Outdoor Photoelectrochemical Characterization of Dyes from *Acalypha wilkesiana 'Haleakala'* and *Hibiscus sabdariffa* as Dye Solar Cells Sensitizers

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Authors' contributions

This work was carried out in collaboration between both authors. Author BCM designed the study, undertook the experimental work, performed the statistical analysis, wrote the protocol, wrote the first draft of the manuscript and managed literature searches. Authors BCM and AP. managed the analyses of the study and literature searches. Both authors read and approved the final manuscript.

Original Research Article

ABSTRACT

An outdoor study on the photoelectrochemical performance of dye-sensitized solar cells (DSSCs) with extracts of leaves of *Acalypha wilkesiana 'Haleakala'* and flowers of *Hibiscus sabdariffa* as sensitizers was undertaken in which the latter was used as a bench mark. Three extraction methods of natural dyes, namely; aqueous, cold ethanol, and Soxhlet hot ethanol, were employed to investigate their impact on the performance of the dye sensitized solar cells. Aqueous and Soxhlet hot ethanol extracts performed the best for *Hibiscus sabdariffa* and *Acalypha wilkesiana 'Haleakala'* respectively. *Hibiscus sabdariffa* yielded the averaged photoelectrochemical performance of light-to-current conversion efficiency (η) of 0.07%, open circuit voltage (V_{oc}) of 0.44 V, short circuit current density (J_{sc}) of 0.23 mA/cm^2; and *Acalypha wilkesiana 'Haleakala'* yielded 0.06% efficiency, V_{oc} of 0.40 V, J_{sc} of 0.26 mA/cm^2. The study for *Acalypha wilkesiana 'Haleakala'* further included varying dye concentrations by dilution. It was found out that the dilution factor D_f of 10 (ratio of 1:9) was optimum at which the best efficiency was achieved.

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1. INTRODUCTION

Renewable and sustainable energy is the only alternative to opt for in these modern days after fossil fuels. Fossil fuels have been the most reliable source of energy for the last decades, but there is an ever growing fear that in the near future these will be scarce commodities. Besides, fossil fuels have had drawbacks that include air pollution, environmental degradation, greenhouse effect, are unreprenishable resource and unsustainable. It is for this reason that intensive studies are being undertaken in renewable energy sources.

Amongst the renewable energy sources is the solar energy. Solar cells are devices that harvest the solar energy and convert it into electric energy. In addition to other solar cells, Dye Sensitized Solar Cells (DSSC) – the third generation solar cells – have attracted much attention in the scientific research. The DSSCs use the wide band gap titanium oxide (TiO_2) and the dye with the aid of an electrolyte to generate electric energy from the sun, the technology that was pioneered by O'Reagan and Grätzel in 1991 [1].

There are of two types of dyes which are synthetic and natural organic dyes (NODs). Synthetic dyes are metal complex dyes that have had better performance of up to 12% [2-4] of light-to-current conversion efficiency (LCCE). These dyes, however, are not favoured because they are expensive to produce, toxic, manufactured from rare materials, and are not biodegradable. On the other hand, NODs have attracted even much more attention because they are also able to absorb solar energy though their LCCE is very low relative to synthetic dyes of which most of them are less than 1% [5-7]. Nevertheless, the interest in these dyes is ever growing because they are user friendly, biodegradable, ecofriendly; the resources are abundantly available and renewable. NODs are plant extracts composed of polyphenol compounds such as anthocyanins that are responsible for colours; red, orange, purple, for instance [8, 9], as well as porphyrin (chlorophyll dyes) that are responsible for green colours, besides other plant constituents. The light absorbance is colour dependent. Anthocyanins, for example, are able to absorb light within the visible and NIR range (400 – 700 nm) whose

maximum absorbance is reported usually to be about 520 nm [10] but this differs depending on the type of anthocyanin [11]. Thus, one function of the natural dye is to enhance absorption of light energy (*hv*) within the visible and NIR regions [12].

In this study, a particular interest was drawn to *Acalypha wilkesiana* plant. It is also known as Fire Dragon, Hoja de Cobre, Beefsteak Plant, Hu-ling, Red leaf, Joseph's coat, Copper leaf, etc. There are different species, but in this article *Acalypha wilkesiana 'Haleakala'* (*AWH*) has been used for observations. Generally, it is an evergreen plant with leaves of red coppery appearance, 1.5 to 1.8 m high and grows shrublike. It is found in many parts of Africa, for example, Tanzania, Kenya, Malawi, Uganda, Nigeria, etc.; and also in other parts of the world. The reddish colour of leaves of this plant indicates the presence of anthocyanin, a phenolic compound that is very useful in DSSC applications, and in our previous study [13] it was observed that anthocyanin – among other plant constituents – was also present in the dye that was extracted from these leaves. *Hibiscus sabdariffa* (rosella) is an edible plant which produces pretty red flowers rich in anthocyanin [13-15], and has been used in various studies for DSSC [11, 16-19] as sensitizers. With the known photoelectrochemical performance, it has been used in this study as a bench mark for *AWH*. The main objective of this study, therefore, was to investigate the photoelectrochemical performance of *AWH* dye as a sensitizer for the dye sensitized solar cell, and also to establish the effect of the three methods of extracting dye from plants on electrical performance of the cell.

2. THE ROLE OF DYE IN DSSC AND PRINCIPLE OF OPERATION OF DSSC

The working principle and the construction of the DSSCs are as illustrated in Fig. 1. The electrode and the counter-electrode are transparent glass substrates with thin fluorine-doped tin conductive oxide (FTO) layer. A thin layer of a wide band gap (~3.2 eV) nanoporous titanium dioxide (TiO_2) is pasted onto the electrode substrate. Due to a wide band gap, the semiconductor TiO_2 needs higher energy (>3.2 eV) – which is within near-ultraviolet (NUV) region and beyond – to excite electrons from the valence band to the conduction band in order to generate meaningful

electricity. The dye is therefore adsorbed onto the semiconductor to help tap lower energies in the visible and near infrared (NIR) regions between 3.1 eV and 1.4 eV [4, 12].

When the incident light from the sun strikes the adsorbed dye on the semiconductor, electrons in the highest occupied molecular orbital (HOMO) of the dye are excited, and then transfer into the lowest unoccupied molecular orbital (LUMO) of the dye. The electron is thereafter injected into the lower energy level of TiO_2 conduction band (CB). This is possible only if the $LUMO_{dye}$ is slightly higher than the $LUMO_{semiconductor}$ [20-22]. Through the thin film conductor, the electrons are conducted to the external circuit, and conducted back to the dye to regain the initial position through the redox electrolyte via the counter electrode (Fig. 1).

Before being energized, the dye is at its ground state (S^o). When excited S*, it loses an electron which it injects to the CB of the semiconductor, and thus becomes oxidized (S+). The electron is transported to the external circuit and back to the cell at the counter-electrode that is regained by the dye through the redox reaction (O/R) of the liquid electrolyte (iodide/triiodide is the generally used electrolyte) which is an electron donor, restoring it back to the ground state of the dye. For as long as there is an exciting energy, this process is continuous. Thus dye plays a very important role not only as a light absorber, but also as an electron generator.

3. MATERIALS AND METHODS

3.1 Preparation of the Natural Dye

The leaves of *AWH* and flowers of *Hibiscus* were air dried till they became invariant in weight. The leaves of *AWH* were ground to small particles by the lab blender (WARING COMMERCIAL, Torrington Connecticut – USA), but the dried flowers of *Hibiscus* were left uncrushed because previous attempts proved failure to extract the dye from crushed samples due to jellification. Three different methods of extraction were used; cold ethanol (*CE*), Soxhlet hot ethanol (*SHE*), and heating in water (*Aq.*). Distilled water was the solvent for aqueous extraction, and ethanol 96% v/v (AVONCHEM Ltd from Wellington House, Macclesfield Cheshire SKII 6PJ) for *CE* and *SHE* extraction methods. For *Aq.* extraction method, each sample was heated at six different temperatures (40°C – 90°C with 10°C step) for 30–50 min after which they were filtered using Whatman No. 41 filter to get rid of solid particles. The optimum extraction temperature of each sample was determined based on the optical absorbance at uniform acidity (pH 3). For each method, 12.5 g of the sample was measured using the analytical scale (OHAUS Corporation – USA) for every 125 ml of the solvent. The optical absorbance of each sample was measured using the SQ2800 Single Beam Spectrophotometer.
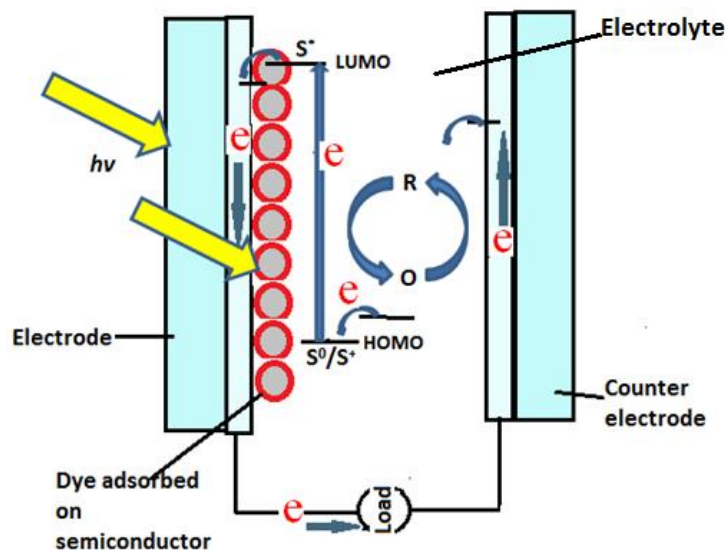

Fig. 1. An illustration of the DSSC principle of operation

3.2 Preparation of Glass Substrates

A 30 cm × 30 cm × 2 mm FTO glass plate (TCO22-7, 15 Ω/sq. Solaronix - Switzerland) was cut into 3 × 3 cm glass substrates using a diamond glass cutter. Cutting was done on the conductive side which was identified by using a multimeter. Another way of identifying the conductive side is to wipe gently on the glass substrate planes with a clean tissue. A 'screeching' sound is heard from a conductive side and not on the other. After cutting, the glass substrates were washed clean with the detergent solution and water, and rinsed twice in distilled water. The substrates were then stored in ethanol till required for use.

3.3 Preparation of Electrodes

The substrates with ethanol were air-dried, and heated on a hot plate with conductive side up at 60°C for 15 min to evaporate the ethanol completely. After cooling, the masking tape was used to make the window (2 cm × 2 cm) for TiO_2 paste on the electrode on the conductive side of the electrode. The ready-to-use TiO_2 paste (Ti-Nanoxide HT, Solaronix– Switzerland) was applied using the doctor blading method. After 10 – 15 min, the masking tape was gently removed. Using the hot plate, the electrode was preheated at 60°C for 15 min, and sintered at 450°C for 30 min. After sintering, the temperature was gradually lowered to 60°C for 5 – 10 min, and finally to room temperature to cool. This was done to avoid cracking of the substrates. Carbon coating was done using the HB lead pencil that was applied on the conductive side of the counter-electrode.

3.4 Assembly of a DSSC and Photoelectrochemical Measurements

The conductive sides of the electrode and the counter-electrode were placed juxtaposed and clamped together by the paper binder clips. The iodide triiodide (I^-/I_3^-) electrolyte (Iodolyte HI-30, Solaronix – Switzerland) was applied which percolated in between the plates by capillary action. The electrical circuit was composed of the cell, resistors, ammeter, and voltmeter (multimeters).

The photoelectrochemical performance was measured using multimeters and a set of resistance boxes with the total resistance of 5

kΩ. Testing was done outdoors using solar illumination from the sun, and the irradiance, *G* was measured using the lux meter. To ensure that maximum instantaneous irradiance from the sun was received, the cell and light sensor were mounted on an adjustable panel (Fig. 2) which was easy to orient in the direction of the sun.

The fill factor *FF* and efficiency η were calculated using the formulae (1) and (2).

$$FF = \frac{J_{mp}V_{mp}}{J_{SC}V_{OC}} \tag{1}$$

$$\eta = \frac{FFJ_{SC}V_{OC}}{P_{in}} = \frac{J_{mp}V_{mp}}{P_{in}} \tag{2}$$

Where J_{mp} and J_{sc} are the maximum power point and short circuit current densities respectively in mA/cm^2; V_{mp} and V_{oc} are the maximum power point and open circuit voltages respectively.

## 4. RESULTS AND DISCUSSION

4.1 Dye Extracts

For each plant specimen, three types of extracts were obtained in the procedure as described above. For *Aq.* extraction method, the optimum extraction temperatures for the two types of extracts were 70°C for *AWH* and 50°C for *Hibiscus* and these were based on optical absorbance of the dyes. The result for *Hibiscus* agrees with other findings in similar studies [11, 18] that were based on photoelectrochemical performance.

4.2 Optical Absorbance

Fig. 3 shows the trend of the spectral optical absorbance of *AWH* and *Hibiscus* dyes that have been extracted by *Aq.*, *CE*, and *SHE* extraction methods. It was observed that for both of the plant species, the *CE* and *SHE* extracts have the similar spectra, and that the latter absorbs slightly higher in the visible range. For water extracts, the absorbance is lower than both ethanol extracts for *AWH* (Fig. 3 (a)), and vice versa for *Hibiscus* (Fig. 3 (b)) in the visible range of the electromagnetic spectrum. The trend for *Hibiscus* agrees with the results obtained by Mulati et al. [18].
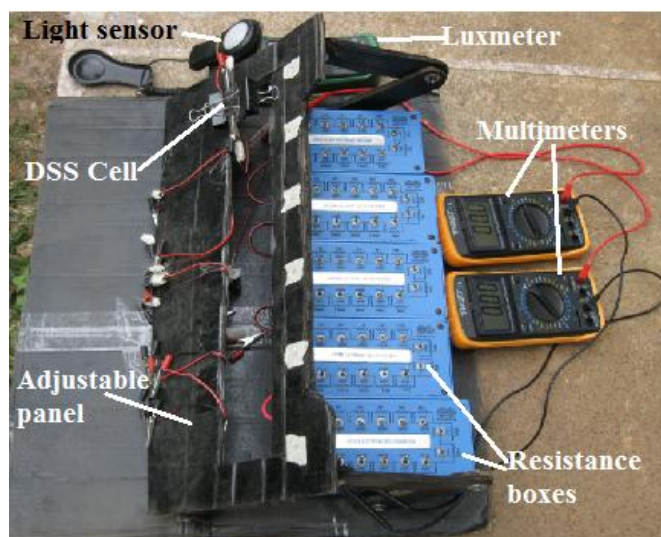
Fig. 2. The DSSC photoelectrochemical performance testing system showing the top view of the system. The panel can hold up to six cells
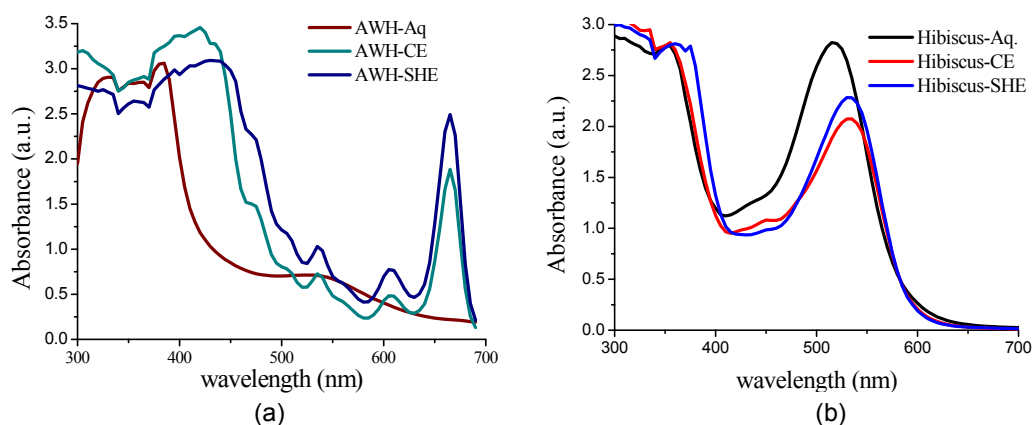
Fig. 3. Optical absorbance spectra of natural dye extracts of (a) *AWH* (b) *Hibiscus* that were extracted using *Aq.* extraction (heating in water), *SHE* and *CE* extraction methods

In the previous study [13] through the phytochemical screening, we found that *AWH* leaves contain quinones, anthocyanins, chlorophyll and other phyto-constituents, and that Hibiscus also has anthocyanins, quinones, carotenoids, etc. For *AWH*, the absorption properties are greatly influenced by chlorophyll whose peaks outstand at wavelengths of 420 and 663 nm while anthocyanin is the main component for Hibiscus whose peaks occur at wavelengths between 500 – 550 nm.

4.3 Photoelectrochemical Performance

The *J-V* photoelectrochemical characteristic curves of the DSSCs fabricated with sensitizers from the six extracts have been shown in Figs. 4

(a) and (b), and Table 1 summarizes the *J-V* parameters as per curves. Since the experiments were conducted outdoors, the test conditions were subject to atmospheric condition variations, besides the systematic errors. In the data presentation, the limits of uncertainties were also considered.

From the effective absorption area of 4.0 cm^2 of the DSSCs, the averaged values of the light-to-current conversion efficiencies (η) of sensitizers, J_{sc}, J_{mp}, V_{oc}, V_{mp}, and *FF* were recorded as presented in Table 1.
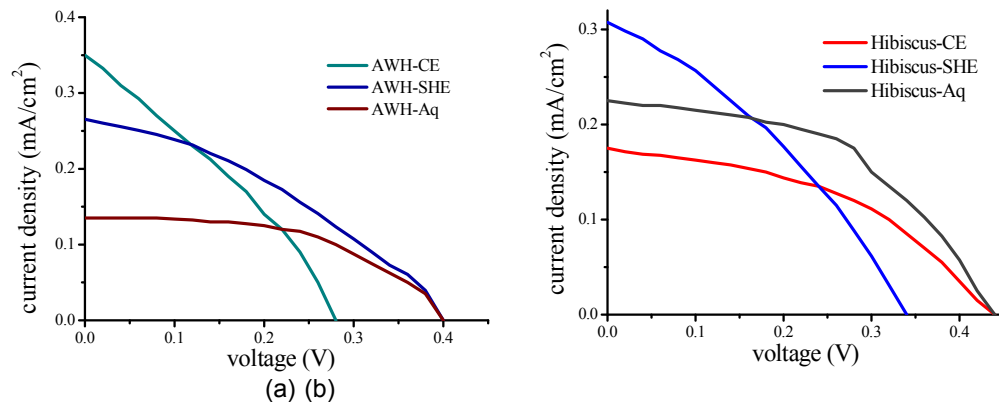
Fig. 4. Photoelectrochemical characteristic *J–V* curves for dye extracts of (a) *AWH* and (b) *Hibiscus*

Table 1. Performance results of the DSSC fabricated using the two dyes extracted by three different extraction methods at pH 3 and D_f =10; irradiance G = 67±4 mW/cm^2

Sample	Extraction method	J_{sc} (mA/cm^2)	V_{oc} (*V*)	J_{mp} (mA/cm^2)	V_{max} (*V*)	*FF*	η (%)
AWH	*CE*	0.35	0.28	0.16	0.18	0.49	0.04±0.01
	SHE	0.26	0.40	0.17	0.24	0.39	0.06±0.02
	Aq	0.14	0.42	0.10	0.28	0.49	0.04±0.01
Hibiscus	*CE*	0.17	0.44	0.12	0.28	0.46	0.05±0.01
	SHE	0.30	0.35	0.17	0.20	0.32	0.05±0.01
	Aq	0.23	0.44	0.17	0.28	0.49	0.07±0.01

It was observed that *AWH-SHE* dye extracts produced better light-to-current conversion efficiency than those of *CE* and *Aq*. of the *AWH* group. Also the dye extract of *Hibiscus-Aq*. shows the highest absorbance of light (Fig. 3 (b)); consequently the conversion efficiency is found to be the best for this sample, and for all (Table 1). The light-to-current conversion efficiencies (Fig. 4) of the DSSCs dyes are relatively consistent with the optical absorption spectra (Fig. 3). Since the *AWH-SHE* dye showed better photoelectrochemical performance than those of the *AWH-CE* and *AWH-Aq*., it was considered for another study on optimization of the level of dilution of the dye for the better performance of the DSSC.

The limit of diluting the dye to achieve the optimum level of performance of the cell for *AWH-SHE* was investigated. The dyes were prepared in five different concentrations by using five dilution factors (D_f) of 1, 5, 10, 15, and 20 where 1 and 20 were the undiluted and the least concentrated dyes respectively, but maintained a pH of 3.

Fig. 5 shows a graph of results of the photoelectrochemical performance of DSSCs

sensitized with *AWH-SHE* in five different concentrations. It was found that a D_f of 10 (i.e. a ratio of 1 part of dye to 9 parts of solvent) was the optimum dilution factor which gave the best performance in efficiency though the difference between D_f 5 and D_f 10 is quite slight. The explanation to this could be that the concentration of phytochemicals (chlorophyll specifically) at D_f greater than 10 was too low for effective absorption of light as concentration reduces with increased dilution; and at D_f less than 10, the concentration was too high that some constituents could not even chelate with the TiO_2 resulting in smearing on top of the chelated constituents. This may have reduced the effective light absorption, and hence reduce the number of electrons to be injected into the conduction band of TiO_2. The dilution factor 10 was also first obtained during the optical absorbance tests experimentally.

The obtained results were compared with other results in other studies that have been shown in Table 2 based on the light source, *J-V* photoelectrochemical parameters, efficiencies and the fill factors for *Hibiscus* as the benchmark, and consequently conclusions were drawn for the performance of *AWH*.

Table 2. Reported photoelectrochemical performance results for different DSSCs of *Hibiscus* dyes

Sample	Extraction solvent	Light source	G (mW/cm^2)	J_{sc} (mA/cm^2)	V_{oc} (V)	J_{mp} (mA/cm^2)	V_{max} (V)	FF	η (%)	Ref.
Hibiscus (rosella)	CE	Simulated	100	2.51	0.488	N/A	N/A	0.58	0.70	[11]
	Aq.	sunlight (AM 1.5)		2.06	0.433	N/A	N/A	0.59	0.52	
	CE	Simulated sunlight (AM 1.5)	100	2.60	0.330	N/A	N/A	0.68	0.58	[19]
	Aq.	Halogen lamp	N/A	0.082*	0.349	0.056*	0.311	0.61	0.43	[18]
	Aq.	Simulated sunlight (AM 1.5)	100	0.17	0.460	N/A	N/A	0.41	0.033	[16]
	Methanol	Sunlight	N/A	4.50	0.0124	N/A	N/A	0.74	0.002	[17]
Azardirachta Indica calyxes	Methanol	Sunlight	N/A	2.50	0.0118	N/A	N/A	0.40	0.00017	

* recalculated from µA to mA/cm^2 based on the reported figures (I_{sc} = 344.4 µA, and I_{mp} = 235.2 µA) and the instruction of the source [18] for preparation of the cell to get area for the current densities
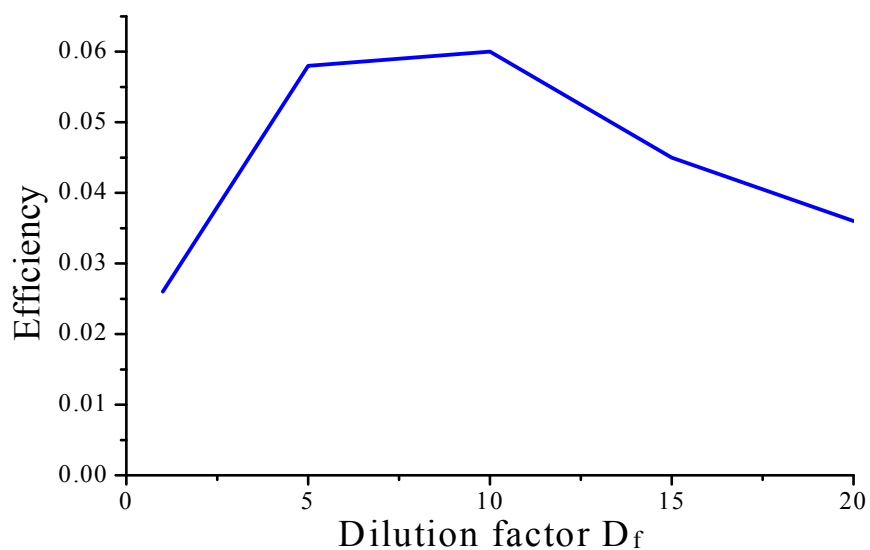
Fig. 5. Variation of efficiencies with dilution factor at pH 3 for *AWH-SHE*

When the light-to-current performance test results [11,19] of Hibiscus as indicated in Table 2 at standard conditions are compared to the results obtained in this study, they are about 10 times as high. This is suggested to be due to the difference in power of the incident light source. The irradiance in this study was about 67 mW/cm^2 on average as compared to 100 mW/cm^2. However, the result that was obtained by Almed et al. [16] as indicated in Table 2 was even lower than ours though tested at standard conditions (i.e. simulated sunlight, air mass of 1.5). The halogen-illuminated cell by Mulati et al. [18] produced higher efficiency but generated the lowest current densities.

On the other hand, when the efficiency results in this study are compared to the other study that was performed under similar conditions (outdoor conditions) by Boyo et al. [17] who found 0.002% and 0.00017% for Hibiscus and Azardirachta indica calyxes respectively, our results are much higher. This may be due to differences in concentrations of phytoconstituents in different parts of the plants. However, the results from this study correspond with the results found in many other similar studies [5-7, 23] whose conversion efficiencies are less than 1.0%.

The meteorological factors may also have effects on the cell performance. In the outdoor performance study of the dye sensitized solar cells by Cornaro et al. [24], it was found that meteorological factors such as wind and temperature have an impact on the performance

of the DSSC. It was established, in their findings, that there was a positive correlation between the cell temperature and the light-to-current conversion efficiency (LCCE) i.e. as the cell temperature rises the LCCE increases. The cell temperature is also regulated by the level of irradiance (which is influenced by the cloud cover) and wind speed. This phenomenon is further emphasized by Peng [25] who described the effect of irradiance and temperature on the DSSC, and Raga and Fabregat-Santiago [26] who also studied and explained the relationship between the DSSC (N719 dye) performance and temperature. The meteorological factors may have played a role in our results, besides other factors like differences in FTO glass substrate specifications, electrode preparations, and dye treatments.

5. CONCLUSION

In this study, we have reported the influence of extraction methods of NODs on LCCE for which the flowers of *Hibiscus* and leaves of *AWH* were the plant samples. It was observed that no predictable trend was established on the effect of the extraction methods on photoelectrochemical performance of the cell. *SHE* and *CE* for *Hibiscus* gave the same efficiencies (0.050%), but *Aq*. was the best with an LCCE of 0.070%. For *AWH*, *SHE* was the best with an LCCE of 0.060% whilst *Aq*. and *CE* gave the same results (0.040%) within limits of uncertainties. Overall, the conversion efficiency results were consistent with the optical absorption results in that the

better the absorbance the better the conversion efficiency.

In comparison with the indoor results in some other studies, the outdoor photoelectrochemical performance tests in our study showed rather very low results. Concurrent performance tests of the indoors and outdoors, however, would provide a proper baseline for the most appropriate conclusion of this difference in performance. Similarly, the results of *AWH* were within the same range as those of *Hibiscus*. Just like *Hibiscus*, *AWH* is a potential candidate material for DSSC applications because both the two have better optical absorption capabilities in the region stretching from the UV through the visible range of the electromagnetic spectrum though the LCCE is low. It remains the case that the LCCEs of most NODs in DSSCs are less than 1.0 %. Further studies, however, can be conducted to improve the performance of the dyes and the cell as a whole.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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