PHYSICOCHEMICAL AND OPTICAL PROPERTIES OF CARBON QUANTUM DOTS SYNTHESIZED FROM WATERMELON PEELS FOR PHOTOVOLTAIC APPLICATION

By

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

January 2024

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

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The characteristics of carbon quantum dots (CQDs) are significantly influenced by various factors such as preparation method, carbon source, doping and others. Therefore, a comprehensive investigation into the optimal properties of CQDs is crucial for their targeted application. In this study, CQDs were synthesized from watermelon peels using a carbonization method. A thorough examination of their properties was conducted, considering key parameters such as the type of solvents used, carbon concentration, and pH values with consideration for their application in dye-sensitized solar cells (DSSC). The solvatochromic effect was observed, manifesting as variations in the photoluminescence (PL) spectra when CQDs were prepared in different solvents. PL measurements conducted at different excitation wavelengths, revealed that the emissions of CQDs were distinctly influenced by the polarity of the solvents. Dynamic light scattering (DLS) analysis confirmed that the size of CQDs ranged approximately from 5.80± 0.4 to 9.74± 0.5 nm, a correlation validated by high-resolution transmission electron microscope (HRTEM) results. In the study on carbon concentration, the findings demonstrate a pronounced impact on the stability of CQDs particles. Zeta potential findings indicated that the stability of CQD particles peaked at low carbon concentration, with zeta potential values of -62.4 mV and -64.3 mV for CQDs dispersed in ultrapure water and methanol, respectively. Additionally, fluorescence intensity increased parallel with carbon concentrations, and the study identified that CQDs synthesized at 0.05-0.07 g/ml concentrations exhibited superior stability in colloidal form. In exploring different pH conditions, consistent pH-independent PL peak emissions were observed, yet the intensities displayed a pH-dependent trend, amplifying from acidic to neutral and diminishing from neutral to alkaline conditions. Moreover, in addressing drying concerns, the study revealed the occurrence of the coffee-ring effect at pH 2.5, while uniform deposition was achieved at pH 7.5. This phenomenon was discussed based on Derjaguin, Landau, Verwey, Overbeek (DVLO) theory. For the application of CQDs in DSSCs, CQDs in methanol demonstrated the highest efficiency of 1.23%, surpassing CQDs in ultrapure water (0.038%) and the control sample N719 dye (0.34%).

Keywords: Carbon quantum dots, photoluminescence, photovoltaic, zeta potential

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

FIZIKOKIMIA DAN SIFAT OPTIK TITIK KUANTUM KARBON SINTESIS DARIPADA KULIT TEMBIKAI UNTUK APLIKASI FOTOVOLTA

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Ciri-ciri titik kuantum karbon (CQDs) dipengaruhi secara signifikan oleh pelbagai faktor seperti kaedah penyediaan, sumber karbon, pendopan, dan lain-lain. Oleh itu, penyelidikan menyeluruh terhadap sifat optimum CQDs adalah penting untuk aplikasi yang ditentukan. Dalam kajian ini, CQDs disintesis daripada kulit tembikai menggunakan kaedah karbonisasi. Pemeriksaan menyeluruh terhadap sifat-sifat CQDs dijalankan, dengan mempertimbangkan parameter utama seperti jenis pelarut yang digunakan, kepekatan karbon, dan nilai pH dengan pertimbangan untuk aplikasi dalam sel suria terpeka warna (DSSC). Kesan *solvatochromic* diperhatikan, yang menampakkan variasi dalam spektrum fotoluminesens (PL) apabila CQDs disediakan dalam pelarut yang berbeza. Pengukuran PL dijalankan pada panjang gelombang pengujaan yang berbeza, mendedahkan bahawa pancaran CQDs dipengaruhi secara nyata oleh kekutuban pelarut. Analisa serakan cahaya dinamik (DLS) mengesahkan bahawa saiz CQDs dianggarkan dari 5.28± 0.4 hingga 9.74± 0.5 nm, korelasi ini disahkan oleh keputusan resolusi tinggi transmisi elektron mikroskop (HRTEM). Dalam kajian ke atas kepekatan karbon, hasil kajian menunjukkan impak yang ketara terhadap kestabilan zarah CQDs. Penemuan potensi zeta menunjukkan bahawa kestabilan zarah CQD mencapai puncaknya pada kepekatan karbon yang rendah, dengan nilai potensi zeta masing-masing -62.4 mV dan -64.3 mV untuk CQDs yang tersebar dalam air ultratulen dan metanol. Tambahan pula, intensiti fluoresens meningkat seiring dengan peningkatan kepekatan karbon, dan kajian mengenal pasti bahawa CQDs yang disintesis pada kepekatan 0.05-0.07 g/ml menunjukkan kestabilan yang lebih baik dalam bentuk koloid. Dalam meneroka keadaan pH yang berbeza, pancaran puncak PL yang tidak bergantung pada pH diperhatikan, namun intensitinya menunjukkan kecenderungan bergantung pada pH, meningkat dari asidik ke neutral dan berkurangan dari neutral ke keadaan alkali. Selain itu, dalam menangani masalah pengeringan, kajian mendedahkan kejadian kesan cincin kopi pada pH 2.5, manakala penyerapan seragam tercapai pada pH 7.5. Fenomena ini dibincangkan berdasarkan teori Derjaguin, Landau, Verwey, Overbeek (DVLO). Untuk aplikasi CQDs dalam DSSC, CQDs dalam metanol menunjukkan kecekapan tertinggi sebanyak 1.23%, mengatasi CQDs dalam air ultratulen (0.038%) dan sampel kawalan pewarna N719 (0.34%).

Kata kunci: fotoluminesens, fotovolta, potensi zeta, titik kuantum karbon

ACKNOWLEDGEMENTS

First and foremost, my sincere gratitude and praise to the Almighty Allah s.w.t, on whom ultimately, we depend for guidance. A very special thanks to my great supervisor, Dr. Mazliana Ahmad Kamarudin, who supervised me since day one of my research. I am very grateful to her for giving me the opportunity, encouragement, and inspiration to work in CQDs, a subject that had never crossed my mind to study. It has been a valuable experience working and studying with her. The experience presenting my work in Montpellier, France, and doing research during the devastating COVID-19 pandemic, would never be forgotten. I would like to extend my appreciation to my co-supervisors, Assoc. Prof. Ts. Dr. Suriati Paiman, Dr. Josephine Liew Ying Chyi, and Dr. Tan Sin Tee for helping me throughout the research. A special thanks to fellow researchers, FYP students, and all fellow friends, who assisted me during synthesis of colloidal CQDs. I appreciate the discussions, ideas, and time spent throughout this research. Not to forget technician and lab assistant who involved in this work especially from Department of Physics UPM, IBS, and ION2. I would also like to take this opportunity to thank SGS and Wazan UPM and for providing me fellowship and JPA through Graduate Research Excellent Programme (GrEP). Finally, to my beautiful wife, Dr. Noor Zuliana, I love you so much, and thank you for helping me a lot in life as well as in research. Also, many thanks to my parents, siblings and all family members for your endless support and love.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CQDs	Carbon Quantum Dots
QDs	Quantum Dots
HRTEM	High Resolution Transmission Electron Microscopy
EDX	Energy Dispersive X-ray
TEM	Transmission Electron Microscopy
FTIR	Fourier transform infrared
PL	Photoluminescence
MetOH	Methanol
EtOH	Ethanol
IPA	Isopropanol
ButOH	Butanol
DOS	Density of states
eV	Electron volt
mV	Millivolt
mW	Milliwatt
DLS	Dynamic light scattering
IEP	Isoelectric point
rpm	Revolution per minute
E_g	Energy band gap
DSSC	Dye-sensitized solar cell
Si	Silicon
λ	Wavelength
A	Absorbance
T	Temperature
CdSe	Cadmium selenide
ZnS	Zinc sulphide
P	Power
E_V	Valence band
E_C	Conduction band
hv	Photon
FWHM	Full width half maximum
Cu	Copper
FF	Fill factor
I_{SC}	Short circuit current density
V_{OC}	Open circuit voltage
P_{max}	Maximum power

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Study of materials at nano-scale has gained a great attention as it fills gap between bulk and molecules or atoms, subsequently improving our understanding of fundamental properties and providing new physical effects. Traditional practice is to use semiconductor quantum dots (QDs), as they provide the information and attention over the years owing the unique combination of optical, chemical, and electrical properties (Gao et al., 2017). QDs had been thoroughly explored for their tunable fluorescence emission and quantum confinement effect, which had allowed them to be used in many applications for many years (Kumar et al., 2022). The photostability and optical characteristics, such as the emission wavelengths, are affected by changes in the particle sizes of semiconductor QDs. These QDs are primarily made of lead sulphide (PbS), cadmium selenide (CdSe), and zinc sulphide (ZnS). In addition to utilising direct raw materials, QDs are also made using chemical vapor deposition and laser ablation, both of which use catalysts made of heavy metals including cobalt, nickel, and titanium (Torres Landa et al., 2022; Hongsith et al., 2022; Zhang et al., 2021). In light of this, the production of conventional QDs may require the use of expensive and toxic materials as well as challenging operating conditions. However, with strict regulatory ban on the use of heavy metals in the consumer products in global especially in Asian market has made major QDs move towards heavy metals free QDs and using low toxic materials like carbon and graphene in their products (Song et al., 2022; Ni & Li, 2018). One of the alternatives is by synthesizing nanoparticle based on the carbon material, also known as carbon quantum dots (CQDs).

CQDs are emerging as a promising class of materials with great potential in various fields such as optoelectronics, biomedicine, energy storage, and catalysis (Xu et al., 2019; Wang & Lu, 2022; Khan et al., 2022). CQDs are small (less than 10 nm), fluorescent carbon nanoparticles, which have unique electronic and optical properties. The carbon-based structure of CQDs makes them a sustainable and eco-friendly alternative to traditional semiconductors. CQDs are discovered in 2004 via purification of fluorescent single-walled carbon nanotube (Xu et al., 2004). The nano-sized carbon particles are broadly classified into two categories, which are CQDs with a size smaller than 10 nm and carbon nanoparticles (CNPs) with a size larger than 10 nm (Azam et al., 2021).

CQDs can be synthesized from a variety of carbon sources, including biomass, and carbon-rich precursors. Among the various waste biomasses, fruit peels have garnered considerable interest due to their abundance, low cost, and high carbon content. Watermelon peels, in particular, have gained attention as a potential precursor for the synthesis of CQDs. Watermelon peels are a readily available waste product generated in large quantities by the food industry. They are composed of organic compounds that contain carbon atoms, making them to easily become the CQDs. The primary components of watermelon peels are carbohydrates, which are organic compound made up of carbon, hydrogen, and oxygen atoms (Sorokina et al., 2021). The main carbohydrate present in watermelon peels are cellulose, hemicellulose, lignin, and other organic compounds. These peels have attracted attention not only for their potential use in sustainable energy production but also for their conversion into value-added materials such as CQDs.

Some solvents can promote aggregation or induce changes in surface chemistry, leading to a reduced stability. The problem can be mitigated by study the solvent-dependent stability, aiding in the development of stable and well-dispersed CQDs (P. Chen et al., 2022). In addition, different applications may require CQDs to be dispersed or used in specific solvents. Therefore, in this research, CQDs have been synthesized by different type of solvents. This helps us to identify solvents that are compatible with CQDs and promote their dispersion, stability, and optimal performance in specific applications. The choice of solvents can influence the stability and aggregation behaviour of CQDs.

The investigation of different carbon concentrations and different pH on the properties of CQDs is essential to comprehensively understand their synthesis, structural variations, and resulting characteristics. Variations in precursor levels affect the size distribution, photoluminescence (PL) behaviour, surface chemistry, aggregation tendency, and stability of CQDs by systematically changing the carbon source concentrations throughout synthesis (Liu et al., 2021). This exploration not only enables the optimization of synthesis procedures to tailor CQDs for specific applications but also sheds light on the underlying mechanisms governing their unique optical and physicochemical properties.

In recent years, there has been a growing interest in the application of CQDs in various fields, including photovoltaics and optoelectronics (Kim et al., 2022) (Batabyal et al., 2023). For example, CQDs have been used as sensitizers in solar cells, and as light-emitting materials in optoelectronic devices (Gaurav et al., 2022; Huang et al., 2020). In this thesis, the CQDs were synthesized via carbonization method from watermelon peels. The optical, structural, and physical properties of the samples are characterized to study the effect of different solvents, carbon concentrations and pH values. Finally, their potential application towards dye-sensitized solar cell (DSSC) where CQDs act as a co-sensitizer are investigated and studied.

1.2 Motivation and problem statements

The motivation behind studying the synthesis of CQDs is to explore the influence of solvent choice, carbon concentration and pH on the properties and characteristics CQDs. CQDs have gained significant attention due to their unique optical, electronic, and chemical properties, making them promising candidates for various applications such as optoelectronics, bioimaging, sensing, and energy storage (Stepanidenko et al., 2021; Fan et al., 2022; Kaur & Verma, 2022).

The decision to utilize watermelon peels as a carbon source was influenced by multiple considerations, encompassing factors such as their availability, sustainability, and the presence of functional groups. Possessing a significant carbon content, crucial for the synthesis of CQDs, renders them a favourable initial material for such processes. As a by-product of agriculture, they represent a cost-efficient source of carbon for CQDs synthesis, concurrently addressing environmental apprehensions and imbuing value into disregarded resources. Additionally, the incorporation of functional groups, such as hydroxyl (-OH) and carboxyl (-COOH), within watermelon peels augments the reactivity of the carbon source, thereby facilitating the synthesis and tailoring of CQDs to meet specific properties.

The choice of solvent during the synthesis process can have a profound impact on the size, morphology, and surface chemistry. Solvents with varying polarity and dielectric constants can influence the emission wavelength, intensity, and stability of CQDs. Therefore, understanding the effects of different solvents on CQDs synthesis can help optimize their properties for specific applications. Thus, improve the performance of optical properties. Investigating these effects can help identify optimal solvents for tuning the optical properties of CQDs for specific applications of DSSC.

The ability to tailor the optical characteristics of CQDs using various carbon concentrations is a further issue that needs to be solved. This is due to carbon concentration influences the size distribution, shape, and aggregation behaviour of the CQDs. This study can provide insights into the relationship between carbon concentration and the resulting CQDs morphology. As consequence, it is possible to adjust the energy bandgap thereby influencing the emission and absorption wavelengths of the CQDs. Investigating these effects can help understand the relationship between carbon concentration and the optical properties of CQDs.

Next, the study is focus on the influence of pH on surface chemistry and functional groups of CQDs. pH level can influence the protonation and deprotonation of surface functional groups, affecting surface passivation, optical properties, and chemical stability (Guo et al., 2018). It is feasible to understand how pH affects the development of surface functional groups and their impact on the characteristics of CQDs by examining the effects of pH on surface chemistry.

Also, as the pH of CQDs varied, the study of drying problems can be investigated based on the DLVO (Derjaguin, Landau, Verwey, Overbeek) theory. The theory involves the van der Waals forces attraction and electrostatic repulsion the charges surface interacting with liquid medium, in this case, the CQDs dispersed in solvents. Finally, the optimized samples based on the systematic study will be tested on dye-sensitized solar cell (DSSC) with the expected efficiency of 1-2%. This expected value was considered higher compared to previously reported CQDs + N719 dye (0.19 %) and CQDs only (0.10 %) (Ghan et al., 2019).

1.3 Objectives of the study

The general aim of this study is to explore the synthesis and characterization of CQDs extracted from watermelon peels. The novelty of this work lies in its sustainable approach towards producing CQDs from agricultural waste, thereby offering an eco-friendly and cost-effective alternative to traditional synthesis methods. Another novelty that can be highlighted is the evaluation of CQD dispersions stability by correlating the zeta potential measurements with colloidal stability parameters such as aggregation behaviour and particle size distribution.

The specific objectives are as follows:

1. to synthesize colloidal CQDs from watermelon peels via carbonization method.
2. to examine the effect of different type of solvents, carbon concentration, and pH on the optical, structural, and physical properties of the CQDs.
3. to evaluate the potential application of CQDs on the dye-sensitized solar cell (DSSC).

1.4 Thesis outline

This thesis starts with **Chapter 1** where the introduction of the CQDs, background of the study and the objectives of the research are presented. In **Chapter 2**, a brief explanation of the background theory of CQDs, low dimensional system, synthesis method, and properties of solvents are described. A description of carrier recombination, generation of electron and holes will be reviewed to give more information related to this research. Next, in **Chapter 3,** the methodology of including materials used to synthesize CQDs, characterization methods, and sample preparation will be explained. Also, in **Chapter 3** explains the theory used in this research. In **Chapter 4,** the characterization results will be analysed and elaborated comprehensively. The results include the effect of different type of solvents in the stability of CQDs, carbon concentration, and pH. The application on the DSSC also will be described in the chapter. Finally, in **Chapter 5**, the conclusion and suggestions for future works will be discussed.

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