UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERISTICS OF POLYURETHANE ACRYLATE GEL POLYMER ELECTROLYTE FOR DYE SENSITIZED SOLAR CELL APPLICATION

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By

CHAI KAI LING

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

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DEDICATION

To
My family

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

SYNTHESIS AND CHARACTERISTICS OF POLYURETHANE ACRYLATE GEL POLYMER ELECTROLYTE FOR DYE SENSITIZED SOLAR CELL APPLICATION

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Solid and liquid electrolytes pose opposing advantages and disadvantages. Solid electrolyte has lower electrochemical performance in terms of ionic conductivity but has wide operating temperatures. However, liquid electrolyte has greater electrochemical performance but they easily leak and corrode components that come into contact with it. Gel polymer electrolyte (GPE) aims to combine the advantages of both solid and liquid electrolyte in one single package. In this work, dye sensitized solar cells (DSSCs) was fabricated from polyurethane acrylate (PUA) GPE which have been enhanced with varying amounts of (i) tetrabutylammonium iodide (TBAI), (ii) TBAI and lithium iodide (LiI) and (iii) TBAI, LiI and 1-butyl-3-methylimidazolium ionic liquid (BMII). The PUA was characterised using wet chemical tests such as Oxirane Oxygen Content test, Acid value test, Hydroxyl value test and Iodine Value test. Furthermore, the PUA was characterised by FTIR (Fourier Transform Infrared spectroscopy) and EIS (Electrochemical Impedance Spectroscopy). With the FTIR spectrum, it proved that PUA was successfully synthesized by epoxidation, hydroxylation and introduction of isocyanate group processes. Based on the Nyquist plot of pure PUA, the ionic conductivity (σ) obtained was 5.60×10^{-6} S cm^{-1}. The PUA polymer was enhanced with various iodide salts to increase its electrochemical performance. All GPE systems prepared were characterised using FTIR, thermal gravimetric analysis (TGA) and EIS. PUA were prepared with TBAI salt as the first GPE system. FTIR was performed to determine the formation of complexation between PUA and TBAI salt. It was observed that 30 wt. % TBAI salt (A3 electrolyte) shows the highest σ of 1.88×10^{-4} S cm^{-1} with the highest charge mobility (μ) of 6.24×10^{-7} cm^2 V^{-1} s^{-1} and diffusion coefficient (D) of 1.60×10^{-8} cm^2 s^{-1} which estimated from fitting the Nyquist plots. The A3 electrolyte recorded the highest solar conversion efficiency (η) of 1.97 %. The highest η was due to low charge transfer resistance (R_{pt}) of 2.54 Ω at the electrolyte/counter electrode interface along with low charge transfer resistance (R_{ct}) of 24.97 Ω at TiO_2/dye/electrolyte interface and the charge diffusion

resistance (R_d) of 34.14 Ω in the redox electrolyte. This A3 electrolyte was then further enhanced with the addition of LiI and was observed that 5.00 wt. % LiI (B2 electrolyte) shows the best result. B2 electrolyte shows the maximum σ of 2.34×10^{-4} S cm^{-1}. This was because B2 electrolyte had the highest μ of 1.65×10^{-6} cm^2 V^{-1} s^{-1} and D of 4.25×10^{-8} cm^2 s^{-1}. B2 electrolyte indicated the highest η of 5.09 % due to low value of R_{pt}, R_{ct} and R_d. B2 electrolyte then further enhanced with the addition of BMII ionic liquid and 6 wt. % BMII (C3 electrolyte) shows the best result. The C3 electrolyte manages to achieve an ionic conductivity value of 4.17 $\times10^{-4}$ Scm^{-1} with highest μ of 2.03×10^{-6} cm^2 V^{-1} s^{-1} and D of 5.20×10^{-8} cm^2 s^{-1}. 5.72 % of η obtained with low R_{pt}, R_{ct} and R_d. This work shows that PUA-based electrolytes have the potential for DSSC applications.

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

SINTESIS DAN PENCIRIAN ELEKTROLIT POLIMER GEL POLIURETANA AKRILAT DALAM PENGGUNAAN SEBAGAI SEL SURIA PEKA PEWARNA

Oleh

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Elektrolit pepejal dan cecair mempunyai kelebihan dan kekurang yang bertentangan. Elektrolit pepejal menunjukkan kemampuan elektrokimia yang lebih rendah berbanding dengan elektrolit cecair, tetapi tahan lasak dan ia juga boleh beroperasi pada julat suhu yang besar. Walau bagaimanapun, elektrolit cecair mempunyai ciri-ciri elektrokimia yang lebih tinggi berbanding dengan elektrolit pepejal tetapi ianya senang bocor apabila bekas yang mengandunginya kemek dan akan mengakibatkan komponen berdekatan terhakis. Elektrolit polimer gel (GPE) dibangunkan untuk mempunyai kelebihan elektrolit pepejal dan elektrolit cecair pada masa yang sama. Dalam karya ini, sel suria peka pewarna telah dibangunkan daripada elektrolit polimer gel poliuretana akrilat (PUA) dengan bantuan (i) Tetrabutilammonium Iodida (TBAI), (ii) TBAI dan Litium Iodida (LiI) dan (iii) TBAI, LiI dan 1-Butil-3-Metilimidazolium (BMII). PUA hos polimer yang telah disediakan akan melalui proses pencirian seperti kandungan oksigen oksiran, nilai asid, nilai hidroksil dan nilai iodin. PUA yang telah disediakan akan melalui proses pencirian inframerah transformasi fourier (FTIR) dan spektroskopi impedansi elektrokimia (EIS). Spektra FTIR PUA menunjukkan bahawa PUA yang telah disediakan dengan jayanya menggunakan proses pengepoksidaan, penghidroksilaan, dan penambahan kelompok isosianat. Berdasarkan plot Nyquist untuk sampel PUA, nilai keberaliran ionik (σ) yang diperolehi adalah 5.60×10^{-6} S cm^{-1}. PUA hos polimer kemudiannya dipertingkatkan dengan garam-garam iodida yang tersenarai di atas untuk meningkatkan keupayaan elektrokimianya. Kesemua GPE yang telah dibangunkan kemudiannya akan melalui pencirian FTIR, analisis gravimetri terma (TGA), dan EIS. FTIR digunakan untuk kaji interaksi garam iodida dengan hos polimer. Campuran PUA dengan garam TBAI akan dijadikan sebagai sistem pertama. Pencirian FTIR telah dilaksanakan untuk mengenal pasti pembentukan kompleks di antara PUA dengan garam TBAI. Elektrolit sistem pertama 30 wt. % TBAI-PUA (Sampel A3) berjaya menunjukkan nilai σ sebanyak 1.88×10^{-4} S cm^{-1} dengan mobiliti caj (μ) yang tertinggi pada nilai 6.24×10^{-7} cm^2 V^{-1} s^{-1} dan pekali resapan (D) sebanyak 1.60×10^{-8} cm^2 s^{-1} yang diperoleh daripada plot Nyquist.

Sampel A3 juga menunjukkan kecekapan penukaran solar (η) yang tertinggi dengan nilai 1.97 %. Ini adalah kerana rintangan pemindahan cas yang rendah pada antara muka elektrolit/elektrod tambahan (R_{pt}) dengan nilai 2.54 Ω manakala antara muka TiO_2/pewarna/elektrod (R_{ct}) dengan nilai 24.97 Ω dan 34.14 Ω pada elektrolit redox (R_d). Sampel A3 kemudiannya dibangunkan lagi dengan menggunakan LiI. Sampel B2 (5.00 wt. % LiI) merupakan sampel yang paling memberangsangkan dengan nilai σ pada 2.34×10^{-4} S cm^{-1}. Sample B2 mempunyai nilai μ tertinggi pada 1.65×10^{-6} cm^2 V^{-1} s^{-1}, nilai D pada 4.25×10^{-8} cm^2 s^{-1} dan nilai η pada 5.09 % kerana nilai R_{pt}, R_{ct} and R_d yang rendah. Sampel B2 ini dibangunkan dengan cecair ionik BMII. Sampel C3 (6 wt. % BMII) berjaya menunjukkan nilai σ sebanyak 4.17×10^{-4} S cm^{-1} dengan nilai μ sebanyak 2.03×10^{-6} cm^2 V^{-1} s^{-1}, nilai D sebanyak 5.20×10^{-8} cm^2 s^{-1}, dan nilai η sebanyak 5.72 %. Dengan itu, elektrolit berasaskan PUA mempunyai potensi tinggi untuk kegunaan dalam sel suria peka pewarna.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

LIST OF ABBREVIATIONS

ε_o	Vacuum permittivity
ε_r	Dielectric constant
R_{Ct}	Charge transfer resistance that occur TiO_2/dye/electrolyte interface
R_d	Resistance related to the diffusion of charge carriers in the redox cycle
R_{pt}	Resistance of charge transfer
k_1^{-1}	Bulk geometrical capacitance of electrolyte
k_2^{-1}	Electrical double layer capacitance at electrode/electrolyte interface
p_1	Ratio of the angle between the diameter of the depressed semicircle and Z" axis to the right angle subtended by the real and imaginary impedance axes
p_2	Skew parameter that control degree of the inclination of the titled spike from the Z' axis
$\frac{\pi p_1}{2}$	Angle between the axis of negative imaginary impedance and diameter of depressed semicircle
$\frac{\pi p_2}{2}$	Angle between real impedance axis and tilted spike line
τ_2	Time constant
μ	Mobility of charge carriers
A	Electrode/electrolyte contact area
AV value	Acid value
BMII	1-butyl-3-methylimidazolium iodide
C_{DL}	Double layer capacitance
CPE	Constant phase element
D	Diffusion coefficient of charge carriers
DMF	Dimethyl formamide
DMSO	Dimethyl sulfoxide

D_o	Diffusion coefficient of charge carriers for electrolyte with only tilted spike Nyquist plot
DSSCs	Dye sensitized solar cells
DTG	Derivative Thermogravimetry
e	Elementary charge
E_a	Activation energy
EC	Ethylene carbonate
EIS	Electrochemical impedance spectroscopy
EJO	Epoxidized Jatropha Oil
EMImSCN	1-Ethyl 3-Methylimidazolium Thio-Cyanate
ε	Permittivity
FF	Fill factor
FTIR	Fourier transform infrared spectroscopy
FTO glass	Fluorine doped tin oxide glass
GPE	Gel polymer electrolyte
H_2SO_4	Sulphuric acid
HEMA	2-Hydroethylmethacrylate
HNO_3	Nitric acid
I^-/I_3^-	Iodide/triiodide
IV value	Iodine value
J_{sc}	Current density
k_b	Boltzmann constant
LiI	Lithium iodide
n	Number of charge carriers
N719	Ruthenizer 535-Bis TBA Dye
NaCl	Sodium chloride

OH value	Hydroxyl number
OOC value	Oxygen content value
PC	Propylene carbonate
PEG	Polyethylene glycol
P_{in}	Total incident power density
PMII	1-Methyl 3-Propylimidazolium Iodide
PUA	Polyurethane acrylate
R	Bulk resistance
R_P	Electron transfer resistance
R_s	Series resistance
R_U	Uncompensated electrolyte resistance
t	Sample thickness
T	Absolute temperature
TBAI	Tetrabutylammonium iodide
TDI	2,4-Toluene-Diisocyanate
TGA	Thermal gravimetric analysis
TiO_2	Titanium dioxide
V_{oc}	Open circuit photovoltage
VTF	Vogel tamman fulcher
Z’	Real impedance axis
Z’’	Imaginary impedance axis
η	Solar conversion efficiency
σ	Ionic conductivity
ω	Angular frequency

CHAPTER 1

INTRODUCTION

1.1 Introduction

Dye sensitized solar cells (DSSCs) is a photovoltaic device that is able to convert sunlight (solar energy) into electrical energy by using dyes and semiconductors. History of DSSCs is related to the early days of color photography. In the year of 1873, Hermann Wilhelm Vogel accidentally found that the photographic film is more sensitive to red light as green dye is added into silver halide photographic emulsion (Peter, 2007). The first known DSSCs were assembled by O'regan and Gratzel in 1991 (O'Regan & Grätzel, 1991). From that, the researchers discovered that DSSC have a lot of advantages. They are low cost, easy to fabricate, environmentally friendly and have high conversion efficiency (Arof et al., 2017; Yahya & Sasongko, 2019). Due to the advantages, DSSC had been used widely in many applications. For examples: solar panel, solar calculator, solar rooftop, solar toy kit etc. Furthermore, DSSCs was sandwiched between fluorine doped tin oxide (FTO) glass coated with titanium dioxide (TiO_2) as photo-anode electrode, dye, electrolyte and FTO glass coated with platinum as counter electrode (Yahya & Sasongko, 2019).

The electrolyte plays an important role in the DSSC in order to obtain good ionic conductivity and solar conversion efficiency. Electrolyte must contain a redox couple (I^-/I_3^-) to regenerate the oxidized dye efficiently (Sharma et al., 2018). As in previous years, liquid electrolyte had been used extensively in DSSC due to its high ionic conductivity value and solar conversion efficiency up to 12.3 % (Yella et al., 2011). However, it has many drawbacks such as solvent evaporation, sealing, electrolyte leakage after damage, desorption of dye and photo-degradation of the dyes (Bandara et al., 2019). Based on the disadvantages of liquid electrolyte, some modification had been done and gel polymer electrolyte had the potential to replace the traditional liquid electrolyte (Bandara et al., 2019). Solid polymer electrolytes are able to provide good mechanical properties and better safety but they have low ionic conductivity of approximately in the order of magnitude of 10^{-8} S cm^{-1} (Baskoro et al., 2019). Gel polymer electrolyte can be said to be the combination between the solid state electrolyte and liquid state electrolyte. In order to further enhance the ionic conductivity and solar conversion efficiency of the gel polymer electrolyte, iodide salt or/and ionic liquid are added into it. Polyurethane acrylate (PUA) based Jatropha oil was synthesized and mixed with two different sizes of iodide salts and ionic liquid. For the first system, tetrabutylammonium iodide (TBAI) salt is added into the PUA gel polymer electrolyte (GPE) system. TBAI salt is a bulky salt. The larger the size, it will be more easy to dissociate to free ions as it has lower lattice energy (513 kJ mol^{-1}) (Arof et al., 2017; Mohamad Sri et al., 2017). This means that with little energy, TBAI salt will readily dissociate into TBA^+ and I^- ions. With the composition of TBAI salt which has the highest value of ionic conductivity and solar conversion efficiency, lithium iodide (LiI) salt is added to produce the second GPE system. The GPE system with mixed iodide salt or binary salt was proven that it was able to increase the value of ionic conductivity and solar conversion efficiency. The small size iodide salt (LiI salt) can enter and absorb at the TiO_2 surface easily compared to TBAI salt. When more amounts of Li^+ ions accumulate at the conduction band, it will downshift the conduction band. Then, less energy is required for the excited electrons to jump into the conduction band. Because of this, the short circuit current will be reduced and the efficiency will increase accordingly. To boost up the ionic conductivity and solar conversion efficiency, 1-butyl-3-methylimidazolium iodide (BMII) ionic liquid is added to produce a third GPE system. BMII ionic liquid is added as a plasticizer.

1.2 Problem Statements

Environmental impact of heavily depending on non-renewable petroleum based polymer and related waste disposal concerns pushes research and development towards finding a sustainable source which is environmentally friendly. The recent development of bio-based polymers which are derived from plants to replace petroleum based polymers are beginning to gain the interest of many researchers worldwide. In this work, non-edible Jatropha oil is used as starting materials to prepare the bio-based polyurethane acrylate (PUA) polymer. This is because jatropha plants contain toxic components such as curcin and other purgatives. The toxicity makes the jatropha oil non-competitive as a food source.

Solid electrolyte based DSSCs present lower conductivity properties and solar conversion efficiency compared to liquid electrolyte based DSSCs. Liquid electrolyte based DSSCs has its various drawbacks as it is susceptible to degradation from solvent evaporation, electrolyte leakage and difficulty of containing the liquid over a long period of time. As a solution, gel polymer electrolyte has a big potential in replacing both solid and liquid electrolyte. Gel polymer electrolyte has many desirable properties such as good chemical, mechanical and thermal stability (Pradhan, Choudhary, & Samantaray, 2008; Ramya, et al., 2008).

Single salt electrolyte has lower ionic conductivity compared to binary salt electrolyte. This is due to the lower number of charge carriers (n) and mobility (μ) in ionic transportation of single salt electrolyte. To further enhance the ionic conductivity of the electrolyte, the number of charge carriers and mobility need to be increased. From the formula $conductivity = n\mu e$, it is proven that n and μ plays an important roles in determining conductivity. In order to improve the number of charge carriers and mobility, binary iodide salts electrolyte has been introduced. Binary iodide salts electrolyte containing a small cation and a big cation in the electrolyte. Using the binary iodide salts electrolyte, the number of charge carriers will increase as well as ionic conductivity will be enhanced. Apart from this, the performance of DSSCs can be improved. This is due to the small cation affecting the energetics of the TiO_2/dye/electrolyte interface and increasing the short circuit current density, J_{sc} as the electron injection into the conduction band of the semiconductor has increased.

Low solar conversion efficiency is another shortcoming in the development of polymer electrolyte. The low number of iodide ions is the main problem of solar conversion efficiency. Several efforts have been done to improve solar conversion efficiency by adding ionic liquid into the polymer electrolyte. Ionic liquid is a plasticizing agent that helps to soften the polymer chain and give rise to easier polymer segmental motion. Addition of ionic liquid into the polymer electrolyte will introduce additional mobile ions. The ionic conductivity will be increased as the ion mobility increased. Other than that, the additional charge carriers will interact with the electron donors of the polymer which cause the polymer-polymer intermolecular interaction to become weaker. This will soften the polymer chains resulting in increased polymer segmental motion. Furthermore, increasing in polymer segmental motion implies that ionic liquid is able to enhance the amorphousness of a polymer electrolyte. Ion conduction only occurs at the amorphous region of the polymer electrolyte. Hence, the ionic conductivity is able to be increased. For the DSSCs performance, addition of ionic liquid not only enhances the current density, it also improves the solar cell efficiency of DSSCs.

The overall stability of current DSSCs electrolyte in general is poor. Although the current DSSCs has good performance in terms of ionic conductivity and solar conversion efficiency, its lifespan is relatively short. This can be observed when the DSSC cells are exposed to sunlight for a long period of time. The DSSC cells begin to show signs of degradation due to solvent evaporation and leakages.

1.3 Objectives

The objectives of this project are as listed below:

a. To synthesise a bio-based polyurethane acrylate (PUA) from Jatropha oil

b. To produce new gel polymer electrolyte based on PUA-TBAI gel polymer electrolyte

c. To determine the transport properties of charge carriers of PUA gel polymer electrolyte with TBAI salt in term of ionic conductivity, solar conversion efficiency and electrochemical behaviour

d. To evaluate the ratio effect of binary salt (TBAI and LiI) in PUA gel polymer electrolyte in order to obtain maximum ionic conductivity and maximum solar conversion efficiency for application in dye sensitized solar cells (DSSCs)

e. To optimize the weight percent of BMII ionic liquid added into the PUA-TBAI-LiI-I_2 gel polymer electrolyte in order to enhance the solar conversion efficiency of DSSCs

1.4 Content of the Thesis

The first chapter in this thesis highlights the overview of the work with a brief description of the introduction, problem statements and objectives of the work. The second chapter describes the classification and comparison of the different types of polymer electrolytes; liquid, solid, and gel. It also highlights the justification of the materials used to prepare the various systems of the polyurethane acrylate (PUA) based gel polymer electrolyte and the background for the workings of dye sensitized solar cells (DSSCs). The third chapter illustrates the methodology for the preparation of the jatropha oil based PUA, the PUA based gel polymer electrolyte systems, the various characterisation of the GPE samples together with the fabrication and performance validation of the DSSC cells. Characterisation of the GPE samples together with a brief background knowledge of the working principle of the characterisation technique are explained in this chapter.

The fourth chapter discusses the results of the wet chemical test which monitors the progress of the synthesis reaction using the oxygen content value (OOC value) and also the characteristics of the polymer produced such as acid value (AV), hydroxyl number (OH value) and iodine value (IV).

The fifth chapter discusses the result for the first GPE system which is the PUA polymer enhanced with the addition of tetrabutylammonium iodide (TBAI) salt with different amounts. The sixth chapter presents the further development of the first GPE system by adding LiI salt with varying amounts. The seventh chapter presents the final development of the second GPE system by the incorporation of BMII ionic liquid. All three GPE systems prepared in this study were subjected to a characterisation study which shows the thermal stability, interactions of the polymer with the iodide salts, electrochemical characteristics at room temperature together with the photovoltaic properties of the DSSC cells fabricated. Further electrochemical characterisation was conducted at various temperatures to further understand the feasibility of the GPE to be used as an electrolyte in DSSC cells.

Chapter 8 concludes the study conducted and suggestions for future work which can further improve upon the work done in this study.

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