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FABRICATION OF SOLAR CELLS WITH TiO_2 NANOPARTICLES SENSITIZED USING NATURAL DYE EXTRACTED FROM MANGOSTEEN PERICARPS

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ABSTRACT

Faced with ever-shrinking reserves of fossil-based energy, in addition to the damaging impacts of the use of fossil-based energy sources, such as the greenhouse effect and global warming, efforts are needed to find energy alternatives. Currently under development as an alternative source of renewable energy, utilizing solar energy as its source, is a device incorporating the dye-sensitized solar cell (DSSC), which works using the simple photosynthetic-electrochemical principle at the molecular level. In this type of device, inorganic oxide semiconductors such as titanium dioxide (TiO_2) offer great potential for the absorption of photon energy from the solar energy source, especially in the form of a TiO_2 nanoparticle structure. In this study, a commercial TiO_2 nanoparticle was used. The as-received TiO_2 nanoparticle was characterized using X-ray diffraction (XRD) and a scanning electron microscope (SEM). For sensitizer, a natural dye extracted from mangosteen (*Garcinia mangostana L.*) pericarps was used. The extracted natural dye was characterized using Fourier transform infrared (FTIR) for the functional groups, whereas ultraviolet-visible (UV-Vis) was used to examine the absorption activity of the extracted natural dye. Performance of the DSSC was analyzed through a precision current versus potential difference (I-V) curve analyzer. The maximum power conversion efficiency (PCE) of the mangosteen natural dye was obtained using ethanol containing 20% distilled water as compared to commercial organic dye with a PCE of 4.02%. This result is convincing and promising for the next development.

Keywords: Anthocyanin; Dye-sensitized solar cell; Hydrothermal method; Mangosteen pericarp; TiO_2 nanoparticle

1. INTRODUCTION

One renewable energy alternative with enormous potential to be developed in Indonesia is solar cells. This is mainly because Indonesia has an abundant source of solar energy that is relatively constant throughout the year. As a country located in the equatorial region, Indonesia has potential for solar energy, with an estimated capacity of 4.800 kWh per square meter per day (Tharakan, 2015).

A solar cell, or photovoltaic cell, is an electronic device capable of converting light energy into electrical energy directly through the photovoltaic effect. A new type of solar cell known as the

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dye-sensitized solar cell (DSSC) has been developed using a simple electrochemical principle that mimics the effects of photosynthesis, i.e., by capturing photon energy at the molecular level and converting it into electrical energy (O'Regan & Grätzel, 1991). The system comprises a semiconductor oxide layer with a wide band gap semiconductor enclosed by a molecular layer of organic dyes and placed in contact with a redox electrolyte.

The performance of the DSSC in converting light energy into electrical energy is determined by the oxide used. An oxide semiconductor is commonly used in photoelectrochemical conversion for its stability against photocorrosion (Kalyanasundaram & Grätzel, 1998), and relatively low band gap energy (< 3.2 eV) is required for more photon energy absorption in the visible light spectrum (Dette et al., 2014). Apart from that, the ability of DSSC-based materials is also influenced by the structure of the oxide layer associated with the characteristic of the high surface area to absorb sensitizing dyes so that system performance can be maximized. In this case, the semiconductor layers most commonly used in DSSCs are metal oxide materials such as TiO_2, ZnO, CdSe, CdS, WO_3, Fe_2O_3, SnO_2, Nb_2O_5, and Ta_2O_5 (Wang et al., 2014; Sholehah et al., 2017).

Titanium dioxide (TiO_2), or titania, is one of the inorganic oxide metals included in the transition class known as transitional metal oxides (TMO) (Liu et al., 2013). This material has long been the subject of in-depth research and industry in the world because of its wide range of fascinating properties and technological applications such as photovoltaic cells, batteries, chemical sensors (Varghese et al., 2003), optical emission, photonic crystals, catalysts, photocatalysts (Livraghi et al., 2005), and purification of the environment (Ikezawa et al., 2001). TiO_2 exists in several crystal structures, namely anatase, brookite, and rutile; however, the most widely used form in DSSC is anatase TiO_2 (Al-Alwani et al., 2015). In addition, anatase $TiO_2$is also used in lithium batteries and electrochemical devices (Hagfeldt & Grätzel, 1995; Kavana et al., 2000; Grätzel, 2001).

The presence of the oxide semiconductor layer plays a very important role in the process of converting light energy into electrical energy. Further, the interaction between the dye and semiconductor oxide particles is instrumental in determining the process of converting light energy into electrical energy (Narayan, 2012). Meanwhile, it has been known that with the small size of the material down to the nanometer scale, the ratio of the surface area to volume (surface-area-to-volume ratio) will be greater, meaning that the possibility of surface interactions with the environment will be higher (Yuwono et al., 2012). It is expected that the use of TiO_2 nanomaterial in the form of nanoparticles, nanotubes, and/or mesoporous as semiconductor oxide layers in a DSSC will improve its system performance (O'Regan & Grätzel, 1991).

Dye sensitivity is an important factor in DSSCs since it acts as a photon absorber and serves to initiate the process of converting solar energy into electrical energy. There are many kinds of natural dye that can be used in DSSCs such as chlorophyll (Kumara et al., 2006), betalains (Zhang et al., 2008), and anthocyanin (Fernando & Senadeera, 2008). The latter has the most promising results in maximizing the efficiency of DSSC (Calogero et al., 2012).

The mangosteen is a plant indigenous to Indonesia (Morton, 1987) and has long been cultivated in many areas. The fruit of the plant is also called mangosteen and is a purplish-red color when ripe, although there are also variants with red skin. In general, only the white and sweet parts of the fruit are used, with the skin or pericarp typically discarded. Based on research carried out to date, it is known that the skin of the mangosteen contains anthocyanin, a flavonoid compound responsible for the purplish-red color of the skin of ripe fruit (Du & Francis, 1977; Chaovanalikit et al., 2012). Up to the present time, this mangosteen pericarp has not been known for use in sensitizing solar cell devices. Therefore, in this study, the pigment contained in mangosteen pericarps was used as a sensitizing dye in TiO_2 nanomaterial-based DSSC. The focus is on extraction of the mangosteen's natural dye using a variety of solvents and the subsequent use of this dye as asensitizer in a DSSC device fabricated with commercial TiO_2 nanoparticles.

2. EXPERIMENTAL SETUP

2.1. Dye Extraction from Mangosteen Pericarp

Fresh ripe mangosteens sourced from a local market were emptied by removing the sweet, white edible fleshy parts. The deepest white layer of skin was scraped and the pericarps were washed thoroughly under running water and allowed to stand, enabling the remaining water to evaporate. The mangosteen pericarps were then chopped in a blender and air-dried for 48 hours. The dried, chopped pericarps were then ground to obtain a very fine powder. The powder was sieved to obtain a fine, homogeneous powder.

Four Erlenmeyer flasks were prepared and each was filled with 10 grams of the fine mangosteen pericarp powder.Added to each Erlenmeyer flask was 100 mL of each of four different solvents, i.e., pure ethanol (Merck KGaA, Germany), ethanol containing 20% distilled water, ethanol containing 1% HCl, and ethanol containing 1% acetic acid. The mix was agitated using a magnetic stirrer at room temperature for 6 hours. After the stirring process, the supernatant was passed through a filter paper to separate the insoluble powder from the solvent. The filtered supernatant was then allowed to stand for a period of time in ambient conditions to evaporate the solvent and obtain a concentrated dye of about 10 mL. The steps of the dye extraction process are given schematically in Figure 1.

Figure 1 Natural dye extraction from mangosteen pericarps

The solution was then ready for characterization by using attenuated total reflectance Fourier transform infrared (ATR FTIR, PerkinElmer Spectrum 2) and ultraviolet-visible (UV-Vis, PerkinElmer Lambda 25) equipment. As a comparison, 2.5 mg commercial organic dye (Sensidizer RK1, Solaronix) dissolved in 100 mL ethanol was also examined.

2.2. DSSC Fabrication

The anode was prepared using commercial TiO_2 nano particles (Degussa P25). One gram of TiO_2 nanoparticle powder was added to 10 mL of ethanol and stirred. Two drops of TRITON X-100 (Sigma Aldrich) were added to the paste and stirred until the homogeneous phase was obtained.

Two fluorine-doped tin oxide (FTO, Solaronix) conductive glass substrates were prepared. One glass substrate was drilled to create two tiny perforations on one side and dry-cleaned using methanol. The other FTO glass was also dry-cleaned using methanol and attached to a flat layer. This substrate was tape-masked and coated with TiO_2 nanoparticle paste using a doctor-blade method under an area of about 1 cm^2. The paste was dried at 450°C for one hour and allowed to cool before soaking in the dye solution prepared from the extraction and air-dried. As a comparison, a commercial dye (Sensidizer RK1, Solaronix) was also used. The other perforated FTO glass substrate was coated in a platinum paste (Platisol, Solaronix) and dried at 450°C for one hour.

Both of the coated glass substrates were then attached to one another, separated by a spacer, and sealed to avoid electrolyte leakage. Once dried, the electrolyte (Iodolyte, Solaronix) was then injected through the two tiny holes drilled on one side of the substrate. The holes were then sealed and the cell was ready for characterization. Cell activity was measured using a simple ammeter, while cell performance was tested using a Semiconductor Parameter Analyzer (SPA, Agilent 4155A) with a standard illumination of 100 mW/cm^2.

3. RESULTS AND DISCUSSION

In order to be effectively adsorbed onto the TiO_2 layer, a dye needs to have specific functional groups. Fourier transforms infrared (FTIR) examination was carried out to observe the functional group characteristic presented in the mangosteen-extracted dye. The spectrum obtained at wave numbers 4000 to 400 cm^{-1} is given in Figure 2. As a comparison, the characteristic of pure commercial dye is also given.

As seen in Figure 2, all of the spectra show the appearance of hydrogen-bonded OH stretching bands ranging from 3250 to 3450 cm^{-1}, and the bands from around 2878 to 2973 represent stretching of the C–H groups (Pereira Jr et al., 2015). The band at 1640 cm^{-1} indicates C=O stretching vibration; it is small except for the natural dye extracted using ethanol containing 20% distilled water. The small peak at 1450 cm^{-1} belongs to C–N, the bands at 1423–1274 cm^{-1} belong to an aromatic compound, the band at 1045 belongs to C–O, and stretching vibration of the C–O–C esters is found at 1046 cm^{-1}. The aldehydes are found in the wave length between 880 cm^{-1} and 800 cm^{-1} due to the organic base natural dye. In this instance, the presence of the carboxyl and hydroxyl group in the dye would interact and strongly bind onto the TiO_2 surface (Al-Alwani et al., 2015). The interaction between the TiO_2 nanoparticle and functional groups of the dye would then drive the electron transfer from the dye molecules to the conduction band of the semiconductor TiO_2 (Narayan, 2012).

The absorption spectrum of the dye in the ultraviolet-visible region was examined using UV-Vis spectroscopy at a wave length from 400 to700 nm, and the results are given in Figure 3. As seen in Figure 3, the UV-Vis absorbance characteristics of the RK1 commercial dye show a specific absorbance peak at around 474 nm, whereas the mangosteen natural dye extracted using ethanol containing 1% HCl has a specific absorbance peak at around 534 nm. The other three spectra from the mangosteen natural dye extracted using ethanol, ethanol containing 1% acetic acid, and ethanol containing 1% HCl have broadened, with no specific absorption peak. The absorption peak indicates the presence of the dye that absorbs a large amount of photon energy at the specific wavelength from the visible light source (Fernando & Senadeera, 2008). It can also be noted from the spectra that the absorption from the mangosteen natural dye extracted using ethanol containing 20% distilled water has broadened the most, in excess of the broadening of the other spectra. This could be an indication that the dye has the widest absorption spectrum in visible light, a statement that is yet to be further confirmed.

The as-received commercial TiO_2 nanoparticle was characterized using X-ray diffraction (XRD) and a scanning electron microscope (SEM). The diffraction patterns from XRD and the secondary electron image from the SEM are shown in Figure 4. As seen in Figure 4a, the as-received material is confirmed to have a mixed anatase crystal structure (JCPDS file No.73-1764) and rutile crystal structure (JCPDS file No.78-1510). The three distinct diffraction peaks observed at the 2θ values of 25.31°, 37.79°, and 48.04° correspond to (101), (004), and (200) of the anatase crystal planes (Lekphet et al., 2017). The three distinct diffraction peaks for rutile (♦) are observed at 2θ27.5°, 36.1°, and 41.3°, corresponding to the (110), (101), and (111) planes, respectively.

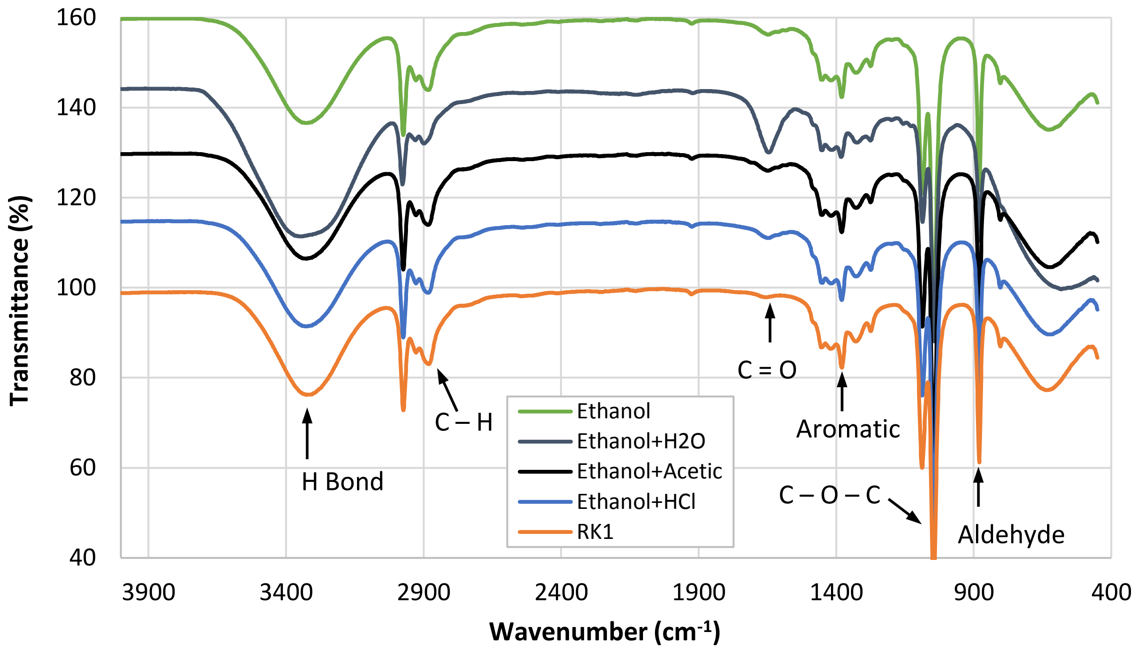

Figure 2 Fourier transform infrared transmittance characteristics of commercial dye and mangosteen dye extracted with various solvents at wave numbers 4000–400 cm^{-1}

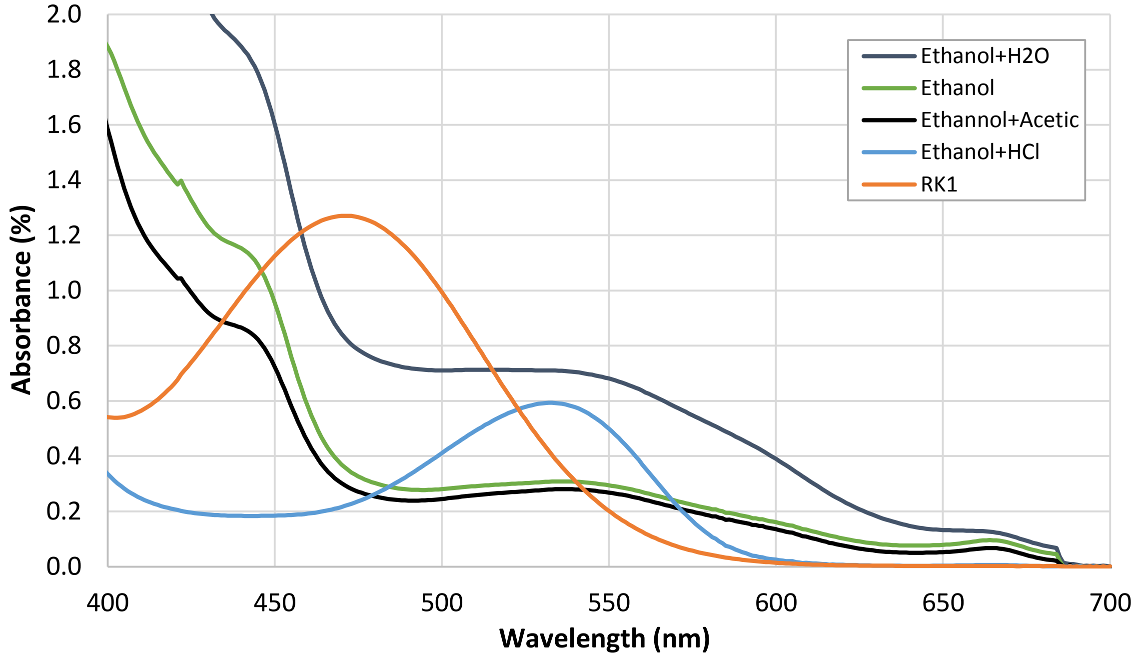

Figure 3 Ultraviolet-visible absorbance characteristics of commercial dye and mangosteen dye extracted with various solvents at a wave length of 400–700 nm

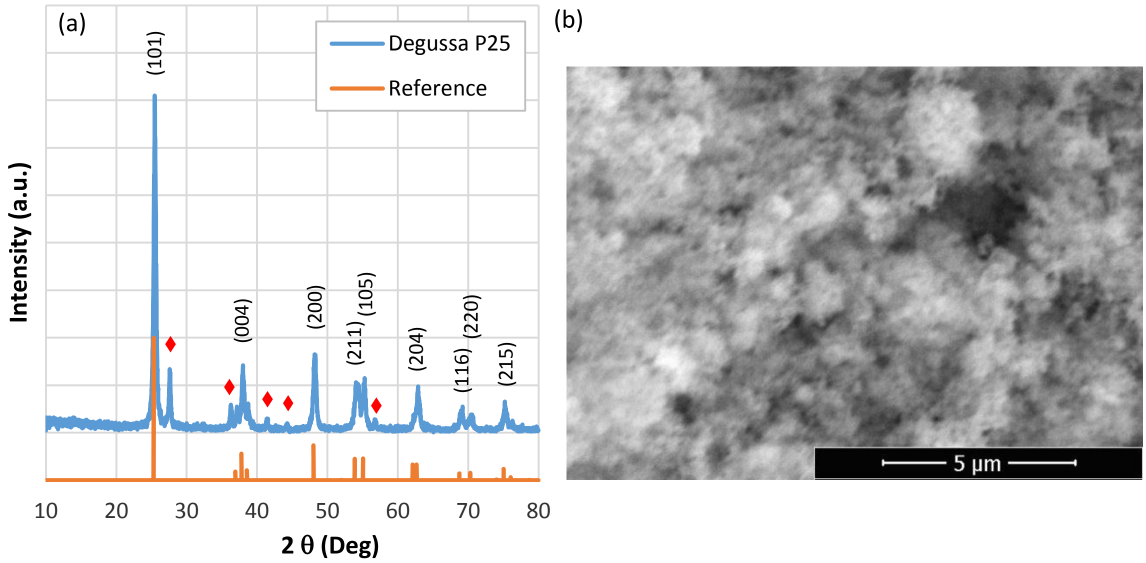

Figure 4 (a) X-Ray diffraction patterns of TiO_2 reference (JCPDS No. 73-1764) and commercial TiO_2 nanoparticle; and (b) a secondary electron image of the commercial TiO_2 nanoparticle morphology

The crystallite size of the TiO_2 nanoparticles was calculated in accordance with the Debye-Scherrer equation (Cullity, 1978):

$$c_s = \frac{k\lambda}{B \cos \theta} \quad (1)$$

where c_s is the crystallite size, λ is the wavelength of the X-ray radiation with Cu $K\alpha$ = 0.15406 nm, k is a constant taken as 0.94, θ is the diffraction angle, and B is the full width at half maximum (FWHM) peak in radians. All of the diffraction peaks were taken into account, from which the average crystallite size of 25 nm was obtained.

An SEM was used to study the surface morphological features of the as-received TiO_2 nanoparticle, and the result is shown in Figure 4b. As seen in Figure 4b, the morphology shows a homogeneous distribution of the nanoparticle. Image analysis revealed the average particle size to be < 80 nm.

Photo activity of the DSSC device sensitized using mangosteen natural dye extracted with various solvents was examined through a J-V curve characteristic, and the results are given in Figure 5. As seen in Figure 5, the photocurrent-voltage characteristic of the DSSC device sensitized using commercial organic dye and the mangosteen natural dye extracted using ethanol containing 20% distilled water have the highest values, with a current density of around 10 mA/cm^2. The other natural dyes extracted using pure ethanol, ethanol containing 1% acetic acid, and ethanol containing 1% HCl have a current density below 70 µA/cm^2.

Based on this J-V curve characteristic, the power conversion efficiency (PCE) was calculated using the following formula (Fernando & Senadeera, 2008):

$$\eta = \frac{FF \times J_{SC} \times V_{OC}}{I_{in}} \times 100 \quad (2)$$

where J_{sc} is the short-circuit photocurrent density (A, cm^{-2}), V_{oc} is the open-circuit voltage (volts), I_{in} is the intensity of the incident light (W, cm^{-2}), and FF is the fill factor defined as:

$$FF = \frac{i_{\max} V_{\max}}{i_{OC} V_{OC}} \quad (3)$$

where i_{oc} is the open-circuit current (mA), whereas $i_{\max}$ and $V_{\max}$ are the maximum photocurrent and voltage, respectively. The $i_{\max}$ and $V_{\max}$ values can be extracted from the maximum power of the I-V characteristics.

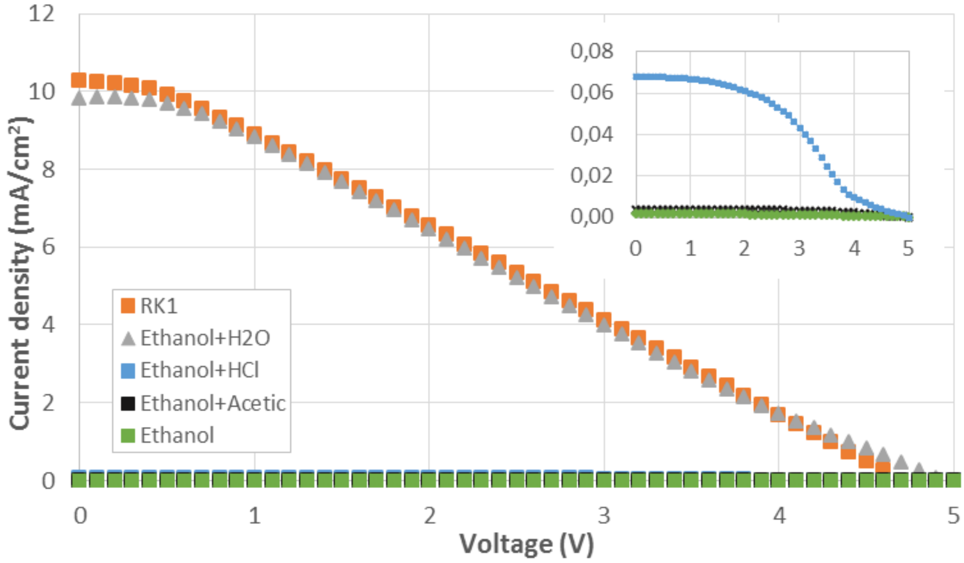

Figure 5 Photocurrent-voltage characteristics of the DSSC device sensitized using commercial dye and mangosteen natural dye extracted with various solvents. Inset is the photocurrent-voltage characteristic of the DSSC device sensitized using ethanol containing 1% HCl, ethanol containing 1% acetic acid, and pure ethanol

Based on the data obtained from the photocurrent-voltage examination of the DSSC device sensitized using mangosteen natural dye, the maximum PCE is found to be 3.91%, as given by the solvent of ethanol containing 20% distilled water. The PCEs from the other natural mangosteen dyes extracted using pure ethanol, ethanol containing 1% acetic acid, and ethanol containing 1% HCl were found to be quite low, as shown in detail in Table 1. At the same time, the PCE for the commercial organic dye was found to be 4.02%, the highest among the others. Compared to the result from the device that used sensitizer from anthocyanin extracted from red apple skin apple with an efficiency of 0.05% (Saputra et al., 2017), the current result is much more convincing and promising for the next development.

Table 1 Power conversion efficiency (PCE) of the DSSC device sensitized using commercial dye (RK1) and mangosteen natural dye extracted with various solvents

	Ethanol	Ethanol+H_2O	Ethanol+Acetic	Ethanol+HCl	RK1
V_{oc} (Volt)	5.000	5.000	5.000	5.000	4.800
I_{oc} (mA)	0.002	9.828	0.004	0.068	10.280
V_{max} (Volt)	0.500	0.400	0.900	1.000	0.400
I_{max} (mA)	0.002	9.784	0.004	0.067	10.060
FF	0.098	0.080	0.176	0.196	0.082
PCE (%)	0.001	3.914	0.003	0.067	4.024

4. CONCLUSION

The extraction of natural dye from mangosteen pericarps has been successfully carried out using various solvents. The extracted dyes have also been successfully applied as a sensitizer for DSSC fabricated on a commercial TiO_2 nanoparticle anode. In this work, the best solvent for extracting natural dye from mangosteen pericarps for use with a DSSC device is found to be ethanol containing 20% distilled water, with a PCE of 3.91%. The dyes extracted using other solvents are found to have low PCEs; however, the stability of the dyes in the DSSC device are yet to be further confirmed.

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