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Full Length Article

Extraction and application of natural pigments for fabrication of green dye-sensitized solar cells

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ABSTRACT

Four dye-sensitized solar cell devices are designed and fabricated based on natural dyes extracted from Celosia Cristata, Saffron, Cynoglossum, and eggplant peel, as photosensitizers. The UV–vis technique has been served to determine maximum absorption of natural extract and pre-dyed photoanode. The Fourier transform infrared (FT-IR) was employed to cover the presence of functional groups. The cyclic voltammetry method has been employed to assess the possibility of charge transfer from dried natural dyes to the photoelectrode. The performance of natural-based dye-sensitized solar cells is determined subsequently. The highest power conversion efficiency was ca. 1.38%, which belonged to Celosia Cristata extract. The devices were examined for higher efficiencies, individually, co-sensitized arrangement and/or in tandem with each other.

1. Introduction

Todays, the primary sources of energy production are fossil fuels such as oil, gasoline, natural gas and coal which cause environmental pollution. On the other hand, these resources are not endless [1]. Dye-sensitized solar cells (DSSCs) are ideal and unlimited options for generating electrical energy [2]. The cause for the spectacular growth of this technology is low cost and environmentally friendly [3]. The photosensitizers as a key component of DSSCs devices play the role of electron production [4]. Several researches have focused on studies of photosensitizers engineering for the improvement of a dye-sensitized solar cell performance [5].

The photosensitizers used in DSSCs devices can be divided into metal-based complex dyes, organic dyes and natural dyes [6]. The literature indicates that metal-based complex dyes and organic dyes have been frequently applied in the manufacture of DSSCs devices, but they could not be good choices for environmental and cost perspective [7]. Natural dyes obtained from flowers, leaves, fruits and roots are promising selection for fabrication of DSSCs device due to inexpensive, abundant and sustainable [8]. Polo and Iha extracted blue anthocyanin from various fruits as sensitizers and applied them in DSSCs, and attained the higher efficiency in case of Jabotica [9]. Chang and Lo used natural dyes extracted from pomegranate leaf and mulberry and reported power conversion efficiency of 0.59 and 0.722% for the resulting cells, respectively [10]. Hosseinnezhad et al. extracted five natural dyes and used in dye-sensitized solar cells and a higher power conversion efficiency of 1.41% has been achieved for radish [11]. Some studies were also constructed to improve the efficiency of natural dye based DSSCs, for instance Sandquist and McHale fabricated a green DSSCs based on beet root and reported an efficiency about 2.70% [12]; and Calogero et al. worked on a different type of berry and reached efficiency of 2.06% in case of Sicilian prickly pear [13]. Hosseinnezhad et al. fabricated a clean and low cost DSSCs based on Sambucusebulus extraction and investigated various pH in the extraction to increase the PCE. The results show that the higher PCE has obtained for 1 N HCl media [14]. Mozaffari et al. developed DSSCs based on Siahkooti fruit natural sensitizers with and without purification. At best, the power efficiency was 0.32% prior to purification [15].

In this study, four sensitizer from nature sources was extracted to enhance the power conversion efficiency of solar cell devices. Thus, different groups of natural dyes are extracted from Celosia Cristata, Saffron petals, Cynoglossum, and eggplant peel of Iran farms. The performance of derivatives towards UV–vis properties of extractions in the solution and on a photoelectrode substrate was then investigated. The FT-IR ATR spectra were examined to assess pre-dye treated TiO_2 substrate. The energy level properties of the aforesaid environmentally dyes as sensitizers were measured on account of their potential in transferring charge from dye molecules to photelectrode. The photovoltaic properties were detected in cells

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configured individually, in co-sensitized arrangement and in tandem with each other, and their efficiencies were compared.

2. Experimental

2.1. Materials and instruments

Natural resources used in this study were Celosia Cristata, Saffron petals, Cynoglossum, and eggplant skin, all received from trees and underbrush grown in Iran. All chemical reagents and solvents were analytical grades provided by Merck Co. and used without further purification. Transparent conducting oxide, FTO (F-doped SnO_2, DyeSol), TiO_2 pastes, scattering layer and Di-tetrabutylammonium Cis-bis(isothiocyanato)bis(2,2′-bipyridyl-4,4′-dicarboxylato)-Ruthenium (II) (N719 Grade) were purchased from Sharif Solar Co. The absorbance spectra of the green sensitizers were measured in the UV–vis range using Cecil 9200 double beam transmission spectrophotometer (Super Aquarius spectrophotometer, UK). FT-IR ATR measurements were carried out on a Perkin Elmer instrument (Spectrum one, wavelength range: 7.800–350 cm^{-1} with beam splitter, USA) to place the functional groups present in the samples.

2.2. Preparation of green sensitizers

The organic extract process was utilized for preparation of green sensitizer. The natural plant (Celosia Cristata, Saffron petals, Cynoglossum, and eggplant peel) was washed with water and vacuum dried at 45 °C. The cleaned and ground plants (10 g) were respectively dipped into the extracting solution which was consisted of ethanol (50 ml) for 48 h under darkness to prevent light decomposition. Then the dye solution was filtered repeatedly and were concentrated with a rotary evaporator at 30 °C to obtain green sensitizers.

2.3. Electrochemical measurements

Electrochemical measurements of the natural dyes were carried out in solution by cyclic voltammetry (CV) method. The oxidation potential (E_{ox}) was measured using three small-sized electrodes. Ag quasi reference electrode (QRE) was used as the reference. Platinum wires were used as the working and the counter electrodes. All electrode potentials were calibrated with respect to ferrocene (Fc)/ferrocenium (Fc^+) redox couplet. An acetonitrile solution of each dye containing tetrabutylammonium perchlorate (0.1 mol dm^{-3}) and ferrocene (ca. 1 mmol dm^{-3}) was prepared. The electrochemical measurements were performed at a scan rate of 100 mV s^{-1} [16].

2.4. Fabrication of DSSCs

In this work, three types of DSSCs containing green sensitizers were fabricated and put into practice, individually, co-sensitized, and tandem.

In case of individual solar cells, a nanocrystalline TiO_2 film was coated on a FTO coated glass support. The dye extracts were adsorbed by dipping the coated glass for 18 h in natural dyes. Then, the film was washed with ethanol solvent. A 1:4 vol ratio of acenonitrile-ethylenecarbonate containing 0.5 mol dm^{-3} tetrabutyl ammonium iodide was employed as electrolyte. Next, dye-adsorbed TiO_2 electrode, the Pt counter electrode and the electrolyte solution were assembled into a sealed sandwich type solar cell [11,16,17]. Reference spectrum was obtained under monochromatic light with a constant photon number of 5×10^{15} photons cm^{-2} s^{-1}. J-V characteristics were determined under illumination with AM 1.5 simulated sunlight (100 mW cm^{-2}) via a shading mask (5.0 mm × 4 mm) by using a Bunko-Keiki CEP-2000 system.

For construction of co-sensitized solar cells, the photoelectrode were prepared by employing two dye layers, where dried TiO_2 was immersed in a first natural sensitizer for 18 h followed by drying at room temperature under dark condition. Tetraethyl ammonium hydroxide in methanol and ethylene glycol solutions were used as desorption solutions. The sensitized TiO_2 was dipped into solution for 30 s and washed with ethanol for 20 s. Desorption process was repeated three times followed by immersion of dried photoelectrode into the second natural sensitizers for 8 h and washed with ethanol [18,19].

The third kind of configuration was the case where two dye-sensitized natural dyes enclosed in tandem. The configuration was as Fluorine doped tin oxide (FTO) glass/TiO_2/dye 1/electrolyte/semi-transparent Pt-FTO glass/FTO glass/TiO_2 /dye 2/electrolyte/Pt-FTO glass. More details on solar cells of this type are available elsewhere [20].

3. Results and discussion

Natural dyes derived from plants, vegetable and minerals are environmentally friendly, low cost and available everywhere compared to organic derivatives used in dye-sensitized solar cells [21]. Figure 1 displays natural dyes used in this study extracted from Celosia Cristata, Saffron petals, Cynoglossumand eggplant peel. Natural dyes based on anthocyanine were obtained from Celosia Cristata, eggplant peel and Cynoglossum and flavonoids was taken from Saffron petals. In this way, different families could be tested for their sensitization effects on power conversion efficiency when used in dye-sensitized solar cells. There was a clue that all natural dyes used in this work could be linked to the TiO_2 surface in DSSCs thanks to the presence of the carbonyl and hydroxyl groups

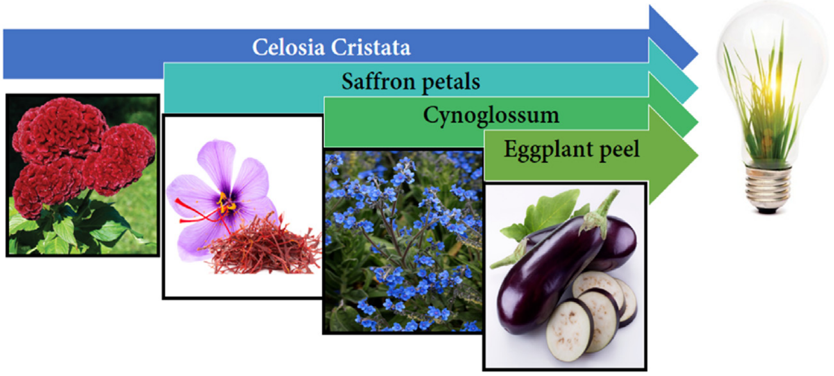
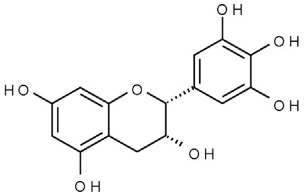
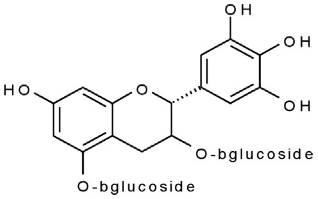
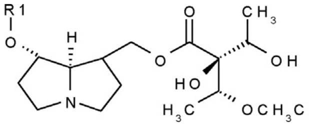
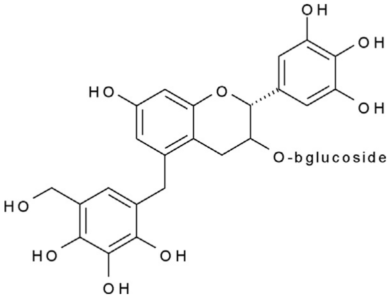


Fig. 1. The origin of the natural dye sources used in this study.

Table 1
Chemical structure of natural dyes.

Plant name	Molecular structure	Ref.
Celosia cristata		[22]
Saffron petals		[23]
Cynoglossum		[24]
Eggplant peel		[25]

[21]. The chemical structure of natural dyes molecules presented in Table 1.

Table 2
Absorption of the natural extracts used in this work.

Natural sources	λ_{max} (nm)[a]	ε ($M^{-1}\,cm^{-1}$)	λ_{max} (nm)[b]	$\Delta\lambda$ (nm)
Celosia Cristata	510	21123	521	11
Saffron	468	22357	483	15
Cynoglossum	573	17130	591	18
Eggplant peel	548	23189	562	14

[a] Maximum Absorption of dyes measured in solution.
[b] Absorption maximum of dye adsorbed on the surface of TiO_2.

3.1. *Absorption spectra of the natural dyes*

Some useful insights can be achieved for the absorption transition of dye molecules featured by absorption spectra [15]. Figure 2 shows the UV–vis absorption spectra of the condensed ethanolic extract of Celosia Cristata, Saffron petals, Cynoglossum, and eggplant peel extraction. The absorption spectra of Saffron petals show that three distinguished peak, but Celosia Cristata, Cynoglossum, and eggplant peels members of anthocyanin group showing a broad absorption [26,27].

Some crucial information, including the wavelength of maximum absorption (λ_{max}) of extracts and λ_{max} of the corresponding extract adsorbed on TiO_2 films are extracted from Fig. 1 and the molar absorption coefficients presented in Table 2. The absorption band of all extracts positioned to TiO_2 substrate show a bathochromic shift compared to the extract in solution. Aggregation of dye molecules on the substrate photoelectrode is the main reason of this phenomena, although it has affected the interaction between dye molecules and polar solvent in solution media [21,28]. That similar observations were reported for battalion dyes by Hemmatzadeh [29] and anthocyanin dyes by Teoli [30].

3.2. *FT-IR spectroscopy analysis*

FT-IR spectra of the dried natural dyes and pre-dye treated TiO_2 nanoparticles were carefully analyzed to assess chemical interactions. The intense peak of the dried natural dyes around 3400 cm^{-1} is indicative of the OH stretching modes, while two peaks appeared at around 2900 and 2800 cm^{-1} were attributed to C—H stretching.

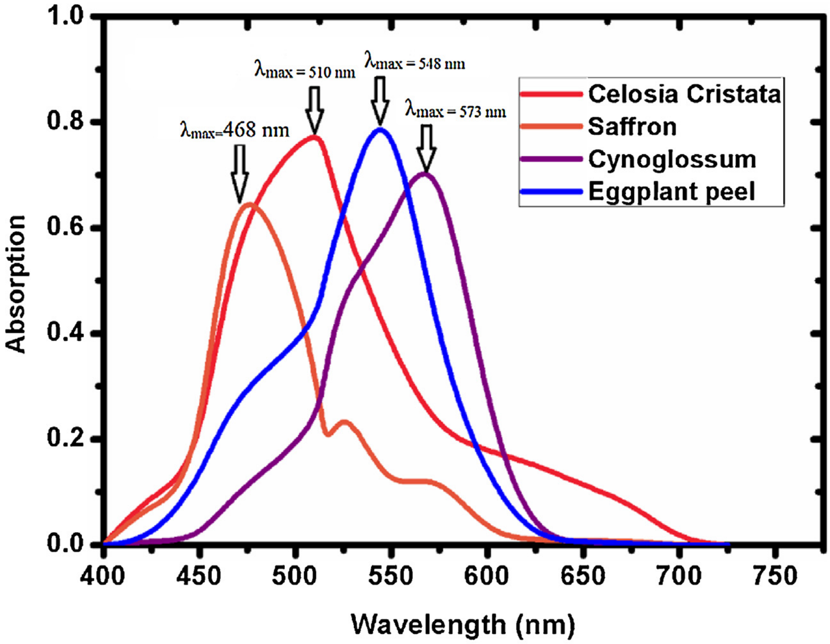


Fig. 2. UV–vis absorption spectra of the ethanolic extract of natural dyes.

Table 3
FT-IR peaks of the natural dyes and pre-dye treated TiO_2.

Natural dye	Dried natural dye			Pre-dye treated TiO_2	
	OH (cm^{-1})	C═O (cm^{-1})	C—H (cm^{-1})	Ti-—O (cm^{-1})	Ti—O—C (cm^{-1})
Celosia Cristata	3451	1717	2931	632, 740	1059
Saffron	3417	1725	2940	639, 745	1063
Cynoglossum	3423	1734	2928	641, 744	1066
Eggplant peel	3441	1728	2938	640, 743	1065

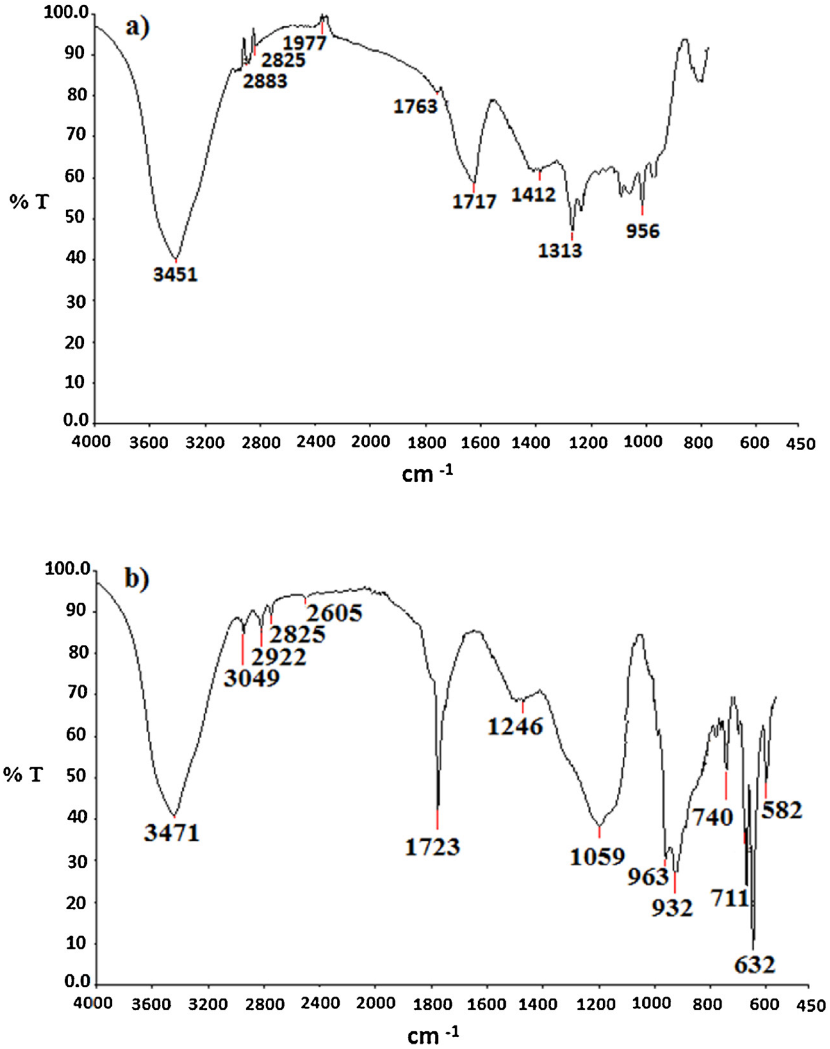


Fig. 3. FT-IR spectra of the extract of Celosia Cristata as (a) free dye and (b) pre dye treated on TiO_2.

The peak around 1700 cm^{-1} is a signature of the C═O stretching [30]. The FT-IR ATR spectra of the pre-dye treated TiO_2 depicts the peak of Ti—O band at 450–1000 cm^{-1}, resulting from the reaction between dye molecules and photoelectrode. To produce a comparative view, FT-IR peaks for all natural dyes are summarized in Table 3, confirming the presence of hydroxyl and carbonyl groups in the molecular structure of natural dye and bonding between dye molecules and TiO_2 nanoparticles. Figure 3 shows the FT-IR spectra of the extract of Celosia Cristata as (a) free dye and (b) pre dye treated on TiO_2.

3.3. *Electrochemical properties*

Inspired by previous studies, the cyclic voltammetry method was applied in evaluating the feasibility of electron transfer from the excited natural dyes to the conduction band of TiO_2 and the dye regeneration by redox electrolyte. The cyclic voltammetry was carried out in the solution [15,31]. The oxidation peal potential (E_{ox}) in a solution for Celosia Cristata, Saffron, Cynoglossum and Eggplant peel was estimated to be +0.80, +0.82, +0.85 and 0.84 V vs. NHE, respectively. The E_{ox} is considered as the highest occupied molecular orbital (HOMO) energy levels. These ground state oxidation potentials of all natural dyes are positive than the redox potential of I^-/I_3^- (0.4 V vs NHE) which indicates that there are enough driving forces for the dyes regeneration. The excited state oxidation potentials (LUMO) in the same order were −1.30, −1.21, −1.38 and −1.40 V, respectively. The results suggest that the LUMO state lies above the conduction band of TiO_2, and makes possible the electron injection from each excited dye molecule to photoelectrode conduction band. This means that the extraction from Celosia Cristata,

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

Dye	J_{SC} (mA cm^{-2})	V_{OC} (V)	FF (%)	η (%)
Celosia Cristata	4.82	0.52	0.55	1.38
Saffron	2.77	0.36	0.52	0.52
Cynoglossum	4.11	0.48	0.57	1.12
Eggplant peel	3.40	0.35	0.55	0.65
N719	13.55	0.88	0.71	8.47

Saffron petals, Cynoglossum, and eggplant peel can admit electron from electrolyte layer due to the HOMO level dye molecules being more positive than the level of energy in the electrolyte. Therefore, they can be all considered in DSSC fabrication as a sensitizer [32].

3.4. Photovoltaic properties of DSSCs based on natural dyes

The photovoltaic properties of DSSCs based on natural dyes were studied by measuring the J-V curve under the standard global AM 1.5 solar irradiation [33]. The potential of obtaining natural dyes to enhance power conversion efficiency was evaluated changing the type and configuration of DSSCs from individual to Co-sensitized and tandem arrangements. Figure 4a) shows typical results of the J-V curves in case of individual DSSCs in which natural dyes are applied as photosensitizers. The photovoltaic parameters are also presented in Table 4. It is apparent that the highest photocurrent was obtained in case of DSSC sensitized with Celosia Cristata from anthocyanin groups. It is also abundant that photocurrent value depends on the binding affinity and previous study suggest that the amounts of adsorbed extractions (natural dye) are sometimes different under the same concentration and thickness of TiO_2 film [28,33]. Therefore, the low photocurrent can be attributed to the weak bonding between dye molecules and the surface of TiO_2 as photoelectrode [28,34]. The highest open circuit voltage was obtained by the DSSC sensitized with Celosia Cristata due to its high conduction band appeared in dye-molecules [31] thus, the best properties were achieved for an anthocyanin family member. The conversion efficiencies of cells based on natural solutions Celosia Cristata, Saffron, Cynoglossum and Eggplant peel are of 1.38, 0.52, 1.12 and 0.65, respectively. The achieved power conversion efficiency for natural dyes are equivalent or even better with respect to previous studies [35–37]. In order to prevent degradation of natural dyes, DSSCs devices should be fabricated under inert atmosphere [38]. Natural dyes mostly have —OH and —O ligands, instead they lack acid substituents. On the other hand, the majority of organic and organometallic dyes are bonded to photoelectrode *via* their anchoring groups such as acrylic acid, cyanoacrylic acid and etc. Lower efficiencies and open circuit voltage values of natural dyes in DSSCs structures compared to organic dyes and organometallic pigments is due to such bonding and slow rate of electron transfer [39,40]. The stability of dye-sensitized solar cells based on natural dyes were tested by monitoring the photovoltaic properties under solar light simulation. Remarkably, no significant alteration was followed after 50 h. The performance of Saffron was decreased, while in case of Celosia Cristata, Cynoglossum and Eggplant peel extract remained unchanged after several hours (100 h). The results presented here in stability state are similar to data reported by other authors [38]. The DSSCs based on N719 were prepared using a standard [16] method to compare with the performance of the DSSCs sensitized by a natural dye. However, the DSSCs sensitized by N719 as standard dye show better performance that the DSSCs based on natural dyes. The DSSCs based on N719 as standard dye, due to reducing the charge recombination at the dye molecules and photoelectrode interface, show higher conversion efficiency. On the other hand, the N719 dye standard contains the expensive ruthenium metal which economically limits its general use.

Table 5
Photovoltaic parameters of Co-DSSCs based on the natural dye extraction.

Co-sensitized dye		J_{SC} (mA cm^{-2})	V_{OC} (V)	FF (%)	η (%)
Dye 1	Dye 2				
Celosia Cristata	Saffron	7.68	0.46	0.54	1.90
Celosia Cristata	Cynoglossum	9.11	0.50	0.51	2.32
Celosia Cristata	Eggplant peel	8.36	0.44	0.53	1.95
Cynoglossum	Saffron	6.98	0.51	0.51	1.81
Cynoglossum	Eggplant peel	7.82	0.44	0.52	1.79
Eggplant peel	Saffron	6.58	0.37	0.50	1.22

Table 6
Photovoltaic parameters of T-DSSCs based on the natural dye extracts.

Tandem DSSCs		J_{SC} (mA cm^{-2})	V_{OC} (V)	FF (%)	η (%)
Top cell	Bottom cell				
Celosia Cristata	Eggplant peel	4.17	0.84	0.52	1.82
Saffron	Eggplant peel	3.11	0.77	0.51	1.22
Cynoglossum	Eggplant peel	3.85	0.79	0.52	1.58
Saffron	Cynoglossum	3.48	0.82	0.52	1.48
Saffron	Celosia Cristata	3.81	0.89	0.51	1.73
Celosia Cristata	Cynoglossum	4.58	1.11	0.51	2.59
Cynoglossum	Celosia Cristata	4.49	1.12	0.52	2.61

With the aim of increasing the power conversion efficiency of cells, all natural dyes are applied in the fabrication of co-sensitized solar cells. The J-V curves of co-sensitized solar cells are shown in Fig. 4b) and photovoltaic properties are summarized in Table 5. Overall, co-sensitization based on any pair of the aforementioned sensitizers with respect to individually apply natural dyes resulted in higher photocurrent values. There is a proof that sensitized processing in co-sensitized solar cells provides opportunity to have the inherent power of dye molecules [18]. The highest power conversion efficiency was obtained in co-sensitization of Celosia Cristata and Cynoglossum about, which was about 2.32%. Overall, the power conversion efficiency of solar cells applied individually based on natural dyes takes values lower than 2%, while co-sensitization could improve the performance of DSSCs [32]. Among reports available in the literature, Co-DSSCs based on natural dyes of Ixora and Canariumodontophyllum showed 1.55% power conversion efficiency [18]. Chang et al. employed two natural dyes extracted from pomegranate leaves and mulberry fruit through cocktails process reaching a conversion efficiency of 0.722% [10]. Although Co-DSSCs containing organic and organometallic dyes show greater efficiency with respect to natural-based Co-DSSCs [34,35], the latter are ecofriendly and low cost sources sensitizers. Interestingly, however, both types of Co-DSSCs, i.e., chemical and natural, represent a similar durability.

The third configuration could be applying two types of solar cells in tandem. Light harvesting from the visible irradiation can be increased using two or more photosensitizer, which is the case when a tandem arrangement is considered [41]. In tandem dye-sensitized solar cells (T-DSSCs), the top component contains dye molecules with shorter wavelength absorption, while the bottom cell is sensitized by dye molecules with longer wavelength absorption [20]. Based on the results obtained in case of individual DSSCs and Co-DSSCs, some tandem configurations are applied in sensitization. In tandem configuration, two single dye-sensitized solar cell are connected in series, hence the amount of efficiency is controlled by the solar cell with the lower photocurrent. As a proof of appropriate connection of cells, the photovoltage of the T-DSSCs takes the value equivalent of the sum of the voltages of the individual DSSCs previously discussed (Table 4). The photovoltaic properties of T-DSSCs are summarized in Table 6. According to the results extracted from the J-V curves of T-DSSCs in Fig. 4c), application of tandem configuration resulted in a three-fold increase in

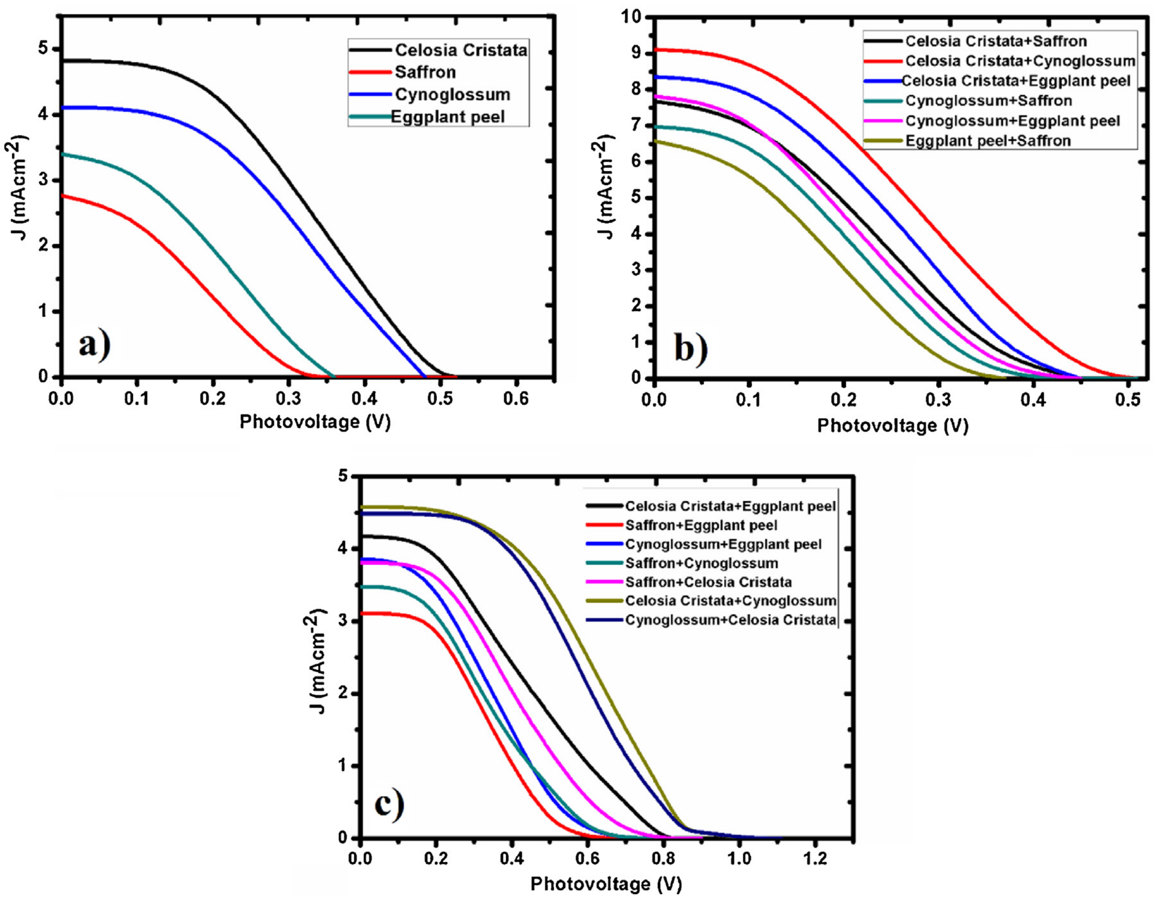


Fig. 4. Photocurrent density-voltage (J-V) curves of the DSSCs based on natural dyes (a) individual DSSCs, (b) Co-DSSCs and (c) T-DSSCs.

the efficiency of natural dye compared to the case where it was applied in cells individually. In some case, such as using Celosia Cristata and Cynoglossum, the efficiency in the tandem configuration is higher than the co-sensitization, because of the fact that we employed natural dyes with high performance in the T-DSSCs. In T-DSSCs, maximum absorption of light has been increased due to using two dyes with complementary absorption wavelength [35]. Thus, choosing the best configuration is determined depending on absorption wavelength needed to be complementary or as high as possible. The maximum absorption of Celosia Cristata and Cynoglossum are very close, 508 nm and 507 nm, respectively, so both possible structures were examined. When the Celosia Cristata is in the top cell, the efficiency is of 2.59% and the efficiency is of 2.61% for a sensitized bottom cell by Celosia Cristata.

In this research, four plants are selected as green photosensitizers, which are all grown in Iran and different configuration of DSSCs devices are assembled and evaluated. Although the power conversion efficiency of prepared DSSCs isn't that high, there is reason behind their benefit as a natural photosensitizer in view of their eco-friendly and low cost [11]. Three arrangements are studied in preparation of DSSCs devices: individual DSSCs, Co-DSSCs and T-DSSCs. The results illustrate that Co-DSSCs present higher efficiency than the individual DSSCs due to expansion of the absorption range [42]. A newly proposed method for increasing of power conversion efficiency of DSSCs is a Tandem structure [39]. The UV–vis evaluation show that saffron and other extractions have different absorption range and could be applied on T-DSSCs configuration. T-DSSCs configuration is a good choice for obtaining high performance because T-DSSCs provides a wider spectrum of absorption. The highest power conversion efficiency was achieved for T-DSSCs based on Cynoglossun and Celosia cristata extraction as natural photosensitizer.

4. Conclusions

Natural dyes extracted from Celosia Cristata, Saffron, Cynoglossum and Eggplant peel grown in Iran are applied in the fabrication of DSSCs. The UV–vis absorption and FT-IR spectra of natural extracts and pre-dye treated TiO_2 confirms the formation of bonding between all dye molecules and photoelectrode substrate. The cyclic voltammetry data indicate that all natural dyes can be used in DSSCs structure and accept electrons from the electrolyte. The dye-sensitized solar cells were then fabricated using natural extracts and the power conversion efficiency of Celosia Cristata, Saffron, Cynoglossum and Eggplant peel followed the order: 1.38%, 0.52%, 1.12% and 0.65%. In order to compare the efficiency and capability of the fabricated cells, different configurations are constructed. The Co-sensitization method was responsible for improving the performance of DSSCs, where 2.32% was marked as the highest efficiency obtained has been obtained for Celosia Cristata and Cynoglossum Co-DSSC configuration. The tandem DSSCs were fabricated from natural extracts and the photovoltaic properties of all devices have been expectedly increased due to $V_{oc\text{-}tan}$ being the summation of two V_{oc} of the individual DSSCs.

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