

## Research Article

# Characterizations of Chitosan-Based Polymer Electrolyte Photovoltaic Cells

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The membranes 55 wt.% chitosan-45 wt.% NH<sub>4</sub>I, 33 wt.% chitosan-27 wt.% NH<sub>4</sub>I-40 wt.% EC, and 27.5 wt.% chitosan-22.5 wt.% NH<sub>4</sub>I-50 wt.% buthyl-methyl-imidazolium-iodide (BMII) exhibit conductivity of  $3.73 \times 10^{-7}$ ,  $7.34 \times 10^{-6}$ , and  $3.43 \times 10^{-5}$  S cm<sup>-1</sup>, respectively, at room temperature. These membranes have been used in the fabrication of solid-state solar cells with configuration ITO/TiO<sub>2</sub>/polymer electrolyte membrane/ITO. It is observed that the short-circuit current density increases with conductivity of the electrolyte. The use of anthocyanin pigment obtained by solvent extraction from black rice and betalain from the callus of *Celosia plumosa* also helps to increase the short-circuit current.

## 1. Introduction

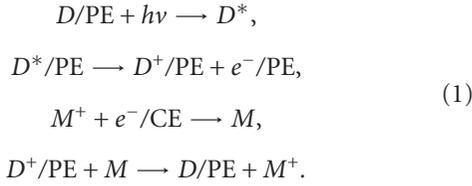
Solid polymer electrolytes are being utilized in the fabrication of solid-state photoelectrochemical (PEC) cells. The configuration of such cells in general is photoactive electrode/polymer electrolyte/counter electrode. The polymer electrolyte basically consists of polymer and salt together with a redox couple. Examples include polyethylene oxide (PEO)-KI-I<sub>2</sub> [1, 2], chitosan-PEO-NH<sub>4</sub>I-I<sub>2</sub> [3], PEO-NH<sub>4</sub>I-I<sub>2</sub> [4], and polyvinylchloride (PVC)-LiClO<sub>4</sub> [5]. In order to enhance performance of the PEC cell, plasticizers have been used either as an additive or as a solvent or cosolvent. Examples include PEO-poly(vinylidene fluoride) (PVDF)-LiI-I<sub>2</sub> [6] which were dissolved in propylene carbonate and dimethoxyethane, polyvinyl pyrrolidone (PVP)-polyethylene glycol (PEG)-KI-I<sub>2</sub> [7], PEO-ethylene carbonate (EC)-propylene carbonate (PC)-KI-I<sub>2</sub> [8], and poly(epichlorohydrin-co-ethylene oxide) (P(EPIEO))-poly(ethylene glycol) methyl ether (P(EGME))-NaI-I<sub>2</sub> [9].

The photoactive electrode is usually a semiconducting material, can be inorganic or organic, and serves to produce photoelectrons when illuminated. The most common inorganic semiconducting material used is TiO<sub>2</sub> [10–13]. Other inorganic semiconductor materials include CdSe [14], ZnTe

[3], and ZnSe [15]. Organic semiconductor materials include the polythiophenes. Examples are poly(3-methylthiophene) (P3MT) [2] and poly[3-(4-octylphenyl)thiophene] (POPT) [16]. Counter electrodes that have been used include graphite [5] and platinum (Pt) [17–20]. A large difference in the Fermi levels between the photoanode and counter electrode under illumination will exhibit a large potential difference and thus will enable a larger current to be delivered by the device.

Types of redox couples or mediators include I<sup>-</sup>/I<sub>3</sub><sup>-</sup> [21–24], 5-mercapto-1-methyltetrazole cesium salt (CsT)/di-5-(1-methyltetrazole) (T<sub>2</sub>) [14], Fe<sup>3+</sup>/Fe<sup>2+</sup> [25], Co(II/III) [26], OH<sup>-</sup>/O<sub>2</sub><sup>-</sup>, and S/S<sup>2-</sup> [27]. The performance of these PEC cells can be improved by soaking the photoactive electrode with a suitable dye material. When the PEC cell is illuminated, electrons in the dye are excited and injected into the conduction band of the semiconductor photoelectrode. The dye-sensitizer is thereby oxidized. The mediator (I<sup>-</sup>/I<sub>3</sub><sup>-</sup> couple) in the electrolyte regenerates the oxidized dye producing photocurrent and photovoltage in the cell. Examples of dye materials are Ru(4,4'-dicarboxylic acid-2,2' bipyridine)<sub>2</sub>(thiocyanate) [28] and (tri(cyanato)-2,2'-terpyridyl-4,4'-tricarboxylate) Ru(II) [29]. Natural dyes are also being used in dye-sensitized solar cells (DSSCs). These include anthocyanin [30], chlorophyll [31], and

carotenoid [32]. The photovoltaic performance of a solid-state DSSC may be explained as follows [33]:



Here  $D$  is the dye, PE is photoelectrode, CE is counter electrode, and  $M$  is redox couple or mediator. In this work, polymer electrolytes consisting of chitosan and  $\text{NH}_4\text{I}$  with ethylene carbonate (EC) as plasticizer and ionic liquid (IL) as ionic dopant were prepared and used in  $\text{TiO}_2/\text{ITO}$  cells. Anthocyanin, a natural pigment that was extracted from black rice, and betalain that was extracted from callus of *Celosia plumosa* (locally known as *balong ayam*) were used as the material to enhance electron injection into the conduction band of the photoelectrode.

## 2. Experimental

Details of electrolyte preparation can be found in previous works [34, 35]. Conductivity of the electrolytes was calculated using the equation

$$\sigma = \frac{d}{R_b A}, \tag{2}$$

where  $\sigma$  is conductivity, and  $A$  and  $d$  are area and thickness of the electrolyte film, respectively.  $R_b$  is the bulk resistance which is derived from the high-frequency intercept on the Cole-Cole plots. The impedance of the samples was measured using the HIOKI 3531-01 LCR Hi-Tester in the frequency range from 50 Hz to 1 MHz from room temperature to 343 K. For solar cell application, the electrolyte was added with some iodine crystals,  $\text{I}_2$  (10% of salt amount) to provide the redox couple  $\text{I}^-/\text{I}_3^-$ .

The anthocyanin pigment was extracted from black rice grains and betalain from callus of *Celosia plumosa*. Black rice and callus were immersed in 95% ethanol and methanol solution, respectively. The pigment solutions were kept at room temperature in the dark for 24 hours. Filtration was done to remove the residues and the pH of the dye was adjusted accordingly by adding hydrochloric acid. Procedure for callus production can be found in [36].

An indium tin-oxide (ITO) glass ( $2.5 \times 2.5 \text{ cm}^2$ ) with sheet resistance of  $5 \Omega \text{ cm}^{-2}$  was cleaned with distilled water and acetone. A part of the ITO layer was covered and the active area of the ITO layer is about  $0.16 \text{ cm}^2$ .  $\text{TiO}_2$  paste (JGC Catalysts & Chemicals Ltd) was doctor-bladed on the ITO substrate to form the photoactive cathode. The  $\text{TiO}_2$  layer was then heated at 773 K for 1 hour. The thickness of the film was controlled using adhesive tape of thickness  $100 \mu\text{m}$ . After cooling to 373 K, the  $\text{TiO}_2$  electrode was immersed in an anthocyanin solution for 24 hours and the ITO sheet resistance of  $11 \Omega \text{ cm}^{-2}$  was obtained. The photoelectrode was washed with water to remove impurities and then with ethanol to remove trapped water from the initial washing.

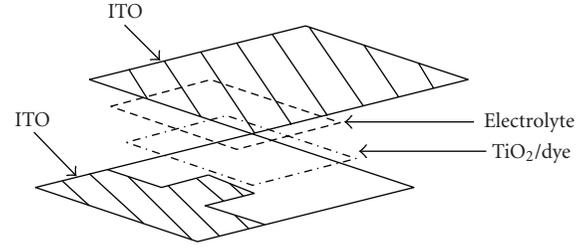


FIGURE 1: Diagram of PEC cell.

TABLE 1: Electrolytes composition and conductivity at ambient temperature.

Sample	Conductivity ( $\text{S cm}^{-1}$ )
55 wt.% chitosan-45 wt.% $\text{NH}_4\text{I}$	$3.73 \times 10^{-7}$
33 wt.% chitosan-27 wt.% $\text{NH}_4\text{I}$ -40 wt.% EC	$7.34 \times 10^{-6}$
27.5 wt.% chitosan-22.5 wt.% $\text{NH}_4\text{I}$ -50 wt.% BMII	$3.43 \times 10^{-5}$

BMII: Buthyl-methyl-imidazolium-iodide.

An equal area of polymer electrolyte was placed above the  $\text{TiO}_2$  photoelectrode. Another ITO glass plate (counter electrode) was placed over the whole assembly and clamped with a paper clip. The  $J$ - $V$  characteristics of the dye-sensitized solar cells were obtained under white light illumination ( $100 \text{ mW cm}^{-2}$ ) using a Keithley 2400 electrometer.

The assembly of the fabricated cell is shown in Figure 1.

## 3. Results and Discussion

Table 1 lists the composition of the polymer electrolytes and the respective room temperature conductivity.

Figure 2 shows the plot of log conductivity versus temperature for each of the electrolyte listed in Table 1. It is noticed in all plots that conductivity increases with temperature. The conductivity-temperature relationship is Arrhenian.

Tables 2 to 4 list the short-circuit current density  $J_{\text{sc}}$ , open circuit voltage (OCV), fill factor (ff), efficiency ( $\eta$ ) of PEC cells and the type of polymer electrolyte, redox couple, and dye material used. The photoactive and counter electrodes of all the PEC cells listed are  $\text{TiO}_2$  and ITO coated with platinum, respectively, except for PEC cells utilizing the chitosan containing electrolyte where the photoactive electrode is  $\text{TiO}_2$  and the counter electrode is ITO glass without any catalytic material coating.

The conductivity of an electrolyte depends on the ability of the polymer host to solvate the salt. Polymers with higher dielectric constant will serve the purpose better. Apart from that, the lattice energy of the salt should be low since this would help to increase dissociation of the salt. Thus, it may be understood why the different electrolyte systems in Table 2 exhibit different room temperature conductivity. From the results in Table 2, it may be inferred that the dielectric constant of PBA [37] is slightly higher than that

TABLE 2: Characteristics of PEC cells using polymer-salt systems.

Sample	Dye	Intensity (mW cm <sup>-2</sup> )	Redox couple	$\sigma$ (S cm <sup>-1</sup> )	$J_{sc}$ (mA cm <sup>-2</sup> )	OCV (V)	ff	$\eta$ (%)	Ref.
91 wt.% Poly(butyl acrylate) + 9 wt.% NaI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$2.10 \times 10^{-6}$	2.20	0.61	0.37	0.51	[37]
87.5 wt.% PEO + 12.5 wt.% NaI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$2.02 \times 10^{-6}$	1.51	0.83	0.61	0.76	[38]
92 wt.% PEO + 8 wt.% KI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$6.33 \times 10^{-5}$	5.04	0.62	0.63	1.96	[1]
89 wt.% P(EPI-EO) + 11 wt.% NaI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$1.9 \times 10^{-5}$	0.55	0.75	—	0.19	[9]
71 wt.% P(EPI-EO) + 29 wt.% NaI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	120	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$1.0 \times 10^{-5}$	0.46	0.71	0.67	0.22	[39]
55 wt.% Chitosan + 45 wt.% NH <sub>4</sub> I	None	56.4	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$3.73 \times 10^{-7}$	0.005	0.15	0.22	—	[35]

TABLE 3: Characteristics of PEC cells using plasticized polymer-salt systems.

Polymer electrolyte Sample	Dye	Intensity (mW cm <sup>-2</sup> )	Redox couple	$\sigma$ (S cm <sup>-1</sup> )	$J_{sc}$ (mA cm <sup>-2</sup> )	OCV (V)	ff	$\eta$ (%)	Ref.
27.2 wt.% PEO + 2.8 wt.% Pr <sub>4</sub> N <sup>+</sup> I <sup>-</sup> + 70 wt.% EC	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$4.9 \times 10^{-5}$	0.051	0.44	0.48	0.01	[40]
PEO + LiI + EC + PC	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	27	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$1.2 \times 10^{-3}$	2.10	0.60	0.62	2.90	[41]
13.5 wt.% PAN + 7.3 wt.% Pr <sub>4</sub> N <sup>+</sup> I <sup>-</sup> + 32.3 wt.% EC + 46.9 wt.% PC	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	60	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$3.0 \times 10^{-3}$	3.73	0.69	—	2.99	[42]
43.5 wt.% P(EPI-EO) + 13 wt.% NaI + 43.5 wt.% P(EGME)	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$1.7 \times 10^{-4}$	1.88	0.7	—	0.52	[9]
33 wt.% chitosan + 27 wt.% NH <sub>4</sub> I + 40 wt.% EC	None	56.4	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$7.3 \times 10^{-6}$	0.007	0.22	0.18	—	[35]

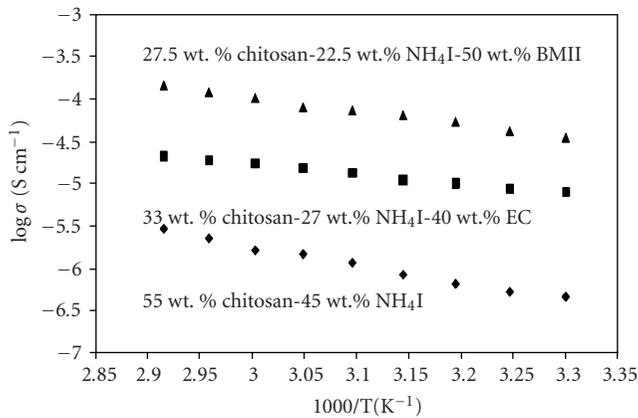


FIGURE 2: Temperature dependence of ionic conductivity.

of PEO [38] because for a higher salt content in the PEO-NaI electrolyte, the conductivity is almost similar to that of PBA-NaI electrolyte at room temperature (assumed 298 K). The composition of the P(EPI-EO)-NaI electrolyte [9] and that of PEO-NaI electrolyte [38] are almost similar. However, the P(EPI-EO)-NaI electrolyte exhibits a conductivity of almost one order of magnitude higher than that of PEO-NaI

electrolyte. This has been attributed to the presence of Cl in P(EPI-EO) that played a part in reducing the crystallinity of the polymer [45, 46], thus resulting in higher conductivity.

Again referring to Table 2, the composition of the PEO-KI electrolyte [1] is almost similar to the composition of PBA-NaI electrolyte [37], but the conductivity of the former is more than one order of magnitude higher. Although it has been inferred that the dielectric constant of PBA is slightly higher than PEO, the higher conductivity of the PEO-KI electrolyte may be attributed to the lower lattice energy of KI compared to that of NaI (according to calculations performed using the Kapunstinskii and Born-Haber equations). Hence, assuming temperature to be the same, the number density of mobile cations is higher in the PEO-KI electrolyte than that in the PBA-NaI electrolyte resulting in a higher conductivity. Another example from Table 2 showing the fact that lower lattice energy of salt results in higher conductivity can be observed from the conductivity of the PEO-KI and PEO-NaI electrolyte systems. At almost similar composition, higher conductivity was observed in the PEO-KI electrolyte compared to PEO-NaI electrolyte. The lattice energy of KI is 614.5 kJ mol<sup>-1</sup> and that of NaI is 674 kJ mol<sup>-1</sup>. The lower conductivity of P(EPI-EO)-based electrolyte with 29 wt.% NaI compared to the same polymer-based electrolyte with 11 wt.% NaI has been attributed to ion pairs formation

TABLE 4: Characteristics of PEC cells using polymer-salt-ionic liquid systems.

Polymer electrolyte Sample	Dye	Intensity (mW cm <sup>-2</sup> )	Redox couple	$\sigma$ (S cm <sup>-1</sup> )	$J_{sc}$ (mA cm <sup>-2</sup> )	OCV (V)	ff	$\eta$ (%)	Ref.
52.5 wt.% PEO + 17.5 wt.% KI + 30 wt.% EMImTFSI	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$8.82 \times 10^{-5}$	4.02	0.77	0.56	1.75	[43]
15 wt.% PEO + 5 wt.% KI + 80 wt.% EMISCN	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	27	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$2.25 \times 10^{-5}$	1.89	0.65	0.52	0.63	[44]
17.5 wt.% PEO + 2.5 wt.% NaI + 80 wt.% EMImTFO	Ru(dcbpy) <sub>2</sub> (NCS) <sub>2</sub>	60	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$4.72 \times 10^{-5}$	5.65	0.79	0.55	2.45	[38]
27.5 wt.% Chitosan + 22.5 wt.% NH <sub>4</sub> I + 50 wt.% BMII	Anthocyanin from black rice	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$3.43 \times 10^{-5}$	0.065	0.23	0.22	—	—
27.5 wt.% Chitosan + 22.5 wt.% NH <sub>4</sub> I + 50 wt.% BMII	Betalain from callus of <i>Celosia plumosa</i>	100	I <sup>-</sup> /I <sub>3</sub> <sup>-</sup>	$3.43 \times 10^{-5}$	0.029	0.14	0.22	—	—

and cross linking sites that hinder segmental motion of the polymer chains thereby decreasing ionic mobility and consequently conductivity [9].

The lower OCV and  $J_{sc}$  exhibited by the cell utilizing the chitosan-NH<sub>4</sub>I electrolyte are probably due to the smaller number of photoelectrons injected into the conduction band of the TiO<sub>2</sub>. In the absence of a catalyst coating such as Pt in the counter electrode leads to a slower rate of I<sub>3</sub><sup>-</sup> reduction to I<sup>-</sup>. This delays the photocurrent and photovoltage generation that accounts for the low  $J_{sc}$  and OCV [35]. According to Yen et al. [47] the I<sub>3</sub><sup>-</sup> + 2e<sup>-</sup> → 3I<sup>-</sup> reaction rate is extremely slow if the ITO counter electrode is not coated with catalytic materials.

From Table 3, conductivity for the electrolyte 43.5 wt.% P(EPI-EO) + 43.5 wt.% P(EGME) + 13 wt.% NaI [9] is nine times higher than that of the unplasticized electrolyte 89 wt.% P(EPI-EO) + 11 wt.% NaI; see Table 2. The employment of poly(ethylene glycol) methyl ether has resulted in more salt to dissociate resulting in a higher conductivity. The conductivity of the PAN + Pr<sub>4</sub>NI + EC + PC electrolyte [42] is greater than the conductivity of PEO + Pr<sub>4</sub>NI + EC [40] by almost 2 orders of magnitude. This could be attributed to the higher dielectric constant of PAN which is 6.27 [48] compared to that of PEO which is 5 [49] and to the presence of the PC plasticizer. With addition of plasticizer to the chitosan-NH<sub>4</sub>I electrolyte, the conductivity has increased to more than one order of magnitude.

The incorporation of ionic liquid to the electrolytes (Table 4) also improved performance of the PEC cell as shown in the work of Singh et al. [38]. The incorporation of 1-ethyl-3-methylimidazolium trifluoromethanesulfonate (EMImTFO) in the PEO-NaI electrolyte has increased the conductivity from  $2.02 \times 10^{-6}$  S cm<sup>-1</sup> to  $4.72 \times 10^{-5}$  S cm<sup>-1</sup>. An increase by a factor of ~4 in  $J_{sc}$  of DSSC was found. The PEC cell employing chitosan-based electrolyte also showed increment in conductivity and in  $J_{sc}$ . The anthocyanin pigment pH is also an important factor affecting  $J_{sc}$ . In this work, anthocyanin extracted from black rice produced a  $65 \mu\text{A cm}^{-2} J_{sc}$  and the cell employing the betalain pigment extracted from callus of *Celosia plumosa*

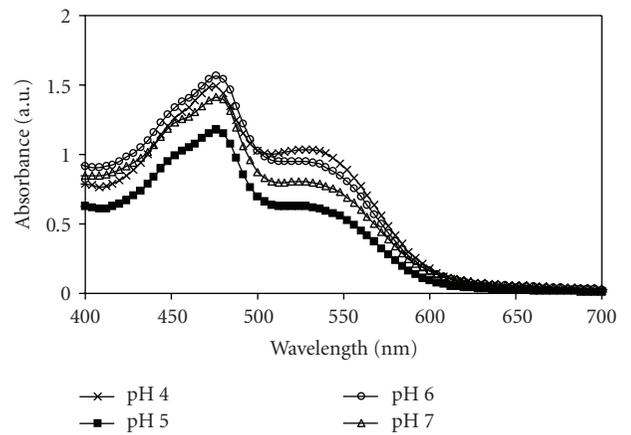


FIGURE 3: The absorption spectra of betalain at different pH.

[50, 51] produced  $29 \mu\text{A cm}^{-2} J_{sc}$ . The absorbance of the betalain pigment solution depends on its pH, as shown in Figure 3.

Rahman et al. [5] investigated the effect of electrolyte conductivity on OCV and  $J_{sc}$  of the ITO/TiO<sub>2</sub>/PVC-LiClO<sub>4</sub>/graphite solar cell. They have shown that both OCV and  $J_{sc}$  increase with electrolyte conductivity. From Table 2, the short-circuit current density  $J_{sc}$  for the PEC cell with PBA-NaI electrolyte is about 1.5 times larger than that of the solar cell employing PEO-NaI electrolyte. The PBA-NaI electrolyte also exhibits higher conductivity compared to PEO-NaI electrolyte. An almost similar situation can be observed in the work of Nogueira and coworkers [9, 39]. The solar cell utilizing the lower conducting electrolyte 71 wt.% P(EPI-EO)-29 wt.% NaI exhibits a short-circuit current density that is smaller than the solar cell utilizing the higher conducting electrolyte 89 wt.% P(EPI-EO)-11 wt.% NaI. This implies that higher conductivity of the electrolyte results in higher  $J_{sc}$  of the solar cell. However, the DSSCs employing PBA-NaI [37] and PEO-NaI [38] electrolytes with lower conductivity exhibit higher  $J_{sc}$  compared to the cells using P(EPI-PEO)-NaI electrolytes [9, 39]. The DSSCs utilizing PBA-NaI and

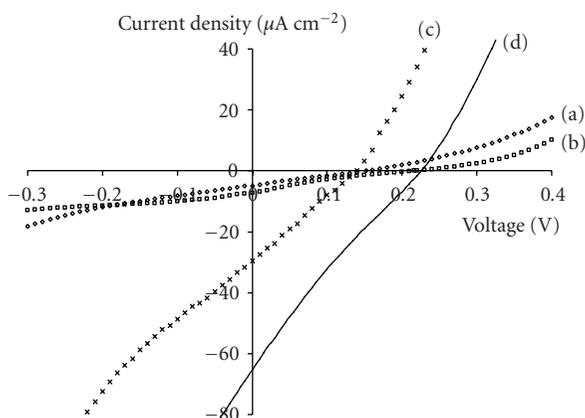


FIGURE 4: Current density versus voltage characteristics under illumination for (a) ITO/TiO<sub>2</sub>/55 wt.% chitosan+45 wt.% NH<sub>4</sub>I/ITO, (b) ITO/TiO<sub>2</sub>/33 wt.% chitosan+27 wt.% NH<sub>4</sub>I+40 wt.% EC/ITO, (c) ITO/TiO<sub>2</sub>/betalain dye (callus of *Celosia plumosa*)/27.5 wt.% chitosan+22.5 wt.% NH<sub>4</sub>I+50 wt.% BMII/ITO, and (d) ITO/TiO<sub>2</sub>/anthocyanin dye (black rice)/27.5 wt.% chitosan+22.5 wt.% NH<sub>4</sub>I+50 wt.% BMII/ITO.

PEO-NaI electrolyte have an extra layer coated on the ITO glass beneath the TiO<sub>2</sub> semiconducting photoelectrode layer. This underlayer consists of Ti(IV)bis(ethyl acetoacetato)-diisopropoxide coating. According to these authors, the Ti(IV)bis(ethyl acetoacetato)-diisopropoxide coating suppresses electron back recombination to the oxidized electrolyte. It is not clear from literature whether the DSSCs employing P(EPI-EO)-NaI electrolyte have this underlayer coating. If this is the case, it may be understood why the  $J_{sc}$  exhibited by these DSSCs is lower when the conductivity of the electrolyte is about one order of magnitude higher than the PBA-NaI and PEO-NaI electrolyte. The recombination is more evident in the absence of the underlayer leading to a depletion of the number of free electrons flowing through the external circuit resulting in a low  $J_{sc}$ . The low OCV exhibited by the cells using the chitosan electrolyte is probably due to the small difference in Fermi level of the TiO<sub>2</sub> and Fermi level of the redox couple. Figure 4 shows the plot of current density versus voltage characteristics for DSSC employing electrolytes of 55 wt.% chitosan+45 wt.% NH<sub>4</sub>I, 33 wt.% chitosan+27 wt.% NH<sub>4</sub>I+40 wt.% EC, and 27.5 wt.% chitosan+22.5 wt.% NH<sub>4</sub>I+50 wt.% BMII.

#### 4. Conclusions

Ethylene carbonate and ionic liquid enhance the conductivity of the electrolyte with chitosan as polymer host and NH<sub>4</sub>I as doping salt. Log  $\sigma$  versus T dependence follows the Arrhenius rule. Betalain pigment from calli of *Celosia plumosa* and anthocyanin pigment from black rice can serve as dye in solid-state solar cell applications. It was found that the dielectric constant of polymer and lattice energy of salt influence the conductivity. The conductivity in turn affects the short-circuit current density of the solar cells.

#### Acknowledgments

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