PREPARATION OF CARBON QUANTUM DOT/CUPROUS OXIDE NANOCOMPOSITE FOR PHOTOCATALYTIC REDUCTION

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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 Master of Science

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PREPARATION OF CARBON QUANTUM DOT/CUPROUS OXIDE NANOCOMPOSITE FOR PHOTOCATALYTIC REDUCTION

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Chair : Associate Professor Dr Suraya Abdul Rashid, PhD
Faculty : Institute of Advanced Technology

Carbon dioxide (CO_2) reduction through photocatalysis is considered a promising way to mitigate the abundance of this greenhouse gas in the earth's atmosphere. In this work, blue-fluorescent carbon quantum dots (CQD) were synthesized *via* a facile top-down hydrothermal method utilizing empty fruit bunch (EFB) biochar of oil palms as the precursor. The as-synthesized CQD with varying loading content (1, 2, 3, 4 and 5 wt. %) were incorporated together with commercial copper (I) oxide (Cu_2O) nanoparticles using a simple wet impregnation method to form CQD/Cu_2O nanocomposites. The CQD, Cu_2O and the prepared CQD/Cu_2O nanocomposites were then applied for gas phase photocatalytic CO_2 reduction. The experiments were performed under visible light irradiation in a self-designated photoreactor which was connected to an online Gas Chromatography (GC). It was demonstrated that the CQD/Cu_2O photocatalyst with 2 wt. % of CQD was able to give the highest photoactivity among all other photocatalysts with an improvement of 54% in photoactivity than that of pure Cu_2O. The physicochemical properties of the best performed photocatalyst were studied to find the correlation of these properties with the marked photoactivity of this particular photocatalyst. The presence of CQD in the composite was confirmed by High resolution transmission electron microscopy (HRTEM) micrographs where CQD with diameter 2.2-5.7 nm were decorated uniformly onto the surface of Cu_2O nanoparticles. X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR) further revealed the deposition of CQD on the surface of Cu_2O. The considerable improvement in the photoactivity of this photocatalyst was due to the improved charge separation efficiency at the CQD-Cu_2O interphase at the optimum loading, where CQD serve as electron acceptor to reduce the charge recombination rate of Cu_2O. This behavior was supported by highest PL quenching of this nanocomposite than that of pure Cu_2O.

Besides, Tauc's relation from absorbance data further revealed the band gap reduction of this nanocomposite. Apart from that, band alignment of CQD/Cu_2O, charge carriers transfer and separation as well as possible reaction pathways for CO_2 photoreduction was proposed based on the research findings. The photocatalyst also exhibited excellent stability under repeated cycles of photoreaction. Overall, the considerable enhancement in the photoactivity of the nanocomposite describes the importance of the CQD to facilitate the high electron demanding CO_2 photoreduction reaction to ethane.

Abstrak tesis yang dikemukakan kepadaSenat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PENYEDIAAN TITIK KUANTUM KARBON DAN KUPRUM (I) OKSIDA NANOPARTIKEL UNTUK PENURUNAN SECARA FOTOPEMANGKINAN

Oleh

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Penurunan karbon dioksida (CO_2) secara foto pemangkinan merupakan cara yang efisen untuk mengurangkan peningkatankadar gas rumahhijau ini di atmosfera bumi. Dalam kajian ini, titik-titik kuantum karbon (CQD) yang memberi pendar kilau cahaya biru telah disediakan melalui kaedah hidroterma *top-down*, dengan meggunakan bio arang daripada tandan kelapa sawit kosong (EFB) sebagai bahanmula. CQD dengan peratus berat yang berbeza (1, 2, 3, 4 and 5 wt.%) digabungkan dengan kuprum (I) oksida (Cu_2O) nanopartikel komersial, melalui kaedah pengisi tepuanbasah untuk menghasikan fotopemangkin CQD, Cu_2O dan pemangkin-pemangkin yang dihasilkan telah digunakan untuk aktiviti penurunan CO_2 berfasa gas. Eksperimen ini dijalankan dibawah sinaran cahaya tampak, di dalam fotoreaktor yang direka sendiri yang disambungkan kepada Kromatografi Gas (GC). Experimen ini menunjukkan bahawa fotopemangkin CQD/Cu_2O dengan 2% berat CQD memberi hasil tertinggi untuk transformasi CO_2 dengan peratus penambahbaikkan sebanyak 54% berbanding Cu_2O. Pemangkin yang memberi hasil penurunan tertinggi telah dicirikan untuk mengkaji korelasi ciri-ciri fizikokimianya dengan aktiviti fotopemangkinan. HRTEM menunjukkan bahawa CQD dengan diameter 2.2-5.7 nm telahmenghiasipadapermukaannanopartikel Cu_2Osecaraseragam. Selainitu, keputusan FTIR dan XPS mendedahkanpemuatanCQD padapermukaan Cu_2O. Peningkatan besar dalam aktivitifoto pemangkinan ini adalah disebabkan oleh pemisahancas yang baik diantara fasa CQD-Cu_2O pada pemuatan optimum, disebabkan peranan CQD sebagai penerimaelekron yang mengurangkan kadar pengabungancas. Ini telah disokong oleh pelindapkejutan PL yang paling tinggi

nanokompositini daripada Cu_2O. Selain itu, perhubungan Tauc juga mendedahkan pengurangan jurang jalur nanokompositini. Selain itu, penjajaran jalur CQD/Cu_2O, pemindahan caspemisah dan pemisahan serta kemungkinan laluan tindakbalas untuk penurunan fotopemangkinan CO_2 dicadangkan berdasarkan penemuan penyelidikan. Pemangkin ini juga menunjukkan kestabilan yang sangat baik dibawah kitaran fotoreaksi yang berulang. Secara keseluruhannya, peningkatan besar dalam aktiviti fotopemangkinan oleh nanokomposit ini menggambarkan kepentingan CQD untuk memudahkan tindakbalas penuruan foto CO_2 kepada etana yang memerlukan kadar elektron yang tinggi.

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I certify that a Thesis Examination Committee has met on 13th July 2018 to conduct the final examination of Tharani Kulandaivaluon her thesis entitled "Preparation of Carbon Quantum Dot/Cuprous Oxide Nanocomposite for Photocatalytic Reduction" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science in Chemical Engineering.

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

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CO_2	Carbon dioxide
IPCC	Intergovernmental Panel on Climate Change
CCS	carbon capture and sequestration
Cu_2O	Cuprous oxide
CQD	carbon quantum dots
TiO_2	titanium dioxide
UV	ultraviolet light
C_2H_6	Ethane
EFB	Empty fruit bunch
HRTEM	High Resolution Transmission Electron Microscopy
XPS	X-ray Photoelectron Spectroscopy
FTIR	Fourier Transformation Infrared
XRD	X-ray Diffraction
PL	Photoluminescence
rGO	reduced graphene oxide
GQD	graphene quantum dots
UCPL	up-converted photoluminescence
GC	Gas chromatography
FID	flame Ionization detector
TCD	thermal conductivity detector
EDX	Energy Dispersive X-ray Spectroscopy
SAED	Selected Area Electron Diffraction
HOMO	highest occupied molecular orbital
LUMO	lowest unoccupied molecular orbital

CHAPTER 1

INTRODUCTION

1.1 Background

Carbon dioxide (CO_2) has been recognized to be the primary anthropogenic greenhouse gas that leads to rise in global temperature, also called global warming. In the Intergovernmental Panel on Climate Change (IPCC) report released on 2015, CO_2 accounted about 76% of total greenhouse gases emitted globally, as shown in Figure 1.1. The major source of CO_2 emission to the atmosphere is through the combustion of fossil fuels to generate energy for various industrial processes, transport and private sectors.

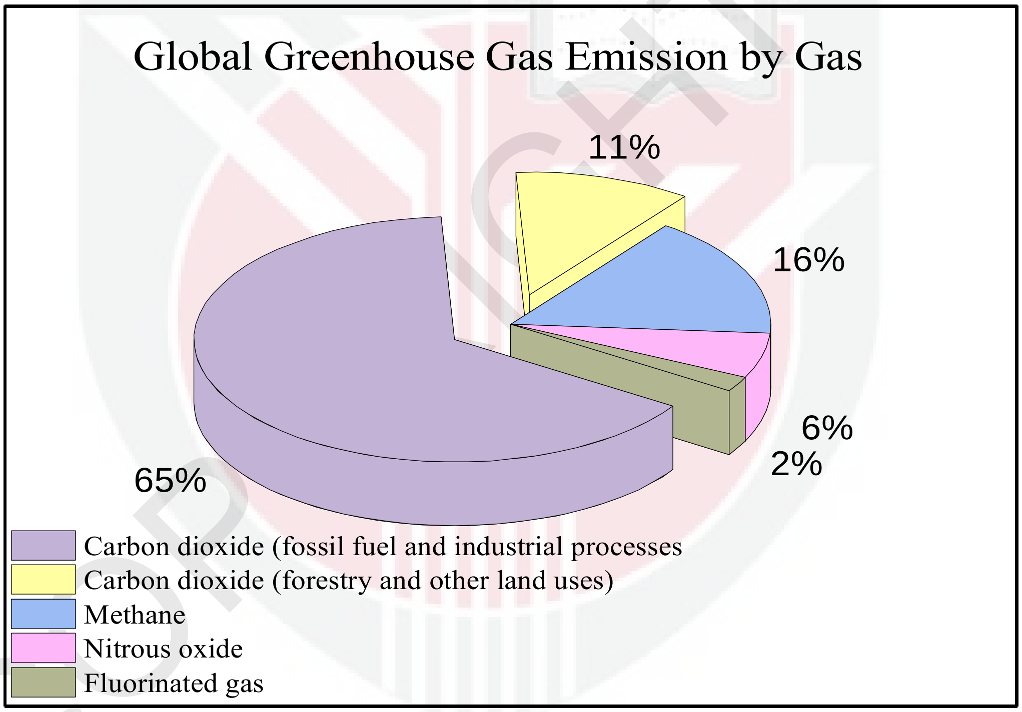

Figure 1.1: Global Greenhouse Gas Emission by Gas, data from IPCC (2015)**.**

Also, United Stated Energy Information Administration has released a report in 2010 which predicts the global CO_2 emission through energy-related activities which depicts in Figure 1.2. This emission is expected to rise from 29.7 billion metric tons in 2007 to 42.4 billion metric tons in 2035 with an increase about 43% of total global CO_2 emission over the projection period. The CO_2 emission of the developing nations outside the Organization for Economic Cooperation and Development (OECD) is progressing rapidly, mainly due to the heavy reliance on fossil fuels to meet the fast-paced growth of energy demand.

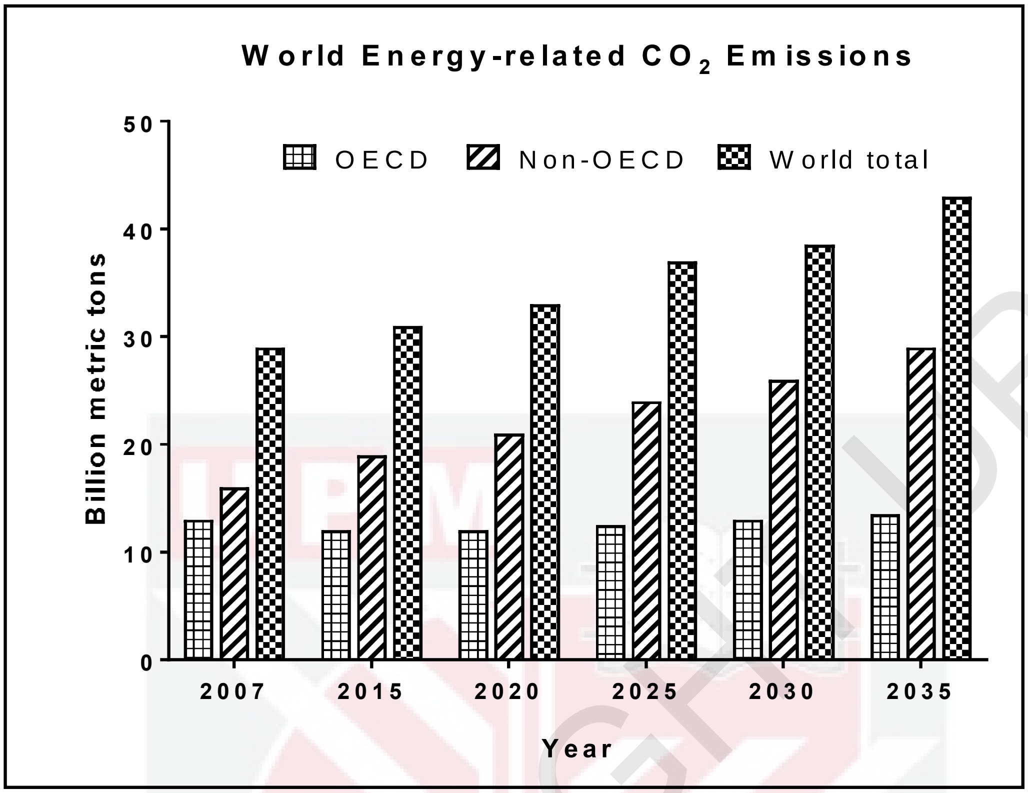

Figure 1.2: World Energy-Related Carbon dioxide Emissions (2007-2035), data adapted from United Stated Energy Information Administration (2010).

The rapid growth of world population and economy creates enormous demand for energy generation, eventually causes the rise in this greenhouse gas emission. This gas may builds up in the atmosphere and absorbs the infrared radiation emitted by the sun, eventually re-emit the radiation in all direction towards the earth before transforming it into heat thereby causing global warming (Hammer et al., 2015). Therefore, there is a desperate need to mitigate CO_2 emissions to the atmosphere.

Though existing robust technology of carbon capture and sequestration (CCS) is regarded the most viable CO_2 mitigation strategy(Leung et al., 2014), its high cost and energy input has drawn the attention of many towards alternative techniques for CO_2 conversion. Many methods had been approached globally which include biological reduction by plants, thermal reduction, electrochemical reduction and photocatalytic reduction using synthetic systems (Kumar et al., 2012; Smestad and Steinfeld, 2012). Among these methods, photocatalytic conversion (or artificial photosynthesis) is identified as the most compelling approach for the CO_2 transformation due to several reasons; (i) the reaction is driven by boundless solar energy, (ii) the reaction could take place at mild conditions (low temperature and pressure), (iii) untreated water can be used as the reductant, (iv) the converted carbon fuels could fulfil the current energy demand, (v) this process is ecologically clean as it does not cause any secondary pollution (Li et al., 2016).

Generally, a photocatalyst is defined as a chemical substance (usually semiconductors) which accelerates the photochemical reaction, without being consumed during the process. An ideal photocatalyst should possesses few characteristics including light sensitivity, chemical inertness, toxic-free and photocorrosion resistance (Ibhadon and Fitzpatrick, 2013). Cuprous oxide (Cu_2O) is a p-type semiconductor, regarded as a potential photocatalyst due to its abundance in nature, low toxicity, and ability to harness visible light (Sharma and Sharma, 2012). Meanwhile, carbon quantum dots (CQD), a new type of carbon nanomaterial opens up a new prospect in the field of photocatalysis for their unique properties such as good absorbance of solar light, excellent photoinduced electron transfer ability, facile functionalization of chemical groups and tunable photoluminescence (R. Wang et al., 2017; Yifan Wang et al., 2017). Combining the fascinating properties of Cu_2O and CQD by forming a composite material is expected to produce an ideal photocatalyst, to achieve a promising photocatalytic performance for CO_2 photoreduction.

1.2 Problem Statement

Generally, there are two key factors that determine the efficiency of artificial photocatalytic process, namely recombination rates of electron-hole pairs and solar radiation absorption capacity of the photocatalysts. Titanium dioxide (TiO_2) emerged as the most investigated semiconductor photocatalyst in the process of CO_2 photoreduction due to its photostability, low cost, and non-toxic property. Extensive research has been made to investigate CO_2 photoreduction by this catalyst, yet TiO_2 is far from the practical applications due to its wide bandgap (3.2 eV) which limiting its photoactivity under ultraviolet (UV) light which comprises only 5% of total solar spectrum (Linsebigler et al., 1995). Therefore, development of visible-light driven photocatalyst is a biggest challenge for this artificial photosynthesis study.

Cu_2O is a potential candidate to replace TiO_2 for its narrow band gap energy making it photosensitive to visible light. On the contrary, Cu_2O is of low stability for its tendency for photocorrosion and fast recombination of electron-hole pairs (Tran et al., 2012). Previous studies demonstrated that the incorporation of Cu_2O with carbon-based materials were able to overcome its limitations in photocatalysis (Mateo et al., 2017; Shi et al., 2015; Yan et al., 2017; D. Zhang et al., 2016; Zou et al., 2016). CQD are opted for this study as this nanocarbon inherited the properties of their parent compound such as good electron transfer property and large electron capacity to enable the extraction of electrons from the semiconductors to alleviate the recombination of charge carriers (R. Wang et al., 2017).

Therefore, combination of photoactive Cu_2O and CQD is targeted to form an attractive visible-light driven composite photocatalyst with extraordinary electron trapping capability to hinder charge recombination for better performance of photoactivity of the composite. However, achieving an appropriate composition ratio of CQD into the host material is crucial to achieve the best photoactivity (Yunus et al., 2017) In addition, there is no study reported yet for gas-phase photoreduction of CO_2 using this composite, hence this work will be a new approach as a strategy for CO_2 mitigation.

1.3 Research Objectives

The ultimate goal of this research objective is to fabricate a visible-light driven CQD/Cu_2O nanocomposite photocatalyst for the photoreduction of CO_2. The objectives of this research are:

a) To investigate the effect of CQD loadings onto Cu_2Oon the photocatalytic CO_2 reduction performance of CQD/Cu_2O nanocomposite photocatalyst

b) To evaluate the optical, physical and chemical properties of the best performed CQD/Cu_2O nanocomposite photocatalyst

1.4 Scope of Study

The above-mentioned objectives were achieved by carrying out a number of research activities throughout the study period. This includes:

- Synthesis of carbon quantum dots (CQD) *via* a top-down hydrothermal method using biochar from empty fruit bunch (EFB) of oil palm as precursor

- Preparation of CQD/Cu_2O nanocomposites by varying the loading ratio of CQD (1, 2, 3, 4 and 5 wt. %) over a fixed amount of Cu_2O *via* a simple wet-impregnation method

- Testing the as-prepared photocatalysts, pure Cu_2O and freeze-dried CQD for CO_2 photoreduction under visible light

- Characterizations of the resulting photocatalysts using HRTEM, XPS, FTIR, XRD, TGA, UV-vis DRS and PL spectroscopy

- Proposal of reaction mechanism of CO_2 photoreduction under visible-light using CQD/Cu_2O nanocomposite photocatalyst

- The catalyst durability or photostability study under repeated cycles of photoreduction process

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