TiO$_2$-Ag AND NATURAL DYE CO-PIGMENTED WITH SALICYLIC ACID FOR DYE-SENSITIZED SOLAR CELL (DSSC) APPLICATION

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ABSTRACT
The Dye-Sensitized Solar Cell (DSSC) is still inefficient. Because low efficiency is caused by electron recombination, this study reduced it by the electrodeposition of Ag metal against TiO$_2$. XRD was used to determine the crystal structure, size, and effect of Ag electrodeposition on TiO$_2$. SEM-EDS was used to investigate the surface morphology and the presence of Ag metal in the TiO$_2$-Ag layer. Anthocyanins were used as dyes. To improve thermal stability and achieve a bathochromic effect, the anthocyanin used was co-pigmented with salicylic acid. The anthocyanin extract used in this study was obtained from the red grape peel (Vitis Vinifera), jengkol peel (Pithecellobium jiringa), senduduk fruit (Melastoma malabathricum L), and mangosteen peel (Garcinia Mangostana L). The dyes were identified using UV-Vis and FTIR. The electrical properties of the resulting DSSC were measured using a multi-meter. According to this study, the electrodeposition of Ag on the TiO$_2$ layer could improve DSSC efficiency. The highest efficiency was achieved in DSSC with TiO$_2$-Ag and co-pigmented dye using mangosteen peel as the dye source.

Keywords: Ag Electroplating, Co-pigmentation, DSSC, Natural Dye.

INTRODUCTION

Energy is extremely important in human life.$^1$ At the moment, the supply of energy resources relies on fossil fuel sources such as oil and gas. Due to a decrease in these energy sources, humans are looking for alternative sources in a competitive manner. Wind power, hydropower, and biomass are alternative renewable energies, but solar cell energy is the most promising source.$^{1,2,3,4}$ Since Indonesia is located on the equator, the use of solar power becomes the most potential solution. Compared to other areas of the Earth's surface, Indonesia receives significantly more radiation. In Indonesia, the total radiation intensity per day exceeds 4500 watt-hours/m$^2$. More than Japan, which only receives 150-180 watt-hours/m$^2$/day in total radiation intensity. Furthermore, due to its equatorial location, Indonesia receives up to 2,000 hours of sunlight per year.$^5,^6$ If the silicon solar cell is compared with the DSSC, the DSSC efficiency remains lower. However, silicon solar cells' manufacturing and assembly processes are more complicated and less environmentally friendly, whereas DSSC is a low-cost energy converter.$^{7,8,9}$ Furthermore, DSSC has a versatile method of converting the most abundant clean energy resources.$^{10}$

The photoelectrode is an important key factor in DSSC since it is responsible for dye molecule adsorption and electron transport.$^{11}$ Photon absorption in sunlight is carried out by dye molecules, which causes electron excitation. The excited electron will be injected into a wide bandgap inorganic semiconductor nanocrystalline, and Titanium Dioxide (TiO$_2$) is a well-known example.$^3$ Titanium dioxide (TiO$_2$) is used for solar cell development based on photoinduction phenomena. TiO$_2$ will absorb photon energy in the phenomena of photoinduction, causing the valence band to be void due to an electron transition to the conduction band. A gap arising in the valence band between electrons in the conduction band can be electrically used for the photovoltaic cell. According to that, TiO2 nanoparticles can be used as dye-based photovoltaic cells.$^{12}$ Furthermore, the cost of TiO2 is relatively low, and it has
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no negative effects on living things or the environment. The issue is that the absorption capacity is still low.

The low efficiency in DSSC using TiO$_2$ is due to the recombination of the injected electron and the redox pair in the electrolyte.\textsuperscript{13} Metal electrodeposition was used in this study to reduce electron recombination and improve DSSC performance. Metal electrodeposition is a technique for creating a barrier layer between a dye or electrolyte and TiO$_2$ to reduce the electron recombination process caused by the distinct interband gap.\textsuperscript{14,15} The metal used in this experiment is silver (Ag). Because of Ag's high conductivity, electrons were transported quickly, causing less electron recombination.\textsuperscript{16} In addition, Ag can boost photocurrent by acting as a scattering center, reflecting incident light.\textsuperscript{17}

At this time, the semiconductor TiO$_2$ thin layer can only absorb 5% of sunlight in the U.V. spectrum (200 nm–400 nm), so the use of dye is critical.\textsuperscript{18} Harvesting sunlight into the DSSC electrical circuits is important in increasing efficiency.\textsuperscript{19,20} To maximize solar power conversion, the dye used in DSSC must absorb well in the visible light range.\textsuperscript{12} Anthocyanins are flavonoid derivatives with red and purple colors, and they are a promising natural dye for use as a sensitizer in DSSC.\textsuperscript{21} The use of anthocyanin as a dye may increase the absorption area, decreasing efficiency.\textsuperscript{22} Anthocyanin, on the other hand, is a natural dye that is easily oxidized or damaged, but anthocyanin has been able to form a more complex compound when interacting with other organic compounds through the co-pigmentation process.\textsuperscript{23} This process is performed by combining anthocyanin with other molecules that increase their stability.\textsuperscript{24} Co-pigmentation with salicylic acid would generate an anthocyanin with better thermal stability and a bathochromic effect. As a result of this effect, molecular absorption shifted to a longer wavelength. The co-pigmentation process not only increases the heat stability of anthocyanins but also increases the activation energy and half-life of anthocyanins.\textsuperscript{25} When used as a dye in DSSC, the better formed co-pigment structure allows anthocyanin to have better stability, durability, and light absorption.

Each device component influences its performance by performing its respective tasks, so optimizing the device components is important.\textsuperscript{26} The purpose of this study was to determine the effect of Ag electrodeposition on TiO$_2$ to DSSC performance using co-pigmented anthocyanin from various sources. The dye was produced by co-pigmenting anthocyanin from jengkol peel (\textit{Pithecellobium jiringa} B.), senduduk fruit (\textit{Melastoma malabathricum} L.), mangosteen peel (\textit{Garcinia mangostana} L.), and red grape peel (\textit{Vitis vinifera}) with salicylic acid.

**EXPERIMENTAL**

**Materials**

The equipment used were glasswares, magnetic stirrer, spatulas, Petri dishes, pestles, batteries, masking tape, furnaces, digital multi-meters, vacuum pumps, storages bottles, rotary vacuum evaporator, refrigerator, spin coating, black glass bottles, spectrophotometer 21, stirring rods and analytical scales, SEM, XRD, UV-Vis Agilent 8435 spectrophotometer, PerkinElmer FTIR.

The materials used were jengkol peel (\textit{Pithecellobium jiringa} B.), senduduk fruit (\textit{Melastoma malabathricum} L.), mangosteen peel (\textit{Garcinia mangostana} L.), and red grape peel (\textit{Vitis vinifera})). salicylic acid, hydrochloric acid (HCl) pa, distilled water, silver nitrate (AgNO$_3$), ITO glass, TiO$_2$ Degussa P-25, methanol pa, ethyl acetate pa, n-hexane pa, Whatman paper No.42, KI (potassium iodide), I$_2$ (iodine), acetonitrile pa, polyethylene glycol (PEG), candle, ITO glass, hydrochloric acid (HCl pa), polyvinyl alcohol (PVA), calcium chloride (KCl) and sodium citrate.

**Preparation of ITO Glass**

ITO glass was cut into 1.25 x 1.25 cm and sanded at the edges. The ITO glass was then soaked in 70% alcohol for 60 minutes before being cleaned with an ultrasonic cleaner. This procedure was designed to remove material that will disrupt the coating process on ITO glass.

**Production of TiO$_2$ Paste**

0.5 grams of polyvinyl alcohol (PVA) was dissolved with 50 mL of aquadest. The solution was stirred and heated at 80°C on a hotplate to form a suspension. The suspension was then added to 0.5 grams of
Dye Preparation
Extraction
100 g of jengkol peel, senduduk fruit, mangosteen peel, and red grape were extracted using 50 mL of 96% ethanol and 10 mL of HCl 1M. The extraction process was carried out in a state without light. After 24 hours, the extraction was filtered with Whatman filter paper No. 42 to separate the residue. The filtrate was then evaporated with a rotary evaporator to remove the solvent. The extract was stored in dark bottles or bottles coated with aluminum. The resulting residue was tested by adding HCl 2M and heating at 100°C for 5 minutes to see if it still contained anthocyanins. If a red color appears, the results are said to be positive. Then, NaOH 2M was added dropwise while observing the color change. When a green-blue color fades slowly, the result is positive. If the residue is positive, it is assumed that anthocyanins are still present, and the extraction is repeated.

Dye Co-pigmentation
Copigmentation was accomplished by mixing 38.89 mg of salicylic acid into 20 mL of each sample extract. The anthocyanins and co-pigments were then mixed for 30 minutes in a shaker.

Dyes Characterization
UV-Vis Testing
The UV-Vis instrument was used to determine the absorbance of the dye, which had been co-pigmented with salicylic acid. UV-Vis was also used to identify the effects of the dye co-pigmentation process. The absorption of dye was analyzed using the UV-Vis Agilent 8435 spectrophotometer. The wavelength of light used was between 400 and 800 nm. The test was conducted at the chemical laboratory of Universitas Negeri Padang.
FTIR Analysis
The FTIR analysis was intended to determine the types of bonds and functional groups of anthocyanins that have been extracted using the PerkinElmer FTIR. The analysis was conducted by looking at a specific spectrum or peak indicating a particular functional group. The test was carried out at the Chemical Laboratory of Universitas Negeri Padang.

Preparation of Semi-Solid Electrolyte
PEG 0.1 M (KI 0.5 M and I$_2$ 0.05 M) was prepared by dissolving 0.498 g of K.I. in 6 mL of acetonitrile. Another solution was made by mixing 0.076 g of I$_2$ in 6 mL of acetonitrile and stirring until it was homogeneous. The two solutions were mixed as an electrolyte solution. A total of 2.4 g of PEG was added to the electrolyte solution that had been prepared and stirred to form a gel.

Preparation of The Counter Electrode
The carbon source used in counter electrode preparation was graphite. Graphite was coated onto ITO glass by heating it in the conductive part of the glass with a candle. Next, ITO coated with graphite was heated at 450°C for 30 minutes. The temperature was gradually reduced until it reached 70°C, after which it was allowed to cool to room temperature for 27 minutes.

Solar Cell Fabrication
After the components of the DSSC had been made, fabrication was carried out to assemble the solar cells. The DSSC components were assembled into a structure similar to a sandwich in the order shown in the picture.

Solar Cell Electric Current Testing
An automated multi-meter was used to monitor the experimental results for solar cell voltages and currents. As a light source, U.V. lighting was used. The solar cell's efficiency can be calculated using the current and voltage values.

RESULTS AND DISCUSSION
Ag-TiO$_2$ Layer
The substrate in this experiment was Indium Tin Oxide (ITO) glass. In the visible light spectrum, ITO has good transparency and low inhibition. ITO is amorphous and has a lower conductivity at low temperatures. Titanium dioxide (TiO$_2$) is first applied to ITO glass. Titanium dioxide is a photon electron transfer semiconductor. TiO$_2$ has high redox selectivity and oxidizing photostability.

The TiO$_2$ in this study was electrodeposited with Ag metal. Electrodeposition is the process of overlaying a chemical specimen on a cathode through an electrolytic cell. In this study, Ag electrodeposition was performed on the TiO$_2$ surface to modify the TiO$_2$ layer and form a barrier film between TiO$_2$, dye, and electrolyte to reduce any electron recombination. The electrodeposition process uses 2 electrodes: a TiO$_2$ electrode and a carbon electrode. The electrolyte solution used was AgNO$_3$ 0.2 M. The reactions that occur in the process of electrodeposition are as follows:

Cathode : $\text{Ag}^{+}(\text{aq}) + e^- \rightarrow \text{Ag}(s)$ (on TiO$_2$ surface)
Anode : $\text{Ag}(s) \rightarrow \text{Ag}^+ (\text{aq}) + e^-$
The counter electrode was made of ITO glass coated with carbon because it has fairly good conductivity, heat resistance, and electrocatalytic activity from triiodide reduction. In Kay and Gratzel's research, they developed the DSSC design using a carbon counter-electrode as the catalyst layer. It has a high surface area and a triiodide reduction reactivity resembling a triiodide reduction on a platinum electrode. The existence of Ag was confirmed by the EDS instrument as shown in figure 3, where the materials composition contained in the layer included oxygen (O) of 28.93%, titanium (Ti) of 19.96%, and silver (Ag) of 51.10%. According to the results, it can be seen that there was a lot of Ag metal that was electrodeposited onto the TiO$_2$ layer.

| Element | Weight % | Atomic % | Net Int. | Error % | Kratio | Z   | A   | F   |
|---------|----------|----------|----------|----------|--------|-----|-----|-----|
| O K     | 28.93    | 67.01    | 152.32   | 11.01    | 0.0363 | 1.2396 | 0.1012 | 1.0000 |
| AgL     | 51.10    | 17.55    | 955.10   | 2.38     | 0.4741 | 0.8615 | 1.0742 | 1.0027 |
| TiK     | 19.96    | 15.44    | 436.30   | 3.96     | 0.1595 | 0.9889 | 0.8035 | 1.0055 |

SEM-EDS analysis was conducted to identify surface morphology and Ag on the TiO$_2$-Ag later. Creating an SEM-EDS image is a physical process of corpuscular interactions between electron sources and the atoms in materials. Compared to the XRD and optical microscope, the data signal generated from SEM-EDS was stronger. On the other hand, the object of observation was perceived to be larger and to contain non-productive components, such as a passivation layer of oxide materials on the surface, which could
cause a low contrast.\textsuperscript{33} The result of SEM showed that Ag electrodeposition formed small clumps distributed evenly on the surface of the TiO\textsubscript{2} layer. Ag clumps on this surface would form a metal contact that would act as a barrier against electron recombination in dye with electrolyte and accelerate the electron stream toward ITO glass, which has a high conductivity.

The Difactogam pattern found in this study was used to determine the crystal structure and crystallite size of TiO\textsubscript{2} and TiO\textsubscript{2}-Ag based on the value of FWHM in various peaks using the Scherrer equation. The interpretation of XRD data can be seen in Tables-1 and 2.

According to the peak data collected from both samples, the resulting crystal was in an anatase phase. The intensity of the anatase structure proved that it had more area. Anatase has a good bandgap and promotes high photoactivity to increase DSSC efficiency. Anatase has a greater bandgap and higher photoactivity because it can significantly absorb ultraviolet (U.V.) light to improve DSSC efficiency.

When TiO\textsubscript{2}-Ag was compared to TiO\textsubscript{2}, the diffraction peak for TiO\textsubscript{2} was at \(2\theta = 25.33\)\(^{\circ}\), and TiO\textsubscript{2}-Ag showed a new peak at \(2\theta = 37.76\)\(^{\circ}\). \(d(A)\) peaks for crystal included 3.5157, 2.3783, 1.8931. The data interpretation card suggested that anatase form was closer to 3.5134, 2.3732, and 1.8935. From the result of XRD interpretation in specifying crystal phase, it is suggested that the intensity of the anatase structure had more areas at TiO\textsubscript{2}-Ag.

In addition, the effect of Ag electrodeposition can be seen from the reduced TiO\textsubscript{2} particle size. The TiO\textsubscript{2} crystal size can affect the efficiency of the resulting DSSC. The smaller TiO\textsubscript{2} crystal size has a larger surface area to accommodate more dye. The more dye accommodated, the more photon absorption will increase, so the efficiency of the DSSC will also increase.\textsuperscript{34}

Absorption with a strong intensity occurred in the area of \(2\theta = 75.08\)\(^{\circ}\), which showed a crystal size of 78.28 nm. The results of the interpretation of the XRD data of the TiO\textsubscript{2}-Ag layer were different from the results of the XRD data interpretation of the TiO\textsubscript{2} layer. As shown in Table 3, the absorption with a strong intensity occurred in the region \(2\theta = 25.28\)\(^{\circ}\), which indicates a crystal size of 63.61 nm. Based on these two measurements, the TiO\textsubscript{2} crystal size decreases to 14.67 nm as it gets smaller. The compact size change was approximately 18.74\% of the initial TiO\textsubscript{2} size.
The change in the size of TiO$_2$ crystals becomes smaller due to the deposition of Ag in the layer, which changes the layer's texture to decrease intensity and reflection.\(^\text{35}\) The smaller the value of 2\(\theta\), the greater the value of FWHM and the smaller the crystal size.

**Dye Characterization**

The dye functions as a maximizer for the absorption of sunlight in the DSSC because it provides a lot of extra electrons. The ideal dye's properties must possess for DSSC: strong absorption in visible light (400-700 nm), strong absorption on the semiconductor surface, stability in the oxidized form, and electrolytes can reduce. The sources of extract dye used in this research were jengkol peel, senduduk fruit, mangosteen peel, and grape peel. In the dye extracts, it is known that there is an anthocyanin compound. According to the literature, anthocyanins are water-soluble pigments and have a red-to-blue color.\(^\text{36}\) Anthocyanin is widely distributed in fruits, flowers, and leaves. Anthocyanin is modified by glycosyl and aromatic or aliphatic alkyl groups, producing hundreds of anthocyanin molecules.\(^\text{37}\) The dye was characterized using two instruments, the UV-Vis spectrophotometer and the FTIR.

FTIR was used to characterize anthocyanin dye's existence by knowing the functional groups present in the dye of jengkol peel, senduduk fruit, mangosteen peel, and grape peel. The results of the analysis can be seen in figure 7. Based on the results of FTIR identification, it can be concluded that in the jengkol skin, senduduk fruit, mangosteen peel, and grape skin dye extract, there were anthocyanin compounds that can be used as dyes in DSSC.

The UV-Vis spectrophotometer test observed the impact of co-pigmentation of salicylic acid in dye extracts. The interaction between co-pigment and dye was intermolecular.\(^\text{38}\) Salicylic acid co-pigmentation will produce anthocyanins with better thermal stability and a bathochromic effect, where the maximum absorbance shift occurs. The results of the UV-Vis test showed that dye extracts from different sources had different maximum wavelengths (\(\lambda_{\text{max}}\)). For the jengkol peel, the absorption shifted from a maximum wavelength of 515 nm in color without any co-pigmentation to 520 nm for co-pigmented dye. Senduduk fruit shifted from 505 nm to 510 nm, mangosteen shifted to 515 nm with an increase in intensity, and grape peel shifted from 525 nm to 535 nm. This enhancement occurred because salicylic acid added electrons \(\pi\) to anthocyanin so that light absorption would shift to a longer wavelength. Co-pigment will improve the coordination between one anthocyanin and another, generating a \(\pi - \pi\) interaction.\(^\text{39}\) Light absorption at a longer wavelength might increase sunlight absorption by DSSC. Complex formation between co-pigment and anthocyanin also affected the absorption of the dye. Based on the data, it can be seen that co-pigmentation revealed an increase in absorption intensity at a wavelength, becoming greater, thus improving light absorption and promoting a positive effect on DSSC.

**Efficiency Measurement**

The measurement of DSSC performance can be done by measuring the assembled DSSC efficiency. It was done with the help of U.V. lighting of 24-watt power and a multi-meter to measure voltage and
The use of light sources from U.V. lamps is intended to obtain an accurate and unchanging source because if tested directly under the sun, it is feared that different light intensities will be obtained, which will affect the calculation of efficiency. The current of the assembled DSSC can be calculated with the equation $V=I.R$. Where $V$ is the voltage generated in volts, $R$ is the resistance in the DSSC in $\Omega$ (ohm), and $I$ is the current flowing in the DSSC (A).

The result of sunlight conversion transforming without Ag electrodeposition was 2.94%, while the efficiency after Ag electrodeposition was 5.21%, revealing an efficiency improvement in DSSC. The formation of a layer on the surface that acts as a barrier layer to reduce the recombination process between the electron injected and the redox pair on the electrolyte results in an increase in DSSC efficiency using Ag electrodeposition. The formation of a layer can mitigate electron recombination due to an additional electron trap provided by Ag.

An increase in efficiency also occurred due to a decrease in TiO$_2$ crystallite size. According to the XRD results, Ag electrodeposition caused the decline in TiO$_2$ crystallite size. The TiO$_2$ crystallite size in nanometers and the anatase phase have a greater surface area to accommodate numerous dyes. The more dye is collected, the more photon absorption and DSSC efficiency will increase. Another improvement is the use of pigmented dyes, which have a maximum $\alpha$ that is longer because of the bathochromic effect to absorb more photons. In Table 4, the DSSC efficiency using dye extracts and Ag electrodeposition can be seen.

| Dye Source       | Output Voltage (V) | Current generated (A) |
|------------------|--------------------|-----------------------|
| Jengkol peel     | 0.4000             | 8.7000 x 10$^{-3}$    |
| Senduduk fruit   | 0.3961             | 8.0340 x 10$^{-5}$    |
| Mangosteen peel  | 0.4340             | 1.8869 x 10$^{-4}$    |
| Grape peel       | 0.4670             | 1.3684 x 10$^{-4}$    |

The performance DSSC produced by TiO$_2$-Ag and co-pigmented dye was 1.4500% from jengkol skin, 1.326% from senduduk fruit, 3.4122% from mangosteen peel, and 2.6683% from the grape skin. The data
in the table showed that DSSC efficiency was sequentially ranked from the lowest to the highest, commencing from DSSC that used dye from senduduk fruit, jengkol peel, grape peel, and mangosteen peel. Mangosteen peel produced DSSC at the highest efficiency. The highest efficiency was the mangosteen peel that had been co-pigmented with salicylic acid, which has a high absorption ability. It can be seen that the absorption area spectrum of the mangosteen peel anthocyanin is quite wide, from 460 to 580 nm. The wider the absorption spectrum line of anthocyanin, the better the solar cell's performance. Better sunlight absorption was obtained through broad and great absorption. The mangosteen peel, which is co-pigmented, causes an increase in the intensity or absorbance ability.

The maximum wavelength of the mangosteen rind was 515 nm, indicating that the anthocyanin and salicylic acid complexes require low energy to excite electrons. The excited electrons from the conduction band to the valence band of the mangosteen rind dye extract will flow to TiO$_2$. The lower the energy required for the electron excitation process, the more electrons will flow to TiO$_2$ and the outer circuit of the DSSC.

**CONCLUSION**

Electrodeposition of Ag on TiO$_2$ reveals the increase in DSSC efficiency:

1. Salicylic acid co-pigmentation on dye can promote dye absorption to longer wavelengths (Jengkol peel at 515 nm to 520 nm, Senduduk fruit at 505 nm to 510 nm, mangosteen peel at 515 nm with an increase in intensity, and grape peel at 525 nm to 535 nm).
2. The performance of the DSSC generated by the TiO2-Ag layer and co-pigmented dye was in jengkol peel of 1.4500%, Senduduk fruit of 1.3260%, mangosteen peel of 3.4122%, and grape peel of 2.6683%.
3. DSSC generated the highest efficiency with Ag electrodeposition on the TiO$_2$ layer using mangosteen peel as the dye source.

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