



UNIVERSITI  
MALAYSIA  
KELANTAN

**Effect of Annealing Temperatures on Carbon-TiO<sub>2</sub> Counter Electrode**

**Muhammad Khairul Amirun bin Norhisham  
J20A0518**

**A report submitted in fulfilment of the requirements for the  
degree of Bachelor of Applied Science (Materials Technology)  
with Honours**

**FACULTY OF BIOENGINEERING AND TECHNOLOGY  
UMK**

**2024**

UNIVERSITI  
MALAYSIA  
KELANTAN

**DECLARATION**

I declare that this thesis entitled “**Effect of Annealing Temperatures on Carbon-TiO<sub>2</sub> Counter Electrode**” is the results of my own research except as cited in the references.

Signature : \_\_\_\_\_

Student's Name : MUAMMAD KHAIRUL AMIRUN BIN NORHISHAM

Date : 2 FEBRUARY 2024

Verified by:

Signature : \_\_\_\_\_

Supervisor's Name : DR HIDAYANI BINTI JAFFAR

Stamp : \_\_\_\_\_

Date : 02 February 2024

## ACKNOWLEDGEMENT

I am grateful to my supervisor, Dr. Hidayani Binti Jaafar, for her encouragement, support, and direction throughout my studies and thesis writing process. I sincerely appreciate the time spent. Supported and corrected my mistakes throughout my undergraduate studies. I'd want to express my gratitude to my family members for their encouragement and support throughout my university studies. I am grateful to my best buddy Muhammad akim, Muhammad Afif, Hidayah, Muhd Faiz for their unending moral support and kindness in assisting me with my education.

Next, I would like to thank the lecturers and staff at UMK Faculty of Bioengineering and Technology for their cooperation and assistance in analysing testing samples. Finally, I'd want to thank everyone who helped, whether directly or indirectly.

UNIVERSITI  
MALAYSIA  
KELANTAN

## Effect of Annealing Temperatures on Carbon-TiO<sub>2</sub> Counter Electrode

### ABSTRACT

This research focuses on synthesizing and characterizing carbon-TiO<sub>2</sub> composites for Dye-Sensitized Solar Cells (DSSCs). Thermogravimetric analysis (TGA) revealed a significant weight loss during decomposition, guiding the selection of annealing temperatures (300°C, 550°C and 600°C). Absorption spectrum analysis showed superior light absorption for the 600°C composite. Tauc plot analysis revealed distinct energy band gaps for each annealing temperature. XRD analysis confirmed anatase crystalline phase with reduced crystal size due to carbon doping. Photovoltaic performance shows a correlation between annealing temperature and efficiency, with the 600°C composite exhibiting the highest efficiency. This study highlights the effect of annealing parameters and carbon source on the structural, optical, and photovoltaic properties of carbon-TiO<sub>2</sub> composites. This research provides fundamental insights for future advances in carbon-TiO<sub>2</sub> synthesis for enhanced DSSC efficiency. Recommendations include exploring alternative carbon sources and optimizing synthesis conditions.

Keyword: Carbon-TiO<sub>2</sub>, Dye-Sensitized Cell, Photovoltaic

UNIVERSITI  
MALAYSIA  
KELANTAN

## Mengesan Suhu Penyepuhlindapan pada Elektrod Pembilang Karbon-TiO<sub>2</sub>

### ABSTRAK

Penyelidikan ini memberi tumpuan kepada mensintesis dan mencirikan komposit karbon-TiO<sub>2</sub> untuk Sel Suria Peka Pewarna (DSSC). Analisis termogravimetrik (TGA) mendedahkan penurunan berat badan yang ketara semasa penguraian, membimbing pemilihan suhu penyepuhlindapan (300°C, 550°C dan 600°C). Analisis spektrum penyerapan menunjukkan penyerapan cahaya yang unggul untuk komposit 600°C. Analisis plot Tauc mendedahkan jurang jalur tenaga yang berbeza untuk setiap suhu penyepuhlindapan. Analisis XRD mengesahkan fasa kristal anatase dengan saiz kristal yang dikurangkan disebabkan oleh doping karbon. Prestasi fotovoltaiik menunjukkan korelasi antara suhu penyepuhlindapan dan kecekapan, dengan komposit 600°C mempamerkan kecekapan tertinggi. Kajian ini menyerlahkan kesan parameter penyepuhlindapan dan sumber karbon ke atas sifat struktur, optik dan fotovoltaiik komposit karbon-TiO<sub>2</sub>. Penyelidikan ini memberikan pandangan asas untuk kemajuan masa depan dalam sintesis karbon-TiO<sub>2</sub> untuk kecekapan DSSC yang dipertingkatkan. Syor termasuk meneroka sumber karbon alternatif dan mengoptimumkan keadaan sintesis.

Kata kunci: Carbon-TiO<sub>2</sub>, Dye-Sensitized Cell, Photovoltaiic

UNIVERSITI  
MALAYSIA  
KELANTAN

## Table of Contents

<b>DECLARATION</b> .....	i
<b>ACKNOWLEDGEMENT</b> .....	ii
<b>ABSTRACT</b> .....	iii
<b>ABSTRAK</b> .....	iv
<b>LIST OF ABBREVIATIONS</b> .....	vii
<b>CHAPTER 1</b> .....	1
<b>INTRODUCTION</b> .....	1
<b>1.1 Background of Study</b> .....	1
<b>1.2 Problem Statement</b> .....	4
<b>1.3 Objective</b> .....	5
<b>1.4 Expected Output</b> .....	5
<b>1.5 Scope of study</b> .....	6
<b>1.6 Significant of study</b> .....	6
<b>CHAPTER 2</b> .....	7
<b>2.1 Introduction</b> .....	7
<b>2.2 Definition and Structure Dssc</b> .....	7
<b>2.3 The Operating Theory Behind Solar Cells and Their Primary Component Materials</b> .....	7
<b>2.4 Counter Electrode</b> .....	8
<b>2.5 Titanium Oxide, TiO<sub>2</sub></b> .....	9
<b>2.4 Carbon</b> .....	10
<b>Chapter 3</b> .....	12
<b>Material and Methods</b> .....	12
<b>3.1 Introduction</b> .....	12
<b>3.2 Research flow</b> .....	12

<b>3.3 Sample Preparation</b> .....	13
<b>3.3.1 Annealing Temperature of C-TiO<sub>2</sub></b> .....	13
<b>3.4 Preparation of C-TiO<sub>2</sub></b> .....	14
<b>3.7 Sample characterization</b> .....	14
<b>Chapter 4</b> .....	15
<b>RESULTS AND DISCUSSION</b> .....	15
<b>4.1 Introduction</b> .....	15
<b>4.5 Modified of working Photoanode for DSSCs application</b> .....	22
<b>CHAPTER 5</b> .....	25
<b>5.1 Conclusion</b> .....	25
<b>5.2 Recommendation</b> .....	25
<b>REFERENCE</b> .....	27
<b>APPENDIX A</b> .....	29

**LIST OF ABBREVIATIONS**

TiO <sub>2</sub>	Titanium Dioxide
C	Carbon
ITO	Indium-Tin-Oxide
XRD	X-ray diffraction
UV-vis	Ultraviolet-Visible
TGA	Thermogravimetric Analysis

UNIVERSITI  
MALAYSIA  
KELANTAN



**LIST OF SYMBOLS**

%	Percentage
°C	Degree Celsius
eV	Electron Volt
Å	Angstrom
nm	Nanometre
mA	Milliampere
mV	Millivolt

UNIVERSITI  
MALAYSIA  
KELANTAN

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

The Carbon-TiO<sub>2</sub> composite material has also proven useful in dye-sensitized solar cells in the role of the counter electrode (DSSCs). The counter electrode in DSSCs is typically made of platinum, which is both pricey and not good for the environment. On the other hand, carbon-titanium dioxide is a more cost-effective and environmentally friendly alternative.

The composite material known as "carbon-titanium dioxide" (or "C-TiO<sub>2</sub>") is made up of carbon and titanium dioxide (TiO<sub>2</sub>). Due to its unique characteristics, this material can be used in a wide variety of contexts. For instance, in photocatalytic processes, where organic molecules and pollutants in water and air are broken down, carbon-TiO<sub>2</sub> is utilized as a catalyst to speed up the process. As a result of its high conductivity and high surface area, it is also utilized in the production of devices that store energy, such as lithium-ion batteries.

The presence of carbon in the composite not only contributes to the improvement of the DSSC's performance but also has a positive effect on the electrical conductivity of the material. In addition, the carbon-TiO<sub>2</sub> composite can be easily prepared by using methods that are not only straightforward but also scalable. In general, the utilization of C-TiO<sub>2</sub> as a counter electrode in DSSCs offers a promising solution for the development of solar cell technologies that are more effective, cost-effective, and friendly to the environment.

In the field of materials science, annealing is a process that is utilized to change the properties of various materials, including metals. It entails heating a material to a

high temperature and then slowly cooling it to modify its internal structure and improve its mechanical properties. The goal of the process is to make the material more durable.

The use of carbon as a material in DSSCs, especially in counter electrodes, offers several advantages. This is because there are several reasons why carbon is usually used first. Cost-effectiveness: Carbon-based materials, this is said because such as carbon black, graphite, or carbon nanotubes, are generally more affordable than precious metals such as platinum (Pt). This makes carbon electrodes a more cost-effective option, which is beneficial for large-scale production and reduces the overall cost of DSSCs.

Next is Good Electrical Conductivity, Carbon materials have excellent electrical conductivity, allowing them to efficiently collect and transport electrons from external circuits. This property ensures a high electrical contact between the counter electrode and the electrolyte, facilitating the reduction of redox couples in the electrolyte. Carbon materials can also exhibit catalytic activity, promoting the reduction of redox couples in electrolytes. Although carbon is not as efficient as platinum in terms of catalytic performance, it can still provide sufficient catalytic activity for DSSC operation. Further stability: Carbon-based counter electrodes generally show good stability in the DSSC environment, especially when compared to certain metal electrodes that may be prone to corrosion or degradation.

The stability of the carbon electrode ensures the long-term performance and durability of the DSSC. Although platinum remains the most efficient and widely used material for DSSC counter electrodes due to its superior catalytic properties, carbon-based materials offer a more cost-effective and stable alternative, making them suitable for many practical DSSC applications. However, it is important to note that ongoing

research and development in this field continues to explore alternative materials and improve the performance of carbon electrodes for DSSCs.

The process of annealing entails heating a material to a temperature higher than the temperature at which it would recrystallize. This causes the atoms contained within the material to rearrange themselves into a configuration that is more stable. Because of this, the material may experience fewer flaws and dislocations, which, in turn, results in an improvement in its ductility, toughness, and strength.

The microstructure of metals, such as steel, copper, and aluminum, and the microstructure of other materials, such as ceramics and glass, can be altered by annealing. To improve the qualities of the materials that are used in production, it is utilized in a wide range of industries, including the aerospace, automotive, and electronic product manufacturing sectors.

The term "annealing" refers to several distinct processes, the most common of which are complete, stress relief, and recrystallization. The characteristics of the material being annealed and the outcomes that are wanted are considered while selecting the parameters unique to each form of annealing. These parameters include the heating and cooling rates, temperature ranges, and holding times.

Depending on the material that needs to be treated, there are a few distinct annealing techniques to choose from. When carbon and  $\text{TiO}_2$  are annealed, the processes and needs that are necessary will be different.

During the annealing process for  $\text{TiO}_2$ , the material is heated to a specified temperature range (usually between 500 and 1000 degrees Celsius) for a certain amount of time. The amount of time required varies depending on the output that is sought. After going through the heating process, the material will then go through a period of controlled cooling, which will allow it to crystallize into the correct shape. The

atmosphere present during the annealing process can affect the qualities of the  $\text{TiO}_2$  material produced, and this is true whether the procedure is done in air or in an inert gas.

In conclusion, the annealing procedure for carbon and  $\text{TiO}_2$  can be different depending on the result intended and the characteristics of the material being treated. While determining the suitable annealing procedure, it is essential to consider the demands of the material in question and the application.

## 1.2 Problem Statement

The presented problem statement is about the influence that the temperature of annealing has on the  $\text{TiO}_2$  activated carbon counter electrode. This is since activated carbon  $\text{TiO}_2$  counter electrodes are frequently utilized in dye-sensitized solar cells. These electrodes have demonstrated excellent results in terms of their electrocatalytic activities and their stability. Adjusting the annealing temperature while the counter electrode is being prepared allows for additional optimization of its properties, which can help achieve a better overall result.

In addition, the preparation of C- $\text{TiO}_2$  composites can be a difficult and pricey procedure that necessitates the use of specialist equipment and the experience of qualified personnel. Because of this, their broad use and financial feasibility may be constrained, particularly in applications of a large scale.

in addition, C- $\text{TiO}_2$  has a poor adherence to the substrate and a moderately low mechanical strength. This may provide a challenge when attempting to disable it on a substrate for a particular application or when employing it in an application that requires it to be structural. This is because carbon is Versatility. Activated carbon is a versatile material that can be used in numerous applications across different industries. It is utilized in water treatment, air purification, gas, and vapor filtration, decolorization,

odour removal, chemical and pharmaceutical processes, and more. Its adsorption capabilities make it suitable for a wide range of purification and separation processes.

In conclusion, the performance of composites made of  $\text{TiO}_2$ , and carbon may be influenced by several parameters. These elements include the purity and quality of the starting material, the processing conditions, and the surface morphology and structure of the composite material. For  $\text{TiO}_2$  carbon composites to attain their maximum potential in terms of performance and stability across a variety of applications, careful management of the parameters is required.

### 1.3 Objective

The objective of this study is to:

- To determine the temperature variation on properties of C- $\text{TiO}_2$ .
- To study effect of annealing temperature on C- $\text{TiO}_2$  as counter electrode
- To determine performance of photovoltaic of activated

### 1.4 Expected Output

The expected output using C- $\text{TiO}_2$  as a counter electrode in a dye-sensitized solar cell (DSSC) can vary depending on several factors such as the combination of Carbon and  $\text{TiO}_2$  provides enhanced stability to the counter electrode. It helps reduce degradation and extend the lifespan of DSSCs. However, here are some potential releases to look forward to.

### 1.5 Scope of study

The scope of research on the use of C-TiO<sub>2</sub> as a counter electrode can include various aspects related to its application in different fields, especially in energy conversion devices such as dye-sensitized solar cells (DSSC). for example, Performance appraisal. this is because assessing the performance of C-TiO<sub>2</sub> as a counter electrode in DSSCs, including efficiency, stability, charge transfer kinetics and charge recombination loss reduction. Next, Examine the Electrochemical behaviour, electrochemical properties of the C-TiO<sub>2</sub> counter electrode, including its redox reaction, catalytic activity, and electron transport properties. This is because it is important to note that the specific scope of the study can be adjusted based on the research objectives, available resources and the level of depth desired for the investigation.

### 1.6 Significant of study

The study of the use of C-TiO<sub>2</sub> as a counter electrode holds important importance because it can be known.

Performance and efficiency of using C-TiO<sub>2</sub> as a counter electrode in energy conversion devices, such as DSSC, can lead to better performance and efficiency of these devices.

This study can provide insight into the enhanced catalytic activity, charge transfer kinetics, and reduced charge recombination loss offered by C-TiO<sub>2</sub> composites, contributing to more efficient solar energy conversion.



## CHAPTER 2

### 2.1 Introduction

This chapter discusses the definition and structure of DSSC. Functions and the next electrode characteristics are discussed in depth. Moreover, the effect is reported using  $\text{TiO}_2$  as the counter electrode. Also, some sintering temperature was explored to ascertain the effect of temperature and  $\text{TiO}_2$  concentration.

### 2.2 Definition and Structure Dssc

One variety of solar cells that belongs to the third generation is called DSSC. The basic idea behind how the DSSC works comes from the process of photosynthesis, which occurs naturally in plants. Since their first study in 1991, Gratzel and his colleagues have made significant progress in the development of the dye-sensitized solar cell (DSSC). This research indicates the benefit of producing the dye-sensitized photoanode by using nanocrystalline  $\text{TiO}_2$  layer (Rassi et al., 2020). DSSCs offer various benefits compared to the much more established silicon-based solar cells. These benefits include a quicker energy payback time, transparency, compatibility with flexible substrates, and most importantly, higher performance in low light settings, particularly artificial illumination.

### 2.3 The Operating Theory Behind Solar Cells and Their Primary Component Materials

All photovoltaic devices go through the same two primary processes to convert sunlight into usable electrical energy: (1) the process of radiation being absorbed by electrons; (2) the process of charge carriers being separated (Sengupta et al., 2017). DSSCs absorb light and separate carriers in a manner that is noticeably distinct from



how conventional p-n solar cells do these things. The typical semiconducting material in a solar cell is responsible for both the light absorption and the separation of charge carriers. Load separation in p-n solar cells take place because of the depletion layer located at the p-n junction.

On the other hand, with DSSCs, the light is absorbed by the dye molecules that are coupled to the semiconductor material (such as  $\text{TiO}_2$ ,  $\text{ZnO}$ , etc.). At the connections between the dye and the metal oxide, the load is separated.

The output voltage of the DSSC is determined by the energy difference between the quasi-Fermi level of the semiconductor and the redox-mediated redox potential while the DSSC's output voltage is determined by the redox-mediated redox potential under illumination. Having more dye mesoporous semiconductors in the photoanode (a larger surface area) leads to the creation of more photogenerated electrons and consequently it leads to an increase in DSSC efficiency. However, with an increase in the interface area between the photoanode and the electrolyte, the probability of recombination of semiconductor conduction electrons (or in any case trapping) and dispersed cations of the redox couple increases, which contributes negatively to the efficiency. Therefore, in order to obtain a good efficiency of the designed dye-sensitized solar cell, the dye in the photoanode should have a high visible light absorption coefficient, and the excited energy level of the dye and the conduction band level of the semiconductor should be suitable for good electron transfer from the excited dye to the band conduction of semiconductor materials, compatible redox potentials that allow effective dye regeneration, and faster counter electrode catalyzation.

## 2.4 Counter Electrode

In DSC, the counter electrode (Ce) is an essential component that is often manufactured by using a glass substrate that has a platinum film deposited on top of it.

The CE's primary responsibility is to perform the function of a catalyst, which it does by lowering the concentration of redox species that play the role of mediators in the process of either the regeneration of the sensitizer (dye) after electron injection or the collecting of holes from hole-transporting materials in a solid-state DSC. Increases in open-circuit voltage (VOC), short-circuit current density (JSC), and fill factor have been the primary focuses of the great majority of DSC research efforts over the past few years (FF). When deciding on a CE, many people go with a pt-coated FTO as their material of choice. When there is an improvement in the CE material, there is a noticeable increase in the fill-factor (FF) of the cells. The series resistance ( $R_s$ ) of the cell is the primary factor that determines the fill-factor (FF) of the cell. The slope of the tangent line that is drawn between current density (J) and voltage (V) at VOC is proportional to the value of  $R_s$ .

The difference in energy between the electron at the semiconductor and the CE, or the difference in energy level between the quasi-Fermi level of the semiconductor under illumination and the Nernst potential of the electrolyte solution, is what determines the VOC for a typical photoelectrochemical cell. Alternatively, the VOC can be determined by the difference in energy between the electron at the semiconductor and the CE. The DSSC can deliver the greatest voltage, denoted by VOC, to an external circuit even when no current is flowing.

## 2.5 Titanium Oxide, $\text{TiO}_2$

Titania ( $\text{TiO}_2$ ) is a sort of semiconductor material that has unique qualities. These properties include the material's low toxicity, low cost, high stability, and exceptional photocatalytic activity. Titania is a form of semiconductor material. This substance is also one of the most promising photocatalysts on the nanoscale (Jiang et al., 2012; Khalid et al., 2012; Poliah et al., 2011; Park et al., 2013).

Anatase, rutile, and brookite are the three distinct types of crystallographic forms that make up  $\text{TiO}_2$ , as stated by Khalid et al. (2012). Brookite is the least common of the three. A broad band gap of 3.2 eV can be seen in anatase-type pure  $\text{TiO}_2$ .  $\text{TiO}_2$ 's photocatalytic activity can be affected by a wide variety of parameters, such as the phase composition, crystallite size, morphology, specific surface area, and energy band gap, to name just a few. In addition to this,  $\text{TiO}_2$  can take on a few other morphologies, including spheres, hollow spheres, nanotubes, and nanowires.

## 2.4 Carbon

Carbon, also known as C, is a carbonaceous substance that is known to be porous and contains a significant number of micropores and mesopores that are open or accessible. Carbon is also known by its more common name, which is charcoal. C is delivered in the form of particulate matter, typically in the form of powder (particle size less than 100 m, average diameter less than 20 m) or granules (particle size ranging from 100 m to several mm). Carbon can be produced from a wide variety of inexpensive carbon-rich and low-inorganic content precursors, including but not limited to wood, lignite, coconut, peat, pistachio shells, walnut shells, saw dust, almond shells, coal, bituminous coal, chocolate coal, petroleum coke, and many more. According to T.J. Mays (1999), the primary considerations in the selection of raw materials for the synthesis of C include things like cost and availability, as well as carbon yield, inorganic content, and simplicity of activation. The final C qualities are determined by the initial materials utilized and the techniques that are followed during preparation. (Ting Lee, 2003)

For its role as a counter electrode, the high electrical conductivity of activated carbon is crucial. The counter electrode and DSCC electrolyte can exchange electrons effectively because of this. This improves the solar cell's overall performance by allowing for more effective dye regeneration and electron collecting. During the redox reaction taking place in the electrolyte, triiodide ions ( $\text{I}_3^-$ ) are reduced by the counter electrode to iodide ions ( $\text{I}^-$ ). By

acting as a catalyst, activated carbon can boost the speed of the redox process and the performance of the solar cell.

When compared to other electrode materials used in DSCC, activated carbon is surprisingly affordable. Its low cost makes it a promising candidate, particularly for mass manufacture of solar cells. The counter electrode can be reused thanks to the DSCC's ability to renew Carbon.



## Chapter 3

### Material and Methods

#### 3.1 Introduction

Due to their appealing qualities and uses, carbon and titanium dioxide ( $\text{TiO}_2$ ) used as a counter electrode in electrochemical systems have drawn interest. While  $\text{TiO}_2$  has good catalytic activity and stability, activated carbon offers a high surface area and conductivity. An overview of the components and procedures for creating a C- $\text{TiO}_2$  counter electrode is provided below.

Materials:

- carbon
- Titanium dioxide

#### 3.2 Research flow

The research flow chart illustrates the simplest procedures of preparation, characterisation, and fabrication portrayed in Table 3.1.

Step	Description
Powder Preparation (C- $\text{TiO}_2$ )	Preparation of carbon-doped titanium dioxide powder
Analysed by TGA	Thermogravimetric analysis to study mass changes
Annealing at 400, 550, and 600 °C	Heat treatment at different temperatures
Analysed by UV-Vis	Ultraviolet-Visible spectroscopy analysis
Characterization by XRD	X-ray Diffraction for crystallographic structure
C- $\text{TiO}_2$ Dye Thin Film Preparation	Preparation of thin films for dye absorption
Fabrication of DSSCs	Assembling Dye-Sensitized Solar Cells

Table 3.1: Research flow for  $\text{TiO}_2$ -C thin film and characterization

### 3.3 Sample Preparation

To prepare C-TiO<sub>2</sub> via milling, first gather titanium dioxide (TiO<sub>2</sub>) and a carbon source. These materials are then processed with equipment like ball mills. This milling method creates a homogeneous mixture and includes carbon into the TiO<sub>2</sub> structure. The duration and conditions of milling vary depending on the material and desired qualities.

After milling, the powder is typically dried to remove any residual moisture. Optionally, the C-TiO<sub>2</sub> powder can be annealed at a specific temperature to increase its characteristics. The third stage is to characterise the C-TiO<sub>2</sub> powder using techniques such as thermogravimetric analysis (TGA), X-ray diffraction (XRD), and ultraviolet-visible spectroscopy (UV-Vis) to better understand its thermal, structural, and optical properties.

Briefly, the preparation consists of milling the TiO<sub>2</sub> and carbon source, drying the powder, selective annealing, and finally characterising the resultant C-TiO<sub>2</sub> material.

#### 3.3.1 Annealing Temperature of C-TiO<sub>2</sub>

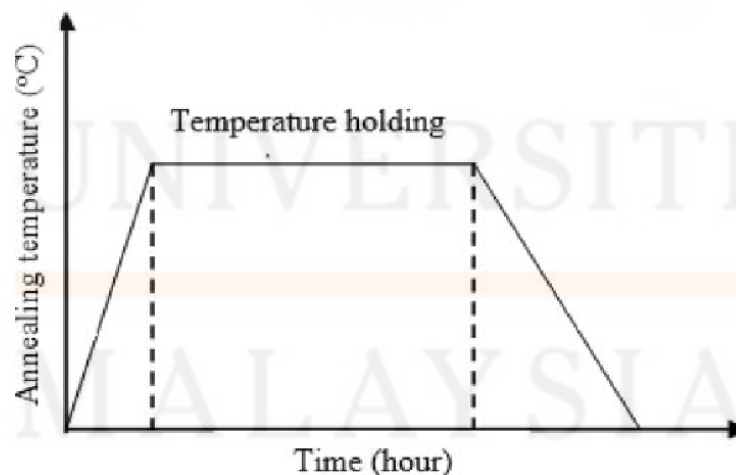


Figure 3.3: The annealing processes.

TiO<sub>2</sub> composite powder with C was annealed at different temperatures, 400°C, 550°C and 600°C, each with a time of 16 hours. as shown in Figure 3.3.

### 3.4 Preparation of C-TiO<sub>2</sub>

The initial step involves a thorough cleaning process for the ITO glass. It underwent washing for 10 minutes using detergent and distilled water, followed by sonication for 15 minutes in distilled water at 50°C. The glass was then treated with ethanol for an additional 15 minutes and then thoroughly dried using a desiccator.

Using the MT-1860 Pro digital Multi-meter Kit, the conductive parts of the indium tin oxide (ITO) coated glass were accurately identified. Placed with the conductive side facing up, the glass is carefully glued on all sides, maintaining about 0.5 cm from the edge.

A solution was prepared by dissolving 1.5g of carbon-TiO<sub>2</sub> in 6.4 ml of 70% ethanol, followed by 30 minutes of sonication. After that, the carbon-TiO<sub>2</sub> paste was carefully created by adding 2-3 drops of acetic acid (CH<sub>3</sub>COOH) and allowing it to stir for 1 hour. After this, a small quantity of the resulting carbon-TiO<sub>2</sub> paste is spread evenly on the ITO surface, with uniform distribution achieved using the doctor's knife technique. The resulting carbon-TiO<sub>2</sub> thin film is then stored in a dark environment at room temperature.

### 3.7 Sample characterization

The structural characteristics of the C-TiO<sub>2</sub> were determined by TGA and X-ray diffraction (XRD). The visible light emissions of all specimens were then determined using a Spectro quant Pharo 300 UV-vis spectrophotometer.



## Chapter 4

### RESULTS AND DISCUSSION

#### 4.1 Introduction

This chapter discussed the structure and properties of C-TiO<sub>2</sub> composites and were analyzed using by XRD and TGA spectra. Then, the absorption spectrum of TiO<sub>2</sub> composite with Carbon was examined for use as a thin film in DSSC fabrication. Natural dyes have been studied based on yield and UV-vis absorption spectra. Finally, photovoltaic parameters were discussed.





## 4.2 Characterization of Carbon-TiO<sub>2</sub> Composite

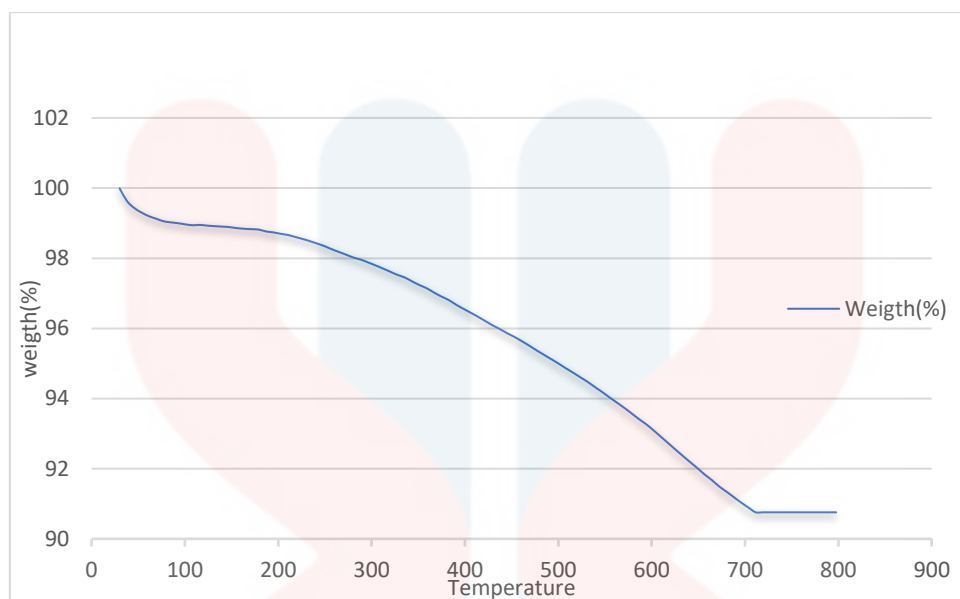


Figure 4.1: Result Thermogravimetric Analysis

Thermogravimetric Analysis (TGA) is a powerful technique for measuring the thermal stability of carbon-tio2. In this method, the change in the weight of the specimen is measured while its temperature is raised. The moisture and volatile content of the sample can be measured by TGA.

TGA of Carbon-TiO<sub>2</sub> sample weight ~ 10 mg was performed using Mettler Toledo 851e TGA/SDTA in pinhole aluminium crucibles at a heating rate of 10 °C/min from 25 to 800 °C under nitrogen purging (10 ml/min) (Fig. 4.1).

Figure 4.1 shows the initial weight loss observed in the TGA analysis of the Carbon-TiO<sub>2</sub> sample was approximately 98.9993% (w/w). This significant weight loss occurs in the temperature range of 25 to 100 °C and is attributed to the removal of water from the sample.

In Figure 4.1, the TGA thermogram discloses a weight loss of 97.7969%, commencing at 300°C and concluding at 700°C (90%) during the transition to carbon-TiO<sub>2</sub>, corresponding to the decomposition of carbon-TiO<sub>2</sub>.

Finally, the selection of temperatures at 300°C, 550°C and 600°C as parameters for the analysis is based on the need to investigate the main thermal events in the material. 300°C is chosen to observe initial stages, 550°C represents an intermediate temperature to capture significant transformations, and 600°C provides insight into potential high-temperature reactions. This temperature point was strategically chosen to study the thermal behaviour and decomposition of the carbon-TiO<sub>2</sub> composite.

### Absorption Spectra of Carbon-TiO<sub>2</sub> Composite

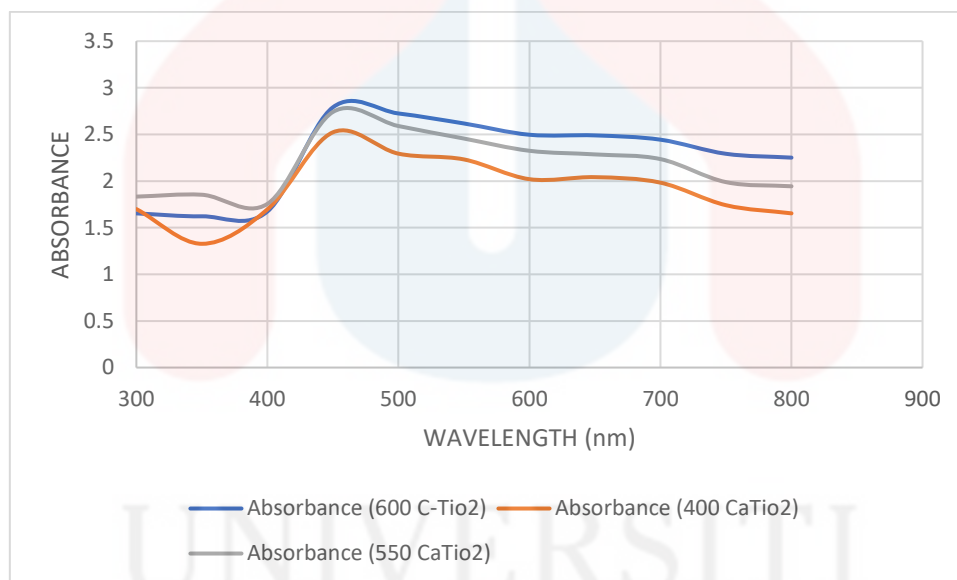


Figure 4.2: Spectra 1-3 were recorded for the same pellet placed differently in the holder. Linear fits for measurements a (red line), b (blue line) and c (green line) are superimposed.

Figure 4.2 shows that 600°C C-TiO<sub>2</sub> absorbs more light than C-TiO<sub>2</sub> 550°C, 400°C in the visible range (around 410-800 nm). This means that 600°C C-TiO<sub>2</sub> TiO<sub>2</sub> coatings can be obtained by mechanical mixing of TiO<sub>2</sub> with poly (vinyl chloride) and heating at temperatures between 600 and 800 °C in an inert atmosphere. Carbon coated TiO<sub>2</sub> particles were found to provide several advantages for TiO<sub>2</sub> catalysts. One of them is the increased adsorption on the

catalyst surface. Another is the prevention of the interaction between TiO<sub>2</sub> and the binding polymer, which easily decomposes under UV irradiation. The absorption peak of 550°C C-TiO<sub>2</sub> is around 450 nm. 400°C C-TiO<sub>2</sub> has a broader and weaker peak around 450 nm.

Table 1. Experimental Values Obtained from Direct Application of Tauc Plots 400°C C-TiO<sub>2</sub>, from Tauc Plots Applied to Differential Spectra 550°C C-TiO<sub>2</sub>, and from Simplified Analysis of Tauc Plots 600°C C-TiO<sub>2</sub>

SAMPLE	ABSORBANCE	ENERGY HV	BANGAP
400 CTio2	3.5	4.1	1.5
600 CTio2	3.844	2.75	1.8
550 CTio2	3.7	4.13	1.8

Table 4 show The Tauc plot analysis involves determining the absorption coefficient ( $\alpha$ ) using the formula.

$$\alpha = \frac{2.303}{d \times A}$$

where d is the sample thickness and A is the absorbance. Subsequently, the photon energy (hv) is calculated using.

$$hv = \frac{hc}{\lambda}$$

where h is Planck's constant, c is the speed of light, and  $\lambda$  is the wavelength.

At each wavelength (400 nm, 550 nm, and 600 nm), the respective absorbance values for C+TiO<sub>2</sub>, TiO<sub>2</sub>, and exposed carbon are used to calculate alpha (absorption coefficient), hv

(photon energy), and  $\alpha h\nu$ . Subsequently, Tauc plots are constructed by plotting  $\alpha h\nu$  against  $h\nu$  for each material.

Table 4 shows the 400°C C-TiO<sub>2</sub> trend is noted around 4.1 eV for energy ( $h\nu$ ) and showing a low level of absorption. This is because it can indicate the energy level at which Carbon-TiO<sub>2</sub> shows significant absorption characteristics. Then the Peak is observed at 3.5. This peak shows significant absorption or transition occurring at this peak level corresponding to the energy flow at 1.5 can provide insight into the electronic structure and optical properties of the carbon-TiO<sub>2</sub> composite.

Another showing the trend of 500°C C-TiO<sub>2</sub> recorded around 1.8 eV for energy ( $h\nu$ ). then the Peak is observed at 3.7. This peak shows a significant absorption or transition that occurs at the level of this peak corresponding to the energy flow at 1.5 eV. Analyzing these absorption characteristics provides insight into the optical properties of the composite. The specific energy levels and transitions observed are important for understanding how materials interact with light, which has implications for applications such as photocatalysis or solar energy conversion.

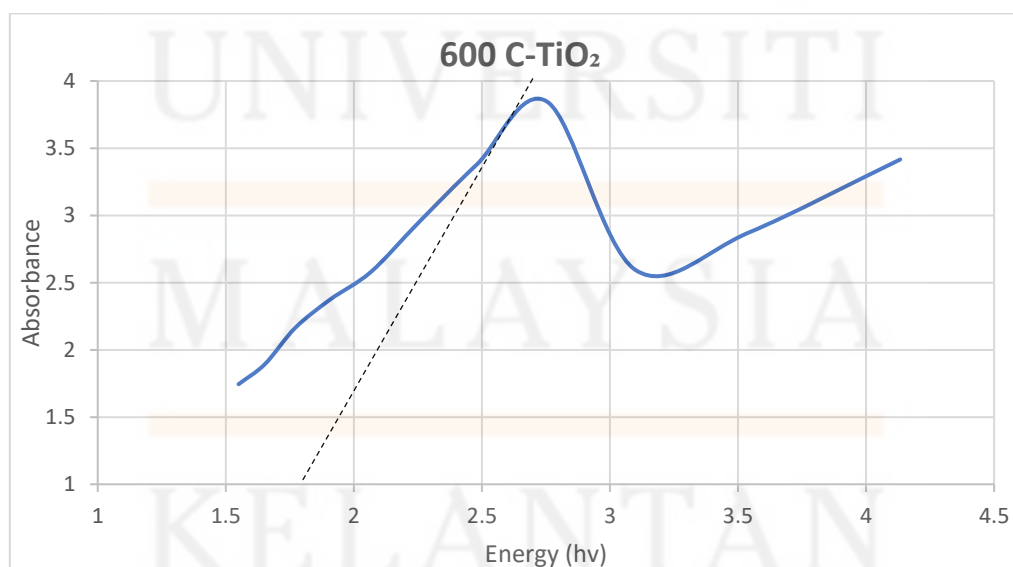


Figure 4.3 The trend of 600°C C-TiO<sub>2</sub>.

Figure 4.3 illustrates 600°C C-TiO<sub>2</sub> by detecting the emission peak at 2.75 (hv) then the Peak is observed at 3.844. Figure 4.2 has illustrated 600°C C-TiO<sub>2</sub> with excitation wavelength. The position of the highest emission peak goes to a larger wavelength as the excitation wavelength increases, and the intensity of carbon-tio2 first decreases and then increases. The excitation-dependent fluorescence properties of C-TiO<sub>2</sub> are related to the abundance of groups on the surface and the size distribution. C-TiO<sub>2</sub> has different conversion fluorescence properties. When light from 300 to 800 nm is used to excite C-TiO<sub>2</sub>, the up conversion PL spectrum of C-TiO<sub>2</sub> appears from 300 to 800 nm. With the absorbance wavelength, the brightest emission peak is centered at 2.755 (Hv). This property is due to an active multiphoton process in which two or more photons are absorbed simultaneously and then shorter wavelength fluorescence is released. As a result, the combination of TiO<sub>2</sub> carbon and UV semiconductor photocatalyst can increase the visible light utilization and photocatalytic capacity.

This explains how the manufacture of carbon mounted TiO<sub>2</sub> followed by carbonization at 400-600 °C can inhibit the phase shift from anatase to rutile at high temperatures while also obtaining a superior crystal structure of anatase.

UNIVERSITI  
MALAYSIA  
KELANTAN

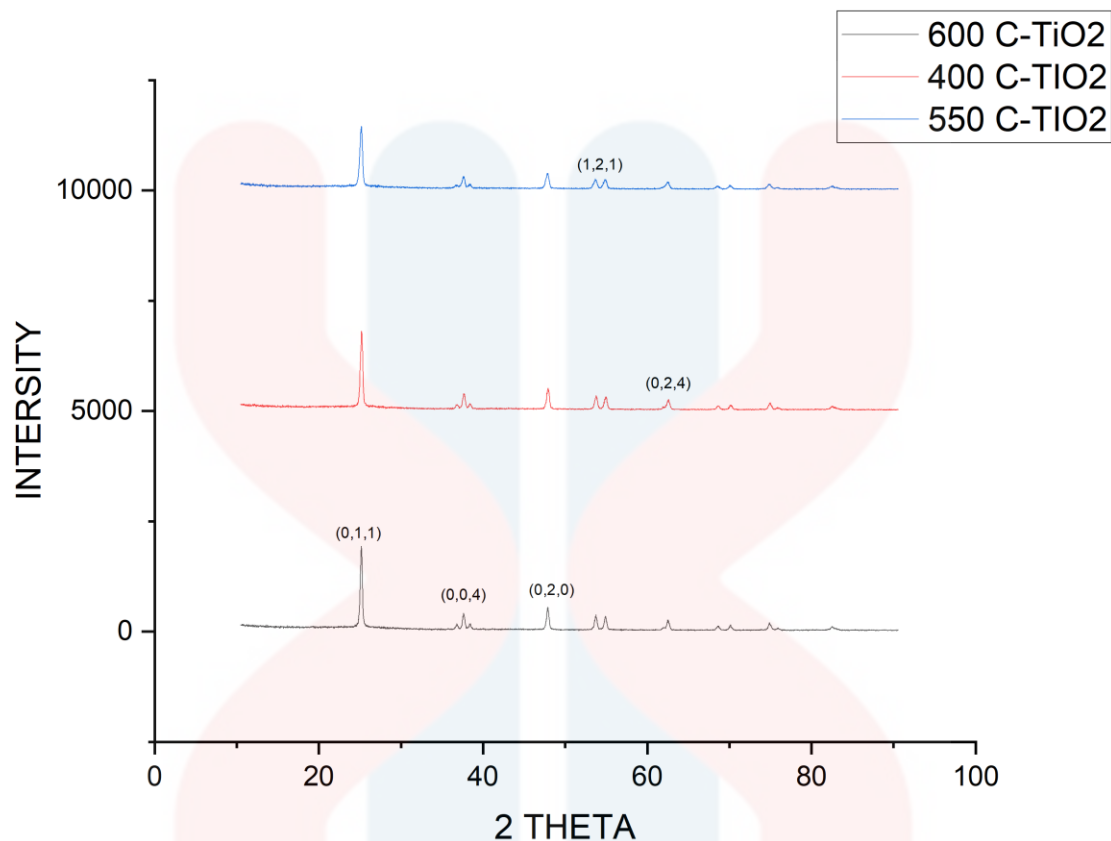


Figure 4: XRD analysis

The XRD spectra of TiO<sub>2</sub>doped with Carbon powder after sintering at various temperatures are shown in Figure 4.1. TiO<sub>2</sub> tetragonal anatase crystal phase is represented in the diffraction peak. This value conforms exactly to the standard data (JCPDS Card No.21-1272), according to (Scarpelli et al., 2018). Any crystalline phase including Ca-TiO<sub>2</sub> cannot be observed by XRD at 400 °C Carbon + TiO<sub>2</sub> due to the very low Carbon content. However, a new peak arises in composite architecture, demonstrating the presence of carbon, particularly at high carbon weight percentages. At  $2\theta \sim 37.288^\circ$ , a new peak (011) was detected in the TiO<sub>2</sub>-carbon composite, corresponding to Curite Carbon.

Based on this configuration, no impurities can be observed. 25 The crystal size (D) of TiO<sub>2</sub> doped carbon is calculated from the diffraction line broadening using the Debye Scherrer formula which is  $D = 0.9\lambda / \beta \cos\theta$ , where  $\lambda$  is the X-ray wavelength,  $\beta$  is the full width at half.

maximum intensity (FWHM) (Tenkyong et al., 2015). The calculated crystallite size for Carbon doped TiO<sub>2</sub> is shown in Table 4.

Table 4.1: Lattice parameter, average crystallite size and d – spacing of C-TiO<sub>2</sub>

Sample	Average crystallite size (nm)	Cell volume Å <sup>3</sup>	a (Å)	c (Å)
600 C-TiO <sub>2</sub>	25.4849588	256.81	3.7892	3.7892
400 C-TiO <sub>2</sub>	22.08870213	136.93	3.7892	9.537
550 C-TiO <sub>2</sub>	19.78083747	136.93	3.7892	9.537

Based on Table 1, it is possible to conclude that carbon doping reduces crystal size. This doping change avoids particle aggregation, resulting in a clear crystalline powder with a large surface area. It demonstrates that the addition of C-TiO<sub>2</sub> crystal formation is inhibited (Trang et al., 2019). However, as indicated by (Ursu et al., 2018), particle size can influence particle surface area and photogeneration recombination electron-hole pairs. Adsorption of reactants on light absorption rises with surface area. The C-TiO<sub>2</sub> band gap grows with particle size due to the quantum confinement effect. Quantum abstinence is a real thing. When the particle size is too small to equal the wavelength of the electron, this event happens. Quantum of electronic particles in nanocrystals provide unique optical and electrical features that have the potential to increase photovoltaic solar cell performance. As a result, the particle size C-TiO<sub>2</sub> influences the wavelength of the light source required for photovoltaics.

#### 4.5 Modified of working Photoanode for DSSCs application.

The photovoltaic parameters were illustrated in Table 4.3. The fill factor (FF) is defined as follows:

$$FF = \frac{(I_{\max} \times V_{\max})}{I_{sc} \times V_{oc}}$$



Where  $I_{max}$  is the maximum photocurrent and  $V_{max}$  is the maximum photovoltage.  $I_{sc}$  denotes the short-circuit photocurrent, whereas  $V_{oc}$  denotes the open circuit photovoltage. Meanwhile for Energy conversion efficiency ( $\eta$ ) is defined as:

$$\eta = \frac{(I_{sc} \times V_{oc} \times FF)}{P_{in}}$$

Material Temperature	$J_{sc}$ (mA/cm <sup>2</sup> )	$V_{oc}$ (mV)	FF (%)	Efficiency (%)
600 C-TiO <sub>2</sub>	1.09	48.6	0.7	0.037
400 C-TiO <sub>2</sub>	1.9	97.7	0.7	0.13
550 C-TiO <sub>2</sub>	1.72	84.4	0.7	0.1

As can be seen from Table 4, the values of  $\eta\%$ ,  $J_{sc}$ ,  $V_{oc}$  and FF are between 0.7, 1.09–19 mA/cm<sup>2</sup>, 48.6–97.7 V of the designed DSSC, respectively. It shows that increasing the annealing temperature increases the current density, implying a reduction of internal resistance, which stabilizes the fill factor (FF).

Max open circuit potential is found 400 C-TiO<sub>2</sub>, resulting in the highest efficiency of the device. One of the reasons for the difference in efficiency between the photoanode is 600 C-TiO<sub>2</sub> has a lower band gap value than 400 C-TiO<sub>2</sub> and 550 C-TiO<sub>2</sub>. Photovoltaic efficiency decreases as Carbon temperature increases. The increase in the antagonistic effect of Graphene content is explained by the fact that only an ideal amount of Graphene acts as a trap for electrons, producing a positive effect on DSSC efficiency. Referring to the fact that the electron transport mechanism in DSSC is a trap-limited electron diffusion process, the trapping state introduced by noble Graphene Carbon influences electrons reunification process. Additionally, noble metals such as copper can be plated semiconductor surface completely, preventing the absorption of light, resulting in a decrease in overall efficiency in composite photoanodes with higher concentrations of noble metals. (Ezealigo et al., 2020). This increase in DSSC efficiency can be caused by an increase in Carbon crystallinity with increasing



annealing temperature. Because it is annealing temperature causes crystal development and reduced agglomeration. The number of particles decreases as the temperature increases, the surface area increases and decreases clumping. Reduced incorporation causes increased dye loading, changing it DSSC performance. Among other samples, those with appropriate composition and temperature had the highest effectiveness.



## CHAPTER 5

### CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Conclusion

The maximum energy conversion is attained with 600 C—TiO<sub>2</sub>. We explored how C-TiO<sub>2</sub> temperature affected DSSC shape, optical absorption, and performance. Several variables can explain the observed rise in light absorption of TiO<sub>2</sub>-C coatings as temperature increases. One probable explanation is that temperature influences the morphological properties of the C-TiO<sub>2</sub> material. High temperatures may alter the crystal structure and surface morphology of C-TiO<sub>2</sub>, resulting in improved light absorption capabilities. Furthermore, higher temperatures can promote crystallinity and eliminate imperfections in the material, resulting in improved electron transport characteristics and, ultimately, DSSC performance.

However, it is important to note that the specific impacts of temperature on C-TiO<sub>2</sub> performance can be modified by a few parameters, including the synthesis method, carbon doping concentration, and the type of the carbon supply. A thorough examination of these variables would be required to properly comprehend the reported temperature-dependent changes in the material's characteristics and their impact on DSSC performance.

#### 5.2 Recommendation

Future research could look at using different carbon sources in the synthesis process to improve the characteristics of C-TiO<sub>2</sub> for better photovoltaic performance. Exploring alternative carbon precursors, such as carbon nanotubes, graphene, or carbon dots, may result in differences in the doping concentration and structural properties of C-TiO<sub>2</sub>. This, in turn, may affect the material's electrical characteristics and applicability for dye-sensitized solar cells (DSSCs).

Understanding the effect of different carbon sources on the final material properties can help adapt C-TiO<sub>2</sub> for specific applications, perhaps leading to increased energy conversion efficiency. Furthermore, thorough examinations of the structural and optical properties of C-TiO<sub>2</sub> generated from various carbon precursors should yield useful information about the most effective carbon doping procedures for photovoltaic applications.

To summarise, researching alternate carbon sources for C-TiO<sub>2</sub> synthesis is a promising route for improving material characteristics and promoting the development of efficient DSSCs.

## REFERENCE

- Mazlan, N. S. C., Yap, M. F. a. a. H., Ismail, M., Yahya, M., Ali, N. A., Sazelee, N., & Seok, Y. B. (2023). Reinforce the dehydrogenation process of  $\text{LiAlH}_4$  by accumulating porous activated carbon. *International Journal of Hydrogen Energy*, 48(43), 16381–16391. <https://doi.org/10.1016/j.ijhydene.2023.01.080>
- Heidarinejad, Z., Dehghani, M. H., Heidari, M., Javedan, G., Ali, I., & Sillanpää, M. (2020). Methods for preparation and activation of activated carbon: a review. *Environmental Chemistry Letters*, 18(2), 393–415. <https://doi.org/10.1007/s10311-019-00955-0>
- Heidarinejad, Z., Dehghani, M. H., Heidari, M., Javedan, G., Ali, I., & Sillanpää, M. (2020). Methods for preparation and activation of activated carbon: a review. *Environmental Chemistry Letters*, 18(2), 393–415. <https://doi.org/10.1007/s10311-019-00955-0>
- Akman, E., & Karapinar, H. S. (2022). Electrochemically stable, cost-effective and facile produced selenium@activated carbon composite counter electrodes for dye-sensitized solar cells. *Solar Energy*, 234, 368–376. <https://doi.org/10.1016/j.solener.2022.02.011>
- Imoto, K., Takahashi, K., Yamaguchi, T., Komura, T., Nakamura, J., & Murata, K. (2003). High-performance carbon counter electrode for dye-sensitized solar cells. *Solar Energy Materials and Solar Cells*, 79(4), 459–469. [https://doi.org/10.1016/s0927-0248\(03\)00021-7](https://doi.org/10.1016/s0927-0248(03)00021-7)
- Chen, J. C., Li, K., Luo, Y., Guo, X., Li, D., Deng, M., Huang, S., & Meng, Q. (2009). A flexible carbon counter electrode for dye-sensitized solar cells. *Carbon*, 47(11), 2704–2708. <https://doi.org/10.1016/j.carbon.2009.05.028>
- Richhariya, G., Kumar, A., Tekasakul, P., & Gupta, B. (2017). Natural dyes for dye sensitized solar cell: A review. *Renewable & Sustainable Energy Reviews*, 69, 705–718. <https://doi.org/10.1016/j.rser.2016.11.198>
- Goncalves, L. M., De Zea Bermudez, V., Ribeiro, H. A., & Mendes, A. M. (2008). Dyesensitized solar cells: A safe bet for the future. *Energy and Environmental Science*, 1(6), 655–667. <https://doi.org/10.1039/b807236a>
- Hudgins, J. L., Simin, G. S., Santi, E., & Khan, M. A. (2003). An assessment of wide bandgap semiconductors for power devices. *IEEE Transactions on Power Electronics*, 18(3), 907–914. <https://doi.org/10.1109/TPEL.2003>

<https://doi.org/10.1016/j.mssp.2013.05.004>810840

Ichimura, M., & Kato, Y. (2013). Fabrication of TiO<sub>2</sub>/Cu<sub>2</sub>O heterojunction solar cells by electrophoretic deposition and electrodeposition. *Materials Science in Semiconductor Processing*, 16(6), 1538–1541.

.Lin, K. Y. A., Chen, Y. C., & Lin, Y. F. (2017). LaMO<sub>3</sub> perovskites (M=Co, Cu, Fe and Ni) as heterogeneous catalysts for activating peroxydisulfate in water. *Chemical Engineering Science*, 160(October 2016), 96–105.

<https://doi.org/10.1016/j.ces.2016.11.017> Low, J.,

Zhang, L., Tong, T., Shen, B., & Yu, J. (2018). TiO<sub>2</sub>/MXene Ti<sub>3</sub>C<sub>2</sub> composite with excellent photocatalytic CO<sub>2</sub> reduction activity. *Journal of Catalysis*, 361, 255–266.  
<https://doi.org/10.1016/j.jcat.2018.03.009> Ludin, N. A., Al-Alwani Mahmoud, A. M., Bakar

Mohamad, A., Kadhum, A. A. H., Sopian, K., & Abdul Karim, N. S. (2014). Review on the development of natural dye photosensitizer for dye-sensitized solar cells. *Renewable and Sustainable Energy Reviews*, 31, 386–396.  
<https://doi.org/10.1016/j.rser.2013.12.001>

UNIVERSITI  
MALAYSIA  
KELANTAN

## APPENDIX A

Figure A.1: Preparation of  $\text{TiO}_2\text{-Cu}_2\text{O}$  sampleFigure A.2: Preparation of  $\text{TiO}_2\text{-C}$  paste

Figure A.3: Measurement of current and voltage for DSSCs