

Research Article

Impact of Solvent Mixtures on Natural Dyes and TiO₂ Nanoparticles for Dye-Sensitized Solar Cells (DSSCs)

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Abstract

Water, as an inorganic solvent, has traditionally been considered inefficient for dye extraction in dye-sensitized solar cells (DSSCs) due to its limited absorbance spectrum, weak bonding with semiconductor surfaces, and rapid dye degradation. Organic solvents like acetone, ethanol, and methanol are typically preferred for enhancing photo-conversion efficiency in DSSCs. This study explores the use of distilled water in combination with ethanol as an alternative solvent mixture to support the global push for low-cost, non-toxic, and eco-friendly DSSCs. Chlorophyll and anthocyanin dyes were extracted from neem leaves (*Azadirachta indica*) and rosella flowers (*Hibiscus sabdariffa*) using distilled water and ethanol. Their absorbance properties were analyzed using UV-Vis spectroscopy, FTIR, and XRD. Among the eight dye samples, the N_e + H_w mixture showed the highest absorbance (3.5 au.) with peaks at 550 nm and 660 nm. FTIR analysis revealed a broad hydroxyl band and a C≡C triple bond trace at 2360 cm⁻¹, indicating enhanced photon harvesting and improved electron transport for higher short-circuit current density. XRD analysis indicated increased crystallinity and a particle grain size of 126.89 nm when adsorbed on a TiO₂-coated FTO substrate. These findings demonstrate enhanced DSSC performance, suggesting improved long-term stability and efficiency.

Keywords

Natural Dyes Mixture, Dye-Sensitized Solar Cells (DSSCs), Distilled Water, Ethanol, Ultraviolet-Visible (UV-Vis) Spectroscopy, Fourier Transform Infrared (FTIR), X-ray Diffraction (XRD)

1. Introduction

Energy, particularly thermal and electrical, is essential for sustaining human life. It has become indispensable as it ful-

fills fundamental daily needs such as cooking, lighting, and heating. Additionally, energy plays a vital role in driving

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technological progress, making life more convenient. Everyday life is nearly impossible without thermal or electrical power. Numerous technological advancements have aimed to address challenges related to energy sources. Solar energy, harnessed from sunlight, is a natural and abundant renewable resource, alongside wind, hydro, biomass, and geothermal energy, providing energy to all living organisms through photosynthesis, essential for growth. Among renewable energy sources, solar energy stands out for its ease of use, limitless supply, quiet operation, and adaptability to various applications [1]. Solar cells come in various types, including p-n junction semiconductor cells, organic solar cells, polymer cells, hybrid solar cells, and dye-sensitized solar cells (DSSCs). DSSCs differ from other types primarily through the use of organic natural dyes as light absorbers and an electrolyte solution forming an electrochemical system [2]. Natural dyes are derived from various plant parts like leaves, bark, flowers, roots, and seeds, each with distinct phytochemical compositions. Over time, several photovoltaic technologies—including organic, inorganic, and hybrid solar cells—have been developed in succession [3].

Dyes play a critical role in dye-sensitized solar cells (DSSCs) by absorbing light and facilitating electron transfer with the semiconductor's conduction band to achieve an optimal quantum yield. In DSSCs, the dye solution functions as a photosensitizer, initiating and sustaining cell operation and performance. The efficiency of natural dye-sensitized solar cells is affected by various factors, including the type of dye sensitizer, extraction solvents, extraction methods, electrolytes, semiconductor materials, transparent conductive oxide (TCO) substrates, and electrodes [4, 5].

Extensive research has been conducted on the effectiveness of single dyes versus dye mixtures, using distilled water and ethanol separately to extract key pigments like anthocyanins and chlorophyll from diverse plant species. Characterization techniques such as UV-Vis spectrophotometry, FTIR, SEM, and EDS have demonstrated the superior performance of ethanol as an extraction solvent over water, as well as the advantages of dye mixtures over single dyes [5-7]. The UV-Vis absorption spectra of dye-loaded photoanodes are notably influenced by the extraction method. Studies comparing mechanical, ultrasonic-assisted, and acidic solvent

extraction (ASE) methods indicate that ASE significantly improves power conversion efficiency when using the same sensitizer [8].

For instance, ethanol mixed with distilled water at a 70:30 ratio for anthocyanin extraction yielded a fill factor of 0.55 and an efficiency of 0.21% [9]. In this study, we focus on using roselle flowers and neem leaves—sources of anthocyanins and chlorophyll, respectively—as photosensitizers. Distilled water and ethanol, along with various combinations of these solvents, were tested to assess their potential for future DSSCs fabrication.

2. Materials and Methodology

Two of the natural plants of interest to this study have been selected mainly because of their abundant supply, low cost and effectiveness in DSSCs fabrication. They are; Neem leaves (*Azadirachta indica*) and Roselle flower (*Hibiscus sardifera*). The dye extracts from these plants are chlorophyll and anthocyanin pigments respectively. Other materials are, distilled water and ethanol.

2.1. Neem Tree (*Azadirachta indica*)

Neem tree is an omnipotent tree and a sacred gift of nature. Neem is mainly cultivated in the Indian subcontinent, and it belongs to the mahogany family called Meliaceae. Neem tree is an incredible plant that has been declared the 'Tree of the 21st Century' by the United Nations (www.unep.org/web/tree-a-day/neem.asp) as it is useful for its multi-various traditional medicine purposes. Neem leaf is very rich in chlorophyll pigment and exists as chlorophyll *a*, its main pigment and chlorophyll *b*, an accessory pigment.

Chlorophyll pigment extract from neem leaves is very stable and does not degrade for a long time. It absorbs light in the visible range spectrum, (peaks at 400 – 490nm and 630 – 700nm) and reflects green color. It is soluble in both organic and inorganic solvents, thus making it a very good source of photo-sensitizer for DSSCs device [5, 10]. The neem fresh leaves with seeds, dry leaves and phytochemical structure are displayed in Figure 1a, 1b, and 1c respectively below.



Figure 1. a: Fresh Neem leaves and seeds. 1b: Dried Neemleaves. 1c: Phytochemical structure of chlorophyll from Neem leaf.

2.2. Roselle Flower (Hibiscus Sabdariffa)

Hibiscus is a genus of flowering plants in the mallow family of Malvaceae. The genus is quite large comprising several hundred species that are native to warm temperate subtropical and tropical regions throughout the world. Members are renowned for their large showy flowers and those species are commonly known as 'hibiscus' or less widely known as rose mallow. Other names include hardy hibiscus, rose of Sharon, and tropical hibiscus (<https://en.m.wikipedia.org>). Hibiscus *Sabdariffa* L commonly called roselleflower, a species of flowering plant

in the genus hibiscus that is native to Africa, most likely West Africa. All true roselle plants, which might be known locally as rosella, red sorrel, Florida cranberry, or Jamaica sorrel, are hibiscus plants, but not all hibiscus plants are roselle. Hibiscus plant is rich in antioxidants such as beta-carotene, vitamin C, and anthocyanin. Antioxidants destroy harmful molecules known as free radicals within the body system (<https://health.clevelandclinic.org>). Phytochemical composition contained in roselle dye is anthocyanin pigment, whose colors range from red to purple. Fresh roselle flower, dry roselle flower, and phytochemical structure are displayed in Figure 2a – 2c.



Figure 2. a: Fresh natural roselle flower; b: Dried roselle flower; c: Phytochemical structure of anthocyanin from roselle flower.

2.3. Experimental Procedure

To prepare chlorophyll extract from neem leaves, 10g of fresh neem leaves were thoroughly washed with distilled water and then air-dried at room temperature. The dried leaves were crushed using a mortar and pestle to form a paste, soaked in 50ml of warm distilled water at 50 °C for 12 hours. The solution was filtered multiple times with Whatman No. 42 filter paper to obtain a pure chlorophyll extract labeled as N_w (Neem in water). Similarly, a paste of 10g of neem leaves was prepared with 50ml of ethanol as the extracting solvent at room temperature, denoted as N_e (Neem in ethanol). The pH levels of both solutions were adjusted to 2.2 using 0.2M of HCl and NaOH, to form a mild pH chosen to optimize DSSCs performance.

For this study, dried roselle flower (commonly called 'zobo') was purchased locally. Each 10g batch was rinsed with distilled water to remove dust, macerated in 50ml of warm distilled water at 50 °C and 50ml of ethanol as extracting solvents in separate containers at room temperature for 12 hours. This temperature range (20 °C to 60 °C) is optimal for anthocyanin extraction, as higher temperatures may lead to thermal degradation, and lower temperatures reduce solubility, producing a lighter-colored extract that decreases DSSCs efficiency [6, 11]. The solutions were then filtered with Whatman No. 42 paper to isolate pure filtrates. While anthocyanin performs best at pH 1.0, the pH was maintained at 2.2 to avoid potential substrate damage from higher acidity [11]. The distilled water and ethanol extracts were denoted as H_w (Hibiscus in water) and H_e (Hibiscus in ethanol), respectively.

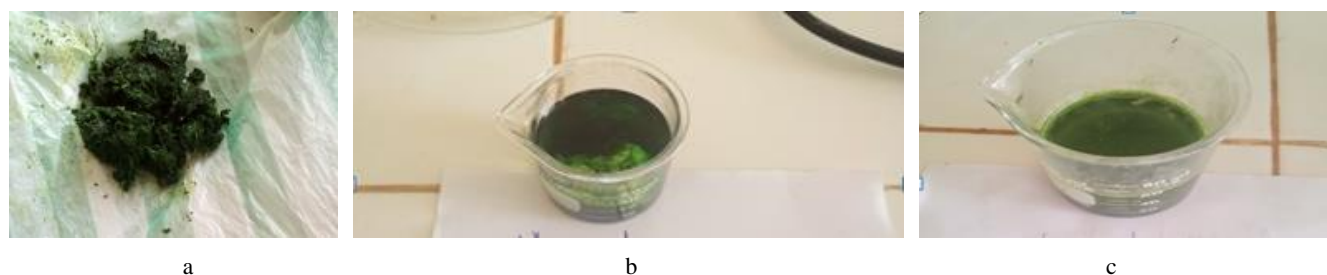


Figure 3. a: Fresh neem leaves paste; b: Chlorophyll extract from ethanol (N_e); c: Chlorophyll extract in water (N_w).



Figure 4. a: Anthocyanin extract from distilled water (H_w); b: Anthocyanin extract from ethanol (H_e).



Figure 5. a: Mixed dye both in ethanol ($N_e + H_e$), b: Mixed dye both in distilled water ($N_w + H_w$), c: Mixed dye in water and ethanol ($N_w + H_e$), d: Mixed dye in ethanol and water ($N_e + H_w$).

Each original extract— N_w , N_e , H_w , and H_e —was used fresh, with all solutions cooled to room temperature before further use. Additionally, eight dye samples were prepared, including four mixed extracts (1:1 ratio) of the four primary dyes, creating the following samples: N_w , N_e , H_w , H_e , ($N_w + H_w$), ($N_e + H_e$), ($N_w + H_e$), and ($N_e + H_w$). Each sample underwent UV-Vis absorbance characterization, with results shown in Figure 6a and 6b, to identify the optimized dye composition that could improve photovoltaic efficiency. Figure 5a, 5b, 5c, and 5d display the various mixtures of chlorophyll and anthocyanin extracts prepared with distilled water and ethanol.

3. Results and Discussion

The following results of various characterizations such as UV-Visible absorbance photospectroscopy, Fourier Transform Infrared (FTIR), and X-ray Diffraction (XRD) were carried out for analysis and discussion.

3.1. Ultraviolet-Visible (UV) Spectroscopy Absorbance Characterization

UV-visible absorbance measurement was carried out using BIOBASE UV-VIS (B-UV1800PC) Spectrophotometer. Figure 6a and 6b show absorbance characteristics of chlorophyll and anthocyanin when extracted in ethanol and distilled water respectively before they are mixed. Each graph shows the absorbance responses and the corresponding peaks of each pigment to light illumination for different extracting solvents. Ethanol seems to be good extracting solvent for both pigments, though better in neem than roselle. The neem displayed two notable peaks in ethanol with the first peak at 440nm which could be attributed to chlorophyll *a* pigment while the second peak occurred at 670nm and attributed to chlorophyll *b* being accessory pigment. The two peaks are typical absorbance wavelength range of chlorophyll pigment. Roselle however has one absorbance peak at 560nm.

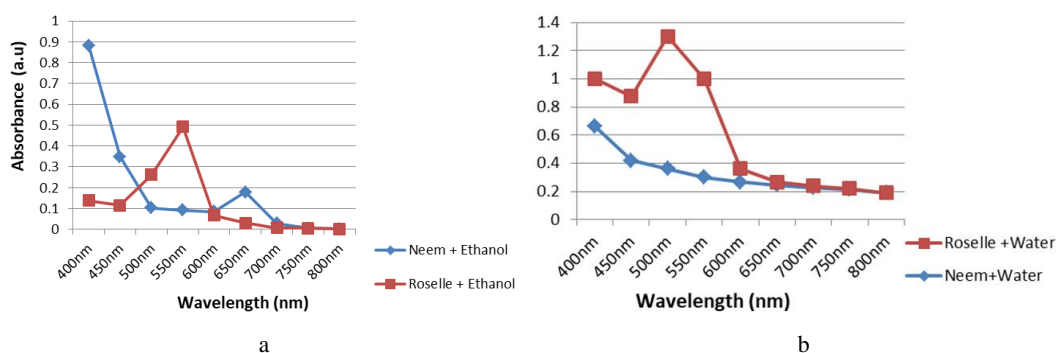


Figure 6. a: Absorbance characteristics of chlorophyll and anthocyanin extracted using ethanol solvent; b: Absorbance characteristics of chlorophyll and anthocyanin extract using distilled water.

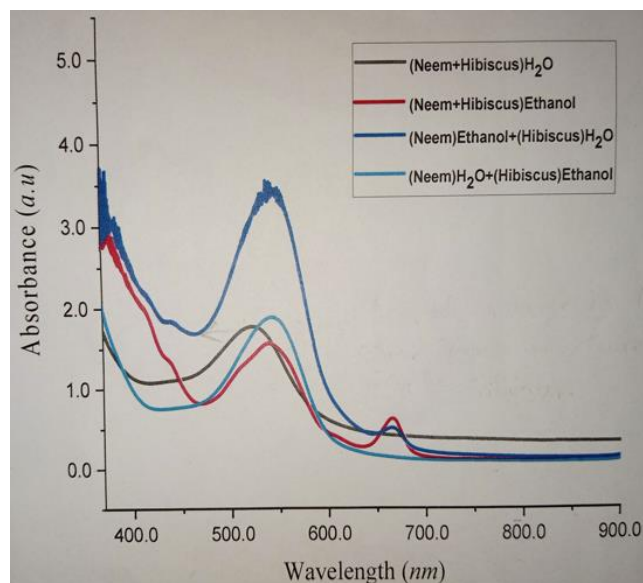


Figure 7. Absorbance Characteristics of Dye Mixtures in different Solvent combinations.

Roselle displayed notable performance over neem in distilled water extraction with an absorbance peak at 530nm while neem peaks at 420nm. It could be inferred that distilled water seemed not to be too good as extraction solvent of chlorophyll pigment from neem leaves, while ethanol served as better extracting solvent for both chlorophyll and anthocyanin pigments. Again, the absorbance values of neem extract with ethanol dropped from 0.88 a.u. to 0.68 a.u., in distilled water. Reverse was the case in roselle extract where the absorbance values increased from 0.5 a.u. in ethanol to 1.3 a.u. in distilled water. The results indicate that anthocyanin extracted with distilled water absorbs more photons which is a requirement for a good sensitizer in DSSCs operation. Similarly, chlorophyll extracted with ethanol also absorbed more light photons than anthocyanin in the same solvent. It is expected that the mixture of these dyes that give maximum photon absorbance would be ideal for DSSCs fabrication as this is clearly revealed in the figure containing dye mixtures below.

Figure 7 compares dye mixture of chlorophyll and anthocyanin extracts from neem leaves and roselle flower in different solvents respectively. All the samples exhibited the first absorption peak almost within the same spectrum range of 530nm and 560nm but having different absorbance values. The second peaks were observed for only dye mixture from neem in ethanol (N_e) and hibiscus in water (H_w), and from neem and hibiscus in ethanol at 660nm wavelength. These peaks are characteristics of typical chlorophyll and anthocyanin especially when extracted in ethanol solvent [6, 12, 13]. Apart from having the highest peak, N_e+H_w also have the broadest band spectrum within the range 470nm and 630nm. This can be attributed to the presence and effect of chlorophyll *b* in addition to chlorophyll *a* when ethanol is used to extract neem leaves. Again, the colors of the dye mixture as in figure

5a, b, c, d could also be responsible for the wider band in the absorbance spectrum [12, 14]. Altogether, the absorbance values of each mixture increased compared with the one obtained in a separate solvent.

Dye mixture of N_e+H_w is a novel one and makes use of the fact that chlorophyll dye extract from neem leaves is more stable in ethanol while roselle flower extract is better in water for long term stability [4, 6]. Generally, dye mixtures aim to widen the wavelength range of light absorption, which can enhance the incident photo-conversion efficiency [5]. Figure 7 displays combined effect of absorption peaks of chlorophyll and anthocyanin in various mixtures of ethanol and distilled water solvents. Before mixture, the absorption peaks of chlorophyll in ethanol were found at wavelengths 430nm and 670nm, while anthocyanin in water peaks at 570nm as shown in Figure 6a and 6b. The combined effect from the broad band spectrum of the mixture dye which incorporates the two peaks in the wider band from 480nm to 630nm with maximum peak at 550nm was shown in Figure 6b. This can be attributed to different solvents used where each of the solvents was affected by the level of polarity of each solvent. The result is to cause a shift in the absorption peak and level of absorbance, which may either, be a longer wavelength (red shift) or shorter wavelength (blue shift) [5].

Although, anthocyanin is more soluble in ethanol and showed a broad absorption band in the visible spectrum region, however, its efficiency dropped drastically when exposed to light illumination. This is due to the photocatalytic decomposition of anthocyanin molecules when in contact with semiconductor oxide (Titanium IV oxide - TiO_2) in the presence of ethanol which makes the color of the photo-anode to become pale within a short time of light illumination. As a result, ethanol is less suitable as an anthocyanin extracting solvent for long term DSSCs operation [6]. In the same vein, though water is not as good as ethanol in the absorption spectrum, however, in this novel mixture, water seems to serve as antidote to ethanol volatility for long time stability of DSSCs operation. This can be seen from the result of UV-Vis absorbance spectroscopy in Figure 7, where this mixture gives the highest absorbance value among the rest of the mixtures. This choice of dye mixture (N_e+H_w) could be used as the optimized dye for DSSCs fabrication for better performance.

3.2. Fourier Transform Infrared (FTIR) Analysis

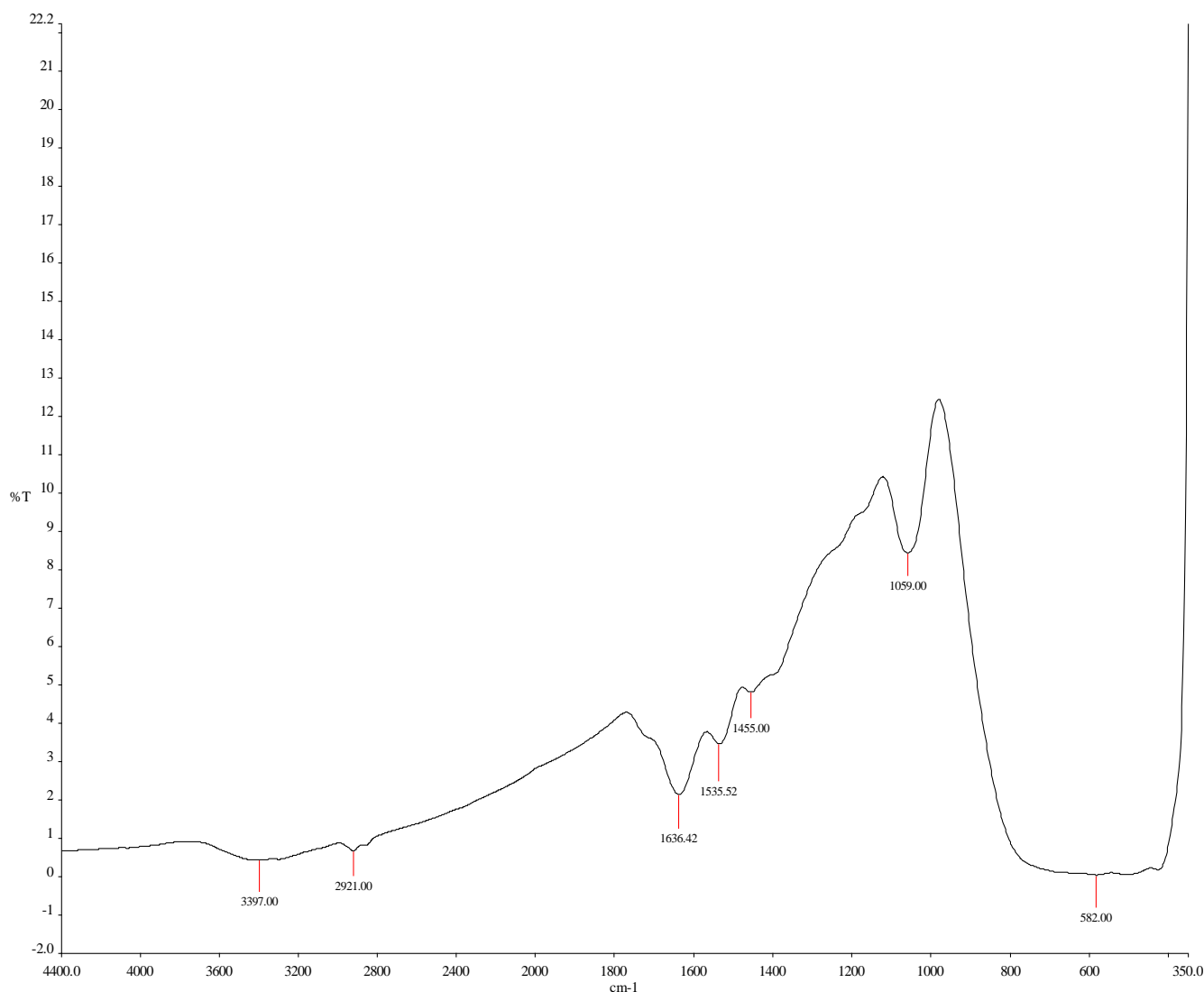
FTIR analysis helps assess a dye's capability to bond effectively with semiconductor nanoparticles, enhancing light-harvesting efficiency. The FTIR spectrum for pure TiO_2 (shown in Figure 8a) reveals several key absorption peaks. A broad band at 3397.00 cm^{-1} indicates the presence of an O-H group, while a peak at 2921.00 cm^{-1} corresponds to a C-H stretch typical of alkanes. These groups promote strong anchoring of the dye onto TiO_2 [14]. Additionally, a peak at

1636.42 cm^{-1} suggests the presence of a carbon double bond within a carboxyl group, and a C-C aromatic ring bond appears at 1535.52 cm^{-1} . A minor peak at 1455.00 cm^{-1} represents a phenolic C-H bond, while peaks at 1059.00 cm^{-1} and 582.00 cm^{-1} indicate the presence of a C-O aromatic ring and an alkyl halide (C-Br bond), respectively [12, 14, 15].

In our optimized dye mixture ($\text{N}_e + \text{H}_w$), FTIR analysis (Figure 8b) reveals various functional groups and bond formations at specific wavenumbers. A broad band, likely due to residual distilled water in the dye mixture, appears around 3376.00 cm^{-1} and represents an O-H stretch. This peak's relative weakness could result from the ethanol and water mixture, which might lower absorbance strength. A peak at 2918.00 cm^{-1} indicates an anthocyanin-associated C-H aromatic ring, with another weak C-H peak at 2853.01 cm^{-1} , likely derived from the chlorophyll in neem leaves [12, 14]. These fall within the alkane bonding category. A peak at 2360.17 cm^{-1} suggests the presence of triple bonds, either

$\text{C}\equiv\text{C}$ or $\text{C}\equiv\text{N}$, corresponding to alkyne or nitrile groups. Chlorophyll *a* appears in the carboxyl group as an aromatic C-C bond at 1595.01 cm^{-1} , possibly contributing to the dye's stability [15].

The C=C aromatic ring bond appears at 1369.00 cm^{-1} [16], while anthocyanin shows up as a C-O stretch at 1032.00 cm^{-1} [12]. A peak at 367.33 cm^{-1} signals an alkyl halide (C-Br bond), indicating the presence of both chlorophyll and anthocyanin in the dye mixture [17]. Comparison of the spectra shows a shift in the O-H group peaks from 3397.00 cm^{-1} in titanium (iv) oxide (TiO_2) to 3376.00 cm^{-1} in the optimized dye, suggesting the dye mixture can capture higher-energy photons when adsorbed onto the TiO_2 surface, thereby enhancing photo-conversion efficiency. Notably, the presence of a rare triple bond ($\text{C}\equiv\text{C}$) around 2360.17 cm^{-1} offers additional photon-harvesting capacity, as the higher energy of triple bonds enables better electron transport onto the TiO_2 surface, supporting improved photo-conversion efficiency.



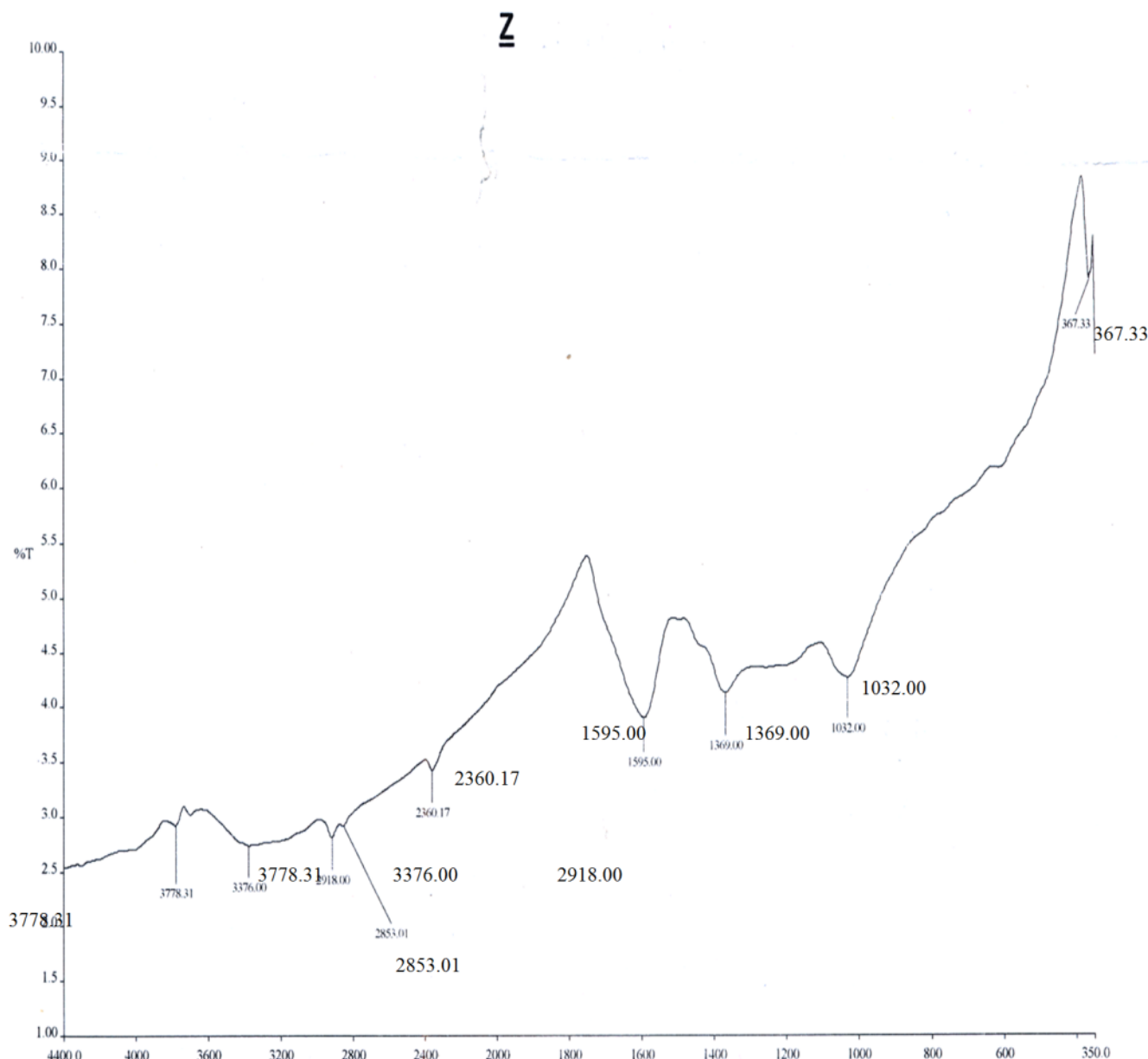


Figure 8. a: FTIR characteristics of pure TiO_2 nanoparticles, b: FTIR characterization of optimized dye mixture of $N_e + H_w$ solution.

3.3. X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis revealed the physical properties of the semiconductor nanoparticles, (TiO_2) and dye sample. XRD measurement was used to determine the crystal structure and phase of TiO_2 nanostructure. The XRD pattern was generated using the Rigaku D/Max-111C X-ray Diffractometer developed by Int. Corp Tokyo, Japan. The equipment was set to produce diffractions at scanning rate of 2° per minute in the range 2° to 50° at room temperature with a CuK α radiation set at 40kV and 20mA. The crystal size was calculated using Scherrer's formula;

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

where D is the crystalline grain size of TiO_2 , k is a dimensionless constant value (0.94), λ is the wavelength of the incident X-ray radiation, β is the full width at half maximum height {rad(FWHM)}, and θ is the angle of diffraction [13].

The average calculated value of the crystalline grain size of TiO_2 was found to be 183.32 nm. This value agrees with the standard size in literature, which is within the range 10nm-300nm with spherical particle having 250nm while cubic particles range between 150 nm and 300 nm in diameter [18].

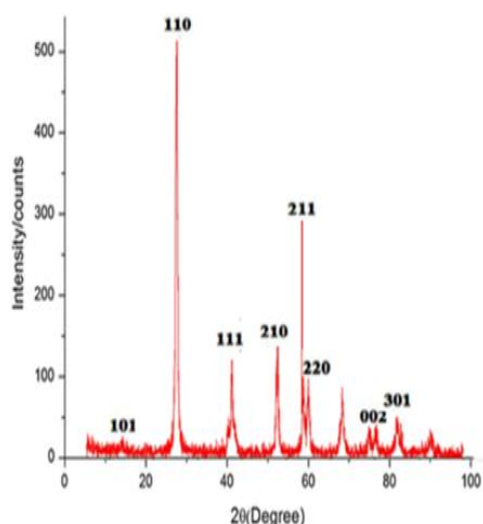


Figure 9. XRD pattern for pure TiO_2 nanoparticles on FTO substrate.

The highest intensity peaks, observed at an angle of 28.58° , strongly indicate the anatase phase of TiO_2 nanoparticles. Other notable peaks with strong intensity appear at angles 40.28° , 46.30° , 59.29° , and 60.185° , all of which align with anatase phases and match the standard spectrum (JCPDS no. 84-1286) [13, 19]. The XRD of the optimized dye solution, as shown in Figure 10a, was compared with the XRD pattern after dye adsorption on the TiO_2 substrate (Figure 10b). A slight decrease in the peak intensity upon adsorption could indicate a chemical change due to intermolecular interactions between the dye and TiO_2 molecules [20]. However, the substrate dye displays sharper peaks and narrower band gaps with a slight increase in other peak intensities compared to the optimized dye alone, as seen in Figures 10a and 10b, respectively. This suggests improved crystallinity and a reduction in particle grain size [21].

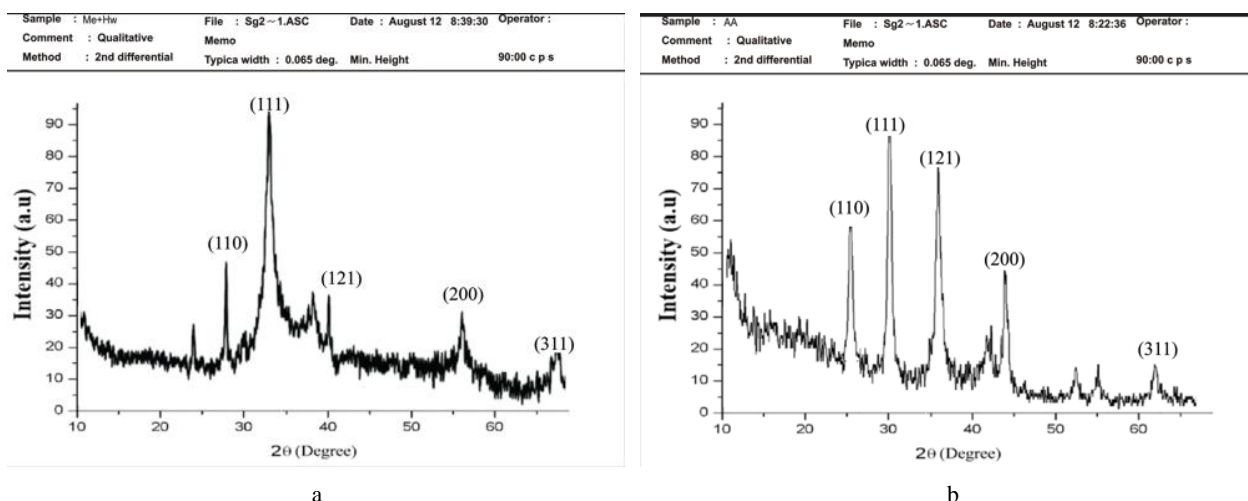


Figure 10. a: XRD spectra of optimized dye solution; b: XRD spectra of Optimized dye on TiO_2 substrate.

The spectrum also exhibits strong diffraction peaks at various angles, including 25.37° , 30.42° , 35.66° , and 44.25° , with the most intense peak at 30.42° . A shift in the highest peak from 35.24° in pure TiO_2 to 30.42° in the TiO_2 adsorbed optimized dye pattern was observed, maintaining the same intensity. The shift is likely result from intermolecular bonding between the TiO_2 and the dye molecules [12]. The average crystalline grain size of the substrate containing TiO_2 and the adsorbed optimized dye was approximately 126.89 nm, compared to 183.82 nm for pure TiO_2 . This reduction in grain size could be attributed to the interaction between the dye and TiO_2 molecules during adsorption. Smaller particle sizes imply a larger band gap, allowing absorption at higher energies, leading to a shift towards shorter wavelengths, known as a blue shift.

4. Conclusion

The effects of distilled water and ethanol as extraction solvents were investigated on a 1:1 blend of natural dyes from neem leaves and roselle flowers, aimed at optimizing light absorption for dye-sensitized solar cells (DSSCs). UV absorbance measurements revealed that the dye mixture ($N_e + H_w$) achieved the highest absorbance at 3.5 a.u., outperforming individual dye samples. FTIR analysis showed a broad hydroxyl band and a trace of a triple $\text{C}\equiv\text{C}$ bond at a wave-number of 2360.17 cm^{-1} , indicating enhanced photon capture and electron transfer, which could lead to higher short-circuit current density. XRD results demonstrated improved crystallinity and a grain size of $126.2\text{ }\mu\text{m}$ upon adsorption on TiO_2 -coated FTO substrates. Characterization techniques,

including UV-Vis spectroscopy, FTIR, and XRD, confirmed that this dye combination provides superior incident photo-conversion efficiency compared to individual dyes by balancing each dye's unique properties. This blend also showed promise for extended stability and efficiency, crucial for the commercial viability of DSSCs. Incorporating suitable additives may further enhance DSSCs stability and performance, suggesting potential directions for future research [22].

Abbreviations

N _w	Neem Leaves Extracted in Water
N _e	Neem Leaves Extracted in Ethanol
H _w	Hibiscus Extracted in Water
H _e	Hibiscus Extracted in Ethanol
DSSCs	Dye-Sensitized Solar Cells
FTIR	Fourier Transform Infrared
XRD	X-Ray Diffraction
UV-Vis	Ultraviolet Visible Light
TCO	Transparent Conducting Oxide
TiO ₂	Titanium Dioxide or Titanium (iv) Oxide
SEM	Scan Electron Microscopy
EDS	Electron Diffraction Spectroscopy
ASE	Acidic Solvent Extraction
FWHM	Full Width at Half Maximum Height
O-H	Oxygen – Hydrogen Bond
C-C	Carbon – Carbon Bond
C=C	Carbon – Carbon Double Bond
C≡C	Carbon – Carbon Triple Bond
C≡N	Carbon – Nitrogen Triple Bond
C – O	Carbon – Oxygen Bond
C-Br	Carbon – Bromine Bond
C-H	Carbon – Hydrogen Bond
a.u	Absorbance Unit
Nm	Nanometer

Conflicts of Interest

The authors declare no conflicts of interest.

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