



**GRAPHENATED CARBON NANOTUBES-BASED COUNTER ELECTRODE FOR  
DYE-SENSITIZED SOLAR CELLS**

By

**YUSNITA BINTI YUSUF**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in  
Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

**June 2023**

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*This thesis is dedicated to my beloved parents; Siti Ausun Hj. Saibi and late father Mahmud Zamhari, mother in law Rohani Khalib, late father in law Zainudin Nagin for their endless love, financial support and encouragement. To my beloved husband, Dr. Afiq Azri Zainudin who always encouraged me until this work was completed, thank you for always being my rainbow after the storm. Without your guidance, I would never complete this work.*

*May Allah bless all of you with mercy and barakah, and reward you with a home in Jannah.*



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in  
fulfilment of the requirement for the degree of Doctor of Philosophy

## **GRAPHENATED CARBON NANOTUBES-BASED COUNTER ELECTRODE FOR DYE-SENSITIZED SOLAR CELLS**

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**June 2023**

**Chairman : Professor Ir. Suhaidi bin Shafie, Ph.D**  
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The counter electrode is one of the most critical components in the dye-sensitized solar cell (DSSC). It catalyzes the reduction of iodide/tri-iodide in the electrolyte, bringing the electrons from external loads connected to the cells. A conventional platinum counter electrode is extensively used; however, the concern with platinum-based is that expose to corrosion in an iodine-based electrolyte, which affects the long-term stability of the cell. Therefore, using the carbon-based material to replace the platinum-based in DSSC can address the mentioned problems. This work synthesized a graphenated-carbon nanotube (g-CNT) via the floating-catalyst chemical vapor deposition method. Then, the g-CNT paste was prepared and deposited for the counter electrode. The morphological results revealed that the g-CNT8 obtained 34.5 S/cm, forming a highly conductive network due to graphene foliates at the sidewalls of CNT. This excellent finding is due to the hybrid structure of the g-CNT8, which provides a high defect structure that creates efficient electron transfer in the materials resulting in higher conductivity. For the counter electrode DSSC, briefly, GCC500 film provided good electrical conductivity of 6.28 S/cm. In addition, the GCC500 counter electrode offered excellent catalytic activity for the iodide/triiodide reaction. That is a significant feature in employing counter electrodes to enhance DSSC performance. Furthermore, the DSSC-based GCC500 exhibited 5.68 % of photovoltaic conversion energy, much higher than platinum (3.79 %). Therefore, the GCC500 is an excellent candidate to replace the conventional platinum as a counter electrode in DSSC.

**Keywords:** g-CNT; carbon-based; counter electrode; DSSC

**SDG:** GOAL 7: Affordable and Clean Energy

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia  
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

## **ELEKTROD BERLAWANAN DENGAN TIUB NANO KARBON BERGRAFENA UNTUK SEL SURIA TERPEKA PEWARNA**

Oleh

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Elektrod berlawanan adalah salah satu komponen paling kritikal dalam sel suria terpeka pewarna (DSSC). Ia memungkinkan pengurangan iodin/tri-iodida yang membawa elektron daripada beban luaran yang disambungkan kepada sel. Elektrod berlawanan platinum konvensional digunakan secara meluas, namun, kebimbangan terhadap bahan berasaskan platinum adalah terdedah kepada kakisan dalam elektrolit berasaskan iodin, yang menjejaskan kestabilan jangka panjang sel. Oleh itu, penggunaan bahan berasaskan karbon untuk menggantikan platinum dalam DSSC dapat menyelesaikan masalah tersebut. Kajian ini mensintesis tiub nano karbon-grafen (g-CNT) melalui kaedah pengendapan wap kimia pemangkin terapung. Kemudian, pes g-CNT disediakan untuk elektrod berlawanan. Hasil morfologi menunjukkan bahawa g-CNT8 mencapai 34.5 S/cm membentuk rangkaian yang sangat konduktif kerana pertumbuhan foliat grafen di dinding sisi CNT. Penemuan cemerlang ini adalah disebabkan oleh struktur hibrid g-CNT8 yang menyediakan struktur kecacatan yang tinggi untuk menghasilkan pemindahan elektron yang cekap dalam bahan, lantas meningkatkan kekonduksian yang lebih tinggi. Untuk elektrod berlawanan dalam DSSC, secara ringkas, filem GCC500 memberikan kekonduksian elektrik yang tinggi 6.28 S/cm. Selain itu, GCC500 menawarkan aktiviti pemangkin yang sangat baik untuk tindak balas iodida/triiodida. Ini adalah ciri penting untuk meningkatkan prestasi DSSC. Tambahan pula, GCC500 menunjukkan 5.68% kecekapan penukaran tenaga, jauh lebih tinggi daripada platinum (3.79%). Oleh itu, GCC500 merupakan calon terbaik untuk menggantikan platinum konvensional sebagai elektrod berlawanan dalam DSSC.

**Kata Kunci:** g-CNT; berasaskan karbon; elektrod berlawanan; DSSC

**SDG:** MATLAMAT 7: Tenaga Mampu Milik dan Bersih

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## LIST OF ABBREVIATIONS

0D	Zero dimensional
1D	One dimensional
2D	Two dimensional
3D	Three dimensional
Ar	Argon
C	Carbon
CB	Conduction Band
CdTe	Cadmium telluride
CE	Counter electrode
CIGS	Copper indium gallium selenide
CNTs	Carbon Nanotubes
CV	Cyclic Voltammetry
DSSC	Dye-sensitized solar cells
EDX	Energy Dispersive X-ray Spectroscopy
FCCVD	Floating-Catalyst Chemical Vapor Deposition
FESEM	Field Emission Scanning Electron Microscopy
FF	Fill factor
FTO	Fluorine-doped Tin Oxide
GCC	Graphenated-carbon nanotubes counter electrode
G-CNT	Graphenated-carbon nanotubes
H <sub>2</sub>	Hydrogen
HOMO	Highest Occupied Molecular Orbital
HRTEM	High Resolution Transmission Electron Microscopy
IEA	International energy agency
LUMO	Lowest Unoccupied Molecular Orbital
NREL	National Renewable Energy Laboratory

Pt	Platinum
PtI <sub>4</sub>	Platinum iodide
RGO	Reduced graphene oxide
Sccm	Standard cubic centimeters per minute
SnO <sub>2</sub>	Tin (IV) Oxide
TCO	Transparent conductive oxide
Ti	Titanium
TiCl <sub>4</sub>	Titanium Tetrachloride
TiO <sub>2</sub>	Titanium dioxide
UV	Ultraviolet
UV-Vis	Ultra Violet Visible Spectroscopy
$\pi$	Pi bond
$\sigma$	Sigma bond



# CHAPTER 1

## INTRODUCTIONS

### 1.1 Background

Emerging economies drive the world's growing demand for energy and an increasing population, especially in developing countries [1]. That leads to global warming resulting from the large-scale emission of the greenhouse effect and air pollution, which can be caused countless illnesses [2]. Nonrenewable energy sources such as petroleum, natural gas, coal, and fossil fuels are used in daily activities [3]. Many strategies have been implemented to tackle the global issue, such as green, sustainable, and energy-efficient [4], [5].

Sustainable and renewable energy resources such as biomass, tidal energy, hydro energy, solar thermal, and solar cell are among the potential option for renewable energy and the most efficient [6]. The most potential among renewable energy is solar cell technology (photovoltaic). This technology has received worldwide attention and turned into a billion, especially in industry. The International Energy Agency (IEA) has predicted that 30% of solar energy is projected to supply the world's power in 2050 and is supposed to increase up to 60% of electricity in 2100 [7].

A solar photovoltaic (PV) system generates electrical power from the sunlight. Semiconducting layers are often referred to as silicon-based PV systems. It is composed of  $p$ -type and  $n$ -type components forming a  $p$ - $n$  junction. The semiconducting material absorbs the incident photons when the system is exposed to sunlight, creating positive or negative charges. The  $n$ -type semiconductor tends to gather electrons, whereas the  $p$ -type semiconductor tends to collect holes. The electricity will readily flow into the cell if an external load is present. Becquerel discovered the PV effect in 1839 through his research on how light impacts electrolyte cells [8], [9]. Later, in 1954, Bell in the United States developed a PV solar cell that was deployed to supply electricity for space satellites [10].

Solar cells can be classified into three generations: first, second, and third. The first-generation solar cells are wafer silicon-based, amorphous silicon, single-crystalline and poly-crystalline silicon, and hybrid silicon cells. Silicon photovoltaics commonly demonstrated 15% to 25% energy conversion efficiency, leading to the photovoltaic market. The advantages of this solar cell technology are good performance and high stability. However, they require an

expensive production cost to build the module technology that demands high-purity silicon.

The second-generation solar cells are made from thin-film semiconductor-based amorphous silicon, cadmium telluride (CdTe), copper indium gallium selenide (CIS or CIGS). They usually recorded 12% to 15% of energy efficiency. The thin film has offered a potential reduction of cost using the active materials between two pieces of glass and flexible cells. However, it still requires high temperatures, complicated module technology, and poor stability, and some materials like cadmium used in thin-film solar cells are toxic to humans.

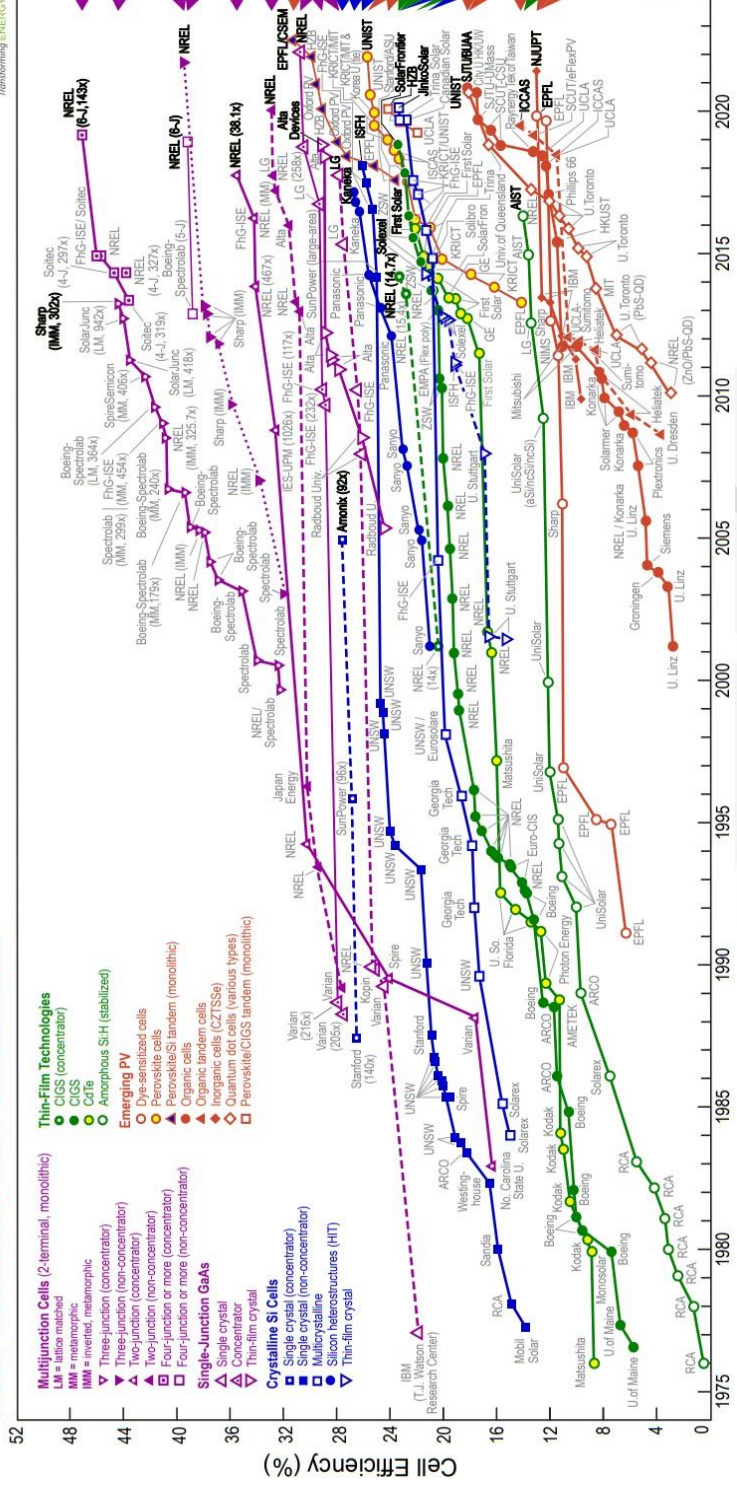
Meanwhile, third-generation solar cells are often described as a new thin-film emerging technology, including organic photovoltaics, polymer photovoltaics, perovskite photovoltaics, dye-sensitized solar cell, and quantum dots solar cells. Yet, low energy efficiency than Si-based solar cells, third-generation solar cells have offered the cheapest photovoltaic technology, are friendly to the environment, and typically demonstrate an energy efficiency of 11% to 14.1 % [11]–[13]. Most recently, the Grätzel group has exhibited a high power conversion efficiency of 15.2 %, which is the new record in DSSC TiO<sub>2</sub>-based and co-adsorbed sensitizers [14].

An effort is being made to develop third-generation solar cells, which it is hoped will soon replace existing solar technology and address its shortcomings. Generally speaking, this technology consists of non-semiconductor (polymer-based and biomimetic) cells, tandem/multi-junction cells, hot-carrier cells, dye-sensitized solar cells (DSSC), and up-conversion technologies. It has attracted attention because its benefit lies in easy fabrication, low-cost production, light, affordable source of renewable energy, outstanding performance under low light conditions, and several options for improving power conversion efficiency [13], [15]–[19].

The dye-sensitized solar cells (DSSC) were invented by Brian O'Regan and Micheal Grätzel in 1991 [20], [21]. The utmost components for common DSSC are wide band gap semiconductor, sensitizer to act as photoanode, catalyst, and redox couple. The photoanode, usually a dye particle coated with the nano porous metal oxide semiconductor film, was deposited on the transparent conductive oxide (TCO) glass as the photoanode, platinum (Pt) catalyst was deposited on TCO substrates as a counter electrode and redox mediator iodide/triiodide electrolyte.

The revolution of solar cells in industry and the prediction of solar cell growth around the world showed in Figure 1.1. The chart reports the highest confirmed conversion efficiencies for different solar cell technologies as written by the National Renewable Energy Laboratory (NREL), the USA to the date of September 2022.

## Best Research-Cell Efficiencies



**Figure 1.1: Chart of highest efficiencies obtained from solar cells of different photovoltaic technologies** (Source: <https://www.nrel.gov/pv/cell-efficiency.html>)

## 1.2 Problem Statements

The counter electrode (CE) is one of the most critical components in the DSSC. It catalyzes the Iodide/Tri-iodide ( $I_3^-/I^-$ ) redox reaction which bring the electrons from external loads connected to the cells and catalyze the  $I_3^-/I^-$  reaction in the electrolyte. Conventional platinum (Pt) CE is extensively used due to its outstanding conductivity and electrocatalytic ability [21]. However, Pt-based CE is a precious metal and obtains low resistance toward corrosion in an iodine-based electrolyte, which decomposes the Pt into platinum iodide ( $PtI_4$ ) [22].

These led many researchers to explore other low-cost materials with low resistance and excellent electrocatalytic ability. For instance, carbonaceous [23], [24], alloys [25], [26], conducting polymers [27], [28], and composites [29]–[31] have been developed as Pt-free CE.

Recently, extensive studies have investigated carbon-based CE materials, such as carbon nanotubes (CNT) [23], carbon fibres [32], [33], carbon black [34], graphene, and graphite [35], [36]. Carbon-based materials are chemically stable in iodine-based electrolytes and do not degrade when exposed to iodine. It also provides active catalytic sites at their edges. Nevertheless, many carbon materials with good catalytic properties do not have the competence for electron transfer. Therefore, initiating the hybrid carbonaceous materials will improve the charge transfer of conducting electrons. CNT alone does not show good improvement in conductivity; hence hybrid can enhance electrical conductivity. Graphene tends to form stacking phenomena, leading to stronger interlayer  $\pi$ - $\pi$  stacking and van der Waals interactions that prevent electron transport and iodide/triiodide ion transfer producing active defect region. It causes an increase in internal resistance and diffusion resistance of the redox reaction. Hybrid can prevent stacking, thereby improving the conductivity of the materials.

The hybrid properties of the graphenated-carbon nanotube structure are hypothesized to provide a way to optimize the hybrid structure by creating a bridging for electrons to improve the conductivity. High defect of basal and edges plane enhancing electrons transfer by electron bridging lead to increasing conductivity. That allowed for superior electronic conductivity and catalytic activity than any of the two materials could achieve independently.

## 1.3 Objectives

This research aims to achieve high power conversion efficiency dye-sensitized solar cells by implementing graphenated-carbon nanotubes cotton as a counter electrode in dye-sensitized solar cells. The working objectives that have been considered in this research are:



- i. To synthesize and characterize graphenated-carbon nanotubes via the floating-catalyst chemical vapor-deposition method and determine the structural and morphological properties.
- ii. To investigate the graphenated-carbon nanotubes films on electrical and electrocatalytic properties.
- iii. To implement and evaluate the graphenated-carbon nanotubes-based counter electrode in DSSC for high power conversion DSSC.

#### **1.4 Scope and Limitations**

This research aimed to examine the performance of carbon-based counter electrodes for DSSC. Since the study focuses only on implementing graphenated/CNT-based counter electrodes, the photoanode  $\text{TiO}_2$ -FTO coated and Pt-based counter electrodes serve as a benchmark for the conventional DSSC. The approached material, hybrid graphenated/CNT (g-CNT) was synthesized and characterized, which would later be used to fabricate a counter electrode. During the g-CNT synthesis, the carbon sources injection rate was fixed at 6 ml/hr and 8 ml/hr due to less formation of g-CNT under 6 ml/hr, whereas at more than 8 ml/hr there is no formation of graphene foliates. Therefore, the selection of injection rate in this work is limited to as stated and other than that injection rates are out of the research scope. As there are different types of electrolytes and dyes, this research focuses on Iodine-electrolyte and synthetic N719 dye. DSSC fabrication and characterization are based on conventional Pt-based counter electrodes and the proposed graphenated/CNT-based counter electrodes. The photoanode fabrication procedure is optimized and characterized, but all parameters related to the photoanode are still out of the research scope.

#### **1.5 Thesis Contents**

The layout of this thesis is: Chapter 1 reviews the background of the study. It also explains solar cell generation and DSSC and enlightens on the previous research that led to the problem statement of this study. Chapter 2 is a literature review of the previous research related to the background on the improvement of DSSC. Besides, the explanation of the operation principles and device structure of DSSC. Chapter 3 explains the research methodology. This chapter briefly describes the method, equipment, and materials used in this research. The synthesis of GCC, preparation of carbon paste for counter electrode, and DSSC fabrication experimental setup involved in the DSSC characterization were explained in detail. Chapter 4 discusses the experimental results from the characterization techniques and analyzes the outcome, including morphological, structural, electrochemical, electrical properties and DSSC performance. Lastly, Chapter 5 concludes the finding and highlights the contribution of this research and recommendations for future research to improve the DSSC performance.

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