



**SYNTHESIS, CHARACTERIZATION AND OPTIMIZATION OF COCOA POD
HUSK CARBON QUANTUM DOTS**

By

NAZATUL AKMAL BINTI NAZIBUDIN

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

November 2022

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in
fulfilment of the requirement for the degree of Master of Science

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November 2022

Chairman : Mohamad Faiz Zainuddin, PhD
Faculty : Forestry and Environment

A quasi zero-dimensional carbon quantum dots were successfully synthesised from cocoa pod husk through an eco-friendly and simple hydrothermal reaction. In addition, different analysis was used to approve synthesise of the cocoa pod husks carbon quantum dots such as x-ray diffraction pattern, high resolution transmission electron microscope, fourier-transform infrared reflection spectroscopy, dynamic light scattering and zeta potential analysis. According to the characterization, the product was amorphous carbon with particle sizes 2.1-4.1 nm and contain various functional groups including hydroxyl and carbonyl. Besides, the photoluminescence (PL) intensity from cocoa pod husk carbon quantum dots can be modified through the optimization of hydrothermal parameters. The PL analysis showed the product has high photoluminescence intensity under optimized synthesis conditions with a quantum yield of 39.3% compared to non-optimized synthesis conditions (25.38%). Overall, the highest PL intensity of CQDs was successfully obtained by setting the hydrothermal temperature to 200°C for 2 hours, at a pH of 10 and a concentration of 1 mg/mL.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

MENSINTESIS, MENCIRI DAN MENGOPTIMUMKAN TITIK KUANTUM KARBON KOKO POD

Oleh

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Titik kuantum karbon (CQD) bersaiz sifar dimensi telah berjaya disintesis daripada sekam pod koko (CPH) melalui tindak balas hidroterma yang mesra alam melalui cara yang mudah. Di samping itu, analisis berbeza digunakan untuk mengesahkan sintesis titik kuantum karbon sekam pod koko seperti corak pembelauan sinar-X, mikroskop elektron penghantaran resolusi tinggi, spektroskopi pantulan fourier-transform inframerah, cahaya dinamik taburan dan analisis potensi zeta. Mengikut pencirian, produk itu adalah karbon amorf dengan saiz zarah 2.1-4.1 nm dan mengandungi pelbagai kumpulan berfungsi termasuk hidroksil dan karbonil. Selain itu, keamatan photoluminescence (PL) daripada titik kuantum karbon sekam pod koko boleh diubah suai melalui pengoptimuman parameter hidroterma. Analisis PL menunjukkan produk mempunyai keamatan fotoluminesensi yang tinggi di bawah keadaan sintesis yang dioptimumkan dengan hasil kuantum (QY) sebanyak 39.3% berbanding keadaan sintesis tidak dioptimumkan (25.38%). Secara keseluruhannya, keamatan PL tertinggi CQD berjaya diperoleh dengan menetapkan suhu hidroterma kepada 200°C selama 2 jam, pada pH 10 dan kepekatan 1 mg/mL.

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LIST OF ABBREVIATIONS

CPH	Cocoa pod husk
CQDs	Carbon quantum dots
CPH CQDs	Cocoa pod husks carbon quantum dots
SQDs	Semiconductor quantum dots
CDs	Carbon dots
CNPS	Carbon nanoparticles
BCDs	Biomass carbon dots
QY	Quantum yield
BPEI	Branched polyethylenimine
PEI	Poly ethyleneimine
RM	Ringgit Malaysia
LKM	Malaysian Cocoa Board
AC	Activated carbon
BSA	Bovine serum albumin
OH	Hydroxyl
C=O	Carbonyl
COO ⁻	Carboxyl
SEM	Scanning Electron microscopy
HRTEM	High resolution transmission electron microscopy
XRD	X-ray diffraction
FTIR	Fourier transmission infrared
CPHE	Cocoa pod husk extract

PL	Photoluminescence
UV	Ultra violet
HOMO	Highest occupied molecular orbital
LUMO	Lowest unoccupied molecular orbital
UCPL	Upconversion photoluminescence
NIR	Near infrared
QCE	Quantum confinement effect
EWGs	Electron withdrawing groups
NaOH	Sodium hydroxide
HCL	Hydrochloric acid
DI	Deionize water
λ_{ex}	Excitation wavelength
<i>et al.</i>	And other

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Theobroma cacao, known as cocoa, is a significant fruit crop grown from cocoa beans that produce chocolate, cocoa powder, and butter. Malaysia is the world's sixth-largest processing and grinding facility for cocoa beans (Bermudez et al., 2020). The cocoa grindings industry in Malaysia is expected to grow further to 400,000 tonnes shortly in 2023. In addition, as a byproduct of the production of cocoa beans, a massive amount of cocoa pod husk (CPH) is produced, and more than 320,00 kilogrammes of CPH are produced annually after processing (De et al., 2022). The vast majority of these wastes are left to deteriorate or are burned in the harvest field, despite the possibility of transforming them into commercially valuable commodities. However, this massive amount of unutilised feedstock might provide a steady supply of raw materials for a potential CPH biomass-based industry. This biomass waste could be turned into fertiliser, composite materials, pulp, and other things that have value (Kilama et al., 2019).

The constituents of CPH biomass are cellulose, hemicellulose, lignin, pectin, oils, and waxes (Diez et al., 2020). Among the following elements of the CPH biomass, cellulose comprises the largest proportion (Zoghلامي & Paes, 2019), providing a feedstock for the production of sustainable value-added materials such as carbon quantum dots (CQDs). CQDs are a new type of carbon nanomaterial, mainly with a diameter of fewer than 10 nanometers in combination with numerous functional groups or polymer chains (Liu et al., 2020; Nazri et al., 2021). Cellulose is a polymer of $(C_6H_{10}O_5)_n$ linked by $-(1,4)$ glucosidic bonds with a regular network of intramolecular and intermolecular hydrogen bonds organised into microfibrils. Crystalline and amorphous segments are repeated and intertwined within microfibrils. The amorphous domain is known to be dissolved during the synthesis of cellulose fibres, resulting in cellulose CQDs. CQDs are categorised based on their carbon core configuration, surface classes, and properties (Liu et al., 2016). The structure of CQDs is made up of a vast number of surface groups or polymer strings, such as carboxyl, hydroxyl, and amine; hence CQDs have exceptional water solubility and are simple to combine with other products without phase separation (Travlou et al., 2018; Zhao et al., 2020).

Waste biomass from alternative sources, e.g., crop residues, wood byproducts, food, and livestock manure, is readily available and abundant. However, these raw materials need to be more consideration and attention, such as wood (Zhou et al., 2020), pineapple leaves (Ravindran et al., 2019), and banana pseudostem (Zhang et al., 2021). Therefore, the synthesis of CQDs from CPH biomass is significant because it would significantly improve the sustainability of cocoa production. In addition, it would provide answers to the multitude of issues associated with CPH disposal (Chandra et al., 2016). In the field of

nanomaterials, a comprehensive study of the physicochemical and optical properties of CPH is required to maximise the use of organic wastes or non-wood fibres to produce CQDs (Akinjokun et al., 2021). The main objective of this work is to investigate the properties of cocoa pod husks carbon quantum dots (CPH CQDs) and the optimisation of synthesis parameters of CPH CQDs. The results obtained in this study could be used in determining further new applications for this kind of product.

1.2 Problem Statement

The expansion of cocoa production increases the proliferation of unwanted residues, such as CPH, on cocoa farms and plantations. Indeed, 10 tons of CPH are produced for every tonne of dry cocoa beans. Consequently, a big space is necessary for disposal. Thus, CPH wastes provide a significant issue for waste management. As biomass waste is enriched with carbon, CPH can be synthesised into functional nanomaterials, known as CQDs, that help manage biomass waste.

However, the characterisation of the CPH CQDs and optimisation of the synthesis procedure to generate high-quality CQDs with desired structures (e.g., size, shape, crystallinity, and amount of functional groups) are limited and cause the formation of CPH CQDs is not explored. Therefore, to create an effective pathway for large-scale processing of high-performing CPH CQDs, the synthesising, characterisation and influences of optimisation synthesise conditions (e.g., temperature, time, pH and concentrations) should be thoroughly investigated. It is very important to summarise synthesise process; the characterisation analysis and the stable synthesise parameters for the CPH CQDs because it is very important to facilitate the fabrication of successful synthetic routes for future potential novel applications.

1.3 Significant of the Study

Waste biomass from crop residues, forestry by-products, and food and livestock wastes is an abundant and cost-effective feedstock source. In this case, using waste materials as feed stocks to synthesise fluorescent CPH CQDs would be more efficient, allowing waste control through CPH CQDs. CQDs feature a variety of low-cost starting materials, ease of synthesis and surface functionalisation, luminescent luminescence, excellent photostability, and outstanding biocompatibility (Sharma et al., 2022; Alaghmandfard et al., 2021). Consequently, CPH CQDs can be developed to replace conventional SQDs, a traditional form of quantum dots that are usually expensive and cause environmental problems due to their heavy metals content (Safranko et al., 2021). A variety of methods have been developed for the synthesis of CPH CQDs, including electrochemical oxidation (Kawamata et al., 2017), hydrothermal (Liu et al., 2019), acid oxidation (Zhou & Wang, 2020), microwaves (Shikir et al., 2018), ultrasound (Askari et al., 2019) and arc discharge (Xiaohui et al., 2020) synthesis methods. However, these methods have some

drawbacks, such as the need for a complex and time-consuming method, high temperatures and harsh synthetic conditions, and the high costs surrounding the production process (Pudza et al., 2019). Therefore, it is highly desirable to explore a new carbon source for the simple, economical, and environmentally friendly synthesis of such CPH CQDs with fluorescent down- and up-conversion properties, especially by focusing on their fluorescent properties in applied technologies.

1.4 Research Aim and Objectives

This study aims to convert the CPH biomass waste into functional materials known as CQDs. To achieve this, the specific objectives listed below need to be met:

- 1) To synthesise the biomass-derived CPH CQDs
- 2) To characterise the physicochemical and optical properties of CPH CQDs
- 3) To determine the optimal synthesis parameters for CPH CQDs

1.5 Research Questions

A few questions that needed to be answered in this study:

- 1) What is the method use to synthesise the CPH CQDs?
- 2) What are the physicochemical and optical properties of CPH CQDs?
- 3) What are the sets of stable parameters of synthesizing fluorescent CPH CQDs?

1.6 Thesis Organization

This thesis contains five (5) main chapters, which provide the outlined information for a better understanding of conducting the study as a whole;

Chapter 2 describes the comprehensive literature review on related topics crucial for this study, such as the summary of CQDs, the synthesis methods, the types of precursors used, CPH biomass waste, the characterisation and the influence of synthesis parameters on the fluorescence of CQDs.

Chapter 3 comprehensively describes the materials and methods utilised for this study. The framework for methodological study has also been supplied to summarise the flow of this investigation. This chapter also covers the analytical

procedures and tools utilised for the characterisation analyses conducted in this study.

Chapter 4 provides the results and research findings of this study. This chapter illustrates the research findings in tables, graphs and figures. The explanations for the research findings, according to the study's objectives, are also discussed.

Chapter 5 summarises all the chapters of this thesis and draws a conclusion based on the fulfilment of the research objectives of this study. This chapter also presented the research contributions and made several recommendations towards the findings.



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