developed Staudenmaier route. Multi-walled carbon nanotube (MWCNT) is prepared from graphite flakes at room temperature and then characterized by using techniques SEM, AFM, TGA, and XRD [10]. The use of the coated electrophoretic deposition method (EPD) for the reduction of graphene oxide is recorded in some of the research[11-16]. The electrophoretic depose method(EPD) is used to get deposition and reduce graphene oxide on carbon steel C45 metal and the use of that layer is to enhance the corrosion protection in the corrosive media of artificial seawater at four temperatures 20, 30, 40 and 50C°. This method gives layers with good adhesion to the metal, where these layers are investigated by techniques: FTIR, SEM, and AFM. It gave good protection, up to 81%[11]. The electrophoretic deposition method(EPD) is used to depose and reduce graphene oxide(rGO) on three metals, namely; stainless steel, Cu, and Al, and the use of that layer to enhance the corrosion protection in the corrosive media of artificial seawater at four temperature 20, 30, 40 and 50C. This method gives layers with good adhesion to the metal, where these layers are investigated by techniques, namely: FTIR, SEM, Raman spectra, XRD and AFM. Besides, it gives good protection up to 95% for stainless steel, while the metals copper and aluminum give protection up to 76.5%, and 68.3, respectively[12]. Have used the electrophoretic deposition (EPD) method to deposit the graphene oxide and its partial reduction at the same time on the copper metal to create a thin, uniform, and transparent layer, the coating is applied after applying 10 volts for one second to a graphene oxide suspension containing 0.03 wt % graphene oxide GO. It is important to mention that the coated layer (GO) is used to reduce the corrosion of copper metal by approximately 54% [13]. Furthermore, the use of the electrophoretic deposition (EPD) method is to deposit hydroxyapatite (HA) and graphene oxide (GO) on the surface of nano-titanium. They use different concentrations of graphene oxide suspension to deposit it on the HA-ATi [14]. They deposit titanium oxide-reduced graphene oxide nanocomposite (TiO_2-rGO) on stainless steel 316L by using the electrophoretic deposition (EPD) method, at first they prepare graphene oxide by Hummers method, then it is coated with the presence of titanium oxide on the stainless steel 316L, where the graphene oxide is reduced and coated simultaneously, then the layer is diagnosed (the morphology and microstructure) by techniques, emission scanning electron microscopy (FE-SEM), Raman spectra and X-ray diffraction (XRD) techniques, while the topography is diagnosed by using atomic force microscopy (AFM) [15]. Have deposited graphene oxide Nitrogen-doped graphene on copper by electrophoretic deposition (EPD) method, while N-doping is performed hydrothermally in the presence of ammonia. It is noteworthy that these copper-coated layers reduce the corrosion of the coated metal[16].

Dye-sensitized solar cell (DSSCs) titanium dioxide nanoparticles since their first discovery by Grätzel, has attracted great interest in research due to their high efficiency and low cost[17]. Dye-Sensitized Solar Cell (DSSCs) consists of four main components, they are the anode electrode, the cathode electrode, the electrolyte, and the dye. Many researchers have resorted to increase in the efficiency either by improving the dye or by using nanomaterials, especially the carbon family (as carbon nanotubes and graphene) [18-33]. Have prepared dye-sensitized solar cell utilization multi-walled carbon nanotubes(MWCNTs) as the counter electrode and titanium oxide TiO_2 nanoparticles as the anode electrode with the use of an industrial dye (cis-diisothiocyanato-bis (2.2'-bipyridyl-4.4'-dicarboxylic acid) ruthenium (II) knows as N3, the multi-walled carbon nanotubes (MWCNTs) are coated on the conductive glass FTO by a spin coating method, giving the cell Voc 626.23 mA, Isc 3.14 mA, I_{max} 2.332 mA, V_{max} 381.95 mV, fill factor (FF) 0.49, conversion efficiency (E%) 1.97 [18]. They have prepared three dye-sensitized solar cells (DSSCs) utilization carbon nanomaterials as the cathode electrodes (graphene oxide GO, reduced graphene oxide rGO, and multiwalled carbon nanotubes MWCNT) and titanium oxide nanoparticles as the anode with ruthenium dye [19]. Through using graphene and reducing graphene oxide (rGO) as a layer on FTO by CVD and spinning coating route respectively as well as using it as cathode electrode for making dye-sensitized solar cell (DSSCs), the graphene layer was diagnosed via HRTEM technique. From the results obtained, it became clear that the expensive platinum in the DSSCs could be replaced by a cheap and easy-to-prepare material, which is graphene-based as a cathode electrode [20]. Have prepared graphene oxide by using the Hummers method and depositing it on FTO through spinning a coating method and mixing graphene oxide with poly (3,4-ethylenedioxythiophene) polystyrene sulfonate PEDOT: PSS to increase the conductivity and promote simultaneously the formation of a good film, thermal reduction of GO (convert the graphene oxide to reduced graphene oxide) to reduce oxides [21]. Three carbon nanomaterials (graphene oxide GO, reduced graphene oxide rGO, and multi-walled carbon nanotubes MWCNT) counter electrodes which are fabricated for preparing three dye-sensitized solar cells (DSSCs), graphene-based films are fabricated through reducing graphene oxide rGO and graphene oxide GO and blending the structure with polystyrene sulfonate doped poly (3,4-ethylenedioxythiophene) PEDOT: PSS on FTO glass sheets using ruthenium dye, the graphene-based films are diagnosed by techniques; Raman spectroscopy, scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM) and the conversion efficiency of the prepared DSSCs was measured; GO/PEDOT: PSS equal to 1.5 %, rGO/PEDOT: PSS equal to 6.62%, MWCNT/PEDOT: PSS equal to 5.97%[22].

The use of natural dye in dye-sensitized solar cells has plentiful benefits, including low cost, with good efficiency, and its use has been recently reported [34-38]

Synthesis of carbon nanotubes

Carbon nanotubes are prepared in two stages: the first stage is to prepare graphene oxide by using the Hummers method while the second stage is used to convert the prepared graphene oxide into carbon nanotubes as shown below:

1. Preparation of graphene oxide (GO)

graphene oxide (GO) is prepared from graphite powder by using the Hummers route [39] with a slight change in some work steps as follows: In the beginning, graphite 2.0 g is added to 46 mL concentrated sulfuric acid H_2SO_4 sp. gr. 1.84, with good stirring at laboratory temperature then, 1.0 g $NaNO_3$ is added while the reaction mixture at 0 ° C under good stirring, 6 g $KMnO_4$ which should be added slowly with noticing that the temperature of the suspension must be maintained at about 2 °C, then the reaction mixture is heated to 35 °C in a water bath with continuous stirring for fifteen minutes, then, 100 mL 1µs.cm of DI deionized water is added and the solution is stirred for 15 min at 90°C, then 332 mL of distilled water is added [40]. The process steps are shown in Fig. 2 below:

Materials and Methods:

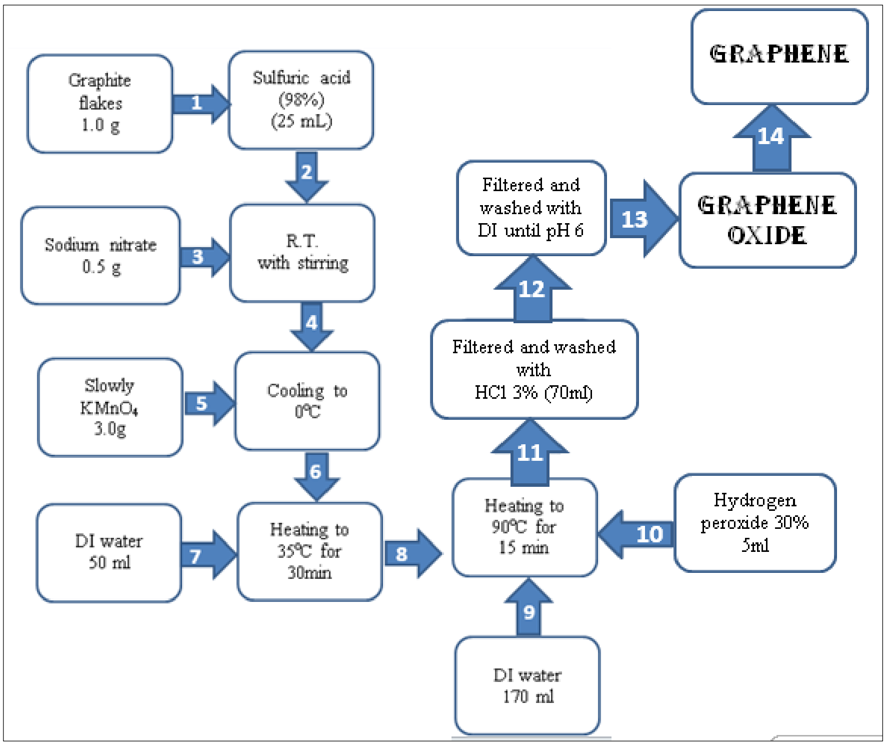

Figure 2. **Shows process flow diagram of Hummers method for graphene oxide synthesis.**

2. Conversion of graphene oxide to carbon nanotubes:

The mixture (graphene oxide) is aged in the air for 10 days at room temperature, and after the aging period ends, It will notice a change in the color of the suspension from yellow to dark brown. So, it is a clear indication of the conversion of graphene oxide into carbon nanotubes. An additional 10 mL of hydrogen peroxide (30%, Seema International) is added to stop the oxidation. after that, the resulting mixture is filtered and rinsed with 170 mL 4% aqueous hydrochloric solution followed by rinsing 130 mL deionized water to remove the acid, then the oxidation product is rinsed until the pH reached 6, i.e, the solution is close to neutral, then filtered and dried. As shown in Fig. 3 below:

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Figure 3. **Shows the steps of preparing carbon nanotubes using a new method.**

Deposition of reduced carbon nanotubes

Coating and reducing the prepared carbon nanotubes on conductive glass type (ITO) through using the electrophoretic deposition (EPD) method for producing diagnosis and preparing the cathode electrode for the synthesis of the dye-sensitized solar cell. In the beginning, the suspended carbon nanotubes are prepared by adding 0.5 g of the carbon nanotubes prepared in the first step to 100 mL of deionized water then the mixture is mixed using an ultrasonic applicator homogenizer (MTI, USA,300W) for 15 minutes to homogenize the mixture.

The electrochemical cell is prepared from a glass cell body (beaker) 25 ml and two electrodes, one of which is a platinum foil that acts as the anode electrode and the other is a conductive glass type (ITO) that acts as the cathode, and the two electrodes are linked to a DC power supply, the carbon nanotubes solution transferred to the glass cell body. The deposition begins with the application of several DC voltages for (five min), as shown in Fig. 4 below. The coated carbon nanotube layers are characterized by using a scanning electron microscope (SEM) and atomic force microscope (AFM).


Figure 4. **Shows the steps of deposited carbon nanotubes on ITO glass using the electrophoretic deposition (EPD) method.**

Synthesis and characterization of the dye-sensitized solar cell

The assembly of the dye-sensitized solar cell is used in four general steps, namely; cleaning of electrodes holder which is ITO glass, preparation of TiO_2 the active anodes, preparation of catalyzed counter electrode for CNTs, and finally the dying and electrolyte obtrusion between the active anode and catalyzed cathode sandwich. The active anode is prepared by coating a layer of TiO_2 nanoparticles on ITO glass using the dipping method, the coating method used is applying a viscous paste of TiO_2 nanoparticles prepared by treating 0.5 g of TiO_2 powder with a few drops of 0.1M HNO_3 of nitric acid) on a conductive side of ITO glass which is determined by the volt-ohm meter, then it is placed in the oven at 450°C for one hour to annealed it, annealed titanium oxide paste is cooled to room temperature gradually and then soaked in a natural dye solution (pomegranate dye) for one day, and then washed with ethanol after soaking, all work steps are shown in Fig.5 below, while the counter electrode is prepared by coating CNTs using the (EPD) method mentioned earlier, as shown in Fig.6 below. assemble the solar cell by adding a few drops of electrolyte (KI/I_2) to the photoelectrode and fix both electrodes (anode and cathode) facing each other using connector clips. As shown in Fig.7 below

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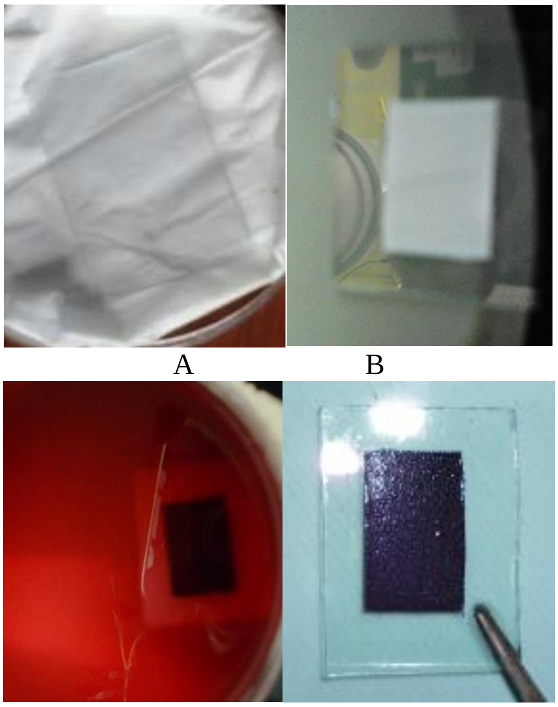

Figure 5. Preparation of TiO_2 photo-anode,(a) cleaned ITO glass,(b) TiO_2 coating,(c)dyeing , and (d) final TiO_2photoanode.

Figure 6. Shows the coated carbon nanotubes on ITO glass (counter electrode).

The emptiness between two electrodes is obtained by using scotch tape from both sides as a spacer.

Figure 7. Shows the final assembled dye-sensitized solar cell.

Finally, the assembled dye-sensitized solar cell is tested by two electrodes potentiostat through using Xenon light source (as shown in Fig. 8 below) and all parameters are established; V_{ocp}, I_{max}, V_{max}, I_{sc}. The scan range is -1000mV to +1000mV at a scan rate of 30 mV/sec, so the total scan time does not exceed a couple of minutes which prevents cell boiling during the test period.

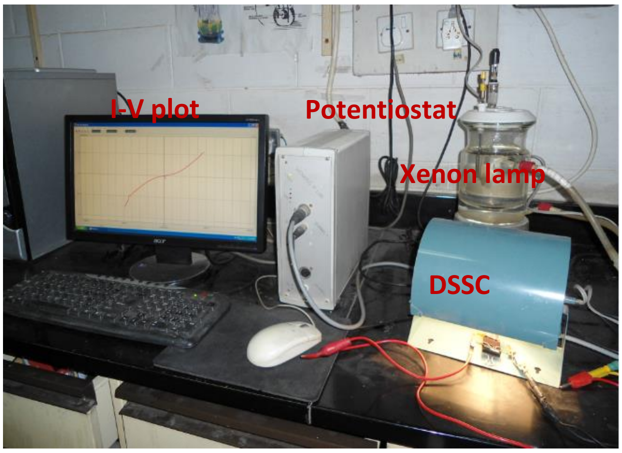

Figure 8. Shows the I-V dye-sensitized solar cell measurement apparatus.

Results and Discussion:

Characterization of the prepared graphene oxide

The graphene oxide (GO) is diagnosed by scanning electron microscope SEM, where the thickness of the nanoplates is between 15-65 nm, as shown below in Fig. 9

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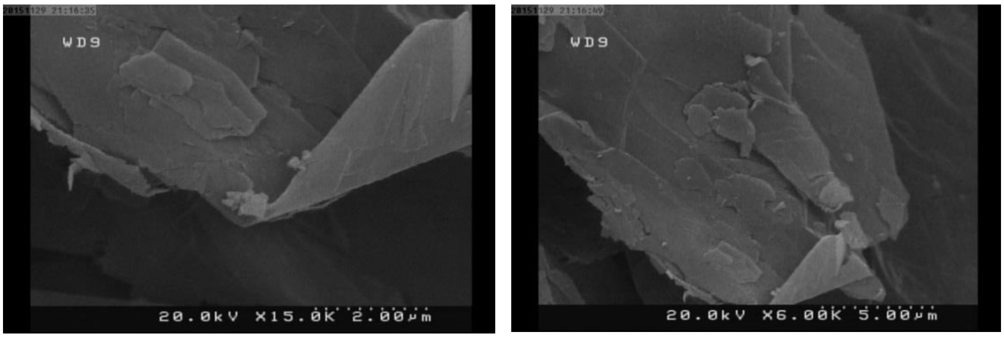

Figure 9. Shows (SEM images) of graphene oxide (GO) sheets are generated through hammers route from graphite flakes.

Characterization of the prepared carbon nanotubes

The prepared CNTs are characterized by (SEM). The scanning electron microscope images in Fig. 10 show the morphology and topographical structure of the produced carbon nanotubes, they represent nanotubes and nanofibers. Through the SEM image, the transformation of graphene into carbon nanotubes and carbon nanotubes is illustrated[8].

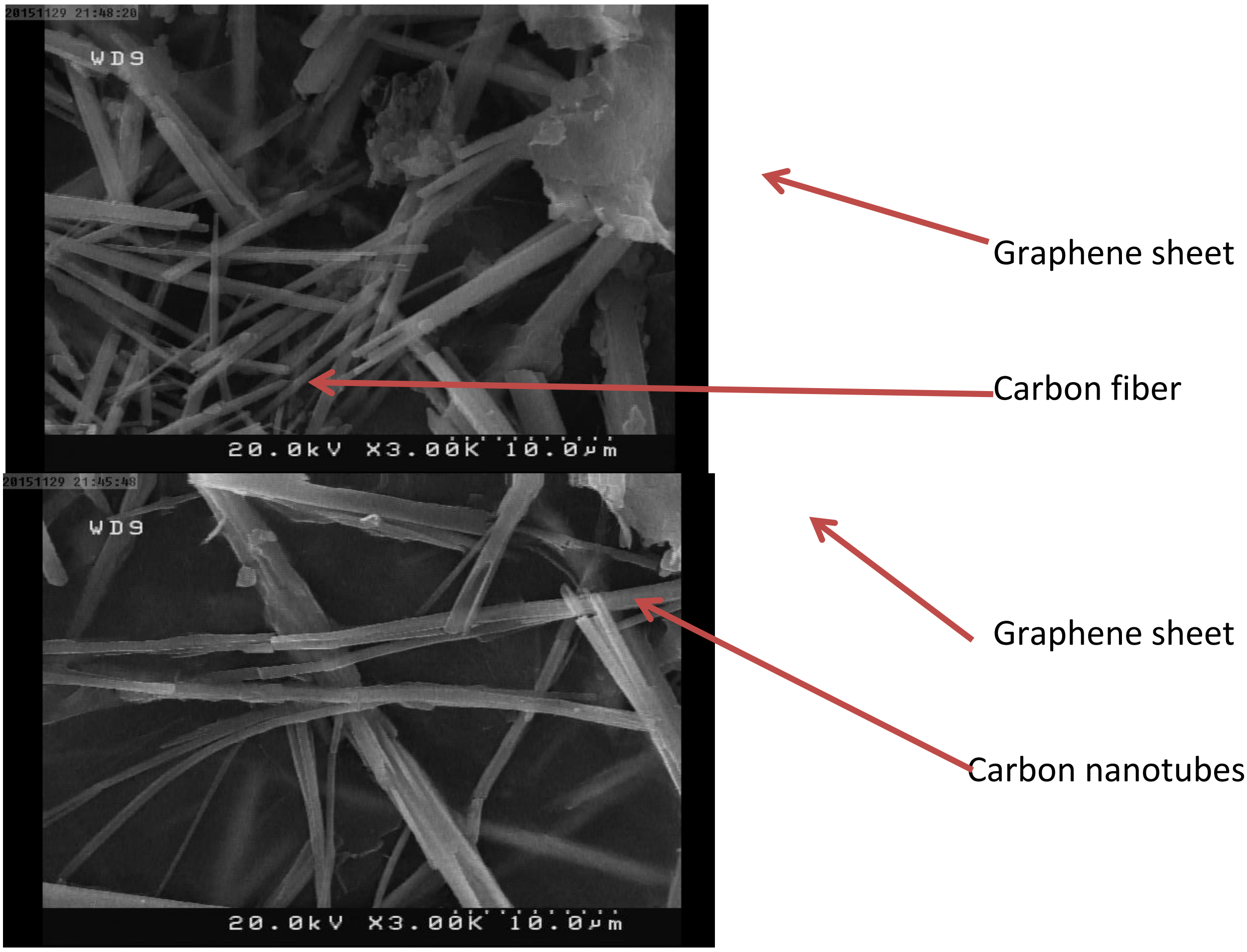

Figure 10. **SEM images of carbon nanotube created through a modified route from graphite flakes.**

Characterization of the dye-sensitized solar cell

After examining the dye-sensitized solar cell with Mlab 200 potentiostat, the i-v plot is obtained from Fig. 11, through the i-v. In the plot, V_{ocp}, I_{max}, V_{max}, and I_{sc} are gotten and from these parameters the full factor (ff) and energy conversion efficiency can be calculated by using the following two equations [40]:

$$ff = \frac{Imax*Vmax}{VIsc*Voc} \qquad 1$$

$$\%\varepsilon = \frac{Isc*Voc}{input\ power} * ff * 100 \qquad 2$$

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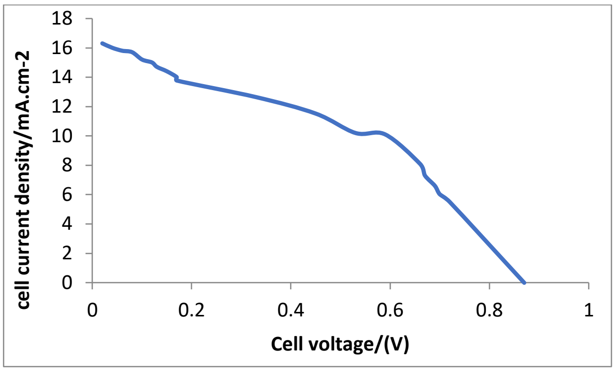

Figure 11. The current / voltage plots of a dye-sensitized solar cell fabricated using; TiO_2 photoanode/ pomegranate dye/ KI/I_2 as electrolyte and counter electrode by using CNTs and denoted on the graphs.

Table 1. Calculated energy conversion (E%) and full factor (FF) for dye-sensitized solar cell fabricated using; TiO2 photoanode/ pomegranate dye / KI/I_2 as electrolyte and counter electrode by using CNTs.

V_{OC} (v)	I_{SC} (mA/cm^2)	V_{max} (v)	I_{max} (mA/cm^2)	E%	FF
0.87	16.2	0.62	8	4.93	0.35

The prepared dye-sensitized solar cell shows a high conversion efficiency 4.93 % which is higher than the efficiency of the dye-sensitized solar cell that uses platinum as a cathode electrode as mentioned in some research, the reason is the high transmittance of carbon nanotubes coated on ITO glass, which reaches 60% in the 430-780 nm wavelength range. Besides, it is found that the transmittance of carbon nanotubes depends on the amount of oxygen functional groups. The mechanism of action of dye-sensitized solar cells is similar to the process of photosynthesis that occurs in plants and mosses, where photons of solar radiation pass during the glass plate shock and absorb the molecules of the organic dye coated on the titanium oxide (TiO_2) and will excite the electrons of the outer orbitals of the molecules of the organic compound and acquires the energy needed for separation, from its nuclei it is liberated to infiltrate the TiO_2 nanoparticles and then move to the upper electrically conductive layer. The electrons lost by the organic dye molecules will be compensated by the electrolyte solution, which is the iodine solution inside the cell, which leads to the oxidation of this compound, converting it to trivalent iodine, therefore, it completes the electrical conduction circuit of the cell during the electrochemical process[41,] as shown in Fig. 12 below.

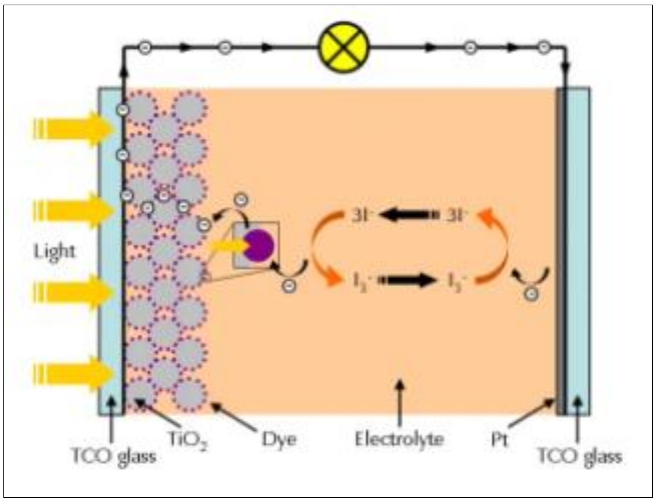

Figure 12. Illustrates the mechanism of action of a dye-sensitized solar cell.

Conclusions:

A new and developed method for preparing carbon nanotubes is presented in this research by using the original Hummers method, but with some changes to the work steps. Reduction and deposition of carbon nanotubes on ITO will be done by using the EDP method. CNTs are prepared and deposited on ITO which is used as a cathode electrode for the manufacture of dye-sensitized solar cells, which that give a high conversion efficiency of approximately 4.93.

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Authors' declaration:

-Conflicts of Interest: None.
-We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
-Ethical Clearance: The project was approved by the local ethical committee in department of Environmental Engineering, University Al-Turath College, Iraq.

Authors' contributions statement:

The first author prepared the carbon nanotubes and interpreted the results, as well as the preparation and measurement of the dye solar cell. The second author drew the curve between voltage and current (i-v plot) from which V_{ocp}, I_{max}, V_{max}, I_{sc}, and from these parameters we can calculate the full factor (ff) and energy conversion efficiency were calculated.

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تحضير انابيب الكاربون النانوية باستخدام طريقة مطورة واستخدامها كقطب محيط في صناعة خلية شمسية صبغية

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الخلاصة:

في هذا العمل ،تم تحضير الأنابيب الكاربون النانوية باستخدام طريقة هامرز(Hummers) مع تغيير طفيف في بعض خطوات العمل، وبالتالي أنشأنا طريقة جديدة بتحضير الأنابيب الكاربون النانوية ، على غرار طريقة هامرز(Hummers) الأصلية التي تم استخدامها لتحضير اوكسيد الجرافين ، بعد ذلك تم تحضير معلق من الأنابيب الكاربون النانوية ونقلها إلى خلية كهروكيميائية بسيطة مكونة من قطبين لترسيب واختزال وطلاء الأنابيب الكاربون النانوية على قطب المهبط (cathode) مصنوع من زجاج الموصل(ITO) بينما كان قطب المصعد (anode) عبارة عن رقيقة البلاتين.تم تشخيص الأنابيب الكاربون النانوية بواسطة تقنية المجهر الإلكتروني الماسح(SEM), أظهرت هذه الصور تكوين الأنابيب النانوية الكربونية.في الجزء الثاني من البحث ، تم تحضير خلية شمسية صبغية (DSSCs) باستخدام أنابيب الكربون النانوية كقطب محيط (counter) ، وجسيمات أوكسيد التيتانيوم النانوية كقطب مصعد (anode) وباستخدام صبغة طبيعية (صبغة الرمان). بعد ذلك تم فحص الخلية بواسطة المجهاد الساكن ذو القطبين باستخدام مصدر ضوء الزينون حيث تم الحصول المعلومات التالية: جهد الدائرة المفتوحة (Vocp) ، دائرة مقصورة (I_{sc}), الفولتية العظمى (V_{max}), التيار الاعظم (I_{max}) ومن هذه المعلومات تم حساب العامل الكامل(full facter) وكفاءة الخلية.

الكلمات المفتاحية: انابيب الكاربون النانوية، الخلايا الشمسية الصبغية الحساسة، طريقة الترسيب الكهربائي، طريقة مطورة، صبغة طبيعية .