



Faculty of Resource Science and Technology

Synthesis and Characterisation of Conjugated Bis-Schiff Base and The Boron Difluoride Complexes as Dye-Sensitiser for Dye Sensitised Solar Cell (DSSC) Application

Nursyafira Adzira Binti Halmi

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Assoc. Prof. Dr. Tay Meng Guan
Faculty of Resource Science & Technology,
Universiti Malaysia Sarawak,
M300 Kota Samarahan,
Sarawak, Malaysia.

Current Address: NO.6, LOT 9937, TAMAN MATANG BARU, JALAN MATANG BARU, 93050,

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Synthesis and Characterisation of Conjugated Bis-Schiff Base and The Boron
Difluoride Complexes as Dye-Sensitiser for Dye Sensitised Solar Cell
(DSSC) Application

Nursyafira Adzira Binti Halmi

A thesis submitted

In fulfillment of the requirements for the degree of Master of Science

(Inorganic Chemistry)

Faculty of Resource Science and Technology

UNIVERSITI MALAYSIA SARAWAK

2023

DECLARATION

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Name: Nursyafira Adzira Binti Halmi

Matric No.: 19020014

Faculty of Resource Science and Technology

Universiti Malaysia Sarawak

Date: 31 August 2023

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ABSTRACT

Schiff base and their metal complexes have been widely used as photovoltaic materials due to their excellent π -electron transfer properties along the molecule. In this study, a total of eleven conjugated symmetrical bis-Schiff base and its complexes with different π -spacers have been synthesised and spectroscopically characterised in order to investigate their conversion efficiency in dye-sensitiser solar cell (DSSC). All compounds were substituted either without any substituent or with hydroxy (-OH) or methoxy (-OMe) as the electron donor and difluoro boron (BF₂) as the electron acceptor. The symmetrical bis-Schiff base compounds (**1a** – **4c**) were synthesised by using condensation reaction and further proceeded to undergo complexation with boron trifluoride diethyl ether to produce boron difluoride complexes (**1d** and **2d**). All compounds were adapted with some modifications from existing works but have not been tested as dye-sensitiser for DSSC. All the synthesised compounds were applied as dye-sensitiser in DSSC using titanium (IV) oxide (TiO₂) coated on a fluoride doped tin oxide (FTO) glass as the working electrode and electric paint containing carbon black and graphite coated indium tin oxide (ITO) glass as the counter electrode. The power conversion efficiencies (PCE) of the eleven bis-Schiff base compounds were tested using a 100W LED light with multimeter and resistance set up. The efficiencies result was compared to N3 Dye as the benchmark standard. The results showed that the compound with aromatic ring bridge as the π -spacer in *para*- position and methoxy (-OMe) substituent gave the highest efficiency at 0.0691% whereas compound with aromatic ring and difluoro boron (BF₂) gave the lowest efficiency at 0.0012%. The presence of strong electron donor and extended π -conjugation effect in the structure allows effective charge transfer along the molecule which affects the conversion efficiency of DSSC device.

Keywords: Conversion efficiency, dye-sensitised solar cells, dye-sensitiser, symmetrical bis-Schiff-base, π -conjugated system

Sintesis dan Pencirian Bes-Schiff Simetri yang Berkonjugasi serta Kompleks Boron Difluorida sebagai Pewarna Foton untuk Sel Solar Peka Pewarna

ABSTRAK

*Bes Schiff serta kompleks logamnya telah digunakan dengan meluas sebagai bahan fotovoltan kerana sifat perpindahan elektron- π di sepanjang molekul. Sejumlah sebelas bes Schiff simetri terkonjugat dan kompleksnya dengan penjarak- π yang berbeza telah disintesis dan dicirikan secara spektroskopi untuk mengkaji kecekapan penukaran mereka di dalam sel solar peka pewarna. Kesemua sebatian telah digantikan sama ada tanpa apa-apa bahan ganti atau dengan hidroksi (-OH) atau metoksi (-OMe) sebagai penderma elektron dan boron difluorida sebagai penerima elektron. Sebatian bes Schiff simetri (**1a** – **4c**) telah disintesis melalui tindak balas pemeluwapan dan seterusnya menjalani pengkompleksan dengan boron trifluorida dietil eter untuk menghasilkan kompleks boron difluorida (**1d** dan **2d**). Kesemua sebatian telah diadaptasikan dengan sedikit ubah suai dari karya sedia ada tetapi belum diuji sebagai pewarna foton untuk DSSC. Kesemua sebatian yang telah disintesis telah digunakan sebagai pewarna foton untuk DSSC menggunakan titanium dioksida (TiO₂) yang disalut di atas kaca oksida timah terdop fluorida (FTO) sebagai elektrod kerja dan cat elektrik yang mengandungi hitam karbon dan grafit disalut di atas kaca oksida indium timah sebagai elektrod pembalasan. Kecekapan penukaran kuasa (PCE) bagi kesebelas bes Schiff telah diuji menggunakan persediaan lampu LED 100W dengan multimeter beserta rintangan. Hasil kecekapan telah dibandingkan dengan pewarna N3 sebagai tanda aras piawai. Keputusan menunjukkan bahawa sebatian yang mengandungi titian gelang aromatik sebagai penjarak- π diposisi para- beserta bahan ganti metoksi (-OMe) memberikan kecekapan tertinggi sebanyak 0.0691% manakala sebatian yang mengandungi titian gelang dan boron difluorida (BF₂) memberikan kecekapan terendah*

sebanyak 0.0012%. Kehadiran penderma elektron yang kuat beserta kesan konjugasi- π yang diperpanjangkan di dalam struktur mengizinkan perpindahan cas yang berkesan disepanjang molekul mempengaruhi kecekapan penukaran peranti DSSC.

Kata kunci: Kecekapan penukaran, sel solar peka perwarna, pewarna foton, bes Schiff simetri, sistem π -konjugasi

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LIST OF ABBREVIATIONS

%	Percent
°C	Degree celcius
¹³ C	Carbon
¹ H	Proton
Ag	Silver
AM	Air mass
BF ₂	Boron difluoride
BF ₃ .Et ₂ O	Boron trifluoride diethyl ether
Br	Bromine
Br ⁻	Bromide
C	Carbon
C=N	Azomethine
C=O	Carbonyl
Cd	Cadmium
CDCl ₃	Deuterated chloroform
CdO	Cadmium oxide
-CH ₃	methyl
CHCl ₃	Chloroform
Cl	Chlorine
Co	Cobalt
-COOH	Carboxyl
CSP	Concentrated solar power
d	Doublet

DAMN	Diaminomaleonitrile
DCM	Dichloromethane
DIPEA	<i>N,N</i> -Diisopropylethylamine
DMSO	Dimethyl sulfoxide
DSSC	Dye-sensitised solar cell
e ⁻	electron
EL	electroluminescent
eV	Electronvolt
F	Fluorine
FF	Fill factor
fs	Femtosecond
FTIR	Fourier-transform infrared
FTO	Fluorine-doped tin oxide
g	Gram
G	global
Ga ₂ O ₃	Gallium(III) oxide
GC/MS	Gas chromatogram/Mass spectroscopy
H ₂ O ₂	Hydrogen peroxide
Hg	Mercury
HOMO	Highest occupied molecular orbital
Hz	Hertz
I ⁻ / I ₃ ⁻	Iodide/triiodide
I ₂	Iodine
ICT	Intramolecular charge transfer
In ₂ O ₃	Indium(III) oxide

IR	Infrared
ITO	Indium tin oxide
J_{sc}	Short circuit current
KBr	Potassium bromide
KOH	Potassium hydroxide
kWh/m^2	Kilowatt hours per square metre
LED	Light-emitting diode
LUMO	Lowest unoccupied molecular orbital
m	Multiplet
m/z	Mass-to-charge ratio
mA	Milliampere
MeOH	Methanol
MHz	Megahertz
ml	Millilitre
mmol	Millimole
mV	Millivolt
N	Nitrogen
N_2O_2	Dinitrogen dioxide
NaOH	Sodium hydroxide
Nb_2O_5	Niobium pentoxide
Ni	Nickel
NLO	Non linear optics
nm	Nanometre
NMR	Nuclear magnetic resonance
O	Oxygen