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Study of Natural Herbal Dyes Photodegradation Effect to Optical Properties for Dye-Sensitized Solar Cells

(Kajian Kesan Fotodegradasi Pewarna Herba Semula Jadi kepada Sifat Optik untuk Sel Suria Peka Pewarna)

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ABSTRACT

The stability of natural dyes for use in dye-sensitive solar cells is known from their photodegradation characteristics. Photodegradation effects using artificial light radiation on natural herbal dyes were investigated. Three natural herbal dyes, namely *Fagraea acuminatissima*, sappanwood, and kulit setong were used in this study. The stability of the natural herbal dyes under artificial light radiation was studied. The optical properties of the natural herbal dyes as indicators of their stability were characterized using FTIR and UV-VIS absorption. Stability testing of natural herbal dyes was conducted in a room with artificial lighting for 5 weeks. Observations of changes in their optical properties were performed weekly. It can be seen that all three natural herbal dyes contained anthocyanin. In addition to anthocyanin, *Fagraea acuminatissima* and sappanwood also contain chlorophyll. In contrast, the kulit setong contains beta-carotene in addition to anthocyanin. The degradation percentage showed that sappanwood degrade to all of its pigments. While an increase occurred in the chlorophyll peak of *Fagraea acuminatissima* and the beta-carotene peak of kulit setong. This clearly shows that *Fagraea acuminatissima* has the highest stability, and potentially increases the efficiency of solar cells.
Keywords: Dye-sensitized solar cell; herb; natural dyes; optical properties

ABSTRAK

Kestabilan pewarna semula jadi untuk digunakan dalam sel suria sensitif pewarna diketahui daripada ciri fotodegradasinya. Kesan fotodegradasi menggunakan sinaran cahaya buatan pada pewarna herba semulajadi telah dikaji. Tiga pewarna herba semula jadi iaitu *Fagraea acuminatissima*, sappanwood dan kulit setong digunakan dalam kajian ini. Kestabilan pewarna herba semulajadi di bawah sinaran cahaya buatan telah dikaji. Sifat optik pewarna herba semula jadi sebagai penunjuk kestabilan mereka dicirikan menggunakan penyerapan FTIR dan UV-VIS. Ujian kestabilan pewarna herba asli telah dijalankan di dalam bilik dengan pencahayaan buatan selama 5 minggu. Pemerhatian terhadap perubahan sifat optiknya dilakukan setiap minggu. Dapat dilihat bahawa ketiga-tiga pewarna herba semula jadi mengandungi antosianin. Selain antosianin, *Fagraea acuminatissima* dan sappanwood juga mengandungi klorofil. Sebaliknya, kulit setong mengandungi beta-karotena sebagai tambahan kepada antosianin. Peratusan degradasi menunjukkan bahawa kayu sappan merosot kepada semua pigmennya. Manakala peningkatan berlaku pada puncak klorofil *Fagraea acuminatissima* dan puncak beta-karotena kulit setong. Ini jelas menunjukkan bahawa *Fagraea acuminatissima* mempunyai kestabilan tertinggi dan berpotensi meningkatkan kecekapan sel suria.
Kata kunci: Herba; pewarna semula jadi; sel suria peka pewarna; sifat optik

INTRODUCTION

The reduced supply of electrical energy by utilizing a limited number of sources can hinder the use of equipment related to the use of electricity sources (Gracheva & Alimova 2019; Toshpulatov et al. 2020). An alternative is needed to substitute for unlimited sources of electrical energy (Stone 1993; Toshpulatov et al. 2020). Abundant natural resources can be used as an alternative source of electricity (Durgawati Vishwavidyalya & Kumar Jawre 2018; Khomsah et al. 2019). One of them is the sun, the process of absorbing radiation from solar light that can be converted into a source of electric power so that the primary source can be used continuously (Dhafina, Daud & Salleh 2020; Pearson 1957). The absorption of solar radiation that hits the solar panels can be utilized and converted into a source of electric power (Ishak et al. 2019; Kaswan, Sonel & Yadav 2022). Solar panels consist of several types of their applications, including silicon-based, thin film, and electrochemical (Diner 2011). Dye-sensitized solar cell (DSSC) is a third-generation solar cell with electrochemical base materials and a light source as the basis for generating electrical energy (Wei 2010). Natural resource supplies can be used to make DSSC solar cells, such as flowers, leaves, wood, and roots found in plants (Gómez-Ortíz et al. 2010; Shakeel Ahmad, Pandey & Abd Rahim 2017). The selection of DSSC solar cell materials that are easy to obtain, friendly to the environment, flexible, and inexpensive can be used as a development to increase efficiency and dimensions in large quantities (Ghann et al. 2017; Shibl et al. 2013). In this study, natural herbal dyes were used, namely *Fagraea acuminatissima*, sappanwood and kulit setong, which contain anthocyanin, chlorophyll, and beta carotene pigments so that they can convert the light to produce electrical energy. The fabrication process is carried out by extracting the essence into powders, which can be dissolved using ethanol to obtain the highest pigment (Amelia & Gunlazuardi 2023; Ruhane et al. 2017). The method used in this study was to vary the amount of ethanol solution with natural dye so that the highest pigment content value was used as the basis for the DSSC solar cell layer (Yusupandi et al. 2022). The research objective was to obtain the pigment lowest degradation value as a coating material for DSSC solar cells so that a high efficiency.

MATERIALS AND METHODS

PREPARATION OF NATURAL HERBAL DYES

In this study, natural herbal dyes were selected, followed by pigment extraction process (Boo et al. 2012; Syafinar et al. 2015). The next step is making a photodegradation chamber and monitoring system for light intensity and temperature inside the chamber (Gautam et al. 2016; Zyoud et al. 2011). Photodegradation of the three natural herbal dyes was conducted for 5 weeks. After completing the photodegradation, characterization was performed to determine the degradation in natural herbal dyes and analyze the results. The following is a brief explanation of each step that will be carried out. *Fagraea acuminatissima*, sappanwood and kulit setong were collected around Terengganu. This selection is because these three natural herbal dyes are known to contain anthocyanins. The fresh *Fagraea acuminatissima* were first washed to remove the dirt and impurities. Then, they were dried in the oven at 60° until their mass remained constant. Once completely dried, they were ground into a fine powder using a grinder. One gram of *Fagraea acuminatissima* powder was soaked in 10 mL of ethanol and kept in the dark for 3 days. After 3 days, the solid residues were filtered out using filter paper to obtain a natural herbal dye solution. The dye solution was wrapped in aluminium foil to protect it from exposure to direct sunlight and stored in the refrigerator. The same extraction process was repeated for sappanwood and kulit setong (Pramananda, Hadyan Fityay & Misran 2021; Taya et al. 2015).

LIGHT INTENSITY, TEMPERATURE ADJUSTMENT AND PHOTODEGRADATION PROCESS

The light intensity and temperature outside the room were checked to adjust the level of radiation in the photodegradation chamber (Ansir et al. 2021; Kim et al. 2015; Mehmood et al. 2017). Photodegradation was performed using artificial lighting generated from 3 incandescent lamps with a power of 60 W each. Photodegradation was carried out in a room with a dimension of 100 × 75 × 50 cm. Overall, the light intensity generated by all incandescent lamps was 30 W/m2 with a temperature inside the photodegradation chamber of 35 °C. The intensity and temperature in the degradation chamber were monitored continuously so that the dye sample in the bottle does not evaporate excessively and can be appropriately degraded. The natural herbal dye was inserted after the light intensity and temperature in the photodegradation chamber were adjusted. An illustration of the photodegradation chamber, lamp and natural herbal dye sample is shown in Figure 1. Using the First-Order reaction approach, the pseudo rate constant can be determined, representing the rate of pigment decrease in natural herbal dye (Lavakusa et al. 2020). The decay of the pigment absorption value in the natural herbal dye is given in Equation (1) as follows,

$$A = A_0 e^{kt} \quad (1)$$

whereas A is the pigment absorption value after artificial light irradiation; is the initial pigment absorption value before irradiation A_0 with artificial light; k is the pseudo rate constant, and; t is the time of artificial light irradiation. If Equation (1) is formed into a linear equation by converting the absorption value into the natural logarithm scale, in the Langmuir–Hinshelwood model; k will be obtained as the slope of the linear equation. The linear equation for the natural logarithm scale is written in Equation (2) herewith (Singh et al. 2018),

$$\ln (A/A_0) = -kt \quad (2)$$

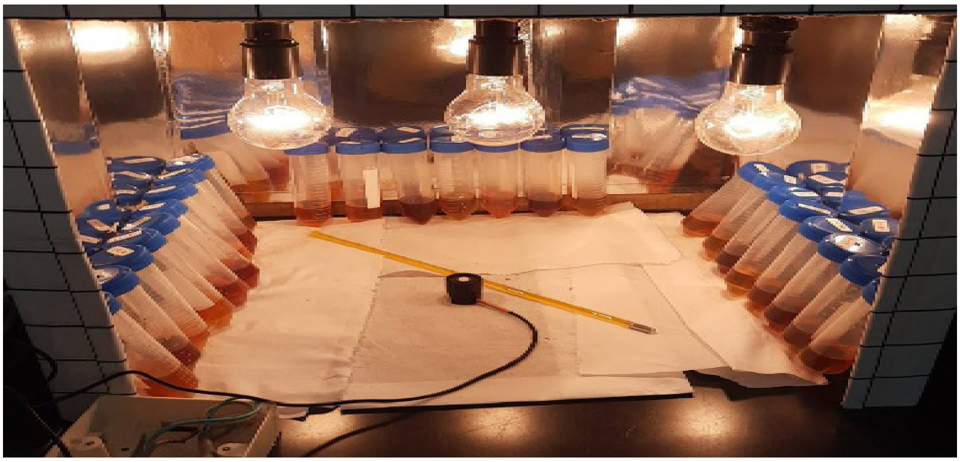


FIGURE 1. Photodegradation natural herbal dyes process

CHARACTERIZATION OF ABSORPTION WAVELENGTH AND FUNCTIONAL GROUP OF NATURAL HERBAL DYES

Photodegradation was carried out for 5 weeks, and each natural herbal dye was analyzed weekly. Measurements for absorption with UV-Vis (Ultra Violet-Visible) spectrometer and functional group content with Fourier Transform Infra-Red (FTIR) to determine pigment degradation were performed (Singh & Koiry 2018). Characterization of absorption values using UV-Vis SHIMADZU UV-1800 in the wavelength range of 350-750 nm. Meanwhile, the functional group content of natural herbal dyes was measured using the attenuated total reflectance Fourier infrared (ATR-FTIR). The spectra were recorded within a spectral window from 500 to 4000 cm-1 using NICOLET iS20 with a 1 cm-1 resolution.

RESULTS AND DISCUSSION

OPTICAL AND STRUCTURAL PROPERTIES OF NATURAL DYES

From the photodegradation process carried out for five weeks, it can be seen that there is a color change in natural dyes. The color change from before to the end of week five is shown in Figure 2. Figure 2 also shows the natural dye before and after degradation with the color density of each dye. After the process of photodegradation, the dye underwent a color change from the Fagraea acuminatissima dye type, which had a thickness from transparent brown to almost dark brown, Sappanwood was degraded from dark brown to transparent brown and kulit setong underwent a change in density from purple to dark purple. The natural photodegradation of all-natural herbal dyes showed changes in pigment composition when exposed to light irradiation. The significant degradation can be attributed to the lack of pigmentation in the dye molecules (color tone becomes lighter). The measurements on the research results were repeated five times with different conductivity area positions. The research was conducted under conditions affected by light (bright) and conditions not affected by light (dark). These pigments absorb the energy of light radiation and have a protective effect on enzymes in other molecular systems. Conversely, the color tone becomes darker in reactions where pigment increases (Falguera, Pagán & Ibarz 2011; Matus et al. 2009).

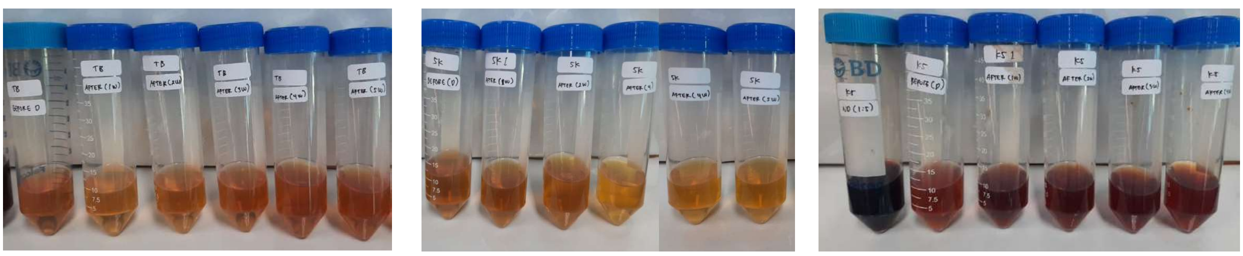
Furthermore, the UV Vis absorption test results are given to observe the absorption peaks at certain wavelengths. These peaks indicate the pigment content in the natural herbal dyes. Natural herbs dye solutions with various concentrations of ethanol were studied. The ratio dye to ethanol was prepared with 1:5, 1:10, 1:15, and 1:20. The absorbance of each natural herb dye with variations in concentrations of dye solution was tested using a UV-Vis spectrophotometer. From the study, the best ratio concentration of dye:ethanol is 1:5. However the results are not represented here. Five gram of samples were put in 100 mL bottles containing 50 mL of ethanol, and then the solutions were kept for three days in the refrigerator. Subsequently, the extracts were filtered with filter paper (Whatman No.1), and purple extract was collected in a 50 mL beaker (Arifin et al. 2021). In Figure 3, the UV Vis absorption plots for the three natural herbal dyes are given. The figure shows the absorption peaks associated with anthocyanin and chlorophyll content in natural. Meanwhile, Table 1 shows the wavelength of the absorption peak of each pigment and its absorption value in the three natural herbal dyes.

Figure 3 shows that the three of natural herbal dyes contain anthocyanin. In Figure 2(a) for Fagraea acuminatissima, the anthocyanin peak appears in two wavelength ranges of 470-550 and 550-600 nm (Ahliha et al. 2018), with peaks at 447 and 652 nm, respectively

(Ge et al. 2015; Mancinelli 1990). Each absorption peak of this pigment has an absorption value of 2.65 and 0.12, respectively. In addition, Fagraea acuminatissima contains chlorophyll, whose absorption peak appears at 400-425 nm with a peak at 400 and has an absorption value of 0.84. In Figure 2b, it can be seen that sappanwood has only one peak of anthocyanin at an absorption wavelength of 500-550 nm with a peak at 525 nm and an absorption value of 0.66. While the content of chlorophyll is seen in two absorption wavelength ranges at 440-460 nm (Arifin et al. 2021) and 650-670 nm (Nurul Huda et al. 2023), with peaks at 481 and 582 nm, respectively. Each absorption peak of this pigment has an absorption value of 2.84 and 0.28, respectively. For kulit setong, the pigment content is clearer. Figure 2(C) shows that the peak of anthocyanin appears in the absorption wavelength range of 550-580 nm with a peak at 588 nm and an absorption value of 0.66. The other content is beta-carotene, whose absorption wavelength peak appears in the 460-500 nm range with a peak at 463 nm and has an absorption value of 2.12. It can be seen that the anthocyanin absorption value in Fagraea acuminatissima is higher than that in sappanwood and kulit setong. While the chlorophyll absorption of Fagraea acuminatissima was lower than that of sappanwood (Agati 1998; Gitelson, Merzlyak & Lichtenthaler 1996). Thus, it can be said that Fagraea acuminatissima is dominated by anthocianin, and sappanwood is dominated by chlorophyll a. While the kulit setong is dominated by beta carotene (Tan & Soderstrom 1989). Although anthocyanin is present, the absorption value of this pigment is comparable to the absorption value of sappanwood. In addition to UV-Vis's absorption, changes in natural herbal dye against artificial light radiation can also be seen from the content of functional groups obtained from FTIR (Institute of Electrical and Electronics Engineers. 2013; Supriyanto, Nurosyid & Ahliha 2018; Tahir et al. 2018). Measurements of the content of functional groups in natural herbal dyes are carried out in the wavenumber range of 500 - 4000 nm (Nurul Huda et al. 2023). The results of FTIR measurements are given in Figure 4 and Table 2. In the three natural herbal dyes before photodegradation, four peaks of O-H stretching, C-H stretching, C-O-C stretching, and C-H bending are well visible. All functional groups have relatively the same wavenumber in all samples. In this section, the UV Vis absorption and functional group measurement results of the natural herbal dye after degradation for 5 weeks are given. In each Figure and Table presented, data before degradation is also included for comparison.

In Figure 5(a) and Table 3, it can be seen that absorption after being irradiated using artificial light for Fagraea acuminatissima is increased. In the first and second weeks, the absorption value for anthocyanin the wavelength range of 470-550 nm was relatively the same. However, the absorption value increased to 1.12 in the third to fifth week. While the absorption value of anthocyanin the wavelength range of 500-550 nm tends to decrease, it reached an absorption value of 0.17 in the fifth week. At the same time, chlorophyll show a different phenomenon where the absorption value increases every week until, in the fifth week, the absorption value reaches 2.54. Figure 5(b) and Table 4 show the decrease in sappanwood absorption values after artificial light irradiation. All pigments decreased from the first week to the fifth week. In the chlorophyll in the wavelength range of 440-460 nm, the absorption value in the fifth week decreased to a value of 1.11. While chlorophyll, in the wavelength range of 650-670 nm, had an absorption value before photodegradation of 0.12, in the fifth week, the absorption value was only 0.02. The anthocyanin also decreased, whose absorption value in the fifth week was 0.17.

From Figure 5(c) and the value given in Table 5, we can see an increasing absorption trend in kulit setong after artificial light irradiation. However, the anthocyanin in the wavelength range of 550-580 nm showed a decreasing trend



(a) (b) (c)

FIGURE 2. Natural herbal dyes color changes before and after artificial light radiation for (a) *Fagraea acuminatissima*, (b) sappanwood, and (c) kulit setong

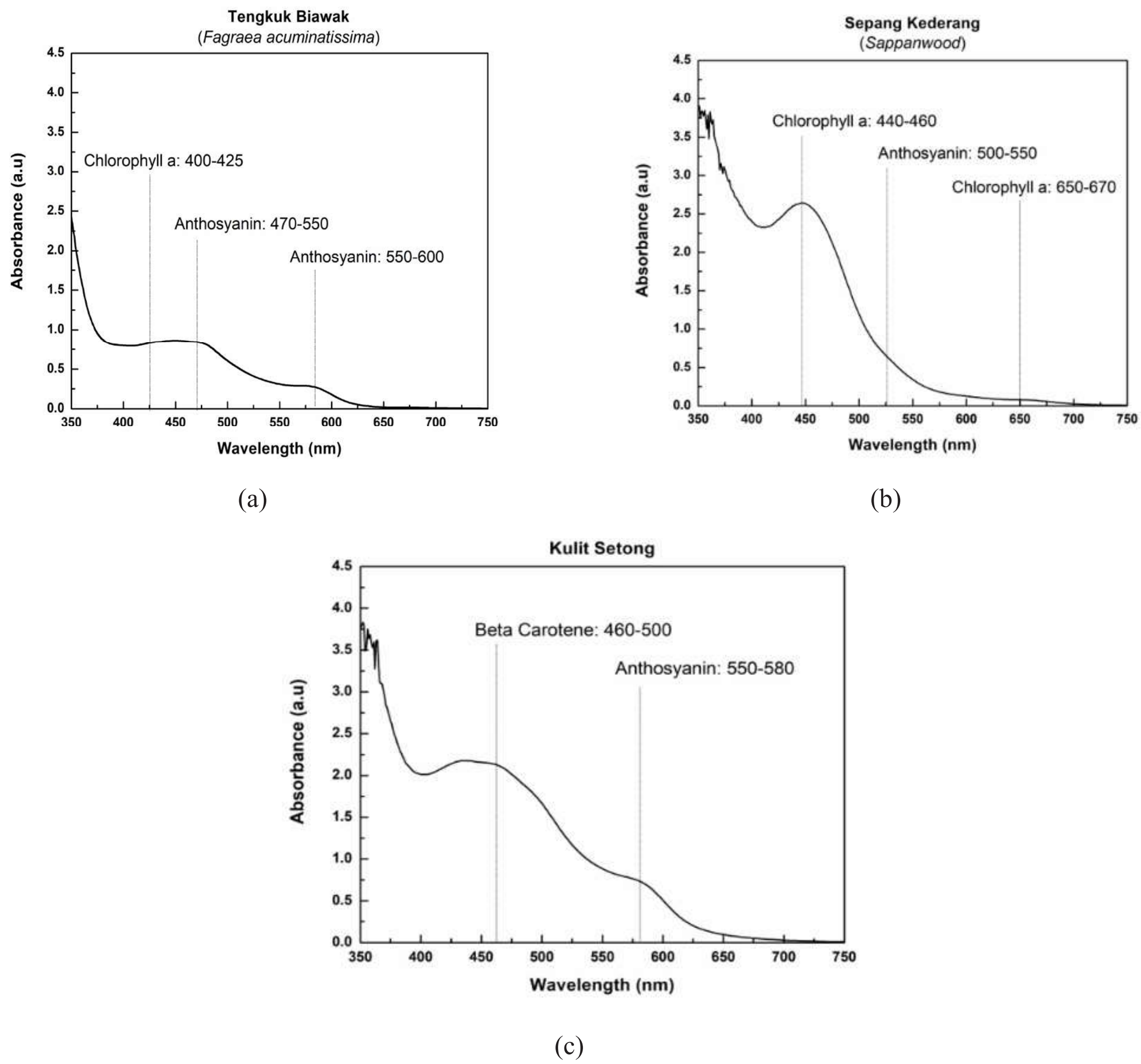


FIGURE 3. UV-Vis's absorption natural herbal dyes before photodegradation

TABLE 1. Peaks and absorption values of pigments from natural herbal dye using UV-Vis before photodegradation

Pigment	*Fagraea acuminatissima*		sappanwood		kulit setong	
	Wavelength (nm)	Absorption (a.u)	Wavelength (nm)	Absorption (a.u)	Wavelength (nm)	Absorption (a.u)
Chlorophyll a	400	0.84	447	2.65		
			652	0.12		
Anthocyanin	481	0.84				
	582	0.28	525	0.66	588	0.66
Beta-carotene					463	2.12

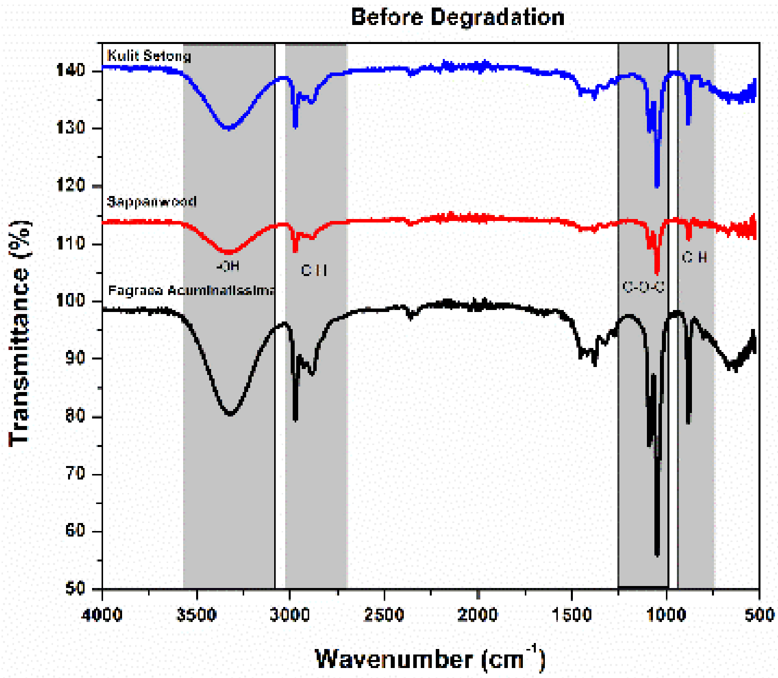
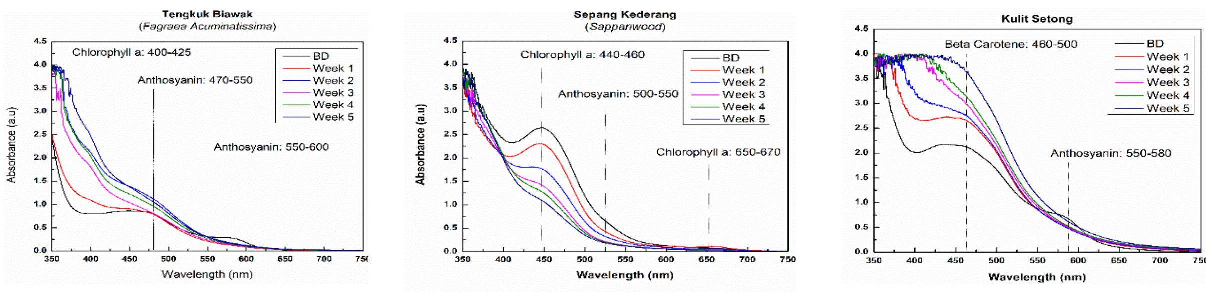


FIGURE 4. FTIR spectra of natural herbal dyes
before photodegradation

TABLE 2. Functional groups and wavenumber of peaks in natural herbal dye before photodegradation

Functional group	Wavenumber (cm^{-1})		
	Fagraea acuminatissima	sappanwood	kulit setong
O-H stretching	3317.93	3332.87	3327.57
C-H stretching	2975.14	2975.14	2973.21
C-O-C stretching	1048.12	1049.09	1048.6
C-H bending	879.381	879.86	879.86



(a) (b) (c)

FIGURE 5. UV-Vis's absorption natural herbal dyes after photodegradation

TABLE 3. Absorption value of each pigment during photodegradation in *Fagraea acuminatissima*

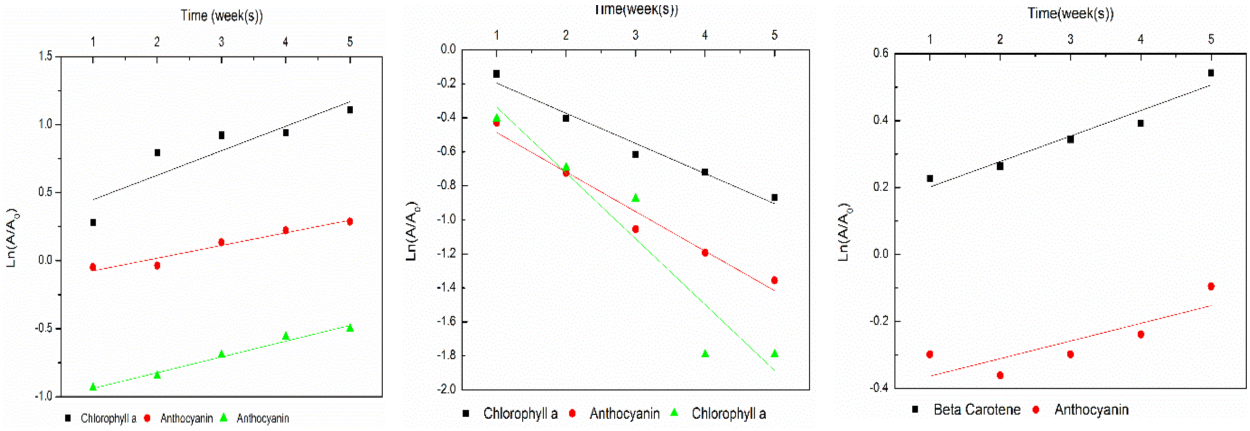
	Pigments absorption value		
	Chlorophyll a (390-420 nm)	Anthocyanin (470-550 nm)	Anthocyanin (500-550 nm)
W_0	0.84	0.84	0.28
W_1	1.11	0.8	0.11
W_2	1.86	0.81	0.12
W_3	2.11	0.96	0.14
W_4	2.15	1.05	0.16
W_5	2.54	1.12	0.17

TABLE 4. Absorption value of each pigment during photodegradation in sappanwood

	Pigments absorption value		
	Chlorophyll a (440-460 nm)	Anthocyanin (500-550 nm)	Chlorophyll a (650-670 nm)
W_0	2.65	0.66	0.12
W_1	2.3	0.43	0.08
W_2	1.77	0.32	0.06
W_3	1.43	0.23	0.05
W_4	1.29	0.2	0.02
W_5	1.11	0.17	0.02

TABLE 5. Absorption value of each pigment during photodegradation in kulit setong

	Pigments absorption value	
	Beta carotene (460-500 nm)	Anthocyanin (550-580 nm)
W_0	2.12	0.66
W_1	2.66	0.49
W_2	2.76	0.46
W_3	2.99	0.49
W_4	3.14	0.52
W_5	3.65	0.6

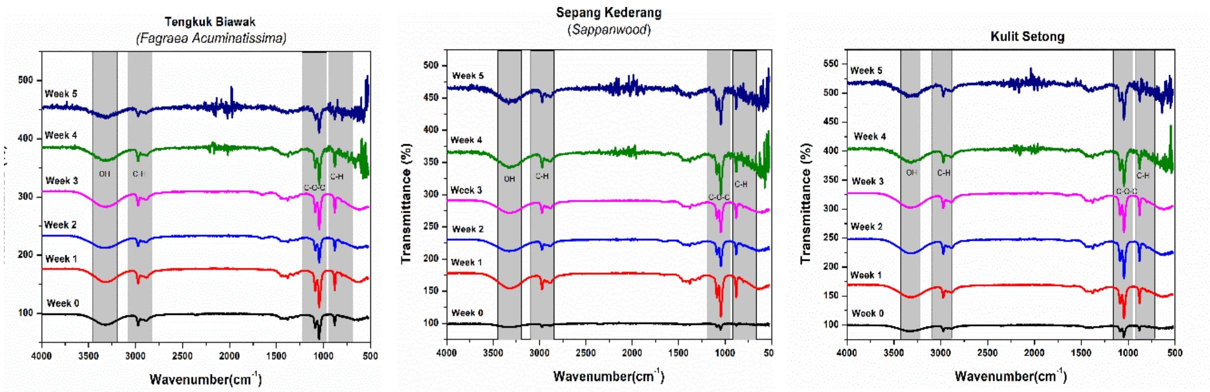


(a) (b) (c)

FIGURE 6. First order pigment degradation in natural herbal dye by artificial light irradiation for (a) *Fagraea acuminatissima*, (b) sappanwood, and (c) kulit setong

TABLE 6. Functional groups and wavenumber of peaks in natural herbal dye at the end of week five after photodegradation

Functional group	Wavenumber (cm^{-1})					
	Fagraea acuminatissima		Sappanwood		kulit setong	
	W_0	W_1	W_1	W_3	W_4	W_5
O-H stretching	3317.93	3320.34	3332.87	3329.98	3327.57	3330.46
C-H stretching	2975.14	2972.25	2975.14	2971.77	2973.21	2975.63
C-O-C stretching	1048.12	1046.68	1049.09	1044.75	1048.6	1048.12
C-H bending	879.381	876	879.86	876.49	879.86	879.86



(a) (b) (c)

FIGURE 7. FTIR spectra of natural herbal dyes after photodegradation

from the first week to the fifth week. The absorption value of beta-carotene increased from 2.12 before photodegradation to 3.65 in the fifth week of photodegradation. From the UV-Vis absorption measurement results, it is known that there is a decrease in the absorption value of anthocyanins in the three natural herbal dyes. In Fagraea acuminatissima, the absorption value of chlorophyll with its wavelength absorption in the range of 390-420 nm increased. In contrast, sappanwood, where the chlorophyll is in the wavelength range of 440-460 nm and 650-670 nm, shows an absorption value that tends to decrease. During irradiation, the absorption value for beta-carotene in the wavelength range 460-500 nm in kulit setong increased. The first order kinetic pigment degradation in natural herbal dye from Equation (2) given in Figure 6.

Figure 6 shows a clear trend of changes in the absorption values of pigments in natural herbal dyes after being irradiated with artificial light. The pseudo rate constant values for all pigments in *Fagraea acuminatissima* and kulit setong are negative (the slope of the linear equation is positive), indicating that the absorption value rate of change tends to increase with irradiation time. The pseudo rate constant value for chlorophyll in *Fagraea acuminatissima* is at -0.18 week^{-1}, and beta-carotene in kulit setong, given the pseudo rate constant value at -0.08 week^{-1}. In contrast to what happened with sappanwood, the apparent rate constants of all pigments are positive (the slope of the linear equation is negative), indicating that the rate of change of absorption value decreases with irradiation time. From the slope of the linear equation for sappanwood, chlorophyll at a wavelength of 650-670 nm has the most significant pseudo rate constant at 0.39 week^{-1}, so it looks like having a rapid decrease compared to pigments at other wavelengths.

Changes in functional groups were observed every week to see the response of the natural herbal dye during artificial light irradiation (Table 6). The functional groups that were visible before photodegradation remained visible during observation from the first to the fifth week. The wavenumber of each functional group did not change significantly. The C-H stretching functional group in Fagraea acuminatissima (Figure 7(a)) has the highest increase in transmittance value compared to the other groups. In the first week of photodegradation, the transmittance ratio of the functional groups is similar to the initial one, where the transmittance of the O-H stretching is highest, and the C-O-C stretching is lowest. In the second week, the O-H stretching changes to C-H stretching. From the second week to the fifth week, the functional group transmittance ratio remains unchanged, where the C-H stretching functional group has the highest transmittance and the C-O-C stretching is the lowest. The C-H bending functional group in sappanwood (Figure 7(b)) has the highest increase in transmittance value compared to the other groups. The C-H bending changes to C-H stretching in the first week of photodegradation. From the second week till the fifth week, the functional group transmittance ratio remains unchanged, where the C-H stretching functional group has the highest transmittance and the C-O-C stretching is the lowest. The changes that occur in the FTIR spectrum of kulit setong (Figure 7(c)) are slightly different. Before photodegradation, the transmittance value of O-H stretching was highest, with C-O-C stretching being the lowest. The first week of photodegradation showed an increase in the transmittance value of C-H stretching, and C-O-C stretching remained the lowest. Changes that occurred in the second week the transmittance value of O-H stretching again became the highest, with C-O-C stretching remaining the lowest. The transmittance ratio of all functional groups remained unchanged from the previous week. While in weeks four and five, the C-H stretching group has the highest transmittance value, and C-O-C stretching remains the lowest. In conclusion, the transmittance value of the relative functional groups increased every week from the beginning of photodegradation until the fifth week for all three natural herbal dyes with almost the similar change in transmittance ratio.

CONCLUSION

Three natural herbal dyes Fagraea acuminatissima, sappanwood and kulit setong dissolved in ethanol were photodegraded at 35 °C under a light power of 30 W/m2. The results obtained showed an increase in chlorophyll in the absorption wavelength range of 400-424 nm and beta-carotene in the absorption wavelength range of 460-500 nm. Of the three natural herbal dyes, the ones that have the potential to be used in solar cell applications with natural dyes are Fagraea acuminatissima with chlorophyll and kulit setong with beta-carotene.

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