

Influences of dye pH on the efficiency of dye-sensitized solar cells using natural dye extracted from *Clitoria ternatea*

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Abstract

The efficiency of dye-sensitized solar cell (DSSC) is dependent on the nature, optimization and compatibility of its components. A strong adsorption of the dye unto the titanium dioxide (TiO_2) is essential for the efficient electron injection into the TiO_2 semiconductor. *Clitoria ternatea* (CT) has a relatively high thermal stability, absorbance level, and anthocyanin content; however, it contains longer aromatic groups which affect the binding of dye molecules into the TiO_2 surface resulting to relatively low efficiency. This study was conducted to determine the influences of CT dye at different pH level, specifically, pH 1.0, 4.0, and 5.8 as dye sensitizer on the efficiency of DSSCs. Results show that CT extracts at pH 5.8 (control) has the highest absorbance followed by pH 4.0, and pH 1.0. However, when the dyes were sensitized in the TiO_2 , pH 4.0 has the highest amount of dye adsorbed and energy conversion efficiency of 0.05%.

Keywords: DSSC, *Clitoria ternatea*, photovoltaic, adsorption, pH

Introduction. Dye-sensitized solar cell (DSSC) developed by Gratzel in 1991 is a third-generation solar cell that uses dye sensitizers to convert visible light into electricity [1]. A typical DSSC is composed of dye-coated mesoporous TiO_2 thin film layer which absorbs light, a liquid electrolyte system composed of I^-/I_3^- redox couple and a platinum or carbon catalyst, in which all three parts are sandwiched between two FTO or ITO conducting glass substrate [2]. The total efficiency of DSSC is dependent on the nature, optimization, and compatibility of each one of the components of the solar cell and more particularly the photoanode which plays a vital role in charge generation and transfer processes [3]. Typically, the strong adsorption of the dye sensitizer with a wide and intense absorption spectrum into the TiO_2 electrode plays a vital role in charge generation and transfer processes and is essential in improving the energy conversion efficiency of the solar cell [3,4].

The most common dye being used in DSSC are Ruthenium Complex dyes; however, due to its cost, toxicity, and environmental impacts, other substitute dyes such as natural dyes are being studied [5]. Natural dyes such as flavonoid, betacyanin, anthocyanin, chlorophyll, tannin, and β -carotene provide natural, non-toxic and low-cost dye sources with high absorbance level of UV, visible and near IR and have been successfully used as dye sensitizers [6,7].

Blue ternate (*Clitoria ternatea* or *C. ternatea*), commonly known as blue pea, is one of the anthocyanin sources that has been incorporated in DSSCs. Having a relatively high thermal stability at 45°C [8], high absorbance level with two peaks at 580 and 620 nm [9] and high anthocyanin content of 227.42 mg/kg [10], *C. ternatea* would make a good dye sensitizer. However, although it contains mostly phenols, alcohols, and carboxylic acids [11], the anthocyanins present in blue ternate were found to be mainly ternatins [8]. Ternatins contain longer aromatic groups which affect the binding of the dye

molecules to the TiO_2 surface [12], making its overall efficiency relatively low. This is the main reason why cyanidins which can be found in red sources such as berries, mangosteen pericarp, and rosella, are preferred over delphinidin sources such as blue ternate that are mostly made up of ternatins [13].

Anthocyanins undergo transformations with changes in the pH, which has a dramatic effect on color [14]. In acidic conditions, the anthocyanins are mainly present as flavylium cations which are more stable, and are more capable in coordinating and forming intramolecular complex with Ti^{4+} sites [15]. Thus, aside from blue ternate having a high anthocyanin content which is expected to have a higher absorption as stated to the Beer-Lamberts' law wherein absorbance is directly proportional to concentration per unit area, by varying the pH of *C. ternatea*, the study aims to determine the pH influences on the performance of natural dye extracted from *C. ternatea* as sensitizer in DSSCs.

Only UV-visible spectroscopy and FTIR analysis were done in dye characterization due to unavailability of specialized equipment such as scanning electron spectroscopy (SEM) and x-ray diffraction (XRD) which can further support the study. Nevertheless, the data gathered from the two equipment were able to provide the necessary and sufficient information for the whole study.

As for the efficiency, only parameters open circuit voltage (V_{oc}) and short circuit current (I_{sc}) were measured due to unavailability of specialized equipment for voltage sweep analysis for the maximum power output (P_{max}). Instead, the actual values of the two were used and derived to calculate the underlying parameter of efficiency; namely, the fill factor (FF). Furthermore, the fabricated DSSCs were not tested with the standard illumination of air-mass 1.5 global (AM 1.5G) having an irradiance of 100 mW/cm^2 that can only be achieved by using a solar simulator.

The *C. ternatea* flowers were acquired from Alta Tierra Village, Jaro, Iloilo City. The whole study was conducted in a span of one month at PSHS-WVC Research Laboratory, October 2018.

The objective of this study is to measure and compare the efficiency of dye-sensitized solar cell with *C. ternatea* dye under different pH conditions as sensitizer. It specifically aims to:

- (i) To characterize the optical absorption of the extracted natural dye from *C. ternatea* under different pH conditions,
- (ii) To determine the adsorption *C. ternatea* dyes under different pH conditions to the TiO₂ surface as sensitizer,
- (iii) To measure the *voltage, current, and power output* of the fabricated DSSC with *C. ternatea* dyes under different pH conditions as sensitizer,
- (iv) To evaluate and compare the efficiency of fabricated DSSCs with *C. ternatea* dyes under different pH conditions as sensitizer

Methods. This experimental study determined if varying the pH of *C. ternatea* dye can improve its performance as natural sensitizer in dye-sensitized solar cells. The methods used were adapted from Vankar et al. (2010), Yang et al. (2014) Mohamed et al. (2017). The dye extraction was carried out using Vankar et al. (2010), while the Yang et al. (2014) and Mohamed et al. (2017) were used for the DSSC fabrication and testing.

Preparation of dyes. *C. ternatea* flowers were harvested at Alta Tierra Village, Jaro, Iloilo City. Petals were separated and cut into smaller pieces for dye extraction. For every 10 g cut petals, 100 mL ethanol 70% with 1 mL 0.1 M hydrochloric acid was added and was left at 25 °C for four days. The solid sediments were then filtered by gravity using a glass funnel with Whatman No. 1 filter paper. The combined filtrates were further concentrated in vacuum at 50 °C which resulted to 65.79% percentage yield.

Addition of buffer solution to dye extracts. The calculated percentage yield of the dye extract determined the amount of buffer solution to be added in every setup. Three setups under varying pH conditions were prepared as shown in Table 1. that corresponds to each pH level: 4.0, 1.0, 5.8 (control). For every 0.8 mL dye extract, 10 mL buffer solutions were used. The pH of each solution was checked using pH electrode (Globisens Enviro).

Dye Characterization. The absorbance spectra of the extracted *C. ternatea* dyes in different pH were characterized by using UV-vis spectrophotometer (UV-1800, Shimadzu). Characterization was done by measuring absorbance to visible light spectrum with spectral range of 400 nm to 700 nm. The functional groups present in the dyes were also characterized by Fourier-Transform Infrared (FTIR Spectroscopy (IR Affinity-1SWL, Shimadzu) using Attenuated Total Reflection (ATR) Method.

Table 1. Solvent proportions of buffer solutions

Setup	Features	Solvent Proportions (v/v) of Buffer Solution
A	Extracted <i>C. ternatea</i> dye (control)	Distilled water
B	Extracted <i>C. ternatea</i> dye at pH 4.0	50 mL 0.1 M potassium hydrogenphthalate (HK(C ₈ H ₄ O ₄)) with 0.1 mL 0.1 M hydrochloric acid (HCl)
C	Extracted <i>C. ternatea</i> dye at pH 1.0	25 mL 0.2 M potassium chloride (KCl) with 67 mL 0.2 M HCl

Adsorption test. Adsorption test was done using the desorption method. TiO₂ coated electrodes were dipped in the dye solution in ethanol and then dried at 25 °C. The TiO₂-dye coated electrodes were then immersed in 0.1 M NaOH solution for 1 hour. The estimated amount of the dye adsorbed was then calculated using the Beer-Lamberts' law [4].

Preparation of DSSCs. Fluorine-doped Tin Oxide (FTO) coated glass (sheet resistance <15 Ω/cm², transmittance ≥ 83%, Kaivo) was used as the conducting glass substrate and cut into pieces with dimensions of 2 cm by 2 cm in a glass and aluminum trading. To prepare the electrode, each glass substrate was first cleaned with distilled water, ethanol, and acetone then coated with TiO₂ paste using doctor blade method. The TiO₂-coated electrode was annealed at 250 °C for 15 min and then at 450 °C for another 15 min [16]. The coated electrode was then sensitized in 10 mL dye solution for 24 hours. The non-adsorbed dye was washed up with distilled water and ethanol and dried in open air. For the counter electrode, graphite layer was obtained by rubbing pencil lead on the conducting glass surface.

Safety Procedure. The study was conducted with proper procedures. The researchers wore lab gowns, gloves and goggles as precaution to chemical spillage. First aid kits were readily-available during the conduct of the study in case of emergency. Material Safety Data Sheet (MSDS) of each chemicals used were taken into consideration for safely handling and working with a particular substance.

Results and Discussion. The effect of pH was investigated in absorbance of *C. ternatea* as a dye sensitizer. Figure 1 shows the UV-vis optical absorption spectra of *C. ternatea* dye extracts at pH 5.8, 4.0, and 1.0. Absorption spectra of a dye reflect optical transition probability between the ground state, the excited state and the solar energy range absorbed by the dye. It can be observed that there is a shift in the absorbance spectrum from right to left as the pH level of the sample decreases.

These shifts indicate the relationship of conjugation and structural changes in the samples as changes in pH were introduced; this can evidently be seen in as pH has a great impact to the color of natural and

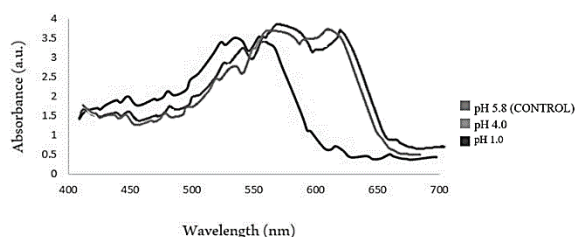


Figure 1. Graph showing the absorption spectra of *C. ternatea* dyes at different pH

and organic dyes. *C. ternatea* extracts at pH 1.0 shows single absorption peak (max) centered at 532.4 nm with 3.848 absorbance. pH 4.0 and 5.8, on the other hand, have two absorption peaks at 565.6 nm and 615.4 nm, and 565.6 nm and 615.2 nm, respectively. It is clear that the two treatments have close max values but differ in terms of absorbance values. As shown in Table 2, the pH extract solution has no significant effect on the absorbance. At 565.6 nm max, pH 4.0 has 3.956 absorbance while pH 5.8 has 3.94. On the other hand, at 615 nm, pH 5.8 with 3.858 absorbance is higher than that of pH 4.0 with absorbance value of 3.666.

Table 2. Absorption peak and the corresponding absorbance *C. ternatea* at different pH

Treatment	max (nm)	Absorbance (a.u)
Control (pH 5.8)	565.6 ; 615.2	3.94 ; 3.858
pH 4.0	565.6 ; 615.4	3.956 ; 3.666
pH 1.0	532.4	3.848

Infrared absorption spectrum of a material or sample has a distinctive pattern so that it is possible to identify the material and to show the existence of the major functional groups in the identified structure. As shown in Figure 3, the FTIR spectral analysis of *C. ternatea* reveals five distinct peaks at 3331, 2361, 2110, 1636, and 608 cm^{-1} . FTIR spectral analysis of *C. ternatea* reveals distinct peaks at 3331, 2361, 2110, 1636, and 608 cm^{-1} . The broad and strong peak at 3331 cm^{-1} indicates the presence of C-H stretching from the CH_2 group. The O-H stretching vibration can be found at the band that appeared at 2361 cm^{-1} . Absorbance at 2110 cm^{-1} , on the other hand, is due to $\text{C}\equiv\text{C}$ stretching vibration, while at 1636 cm^{-1} shows presence of $\text{C}=\text{O}$ or N-H vibration of either -COOH and amide groups. The band at 608 cm^{-1} arises from b-glucosidal linkage. Hence, FTIR spectral analysis shows the presence of chemical groups like -CH, -OH, $\text{C}\equiv\text{C}$ and $\text{C}=\text{O}$ in *C. ternatea*.

Table 3 also presents the wavenumber of each five peaks together with their corresponding percent transmittance (%T). As absorbance (A) = $-\log (\%T)$, higher peak intensity indicates low transmittance and hence, large absorption. This shows that the extracted *C. ternatea* dye contains mostly carbonyl (74.98 %T), CH_2 group (58.69 %T), and b-glucosidal linkage (45.60 %T).

Table 3. Extracted *C. ternatea* dye peaks, %T and present functional groups

Wavenumber (cm^{-1})	Transmittance (%T)	Functional group present
3331.07	58.69	C-H from CH_2
2360.87	95.12	O-H
2110.12	95.02	$\text{C}\equiv\text{C}$
1635.64	74.98	COOH and amide group
607.58	45.60	B-glucosidal linkage

Desorption method was done to estimate the amount of dye adsorbed in the photoanode. The higher the concentration of the adsorbed dyes, the more efficient the charge generation and transfer processes of the solar cell. Table 3 shows the absorbance and the calculated concentration of the dye adsorbed in the TiO_2 layer. It can be seen that pH 4 and pH 5.8 are relatively close than pH 1. In terms of absorbance, pH 4.0 yields the highest with 3.633, followed by 3.051 of pH 1.0, and 2.408 of pH 5.8. This is also the same in terms of the concentration adsorbed 1.35×10^{-4} mol/L in pH 4.0, 1.13×10^{-4} mol/L in pH 5.8, and 8.95×10^{-5} mol/L of pH 1.0.

Table 4. Absorbance and Concentration of desorbed dyes in varying pH

Treatment	Absorbance	Concentration (adsorbed)
Control (pH 5.8)	3.051	1.13×10^{-4} mol/L
pH 4.0	3.633	1.35×10^{-4} mol/L
pH 1.0	2.408	8.95×10^{-5} mol/L

Table 4 shows the actual measurements of the short circuit current (I_{sc}) and open circuit voltage (V_{oc}), and the theoretical value of the fill factor (FF) of each fabricated solar cell at different pH. The table shows that pH 4.0 has the highest energy conversion efficiency (η) with 0.180% efficiency followed by 0.070% of pH 5.8 (control), then 0.010% of pH 1.0. This was also confirmed by the three parameters--open circuit voltage, short circuit current, and fill factor, as pH 4.0 obtained the highest values in all three. Although pH 5.8 and pH 4.0 are relatively closer in terms of V_{oc} , it is obvious that their I_{sc} values are different having 2.86A at pH 5.8 while 6.44A at pH 4.0. The values measured from the short circuit current somehow determined which of the fabricated dye sensitized solar cells are more efficient than the others.

Anthocyanins may exhibit different colours, depending on their structure. At a given pH, equilibrium exists between anthocyanin structures: a blue quinoidal (anhydro) base (A), a red flavylium

Table 5 Average Isc, Voc, FF and efficiency from the fabricated solar cells at different pH

Treatment	Voc (mV)	Isc (A)	FF	N (%)
Control (pH 5.8)	81.64	2.86	0.59	0.070
pH 4.0	92.50	6.44	0.63	0.180
pH 1.0	28.13	0.70	0.56	0.010

cation (AH⁺), the colourless carbinol pseudobase (B), and chalcone (C). At neutral or slightly acidic condition, anthocyanins exist predominantly in their colourless forms, due to the instability of the anhydro base. This is confirmed by the absorbance spectra found at pH 5.8, as aside from having a blue color, it also exhibits two absorbance peaks which is another distinct characteristic of a blue quinoidal base. Lowering the pH is largely attributed to the higher concentration of the flavylium cations, which can be seen in pH 1.0 having a red color. UV-vis spectroscopy results showed no significant difference in terms of absorbance. Thus, it can be observed that the only differences between the different treatments can be attributed to their structure. Anthocyanins in flavylium cation forms are more stable as compared to those in quinoidal base forms. Thus, highly acidic conditions such as pH 1.0, anthocyanins have high stability and absorbance [17]. At pH 4.0, the stability of the anthocyanins is attributed to the acylated anthocyanins present in the extract [18]. The highest absorbance showed by the CT extract at pH 4 shows that polyacylated anthocyanins are also stable in weak acidic conditions as they undergo B-ring substitution [19]. The common characteristics shown by acylated B-ring substituted anthocyanins is the additional absorbance as the acidity of the extracts decreases.

The efficiency of the cell depends on the cells ability to collect and generate photons [20] measured by the short circuit current (Isc), ability to replenish electrons within the system [21] measured by the open circuit voltage (Voc), and the ratio of the maximum power from an actual solar cell to the maximum of an ideal solar cell [22], the fill factor (FF). Efficiency is mainly affected by Isc, Voc and FF. In this study, pH 4.0 has the highest energy conversion efficiency followed by the control, and lastly, pH 1.0. This is because the Voc and Isc of pH 4 is higher than other treatments. The separation of the electron and the hole quasi-Fermi levels defines the maximum achievable Voc. Higher bandgap of the material leads to higher Voc [23], meaning Voc is directly proportional to the bandgap of the semiconductor. Bandgap refers to the energy required to transfer an electron from valence to conduction band [24] and is dependent on the absorbance of the material [25]. This can be correlated with the results of the UV-vis spectroscopy wherein pH 4.0 and 5.8 has relatively higher Voc having two absorbance peaks compared to the pH 1.0 with only one absorbance peak. The reason why pH 4.0 has a higher Voc measurement than pH 5.8 might be caused by the B-ring substitution and acylation that occurred in pH 4 which enhances the stability of the anthocyanin through intermolecular co-pigmentation [26]. The results show that there is a

correlation between efficiency and stability as a function of dye acidity. Additionally, Isc is the rate of collecting photons from the solar cell. The illumination of the light source to the solar cell affects the Isc [27]. The photon collection property affects the Isc. As Isc is also related to the absorption, the trend with pH 4.0 having the highest, followed by 5.8, and 1.0 can still be observed. Higher FF means that the quality of the solar cell is good while low FF indicates that solar cell is in low quality condition [21]. In the equation on calculating the FF, it can be observed that Voc mainly affects the FF of the solar cell. Efficiency is the measure output power at a given input power. It can be calculated by multiplying Voc, Isc and FF. pH 1 exhibits the lowest efficiency. This might be due to acid leaching that leads to deterioration of cells. This can also be reflected in the adsorption test wherein the concentration of dye adsorbed 1.35×10^{-4} mol/L in pH 4.0, 1.13×10^{-4} mol/L in pH 5.8, and 8.95×10^{-5} mol/L of pH 1.0.

It is found out that by lowering the pH through buffer solutions, *C. ternatea* undergoes structural transformation which further improve the dye adsorption as well as the stability of the dye itself; however, at highly acidic conditions like pH 1.0 exhibits the lowest efficiency of 0.01% that is caused by acid leaching that occurred in the TiO₂ semiconductor. The photocatalytic activity of the TiO₂ decreases due to the damage located at the surface the semiconductor. The highest efficiency was exhibited by CT extract at pH 4.0 due to the acylation occurred with B-ring substitution that enhances that stability of the anthocyanin as well as its absorbance at broader wavelengths.

Error Analysis. The overall performance of fabricated DSSC was compared with theoretical value. It was observed that there is 50% percent error in efficiency. There are several factors which affects the performance of a fabricated DSSC. One of these factors is the drying of liquid electrolyte solution. It plays an important role in the process of light-to-electricity conversion in DSSC. Contamination of dust in the surface of the TiO₂ also affects the photocatalysis of semiconductor. The internal resistance of the FTO glass affects the efficiency of the solar cell as higher internal resistance of the glass substrate decreases the current density-voltage curve area of the fabricated solar cell. Lastly, the illumination of the light source to the solar cell affects the Isc.

Conclusion. The most efficient dye-sensitizer extracted from *C. ternatea* out of the three setups is the dye pH 4 that exhibits a high absorbance and adsorption on the TiO₂ surface has the highest efficiency out of the three setups as the two prerequisites are directly proportional to the amount and intensity of the trapped light in the DSSC. It was observed that the adsorption and absorption of the dyes depends on the chemical structure present in the dyes. Therefore, by lowering the pH of *C. ternatea* dyes as sensitizer, which changes the structure of the dyes, could improve the efficiency of the DSSC; however, strong acidic environment such as pH 1.0 resulted in leaching of the adsorbed dye from the TiO₂ surface which can damage the surface and the active area of the semiconductor, affecting the charge

generation and transfer processes of the entire solar cell.

Recommendations. Further studies should explore on other pH values of dye extracted from the petals of *C. ternatea*. Also, to better understand the adsorption and interactions between the dye molecules and other components, it is also recommended not only to use UV-vis spectrophotometer and FTIR spectrometer, but other methods of analysis as well, such as scanning electron microscopy (SEM) analysis, x-ray diffraction (XRD), and transmission electron microscopy (TEM). The actual maximum power output (Pmax) must also be measured in order to generate an I-V (current vs. voltage) curve for a more accurate measurement of the efficiency of the solar cell. Furthermore, it may also be beneficial to have the fabricated DSSCs tested using a solar simulator with the standard illumination of air-mass 1.5 global (AM 1.5G) having an irradiance of 100 mW/cm² for a more reliable and accurate results. Although there is a 50 percent error between the theoretical and the actual efficiency values, the present study suggests that if the factors affecting the Isc and Voc values were lessened and if all the measured values were maximized up, *C. ternatea* dye may be compared with existing organic dyes with high efficiency.

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