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Efficiency enhancement of natural cocktail dyes in a TiO₂-based dye-sensitized solar cell and performance of electron kinetics on the TiO₂ surface

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In this study, natural dye extracts were prepared from the dried leaves of *Andrographis paniculata* and *Psidium guajava* (APPG). The study's objective was to increase the light harvesting phenomenon from solar energy utilizing natural dye from APPG, and the problem statement was to harvest the optimum solar radiation and convert it into electrical energy. Acetone and ethanol were used as solvents during the preparation process. Based on this research, the crystallite size of TiO₂ nanoparticles was assessed, the impact of acetone and ethanol on APPG dye was compared, and the absorption, FTIR, and UV-Vis spectra of the solar cell fabrication process using solvents were experimentally explored. APPG leaf extract functions as a dye sensitizer. Cells are precisely sandwiched with a photoanode, TiO₂ nanoparticles, an electrolyte (I⁻/I₃⁻), and a cathode. The JV properties of dye extracts utilizing acetone and ethanol were measured using a solar simulator equipped with a 100 mW/cm² Xenon light and a Keithley 2400 Graphical Series SMU. An experimental DSSC with dye extraction and utilizing acetone solvent yielded a maximum photo-conversion efficiency of 0.6914%, while ethanol yielded a photo-conversion efficiency of 0.5630%. Furthermore, an energy-level diagram was used to explain the electron kinetics of DSSC, and the time required for transfer electron injection in the TiO₂ surface from a dye-excited state was 150 ps.

KEYWORDS

Andrographis paniculata, *Psidium guajava*, APPG, TiO₂ nanoparticles, sensitizer

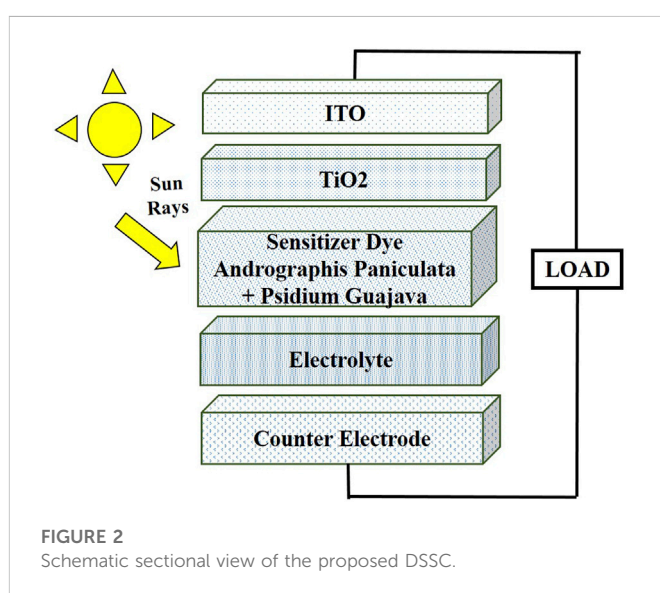
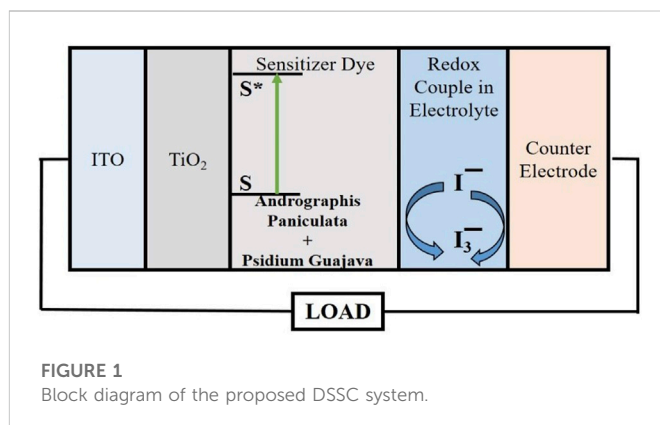
1 Introduction

The industrial revolution commenced in the 19th century with fossil fuels as the primary energy source for electrical power generation. As the effects of environmental degradation became apparent, countries began to shift toward carbon-neutral footprints and reduced emissions. Various efforts have been made to find an alternative to traditional power generation, and renewable energy sources, such as solar energy, play a vital role in electrical power generation. Conventionally, silicon (Si)-based solar cells for photovoltaic devices are globally more popular, but dye-sensitized solar cells (DSSCs) have received great attention over the last three decades. DSSCs have been widely used due to their simple preparation methodology, low cost, ease of production, and low toxicity. Researchers from all over the world have expressed an interest in DSSC since it has a variety of FTO layers, physical methods for depositing metal oxide on FTO thin films, and materials for producing various DSSC layers and there are also various types of dyes included in DSSC, and their components make it much easier to fabricate the solar cell effectively and

efficiently. Solar energy is either converted into thermal energy or through the photo-voltaic method. From various references and a conducted literature survey, the green dye rich in chlorophyll pigments present in fruits, flowers, leaves, and vegetables of naturally abundant pigments superiorly harness light energy results in the photovoltaic current.

Red and green dyes were extracted at a ratio of 4:1 from Malabar spinach and red spinach, respectively, and employed as a sensitizer in DSSC by Kabir et al. (2019). The survey's findings show that the cell efficiency was .847% compared to the dyes' individual attributes of .466% and .531%. Anthocyanin was isolated from *rubra* flowers and chlorophyll from androgynous leaves by Dhafina et al. (2020) who also evaluated the efficiency of dye molecules on the ZnO surface. It was discovered that the anthocyanin dye has a greater adsorption and bonding with ZnO surfaces, which results in an improved power conversion efficiency of .038%. Red dye from beetroot and green dye from spinach were extracted by Bashar et al. (2019) and combined in varying quantities, which acts as a sensitizer in DSSC. It was reported that a solar cell containing 80% red and 20% green dyes had a .99% higher efficiency than a cell with red and green dyes. To create sensitizers for DSSC (Rajkumar et al., 2019), dyes were collected from *Murraya koenigii* fruit and *Hibiscus sabdariffa* flower. It was observed that the dye mixture produced a voltage of 267 mV. Yellow and orange nasturtium (*Tropaeolum majus*) petals were employed by Singh et al. (2021) to extract dyes and act as a sensitizer in DSSC. According to the cell's categorization and HOMO-LUMO data, its fill factor is 0.70 and its photon to electricity conversion is 0.28%. Gokilamani et al. (2013) used natural dyes with anthocyanin bases to fabricate DSSC. The effectiveness of anthocyanin-presented extracts from red cabbage and blue peas with ethanol and concentrated HCL was 0.73% and 0.67%, respectively. This was more efficient when compared to earlier results that had been published. A TiO₂-based DSSC was created by Zakar et al. (2021) using floral extracts. For example, nursery flowers from Duhok and mountain flowers from Gara were collected. The results of the experiments with various Gara Mountain flower dyes suggest that cowslips (*Primula veris*) exhibit the best efficiency at 4.05%, while yellow nursery flowers (*Gazania rigens*) exhibit the highest efficiency at 4.19%. A curcumin-based natural dye was extracted by Sreekala et al. (2012) from acetone, dimethyl formamide, dimethyl sulfoxide, and ethanol; according to the experimental findings, the solar cell had an energy conversion efficiency of .63%, .44%, .38%, and .31%, respectively. The natural colors were extracted from spinach and *Ipomoea* by Chang H et al. (2009). Guo et al. (2015) described the graphene-based materials for the photoanode and their properties.

A comparison of the leaf extract of *Acacia nilotica* (green dye) and beetroot (red dye) combined with coconut shell as an activated carbon source showed experimental results of 0.69% and 0.504% by Parasuraman (2022). The green dye with activated carbon showed a maximum photo-conversion efficiency compared to the red dye. Similar improvements can also be applied to solar thermal systems using a heat-reflective coating by Kumar and Ramakrishnan (2022). This minimizes dye degradation and helps increase the lifetime of the solar cell. The natural dye prepared from *Kerria japonica* and *Rosa chinensis* flower showed an efficiency that was improved by the sugar molecule and increased to 0.29% and



decreased to 0.27%, respectively, by Hemalatha et al. (2012). Oprea et al. (2012) conducted a hybrid theoretical and practical analysis to determine if betalain pigments meet the requirements for DSSC TiO₂-sensitizing dyes. Based on DFT calculations, they examined the adsorption onto the substrate and revealed that all betalain dyes favor the same type of bonding to the TiO₂ nanocrystal, i.e., *via* the COO group connected to the sp²C of the six-member heterocycle. Zolkepli et al. (2015) showed that the 1:4 mixture of *Tradescantia spathocia* and *Ixora coccinea* pigments had the highest cell efficiency (= 0.80%).

Koyyada et al. (2019) systematically introduced alternative donors and anchoring groups, and we created a new class of five EDOT-based co-sensitizers with N749 (black dye). The power conversion efficiency (PCE) of DSSC devices with a mixture of co-sensitizers and N749 was the maximum, which was higher than the PCE of a single N749 dye (6.18%). Pan et al. (2017) incorporated electron-donating diphenylamino and electron-withdrawing benzothiadiazole moieties with a series of newly created push-pull porphyrin dyes that added more donors and acceptors. At an optimum molar ratio of 10:1 for the two porphyrin dyes, a high photovoltaic efficiency of 8.6% was achieved. Ogomi et al. (2010) described that the dye adsorption was aided by a cocktail of organic dye NK3705 and inorganic

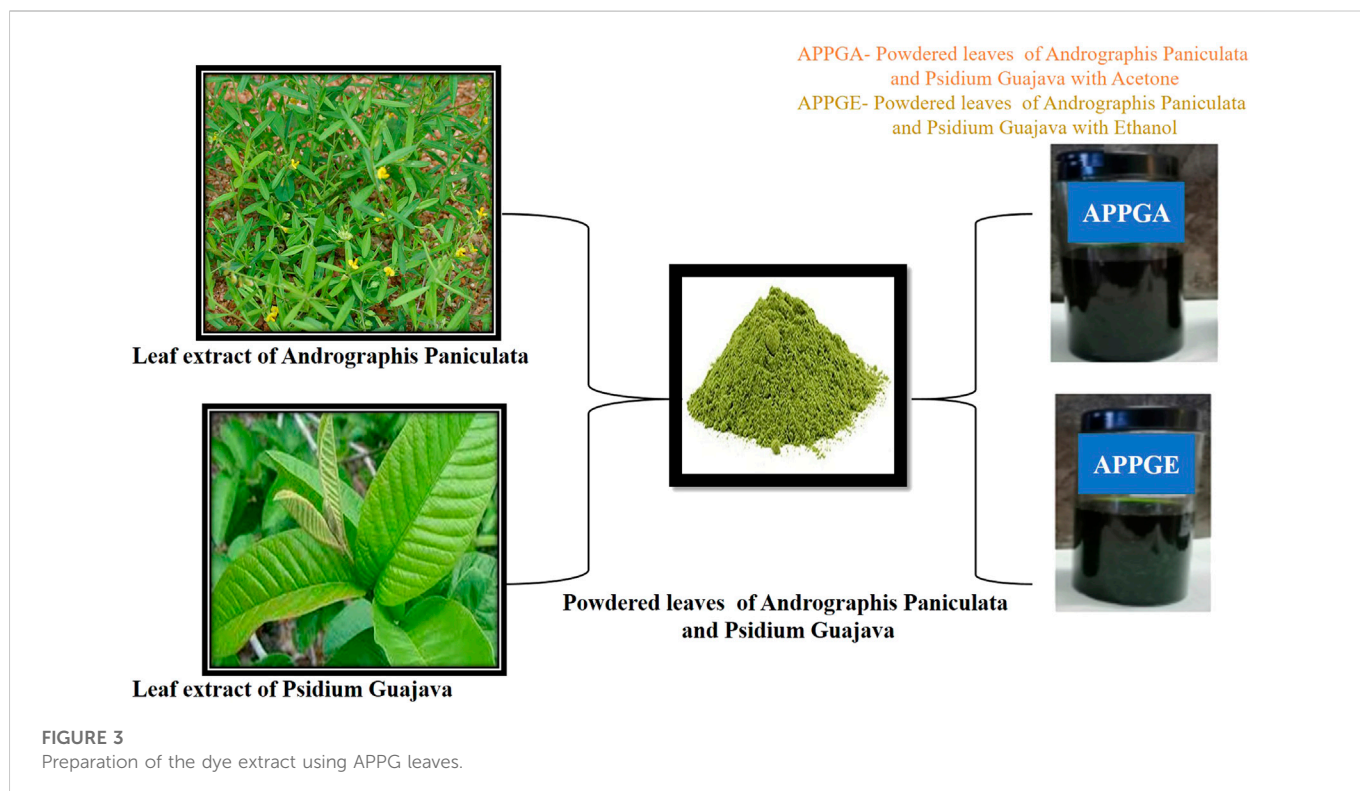


FIGURE 3
Preparation of the dye extract using APPG leaves.

ruthenium-based dye Z907 and achieved a 7.1% efficiency compared with a single dye. Lim et al. (2016) demonstrated natural cocktail dyes derived from *Ixora coccinea* (RX) and *Bougainvillea* at a 1:1 ratio and observed an efficiency of .40%.

According to references and extensive analysis, a dye cocktail was more effective than individual dyes. The blend of *Andrographis paniculata* and *Psidium guajava* was employed in this study with the frequently available acetone and ethanol solvents and made the task efficient. Figure 1 depicts the proposed DSSC system's block diagram and charge transportation. This dye promotes light harvesting and electron transport to TiO_2 nanoparticles, which results in a photoelectric current. In comparison to fruits and flowers, leaf extraction is simpler and more abundant. The presence of chlorophyll pigment and a green color indicates that electrons are transported effectively from the photoanode to the cathode via the load. Research on natural dyes is competing against the results of the suggested solar cell and promotes the utilization of light as a photosensitizer. Figure 2 depicts a schematic sectional view of the proposed DSSC.

2 Preparation of the dye

The leaves of *Andrographis paniculata* and *Psidium guajava* can be found all over India and were grown with other plants. Separately, 150 g of each fresh leaf was collected and placed in distilled water for 48 h before drying in the shade. The dried leaves were pulverized into a powder using a mortar and pestle at a 1:1 ratio. According to the findings and conclusions, natural dyes based on ethanol and acetone are more efficient than other solvents. The dye extraction technique is

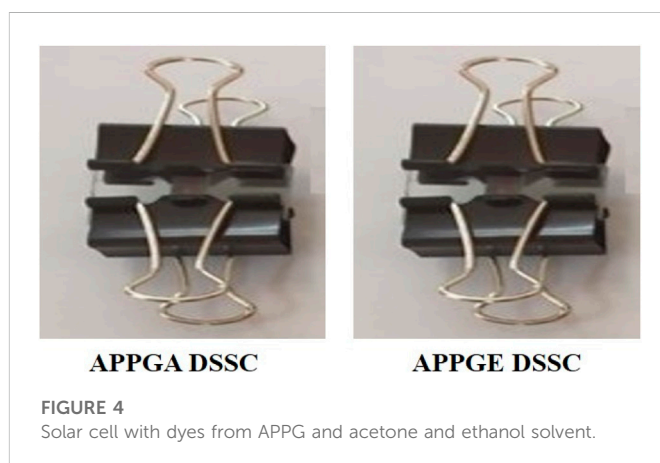
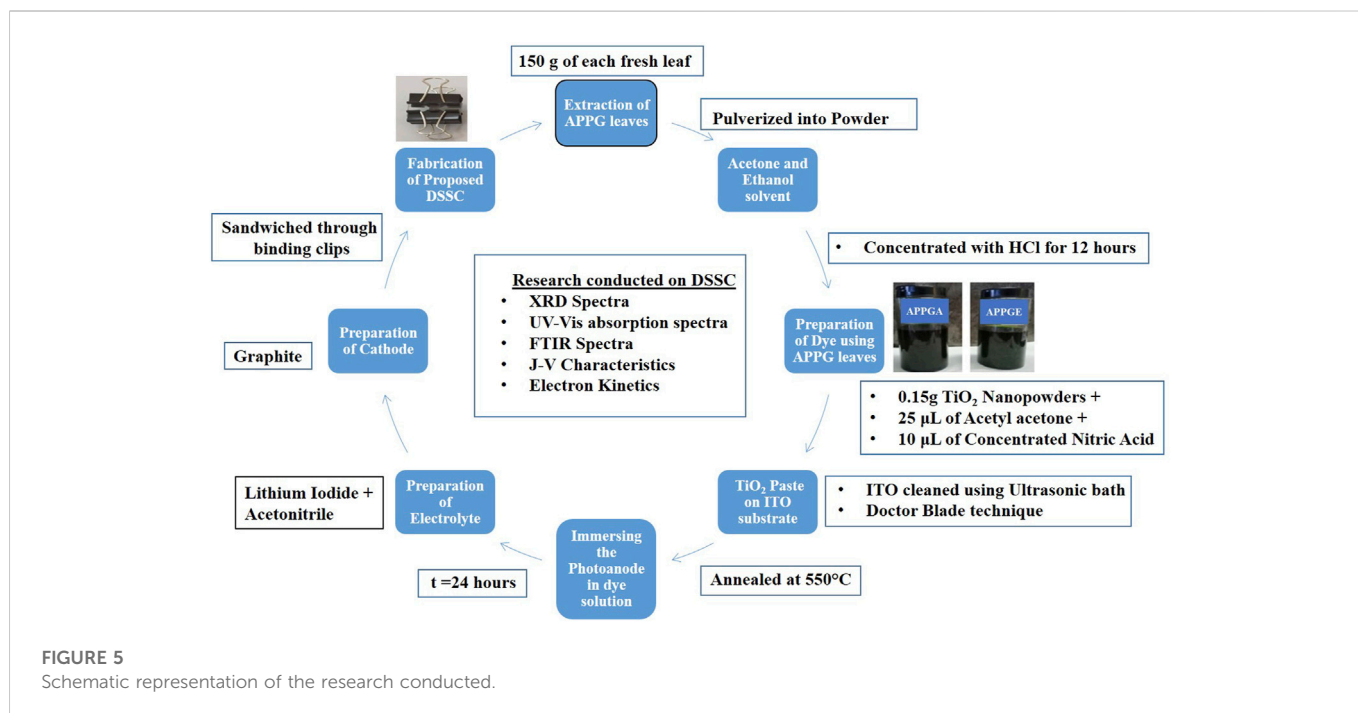


FIGURE 4
Solar cell with dyes from APPG and acetone and ethanol solvent.

depicted in detail in Figure 3. The powdered leaves were separated and steeped in ethanol and acetone with varying concentrations of hydrochloric acid for 12 h. Aluminum foil was utilized to protect the dye from light exposure. The residues are then filtered through a Whatman filter paper to form the dye solution.

FTO conductive glass has an area of $4 \times 4 \text{ mm}^2$, a resistivity of $6/\text{sq.}$, a thickness of 0.22 cm, and a transmittance of 79% for both TiO_2 -coated FTO and graphite counter electrodes. To remove foreign particles, FTO was washed in an ultrasonic bath and then soaked in distilled water for 15 min. TiO_2 paste is made from 0.15 g TiO_2 nanopowders, 25 μL of acetylacetone, and 10 μL of concentrated nitric acid. Using a porcelain mortar, the paste was

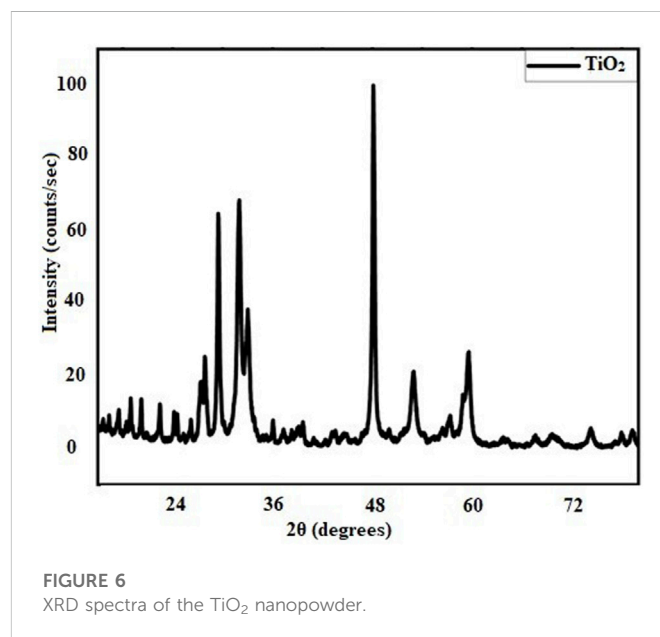


made by layering TiO_2 nanopowder and adding acetyl acetone and concentrated nitric acid one drop at a time. Then, a pestle was used to fully mix the paste for 6 min. Using the doctor blade technique, the prepared TiO_2 was coated on FTO. To ensure homogeneity, the coated FTO was annealed at 550°C . After treating at a room temperature of 70°C , the annealed photoanode was thoroughly immersed in prepared APPG dyes for 24 h. As a counter cathode, prepared FTO conductive glass was uniformly coated with graphite, and lithium iodide was made with a few drops of acetonitrile for the electrolyte solution.

Six solar cells of each APPGA and APPGE were produced, and two solar cells of each solvent were fabricated and perfectly sandwiched. Figure 4 resembles a solar cell with dyes from APPG with acetone and ethanol solvents. The photoanode of the coated TiO_2 paste faces downward with the conductive side of the counter electrode, and the prepared strong electrolyte is introduced between the photoanode and cathode through the holes *via* the counter electrode. To prevent electrolyte leakage, the binding clips were placed, and this comprises the dye-sensitized solar cell (DSSC). Figure 5 represents a schematic representation of the research conducted in this study.

3 Characterization and measurement

A Shimadzu UV-1700 series UV-visible spectrometer was used to perform the UV-visible spectroscopic study of the dyes, and for the FTIR spectroscopic study, IRAffinity with MIRacle 10 in ATR mode was utilized. XRD was recorded for the semiconductor oxide after annealing using the 3rd generation Empyrean, Malvern Panalytical multipurpose diffractometer with MultiCore Optics and an EVO 18 model Scanning Electron Microscope with low vacuum facility and ALTO 1000 cryo attachment. A solar simulator with a 100-mW/



cm^2 power Xenon lamp incorporated with Keithley 2400 Graphical Series SMU was used to record the JV characteristic study for the dye-sensitized solar cell. The study of electron injection was employed by ultrafast transient absorption spectroscopy.

4 Results and discussion

4.1 XRD spectra of TiO_2

Using the sol-gel method, titanium dioxide (TiO_2) nanopowder was synthesized and characterized using XRD to determine the crystallite size.

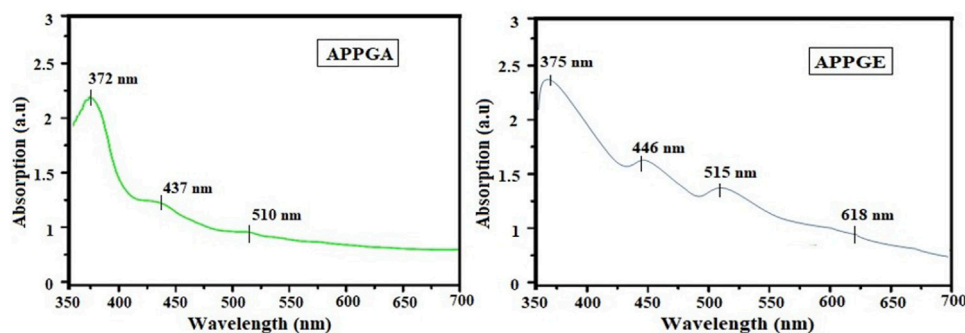


FIGURE 7
UV-Vis spectroscopy of the APPG leaf extract with acetone and ethanol.

XRD was used only for TiO₂ and not TiO₂ coated over ITO. The literature indicates that TiO₂ nanopowder thermally treated below 500 °C has both the crystalline anatase and rutile phases. As the anatase phase was more dominant than rutile (2θ values at 32.9, 46.7, 58.22, 68.1, 77.3, and 86.3 for the anatase phase and 2θ values at 38.8, 40.4, 55.5, 60.7, and 85.7 for the rutile phase), it was observed that the anatase phase was dominant with high intense peaks. For the evaluation of the crystallite size, Debye Scherrer's formula was utilized, and TiO₂ nanoparticles were obtained by comparing the x-ray diffraction peaks with the JCPDS data.

From Debye Scherrer's Eq. 1, the crystallite size D can be evaluated and is given by

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where K is the Scherrer constant equal to 0.9, λ is the wavelength of the X-ray beam, β is the peak width of the half maximum, and θ is the diffraction angle.

Figure 6 shows the XRD pattern of TiO₂ nanoparticles, which has been drawn with obtained data, i.e., the intensity of diffraction is plotted against the 2θ values.

TiO₂ powder reflects the monoclinic structure, which is confirmed with the JCPDS data sheet that there is no anatase (PDF card 98-011-1319) or rutile (PDF card 96-900-7532) and can be indexed due to its orthorhombic nature. The most intense 2θ peak at 32.9 is indexed at (0 2 0). The Debye Scherrer equation was used to measure the average crystallite size of the TiO₂ as 16.59 nm. The XRD data revealed the structure, which occurs under the pbcn space group. The cell parameter is a = 4.52 Å, b = 5.50 Å, and c = 4.90 Å.

4.2 UV-Vis spectra

UV-Vis spectroscopy was used to measure the absorption wavelength of APPG leaves. The UV-Vis spectroscopy of APPG leaves with acetone (APPGA) and ethanol (APPGE) is shown in Figure 7. The presence of the chlorophyll pigment and green color in nature is confirmed by the absorption peaks of APPGA at 372 nm, 437 nm, and 510 nm. Simultaneously, APPGE absorption peaks at 375 nm, 446 nm, 515 nm, and 618 nm confirm the presence of chlorophyll pigments. According to these findings, the cocktail dye

consisting of AP and PG operates as a photosensitizer and is excellent in light-harvesting phenomena. Solar radiation clearly illuminates the cell, and energy is absorbed by the dye molecules. UV-visible spectroscopy indicates that dye molecules may absorb solar light in the UV and visible regions of the electromagnetic spectrum, which ranges from 350 to 780 nm depending on the source. The dye molecules sensitize and photoexcite the electrons faster that are attached to the TiO₂ conduction band, which causes them to move into the ITO material. The electrons are now flowing to the counter electrode *via* the load and form a current.

4.3 FTIR spectroscopic study

To achieve an optimal photo conversion efficiency when using the prepared dye for fabrication, there must be an effective bonding between TiO₂, and dye molecules must have specific functional groups. The FTIR analysis was conducted using an FT/IR-4600 type A spectrometer.

Stretching modes identified in FTIR spectra show that the functional groups of APPG and APPGA were compared and determined to be identical, namely, -C-H, N-H, =C-H, C=C, O-H, and C-N (Table 1). Figure 8 shows the FTIR spectroscopy of an APPG cocktail leaf extract using acetone and ethanol. These studies demonstrate the strong bonding between TiO₂ and dye because of the presence of functional groups such as the carboxylic group, the hydroxyl group, and the carbonyl group. The sensitized dye becomes tightly bonded, and the electron transfer in TiO₂ later transmits them to the counter electrode *via* the load, improves current flow, and enables DSSC to reach a maximum power conversion efficiency.

4.4 JV characteristics study

Fabricated DSSC's are examined under a solar simulator with a 100 mW/cm² power Xenon lamp incorporated with a Keithley 2400 Graphical Series SMU. From this study, maximum current density (J_m), maximum voltage (V_m), open-circuit voltage (V_{oc}),

TABLE 1 Absorption frequency region from FTIR spectra of APPGA and APPGE.

Absorption frequency region (cm ⁻¹)	Functional group	Absorption frequency region (cm ⁻¹)	Functional group
	APPGA		APPGE
982.345	C-H bonding	998.516	C-H- bonding
1,432.678	C-N bonding	1,589.576	C-N bonding
2056.347	C=C bonding	1998.125	C=C bonding
2,187.785	O-H bonding	2,756.214	O-H bonding
2,756.971	O-H bonding	2,995.578	=C-H bonding
2,985.741	=C-H bonding	3,312.429	N-H bonding
3,357.456	N-H bonding		

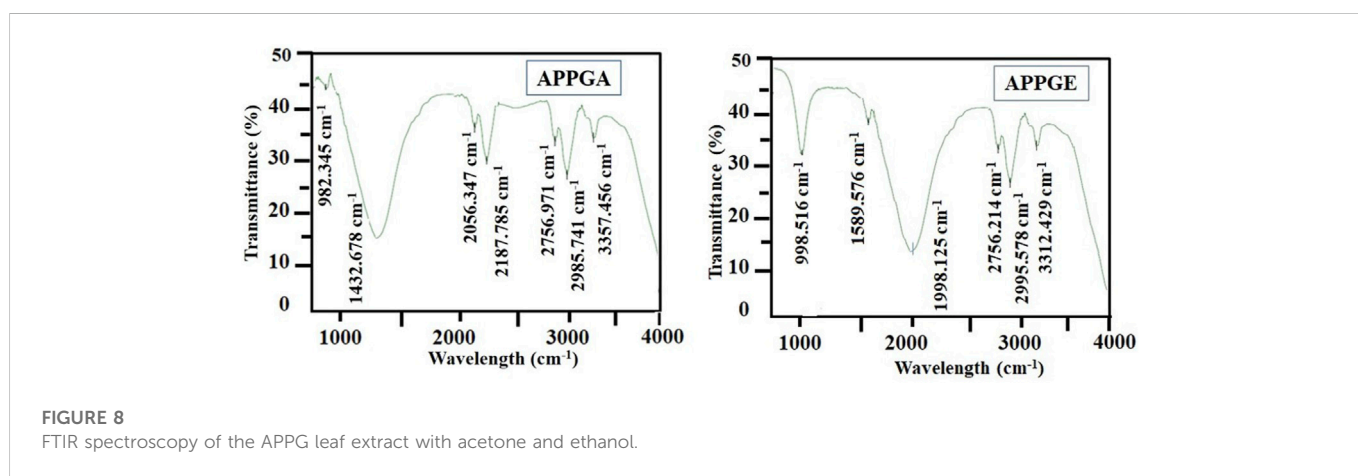


FIGURE 8 FTIR spectroscopy of the APPG leaf extract with acetone and ethanol.

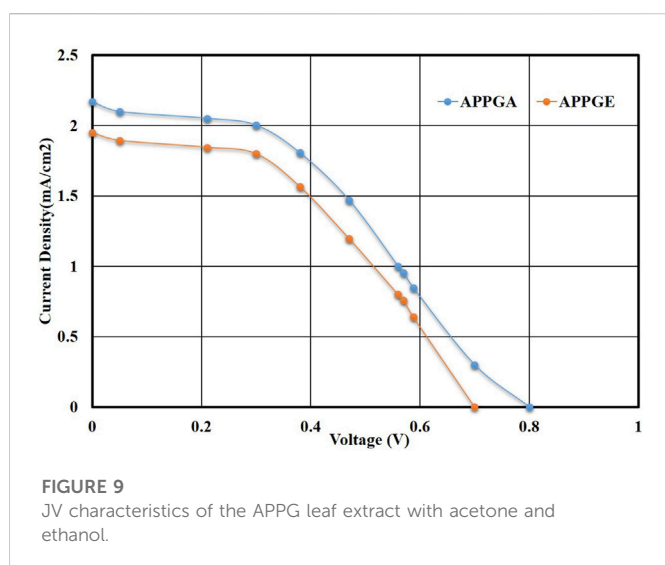


FIGURE 9 JV characteristics of the APPG leaf extract with acetone and ethanol.

TABLE 2 List of parameters evaluated for DSSC with APPGA and APPGE.

Parameter	APPGA	APPGE
J _{sc}	2.17	1.95
V _{oc}	0.8	0.7
J _m	1.4711	1.198
V _m	0.47	0.47
FF	0.3982	0.3535
Efficiency (%)	0.6914	0.5630

$$\text{Fill Factor (FF)} = \frac{J_m \cdot V_m}{J_{sc} \cdot V_{oc}} \tag{2}$$

$$\text{Efficiency } (\eta) = \frac{FF \cdot J_{sc} \cdot V_{oc}}{P_{in}} * 100 \text{ (\%)} \tag{3}$$

Figure 9 represents the JV characteristics of the APPG leaf extracts of acetone and ethanol. The JV properties of a solar cell using the APPG dye with solvent acetone (APPGA) and ethanol (APPGE) have a substantial impact on electron injection, charge transfer, and redox couple reduction. It was discovered that oxidation of the dye after photon energy absorption and regeneration of dye molecules by reduction occurred within a

TABLE 3 Half-time-scale process in DSSC.

Execution process	Time-scale (second)
Electron injection	150 ps
Dye excited to the ground state	12 ns
Dye regeneration	1.8 μ s
e ⁻ (TiO ₂) recombination in dye	5 μ s
e ⁻ (TiO ₂) recombination in electrolyte	2 ms

picosecond. The cocktail dye produced from the leaves of APPG contains a flavonoid pigment that forms a strong link with the semiconductor oxide material. As a result, the electron injection into the TiO₂ material increases, and the electrons reach the ITO transparent substrate. Through an external circuit that makes up the photocurrent, the injected electrons travel in the direction of the counter electrode.

Table 2 describes the experimental results of APPGA and APPGE J_m, V_m, V_{oc}, I_{sc}, FF, and efficiency. A cocktail of dyes was shown to have higher efficiency than the dye that acted separately. From the literature survey and experimental results, the acetone solvent is more efficient compared to ethanol, and the photo-conversion efficiency (PCE) of APPGA and APPGE was 0.6914% and 0.5630%, respectively.

5 Electron kinetics in DSSC

The efficiency enhancement of DSSC not only depends on the electron injection of TiO₂ (Γ_{inj}) but also on losing mechanisms such as

the dye excited state to the ground state (Γ_{S-G}), the recombination of injected electrons with sensitizer (Γ_D), and the redox electrolyte (Γ_R). This cyclic process of electron transfer results in the separation of electrons and holes by Manikandan et al. (2019). In natural-based dyes, the photosynthesis process takes place, which excites the photons and achieves forward electron transfer and loss due to the excited state to the ground state. The essential factors to follow to obtain maximum photo-conversion efficiency are based on organic and inorganic dyes, where the excitation of dye molecules occurs in picoseconds to nanoseconds. Table 3 represents the half-time scale process in DSSC. The rate of electron kinetics varies and depends on the dye used for fabrication and the study of electron injection is employed by ultrafast transient absorption spectroscopy. The injected electron efficiency to the metal oxide semiconductor is derived mathematically using Eq. 4:

$$\eta_{inj} = \frac{\Gamma_{inj}}{\Gamma_{inj} + \Gamma_{S-G} + \Gamma_D + \Gamma_R} \quad (4)$$

a) Regeneration due to electrolytes:

From Figure 8, the natural dye APPG regeneration is sensitized due to iodide in 1.8 μ s at 250 mV, and the inorganic dyes normally take 1 μ s at 600 mV. This results in less energy lost for dye regeneration due to the electrolyte compared to inorganic dyes such as N719 ruthenium dyes.

b) e⁻ TiO₂ recombination in the dye and electrolyte:

In the absence of an electrolyte, the e-TiO₂ recombination in the oxidized dye reacts with the dye at the ground state in 5 μ s, which results in faster electron transfer to the excited state and optimal photo-conversion

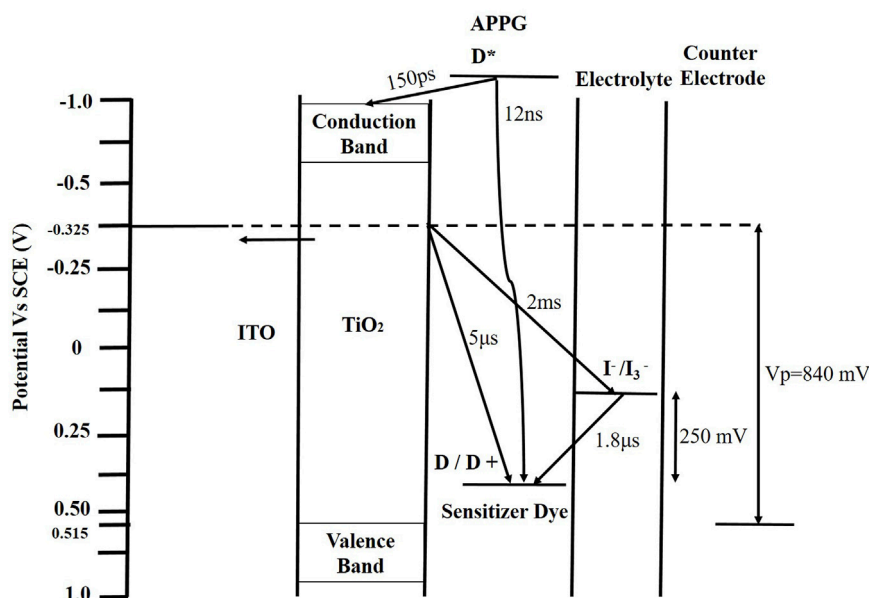


FIGURE 10 Time-scale process of electron kinetics in DSSC.

efficiency. The process of Γ/I_3^- , which is due to the electrolyte for electron release to the dye ground state, excites the HOMO level for continuous current in 2 ms and obtains the highest power conversion efficiency. Furthermore, the solar energy stimulates the dye molecules to an excited state, which increases the efficiency of the DSSC (Al-Alwani et al., 2016).

c) Electron injection:

The excited state HOMO level injects an electron to mesoporous TiO₂ nanoparticles in 150 ps. The process of the liberated electron from nanoparticles transfers to the counter electrode through the load, which in turn, reacts with electrolytes to excite the oxidized dye to form a continuous operation of DSSC. The time scale process is given in Figure 10, which studies the electron kinetic movement of the proposed solar cell.

6 Conclusion

A cocktail dye sensitizer can be made by blending different natural dyes in a 1:1 ratio. This could be an alternative for boosting the band absorption and improves the efficiency of the DSSC. As a result, natural cocktail coloring is the most cost-effective alternative. According to FT-IR analysis, nitrogen-containing compounds predominate in the colors. The conversion efficiency of a cocktail dye made by mixing chlorophyll dyes in a 1:1 ratio with acetone and ethanol was 0.6914% and 0.5630%, respectively. According to the findings, the dye with ethanol solvent has less effect on the cell efficiency due to minimal anchoring with the dye molecules, and acetone shows the maximum power conversion efficiency. The use of TiO₂ nanocomposites in the photoanode can also improve results by combining diverse pigments found in natural dyes. The extraction of the dye from the leaves of APPG is significant in

effectively absorbing solar radiation and converting its energy to the electron for the formation of a photocurrent.

Data availability statement

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

Author contributions

PD: validation, formal analysis, investigation, methodology, and writing—original draft. RM: writing—review and editing.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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