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Article

# Using Various Solvents for Extracting Grape Juice as Dye and Applying It in Fabrications of Dye-Sensitized Solar Cells

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Abstract: Using grape juice as a natural dye source and employing a solvent extraction to isolate anthocyanins and subsequently harnessing them as sensitizers, dye-sensitized solar cells (DSSCs) were fabricated in this study. The extraction process required diverse solvents, including tert-butanol, acetonitrile, acetone, and ethanol, with multiple adjustments and parameter optimizations to determine the most effective ratio in extraction. In the preparation of the photoanode, commercial titanium dioxide powder, which combined 20 mol% anatase phase and 80 mol% rutile phase, was utilized and applied through a doctor blade technique. The prepared photoanodes were immersed in the extracted anthocyanin dye and subjected to being exposed at various times to ensure complete light exclusion throughout the process. For electrode preparation, a platinum film approximately 1 nm thick was sputter-coated onto an indium tin oxide (ITO) glass substrate. The photoanodes treated in a dye-soaking process were then sealed with the electrodes. We measured incident photon-to-electron conversion efficiency (IPCE) with ultraviolet-visible spectroscopy (UV-VIS) and a solar simulator to comprehensively assess the performance of the manufactured dye-sensitized solar cells.

Keywords: Grape juice, Natural dye source, Solvent extraction, Dye-sensitized solar cells

# 1. Introduction

Dye-sensitized solar cells (DSSCs) employ conductive glass as an essential electrode material, commonly referred to as transparent conductive oxide (TCO). Notable TCO materials include tin dioxide (SnO<sub>2</sub>) and indium tin oxide (ITO) for their remarkable transparency and robust electrical conductivity [1,2]. These transparent conductive oxide films allow the functionality of DSSCs. However, the sheet resistance of indium tin oxide (ITO) films tends to increase at high temperatures. In contrast, films made of fluorine-doped tin oxide (FTO) display minor changes in impedance. Consequently, when crafting dye-sensitized solar cells composed of titanium dioxide (TiO<sub>2</sub>), high-temperature annealing is frequently employed with FTO substrates as a common choice [3,4]. The selection between ITO and FTO substrates in DSSCs is significant, as it directly influences the overall performance and stability of the device. The impact is especially pronounced in response to temperature fluctuations and during the annealing process. The intricate interplay between electrode materials and TiO<sub>2</sub>'s sensitizing properties underscores the critical role of meticulous material selection in the design and optimization of DSSCs.

In DSSCs, the photoanode plays a crucial role as it absorbs dye molecules and facilitates charge transfer as a key component in converting sunlight into electricity. Common photoanode materials include semiconductors such as zinc oxide (ZnO) [5], titanium dioxide (TiO<sub>2</sub>) [6], and tin dioxide (SnO<sub>2</sub>) [7] with titanium dioxide being the predominant choice. TiO<sub>2</sub> exhibits three distinct crystal structures: anatase, rutile, and brookite. Anatase possesses remarkable light scattering properties, enhancing light absorption, while rutile offers lower resistance to electron transport, promoting efficient electron transfer on the surface of titanium dioxide. Consequently, in DSSCs, titanium dioxide nanoparticles are often employed with anatase and rutile crystal phases. The commercially available TiO<sub>2</sub> powder typically comprises 80% of the rutile phase and 20% of the anatase phase. The selection of the photoanode material and its specific crystalline structure significantly impacts the performance and efficiency of DSSCs. Researchers and engineers continually explore various combinations and modifications to optimize the photoanode to improve solar energy conversion for the advancement of renewable energy technology. Commercially available TiO<sub>2</sub> powder with its specific phase composition is used owing to its importance in tailoring the properties of the photoanode and the device's effectiveness in harnessing solar energy. In DSSCs, the prevailing choice for sensitizer materials has long been ruthenium-based dyes such as N3, N719, Black dye, and others. These ruthenium dyes possess a range of favorable characteristics, including exceptional light

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absorption capabilities, high photoconversion efficiency, and remarkable photostability. However, the scarcity and high cost of ruthenium as a base metal are their limitations. Furthermore, the inherent toxicity of ruthenium, coupled with the potentially adverse environmental consequences associated with its extraction and production processes, poses challenges. Consequently, more researchers are increasingly turning their attention towards the exploration of natural dyes as viable sensitizers [8–10].

The transition towards natural dyes as sensitizer materials signifies a notable advancement in DSSC manufacturing for the following key reasons.

- (1) Environmental Benefits: Natural dyes are sourced from readily available materials such as fruits, vegetables, or plants. Their extraction processes typically entail fewer environmental risks compared to the chemical synthesis of ruthenium-based dyes.
- (2) Cost Reduction: The adoption of natural dyes holds the potential to significantly reduce the manufacturing costs of solar cells, rendering them more affordable and accessible to a broader demographic.
- (3) Sustainability: The use of natural dyes harmonizes with the principles of sustainability and renewable energy. It is a more ecofriendly alternative, aligning with the overarching eco-conscious of renewable energy technologies.
- (4) Biocompatibility: Natural dyes exhibit biocompatible properties, paving the way for their potential applications in biophotovoltaics and wearable solar devices being compatible with living organisms and human users. This biocompatibility opens new avenues for harnessing solar energy in novel and innovative ways.

Anthocyanins belong to the flavonoid group of natural pigments and are found in many fruits and vegetables. They are watersoluble pigments that often form glycosides with carbohydrate molecules. The stability of anthocyanins varies due to various factors such as pH, temperature, and the specific type of anthocyanin. pH directly affects the color of anthocyanins. Depending on the number and position of hydroxyl (OH) or methoxy (OCH<sub>3</sub>) groups in the cation, anthocyanins are categorized into six types: malvidin, cyanidin, peonidin, pelargonidin, petunidin, and delphinidin. Researchers showed that TiO<sub>2</sub> and anthocyanins bind together [11,12]. Anthocyanins are known for their vibrant red, purple, or blue colors and are responsible for the striking pigmentation in many plant-based foods. They have gained attention not only for their aesthetic qualities but also for their potential health benefits, including antioxidant properties and other possible contributions to human health. Furthermore, their ability to interact with materials such as TiO<sub>2</sub> attracts attention in various fields, including food science, cosmetics, and solar cell technology. Thus, they can be used as sensitizers for DSSCs to harness solar energy efficiently.

## 2. Materials and Methods

In DSSCs, the electrolyte is a key component for transmitting charge and ions. It serves as the medium for electrons and ions, facilitating the smooth conversion of solar energy into electrical energy within the solar cell. DSSC electrolytes are a liquid or gel form, containing ions or molecules that are soluble. The most common electrolyte in DSSCs consists of iodine/iodide ions  $(I_3^-/I^-)$ . At the positive electrode (anode) of the DSSC, solar energy is absorbed by dye molecules, exciting the electrons within these dye molecules to transition to higher energy levels, resulting in the formation of positively charged dye cations. These cations traverse through the electrolyte, while the iodide ions in the electrolyte accept these electrons at the cathode and combine with them to form iodine molecules. The role of the electrolyte in DSSCs is to maintain a continuous flow of charge and enable the photoconversion process to ensure that the electrons generated by the absorption of light are efficiently transported and collected at the electrodes and allow for the generation of electrical current. The electrolyte and its properties significantly influence the performance and efficiency of DSSCs, necessitating research in the development of solar cell technology.

In preparing natural dyes, blueberries were ground in a mortar. The measured quantity of ground blueberries was then introduced into an extraction solvent. A variety of extraction solvents were used, including ethanol, acetone, acetonitrile, tert-butanol, and combinations such as ethanol mixed with acetone, acetonitrile mixed with tert-butanol and others. These solvents were prepared at a concentration of 200 g/L to ensure a consistent dye concentration. The obtained dye solution was transferred into a glass container and heated while being stirred using an electromagnetic heating stirrer. Afterward, the extracted dye was filtered to eliminate solid residues with filter papers. For the fabrication of the electrodes, the previously cleaned, dried, and perforated ITO glass was placed within the sputtering chamber of a gold-plating machine. The chamber was evacuated, and the process parameters were configured for 60 s at a current of 10 mA. A sputtered thickness was approximately 10 nm for a platinum (Pt) target material. In this step, the counter electrode's preparation was completed. In the procedure, natural dyes from blueberries were extracted to manufacture essential components for applications, including DSSCs and various optical devices. The sorts of extraction solvents and sputtering materials significantly influenced the properties and performance of the final devices.

The electrolyte was formulated by blending the following components in 3-methoxypropionitrile (MPN): 0.1M lithium iodide (LiI), 0.05M iodine (I2), 0.5M 4-tert-butylpyridine (TBP), and 1M 1,2-dimethyl-3-propylimidazolium iodide (DMPII). Ultrasonic agitation was used to complete the electrolyte preparation. For the preparation of the photoanode slurry, tert-butanol, TiO<sub>2</sub>



nanoparticles, and deionized water were combined at a fixed ratio of 10 wt% concentration and stirred for a minimum of 6 hours. The photoanode fabrication process employed the doctor blade method. Initially, the cleaned and dried FTO glass was placed on a custom-made holder, achieving the desired film thickness. After natural air-drying, the thin film was pressure-treated at a hydraulic press at 20 kg/cm<sup>2</sup> for 60 s. Subsequently, the TiO<sub>2</sub> film was subjected to a high-temperature annealing process, involving heating at 150°C for one and a half hours, followed by a 30-minute treatment at 550°C. In the final step, the annealed TiO2 film was exposed to UV light for 10 min to eliminate impurities generated during annealing, completing the photoanode preparation. This process involved the precise materials and steps to ensure the successful fabrication of the photoanode and electrolyte for DSSCs, where the performance and characteristics of these components affected device efficiency significantly.

Following the completion of the annealing process, the photoanodes underwent a sensitization procedure involving immersion in various natural dyes, each for specific durations of 6, 12, and 24 h. To minimize the influence of light exposure, all these steps were carried out indoors. After soaking, the photoanodes were carefully retrieved, and any surplus dye was eliminated using appropriate solvents. The sensitization of the photoanodes was completed by drying the photoanodes. For solar cell encapsulation, we used a 0.06 mm-thick thermoplastic polymer film. This film was precision-cut into dimensions measuring 2 x 1.2 cm, securing a central working area designated for the photoanode measuring 0.6 x 0.6 cm. The openings for the electrodes were aligned with the lower edge of the photoanode and placed on a sealing heating platform for the encapsulation process. This initial encapsulation step resulted in the formation of a rudimentary sandwich structure for the dye-sensitized solar cell. Subsequently, a syringe was used to inject the electrolyte into the designated electrode holes. Finally, the holes were securely sealed using a thermoplastic polymer film and an ITO glass to complete the assembly of the dye-sensitized solar cell. To analyze the absorbance spectra of the blueberry juice in various solvents, ultraviolet-visible (UV-VIS) spectroscopy was employed. Once a DSSC was fully packaged, incident photon-to-current efficiency (IPCE) was measured to evaluate how effectively a dye converted absorbed light into current at different wavelengths. Additionally, a solar simulator was used to measure the precision and electrical efficiency of the solar cells in generating electrical energy for the evaluation of their performance.

#### 3. Results

In this experiment, a solvent extraction method was used to extract compounds from blueberries and grapes using various solvents, including ethanol, acetone, acetone-ethanol mixture, acetonitrile (ACN), tert-butanol, and a tert-butanol-acetonitrile mixture. The UV-VIS analysis was conducted to examine the absorption and transmission characteristics of the extracted dyes. Grape dye exhibited distinct absorption peaks around 550 and 660 nm, indicating the presence of anthocyanins (Fig. 1). Solvent extraction is commonly used to isolate and analyze specific compounds or pigments from natural sources such as fruits. In this case, the experiment was carried out to extract and analyze anthocyanins which show red, purple, and blue colors in various fruits including grapes and blueberries. The UV-VIS analysis allows researchers to precisely identify the wavelengths at which these compounds absorb light, providing information on their presence and concentration in the fruit extracts. This information is crucial for various applications, including the development of natural dyes, food additives, and antioxidants.



Fig. 1. UV-VIS spectra of grape dye extracted using different solvents.



The efficiency of DSSCs depends on the light-absorbing capabilities of the photosensitizer and the smooth transport and diffusion of electrons through the  $TiO_2$  film. Figure 2 displays the UV-VIS spectra of  $TiO_2$  films soaked in different solvent-extracted dyes. The enhancement in absorbance indicates the anchoring and chemical interactions between dye molecules and the  $TiO_2$  film. Tert-butanol-extracted dyes exhibited the most robust absorption in various solvents, while the dye extracted with acetone showed lower absorption intensity. This variation was attributed to the chemical properties of acetone, which were relatively stable and less prone to react with plant components. Additionally, as acetone was volatile, it reduced the yield of anthocyanin dye molecules in the extraction process. Consequently, it was important to select an appropriate solvent for dye extraction as the dye significantly impacts the interaction and binding of dye molecules to the  $TiO_2$  film and influences the overall performance of DSSCs.



Fig. 2. UV-VIS spectra of TiO<sub>2</sub> films soaked in different solvent-extracted dyes.

IPCE is an essential parameter to assess the effectiveness of converting photons into electrons. Unlike overall efficiency, IPCE is measured with a spectrometer to selectively filter wavelengths of light emitted by a xenon lamp. The resulting photocurrent at each wavelength is then quantified. The primary objective of IPCE measurements is to investigate how efficiently a specific dye converts absorbed light into electrical current across different wavelengths. An ideal IPCE chart exhibits variations that align with the UV absorption spectrum, demonstrating the dye's capacity to efficiently convert light energy at various wavelengths into photocurrent. Conversely, when positions of absorption on the UV absorption spectrum do not coincide with the IPCE graph, the dye's efficiency in converting light into electricity is suboptimal. Such discrepancies are attributed to substantial resistance during electron transfer, rapid recombination of electrons to their ground state (electron-hole recombination), or other hindrances. IPCE is important in evaluating the efficacy of dye sensitization and the impact of such factors. In co-sensitization, where a mixture of organic dye molecules is used, the IPCE chart is compared with the UV absorption spectrum. This comparative analysis enables the assessment of the extent to which each dye contributes to the release of electrons after absorbing light at different wavelengths. It provides a comprehensive understanding of the individual and collective contributions of various dyes across the spectrum of wavelengths, facilitating informed decisions in the development of more efficient DSSCs.

IPCE, in other words, is a measure of the percentage of photons converted into electrons across different wavelength ranges. The intensity of light does not significantly affect a solar cell's IPCE. As depicted in Fig. 3, the dye extracted with tert-butanol enhanced light absorption more effectively than with other solvents. The DSSC prepared with grape dye reached an IPCE of up to 9%, showing nearly a two to three-fold increase in IPCE compared to its poorer-performing counterpart. The grape dye enhanced light absorption more efficiently and improved the efficiency of the DSSC. This, in turn, helped the photoanode to capture more photons after being excited, generating more electrons and consequently an increase in current and effectively boosting the overall performance of the DSSC. The IPCE values obtained in this analysis reflected the ability of the DSSC to convert absorbed light into electrical current, which stood for the device's performance. The substantial improvement in IPCE with tert-butanol-extracted dyes implied the significance of the choice of solvent in dye extraction. It directly impacted the light absorption capabilities of the dye and, consequently, the efficiency of the DSSC. These findings provided valuable foundation for researchers to optimize DSSCs and enhance their light-harvesting capabilities for more efficient solar energy conversion.

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Fig. 3. IPCE measurement results of grape dye DSSCs extracted with different solvents.

Figs. 4–9 depict the I-V characteristic curves of DSSCs prepared with grape dye extracted using different solvents and soaked for 6, 12, and 24 h. Relevant parameters are presented in Table 1. The results demonstrated that the appropriate soaking time of TiO<sub>2</sub> effectively enhanced the overall performance of DSSCs. The efficiency peaked at 12 h and declined afterward. Among the DSSCs prepared with these solvents, those containing dye extracted with tert-butanol exhibited higher cell efficiency, reaching up to 0.43% with a fill factor of 65.40%. The key factor for the improvement in cell efficiency was the current density, while the decline in efficiency was attributed to dye decomposition. Since natural dyes are prone to degradation, prolonged soaking broke the dye down, subsequently reducing cell efficiency. Another possible reason was the thickening of the dye layer. Excessive soaking might cause the dye layer to become too thick, which shortened the electron diffusion distance. This reduction in electron transfer and injection decreased cell efficiency. The findings underscore the delicate balance required in the preparation of DSSCs, where optimizing the soaking time and solvent choice is critical for achieving the highest possible cell efficiency. This research result provides a basis to improve the performance of DSSCs and understand the impact of various factors on their overall efficiency.



Fig. 4. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with ethanol solvent.



Fig. 5. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with acetone solvent.



Fig. 6. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with acetone-ethanol mixture solvent.



Fig. 7. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with acetonitrile solvent.



Fig. 8. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with tert-butanol solvent.



Fig. 9. Time-dependent I-V characteristic curves of grape dye DSSCs extracted with acetonitrile-tert-butanol mixture solvent.

Sample	Jsc (mA/cm <sup>2</sup> )	Voc (V)	Fill Factor (%)	Efficiency (%)
Ethanol-6H	0.68	0.59	62.61	0.25
Ethanol-12H	0.84	0.59	61.76	0.30
Ethanol-24H	0.77	0.59	60.98	0.27
Acetone-6H	0.42	0.59	59.88	0.15
Acetone-12H	0.54	0.59	59.29	0.19
Acetone-24H	0.47	0.6	58.16	0.16
Acetone-Ethanol-6H	0.54	0.59	59.29	0.19
Acetone-Ethanol-12H	0.69	0.6	58.81	0.24
Acetone-Ethanol-24H	0.6	0.6	62.93	0.22
Acetonitrile-6H	0.86	0.55	63.73	0.3
Acetonitrile-12H	0.91	0.56	68.13	0.34
Acetonitrile-24H	0.89	0.54	66.61	0.32
Tert-Butanol-6H	1.01	0.56	66.72	0.38
Tert-Butanol-12H	1.12	0.58	65.4	0.43
Tert-Butanol-24H	1.08	0.57	66.15	0.41
Tert-Butanol-Acetonitrile-6H	0.78	0.55	63.7	0.27
Tert-Butanol-Acetonitrile-12H	0.86	0.54	66.58	0.31
Tert-Butanol-Acetonitrile-24H	0.76	0.56	66.77	0.29

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The use of solar simulators primarily aims to replicate a sunlight spectrum closely resembling that of real-world solar radiation. In reality, solar radiation is often attenuated by atmospheric components such as oxygen, ozone, water, and carbon dioxide. Consequently, solar radiation reaching the Earth's surface is already diminished after traversing the atmosphere. The solar simulators commonly employed in laboratories are typically steady-state solar simulators, capable of providing a continuous and stable light output. When measuring the photovoltaic conversion efficiency of solar cells, particularly through techniques including I-V (currentvoltage) and P-V (power-voltage) curves, these simulators enable more accurate and consistent quantification of the electrical energy production efficiency of solar cells. The I-V and P-V curve analysis is conducted to evaluate the performance of solar cells under different operating conditions. By employing solar simulators to generate controlled light conditions, it can be measured how a solar cell responds to varying levels of illumination and voltage. This information is vital for understanding a solar cell's behavior and optimizing its performance, ensuring its operations at its maximum potential. Fig. 10 displays the I-V characteristic curves for different solvents used to extract grape dye, each at their optimal soaking times. The relevant parameters are briefed in Table 2. The results indicated that the optimal solvent for grape dye extraction was tert-butanol with an efficiency of 0.43% and a fill factor of 65.40%. Thus, tert-butanol was found to be the most effective solvent. The superiority of tert-butanol can be attributed to the quantity of dye molecules extracted, which enhances light absorption and, in turn, improves efficiency. The solvent was a critical factor for the performance of DSSCs. The results emphasized that selecting the right solvent is crucial in optimizing DSSCs, and tert-butanol demonstrated its effectiveness in extracting the grape dye and improved the device's overall performance. The selection of appropriate solvent is significant in dye extraction as it influences the photovoltaic performance of DSSCs. Researchers and engineers can refer to the present results to fine-tune their DSSC fabrication processes for enhanced solar energy conversion efficiency.

Table 2. J-V	/ characteristics c	of grape dye	extracted with	different solvents	at optimal	soaking times
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Sample	Jsc (mA/cm <sup>2</sup> )	Voc (V)	Fill Factor (%)	Efficiency (%)
Ethanol-12H	0.84	0.59	61.76	0.30
Acetone-12H	0.54	0.59	59.29	0.19
Acetone-Ethanol-12H	0.69	0.6	58.81	0.24
Acetonitrile-12H	0.91	0.56	68.13	0.34
Tert-Butanol-12H	1.12	0.58	65.40	0.43
Tert-Butanol-Acetonitrile-12H	0.86	0.54	66.58	0.31



Fig. 10. I-V characteristic curves of grape dye extracted with different solvents at optimal soaking times.

# 4. Conclusions

We investigated various solvent extraction techniques for grape dye, their influence on the light absorption characteristics of  $TiO_2$  films, the IPCE of DSSCs, and the effects of different immersion times on DSSC performance. The findings demonstrated that dye extraction with tert-butanol exhibited the highest light absorption efficiency, whereas acetone-extracted dye displayed relatively lower absorption intensity. This distinction can be attributed to the chemical stability of acetone, which reduces its reactivity with plant components and its volatility, resulting in a reduced yield of anthocyanin dye molecules during extraction. The IPCE



measurements further indicated that DSSCs prepared with blueberry dye achieved a peak IPCE of 9%. Solar simulator measurements revealed that the appropriate immersion time for  $TiO_2$  significantly enhanced the overall performance of DSSCs, with the optimal immersion duration of 12 h, as efficiency declined afterward. Furthermore, tert-butanol was found to be the most suitable extraction solvent. DSSCs fabricated with blueberry dye using tert-butanol achieved an efficiency of 0.43% and a fill factor of 65.40%. These results underscored the critical role of a type of the solvent and immersion time in optimizing DSSC performance, offering valuable insights for enhancing the efficiency of DSSCs.

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