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Electrochemical Properties of Natural Sensitizer from *Garcinia mangostana* and *Archidendron pauciflorum* Pericarps for Dye-Sensitized Solar Cell (DSSC) Application

(Sifat Elektrokimia Pemekaan Semula Jadi daripada Perikarpa *Garcinia mangostana* dan *Archidendron pauciflorum* untuk Penggunaan Sel Suria Peka Pewarna (DSSC))

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ABSTRACT

Dye-sensitized solar cells (DSSC) create imitation photosynthesis by using chemical reactions to produce electricity from sunlight. DSSC has been pursued in numerous studies due to its capability to achieve efficiencies of up to 15% with artificial photosensitizer in diffuse light. However, artificial photosensitizers present a limitation because of the complex processing of metal compound. Therefore, various types of sensitizers were developed and synthesized to surpass the artificial sensitizer performances such as natural sensitizers from bio-based materials including plants, due to simple processing techniques and low environmental impact. Thus, this study examines the potential and properties of natural sensitizers from the waste of bio-based materials from Garcinia mangostana *(mangosteen fruit) and* Archidendron pauciflorum *(*jering *fruit). Both fruits pericarps have dark color pigments as dark purple and dark brown, respectively, which promise a good absorption and has potential to be used as sensitizer for DSSC. Each pericarps dye extracted using cold extraction method in methanol solvent. Electrochemical properties and photovoltaic properties of the natural photosensitizers were studied. The highest peaks of photoluminescence spectra of mangosteen and* jering *sensitizers were at 490 and 670 nm, respectively, due to their different types of dye pigment extracted. We also obtained the absorption spectra for both mangosteen and* jering *sensitizers at 380-500 and 400-600 nm, respectively, in blue shift behavior. The redox reaction was also studied using cyclic voltammetry and identify their energy levels. The DSSC device with mangosteen sensitizer achieved an efficiency of 0.38% with 35.43% (IPCE at 337 nm) and 37.75 Ω (Rs), whereas that with* jering *sensitizer has efficiency of 0.07% with 25.31% (IPCE at 337 nm) and 490.70 Ω (Rs). Performance studies for both photosensitizers were weak due to their HOMO-LUMO levels, but the results show that both natural dyes can be potentially applied as photosensitizer in DSSC.*

Keywords: Absorption; DSSC; efficiency; natural dye; photosensitizer

ABSTRAK

Sel suria peka pewarna (DSSC) membuat fotosintesis tiruan dengan menggunakan tindak balas kimia untuk menghasilkan elektrik daripada cahaya matahari. Peranti DSSC telah digunakan dalam banyak kajian kerana kemampuannya untuk mencapai kecekapan hingga 15% dengan pemeka warna buatan dalam cahaya yang resap. Walau bagaimanapun, pemeka warna buatan mempunyai batasan kerana mengandungi sebatian logam yang kompleks. Oleh itu, pelbagai jenis pemeka warna dikaji dan disintesis untuk mengatasi prestasi pemekaan buatan seperti pemeka semula jadi daripada bahan berasaskan bio termasuk tanaman, kerana teknik penghasilan pemeka yang mudah dan kesan persekitaran yang rendah. Oleh itu, kajian ini mengkaji potensi dan sifat pemekaan semula jadi daripada bahan buangan berasaskan bio daripada Garcinia mangostana *(buah manggis) dan* Archidendron pauciflorum *(buah jering). Kedua-dua buah masing-masing mempunyai kulit perikarpa yang berwarna gelap iaitu ungu gelap dan coklat gelap yang boleh membantu mendapatkan penyerapan cahaya yang baik dan berkebolehan dijadikan pemeka untuk peranti DSSC. Pewarna diekstrak menggunakan kaedah pengekstrakan sejuk di dalam larutan metanol. Sifat elektrokimia dan fotovoltaik pemeka warna semula jadi ini dianalisis dalam kajian ini. Puncak tertinggi spektrum fotoluminesen pemeka manggis dan jering adalah masing-masing pada 490 dan 670 nm, kerana pelbagai jenis pigmen pewarna yang diekstrak. Kami juga dapat memperoleh spektrum penyerapan untuk kedua-dua pemeka manggis dan jering pada masing-masing 380-500 dan 400-600 nm, dalam tingkah laku anjakan biru. Tindak balas redoks juga dikaji menggunakan* *voltametri berkitar dan mengenal pasti tahap tenaga pemeka tersebut. Peranti DSSC dengan pemeka warna manggis mencapai kecekapan peranti 0.38% dengan 35.43% (IPCE pada 337 nm) dan 37.75 Ω (Rs), sedangkan pemeka warna jering mempunyai kecekapan peranti 0.07% dengan 25.31% (IPCE at 337 nm) dan 490.70 Ω (Rs). Kajian prestasi peranti untuk kedua-dua pemeka warna tersebut lemah kerana paras HOMO-LUMO pewarna, tetapi hasil kajian menunjukkan bahawa kedua-dua pewarna semula jadi berpotensi digunakan sebagai pemeka cahaya dalam DSSC.*

Kata kunci: DSSC; kecekapan; pemeka warna buatan; penyerapan; pewarna semula jadi

Introduction

Solar energy is a renewable energy that had been pursued in decades either in commercial or research field. Thus, new generations of devices were created for good performances. The DSSC device is a third-generation photovoltaic device which consists of photoelectrode, counter electrode (CE), sensitizer and electrolyte, and each of these components plays an important role in device mechanisms (Zulkifili et al. 2015).

A sensitizer helps in harvesting sunlight and produces photo-excited electrons (Shahid et al. 2013). Several properties of sensitizer are needed to obtain an efficient DSSC with strong anchoring groups, such as carboxyl and hydroxyl, a potent absorption in the visible region of 400 to 700 nm and into the semiconductor surface and high extinction coefficient (Ye et al. 2014). Energy levels are crucial, and a sensitizer requires a competent electron injection with a more positive lowest unoccupied molecular orbital (LUMO) than the conduction band (CB) of the semiconductor and more negative highest occupied molecular orbital (HOMO) than the electrolyte potential (Safie et al. 2017a). O'regan and Grätzel (1991) successfully developed a DSSC with low cost and high efficiency of 12% in diffuse light by using colloidal TiO_2 films and newly developed ruthenium (Ru(II)) photosensitizers. Ru (II) sensitizers can exhibit high efficiency because of its wide absorption range below 550 nm in visible light range and metal component inside its chemical bonding. However, due to this condition, Ru (II) sensitizer exhibits high toxicity and entails high cost. Researchers prepared different types of sensitizers, including black dye, coumarin, and porphyrins. Other approaches use natural products as sensitizer.

Natural products can be obtained from trees, leaves, flowers, and fruits. Most researchers use natural products consisting of wastes from human usage, including dead tree barks, fruit seeds, fruit pericarps, and fallen leaves or flowers, thereby helping to reduce produced waste (Attanayake et al. 2013). Natural products were also selected due to their organic substances, readily available source and low cost. Natural products also consist of natural carboxyl and hydroxyl groups. Towards 2017, Sathyajothia et al. (2017) conducted the latest study on natural products by using henna and beetroot dye extracts as DSSC sensitizers and obtained efficiencies of 1.08 and 1.3%, respectively. *A. jiringa*, also known as dogfruit or *jering* in Malaysia, is widely used in Asia regions as herbal medicines to cure minor diseases, such as fever, diarrhoea, headaches, stomachache and coughs and is also utilised as traditional fitness supplements. In the use of *jering* as herbal medicine, only the inside seeds in pods and leaves are utilized, and the external pod, known as pericarp, is discarded due to the absence of nutrients in this part, used as dye for silk and timber wood crafting or burned as firewood (Bunawan et al. 2013). Other natural products that we used in this study included pericarps from *Garcinia mangostana*, which is locally known as mangosteen. Mangosteen can easily be found in the South Asian region. Locals eat the sweet edible part of the fruit and throw away its outside pericarps. However, other researchers also use mangosteen pericarp for studies on cancer and Alzheimer treatment (Jung et al. 2006).

In DSSC research field, mangosteen pericarps dye had been known to have high efficiency comparing with other natural dye where Maiaugree et al. (2015) study mangosteen pericarps in acetone solvent and obtained efficiency of 1.99% and Tontapha et al. (2017) studied the mangosteen pericarps in ethanol solvent obtained efficiency of 0.54%. Both studies were observed with surface area of 0.25 cm^2. However, *jering* pericarps has not been studied in DSSC research field. Therefore, in this study, the pericarps of both fruits are extracted with methanol solvent to obtain all target components for anchoring into TiO_2 film surfaces. In this study, we utilized two different types of waste from natural products for dye extractions and studied their characteristics to determine their compatibility for DSSC application.

Methods

Material

Pericarps of mangosteen and *jering*, 99.8% methanol as solvent, fluorine tin oxide (FTO) glass, titanium oxide paste (TiO_2), platinum (Pt) paste, and iodine electrolyte were used in this study.

NATURAL DYE PREPARATIONS

Jering and mangosteen pericarps were obtained from the waste residue of eaten fruits. All pericarps were dried inside a 45 °C oven for 48 h and then cut into small pieces for easy crushing into powder by using a blender. After being pulverised, pericarps of the mangosteen and *jering* were immersed inside the methanol solvent at a ratio of 1:10 for 24 h (Al-Alwani et al. 2017). This method is known as cold extraction. After 24 h, extracts were filtered with a filter paper to obtain dye extracts and to separate the powder residue. The extraction method was repeated twice at durations of 3 days to fully extract the pigment from the pericarp powder. After the extraction was completed, the extract was concentrated to suitable transparency for DSSC application using a rotary evaporator. Then, the extracts were stored inside a refrigerator until further use in DSSC fabrication.

FULL DSSC DEVICE FABRICATION

In this study, we used FTO glasses as substrate for both photoanode and CE at different resistivities of 15 and 8 Ω/sq, respectively. The substrate was cut using a diamond cutter into dimensions of 3 × 2 cm. The photoanode consists of two different layers of TiO_2 deposited by using doctor-blade method at the active area of 1 cm^2. The first layer helps absorb the light, followed by the second layer on top annealed to the first layer helping to fully scatter the light inside the layers. Both layers were annealed at a temperature of 450 °C for 30 min. The photoanode substrate was then immersed inside the selected dye extract for 24 h after heating at 100 °C in a furnace. Platinum was deposited into a substrate for CE by using the same method of doctor-blade method and annealed at the same settings of 450 °C for 30 min. Both dye-immersed photoanode and CE were sandwiched together with surlyn adhesion by using a heat press machine. Then, the electrolyte was inserted inside the sandwiched device and sealed with a polymer glue.

CHARACTERIZATIONS

Electrochemical properties of the sensitizer were characterised using Fourier transform infrared (FT-IR)–near-infrared (NIR) spectroscopy, Ultraviolet/Visible spectrometer (UV-vis), photoluminescence spectroscopy (PL) and cyclic voltammetry (CV). Chemical compound inside the sensitizer was studied using FT-IR spectra measured using Perkin Elmer Spectrum 400 FTIR/FT-NIR and Spotlight 400 imaging systems at wavenumber 4000 to 650 cm^{-1} (Kushwaha et al. 2013; Mariey et al. 2001). Absorption spectral range for selected sensitizer was studied using Perkin Elmer UV-vis Spectrometer Lambda 35. The absorption spectral wavelength was measured from 400 to 700 nm. PL was measured using FLS920 Edinburgh Instrument with excitation value of 400 nm. By using PL spectral graph, we obtained energy gap (E_g, eV) by using energy formula (1), where h is the Planck's constant (6.62607004 × 10^{-34} m^2 kg s–^{1}), c is the speed of light (3.00 × 10 m s^{-1}) and λ is the wavelength on the highest peak (Rajkumar & Suguna 2016).

$$Eg = hc / \lambda \tag{1}$$

CV was measured by using METROTHM AUTOLAB and NOVA software to study the oxidation and reduction occuring inside the sensitizer. CV measurements involved electrochemical cells comprising glossy carbon as working electrode, Ag/AgCl immersed in 3 M NaCl as reference electrode, platinum as CE, dye sensitizer consisting of 0.1 M lithium perchlorate and 99+% from Acros Organics acting as electrolyte. We calculated HOMO and LUMO from CV by using (2) and (3) and E_g from the PL spectra. E_{ox} onset at which oxidation occurred was obtained from the CV graph (Leonat et al. 2013).

$$E(HOMO) = -e[Eox_{onset} + 4.4] \tag{2}$$

$$E(LUMO) = E(HOMO) + Eg \tag{3}$$

$$\eta = \frac{Voc \times Jsc \times x\, FF}{Pin} \tag{4}$$

The performances of fully fabricated DSSC were then measured using current–voltage (*I–V*) characteristics, incident photon current efficiency (IPCE) and electrical impedance spectroscopy (EIS). *I–V* characteristics were measured using a Keithley 2400 source meter and solar simulator from San-El Electric, with the luminosity of solar light requiring 1000 W/m^2 measured with the Daystar meter. *I–V* characteristics facilitates the measurement of cell efficiency, which can also be calculated using (4). The formula consists of open circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (FF) and Pin = 100 mW/cm^2 (Park et al. 2003). IPCE describes the light-to-electricity conversion efficiency of a photosensitive device. This parameter is dependent on the absorption range of the dye. We used *Bentham* Photovoltaic Spectral Response PVE300 IPCE. IPCE can also be calculated using (5).

$$IPCE\% = \frac{I_{sc}}{P} \times \frac{1240}{\lambda} \times 100 \tag{5}$$

EIS measurement was used to study the electrocatalytic activity of the DSSC device by using Autolab Metrothm impedance module. V_{oc} from I–V measurement is required for measuring EIS. We fit and studied the device impedance using the graph. In fitting analysis, we used the equation R(RQ)(RC), where the first R represents the sheet resistance (R_s), followed by charge transfer resistance (R_{ct}) between

TiO_2 CB and FTO and Q as constant phase element. The last RC is for the CE side. We focused on R_s and R_{ct} at the dye and TiO_2 location (San Esteban & Enriquez 2013).

RESULTS AND DISCUSSION

ABSORPTION SPECTRA

An efficient sensitizer exhibits a broad wavelength especially within the visible light spectra (400 to 700 nm) to absorb additional light from sunlight radiation. Both *jering* and mangosteen exhibit different peaks of absorbance (Figure 1), which are due to different colours of pigment extract from each pericarp (Alias & Yaacob 2016). Both extracts show the highest peak at 400 nm, but *jering* exhibits a lower peak of 0.05 A compared with that of mangosteen of 0.20 A. Moreover, *jering* show an additional small peak at 664 nm. Both extracts exhibit blue shifts with decreased performance when reaching high wavelength which were affected by the solvent used and types of pigment inside the extract (Kumar et al. 2019).

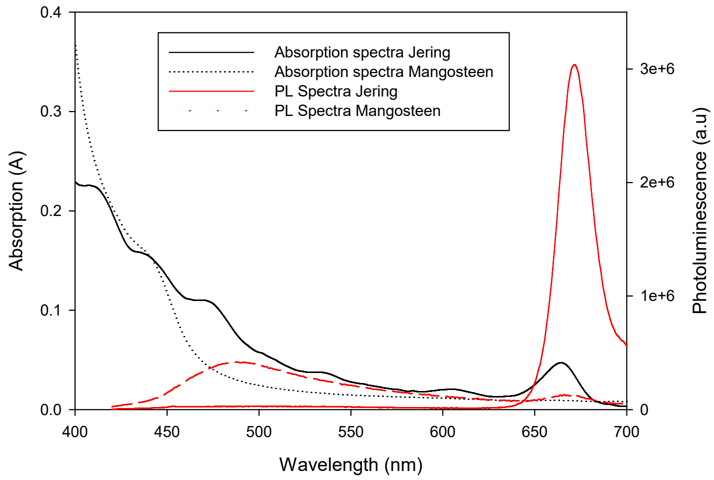

FIGURE 1. Absorption spectra vs photoluminescence spectra for *jering* and mangosteen dye extracts

PHOTOLUMINESCENCE (PL) SPECTRA

PL spectra were studied when substances in electronic states were excited by photon, releasing energy as light at different wavelengths. The spectra can be used to calculate energy gap (E_g) using (1) by obtaining the highest peak wavelength. Both sensitizers also exhibit different spectral behaviour as shown in Figure 1. The highest peaks for the mangosteen and *jering* sensitizers were at 487 and 671 nm, respectively. Equation (1) was used to obtain E_g for each sensitizer, and E_g values of 2.54

REDOX REACTION

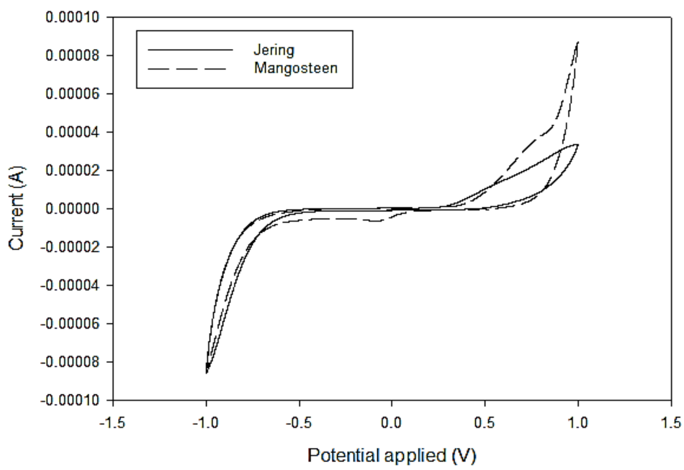

FIGURE 2. Cyclic voltammetry (CV) of *jering* and mangosteen extractions

and 1.85 eV were obtained for mangosteen and *jering* dyes, respectively. Different E_g values for both sensitizers were affected by the colour of the dye extracted from pericarp, that is, dark violet and transparent dark brown for mangosteen and *jering*, respectively. The small energy gap affected the device, leading to excited electrons returning to ground state levels and consequently affecting electron recombination (Table 1).

TABLE 1. Optical band gap, homo, and lumo calculations

Dye	λ (NM)	EG (EV)	E_{ox}^{onset} (V)	HOMO (eV)	LUMO (eV)
Jering	671	1.85	-0.671	-5.074	-3.232
Mangosteen	487	2.54	-0.41	-4.810	-2.270

Reduction and oxidation reaction of dye sensitizer can be studied in electrochemical cells in CV. HOMO and LUMO can be calculated by obtaining E_{ox} onset from the CV graph (Figure 2) by the inserted value and E_g from PL analysis into (2) and (3). The energy levels were tabulated in Table 2 and illustrated in Figure 3. LUMO should be more positive than the CB of TiO_2 (−4.0 eV to −4.3 eV), and HOMO should be more negative than the redox I_3^-/I^- redox potential (4.6–5.0 eV). Required differences of more than 0.2 eV between the LUMO level of sensitizer and CB of TiO_2 was observed by Ooyama et al. (2013). According to the result that we obtained from the CV graph, both sensitizers achieved the requirement, with the differences for *jering* and mangosteen being 1.0 and 1.9 eV, respectively. However, HOMO levels for both sensitizers are within the range of electrolyte redox potential by differences of 0.1 and 0.4 eV for *jering* and mangosteen, respectively. This condition affects DSSC performance by contributing to poor dye regeneration (Safie et al. 2017a).

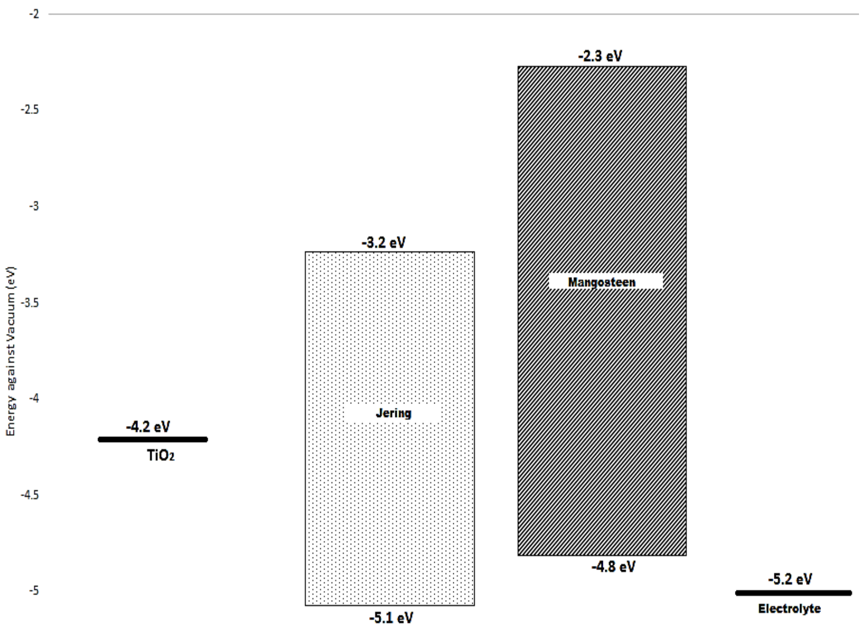

FIGURE 3. Energy level of *jering* and mangosteen dye sensitizer

CURRENT-VOLTAGE CHARACTERIZATION

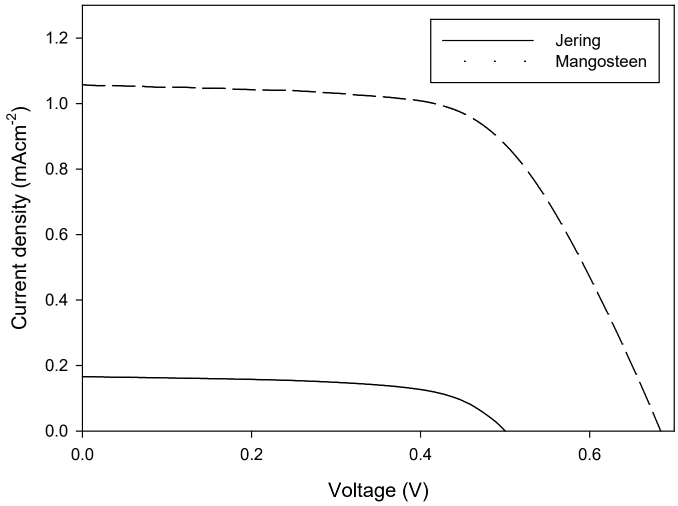

FIGURE 4. *I-V* characteristics of *jering* and mangosteen DSSC devices

Jering and mangosteen dye extracts were applied as sensitizer for the DSSC device, and we studied their photovoltaic properties from *I–V* characteristics in Figure 4 and Table 2. Figure 4 shows that the DSSC device with the mangosteen sensitizer exhibits superior performance to that with the *jering* sensitizer. This finding is demonstrated in Table 2, where mangosteen exhibits superior efficiencies of 0.38 and 0.074% to that of the *jering* sensitizer. Performance can be influenced by current density, which was measured for both DSSCs with *jering* and mangosteen as 0.20 and 1.03 mA/cm^2, respectively. This result shows that the mangosteen sensitizer presented a large amount of electron transfer from the sensitizer to the TiO_2 surface (Jinchu et al. 2016). The low efficiency obtained by *jering* dye compared to that of mangosteen dye is caused by the HOMO level of *jering* dye being relatively near to electrolyte redox potential and smaller band gap as seen in Figure 3 (Safie et al. 2017b).

TABLE 2. Photovoltaic parameters of the devices

Dye	J_{sc} (mA/cm^2)	V_{oc} (V)	*FF* (%)	η (%)
Jering	0.2	0.51	72.48	0.07
Mangosteen	1.03	0.64	57.32	0.38

IPCE

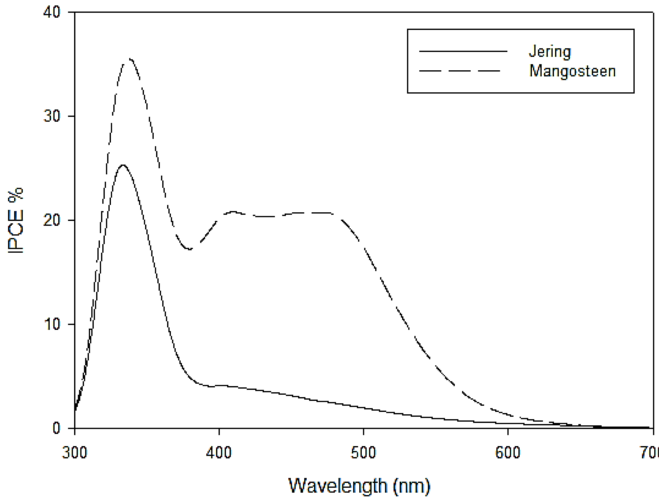

FIGURE 5. IPCE spectra for *jering* and mangosteen as DSSC sensitizer

Photon conversions to electron efficiency can be studied using IPCE from the spectral range of 300 to 700 nm. The IPCE measurements can be seen in Figure 5, which shows the highest peak for the DSSC devices with *jering* and mangosteen at 337 nm and efficiencies of 25.31 and 35.43%, respectively. These peaks represent TiO_2, to which the attached dye helps improve the efficiency of photon conversions (Godibo et al. 2014). The action spectra occurring from 400 to 700 nm represent the dye itself. The mangosteen sensitizer exhibited constant photon conversion from 406 nm to 480 nm and then displayed a blue shift that is characteristic of the absorption spectra in Figure 1. The DSSC device with *jering* dye as sensitizer did not show significant behaviour under visible light range. The dye colour improved device performance until the wavelength range light entered the devices.

IMPEDANCE ANALYSIS

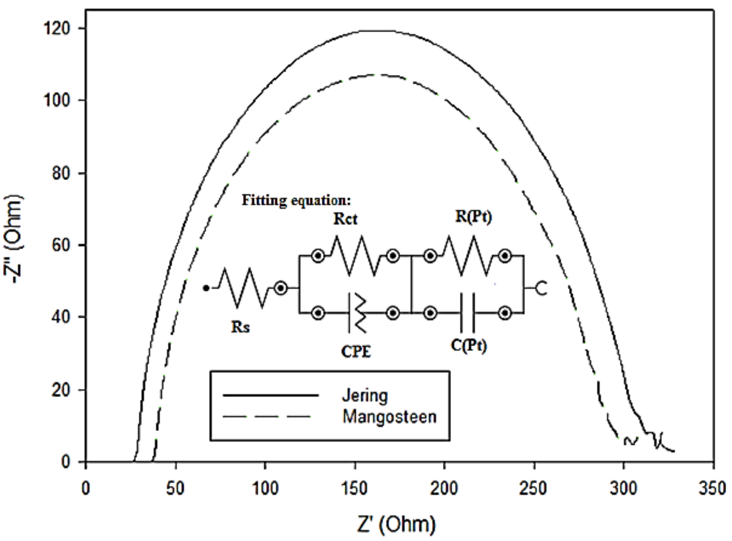

FIGURE 6. Nyquist curve and fitting equation for *jering* and mangosteen sensitizers for DSSC

Electrocatalytic reaction within DSSC device can be studied with EIS measurement, where impedance can be measured using a suitable fitting equation. The fitting equation used in this study was R(RQ)(RC) as shown in Figure 6, and the equation was simulated in NOVA software and fit in a circle with the EIS graph. From the fit and simulation, we were able to obtain DSSC R_S and R_{ct} as tabulated in Table 3. The DSSC device with mangosteen as sensitizer has smaller sheet resistance R_s at 37.75 Ω compared with the DSSC device with *jering* at 490.70 Ω. This finding showed that the mangosteen sensitizer exhibits more efficient electron transfer within the device compared with the *jering* dye sensitizer. This result was due to the increase in dark colour of the dye photon-electron conversion, therefore increasing electron transfer and decreasing resistance (Zhou et al. 2011).

TABLE 3. Current – voltage characteristics

Dye	χ^2	R_s (Ω)	R_{ct} (Ω)
Jering	0.02	490.70	282.68
Mangosteen	0.02	37.75	256.77

Conclusion

Garcinia mangostana and *Archidendron pauciflorum* can be easily found in South Asia, where the fruits are used for their nutrients as healthy or daily snacks. In this study, we used fruit pericarps, which was extracted with methanol solvents to obtain colour pigments and used as DSSC sensitizer. Each extract contains different colours pigment, affecting the UV–Vis spectra, PL and Eg. Mangosteen sensitizer has wider Eg of 2.54 eV compared with *jering* sensitizer of 1.85 eV. Mangosteen sensitizer also affects the HOMO and LUMO levels. Mangosteen sensitizer has significant electrochemical properties compared with *jering* sensitizer, therefore, influence the photovoltaic properties when the extracts were applied as DSSC sensitizer. When the photoanode layer was immersed in both sensitizers, namely, *jering* and mangosteen, mangosteen-DSSC-based sensitizer has darker colour than the *jering*-DSSC-based sensitizer. Therefore, mangosteen dye as sensitizer has an efficiency of 0.38%, and *jering* dye as sensitizer has 0.07%. Both sensitizers have a low efficiency compared with the artificial sensitizer. From this study, we were able to confirm that natural products especially mangosteen and *jering* have the capability to be used as DSSC sensitizer, but their performance can be improved in future works by added suitable catalyst as co-adsorbance or dye purification.

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