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Effects of Iodide/Triiodide (I^-/I_3^-) Ratios on Palm Based Polyurethane Polymer Electrolyte for Solid-State Dye-Sensitized Solar Cell

(Kesan Nisbah Iodida/Triiodida (I^-/I_3^-) ke Atas Elektrolit Polimer Poliuretana Berasaskan Sawit untuk Sel Suria Terpeka Pewarna Keadaan Pepejal)

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ABSTRACT

In this investigation, optimization of iodide/triiodide (I^-/I_3^-) ratios on palm-based polyurethane (PU) polymer electrolyte's composition for solid-state dye sensitized solar cell (DSSC) application is explored. Polyurethane polymer electrolyte is synthesized under nitrogen gas atmosphere by pre-polymerization reactions between palm kernel oil monoester-OH (PKO-p) and methylene diphenyl diisocyanate (MDI) at different LiI/I_2 mole ratios. The effects of I^-/I_3^- mole ratios optimization on photovoltaic characteristics (short circuit current density, open circuit voltage, fill factor and power conversion efficiency), photo-response behavior (internal quantum efficiency) and internal charge transport properties (charge transport and recombination resistance, and chemical capacitances) of solid-state DSSC is examined by light-current-voltage (LIV), incident photon-to-current efficiency (IPCE) and electrochemical impedances spectroscopy (EIS) measurements. The current-voltage characteristic of FTO/TiO_2-dye/PU-LiI-I_2/Pt at LiI/I_2 1:0.125 mole ratio demonstrated a photovoltaic response with a power conversion efficiency of 1.8% under a standard AM 1.5G illumination with 42% of internal quantum efficiency. The short circuit current density (J_{sc}) and open circuit voltage (V_{oc}) are measured at 8.7 mA cm^{-2} and 0.68 V, respectively. These promising results could be a first step toward a new generation of low-cost and effective solid-state DSSC.

Keywords: Bio-based polyurethane; dye sensitized solar cell; iodide/triiodide; polymer electrolytes

ABSTRAK

Dalam kajian ini, pengoptimuman nisbah iodida/triiodida (I^-/ I_3^-) pada komposisi elektrolit polimer poliuretana (PU) berasaskan sawit untuk aplikasi sel suria terpeka pewarna (DSSC) diterokai. Elektrolit polimer poliuretana disintesis di bawah keadaan gas nitrogen dengan tindak balas pra-pempolimeran di antara minyak mentah sawit monoester-OH (PKO-p) dan metilena difenil diisosianat (MDI) pada nisbah mol LiI/I_2 yang berbeza. Kesan pengoptimuman nisbah mole (I^-/ I_3^-) ke atas ciri-ciri fotovoltaik (ketumpatan arus litar pintas, voltan litar terbuka, faktor pengisian dan kecekapan penukaran kuasa), kelakuan rangsangan cahaya (kecekapan kuantum dalaman) dan ciri-ciri pengangkutan cas dalaman (pengangkutan cas dan rintangan penggabungan semula, dan kapasitan kimia) untuk sel suria terpeka pewarna keadaan pepejal diuji oleh pengukuran arus voltan cahaya (LIV), kecekapan pencahayaan tuju kepada arus (IPCE) dan spektroskopi impedans elektrokimia (EIS). Ciri-ciri arus voltan FTO/TiO_2-dye/PU-LiI-I_2/Pt pada nisbah mol LiI/I_2 1:0.125 menunjukkan respons fotovoltaik dengan kecekapan penukaran kuasa sebanyak 1.8% di bawah pencahayaan AM 1.5G dan kecekapan kuantum dalaman sebanyak 42%. Ketumpatan arus litar pintas (J_{sc}) dan voltan litar terbuka (V_{oc}) diukur pada 8.7 mA cm^{-2} dan 0.68 V, masing-masing. Dapatan yang menjanjikan ini boleh menjadi langkah pertama bagi sel suria terpeka pewarna keadaan pepejal generasi baru yang berkos rendah dan berkesan.

Kata kunci: Poliuretana berasaskan bio; sel suria terpeka pewarna; iodida/triiodida; elektrolit polimer

INTRODUCTION

According to world energy outlook 2010, the growth of global population in combination with industrial developments will pose an enormous challenge in meeting the rising energy demand in the near future (Birol 2010). Electricity is the fastest growing form of end-use energy, and the net global electricity generation is projected to grow by 2.2% per year from 2008 to 2035. The rising energy demand, coupled with environmental concerns, as well as the higher prices of fossil fuels have motivated governments to seek alternative, environmentally-friendly low-carbon energy sources. There are various energy sources to meet the increasing energy needs such as fossil fuels and nuclear energy which are the biggest supply of electricity generations. However, the combustion of fossil fuels produces greenhouse gas emissions such as carbon dioxide, which are believed to impose a significant impact on global warming and climate change. Moreover, the disposal of the radioactive waste has been a serious challenge in the nuclear energy industry. Therefore, renewable energy sources, such as solar, hydropower, wind and biomass are expected to play an essential role in moving the world to a more secure, reliable and sustainable energy system (Birol 2010).

Solar energy is the most abundant permanent energy resource on earth. Solar cells or photovoltaic (PV) cells, are electrical devices that directly convert sunlight into electricity. The first generation of solar cells from industrialization point of view are based on crystalline silicon. This type of solar cells is currently dominant in the PV market due to the high efficiency of up to 25%. However, the manufacture of silicon-based solar cells involves high purity silicon, the manufacturing processes of which are extremely expensive. The second generation of solar cells are normally referred to as thin-film solar cells. Amorphous silicon, cadmium telluride (CdTe), gallium arsenide (GaAS) and copper indium gallium selenide (CIGS) are the three most commonly used materials for the second-generation solar cells (Sakid & Ahad 2018). The thin-film technology allows the second-generation solar cells to use far less materials required in a solar cell, which significantly reduce the production cost in contrast to the first-generation solar cells. However, the efficiencies of thin-film solar cells are lower than the first-generation solar cells, ranging between 10-20% (Green et al. 2011).

Dye-sensitized solar cells (DSSCs), also known as Grätzel cells, were discovered in 1991 by O'Regan and Grätzel. In contrast to conventional systems where the semiconductor takes both the function of light absorption and charge carrier transport, these two functions are separated in DSSCs. The light absorption is performed by a monolayer of dye molecules attached to a mesoporous layer of a wide band gap semiconductor. Charge separation takes place at the semiconductor/dye interface. Charge carriers are transported in the conduction band edge (CB) of the semiconductor to the charge collector (Grätzel 2003). DSSCs are considered to be a technology between the second and third generation solar cells (Hagfeldt et al. 2010). The record efficiencies of DSSCs have shown up to 12% in small cells (Yu et al. 2010; Yella et al. 2011) and 10% in sub-modules (O'Regan & Grätzel 1991). Of particular interest is the low production cost for the fabrication of a DSSC. The materials used in DSSCs are inexpensive, for instance, the commonly used semiconductor, titanium is very cheap and is widely used as pigment in white paint.

Photo-electrochemical solar cells based on nanostructured dye-sensitized titanium dioxide (TiO_2) have attracted worldwide attention as a low-cost alternative to conventional silicon solar cells. Such solar cells have impressive conversion efficiencies reaching around 10% where the electrolyte is typically based on liquid acetonitrile solvent and a I^-/I_2 redox couple. However, using liquid electrolyte in the DSSC also raise a few problems such as losing electrolyte by the leakage and/or volatility of the electrolyte solution. It also has low durability, high temperature instability, flammability and technical difficulties in sealing. Su'ait et al. (2014) has suggested a cheap bio-based polyurethane polymer electrolyte as an alternative to act as electrolyte in DSSCs. It has been proved that polyurethane electrolyte gives better stability, no leakage of electrolyte and also has adhesive properties that can deny the use of sealing. The photovoltaic characteristic was observed and reported by Su'ait et al. (2014) was 0.003% with a room for improvement.

In this work, the optimization of bio-based polyurethane polymer electrolyte for DSSCs application was explored. Polyurethane polymer electrolyte was synthesized by pre-polymerization reaction between palm kernel oil monoester-OH (PKO-p) with methylene diphenyl diisocyanate (MDI) with the presence of lithium iodide/iodine (LiI/I_2) under nitrogen gas atmosphere. The purpose of this study was to investigate the effect of different ratio of LiI/I_2 and its optimization on photovoltaic characteristics, photo-response behavior, and internal charge transport properties of DSSC by light current voltage (*I-V*), incident photon to current efficiency (IPCE) and electrochemical impedance spectroscopy (EIS) measurements.

MATERIALS AND METHODS

MATERIALS

2,4'-Methylene diphenyl diisocyanate (MDI) was commercially obtained from Cosmopolyurethane (Malaysia) Sdn. Bhd. Palm kernel oil monoester-OH (PKO-p) from palm kernel oil was synthesized by polyesterification reaction in UKM laboratory using established method by Badri et al. (2000). Lithium iodide (LiI), ethylene carbonate (EC), iodine (I_2) and titanium dioxide (TiO_2) were supplied by Sigma Aldrich (St. Louis, Mo, USA). DSSC component, di-tetrabutyl ammonium cis-bis(isothiocyanato) bis(2,2' -bipyridyl-4,4'-di carboxylato) ruthenium (II) dye (N-719), platinum (Pt) paste under commercial name Platisol T, conventional electrolyte consist of lithium iodide salt in ionic liquid and pyridine derivative based electrolyte with 50 mM of tri-iodide in acetonitrile (Iodolyte AN-50) and 3 mm thick fluorine-doped tin oxide (FTO) with 8 and 15 Ω cm^{-2} as transparent conductive oxide were purchased by Solaronix (Aubonne, Switzerland). Acetone and ethanol 99.9% were supplied by SYSTERM ChemAR (Kielce, Poland). All materials were used without further purification.

SYNTHESIS OF POLYURETHANE

PKO-p was mixed together with 2,4'-MDI in 5 mL acetone at 1:2 ratio (NCO:OH) at ambient temperature under Argon gas atmosphere. These solutions were stirred to a homogenous solution for 5 min. EC was added in the mixture as the plasticizer at a fix amount of 20% (*w*/*w*). It was dissolved in acetone together with varying ratio of LiI:I_2 from 1:1 to 1:0.05 and fixed amount of LiI 25% (*w*/*w*). This mixture was stirred for an hour prior to inclusion to PU pre-polymer, and further mixed for another 5 min. Half of the prepared solution will then casted into Teflon mould and allowed to evaporate at room temperature for an hour and the remaining will be stored for further use. The reaction involved in the polymerization synthesis between MDI and PKO-p was additional polymerization *via* nucleophilic substitution reactions as proposed by Su'ait et al. (2014).

FABRICATION OF DSSC

Standard cell fabrication procedures were followed based on the instructions from Solaronix Company. TiO_2 paste was spread on the FTO using doctor-blade technique with the support of adhesive tapes on both sides. The TiO_2 paste was sintered at 500°C for 30 min to activate quasi transparent platinum layer and was sensitized in 3 mM solution of N719 dye solution at room temperature for 12 h. Platinized FTO counter electrode was prepared by brush-painting technique, firing at 450°C for 30 min. The TiO_2 photo-anode electrode was then assembled with a platinum counter electrode and filled with iodide-based electrolyte with 50 mM of tri-iodide in acetonitrile. The PU-Li/I_2 based electrolytes were fabricated by the same procedures according to Su'ait et al. (2014).

LIGHT-CURRENT VOLTAGE (*I-V*) ANALYSIS

Light-current voltage (*I-V*) measurement is the most important and conventional technique for the assessment of the photovoltaic performance in DSSC devices. A standard illumination of air-mass 1.5 global (AM 1.5 G) with an irradiance of 100 mW/cm^2 is normally used for the *I-V* characterization of DSSCs. The *I-V* characteristics are monitored under solar irradiation by changing the external load from zero load (short-circuit conditions) to infinite load (open-circuit conditions). The most important photovoltaic parameter to evaluate the performance of DSSC devices is the overall light-to-electricity conversion efficiency (η), which is determined by the product of the short-circuit current density (J_{sc}), open-circuit voltage (V_{oc}), and fill factor (*FF*) divided by the intensity of the incident light (P_{in}) in Equation (1):

$$\eta = \frac{V_{oc} \cdot J_{sc} \cdot FF}{P_{in}} \tag{1}$$

These three parameters J_{sc}, V_{oc}, and *FF* can be extracted from the *I-V* curves. The V_{oc} of DSSCs is defined as the difference between the quasi fermi level of the electrons in the mesoporous TiO_2 film and the redox potential of the electrolyte. The *FF* is defined as the ratio of the maximum power ($P_{\max}$) of the solar cell divided by the product of V_{oc} and J_{sc}. The $P_{\max}$ is defined as the product of J_{sc} and V_{oc} at the maximum power point according to Equation (2):

$$FF = \frac{P_{\max}}{J_{sc} \cdot V_{oc}} \tag{2}$$

QUANTUM EFFICIENCY (QE) ANALYSIS

Quantum Efficiency (QE) is performed by incident photon to current efficiency (IPCE) model Bentham. QE is a measure of how efficient the device converts the incident light into electrical energy at a given wavelength. There are two types of QE: external quantum efficiency (EQE) and internal quantum efficiency (IQE). EQE is the ratio between the number of collected carriers and the number of all the incident photons on the device active area at a given wavelength. IQE is the ratio between the number of collected carriers and the number of all the absorbed photons by only the active absorber at a given wavelength. The difference between IQE and EQE is important for distinguishing loss mechanisms between optical absorption properties of the entire device and photo-conversion properties of the absorbing materials. IPCE is also useful for studying degradation properties of devices. The general reduction in the IPCE during time points to deterioration of photo-conversion properties of the active material, while the change of the shape of the IPCE curve may point to morphological alterations in the absorbing layer. The IPCE measurement is also quantitatively evaluate the spectral response of a DSSC device. The IPCE value corresponds to the ratio of photocurrent density in the external circuit divided by the monochromatic photon flux, and can be calculated according to Equation (3):

$$IPCE = 1240 \frac{J_{ph}}{\lambda \cdot P_{in}} \tag{3}$$

Where, J_{ph} is the short-circuit current density generated by the monochromatic light, λ and P_{in} are the wavelength and light intensity, respectively. The IPCE describes how efficiently the incident photons are converted to electrons. The IPCE value can also be expressed as Equation (4):

$$IPCE = (LHE)\,(\Phi_{inj})(\Phi_{reg})(\eta_{cc}) \tag{4}$$

Where, LHE is the light harvesting efficiency; Φ_{inj} and Φ_{reg} represent the quantum yields of electron injection and dye regeneration, respectively; and η_{cc} is the charge collection efficiency. J_{sc} in full sunlight can be calculated by the integral of the obtained IPCE spectrum. Note that a discrepancy of the J_{sc} between the value from the *I-V* measurement and the integrated one from the IPCE spectrum might exist since the IPCE is typically recorded at lower light intensities.

IMPEDANCE (EIS) ANALYSIS

Internal charge transport properties can be measured using electrochemical impedance spectroscopy (EIS). EIS measurements were performed with an Autolab Potensiostat Galvanostat with frequency response analyzer (FRA) module. Impedance measurements were implemented in the frequency range 1 MHz to 0.05 mHz, using 10 mV AC amplitude, with illumination provided by a Xenon lamp (XPS-150). Impedance was measured under illumination and under dark conditions. Both conditions were set to a range of intensities and impedance was performed at current density, maximum voltage and open-circuit condition. From EIS, several parameters can be obtained, such as charge-transfer resistance (R_{pt}) at the CE, electron recombination resistance (R_{ct}) at the TiO_2/dye/electrolyte interface as well as diffusion resistance in the electrolyte etc.

RESULTS AND DISCUSSION

LIGHT-CURRENT VOLTAGE (*I-V*) ANALYSIS

Table 1 shows photovoltaic characteristics of different PU electrolyte compositions, under light intensity of 100 W m^{-2}. J_{sc} (mA cm^{-2}) and V_{oc} (V) values, obtained for the polyurethane polymer electrolyte solar cells of the type, FTO/TiO_2/dye/ Pu-LiI/I_2/Pt, fabricated using the electrolyte compositions of LiI to I_2 ratio (1:1 to 1:0.05). In this system, LiI and I_2 salt was used as redox couples in bio-based PU polymer electrolyte according to stochiometric mole ratios according to the Equation (5) as follows:

$$PU + LiI \rightarrow Li^+ I^- \quad (5)$$

$$I^- + I_2 \leftrightarrows I_3^- \quad (6)$$

$$I^- + 2e \leftrightarrows 3I^- \quad (7)$$

It can be seen from Table 1 that out of all the six samples studied, the optimized power conversion efficiency of 1.8% (η) were obtained at LiI to I_2 ratio of 1:0.1250 with J_{sc} of 8.7 mA cm^{-2}, V_{oc} of 0.67 mV and FF of 30.48%.Whereas, solar cell G was used as control standard DSSC using Iodolyte AN-50 as electrolyte. It is clear that from our data, power conversion efficiency and J_{sc} increased from cell A to C along the increase of iodine ratios and afterwards, dropped with the increase weight of iodine in bio-based PU polymer electrolyte. According to Papageorgiou et al. (1996), by increasing the presence of iodine in electrolyte, it will also increase triiodide ion which will help chemical availability of the triiodide ion in the electrolytes to regenerate the oxidized N719 dyes. Another reason for the positive increment in power conversion energy is mass transport limitation of the triiodide ion. It has been proposed that a Grotthuss-type mechanism can partially facilitate the transport of I_3^- in ionic liquid electrolytes, when high concentrations of iodide and triiodide are present (Papageorgiou et al. 1996).

As we can observe from the Equation (5) to (7), the ratio 1:1 LiI/I_2 is the highest ratio since LiI is the limiting reactants. Theoretically, triiodide ion will fully utilize for the ratio 1:1 but instead its show the lowest J_{sc} and power conversion efficiency as indicted in cell F. Higher concentration of iodine also can reduce the electrolyte charge and mass transport limitation. It can be achieved by allowing charge transport without a net mass transport through the exchange of iodide ions. Thus, cell A, B and C showed increment in power conversion efficiency; whereas, cell D, E and F show the opposite behavior.

TABLE 1. Photovoltaic characteristic of different PU electrolyte compositions, under light intensity of 100 W m^{-2}

Cell	Ratio LiI/I_2	J_{sc} (mA cm^{-2})	V_{oc} (mV)	FF	η (%)
A	1:0.0500	5.0	0.68	34.00	1.1
B	1:0.0625	5.7	0.70	28.14	1.1
C	1:0.1250	8.7	0.67	30.48	1.8
D	1:0.2500	7.0	0.62	33.13	1.4
E	1:0.5000	3.6	0.56	37.72	0.8
F	1:1.0000	3.4	0.54	36.21	0.6
G	Iodolyte AN-50	10.8	0.75	32.96	2.7

One major reason for this lower J_{sc} achieved by the PU solar cells could be attributed to visible light absorption by the I_3^-, the absorption tail of which extends to up to 500 nm. This light absorption severely interferes the light harvesting efficiency of the dye molecules (Nakade et al. 2005). Increasing the amount of iodine to the electrolyte resulted in more electron recombination between the injected photoelectron in the TiO_2 and the I_3^- (Nakade et al. 2005).

QUANTUM EFFICIENCY (QE) ANALYSIS

Figure 1 shows the internal quantum efficiency (IQE) – wavelength (nm) spectra of the DSSCs prepared with Iodolytes liquid electrolyte and polyurethane electrolytes with different LiI/I_2 mole ratios. It can be seen that, the solid-state DSSCs prepared with the polymer electrolytes exhibit lower IPCE performance compared to the cell prepared with the liquid electrolyte. The solid-state cell prepared with the polyurethane – LiI/I_2 polymer electrolytes with mole ratio 1:0.125 electrolyte shows much higher IQE in the wavelength region of 450-550 nm in comparison to 1:1 mole ratio. It can be seen from this figure that polyurethane polymer electrolyte with mole ratio 1:0.125 of LiI/I_2 leads to a substantial increase in the maximum IQE value.

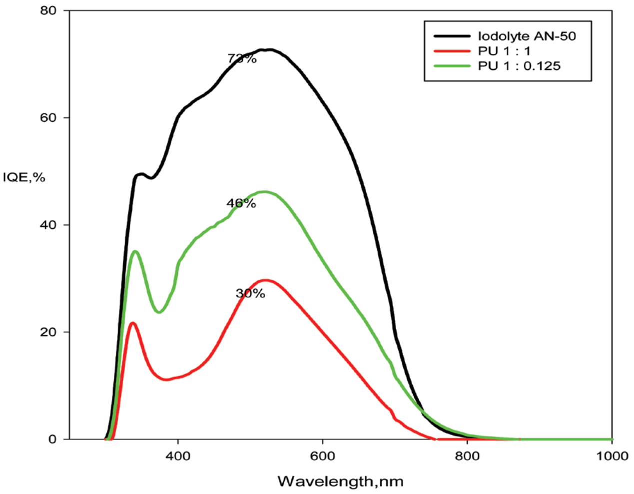

FIGURE 1. IQE spectra of DSSCs based polyurethane electrolytes with different LiI/I_2 mole ratios

However, with further increase of this ratio to 1:1 caused to a decrease in the IPCE value. This observation is consistent with the optimum power conversion efficiency result that shown in previous Table 1. Internal quantum efficiency, IQE was used because it is typically done by recording and excluding the light reflected from the device and using only the absorbed portion of light for the calculation of quantum efficiency. Since the absorbed light is typically less than the total incident light as there will always be some loss of light due to reflections.

TABLE 2. Electrical parameter characteristics of different PU electrolyte compositions, under light intensity of 100 W m^{-2}

LiI/I_2 Ratio	R_s	R_{pt}	C_{pt}	R_{ct}	C_μ	R_e	C_e
at $V = 0$							
1:0.0625	36.36	244.40	9.25×10^{-5}	2.56×10^{2}	4.73×10^{-3}	–	–
1:0.1250	29.25	160.00	1.21×10^{-4}	1.60×10^{2}	1.09×10^{-2}	–	–
1:0.2500	35.85	201.80	3.31×10^{-5}	1.00×10^{2}	3.58×10^{-9}	–	–
1:0.5000	28.64	731.40	3.99×10^{-5}	–	–	–	–
1:10000	73.38	929.50	2.52×10^{-7}	7.38×10^{4}	1.35×10^{-7}	–	–
at V_{max}							
1:0.0625	35.96	283.60	1.10×10^{-4}	108.1	0.16	–	–
1:0.1250	30.15	17.20	2.65×10^{-5}	7.39	0.11	–	–
1:0.2500	25.94	17.29	1.08×10^{-3}	8.00	1.66×10^{-4}	–	–
1:0.5000	29.03	44.43	1.07×10^{-5}	23.72	1.00×10^{-4}	–	–
1:1.0000	62.71	1.60×10^{3}	7.11×10^{-7}	1.40×10^{4}	2.42×10^{-6}	–	–
at V_{OC}							
1:0.0625	26.29	35.37	3.21×10^{-9}	–	–	–	–
1:0.1250	24.83	4.71	2.28×10^{-4}	10.11	4.58×10^{-2}	12.27	9.53×10^{-3}
1:0.2500	23.39	4.85	1.12×10^{-9}	17.50	1.03×10^{-3}	36.86	5.73×10^{-2}
1:0.5000	28.84	8.92	7.23×10^{-2}	11.62	1.03×10^{-3}	61.50	1.8×10^{-4}
1:1.0000	71.99	4.05×10^{3}	1.67×10^{-6}	6.86×10^{3}	3.15×10^{-6}	–	–

IMPEDANCE (EIS) ANALYSIS

Figure 2 indicates EIS spectra of DSSCs based polyurethane electrolytes with 25 wt.% LiI/I_2 at different applied voltages ($V = 0$, V_{max} & V_{oc}) and the electrical parameters were extracted to Table 2. Electrical characteristic of R_{pt}, C_{pt}, R_{ct}, C_μ, R_e, C_e and R_s at different PU electrolyte compositions, under light intensity of 100 W m^{-2} was defined as follows; R_{pt} and C_{pt} are charge transfer resistance and capacitance respectively at counter electrode/electrolyte interface; R_{ct} and C_μ charge transfer resistance and chemical capacitance respectively due to recombination of electron at TiO_2/dye/electrolyte; R_e and C_e are charge transfer resistance and capacitance for electron recombination and charge accumulation at the FTO/electrolyte; and R_s is a sheet resistance or series resistance of the FTO (Omar et al. 2013). It is observed that the R_{ct} of the highest efficiency is reduced drastically from ~160 Ω at zero potential, 0 V to ~5 Ω at open circuit voltage, 0.66 V indicating the lowest barrier for the mass transport inside the cell. On the other hand, R_e and C_e are only appeared at V_{OC} where the resistant value closed to the bulk resistance (estimated conductivity around ~$10^{-3}/10^{-4}$ S cm^{-1}) of the electrolytes reported by Su'ait et al. 2014.

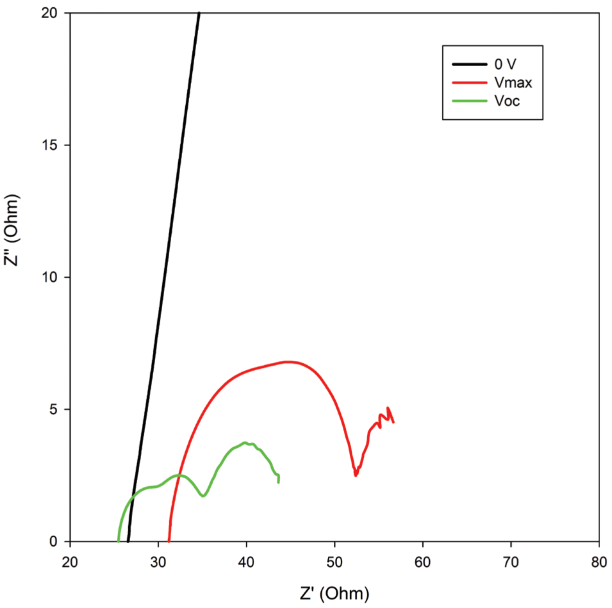

FIGURE 2. EIS spectra of DSSCs based polyurethane electrolytes with $LiI:I_2$ mole ratio of 1:0.125 at different applied voltages

CONCLUSION

Bio-based polyurethane electrolyte from palm kernel oil with the inclusion of varied lithium iodide-iodine salts, LiI/I_2 has been successfully prepared by solution casting technique and has been tested in dye-sensitized solar cell. A DSSC of FTO/TiO_2-dye/PU-LiI-I_2/Pt with optimized mole ratio of 1: 0.125 LiI/I_2 give a response under light intensity of 100 mW cm^{-2} indicated the photovoltaic effect with the J_{sc} of 8.7 mA cm^{-2} and V_{oc} of 0.68 V respectively. These cell gives a 1.8% of η with a 42% of IQE and charge transfers value.

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