Formulation Optimization of Swamp Vegetation Natural Dyes for DSSC Using Simplex Lattice Design

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Article Info

Abstract

Dye-sensitized solar cells (DSSC) are third-generation photovoltaic devices capable of using natural pigments as photosensitizers. This study aims to optimize the efficiency of DSSC by using formulations of a combination of natural dyes extracted from swamp vegetation: water hyacinth (Eichhornia crassipes), senduduk fruit (Melastoma malabathricum), and primrose willow (Ludwigia peruviana). These dyes, which are rich in chlorophyll, anthocyanins, and carotenoids, respectively, were formulated in different volume combinations using the Simplex Lattice Design (SLD) method. The experimental results show that the mixture of natural dyes can expand the absorption range of visible light wavelengths and enhance the performance of DSSC. The optimal dye combination, consisting of 1.827 mL water hyacinth and 3.173 mL senduduk fruit, yielded an efficiency of 0.050% with a desirability score of 1. This study highlights the potential of swamp vegetation as a sustainable and low-cost alternative for DSSC applications and demonstrates the effective use of statistical modeling to optimize dye formulations.

Introduction

Dye-sensitized solar Cells (DSSCs) are promising third-generation photoelectrochemical solar cells due to their easy fabrication, low cost, and eco-friendly potential [1]. However, reliance on synthetic dyes as photosensitizers raises several concerns, including high toxicity, environmental risks, and high production costs [2]. Therefore, many researchers are limiting the use of synthetic dyes and developing alternatives in the form of natural dyes that are more ecological, biodegradable, environmentally friendly, sustainable, and a low-cost alternative for DSSC applications [3], [4].

Several natural dye sources, such as fruits, leaves, and flowers, have been successfully used as natural dyes in DSSC, including extracts from *Hylocereus polyrhizus*, *Carica papaya L*, and

Hibiscus Sabdariffa [5], [6], [7]. Although these studies demonstrated the feasibility as natural dyes, most of them only explored the type of material as a single dye or concentration variation, without considering the synergistic effect between different pigments. The mixture of multiple pigments from natural dye extracts with different absorbance can be an effective technique to increase the absorption of light wavelengths and can promote better performance than a single dye [8],[9]. In addition, optimization of dye mixtures for DSSC applications is still limited, especially in studies that utilize unexplored resources such as swamp vegetation.

One interesting aspect of swampland vegetation is its potential as a natural dye source for dye-sensitized solar cells. Swamp plants such as water hyacinth (*Eichhornia crassipes*) [10], senduduk (*Melastoma malabathricum*) [11], and primrose willow (*Ludwigia peruviana*) [12], are potentially abundant, fast-growing, and rich in chlorophyll, anthocyanin, and carotenoid pigments. Despite their potential, their combined use as photosensitizers for DSSC has not been widely reported. In addition, research on DSSC optimization has been successfully conducted by [13], [14], [15], [16], but most of these optimizations only focus on the DSSC fabrication process, such as dye concentration optimization using D-Optimal or RSM. Therefore, this research will optimize the mixture formulation of the three types of dye.

Compared to other optimization methods, such as Taguchi design, One Factor at a Time (OFAT), or Response Surface Methodology (RSM), Simplex Lattice Design (SLD) offers greater predictive efficiency in exploring mixture composition. These other methods are less able to capture interaction effects between components, and sometimes require more experiments, which is an important limitation when studying complex natural dye systems. In contrast, SLD can systematically evaluate the combination effects of a fixed number of components, enabling accurate predictions for synergistic effects and more effective experimental methods with fewer trials [17]. Several SLD studies have been successfully reported by [18], [19], [20], [21] in identifying the optimal volume in material formulations conducted in the pharmaceutical and food fields.

In addition, this study emphasizes the synergistic interaction of several natural pigments, chlorophyll, anthocyanin, and carotenoids, which are extracted from swamp vegetation. Each type of pigment contributes differently to light absorption; for example, chlorophyll absorbs in the red and blue regions, anthocyanins in the green-blue region, and carotenoids primarily in the blue-green region. When pigments are mixed, they can expand the light absorption spectrum in the visible range and improve photon capture. Additionally, electron transfer between pigments or resonance energy transfer (RET) can occur, accelerating charge separation and minimizing recombination losses. These interactions can also lead to bandgap engineering, effectively lowering the energy threshold required for electron excitation and enhancing the overall efficiency of DSSC [22].

Despite these advantages, there are still few reported studies on mixture design optimization and synergistic photovoltaic analysis of pigments in DSSC. Most existing studies have not investigated the absorbance properties and photovoltaic parameters affected by different combinations of natural pigments. Therefore, the main objective of this study is to optimize the formulation of natural dyes derived from swamp plants using SLD to improve the efficiency of DSSC. The novelty of this study lies in incorporating pigment-rich extracts from marsh vegetation and modeling their optimal composition using SLD. This approach also overcomes the current limitations in natural dye DSSC research. The results of this study are

expected to contribute to the development of environmentally friendly and low-cost solar cell technology.

Experimental Method

Instrument and Material

The instruments used in this research are: luxmeter (HS1010), Ohaus digital balance, Kirin oven, glass cutter (PKC593), 10 kΩ potentiometer, digital multimeter (FLUKE 77 iii and HELES UX-369c), project board, refrigerator, solar simulator (PEC-l01), voltmeter, wattmeter, Design Expert 13 software (StatEase), and OriginLab 10 software. The materials used are water, acetic acid (SMART-LAB), distilled water (OneLAB), TiO2 powder (Merck), water hyacinth leaves, senduduk fruit, primrose willow obtained from swamp land in the Tanjung Batu area of South Sumatra, 0.1 N iodine (Merck), TCO glass (Mito T500 tablet), and carbon catalyst (candle soot).

Extraction of Natural Dye

The natural dye materials used were extracts of water hyacinth (*Eichhornia crassipes*), senduduk fruit (*Melastoma malabathricum*), and primrose willow (*Ludwigia peruviana*). Natural pigments were extracted by macerating 50 g of each raw material with 100 mL of Aquadest (Waterone, ONELAB) (1:2 b/v) for 24 hours at room temperature. The resulting mixture was filtered using Whatman No. 42 paper, and the filtrate was stored at 4°C until use.

Dye Formulation using Simplex Lattice Design

The formulation of a natural dye mixture was carried out using a three-component simplex lattice design (SLD) through Design Expert 13 software (StatEase Inc., USA). Three components were used from dye extracts: A (water hyacinth), B (senduduk fruit), and C (primrose willow), with 5 mL of each mixture. The SLD model resulted in 14 experiment runs. This design aims to identify the optimal combination to maximize the fill factor and efficiency of the DSSC. Mixed boundary ranges of three vegetation types can be seen in Table 1 and formulation design in Table 2.

Table 1. Mixed boundary ranges of three vegetation types

Component	Name	Units	Type	Minimum	Maximum
A	Water Hyacinth	mL	Mixture	0	5
B	Senduduk fruit	mL	Mixture	0	5
C	Primrose willow	mL	Mixture	0	5

Each formulation was prepared and measured once under identical, controlled laboratory conditions due to resource/time constraints. We acknowledge the absence of replicate measurements as a limitation of the present study; therefore, results are presented as single-run values and discussed primarily as exploratory/optimization outcomes. Future work will include replicate fabrication and measurement (triplicate) to quantify experimental variance and provide statistical error bars.

Table 2. Design formulation of a natural dye mixture from three vegetations

Run	A: Water Hyacinth (mL)	B: Senduduk fruit (mL)	C: Primrose willow (mL)
1	0	5	0
2	0	0	5
3	2.5	2.5	0
4	2.5	0	2.5
5	0.833333	0.833333	3.33333
6	0	0	5
7	0.833333	3.33333	0.833333
8	0	5	0
9	3.33333	0.833333	0.833333
10	2.5	2.5	0
11	5	0	0
12	0	2.5	2.5
13	5	0	0
14	1.66667	1.66667	1.66667

DSSC Assembly

TiO$_2$ photoanodes were prepared by coating TCO glass with TiO$_2$ paste and oven at 105°C for 5 minutes. The prepared electrodes were then immersed in each natural dye solution for 24 hours to allow dye adsorption. The DSSC was assembled in a sandwich configuration using a dye-sensitized photoanode, electrolyte (I-/I$_3$- redox couple), and carbon-coated comparison electrode, and the assembly was clamped with binder clips. An electrolyte solution of 0.5 mL was injected between the electrodes.

Bandgap Energy Analysis

The absorbance spectra of each dye extract were recorded using a UV-Visible spectrophotometer in the range of 300–800 nm to evaluate their light-harvesting capabilities. To estimate the optical bandgap energy (Eg), the Tauc plot method was applied based on the absorbance data. Although the Tauc method has traditionally been used for crystalline semiconductors, it has been applied in various studies to provide qualitative estimates of the optical gap in organic dye films and sensitized dye systems. The results of the Tauc plot should be interpreted qualitatively in the context of molecular dye systems rather than as absolute crystalline optical gap values [23] [24].

$$(\alpha h v)^2 = (h v - Eg) \tag{1}$$

where α is the absorption coefficient (derived from absorbance), hv is the photon energy, and Eg is the optical bandgap. A plot of $(\alpha h v)^2$ versus hv was constructed, and the linear portion was extrapolated to intersect the x-axis, giving the estimated bandgap energy. This approach provides a comparative indication of the electronic transition threshold between different dye mixtures, although the absolute Eg values should be interpreted with caution due to the molecular complexity and non-crystalline nature of natural dyes [25].

DSSC Performance Characterization

The efficiency of DSSC is determined from measurements of current-voltage curves and power-voltage density. The DSSC performance measurement used a solar simulator (PEC-L01). A 10 kΩ potentiometer and a digital multimeter (Fluke 77 III and Heles UX-369C) were used to obtain the maximum output voltage value. I-V and P-V curves were used to determine the fill factor (FF) and efficiency values.

Statistical and Regression Analysis

ANOVA was conducted to evaluate the significance of factors affecting the DSSC efficiency using Design Expert software. The responses (efficiency and fill factor) were modeled using linear, quadratic, and special cubic polynomial equations. Model fit was evaluated by R^2, adjusted R^2, and p-values (<0.05). Optimization was done by maximizing desirability criteria [26].

The overall research procedure is summarized in the research flow chart presented in Figure 1. This diagram illustrates the sequential steps starting from sample preparation and dye extraction, followed by dye formulation, DSSC fabrication, DSSC performance, and statistical analysis.
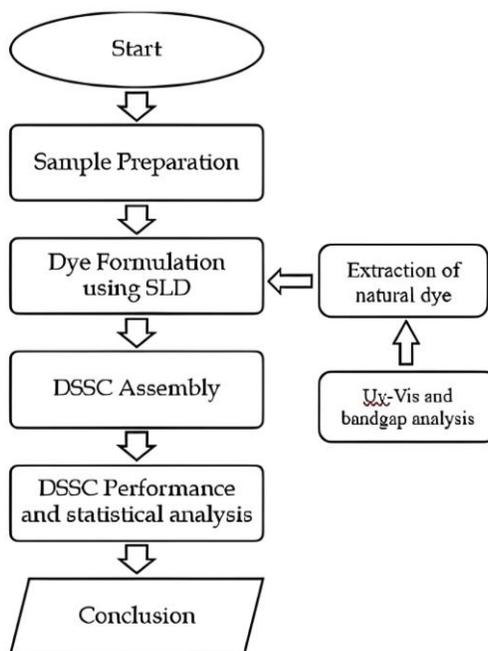

Figure 1. Flow chart research

Result and Discussion

Bandgap Energy of Natural Dye

The natural dyes extracted from water lotus (chlorophyll), senduduk fruit (anthocyanins), and primrose willow(carotenoids), used as sensitizers in this study, have varying band gap energies. Each pigment exhibits different light absorption behavior, contributing to complementary spectral coverage. Chlorophyll absorbs strongly in the red (600-700 nm) and

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blue (400-500 nm) regions, anthocyanins absorb around 500-600 nm, and carotenoids generally absorb between 400-500 nm. These characteristics allow for spectral overlap when dyes are mixed.

This overlap is clearly visible in the UV-Vis absorption spectrum of the three-dye mixture in Figure 2, which shows broad absorption from 400-700 nm. This broadband nature promotes pigment synergy in enhancing the absorption of photons from sunlight. The presence of various chromophores allows for better photon capture and electron excitation, which in turn increases charge generation. In addition, we also measured the UV-Vis data of single pigments, which are presented in the supplementary data [27].
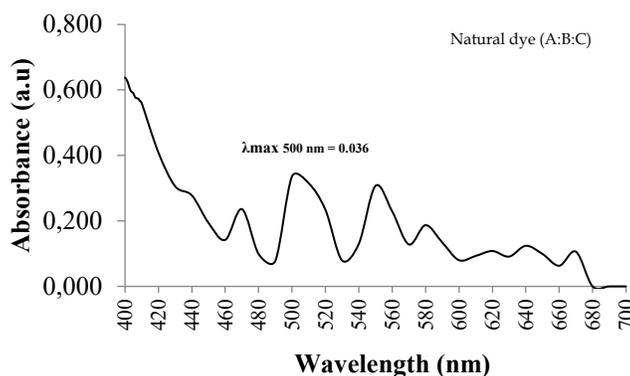

Figure 2. UV-Vis absorption spectrum of the mixture of three dyes

Based on the results of Tauc plot analysis of UV-Vis data, the band gap energy results ranged from 1.75 eV to 2.86 eV, which can be seen in Table 3, consistent with previous reports on similar pigments [28], [29]. Among the single dye extracts, senduduk fruit (B) showed the lowest band gap energy of 1.99 eV, which corresponds to the absorption of visible light at 532 nm. Water hyacinth extract (A) showed two values, 1.75 eV and 2.86 eV, indicating a possible double transition, possibly related to chlorophyll a and b. Primaryose willow extract (C), which is rich in carotenoids, showed a band gap of 2.36 eV, corresponding to the absorption of visible light at 470 nm.

The mixed dye formulation (A:B = 2.5:2.5 mL) resulted in a band gap of 2.25 eV, which indicates an intermediate and overlapping spectral response. This synergy allows the DSSC to absorb photons of solar light over a broader and wider range of the visible spectrum. Figure X confirms this, showing that the UV-Vis absorption spectrum of the dye mixture is significantly wider in the 400-700 nm range. These overlapping spectra indicate that the combined dye acts as a broad-band absorber, improving the light-harvesting efficiency.

From a photoelectric point of view, having multiple chromophores increases the ability to capture photons and promote electron excitation from the LUMO of the dye to the conduction band of TiO_2. These varied dye properties can also minimize recombination losses by spatially isolating charge carriers and encouraging directional electron movement. This cooperative interaction among dye components favors improved DSSC performance.

Table 3. Bandgap energy values of natural dyes

Natural dye	Volume (mL)	Bandgap energy (eV)
A	5:0:0	1.75 and 2.86
B	0:5:0	1.99
C	0:0:5	2.36
A:B	2.5:2.5:0	2.25
B:C	0:2.5:2.5	2.30
A:C	0:2.5:2.5	2.28
(A:B:C)	0.8:0.8:3.3	2.31
(A:B:C)	0.8:3.3:0.8	2.26
(A:B:C)	3.3:0.8:0.8	2.5
(A:B:C)	1.6:1.6:1.6	2.25

Description: (A: water hyacinth, B: senduduk fruit, C: primrose willow)

DSSC Performance

The results of DSSC performance measurements are shown in Table 4. Among the single natural dyes, the DSSC based on natural dye from senduduk fruit (B) yielded the highest efficiency value of 0.024%, which was due to the strong absorption of visible light due to its low band gap energy of 1.99 eV. The other dyes, water hyacinth (A) and primrose willow (C) showed lower performance of 0.003% and 0.004%, respectively, corresponding to less optimal absorption regions [30].

The combination of dyes significantly improves DSSC performance. A mixture of water hyacinth and senduduk fruit (A:B = 2.5:2.5 mL) with an intermediate band gap of 2.25 eV significantly improves performance, resulting in an efficiency of 0.049% and a fill factor (FF) of 0.270. This improvement in performance is due to the mixture of chlorophyll and anthocyanin, which increases the photon absorption area. Chlorophyll absorbs mainly in the (400-500 nm) and (600-700 nm) range, while anthocyanin covers the (450-600 nm) range. Meanwhile, the highest recorded efficiency of 0.050% was achieved in the formulation (A:B:C = 0.833:3.333; 0.833 mL), while the highest filling factor (0.438) was found in a different formulation (A:B:C = 3.333:0.833:0.833 mL). In the formulation (A:B:C = 3.333:0.833:0.833 mL), the efficiency was only 0.022%, despite having the highest filling factor, indicating that insufficient photon absorption or recombination limited the overall output. These results are consistent with the theory proposed by [29], that as the absorbed wavelength becomes longer, the photon energy decreases, and this is consistent with semiconductors that have smaller band gap energies.

This difference shows that a high fill factor value (which indicates lower series resistance) does not guarantee higher efficiency if photon absorption is insufficient in the active spectral region, or if incomplete charge recombination and electron injection reduce the photocurrent. The

interfacial resistance between the photoanode and electrolyte is often related to the fill factor. A higher fill factor indicates better charge extraction and lower series resistance. Conversely, light absorption and electron injection, as well as interfacial dynamics, increase efficiency when dyes with complementary band gaps are synergistically coupled.

For further clarification, we present Figure 3, the I–V curve of the mixed dye formulation that produces the highest photovoltaic performance. Additionally, I–V data for curves sensitized with single dyes (water hyacinth, senduduk fruit, and primrose willow) are provided in the supplementary Information for reference, allowing readers to compare the performance of single dyes and confirm the improvement achieved by the mixture.

Table 4. DSSC performance measurement of each design formulation

| Std	Run	Component 1 A: Water Hyacinth mL	Component 2 B:Senduduk fruit mL	Component 3 C:Primrose willow mL	Response 1 Efficiency %	Response 2 Fill factor
2	1	0	5	0	0.024	0.2529
13	2	0	0	5	0.004	0.255
14	3	2.5	2.5	0	0.049	0.27
5	4	2.5	0	2.5	0.017	0.262
9	5	0.833333	0.833333	3.33333	0.026	0.36
3	6	0	0	5	0.004	0.255
8	7	0.833333	3.33333	0.833333	0.050	0.342
12	8	0	5	0	0.024	0.252
7	9	3.33333	0.833333	0.833333	0.022	0.438
4	10	2.5	2.5	0	0.049	0.270
1	11	5	0	0	0.003	0.273
6	12	0	2.5	2.5	0.012	0.2826
11	13	5	0	0	0.003	0.2730
10	14	1.66667	1.66667	1.66667	0.019	0.2623
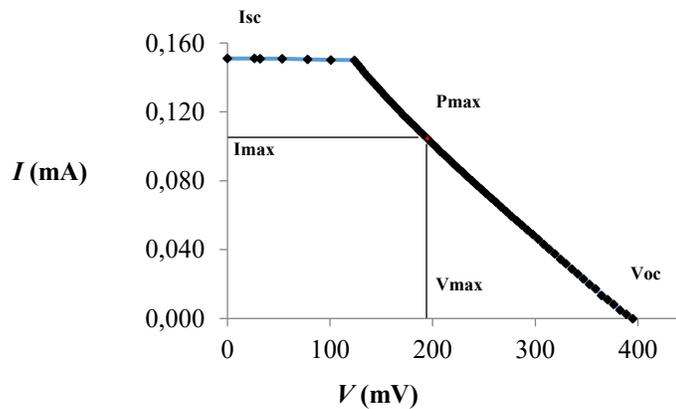
Figure 3. Characteristic curve (*I-v*) of the mixture formulation (A:B:C = 0.833:3.333; 0.833 mL)

Optimization and Statistical Analysis

The simplex lattice design provided 14 formulation trials. The resultant efficiency values ranged from 0.003% to 0.050%, and fill factors from 0.252 to 0.438, as shown in Table 4. Furthermore, Table 5 shows the results of the regression analysis to predict the efficiency of DSSC with various models. The linear model has a p-value of 0.0913 and an Adjusted R^2 value of 0.2352, indicating that it is not significant and is only able to explain a small portion of the variation in the data, with a negative predicted R^2 (-0.0651), signifying poor predictive ability. The quadratic model, on the other hand, showed significant results with a p-value of 0.0037, an Adjusted R^2 of 0.7889, and a Predicted R^2 of 0.7250, indicating this model was able to explain 78.89% of the variation in the data and had good predictive ability, so it was recommended for use. The special cubic model and the cubic and other models were not significant with a negative predicted R^2, so they were not suitable for use in this analysis and were considered aliased. Overall, the quadratic model proved to be the best fit in predicting DSSC efficiency.

Table 5. Response regression analysis

Source	Sequential p-value	Adjusted R^2	Predicted R^2	
Linear	0.0913	0.2352	-0.0651	
Quadratic	0.0037	0.7889	0.7250	Suggested
Special Cubic	0.6238	0.7675	-0.6334	
Cubic	0.2125	0.8248	-7.0515	Aliased
Sp Quartic vs Quadratic	0.3120	0.8248	-7.0515	
Quartic vs Cubic		1.0000		Aliased
Quartic vs Sp Quartic		1.0000		Aliased

Based on the advanced ANOVA results presented in Table 6, the model analyzed for efficiency showed high significance, with an F value of 10.72 and a p-value of 0.0022, which is below the general significance limit of (<0.05). This indicates that the model as a whole can explain the significant variation in DSSC efficiency. The linear mixture component also showed a significant effect with an F value of 10.87 and a p-value of 0.0052. For A-B (chlorophyll-anthocyanin) interaction had the highest effect (F = 29.64, p < 0.001), indicating their synergy in improving DSSC performance. In contrast, the A-C and B-C interactions were not significant (p > 0.05), indicating weaker compatibility. This indicates that the linear mixture of components A (water hyacinth) and B (senduduk fruit) contributed significantly to the efficiency.

Table 6. ANOVA of efficiency response

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0032	5	0.0006	10.72	0.0022	significant
[1]Linear Mixture	0.0013	2	0.0007	10.87	0.0052	
AB	0.0018	1	0.0018	29.64	0.0006	
AC	0.0001	1	0.0001	2.14	0.1818	
BC	1.042E-07	1	1.042E-07	0.0017	0.9679	
Residual	0.0005	8	0.0001			
Lack of Fit	0.0005	4	0.0001			
Pure Error	0.0000	4	0.0000			
Cor Total	0.0037	13				

By considering the molecular orbital interaction between the dye and the titanium dioxide semiconductor, this statistical synergy can be photoelectrically rationalized. Chlorophyll and anthocyanin dyes have different dipole orientations and HOMO-LUMO gaps. Their LUMO levels may be more aligned with the conduction band of TiO_2 (3.0-3.5 eV) when co-sensitized [30]. This facilitates effective electron injection. Dyes may also increase the density of the interfacial electronic states, which allows a wider energy window for charge transfer.

In addition, the different light absorption of each dye on the TiO_2 surface complements their spatial and electronic distribution. This results in better coverage and fewer vacant sites. This reduces the amount of recombination and increases the effectiveness of charge separation. Therefore, the statistical optimization is not only performed mathematically, but also supported by the molecular-level interaction between the surface energy of titanium dioxide and the orbital structure of the dye.

In contrast, the insignificant interaction of the A-C and B-C mixtures could be due to poorer spectral overlap, poor energy level alignment, or less compatibility in binding affinity with the titanium dioxide surface.
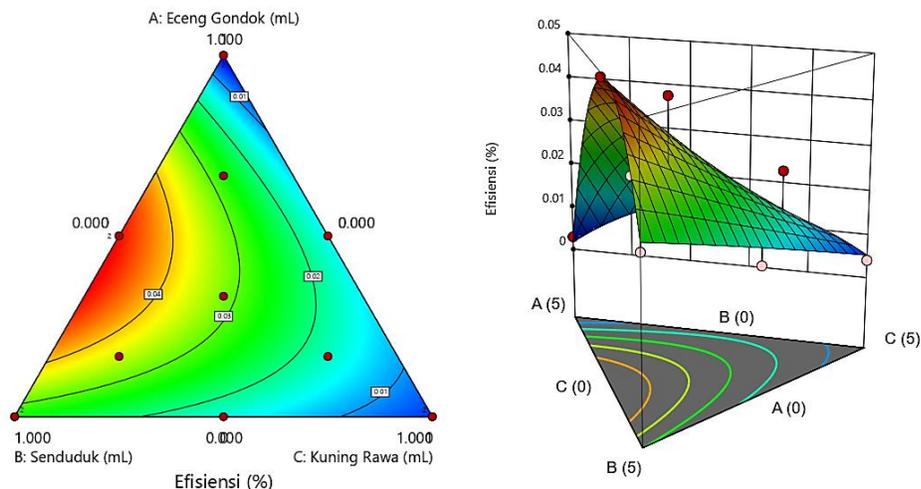
Figure 4. 3D contour and surface plots against response

To illustrate the interaction between each parameter to the response in Figure 4. 2D contour plot and 3D surface of the efficiency in response to the variable mixture of three natural dyes. The figure shows that the efficiency tends to increase as the volume of water hyacinth and senduduk fruit natural dyes increases.

Optimal Formulation

Optimization results using Design Expert 13 software suggested nine potential dye formulations, with the top ranked: A (Water hyacinth) = 1.827 mL, B (senduduk fruit) = 3.173 mL, C (Primrose willow) = 0.000 mL. These formulations achieved a potential efficiency of 0.050% with a desirability score of 1.0, indicating that the predicted results were statistically and experimentally accurate.

In Figure 1, the surface and contour plots also confirm that the efficiency values on the DSSC increase with higher proportions of A and B, supporting the statistical results and confirming that C (Primrose willow) may be less effective or redundant in the optimized mixture.

Table 7. Formulas generated in the optimization process.

No	Water Hyacinth	Senduduk fruit	Primrose willow	Efficiency	Desirability	
1	1.827	3.173	0.000	0.050	1.000	Selected
2	2.212	2.788	0.000	0.050	1.000	
3	1.957	3.043	0.000	0.050	1.000	
4	2.093	2.907	0.000	0.050	1.000	
5	2.378	2.622	0.000	0.049	1.000	
6	2.288	2.712	0.000	0.049	1.000	
7	1.718	3.282	0.000	0.049	1.000	
8	2.126	2.825	0.049	0.049	1.000	
9	2.500	2.500	0.000	0.049	0.993	

Conclusion

This study shows that optimizing natural dye mixtures from wetland vegetation using Simplex Lattice Design (SLD) can identify synergistic pigment combinations that enhance photon absorption and improve DSSC performance. The optimized mixture (A = 1.827 mL, B = 3.173 mL, C = 0.000 mL) achieved the highest measured efficiency (0.050%) under current experimental conditions, confirming that co-sensitization and rational mixture design can enhance light absorption and charge generation in DSSC. The novelty of this research lies in the use of under-explored wetland vegetation and its potential as a renewable dye source and the systematic application of SLD to identify synergistic effects among natural pigments. Future work will involve applying repeated trials under controlled conditions to strengthen the statistical reliability of performance data, along with refining spectral characterization to obtain clearer absorption features and bandgap estimations. Expanded mixture optimization will also be performed to further explore pigment synergy and maximize light-harvesting capability.

Acknowledgment

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