Al-Kimia, 13-1-2025, 23-34
Available online at: http://journal.uin-alauddin.ac.id/index.php/al-kimia

Characterization of Sappanwood Dye (*Caesalpinia sappan* L.) from Choloroform: Methanol Fraction as Photosensitizer in Dye Sensitized Solar Cell Using UV-Vis and FTIR

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Received: February, 12, 2025/Accepted: June, 21, 2025
doi:10.24252/al-kimia.v13i1.55418

Abstract: *The ongoing electricity crisis in Indonesia, due to the depletion of fossil fuels, necessitates the development of alternative energy sources such as Dye-Sensitized Solar Cells (DSSCs). This study explores the potential of secang wood (Caesalpinia sappan L.) extract as a natural photosensitizer in DSSCs. The extract was obtained using a chloroform: methanol solvent system and tested at pH 4, 7, and 10 to determine optimal performance. The highest efficiency (0.05497%) was recorded at pH 7. Subsequent purification using Vacuum Liquid Chromatography (VLC) with solvent ratios of 9:1, 5:5, and 1:9 resulted in markedly lower efficiencies of 0.00213% and 0.00383%. UV-Vis analysis characterization revealed 243, 285, 445, and 541.4 nm absorption peaks. GC-MS confirmed the presence of brazilin (m/z 286). At the same time, FTIR identified functional groups including –OH, C–H, C=C, C–O–C, and aromatic rings, which are crucial for light harvesting and electron transfer. The reduced efficiency after purification suggests that the synergistic effects of crude extract components enhance DSSC performance. Therefore, pH adjustment is more impactful than purification in optimizing the extract, highlighting neutral pH sappan wood extract as a promising natural photosensitizer for DSSC applications.*

Keywords: *Characterizations, Dye Sensitized Solar Cell, Sappanwood.*

INTRODUCTION

The electricity crisis in Indonesia has recently become a primary focus, especially with the increasing frequency of rolling blackouts in several regions, such as Kalimantan and Sulawesi. This phenomenon indicates an imbalance between energy supply and demand. The decline in fossil fuel reserves, which have been the main source of power generation, is the primary cause of this crisis. At the same time, the demand for electrical energy continues to increase year after year. Based on data from Statistics Indonesia (2024), electricity distribution in South Sulawesi increased from 6,597.87 GWh in 2021 to 7,856.64 GWh in 2022. This increase reflects a significant growth in electricity demand.

As a tropical country on the equator, Indonesia has great potential in utilizing renewable energy, including solar energy. Solar energy is estimated to produce energy of approximately $\pm 3 \times 10^{24}$ J per year (Grätzel, 2001), which can be converted into electricity through photovoltaic devices such as solar cells. However, using silicon (Si) as the base material for conventional solar cells has various challenges, such as high production costs,

complexity in the fabrication process, and environmental impact. Therefore, developing alternative solar cell technology becomes important to address these challenges.

One of the promising solar cell technologies is the Dye Sensitized Solar Cell (DSSC). This technology is developed with a working principle resembling natural photosynthesis, where the dye acts as a light absorber and electron producer, which is then transferred to the semiconductor electrode. DSSC has advantages such as lower production costs, a simple fabrication process, and the use of more environmentally friendly raw materials (Nasyori et al., 2020). The dye in DSSC can be derived from natural materials such as flowers, leaves, fruits, and even wood, which are extracted using specific solvents. Using natural dyes as sensitizers offers several advantages, including being biodegradable, easily obtainable, and containing active compounds such as flavonoids and anthocyanins that can effectively absorb visible light (Mejica et al., 2022). However, natural dyes also have limitations, such as low photochemical stability, susceptibility to degradation by UV light and oxygen, and variability in the composition of active compounds influenced by plant type, season, and extraction method (Kabir et al., 2022). One promising example of a natural dye is sappanwood extract (*Caesalpinia sappan* L.), which has been extensively researched due to its active compound content that can support the energy photoconversion process in DSSC.

Using sappanwood extract as a photosensitizer in DSSC has shown great potential because it contains active compounds such as brazilin and hematoxylin that function as chromophores and electron donors. Research by Simanjuntak et al. (2022) noted that using an ethanolic extract of secang wood produced DSSC efficiency of up to 0.196629%. However, this efficiency is still hindered by stability and spectral absorption range limitations, which pose challenges in further development towards commercial applications (Nasyori et al., 2020). Furthermore, based on the existing literature, the type of solvent indeed plays a significant role in the light absorption capacity of natural dye extracts used in DSSC. Research shows that solvents with polar properties increase the solubility of dye compounds, thereby improving the interaction between the active compound molecules and the TiO_2 surface. This can enhance the adsorption of dye molecules on the semiconductor, thereby increasing light absorption capacity (Boyo, 2020). However, despite the relatively higher light absorption intensity, the energy conversion efficiency of the resulting DSSC remains relatively low. The low efficiency factor is partly influenced by the fragility of the complex structure of natural dye compounds, which makes the electron injection mechanism and charge recombination less optimal (Yelkovan et al., 2025).

The strategy to improve the performance of DSSC is to separate the fractions of active compounds so that the compounds important for electron transfer can be enriched, and other interfering components can be removed. This fractionation helps improve electron transfer efficiency from the dye to the TiO_2 layer. It reduces the recombination rate between the injected electrons and the remaining holes, enhancing the cell's stability and performance (Gu et al., 2020). In practice, fraction separation through chromatography techniques or other methods allows for selecting components with a high affinity for the semiconductor surface and better light absorption capabilities, contributing to the overall performance improvement of DSSC.

Overall, the effectiveness of natural dye extraction using polar solvents is reflected in the increased light absorption. In this context, methanol is the solvent of choice due to its polar nature and its ability to extract flavonoid compounds, which are also polar (Zreen et al.,

2022). Additionally, the low boiling point of methanol (64.7 °C) facilitates the evaporation process without damaging the active compounds, and it can also enhance the yield and stability of the extract compared to non-polar solvents like hexane. However, other factors such as the composition of the compound mixture and the interactions between different fractions still affect the efficiency of electron transfer. Therefore, the separation of active compound fractions is a promising approach to overcoming the limitations of energy efficiency in DSSC, as this fractionation allows for the improvement of electron transfer pathways and the long-term stability of the device (Boyo, 2020).

Based on that background, this research aims to characterize the fraction of secang wood extract produced from a combination of chloroform: methanol solvents and evaluate its efficiency as a natural photosensitizer in DSSC. The combination of chloroform (a non-polar solvent) and methanol (a polar solvent) is widely used in phytochemical extraction because it separates a wide range of compounds with varying polarities. Chloroform effectively dissolves non-polar compounds such as lipids and some pigments. At the same time, methanol extracts polar compounds such as flavonoids and phenolic acids, which are known to play a key role in light absorption and electron transfer in DSSC applications. This approach is expected to provide information and contributions related to the optimizing of solvent fractions towards the more stable and efficient photovoltaic activity of natural materials.

RESEARCH METHODS

Materials and Tools

The tools used in this study were Scanning Electron Microscopy (SEM) Tescan Vega3SB, Fourier Transform InfraRed (FTIR) Shimadzu IRPrestige-21, Gas Chromatography-Mass Spectroscopy (GC-MS) Thermo, Ultraviolet-Visible Spectrophotometer (UV-Vis) Shimadzu UV-2600 Series, evaporator, ultrasonic, digital multimeter, oven, balance, hot plate, vacuum pump, TCO glass resistance 100 Ω/sq, potentiometer and glassware.

The materials used in this study were sappanwood (*Caesalpinia sappan* L.), titanium dioxide (TiO_2) food grade, aluminum foil, aquades (H_2O), ethanol (C_2H_5OH) 96%, ice cubes, iodine (I_2), potassium iodide (KI) p.a, carbon, filter paper, chloroform ($CHCl_3$) Merch, methanol (CH_3OH) Merch and silica purchased from CV Intraco.

Methods

Dye Extraction from Sappanwood

The dye extract was prepared by modifying the method of Zulenda et al., (2019). The sappanwood samples were cut into pieces and dried at room temperature. The small-sized samples were weighed as much as 500 grams and then put into a macerate container then methanol solvent (CH_3OH) was added thrice for 1 x 24 hours by changing the solvent every 1 x 24 hours. The sappanwood extract that had been obtained was put into an evaporator flask to be evaporated, to obtain a thick extract. The thick extract was dissolved with its solvent, and then the pH varied (pH 4, 7, and 10). Then, it was tested with DSSC to determine its efficiency. The dye from the pH variation that had the highest efficiency was continued in the VLC process.

Vacuum Liquid Chromatography (VLC)

The preparation of the anode was carried out by modifying the method of Mutmainnah et al., (2017). Silica 7730 was weighed at 20 grams and then packed into a chromatography column, then flowed with n-hexane solvent until there were no air bubbles in the silica. The sappanwood extract that had the highest efficiency was impregnated with 3 grams of silica 7733. After that, the impregnation results were put into the chromatography column and then covered with paper according to the column diameter. Then, the column was eluted with an eluent of increasing polarity, namely chloroform: methanol (9:1, 5:5, and 1:9) volume 50 mL. The resulting fractions were collected into separate containers according to the eluent used. The fractions produced are each tested for efficiency; the fraction with the highest efficiency will be tested on the UV-Vis instrument at wavelengths of 200-800 nm, GC-MS, and FTIR transmission method.

DSSC

Preparation of Electrolyte Solutions

Potassium iodide (KI) was weighed as much as 0.9306 grams, then put into a container containing 10 mL of distilled water and stirred until dissolved. Iodine (I_2) was added as much as 0.1270 grams to the mixture and stirred until homogeneous (Wahidah Febriya Ramadhani, Aisyah A, Suriani S, 2019).

Preparation of TiO_2

TiO_2 was mixed with distilled water and heated to a boil on a hot plate. After cooling, TiO_2 was filtered to separate it from the distilled water, then transferred to a container, and ethanol (C_2H_5OH) was added, then stirred until homogeneous (Wahidah Febriya Ramadhani, Aisyah A, Suriani S, 2019).

Preparation of Counter Electrode

TCO glass is washed using an ultrasonic tool, then dried with tissue. After drying, the conductive side of the glass is heated with a candle flame until a black color forms (Wahidah Febriya Ramadhani, Aisyah A, Suriani S, 2019)

DSSC Circuits

TiO_2 paste is spread on the TCO glass using a spatula or capillary pipette, then the TCO glass is soaked in tomato color extract and attached to a reference electrode. Electrolyte solution is dripped onto both electrodes, and the DSSC circuit is measured for current and voltage using a multimeter. The intensity of sunlight is measured using a luxmeter (Wahidah Febriya Ramadhani, Aisyah A, Suriani S, 2019).

Characterization of Sappanwood

The results of the purification of sappanwood extract were characterized using Ultra Violet-Visible (UV-Vis), Fourier Transform Infrared (FTIR), and Gas Chromatography-Mass Spectroscopy (GC-MS).

RESULTS AND DISCUSSION

Efficiency of DSSC

The DSSC testing of sappanwood extract was conducted to determine the efficiency based on pH variations.

Table 1. Results of DSSC Testing at Various pH Levels of Sappanwood Extract

No	Sample	pH	P_{in} (mWatt/cm^2)	P_{out} (mWatt/cm^2)	η (%)
1	Sappanwood	4	12.021082	0.0020604	0.01714
2	Sappanwood	7	12.035724	0.0066164	0.05497

3	Sappanwood	10	12.108934	0.0038437	0.03174

Based on the data in Table 1, which shows the performance test results of DSSC using sappanwood extract at various pH levels (4, 7, and 10), it can be seen that the highest efficiency level is achieved at neutral pH (pH 7), which is 0.05497%. This efficiency value correlates with the highest output power (P_{out}) value, which is 0.0066164 mWatt/cm², compared to pH 4 (0.0020604 mWatt/cm²) and pH 10 (0.0038437 mWatt/cm²). In contrast to the research conducted by Ekpunobi et al (2022), which showed the highest efficiency at pH 3 using Alstonia boonei extract, similar results were also found in Abidin et al (2020), where the highest output voltage and current were obtained at pH 1,5. Meanwhile, different results were shown by Mejica et al (2022), who used malabar spinach (Basella alba) extract, with the highest efficiency achieved at pH 9.

This phenomenon supports that neutral pH is optimal for stabilizing active compounds such as brazilin in sappanwood extract. At neutral pH, the chemical structure of the dye compound is more stable and remains active in absorbing light and efficiently transferring electrons to the TiO_2 semiconductor. The value of P_{in} (input power) is relatively constant under the three conditions, so the difference in efficiency is primarily influenced by how effectively the dye converts light into electricity (through P_{out}).

The significantly increased efficiency value from 0.01714% (pH 4) to 0.05497% (pH 7) indicates that acidic pH conditions can cause degradation or protonation of phenolic groups, reducing the interaction of the dye with the TiO_2 surface. On the other hand, at basic pH (pH 10), although its efficiency is higher than at pH 4, it is still lower than at pH 7. This could be due to the possibility of excessive deprotonation of the –OH group, which can disrupt efficient charge transfer and the stability of the dye's molecular structure. Based on the data, the highest efficiency value was obtained from the sappanwood extract at pH 7, which was 0.05497%. This sample was further tested using VLC.

Table 2. Results of DSSC Testing After VLC

No	Solvent Comparison (Dye)	P_{in} (mWatt/cm²)	P_{out} (mWatt/cm²)	η (%)
1	Cloroform:Methanol 9:1	-	-	-
2	Cloroform:Methanol 5:5	12.328564	0.0002622	0.00213
3	Cloroform:Methanol 1:9	12.22607	0.0004687	0.00383

Based on Table 2, after the sapanwood extract with an optimum pH (pH 7) that has the highest efficiency (0.05497%) underwent a purification process using VLC, it was observed that the DSSC efficiency experienced a significant decrease. Two fractions resulting from the separation of the chloroform: methanol solvent with ratios of 5:5 and 1:9 showed much lower efficiencies than the crude extract, namely 0.00213% and 0.00383%, respectively. Meanwhile, the 9:1 fraction showed no efficiency, possibly due to the absence of significant photovoltaic activity or the active dye compounds not being present in that fraction.

This decrease in efficiency indicates that the separation process through VLC can lead to the loss of synergy between active compounds previously present in the crude extract. These results support the understanding that the crude extract at pH 7 provides the best performance because it maintains the natural chemical complexity of the material, which includes a combination of hydroxyl, aromatic, and ether groups that synergistically absorb

light and bind to the TiO_2 surface. Therefore, although purification with VLC aims to increase the purity of the active compounds, in the context of natural DSSC, this could have the opposite effect on the overall performance of the solar cell.

Scanning Electron Microscope (SEM)

SEM analysis was conducted to observe the morphology of TiO_2 modified with dye. The interaction between TiO_2 and the compounds occurs due to forming bonds with reactive groups, such as hydroxyl and carbonyl groups, which can form chelates, as described in cyanine compounds (Smestad & Grätzel, 1998).). In the brazilin compound, hydroxyl groups are present, while brazilein contains hydroxyl and carbonyl groups, enabling bond formation with TiO_2. This interaction also involves forming hydrogen bonds between the dye and TiO_2.

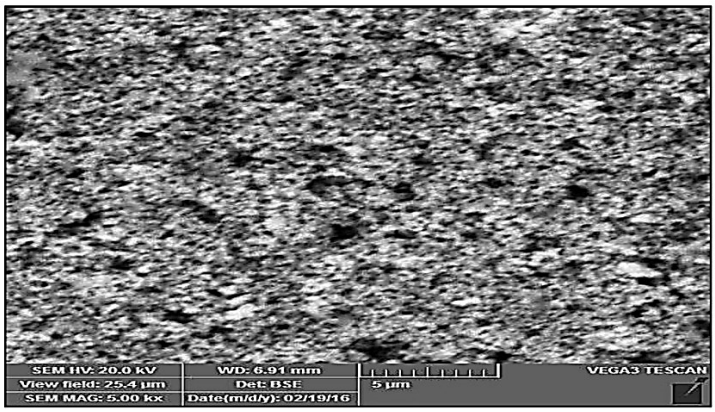

Figure 1. The morphology of TiO_2 modified with dye

According to several studies, this interaction also helps to expand the light absorption spectrum by DSSC because natural dyes such as brazilin and brazilein can absorb light over a wide wavelength range. This is important for improving energy conversion efficiency from light to electricity (Robledo et al., 2023). In addition, the surface morphology shown by the SEM images supports optimal dye absorption and efficient electron transfer from the dye to the TiO_2 semiconductor (Ferreira et al., 2018).

Ultraviolet Visible (UV-Vis)

UV-Vis testing was conducted at a wavelength range of 200–800 nm to determine the maximum wavelength absorbed by the fractionated sappanwood extract. This provides an initial overview of the compound content in the fractionated sappanwood extract.

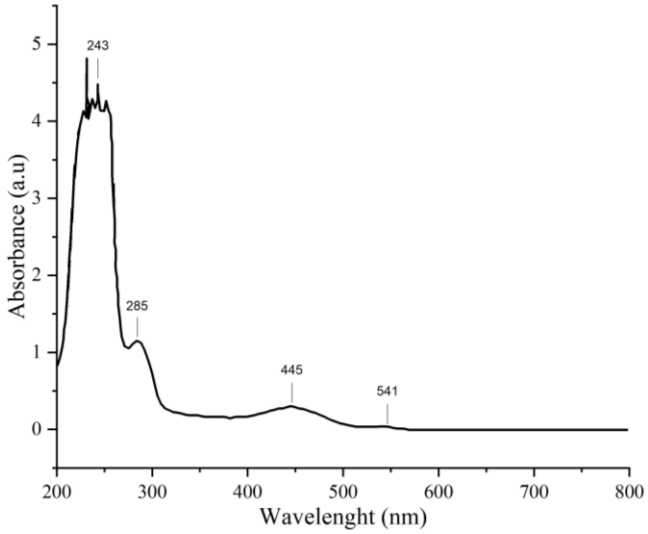

Figure 2. UV-Vis Spectrum of Sappanwood Extract After VLC with Chloroform:Methanol (1:9)

Table 3. UV-Vis Instrument Data Results

Dye	Wavelength (λ)	Absorbance
Cloroform: Methanol 1:9	243.00	4.605
	285.00	1.166
	445.00	0.295
	541.50	0.044

Natural dyes such as sappanwood extract fraction chloroform: methanol, which contains the active compounds brazilin and brazilein, exhibit broad absorption spectral characteristics in the ultraviolet (UV) and visible regions. Based on the UV-Vis spectrum data, the highest absorption peaks were observed at wavelengths of 541.50 nm, 445 nm, 285 nm, and 243 nm. The wavelengths of 285 nm and 243 nm lie within the UV region and are associated with the absorption of phenolic compounds, consistent with the general characteristics of molecules containing hydroxyl groups (Lioe et al., 2012). Meanwhile, the wavelengths of 445 nm and 541.5 nm in the visible region are caused by $\pi \rightarrow \pi^*$ excitation occurring in conjugated double bonds and $n \rightarrow \pi^*$ excitation from free electron pairs in hydroxyl and carbonyl groups (Furniss et al., 1989). The solution's pH changes can affect the active compound's chemical form. Vitorino (2012) n oted that the extract of secang wood showed a maximum peak at 446 nm in acidic conditions and 540 nm in basic conditions. This indicates the presence of different internal electron tunneling depending on the environmental conditions. In the study by Padmaningrum et al (2012), the ethanol extract at pH 6.3 showed two significant absorbance peaks at 537 nm and 445 nm, reinforcing the evidence that this dye is effective in absorbing energy in the range relevant for light energy conversion.

The broad absorption spectrum and the presence of π-electrons in the aromatic structure of compounds like brazilin make secang wood an ideal candidate for a photosensitizer in DSSC (Malashi et al., 2024). The conjugated π-system enhances the molecule's ability to absorb a wide range of visible light, facilitating efficient solar energy capture and electron excitation. This characteristic is crucial because the delocalized π-electrons can easily transition from the HOMO to the LUMO and subsequently inject into the conduction band of TiO_2, initiating the photovoltaic process (Prakash & Janarthanan, 2023).

According to Guo et al (2023) Delocalized electrons in aromatic rings, such as in the structure of brazilin benzene, can undergo resonance and transfer to the conduction band of semiconductors (such as TiO_2), creating an electric current. In other words, the excited electrons can move from the HOMO to the LUMO, and then to the semiconductor, which is a key step in the working cycle of DSSC.

Moreover, the presence of polar functional groups such as hydroxyl (-OH) is also significant, as they facilitate binding with the TiO_2 surface through hydrogen bonding or chelation, enhancing the dye's adsorption and improving charge transfer efficiency (Akinsola et al., 2021). The stronger the anchoring between the dye and semiconductor, the better the charge injection and overall cell performance. Natural dyes rich in flavonoids, tannins, and anthocyanins, such as those found in sappanwood, demonstrate these critical properties, leading to improved DSSC efficiencies (Nasyori et al., 2020).

Therefore, the spectral characteristics and chemical structure of natural dyes, such as those from sappanwood, are crucial in determining the energy conversion efficiency in DSSC. Their ability to absorb across a wide range of light spectra and form strong bonds with the semiconductor surface allows for more optimal utilization of sunlight, thereby enhancing the performance and stability of dye-based photovoltaic devices.

Fourier Transform InfraRed (FTIR)

FTIR testing was conducted to identify the functional groups present in the fractionated sappanwood extract fraction, chloroform: methanol. The data obtained complements the interpretation of the UV-Vis data. The chloroform: methanol fraction of the sappan wood extract shows increasingly promising potential as a natural dye in DSSC applications, particularly due to its ability to more selectively extract active compounds such as brazilin (Arsyad et al., 2024). The FTIR spectrum of this extract shows a characteristic peak around 3415 cm^{-1}, indicating the presence of hydroxyl (–OH) groups from phenolic compounds, which are important in forming bonds with the TiO_2 surface and enhancing adsorption stability (Surana et al., 2021).

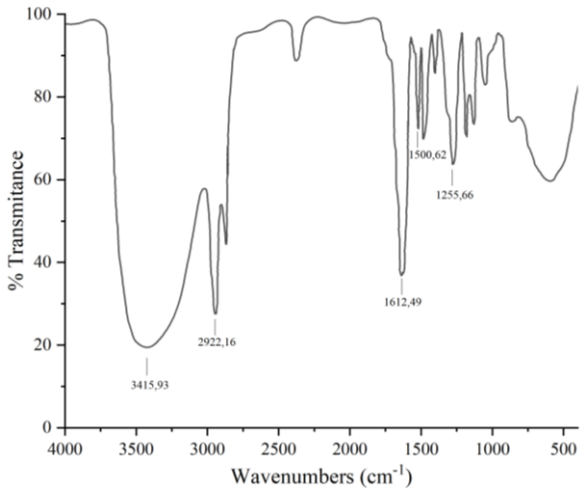

Figure 3. IR Spectrum of Sappanwood Extract

Extraction with a chloroform: methanol mixed solvent also revealed the presence of C=C and C–O–C groups through strong absorption in the band around 1612–1255 cm^{-1},

indicating the presence of aromatic and cyclic ether structures of isoflavonoid compounds such as brazilin. This is very important because the structure allows for a wide light absorbance, including in the visible and near-infrared spectrum (Nasyori et al., 2020).

Table 4. FTIR Interpretation Data Results

Dye	Absorption (cm^{-1})	IR Absorption Database (cm^{-1})	Interpretation
Chloroform: Methanol 1:9	3419.93	3200 – 3600	O-H
	2922.16	2850 – 2950	C-H (saturated)
	1612.49	1600 – 1700	C=C
	1500.62, 1460.11	1400 – 1500	C-C (aromatic ring)
	1255.66	1050 – 1270	C-O-C

FTIR spectra from this extract also exhibit broad O–H stretching vibrations near 3419.93 cm^{-1}, confirming the presence of hydroxyl groups. These hydroxyl groups play a dual role: they enhance solubility and provide anchoring sites for chemical bonding with the TiO_2 surface, forming strong hydrogen or coordination bonds that facilitate efficient electron transfer from the dye to the semiconductor (Supriyanto et al., 2021) This interaction reduces charge recombination and enhances the photoelectric conversion process.

Furthermore, the separation using semi-polar fractions such as chloroform: methanol yields fractions with higher concentrations of brazilin, resulting in a more intense and stable red-orange color, crucial factors for photon harvesting efficiency in DSSC (Surana et al., 2021). The use of this fraction in DSSC shows an increase in photoelectric efficiency compared to the crude extract, as evidenced by the performance testing of TiO^{2-} based DSSC doped with brazilin dye from this fraction (Arsyad et al., 2024). This further strengthens the argument that the chloroform: methanol fraction of the secang wood extract is one of the candidates for an environmentally friendly and efficient natural dye in the next generation DSSC.

Gas Chromatography – Mass Spectroscopy (GC-MS)

The analysis using GC-MS instrumentation strengthened the identification of compounds in sappanwood by examining the molecular weight detected in the MS of GC cuts. The instrument used a 50 m column with helium gas as the carrier, and the analysis was conducted for 22:34 minutes, producing several peaks on the chromatogram. At a retention time of 9.54 minutes, a peak with m/z = 286 was detected, which corresponds to the molecular weight of the brazilin compound.

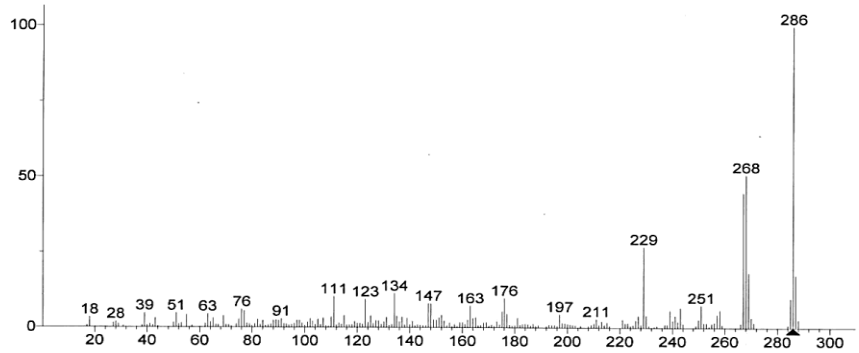

Figure 4. Molecular Fragmentation Pattern of Sappanwood Extract Chloroform:Methanol (1:9) Fraction at Retention Time 9.54

The molecular ion at m/z 286 originates from the $C_{16}H_{14}O_5^+$ cation as the base peak. The loss of the OH group results in a fragment with m/z 268 (M-18), followed by the release of the $C_3H_3^{\cdot+}$ radical, forming m/z 229. A fragment at m/z 163 is produced by the loss of a CHO^+ molecule, leading to the formation of the molecular ion at m/z 134 (Figure 4).

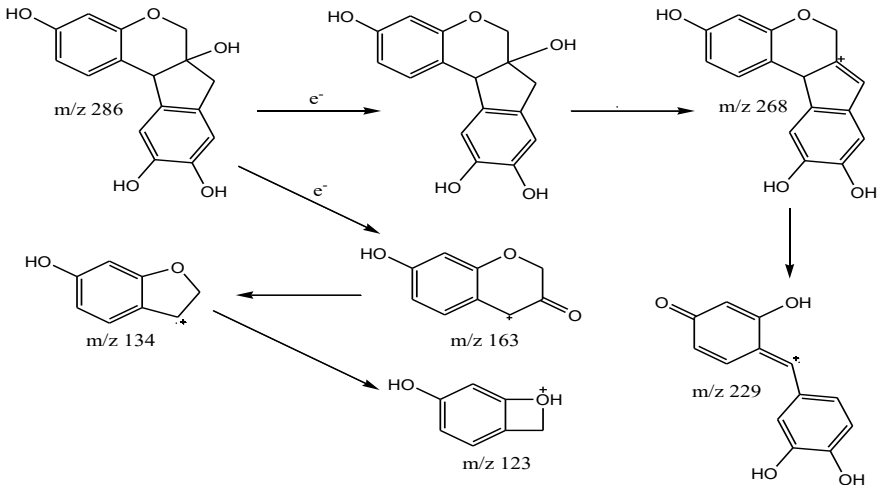

Figure 5. Fragmentation of m/z 286

The fragmentation pattern obtained in this study shows similarities to the brazilin fragmentation pattern reported by the Editorial Committee of the Chinese Pharmacopoeia (2010), which includes fragments at m/z 285, 267, 229, 163, 135, and 121.

CONCLUSIONS

Sappan wood extract at neutral pH (pH 7) showed the highest efficiency in DSSC of 0.05497% due to the optimal stability of active compounds such as brazilin and brazilein. After purification using VLC with chloroform: methanol solvents (5:5 and 1:9 ratios), the efficiency decreased drastically to 0.00213% and 0.00383%, indicating that compound separation reduced the synergy of active components in the crude extract. UV-Vis, FTIR, and GC-MS analyses confirmed the presence of active groups -OH, C=C, and aromatic rings, as well as the identification of brazilin (m/z 286), supporting light absorption and electron transfer to TiO_2. The best results were still obtained from the pH 7 crude extract as it retained the natural complexity of the active compounds. Optimization of extraction conditions, especially pH, has more influence on DSSC efficiency than purification. These findings open up opportunities for development by combining of crude extracts and selected fractions and compound stabilization techniques such as encapsulation. However, this study is limited to one separation method and does not include long-term stability tests against sunlight or real environmental conditions.

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