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# OPTICAL CHARACTERIZATION USING NATURE BASED DYE EXTRACTED FROM HIBISCUS'S FLOWER

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## ABSTRACT

Nature has the best way of providing the solution for the next energy harvesting mechanism from fruits plants and flowers. This paper proposes the use of a nature based dye extracted from Hibiscus flower which has potential light absorbing mechanism in dye-sensitized solar cell, an electrochemical oriented solar cell. Ultrasonic extraction treatment was enforced to Hibiscus petals with a temperature, time and frequency rating of 30 ºC, 30 minutes and 37 Hz, respectively. Different extracting solvent from ethanol and distilled water were used to observe the absorption spectra by using UV-Vis absorption spectroscopy. Fourier transforms infrared (FT-IR) were used to characterize the functional components of dyes. From FT-IR result, the presence of carbonyl (C=O) and amines group (N-H) represented the existence of betacyaninfrom the betalains group. The result shows that dye extracted under ethanol treatment has the highest absorbance rate compared to extraction by distilled water with the peak of 460 nm. As for the photon energy, results shows a narrow energy gap of 2.70 eV with higher absorption coefficient of 2.35k $m^{-1}$ in the visible light spectrum.

**Keywords:** natural dye, hibiscus, UV-Vis absorption, optical, photon energy, FT-IR.

## INTRODUCTION

Dye is an important element in dye-sensitized solar cell (DSSC) by enhancing the absorption of photon and transforming it into electrical energy. The wide band gap semiconductor can be sensitized by using these dyes due to high absorption of visible light spectrum from the red to blue wavelength (Susanti *et al.* 2014). So far, Ruthenium is the preferred sensitizer due to its high photon to electrical efficiency being in the range of 10-11%, yet high cost and limited resources factor tend to introduce the usage of nature based dyes into the DSSC which promises environmental friendliness and easy extraction method.

Natural dyes extracted from plants, fruits and flower contains pigments such as anthocyanin, chlorophyll, betalains and many more which could interact with the wavelengths of visible light to either being reflected or transmitted by plant tissues (Shelke *et al.* 2013). Low cost, complete biodegradation and non-toxicity of natural dyes are the reasons for extensive research being conducted throughout the world.

Malaysia's national flower which is the Hibiscus (Figure-1) or locally knows as the 'Bunga Raya' has been investigated as sensitizer dye-sensitized solar cell (DSSC). The Hibiscus, also known as Hibiscus-Rosa-sinensis comes from the Malvaceae family group. Hibiscus (H) was extracted by using distilled water (DI) and ethanol (Eth) as extract solvents. These extracted dyes were characterized by UV-Vis Spectrophotometer to observe on the absorption spectra and FT-IR spectral analysis to determine the functional group in the nature dye.

**Figure-1.** Hibiscus's Flower.

### DSSC components

The typical configuration of DSSC has shown in Figure-2 consists of two transparent glass coated with ITO (Indium Tin Oxide) to give the conductivity to the glass. The semiconductor material are the $TiO_2$ (Titanium Dioxide), iodide/tiiodide ($I^-/I_3^-$) and the counter electrode. The main parameter in DSSC is the dye itself. The dyes should fulfill the requirement such as having an intense absorption in the visible light spectrum. Besides, the dyes must possess the =O or OH stretching vibrations to help the $TiO_2$ surface chelate with the Ti(IV) sites (Narayan, 2012).

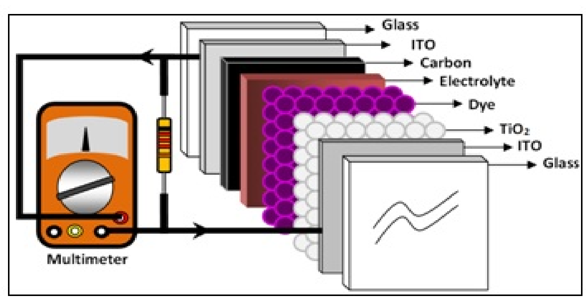


**Figure-2.** Cross section of DSSC.

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**DSSC operation**

In DSSC,the photoexcited dye transfers an electron to the semiconducting $TiO_2$ layer via electron injection as shown in Figure-3 which is the operation principle of DSSC. The injected electron is then transported through the porous $TiO_2$ layer and collected by the conductive ITO layer on the glass surface. Within the electrolyte, the mediator ($I^-/I_3^-$) trough redox couple which undergoes oxidation at the dye and regeneration at the catalyst-coated counter electrode. The cell is illuminated and current flows through the electrical load. The reactions found in the solar cell are:

(1) Dye + Light = Dye*
(2) Dye* + $TiO_2$ = $e^-$ ($TiO_2$) + Oxidized dye
(3) Oxidized dye + 3/2 I = Dye + ½ $I_3^-$
(4) ½ $I_3^-$ + $e^-$ (counter electrode) = 3/2 $I^-$

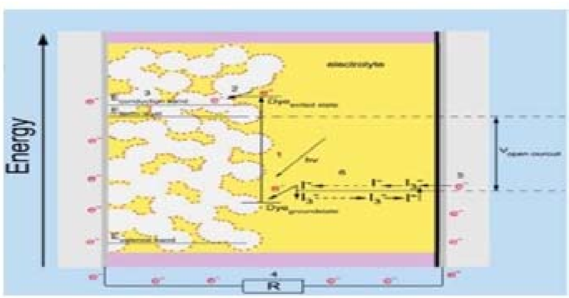


**Figure-3.** Operation principle of a dye-sensitized solar cell (Karki *et al.* 2013).

**Natural dye sensitizer**

Betalains represent the pigment in plant of Caryophyllales. Betalains replace the anthocyanin pigment in fuits and plant which is divided into two types, namely betacyanin (red-violet) and betaxanthins (yellow-orange). Betalains have ability to absorb in the range of 476 nm to 600 nm which represent the group of betacyanin and betaxanthins. Betalains, havinga carboxyl group compared to anthocyanin group whichcontains hydroxyl functional group as well. Carboxyl group creates a stronger electron coupling bound (Al-Alwani et l., 2015). Hibiscus represents the betacyanin group which represented the range color of red-violet. So far, the best performance of pigmentation was Betalains (Narayan, 2012).

**Previous research on natural based dye**

DSSC usually uses Ruthenium (II) complexes as sensitizers, but due to expensive in costing, natural dye has been promoted due to low cost and biodregadation. Several researches have focused on the electrical characterization of DSSC.Polo *et al.* investigated the natural dye from Calafate and Jaboticabaand shows result of Jaboticabato having a higher fill factor as much as 0.54 compared to Calafatewhich is only 0.36(Polo *et al.* 2006).Garcia *et al.* reported that by using Cabbage palm fruit as a natural dye sensitizer could produce higher fill factor at about 0.61 compared to chaste tree fruit and mulberry which are onlyat 0.51and 0.45, respectively (Garcia *et al.* 2003).This research is focused on the optical characterization of dye which is important in developing high efficiency of DSSC. Oviri and Ekpunobi stated that each dyes can be characterized from band gap analysis by using calculation to see whether the dyes has the potential to promotes light harvesting and highest absorption coefficient (Oviri and Ekpunobi, 2013).

**MATERIALS AND METHOD**

**Preparation of Natural Dye**

10 g of Hibiscus is mixed into 15 ml distilled water (DI) (and ethanol at separate sample) at room temperature. The Hibiscus is mashed using a mortar into paste. The samples are placed into an ultrasonic cleaner for 15 minutes with the frequency of 37 Hz using 'degas' mode at the temperature of 30 $^{O}$C shows in Figure-4. The coloring of the samples is segregated via 2500 rpm of centrifugal force for 25 minutes. All the procedure is shown in Figure-5 and Figure-6. The complete extracted dyes are shown in Figure-7.

**Figure-4.** Elmasonic P Ultrasonic Cleaner.

**Figure-5.** Preparation of natural dye sensitizer.

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**Figure-6.** Preparation of natural dye sensitizer.

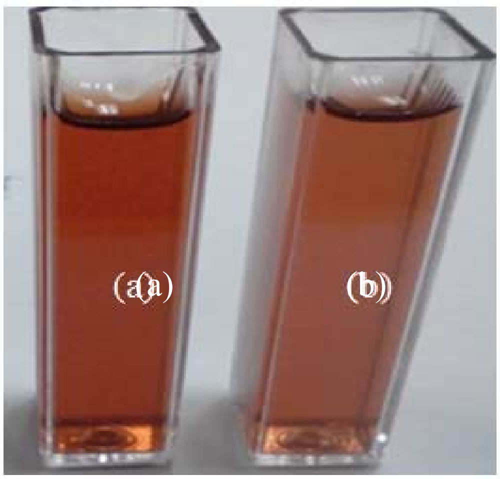


**Figure-7.** (a) Hibiscus with ethanol b) Hibiscus with DI water.

Notice the coloring of the dye represent the color of Hibiscus (H) extracted using distilled water and ethanol. Figure-1 (a) and (b) shows the colours of hibiscus remain the same which is light red (b) from Di-water and dark red (a) from ethanol solvent.

**Characterization and measurement**

The absorption spectra of the dyes were performed using Evolution 201 UV-Vis Spectrophotometer shown in Fig. 8. UV-Vis spectrophotometer is used to measure the absorbance rate in visible light spectrum. Fourier Transform Infrared spectra of the extracts were recorded in the range of 650-4000 cm$^{-1}$. The samples of dyes are filled into cuvette tube and place in sample holder in UV-Vis machine.The procedures to test the liquid sample is stated on (Gomesh *et al.* 2014). The photon energy; E, absorbed by these dyes is calculated by using formula in Eq. (1) in which h is represented by Planck's constant, λ is the wavelength of the absorbed visible light and c is the speed of light. The numerical values of the symbols are:-

$$E = \frac{hc}{\lambda}$$

(1)

$h$ = $6.63 \times 10^{-34}$ Js

c = $3.0 \times 10^{8}$ m/s

λ = Wavelength (nm)

The absorption coefficient determines how far into a material, light of a particular wavelength can penetrate before it is absorbed. The absorption coefficient of the respective wavelengths is obtained by the division of the absorbance with the wavelength shown in Eq. (2) using K as Boltzman constant;

$$Absorption\ coefficient = \frac{4\pi K}{\lambda}$$

(2)

Where λ stands for the cutoff wavelength of the dyes and Boltzman constant with the value of $8.617 \times 10^{-5}$ eV.

**Figure-8.** Evolution 201 UV-Vis Spectrophotometer.

## RESULT AND DISCUSSIONS

**FT-IR Spectra**

Figure-9 shows the FTIR spectra of the Hibiscus dyes extracted using Di-water (H-DI) and Ethanol (H-Eth). For H-DI, the peak at 1553 cm-1 represented N-H stretch. The peak at 1657 cm$^{-1}$ are attributed to the C=O stretch. The peak at wavenumber 3384 cm$^{-1}$ corresponds to the O-H stretching vibration of Hibiscus. The strong and broad bands at 3000-3700 cm$^{-1}$ occur due to the –OH groups of water. The presence of carbonyl (C=O) and amines group (N-H) is represented the existing of betacyanin group of betalains pigments as shown in Fig. 10 (Narayan, 2012).

FTIR spectra of the H-Eth starting from the peaks at 1050 cm$^{-1}$ and 1092 cm$^{-1}$ are attributed to the C-O stretching vibrations which represent the esters group. The peak at 1423 cm$^{-1}$ corresponds to the C-C stretching vibration in aromatic group. The two peaks at 2982 and 3398 cm$^{-1}$ represented the hydroxyl group which contained O-H stretching vibration. Amines group exhibit most characteristic group frequencies with the N-H bond.

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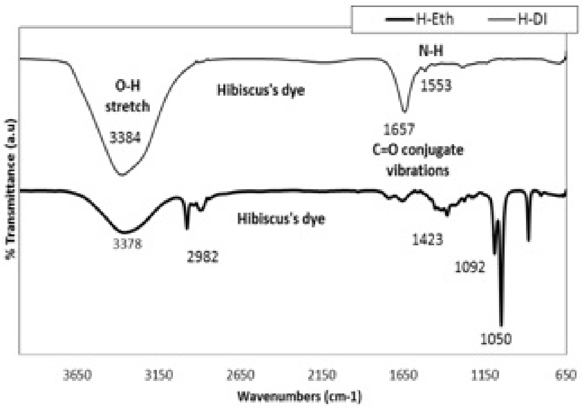


**Figure-9.** FTIR spectrum of the Hibiscus natural dye extracted by DI-water (H-DI) and Ethanol (H-Eth).

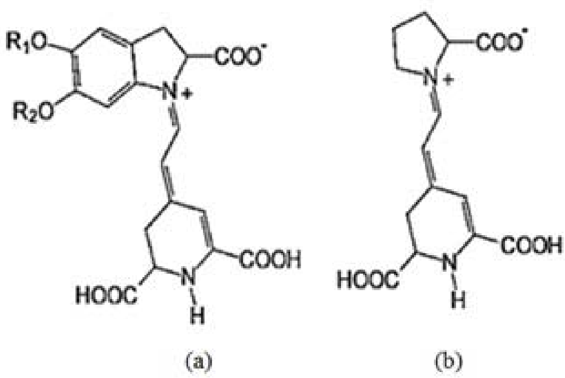


**Figure-10.** Structure of the betalain dyes (a) betacyanin and (b) indicaxanthin R1 and R2 = H (betanidin) or R1 = β-D-glucose and R2=H (betanin) (Narayan, 2012).

### UV-Vis Absorption Spectra

Based on Figure-11, the H-DI sample desorbs from 400 nm to the peak absorbance at 550 nm with its tail desorbs to broader wavelength while H-Eth desorbs from 400 nm to 420 nm and absorbs to the peak of 460 nm and towards the second peak at 550 nm and desorbs at further wavelength. Absorption with different wavelength will help the cells capture photons at two different energies. This result is concurrent with the finding from FTIR, which shows the betalains functional group being present in the sample from Hibiscus. The presence of hydroxyl groups in hibiscus will improve the electron transfer and enhance the efficiency of DSSC.

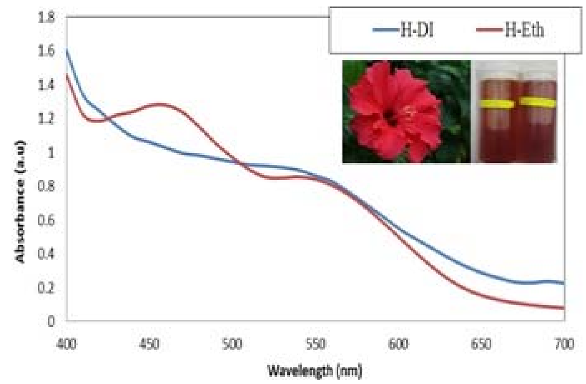


**Figure-11.** Absorption spectra of Hibiscus extracted by using Di-water (H-DI) and Ethanol (H-Eth).

### Optical characteristics of dyes

Table-1 shows the photon energy and the absorption coefficient (α) of the Hibiscus with different extract solvent from DI water and ethanol. From the table, the suitable extract solvent for Hibiscus was ethanol with the photon energy (eV) of 2.70 eV and highest absorption coefficient (α) with 2.35km$^{-1}$. Figure-12 shows the dependence of the absorption coefficient on the wavelength of the dye.

**Table-1.** The photon energy and absorption coefficient (α) of the Hibiscus with different extraction solvents from DI water and ethanol.

| Dyes | Extract solvent | Peak Absorbance (nm) | Absorption range (nm) | Photon energy (eV) | Absorption coefficient (α) k m$^{-1}$ |
|---|---|---|---|---|---|
| Hibiscus | Ethanol | 460 | 500-600 | 2.70 | 2.35 |
| | Distilled water | 550 | 500-600 | 2.26 | 1.97 |

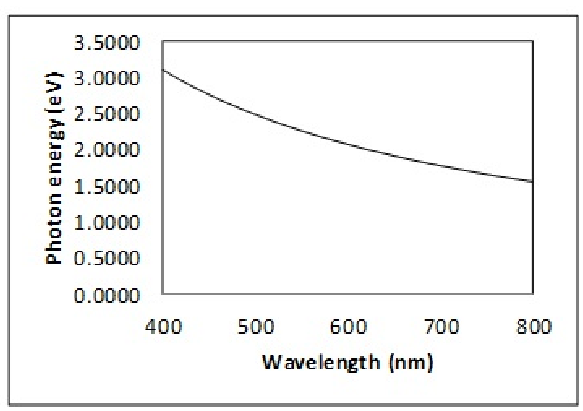


**Figure-12.:** Dependence of absorption coefficient on the wavelength of dye.

## CONCLUSIONS

A natural dye from Hibiscus was successfully extracted from flowers using a simple extraction of boiling process in an ultrasonic cleaner. The broad absorption peak for the dyes diluted with different extract solvent show different absorption rate with different wavelength in the visible light spectrum (400-700 nm) which indicates that a different extract solvent gives a different sensitizing performance. The present of carbonylgroup in Hibiscus was proven by the existing of N-H, C=O and OHstretches, which are an important spectroscopic measurement for betalains pigment.

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