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Characterization of a green and environmentally friendly sensitizer for a low cost dye-sensitized solar cell

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ABSTRACT

Clean and cheap device, namely dye-sensitized solar cells (DSSCs) were fabricated using a natural dye extracted from *Sambucus ebulus*. We prepared five sample solutions with various pH in the extraction process to improve power conversion efficiency. The UV–visible absorption investigation of sample solutions and on photoanode show the dyes from J-type aggregation on a photoanode substrate. Redox properties of all sample solutions certify thermodynamically a charge transfer from excited state to conduction band TiO₂. The optical properties of various dye solutions were investigated and results showed darkness and bluish tint effect of dye solutions extracted in basic environment rather than those extracted in acidic condition. Moreover, in comparison to the basic condition, the dye solutions extracted in acidic environment were more saturated and colorimetrically less different from that one which extracted in neutral condition. Photophysical and photoelectrochemical performance of natural extraction dyes have been studied in dye-sensitized solar cell devices. The results show the rather high conversion efficiency of 0.57%, 1.15%, 1.02%, 0.35% and 0.15% of each individual dye extraction, respectively.

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1. Introduction

Dye-sensitized solar cells (DSSCs) are key photovoltaic devices for production of clean and renewable energy [1,2]. Numerous studies on the DSSCs have been carried out in the last years and the results show that the photosensitizers are important materials in DSSCs [3,4]. DSSCs efficiency was determined by a dye absorption spectrum and the amount of anchoring to the photoelectrode surface. Thus, the selection of a suitable dye sensitizer in DSSCs is very important [5,6].

One of the photosensitizers in DSSCs is the natural dyes that can be extracted from some flowers, leaves and fruits [6,7]. Since, natural dyes are low cost, complete biodegradation, easy preparation and environmentally friendly, there is interest toward using these for dye-sensitized solar cells in the future research [8–10]. To the best of our knowledge, the highest efficiency of 1.6% was achieved from the methanol of the Mulberry [11]. Alhamed et al. reported a power conversion efficiency of 1.50% using the natural dye extracted from Raspberriec fruit [12]. The leaves of *Ficusreusa* and *Hibiscus surattensis* were employed as photosensitizers in a

DSSCs' structure by Fernando et al. [13] and Lai et al. [14], while their power conversion efficiency were 1.14% and 1.18%. Hamadani et al. showed that using Reseda Luteola as photosensitizer, J_{sc} of 0.54 mA cm⁻², V_{oc} of 0.64 V and power conversion efficiency of 0.22% can be achieved [15]. Zhou et al. used 20 natural dyes as sensitizers in a dye-sensitized device and the results show that the power conversion efficiency of the ethanol extract of Mangosteen pericarp without purification reached 1.17% [16].

In the current study, a natural dye containing ebulosid extracted from the fruit of *Sambucus ebulus*, which grows in Iran, was used as photosensitizers in the manufacturing DSSCs. To the best of our knowledge, the natural dye extracted of this plant sources have never been used as photosensitizer in DSSCs and the investigation presents a new view on the application of this extract in the DSSCs. The spectrophotometric properties of the prepared natural dyes in a solution with various pH and on a nanoanatase titanium dioxide (TiO₂) substrate were examined. The absorption maxima and intensities of the resultant natural dyes were also obtained. Finally, the optical and photovoltaic of DSSCs made of this extract, as well as their colorimetric attributes were determined and discussed.

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2. Experimental

2.1. Materials and instruments

The samples of *S. ebulus* used in this study were obtained from underbrush are grown in north of Iran (Mazandaran-Ramsar). All chemical materials and solvents utilized in this study were analytical grades provided by Merck Co. without further purification. Transparent conducting oxide, fluorine doped tin oxide (FTO, F-doped SnO₂, DyeSol), TiO₂ pastes, scattering layer (Sharif Solar Co.) and di-tetrabutylammonium cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato) ruthenium(II) (N719, Sharif Solar Co.) were purchased. Ultraviolet–visible (UV–vis) spectrophotometry was carried out on a Cecil 9200 double beam transmission spectrophotometer.

2.2. Preparation of a natural dye

The extract of *S. ebulus* was obtained from a fresh fruit. The clean fruit was extracted from 100 mL ethanol. Four extracts were prepared by immersing the fresh fruit in HCl (0.1 N and 1 N) and NaOH (0.1 N and 1 N) solutions (ethanol extract). The mixture was kept overnight at 4 °C, then solid residues were filtered out to obtain the clean dye solution. All solution was protected from direct light and stored in the refrigerator at 4 °C until tested (Table 1).

2.3. Colour measurement

In order to compute the colourimetric attributes, the percentage of light transmitted from the natural dye solutions were measured over the visible wavelengths from 400 to 700 nm by 10 nm intervals. The measured transmitted spectra were then converted to the colourimetric properties of natural dye solutions. The CIEXYZ tristimulus values of solutions were computed by using Eqs. (1)–(3) where T indicates to the dyes solutions transmittance spectra and x, y and z refers to the colour matching functions (CMFs) under a D65 standard illuminant [17].

$$X = \int_{\lambda=400}^{\lambda=700} S(\lambda)\bar{x}(\lambda)T(\lambda)d\lambda \quad (1)$$

$$Y = \int_{\lambda=400}^{\lambda=700} S(\lambda)\bar{y}(\lambda)T(\lambda)d\lambda \quad (2)$$

$$Z = \int_{\lambda=400}^{\lambda=700} S(\lambda)\bar{z}(\lambda)T(\lambda)d\lambda \quad (3)$$

where $S(\lambda)$ is the spectral power distribution (SPD) of the light source that is normalized by applying of Eq. (4) [17,18]:

$$100 = \int_{\lambda=400}^{\lambda=700} S(\lambda)\bar{y}(\lambda)d\lambda \quad (4)$$

In the next step, the CIELAB colorimetric attributes [18–20] of natural dye solutions were computed via Eqs. (5)–(7):

$$L^* = 116(Y/Y_n)^{1/3} - 16 \quad (5)$$

$$a^* = 500[(X/X_n)^{1/3} - (Y/Y_n)^{1/3}] \quad (6)$$

$$b^* = 200[(Y/Y_n)^{1/3} - (Z/Z_n)^{1/3}] \quad (7)$$

where, L^* , a^* and b^* are the colourimetric coordinates of objects in a CIELAB colour space. Fig. 1 schematically shows the CIELAB colour order system. Based on Fig. 1, the horizontal axes of CIELAB colour space indicate to the redness-greenness (from the positive a^* values to the negative one) and yellowness-blueness (from the positive b^* values to the negative one) properties of objects. Based on Fig. 1,

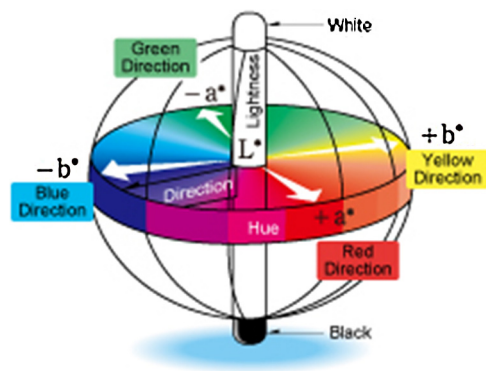


Fig. 1. The CIELAB colour order system [20].

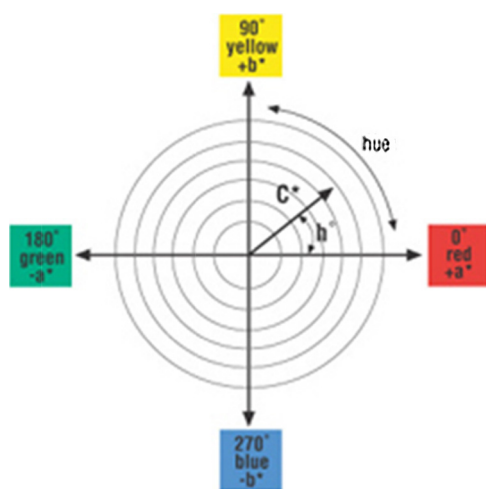


Fig. 2. The schematic definition of objects' colourimetric saturation (chroma) and hue angle [21].

the vertical axis of figure shows the lightness attribute of objects while varies from the $L^* = 0$ (for the ideal black) to the $L^* = 100$ (for the ideal white) [19].

On the other hand, the chroma values and hue angle of natural dye solutions were obtained by applying of Eqs. (8) and (9), respectively.

$$C^* = (a^{*2} + b^{*2})^{0.5} \quad (8)$$






$$\text{hue angle} = \tan^{-1}(b^*/a^*) \quad (9)$$

where the L^* , C^* and hue angle are the colourimetric coordinates of a CIELCH colour space which is the cylindrical representation of the CIELAB colour order system. Fig. 2 defines the saturation (chroma) and hue angle properties of objects, schematically. The chroma attribute shows that how much the colour appearance of objects is saturated and the hue angle indicates to their tint attribute [19].

2.4. Fabrication and characterization of DSSCs

A nanocrystalline TiO₂ film was coated on a fluorine doped tin oxide (FTO) coated glass support. The dye solutions were adsorbed by dipping the coated glass for overnight in an ethanolic solution of the dye. Finally, the film was washed with an ethanol solvent. Acenonitrile-ethylenecarbonate ($v/v = 1:4$) containing tetrabutyl ammonium iodide (0.5 mol dm⁻³) was used as an electrolyte. The dye-adsorbed TiO₂ electrode, the Pt counter electrode and the electrolyte solution were assembled into a sealed sandwich type solar cell [22]. An action spectrum was measured under monochromatic

Table 1
Sambucus ebulus extraction samples.

Sample	Solvent	Diluted	Sample
A	0.1 N HCl	Ethanol	
B	1 N HCl	Ethanol	
C	–	Ethanol	
D	0.1 N NaOH	Ethanol	
E	1 N NaOH	Ethanol	

light with a constant photon number (5×10^{15} photon $\text{cm}^{-2} \text{s}^{-1}$). J - V characteristics were measured under illumination with AM 1.5 simulated sun light (100 mW cm^{-2}) through a shading mask ($5.0 \text{ mm} \times 4.0 \text{ mm}$) by using a Bunko-Keiki CEP-2000 system.

3. Results and discussion

3.1. Natural dye extraction as photosensitizers

S. ebulus with the common name of Dwarf elder or Elderberry is a member of the Adoxaceae group [23] and possible functions of Adoxaceae in leaves, flowers and fruits have long attracted scientific studies [24]. Adoxaceae are polar pigments with carbonyl and hydroxyl groups (Fig. 3) that may appear in various colours from pink to blue depending on pH [25]. These groups can be linked to the photo-anode surface in a DSSCs' structure [26]. The general chemical structure of anthocyanin and a picture of the investigated natural dye source is shown in Fig. 3. In this study, natural dye source, *S. ebulus*, was obtained from underbrush are grown in Ramsar forest (Iran-Mazandaran). The extracts obtained from whole fruits with the different pH were prepared (Table 1) and their photovoltaic properties were investigated.

Table 2
Absorption of the ethalonic solutions.

Dye	λ_{max} (nm) ^a	ε ($\text{M}^{-1} \text{cm}^{-1}$) ^b	λ_{max} (nm) ^c	$\Delta\lambda$ (nm)
A	509	41123	518	9
B	515	40750	519	4
C	532	40258	545	13
D	558	41024	569	11
E	603	41587	618	15

^a Absorption maximum of dye measured in ethanol.

^b The molar extinction coefficient at λ_{max} in solution.

^c Absorption maximum of dye adsorbed on the surface of TiO_2 .

3.2. UV-vis absorption spectra

The UV-vis absorption of the ethanolic extract in various pH are shown in Fig. 4 and the wavelength of maximum absorption (λ_{max}), the molar extinction coefficients (ε_{max}) and λ_{max} of the corresponding extract adsorbed on TiO_2 films are presented in Table 2. The λ_{max} of A, B, C, D and E appeared at 509, 515, 532, 558 and 603 nm and the corresponding molar extinction coefficients (ε) were 41123, 40750, 40258, 41024 and 41587 $\text{M}^{-1} \text{cm}^{-1}$, respectively. Solution of the natural dye extract in acidic environments has strong absorption due to the colour combination [27]. In comparison to two acidic environments, the λ_{max} of B showed strong absorption intensity, which could be attributed to the variation in the electrical current of the cells [10]. Note that the similar experimental phenomena have been achieved for batalains dyes by Hemmatzadeh [10] and anthocyanin dyes by Teoli [28]. The maximum absorption wavelengths (λ_{max}) of A, B, C, D and E on TiO_2 substrate appeared at 517, 528, 541, 569 and 611 nm. As expected, the absorption maxima of natural dyes applied on the surface of a photoanode gave bathochromic shifts compared to the corresponding dye spectra in ethanolic solution due to the J-aggregation [29].

3.3. Electrochemical properties

The oxidation potential (E_{ox}) of natural dyes was measured in ethanol by cyclic voltammetry (CV) to estimate the possibility of electron injection and dye regeneration. The voltammogram shows two distinct redox waves. The first oxidative wave (I) was due to the oxidation of the external standard of Ferrocene, whereas the second wave (II) is due to the electrochemical oxidation of each dye [30]. The oxidation peak potential (E_{pa}), which is corresponded to the highest occupied molecular orbital energy (HOMO) of A, B, C, D and E, can be calculated to be +0.93 V, +0.95 V, +0.96 V, +0.95 V and +0.96 V vs. Fc/Fc^+ (as an external standard Ferrocene/Ferrocenium redox couple) in acetonitrile, respectively. The energy levels of HOMO of all natural dyes are sufficiently more positive than the I_3^-/I^- redox potential (0.42 V), indicating the sufficient thermodynamically driving force for dye regeneration [31]. The E_{red} level of A, B, C, D and E is calculated to be -1.22 V , -1.27 V , -1.25 V , -1.28 V and -1.31 V , respectively vs. Fc/Fc^+ in acetonitrile. The E_{red} level, which corresponded to the lowest unoccupied molecular orbital energy (LUMO) of natural dyes, are more negative than the conduction band gap edge of TiO_2 (-0.5 V). Thus, the electron injection process from each excited dye molecule to the TiO_2 conduction band and the subsequent dye regeneration are energetically permissible [30].

3.4. Colourimetric attributes of natural dye solutions

The colourimetric attributes of five natural dye solutions have been shown in Table 3. By considering the colourimetric attributes of neutral *S. ebulus* solution (sample C), it is found that the natural extract benefits from the purple colour with the lightness value (L^*) of 82.7 and redness (a^*) and blueness (b^*) values of 27.58 and -6.81 ,

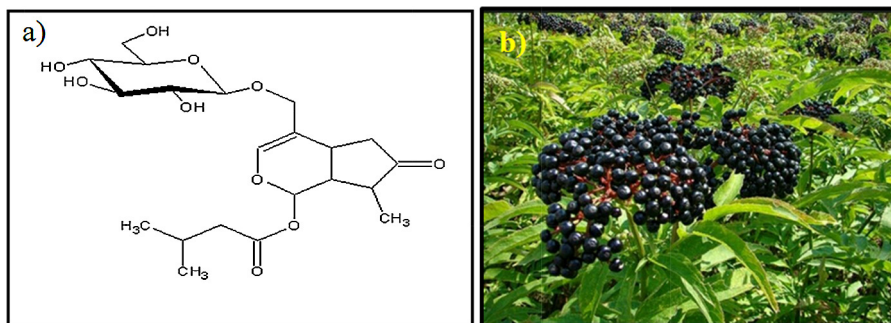


Fig. 3. (a) General structure of Adoxaceae [23] and (b) investigated natural dye source.

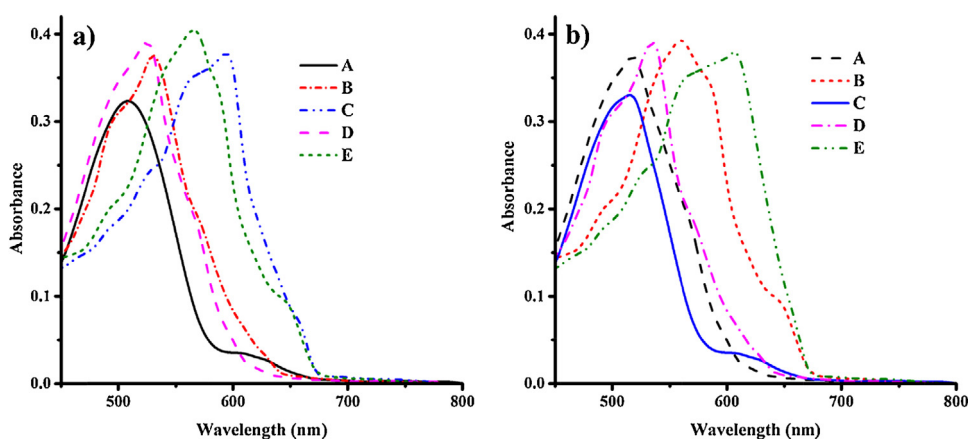


Fig. 4. UV-vis absorption (a) in solvent and (b) on TiO₂ substrate.

Table 3
The colourimetric characteristics of five natural dye solutions.

Sample	L^*	a^*	b^*	C^*	Hue	ΔE_{ab}^*
A	87.57	24.01	1.09	24.04	2.59	9.94
B	83.85	29.48	-2.76	29.61	354.66	4.62
C	82.70	27.58	-6.81	28.41	346.13	0
D	77.95	15.32	-16.92	22.83	312.18	16.58
E	78.99	5.41	-16.24	17.12	288.43	24.38

respectively. As the L^* parameter in Table 3 shows, *S. ebulus* extracts in presence of HCl (0.1 N and 1 N) (samples A and B) are lighter than the neutral dye solution (sample C), as well as those which extracted in presence of NaOH (0.1 N and 1 N) (samples D and E). Besides, the C^* values in Table 3 show that the neutral *S. ebulus* solution (sample C) and those which extracted in presence of HCl (0.1 N and 1 N) (samples A and B) are more saturated than those solutions extracted in presence of NaOH (0.1 N and 1 N) (samples D and E). Meanwhile, the b^* values of solutions A, B, C, D and E decrease continuously that indicate to the increase in bluish tint effect of natural dye solutions and explain their darkness enhancement.

In order to better show the optical difference between solutions extracted in presence of HCl or NaOH with the neutral dye solution, the colourimetric differences between all samples with sample C were computed by applying of Eq. (10) [32,33] and have been shown in Table 3:

$$\Delta E_{ab}^* = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{0.5} \quad (10)$$

where;

$$\Delta L^* = L_{\text{samp}}^* - L_{\text{ref}}^* \quad (11)$$

Table 4
Photovoltaic parameters of DSSCs based on the natural dye solutions.

Sample	V_{oc} (V)	J_{sc} (mA cm ⁻²)	FF (%)	η (%)
A	0.54	1.70	0.62	0.57
B	0.6	3.30	0.58	1.15
C	0.58	3.25	0.54	1.02
D	0.62	0.94	0.60	0.35
E	0.62	0.40	0.61	0.15
N719	0.88	13.55	0.71	8.47

$$\Delta a^* = a_{\text{samp}}^* - a_{\text{ref}}^* \quad (12)$$

$$\Delta b^* = b_{\text{samp}}^* - b_{\text{ref}}^* \quad (13)$$

In Eqs. (11)–(13) the natural dye solution extracted in neutral condition (sample C) is considered as the reference solution.

According to Table 3, in comparison to dye solutions extracted in presence of HCl, the dye solutions extracted in basic condition (in presence of NaOH) are more different rather than the reference solution (sample C), colourimetrically. It means that, extracting of dye solutions in basic environment results in higher optical differences rather than extracting the dye solutions in acidic conditions.

3.5. Photovoltaic properties of DSSCs

All photocurrent density–voltage (J – V) curves and incident photo to current conversion efficiency (IPCE) spectra of DSSCs based on C solution are presented in Fig. 5 and all photovoltaic properties are summarized in Table 4. According to the results presented in Table 4, under the standard global AM 1.5 solar condition, the conversion efficiencies of cells based on natural solutions A, B, C,

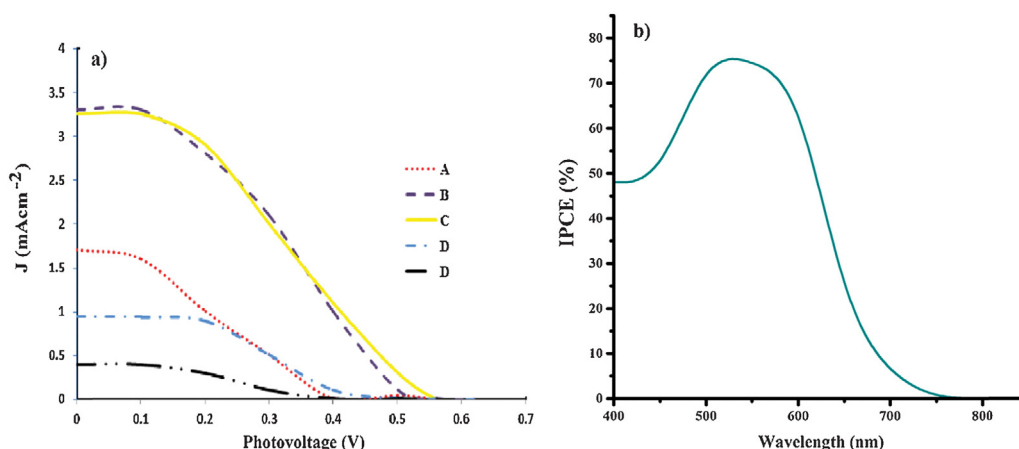


Fig. 5. (a) Photocurrent density–photovoltage curves of all devices and (b) IPCE spectra of DSSCs based on C solution.

D and E are 0.57%, 1.15%, 1.02%, 0.35% and 0.15%, respectively. The best fill factor (FF) was obtained by the natural extract in the presence of HCl 0.1 N (A solution), showing V_{oc} and J_{sc} values of 0.54 V and 1.70 mA cm⁻², respectively, with an active area = 0.62 cm². The common values of FF for DSSCs based on a natural extract are around 0.7 [34,35] so, the results achieved from the current work are in agreement with those reports.

The results show that the DSSCs based on acidic extraction presents a higher power conversion efficiency than other devices due to a low aggregation on TiO₂ substrates that these results are in agreement with those obtained in research [10]. Two natural pigments containing betalains and anthocyanins were prepared by Calogero et al. [36]. These natural dyes were utilized as photosensitizer in DSSCs device with conversion efficiency of 1.13% and 1.07%, respectively. The mentioned power conversion efficiencies are very close to the natural dye solutions have been used in this research in presence of HCl 1 N, as well as in inert solution. Lim et al. [37] utilized two natural dyes from Ixoracoccinea (RX) and Bougainvillea (BG) for an investigation of a DSSCs performance. The power conversion efficiencies of the DSSCs based on mentioned natural dyes were 0.76% and 0.2%, respectively, which are less than natural dyes employed in the current research. Fig. 5b shows the maximum IPCE value for the inert natural dyes (C sample). The natural dye shows IPCE ranged from 75%. However, the DSSCs with N719 presents much better properties rather than the DSSCs based on natural dyes due to a higher photovoltage value (0.88 V) as compared to those natural dyes (0.54–0.62 V). But, the presence of ruthenium as expensive metal has been limited in its general use.

4. Conclusion

In this study, we have reported an investigation on the use of extracting natural dye from *S. ebulus* in various pH as photosensitizers in the fabrication of DSSCs. We tried to improve the power conversion efficiency of clean DSSCs using changes in the solution properties as an assembling parameter. The UV-Visible properties of extract dyes in solvent and on a photoelectrode substrate were studied. The results show that the absorption maxima of all natural dyes applied on TiO₂ substrate are bathochromically shifted by 9, 4, 13, 11 and 15 nm for A, B, C, D and E compared to the corresponding dye spectra in solutions due to partial J-aggregation of dye molecules. Cyclic voltammetry (CV) data show that the energy levels of HOMO of all extractions are more positive than the electrolyte potentials and the energy levels of LUMO of all natural dyes are more negative than the conduction band of TiO₂. Thus, electron injection and natural dye regeneration are permitted and all natural

dyes are suitable for use in a DSSCs' device. Besides, the colourimetric attributes of five natural dye solutions were investigated. Results showed that dye solutions extracted in basic environment are darker and less saturated than the dye solutions extracted in acidic condition and the neutral one. Meanwhile, the neutral dye solution, as well as those extracted in acidic environment benefit from more redness tint effect while the dye solutions extracted in basic condition are more bluish. Moreover, the dye solution extracted in basic environment show more optical differences with the neutral dye solution rather than those dye extracted in acidic condition. Finally, DSSCs devices were fabricated by employing natural dyes as photosensitizers. Power conversion efficiency of 0.57%, 1.15%, 1.02%, 0.35% and 0.15% were achieved for A, B, C, D and E, respectively. The results show that the photocurrent and power conversion efficiency increased by the acidification of dye extracted from *S. ebulus*.

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