

Solvent selection for anthocyanin dye extraction from *Kigelia Africana* and *Hibiscus sabdariffa* for dye sensitized solar cells

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ABSTRACT

The main flavonoid pigment in the petals of *Kigelia Africana* and the calyx of *Hibiscus sabdariffa* is anthocyanin, responsible for the vibrant red, maroon, and purple hues in flowers. This pigment can modulate incident light on flowers, prompting its selection for detailed investigation. TiO₂ nanostructures were synthesized using a one-step hydrothermal method, revealing the formation of nanorods and a single-phase rutile structure through FESEM and XRD analyses, respectively. The study aimed to assess the impact of various solvents on the extraction of natural dyes, which were subsequently sensitized on TiO₂ photoanodes for DSSC applications. Four solvents—water, water with HCl, ethanol, and citric acid—were employed to extract natural dyes from *Kigelia Africana*'s petals and *Hibiscus sabdariffa*'s calyx. Notably, dyes extracted with citric acid demonstrated promising results. The conversion efficiency of DSSCs fabricated with *Kigelia Africana* dye and *Hibiscus sabdariffa* dye, extracted using citric acid as the solvent, was found to be 0.87 % and 0.92 %, respectively. The implications of these findings are discussed.

1. Introduction

The utilization of natural pigments in dye-sensitized solar cells (DSSCs) has garnered significant attention due to their eco-friendly and sustainable characteristics. Natural pigments, often derived from plant sources, offer an alternative to synthetic dyes in enhancing light absorption for solar energy conversion. Anthocyanins, compounds classified as glycosides of phenyl-2-benzopyrylium or flavylum salts, have garnered scientific attention due to their diverse structures. Approximately 247 anthocyanin pigments have been documented, with 17 distinct structures reported so far [1,2]. Categorization is based on the number of sugar molecules, yielding monosides, biosides, trisides, etc. The compound count can be expanded by considering sugar diversity and potential glycosylation structural points [3].

In the realm of research, anthocyanins hold prominence in Dye-Sensitized Solar Cells (DSSCs) owing to their capacity to efficiently absorb and be energized by light. This results in broad absorption in the visible region, with minimal energy requirements for transitions from

Highest Occupied Molecular Orbital (HOMO) to Lowest Unoccupied Molecular Orbital (LUMO) [4,5]. Anthocyanins, featuring carbonyl and hydroxyl groups, exhibit facile binding to Ti (IV) sites, facilitating electron excitation and transfer to the conduction band of TiO₂ [6,7]. While stable under acidic conditions, anthocyanins are susceptible to degradation and color loss influenced by factors like structure, pH, co-pigmentation, temperature, enzymatic activity, and the presence of metal ions.

This study focuses on synthesizing two distinct DSSC configurations, both configurations employ TiO₂ nanorods as the photoanode, Pt-coated counter electrode, and iodide-triiodide electrolyte. Sensitizer plays an important role in determining the performance of DSSC. There is a need for selecting a right sensitizer and therefore natural dyes with anthocyanins as the major pigments are proved to be versatile in improving its performance. In our study, we have addressed the use of two different dyes, (i) Natural dye extracted from a maroon and velvety flower "*Kigelia Africana*". This dye has been reported in the literature for the first time in fabrication of DSSC and the results are encouraging (ii)

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Natural dye extracted from the calyx of a soft red flower “*Hibiscus Sabdariffa*”. Both the dyes are found to show good colour stability (visual examination) with time when compared to few other reported dyes that causes browning or formation of precipitates in the solution. The chemical constituents of *Kigelia Africana* and *Hibiscus sabdariffa* have been previously reported in our reports [8,9].

The investigation also delves into improving the stability of anthocyanins from *Kigelia Africana* and *Hibiscus sabdariffa* by studying the impact of the extracting solvent on the stability of the dyes. Hence four different solvents viz water, water-HCl, ethanol and citric acid were used in extraction of the dyes. The study concludes by discussing the performance of the fabricated natural dye-based DSSCs.

2. Experimental details

2.1. Extraction of natural dye using different solvents

For extraction of dyes, 25 g of *Kigelia Africana* petals and 25 g of *Hibiscus sabdariffa* calyx were washed and dried. They were then cut into small pieces and soaked in 30 mL of different solvents (Water, Water-HCl, Ethanol and Citric acid) for 24 h in dark. Both the extracts were filtered and used directly for sensitizing the TiO₂ film.

2.2. Synthesis of TiO₂ nanorods by hydrothermal method and sensitization of TiO₂ film

Fluorinated tin oxide (FTO) substrates were cleaned ultrasonically using soap solution, distilled water, acetone and ethanol for 10 min and dried. Synthesis of TiO₂ nanorods by hydrothermal method and sensitization of TiO₂ film using the natural dyes was carried out according to

our previous reports [10]. Fig. 1 displays the photograph of flower/-calyx, extracted dye and TiO₂ sensitized with dye

3. Results and discussions

3.1. Characterization of TiO₂ films

Fig. 2(a), accompanied by an inset, presents a top-view Field Emission Scanning Electron Microscopy (FESEM) image depicting TiO₂ nanorods cultivated at 170 °C for a duration of 4 h. The images unveil densely packed, well-aligned nanorods of uniform dimensions. The average diameter and length of the nanorods measure 220 nm and approximately 2 μm, respectively. Notably, the top surface of the nanorods exhibits a comparatively rough texture with discernible step edges, in contrast to the smoother sides of the rods.

The observed characteristics suggest that the initiation and sustained growth of the nanorods occur through the incremental addition of Titanium (IV) isopropoxide precursor. The square-shaped top surface observed in the image supports the anticipated growth of a tetragonal crystal. Quantitative elemental analysis, as depicted in Figure S1 of the supporting information, reveals the prevalence of titanium and oxygen as the primary elements in the sample. The atomic percentage of titanium (37.73) and oxygen (62.27) indicates a nearly stoichiometric composition, with a Ti:O ratio of 1:2.

In Fig. 2 (b), presence of lattice fringes with interplanar spacing $d_{110} = 0.33$ nm corresponds to the rutile phase of TiO₂. The selected area electron diffraction (SAED) pattern (Fig. 2 (c)) shows the presence of sharp spots indicating that the samples are highly crystalline in nature. Fig. 2 (d) shows the XRD plot of TiO₂ nanorods and it is clearly seen that all the peaks observed at 2θ positions of 27.39°, 36.08°, 39.10°, 41.3°,

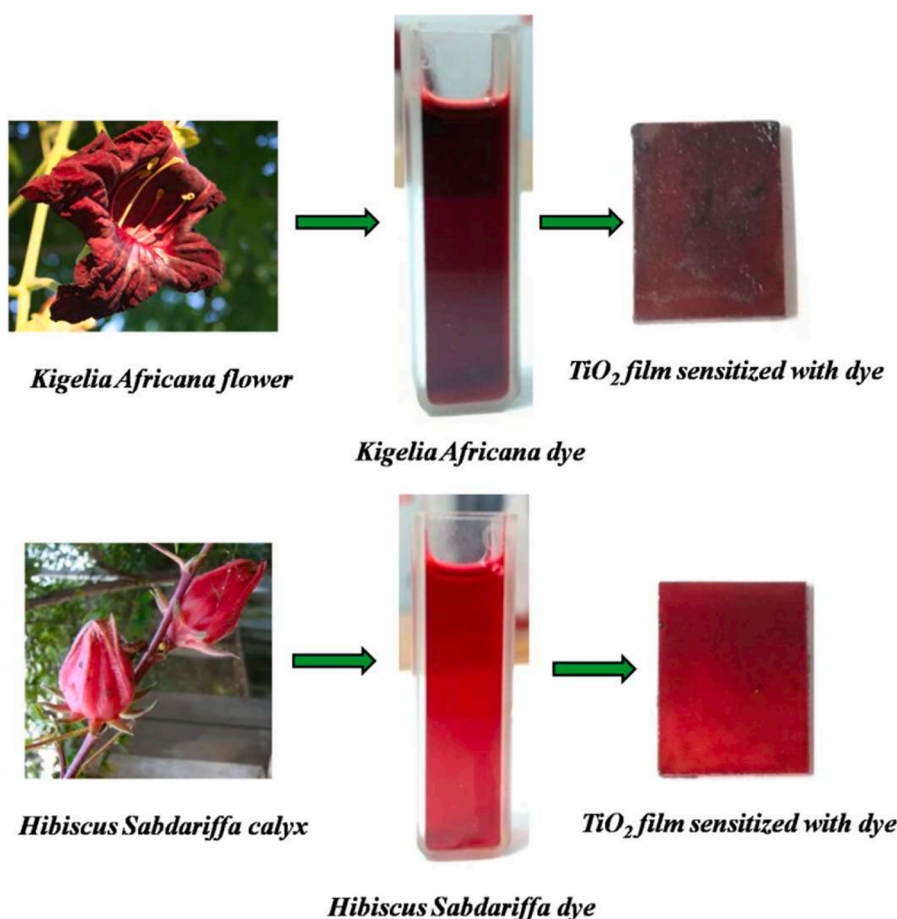


Fig. 1. Photograph of flower/-calyx, extracted dye and TiO₂ sensitized with dye.

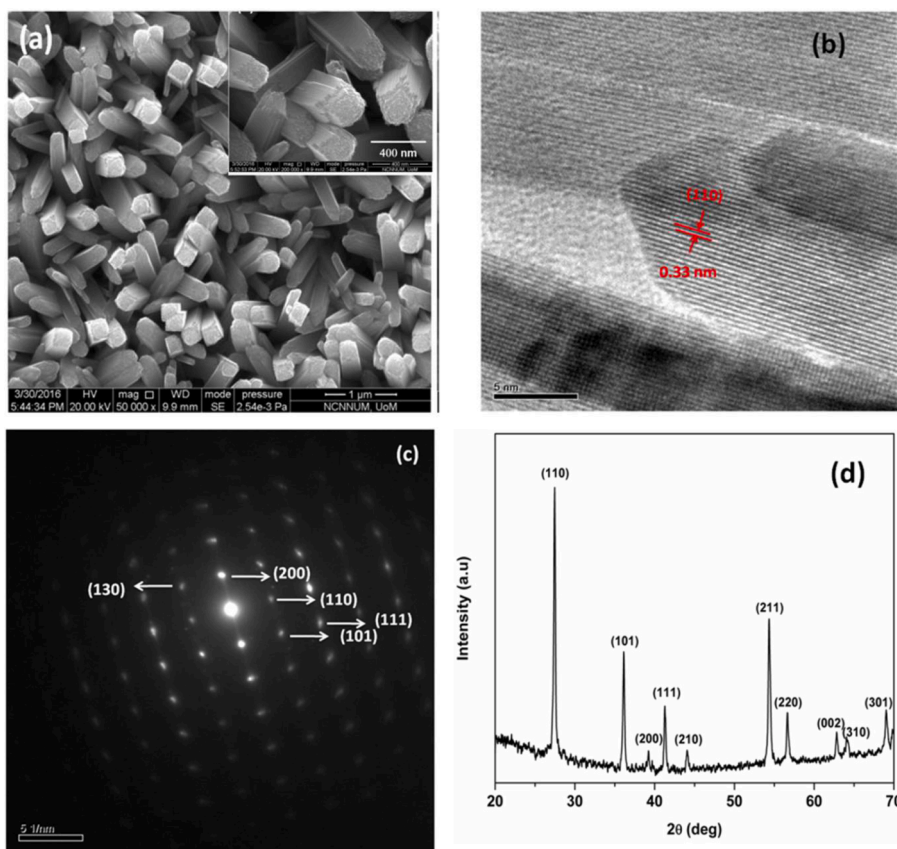


Fig. 2. (a) FESEM image (b) Lattice fringe pattern (c) SAED pattern (d) X-ray diffractogram of TiO_2 nanorods.

43.97°, 54.36° and 56.68° corresponds only to the tetragonal rutile phase TiO_2 (JCPDS No. 089-0555). No additional peaks corresponding to other phases of TiO_2 such as anatase or brookite were observed.

3.2. Effect of solvent on dye extraction

Anthocyanins are glycoside molecules with hydroxyl sugar attached to them and so choice of polar solvents is essential for its extraction of anthocyanins. Four different solvents; namely water, water-HCl, ethanol and citric acid were chosen for the extraction of dye from the flowers of *Kigelia Africana* and calyx of *Hibiscus sabdariffa*. Because of the

instability of the compounds, dye extraction was carried out at temperature below 50 °C, to prevent decay of pigments. In order to prevent the pigment degradation, weak acid such as formic acid or citric acid can be used. Citric acid is considered as a safe solvent as it is used mainly in food industries [11]. Citric acid is less corrosive than HCl and helps in stabilizing the dye. In general, the degradation of the dye is severely due to the polyphenoloxidase [12]. This polyphenoloxidase degrades the anthocyanin and phenolic content to a greater extent. The usage of citric acid as solvent extensively inhibits the role of polyphenoloxidase in de-pigmentation and browning of flowers and fruits [13]. Hence we have concentrated on the use of citric acid as the solvent for extracting

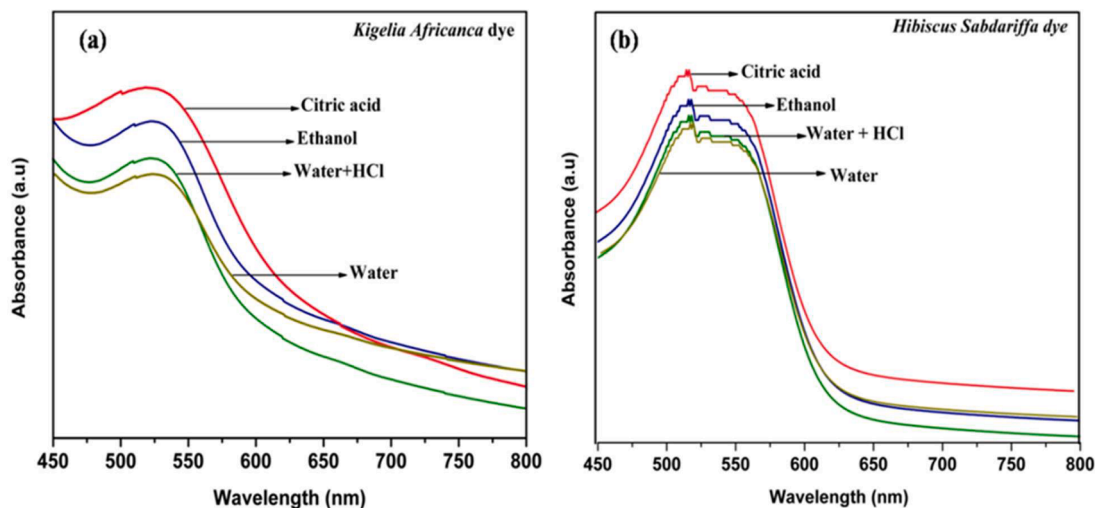


Fig. 3. Absorption spectra of *Kigelia Africana* and *Hibiscus sabdariffa* dye extracted with various solvents.

the dyes.

3.2.1. Absorption spectra

The absorption spectra of *Kigelia Africana* and *Hibiscus sabdariffa* dye extracted with various solvents are shown in Fig. 3.

The presence of anthocyanin can be confirmed from the peak maxima observed around 520 nm [14]. It is evident that both the dyes extracted with citric acid as solvent are found to show good absorbance, confirming more anthocyanin content in them. The total amount of anthocyanin present in both sample solutions is calculated and discussed in the next section.

3.2.2. Total anthocyanin content

The quantification of anthocyanin content in the dye employed the pH differential method, as outlined by Giusti and Wrolstad [15]. This method revolves around assessing the anthocyanin levels at two distinct pH values: pH 1.0, where anthocyanins manifest in a colored oxonium form, and pH 4.5, where the anthocyanins transition into a colorless hemiketal form. To execute this analysis, samples were dissolved in two buffer solutions adjusted to pH 1.0 and pH 4.5. The absorbance of these samples was measured within the wavelength range of 520 nm to 700 nm. The buffer solution at pH 1.0 comprised potassium chloride (0.025 M), while sodium acetate (0.4 M) was used for pH 4.5. The pH adjustments to 1.0 or 4.5 were accomplished using hydrochloric acid (HCl). The initial determination involved establishing the quantity of the sample to be dissolved, i.e., the dilution factor. A small fraction of the sample was diluted in the buffer solution, and absorbance was subsequently measured. Dilutions with buffers continued until the absorbance at 520 nm wavelength fell < 1.2 absorbance units (AU). The selection of the 520 nm wavelength for absorbance measurement was based on its representation as the midrange for various anthocyanin pigments [14], displaying maximal absorption characteristics across the spectrum.

Measurement of absorbance at a wavelength of 700 nm is for haze. Now, the samples were separately diluted with both the buffer solution and absorbance was measured at 520 nm and 700 nm. The difference in absorbance was calculated using the given equation

$$A = (A_{520nm} - A_{700nm})_{pH=1.0} - (A_{520nm} - A_{700nm})_{pH=4.5} \quad (1)$$

The total anthocyanin content was then determined using the following equation and the values are plotted for both the dyes (Fig. 4)

$$TAC(mg/l) = \frac{A \times MW \times DF \times 1000}{\xi \times L} \quad (2)$$

Where A is the absorbance

MW is the molecular weight of cyanidin-3-glycoside (449.2 g/mol)

DF is the dilution factor

L is the standard path length (1 cm)

ξ is the molar extension coefficient (26,900 L mol⁻¹ cm⁻¹ for cyanidin-3-glycoside)

Factor 1000 is to convert from g to mg

The quantification of total anthocyanin content was conducted through the application of Eq. (2), and the resultant values are graphically represented for both dye samples in Fig. 4. Analysis of the plotted data unequivocally reveals that the total anthocyanin content within the dye solutions is significantly higher when the samples are extracted utilizing citric acid as the solvent.

3.2.3. pH stability

Anthocyanins are easily susceptible to pH changes and undergo structural changes on varying the pH namely the quinonoidal base, the flavylium cation, the carbinol and the chalcone. The sample solutions with pH values 1.1, 2.2, 3.8, 5, 6, 7, 8, 10 were prepared and stored in refrigerator (see Figure S2 in supporting information). The amount of anthocyanin present in the samples at different pH was calculated at regular intervals of days as 0th day, 5th day, 10th day, 15th day, 20th day, 25th day and 30th day. Residual rate of anthocyanin (RR) present was calculated using the formula

$$\%RR = \frac{TAC}{TAC_i} \times 100\% \quad (3)$$

where TAC is the amount of anthocyanin present after the specified day and TAC_i the amount of anthocyanin present initially.

Fig. 5 shows the residual rate of anthocyanin present in the sample solutions on varying the pH values. From the graph it can be seen that both the dyes show decrease in residual anthocyanin rate (% RR) with time. Sample solutions at pH 7, pH 8, pH 9 shows fast decrease in residual anthocyanin content within a period of 5 days as compared to the remaining solutions. Moreover, the percentage of anthocyanin present in the samples prepared at more basic conditions decrease faster and reaches a very low value with increase in time. Similar results have been reported by Prabavathy et al. [16] in their study on extraction and stability of anthocyanin from the petals of *Caesalpinia pulcherrima*.

Therefore, samples prepared at acidic conditions seem to possess considerable rate of anthocyanin and are stable even after one month. Sample solutions at pH 2.2, i.e., samples extracted with citric acid shows promising result and nearly 85 % of anthocyanin is still present in the solution even after one month of time.

3.2.4. Color intensity

Color intensity of anthocyanin was determined by the method as

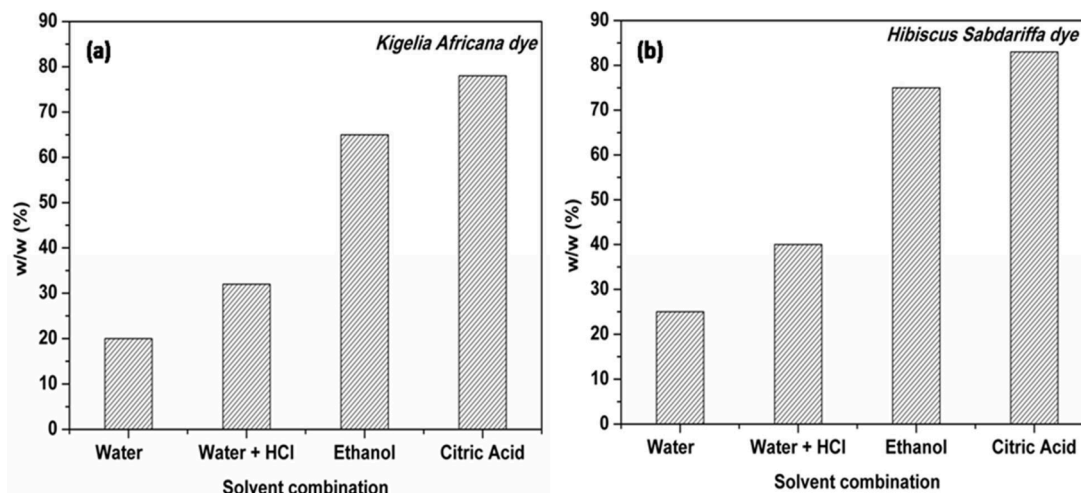


Fig. 4. Total anthocyanin content vs different extracting solvents in (a) *Kigelia Africana* dye (b) *Hibiscus sabdariffa* dye.

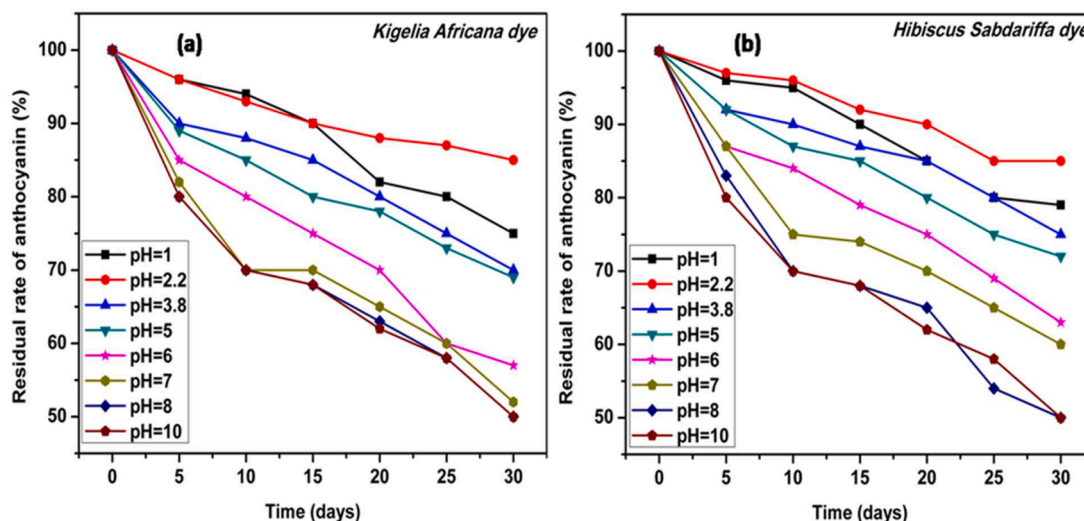


Fig. 5. Residual rate of anthocyanin present in the sample solutions (a) *Kigelia Africana* dye (b) *Hibiscus sabdariffa* dye on varying the pH values.

suggested by food and agriculture organization in 1984. Required amount of citric acid and sodium hydrogen phosphate was mixed in a 25 mL volumetric flask and adjusted till pH of the buffer solution turns 3.0. Then the absorbance at 520 nm was measured by diluting 2 mL of sample in 10 mL of buffer solution. Citric acid-sodium hydrogen phosphate buffer solution was used as blank for measuring the absorbance. The same process was repeated from 0 to 30 days with an interval of 10 days.

Fig. 6 shows the colour intensity of the dyes over a period of 30 days on extracting with different solvents. From the graph it can be seen that the absorbance of both the natural dyes extracted with citric acid as solvent is found to be high. The absorbance then slowly decreases for both the dyes from 0 to 30 days proving the degradation of anthocyanin with time.

3.2.5. Light and thermal stability

In the context of light stability investigations, the sample solutions underwent a regimen of exposure to both dark and light conditions over a period of 30 days, all conducted at room temperature. The residual anthocyanin content was quantified at intervals of 5 days throughout this duration. The results of these experiments are graphically depicted in Fig. 7.

Thermal stability studies were conducted on *Kigelia Africana* dye and *Hibiscus sabdariffa* dye. The sample solutions were subjected to heating

for a duration of 10 min within a water bath, with incremental temperature settings ranging from 30 °C to 100 °C. Subsequently, the absorbance of the solutions was determined following complete cooling. The observed absorbance values provide insights into the thermal stability profiles of the respective dyes under varying temperature conditions.

Dyes obtained from *Kigelia Africana* and *Hibiscus sabdariffa*, extracted using citric acid as a solvent and stored in the absence of light, exhibited promising stability. The residual anthocyanin content remained relatively unchanged for up to 15 days, with approximately 90 % of the initial anthocyanin concentration persisting even after a 30-day duration. Conversely, when alternative solvents were employed for dye extraction, a faster degradation rate was observed, resulting in only 60–80 % of the initial anthocyanin content remaining after 30 days.

In contrast, when the dyes were subjected to light exposure, a gradual reduction in the residual anthocyanin content was observed from the outset. Consequently, through a comparative analysis of both storage conditions, it is deduced that the long-term preservation of anthocyanin integrity is more effective in the absence of light. This observation aligns with findings reported by Contreras-Lopez et al. [17] in their investigation of the light-induced stability of anthocyanins extracted from *Rubus fruticosus*.

Fig. 8 illustrates the outcomes of thermal stability investigations conducted on the dyes extracted from *Kigelia Africana* and *Hibiscus*

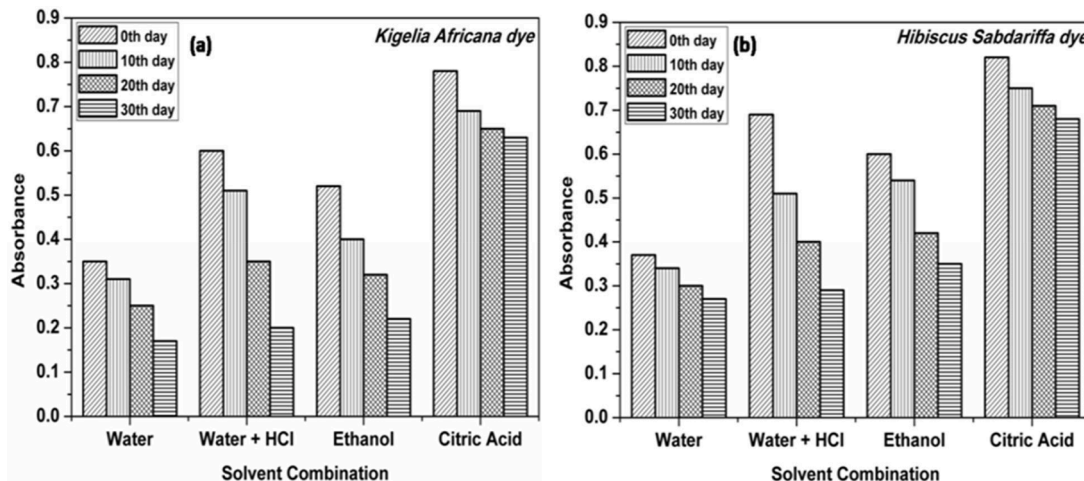


Fig. 6. Colour intensity of (a) *Kigelia Africana* dye (b) *Hibiscus sabdariffa* dye over a period of 30 days on extracting with different solvents.

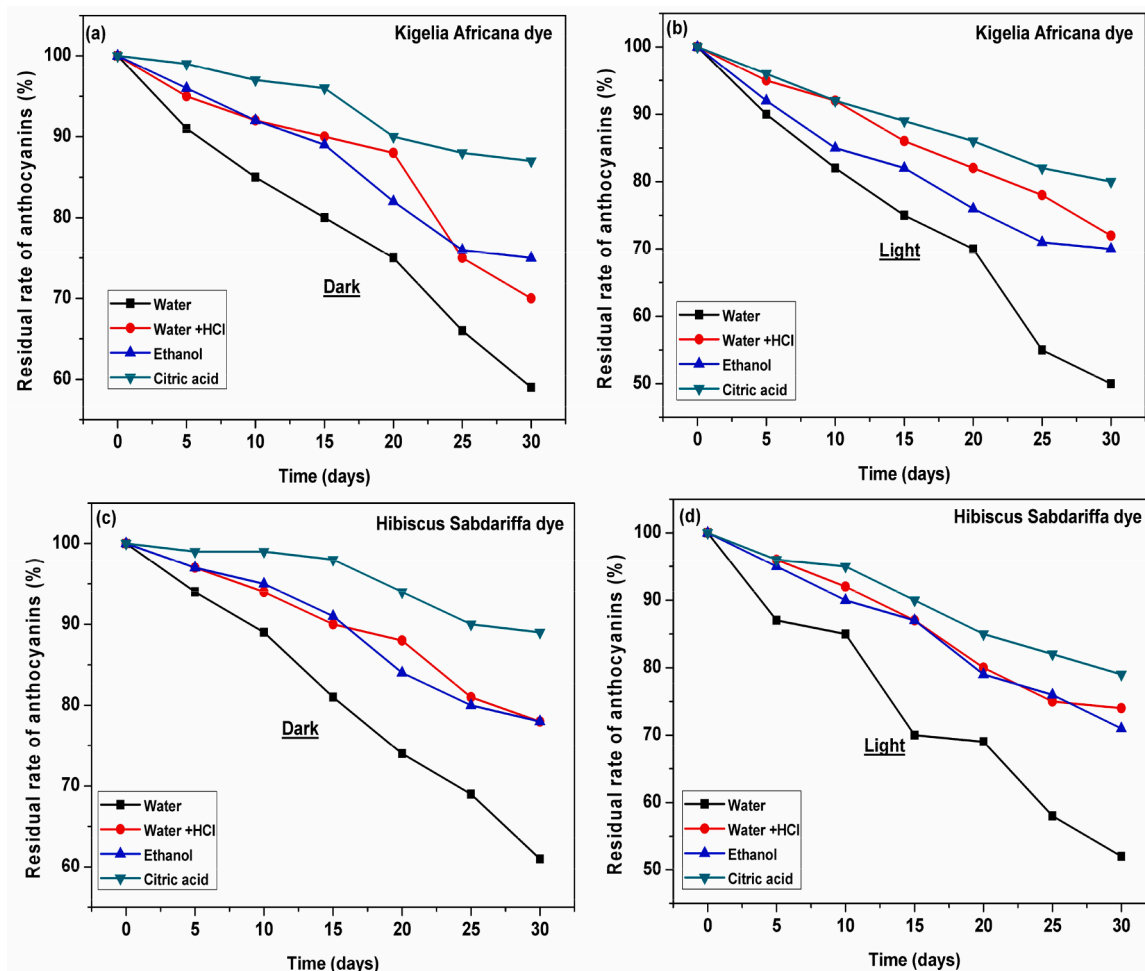


Fig. 7. Light stability studies under dark and light conditions (a & b) *Kigelia Africana* dye (c & d) *Hibiscus sabdariffa* dye.

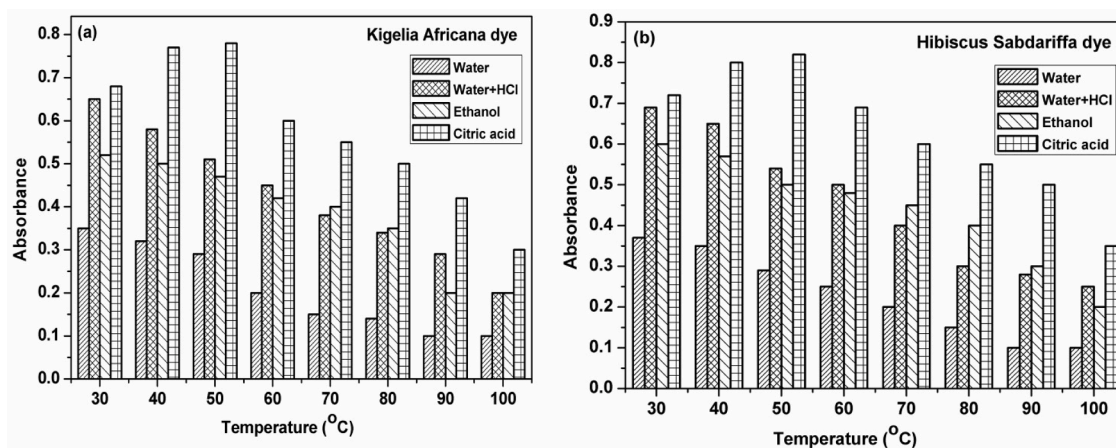


Fig. 8. Thermal stability studies of (a) *Kigelia Africana* dye (b) *Hibiscus sabdariffa* dye.

sabdariffa. The absorbance profiles of the dye solutions obtained through various extraction solvents, namely water, water with HCl, ethanol, and citric acid, were monitored during the gradual heating of the samples from 30 °C to 100 °C.

In the case of water, water with HCl, and ethanol extractions, a progressive decrease in absorbance was observed, indicative of thermal degradation. The presence of acid or acidified water during the extraction process contributed to the degradation, leading to the hydrolysis of

acyl groups and co-pigments, consequently compromising the stability of the dye.

Remarkably, the dye solutions extracted with citric acid as the solvent exhibited a distinct behavior. The absorbance of anthocyanin steadily increased as the samples were heated from 30 °C to 50 °C, reaching a maximum at the latter temperature. Subsequent heating beyond 50 °C resulted in a slight reduction in anthocyanin absorbance at 60 °C, followed by a consistent decline. Comparable findings were

reported by Yusoff et al. [18] in their investigations on the temperature stability of pigments from tropical plants.

The observed enhancement in thermal stability at 50 °C for anthocyanin extracted with citric acid is attributed to the inhibitory effect of citric acid on the formation of polyphenoloxidase. This inhibition mitigates the degradation of anthocyanin. Conversely, the decline in absorbance beyond 50 °C may be attributed to the formation of unstable chalcone-type structures, leading to the distortion of anthocyanins. Therefore, these results underscore the favorable thermal stability of anthocyanin extracted with citric acid at 50 °C, offering valuable insights into optimizing extraction methodologies for applications requiring robust thermal performance.

3.3. Fabrication of natural dye sensitized TiO₂ nanorods based solar cells

Finally, DSSC (active area of the cell is 1 cm²) was formed with natural dye-sensitized TiO₂ nanorods grown on fluorine-doped tin oxide as photoelectrode, with standard iodide-triiodide electrolyte and platinum-coated glass counter electrode. Fig. 9(a) shows the schematic of the fabricated DSSC. J-V measurements were carried out using PECCELL solar simulator (with an irradiance of 100 W/m²) interfaced with Keithley 2401 source meter and software to obtain the fabricated solar cell characteristics.

Fig. 9 (b & c) presents the J-V characteristics of dye-sensitized solar cells (DSSCs) employing natural dyes extracted from *Kigelia Africana* and *Hibiscus sabdariffa*. Table 1 provides a comprehensive summary of key parameters, including open circuit voltage (V_{oc} , V), short circuit current density (J_{sc} , mAcm⁻²), fill factor (FF), and cell efficiency (η ,%).

Analyzing the data reveals that the DSSC sensitized with *Kigelia Africana* dye extracted using water as a solvent attains an efficiency of 0.66 %, accompanied by V_{oc} of 0.37 V, J_{sc} of 3.9 mAcm⁻², and FF of

Table 1

Consolidation of all the photovoltaic parameters of TiO₂/natural dye sensitized DSSC.

TiO ₂ /natural dye based DSSC	<i>Kigelia Africana</i> dye				<i>Hibiscus sabdariffa</i> dye			
	J_{sc}	V_{oc}	FF	η	J_{sc}	V_{oc}	FF	η
Water	3.9	0.37	0.457	0.66	4.0	0.4	0.406	0.65
Ethanol	4.3	0.41	0.440	0.71	4.1	0.41	0.464	0.77
Water + HCl	4.6	0.42	0.413	0.80	4.5	0.42	0.444	0.83
Citric acid	5.0	0.48	0.362	0.87	4.8	0.45	0.428	0.92

Foot note: J_{sc} in mAcm⁻², V_{oc} in V, η in%.

0.457. In the case of *Hibiscus sabdariffa*, the DSSC exhibits a V_{oc} of 0.4 V, J_{sc} of 4.0 mAcm⁻², FF of 0.406, resulting in an efficiency of 0.65 %. Shifting focus to natural dyes extracted with ethanol as a solvent, *Kigelia Africana*-sensitized DSSC achieves a V_{oc} of 0.41 V, J_{sc} of 4.3 mAcm⁻², FF of 0.440, and an efficiency of 0.71 %. In comparison, the *Hibiscus sabdariffa*-sensitized DSSC yields a V_{oc} of 0.41 V, J_{sc} of 4.1 mAcm⁻², FF of 0.464, and an efficiency of 0.77 %. Ethanol, possessing both polar and nonpolar components conducive to hydrogen bonding, proves effective in anthocyanin extraction, enhancing DSSC efficiency compared to water as a solvent.

Furthermore, DSSCs sensitized with *Kigelia Africana* and *Hibiscus sabdariffa* dyes extracted using acidified water display V_{oc} values of 0.42 V, J_{sc} values of 4.6 mAcm⁻² and 4.5 mAcm⁻², FF values of 0.413 and 0.444, and efficiencies of 0.80 % and 0.83 %, respectively.

Notably, citric acid, a weak acid, emerges as a promising solvent. DSSCs sensitized with *Kigelia Africana* and *Hibiscus sabdariffa* dyes extracted using citric acid exhibit efficiencies of 0.87 % and 0.92 %, with corresponding V_{oc} values of 0.48 V and 0.45 V, J_{sc} values of 5.0 mAcm⁻² and 4.8 mAcm⁻², and FF values of 0.362 and 0.428. While acids are

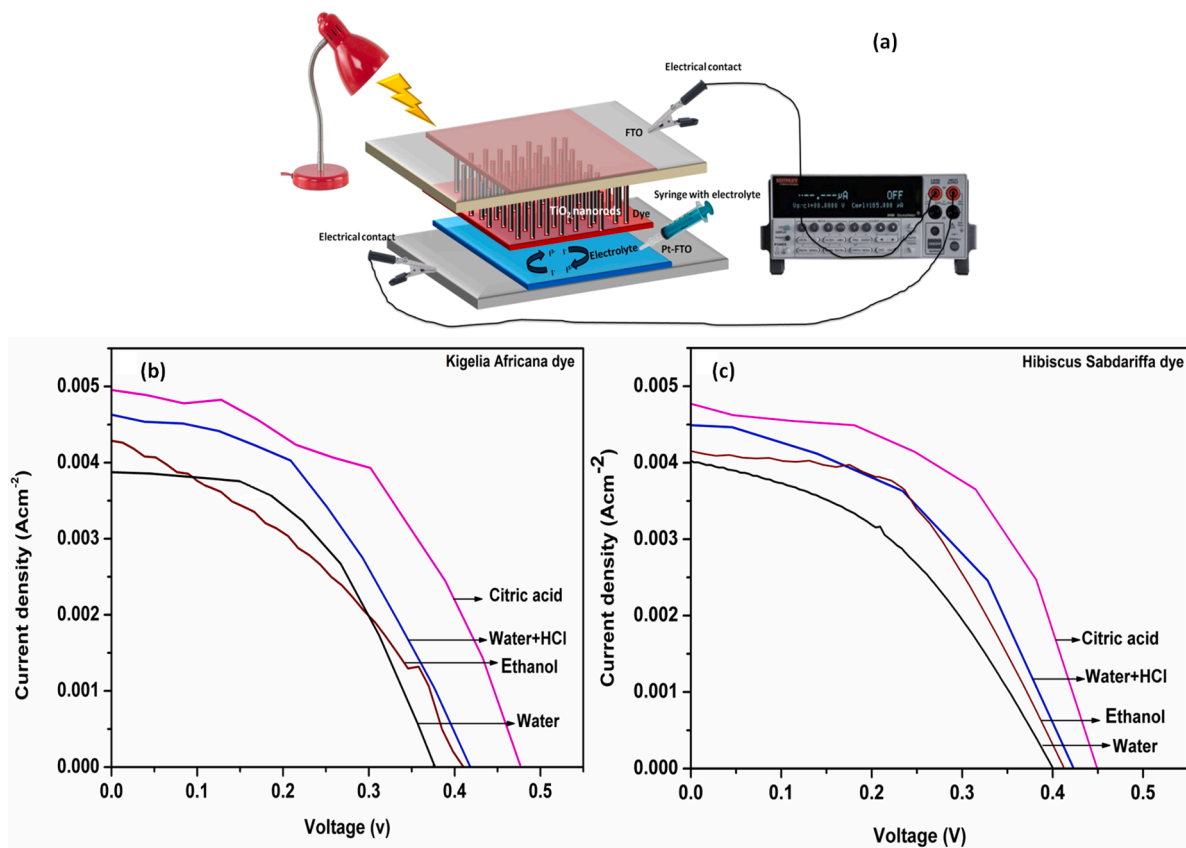


Fig. 9. (a) Schematic of the fabricated DSSC interfaced with Keithley 2401 source meter (b) J-V characteristics of *Kigelia Africana* dye sensitized DSSC (c) *Hibiscus sabdariffa* dye sensitized DSSC.

essential for effective anthocyanin extraction, the use of HCl is cautioned due to corrosion and polyphenoloxidase-induced browning, leading to dye degradation and reduced efficiency. In contrast, citric acid proves effective in inhibiting polyphenoloxidase, mitigating dye degradation, and consequently enhancing DSSC efficiency.

Conclusions

In the current investigation, TiO₂ nanorods, characterized by a single-phase rutile structure, were synthesized using a one-step hydrothermal method. Extraction of dyes from *Kigelia Africana* petals and *Hibiscus sabdariffa* calyx, employing citric acid as the solvent, exhibited promising outcomes. Notably, in terms of pH stability, samples extracted with citric acid retained approximately 85 % of anthocyanin content even after one month. Optical absorbance analysis indicated a gradual decrease over 30 days, signifying the degradation of anthocyanin with time.

In the context of light stability studies, dyes extracted with citric acid and stored in the dark demonstrated a notable resilience, with around 90 % of the anthocyanin content remaining after 30 days. Conversely, dyes extracted with other solvents exhibited a faster rate of degradation, leaving only 60–80 % of anthocyanin in the extract after 30 days. Regarding thermal stability, absorbance in both dye solutions extracted with citric acid increased gradually when heated from 30 °C to 50 °C, reaching a maximum at the latter temperature.

The conversion efficiency of Dye-Sensitized Solar Cells (DSSCs) fabricated with *Kigelia Africana* and *Hibiscus sabdariffa* dyes extracted using citric acid was measured at 0.87 % and 0.92 %, respectively. This enhancement in efficiency was attributed to the inhibition of polyphenoloxidase, effectively mitigating dye degradation and consequently improving DSSC efficiency.

CRedit authorship contribution statement

T. Satish Kumar: Writing – original draft, Validation, Investigation, Conceptualization. **S. Shalini:** Writing – original draft, Methodology, Investigation, Conceptualization. **T. Anurag Roy:** Writing – review & editing, Validation, Methodology. **S. Prasanna:** Writing – review & editing, Resources, Methodology. **R. Balasundaraprabhu:** Writing – review & editing, Visualization, Supervision. **Senthilarasu Sundaram:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.jpap.2024.100233](https://doi.org/10.1016/j.jpap.2024.100233).

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