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Eco-friendly natural dyes as sensitizers for dye-sensitized solar cells (DSSCs)

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Abstract

Six natural dyes D1–D6 were extracted from various parts of *Hibiscus rosa sinesis, Brassica oleracea, Celosia argentea, Rosa damascena, Rosa macdub* (red and yellow) by maceration with ethanol for 24 hours for dye-sensitized solar cells. Their photophysical, electrochemical and photovoltaic studies were carried out. UV-Vis absorption spectra of the dyes D1–D6 showed two distinct bands i.e. $\pi - \pi^*$ and ICT band. Out of all the extracted dyes, the dye D1 gave best absorption of ICT band at λ_{max} 534 nm. Cyclic voltammetry of the dyes D1–D6 was carried out which revealed the distinct oxidation and reduction peaks in D1 and D2 dyes while in other dyes these peaks were weak. The photovoltaic performance of the dyes D1–D6 was also measured by making sandwich type DSSCs. Their *J-V* curves showed open circuit voltage values of the dyes were 0.36V, 0.37V, 0.36V, 0.34V, 0.34V, 0.39V and short circuit current density values are 5.48 mA/cm^2, 5.69 mA/cm^2, 1.71 mA/cm^2, 3.99 mA/cm^2, 2.71 mA/cm^2 and 5.18 mA/cm^2 respectively. The best sunlight to energy conversion efficiency was obtained from D1 dye which has better UV absorption and it gave higher short circuit current density which leads to highest efficiency. Other dyes gave the efficiency in the following order D2=0.9% > D6=0.8% > D4=0.5% > D5=0.3% > D3=0.2%.

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Introduction

The energy sector has vital role in sustaining the modern social and economic world. The production of energy has been one of the core issues confronting mankind till today. World population was 2500 million in 1950, which has already increased to 7000 million till 2011 and an increase of 90 million peoples every year on this earth has been estimated (Pekka, 2013). With the rapid increase in population, requirement of energy has increased enormously. Hoffert *et al.*, 1998 projected that world energy consumption rate will double from 13.7 TW to 27 TW by 2050 and will triple to 43 TW by 2100.

World is facing the energy shortage and this shortage is even going more worse in the developing countries (Liu *et al.*, 2008; Karakus *et al.*, 2017). At present, around 1.317 billion people all over the world are living without electricity (Abdullah *et al.*, 2017). Demand of energy is continuously increasing due to overpopulation and industrial proliferation (Wakeel *et al*, 2016). Substitution of conventional sources of energy such as fossil fuels with the renewable energy resources like solar power is the main challenge of today's world (Nazeeruddin *et al.*, 2011). The sun offers an ultimate way out to this challenge. The surface of earth receives about 13.6 TW of energy from the sun in only one hour which is more than what we consume annually (Calogero *et al.*, 2015). Photovoltaic devices convert sunlight into electricity (Parida *et al.*, 2011). Out of all photovoltaic techniques, dye-sensitized solar cells (DSSCs) also known as third generation solar cells have gained significant attention for the conversion of solar power into electricity with the help of sensitizing agents (O'Regan and Gratzel, 1991; Hao *et al.*, 2006) synthetic, natural, metallic, organic or inorganic.

Synthetic organic dyes with metal or without metal have been synthesized by various groups and successfully applied for the dye-sensitized solar cells. Though these dyes gave good results but they have much complication in synthesis and purifications. (Ayalew and Ayele, 2016). Furthermore, synthetic dyes are not only expensive but also tend to undergo degradation on the photoanode surface due to environmental influences (Zhang *et al.*, 2008).

It has been reported by Enciso and Cerda (2016) that DSSCs fabricated with natural dyes evidenced the efficiency values up to 2% along with good stability. In 2008, Calogero and Marco acquired an efficiency of 0.66% using juice of red Sicilian orange as natural sensitizer for DSSCs. Coumarin dyes were also used as sensitizers with reported efficiency of 7.6% (Wang *et al.*, 2007). The efficiency of DSSCs mainly depends on the absorption spectrum of the dye being used as photosensitizer and the electron transfer activity to the electrode (Dey *et al.*, 2016). Anthocyanins from a variety of plants gave various photo-sensitizing performances. They are mainly responsible for cyanic colors ranging from pink to red then violet and deep blue (Narayan, 2012). According to the findings of Shahid *et al.*, (2013), a variety of natural dyes illustrated different solar conversion efficiencies depending on dye source and its composition and interaction between dye molecules and nanostructured photo-electrode.

Thus attention has been diverted toward natural dyes as these are versatile and easily available all over the world. Major natural dyes and pigments include chlorophyll, anthocyanins, flavonoids and carotenoids, which have been applied for dye-sensitized solar cells (Hernandez-Martinez *et al.*, 2013; Lim *et al.*, 2015). Chlorophyll is well-known and prime important pigment in term of absorbing specific wavelength of the visible light and its magnesium ion in the centre play an important role in absorbing sunlight (Rosana, 2015). But chlorophyll is not an active sensitizer due to lack in binding sites with the TiO_2 surface (Kartini *et al.*, 2015). Hence different approaches were adopted to improve the conversion of sunlight to electricity from natural resources. Which include synergetic influence of natural dyes including anthocyanins, carotenoids, flavonoids etc because they have carboxyl as well as hydroxyl functional groups that can bind easily with the TiO_2 surface (Vargas *et al.*, 2000). Furthermore, chlorophyll absorb light over the wide range and have longer π-conjugation system while other dyes and pigments played role for stabilization and higher molar extinction coefficient (Kim *et al.*, 2013).

Owing to fundamental advantages of natural dyes of being easily available, lower cost, simplified extraction process, green technology and ability to stabilize in the environmental stress, these are preferred over synthetic dyes (Narayan, 2012; Hambali *et al.*, 2014). Keeping in view of all factors, the current study was designed to evaluate the six natural dyes for dye-sensitized solar cells. Six different type of flowers from *Hibiscus rosa sinesis* (Red colored flower), *Brasicca oleracea* (Purple colored flower), *Celosia argentea* (Deep red color), *Rosa damascena* (Bright Red color) and *Rosa macdub* (yellow and red flowers) for application in dye-sensitized solar cells. Their photo-physical, electrochemical and photovoltaic studies are carried out.

Materials and methods

Natural Dye Extraction from Flowers

Extraction of natural dyes from the plant material was carried out via the reported procedure by Senthil *et al.*, 2014 with minor modifications. 250 gm of six different types of locally available flower species namely *Hibiscus rosa sinesis, Brasicca oleracea, Celosia argentea, Rosa damascena* and *Rosa macdub* were collected and codes were assigned to each sample from D1–D6 respectively. After collection, for the flower species like D1, D3, D4, D5 and D6, petals were separated and cleaned in order to remove dust, buds and pollens. Well-cleaned samples were finely chopped and soaked in 1.5 L of ethanol with few drops of conc. nitric acid in amber colored bottles for 24 hours. The residual solids were filtered. The filtrate was collected in a separatory funnel and washed with hexane several times to remove all fatty materials and chlorophyll present in the extract. The dyes D1, D2, D4 and D6 have bright red color while dyes D3 and D5 have yellow color. All these dyes were collected and 100mL of each solvent was removed through rotary evaporator. The extracted dyes (50mL) were stored in reagent glass bottles in dark in a refrigerator.

UV-Vis Absorption Spectroscopy

The photo-absorbance of natural dyes were measured through UV-visible spectrophotometry. The extracted dyes were diluted 10 times with ethanol. The absorption spectrum was recorded within the spectral range of 350-800 nm after adjusting the baseline with the help of Hitachi U-3300 spectrophotometer.

Cyclic Voltammetry Measurements

Electrochemical properties of the extracted natural dyes D1–D6 were analyzed through cyclic voltammetry by using the conventional three-electrode system in a compartment using an Eco Chemie Autolab PGSTAT 302 Potentiostat/ Galvanostat drived by GPES 4.9 software (Utrecht, The Netherlands) by following the reported method by Kavitha *et al.*, 2017 with slight alterations. Platinum was used as working electrode in addition to counter electrode and Ag/AgCl wire was used as the reference electrode at a scan rate of 50 mV/s. All three electrodes were immersed in supporting electrolyte solution containing natural dye at room temperature. 0.1 M Tetrabuty lammonium Perchlorate (TBAClO$_4$) solution in distilled water was used as supporting electrolyte. All solutions were degassed with argon before analysis.

Preparation of Working Electrode (TiO$_2$ Coated FTO Glass)

The cells were assembled by using FTO glass as working electrode coated with nanostructured particles of TiO$_2$. A suspension of TiO$_2$ nanoparticles was prepared by following the procedure previously adopted by Ghani *et al.* (2014). At first, FTO glass was cut into 1x1.5 dimensions pieces. The glass pieces were thoroughly washed with detergent using ultrasonic bath for 20 minutes and a TiO$_2$ blocking layer was coated on the FTO glass substrates by immersing in 50 mM TiCl$_4$ ethanol solution for 30 min at a temperature of 70 ^{o}C. The nanoparticles colloid was prepared by combining 2 g TiO$_2$ powder, 4 mL deionized water (DI), 0.1 mL Triton X and 0.8 mL of acetylacetone. The mixture was placed for stirring in an ultrasonic bath for 2 hours. The mesoporous film of colloid was homogenously deposited on TiO$_2$ blocking layer through doctor blade method and sintered for 450 ^{o}C for 30 minutes. After cooling down, the TiO$_2$ anode was immersed in concentrated natural dye extract for 24 hours at room temperature.

Fabrication of DSSCs with Natural Dyes

The light harvesting properties of the DSSCs fabricated with six extracted natural dyes were analyzed after drying the TiO_2 coated photoanode. The photoanode and counter electrode were sandwiched together face to face. The two electrodes were held jointly with the help of binder clips. The fabrication of DSSC was completed by injecting a drop of Iodine/Tri Iodide (I^-/I^{-3}) electrolyte in the aperture between photoanode (working electrode) and counter electrode which was made with graphite. The electrolyte helps to maintain the electrical path among both electrodes. A solar simulator (SS80AAA by Photo Emission Tech. Inc. USA) was used under solar irradiation power of 100 mW/cm^2 to scrutinize the photoelectric conversion efficiency of DSSCs. The *J-V* curves were obtained using a digital electrometer which is connected to a computer. The short circuit current density J_{sc} and open circuit voltage V_{oc} values were evaluated from the *J-V* curves. The fill factor *ff* and average conversion efficiency (η) of the dye-sensitized solar cell was calculated for each dye sample with the help of following current voltage relationship equations.

$$ff = I_{max} \times V_{max} / J_{sc} \times V_{oc} \quad (1)$$

$$\eta = ff \times J_{sc} \times V_{oc} \quad (2)$$

where I_{max} is the maximum power point current (mA/cm^2) and V_{max} is the maximum power point voltage (V).

Results & discussion

UV Absorption of Natural Dyes

A total number of six natural dyes were used as sensitizers for DSSCs. The absorption of the natural dyes extracted and diluted with ethanol was measured using UV-Visible spectrophotometer. The UV-Vis absorption data of the dyes D1–D6 extracted with ethanol from flowers is enlisted in Table 1.

Fig. 1. shows the representative UV-Vis light absorption spectra for the ethanolic extracts of all the dyes. It is evident from the Fig. 1. that the absorption peaks of all the D1–D6 dye solutions were within the UV and Visible range of spectrum from 358 nm to 534 nm.

All the dyes have two distinct bands one π-π* band (225-425 nm) and ICT band (500-625 nm). The maximum absorption 534 nm at ICT band was observed in dye D1 followed by D2 with 532 nm absorption. The intensity of absorption was D1>D2, D5, D6>D4>D3 respectively. The absorption spectra of three of the dyes D2, D5 and D6 shows an absorption peak at 532 nm. D4 dye has an absorption peak at 412 nm while the lowest absorption peak is observed in case of D3 dye extract which has a yellowish color. As shown in Fig. 1. D1 peak is at highest wavelength while other five samples also have similar absorption patterns. This absorption peak attributes the presence of anthocyanins, which are known as group of natural phenolic compounds and is the main component of all these natural dye extracts.

Table 1. Wavelength and maximum absorption of natural extracts in visible region of spectrum.

Sample code	Botanical Name	Part used for dye extraction	Wavelength ICT band (nm)	Absorbance
D1	*Hibiscus rosa sinensis*	Petals	534 nm	1.125
D2	*Brassica oleracea*	Flower	532 nm	0.686
D3	*Celosia argentea*	Flower	358 nm	0.642
D4	*Rosa damascena*	Petals	412 nm	0.78
D5	*Rosa macdub*	Petals	532 nm	0.199
D6	*Rosa macdub*	Petals	532 nm	0.61

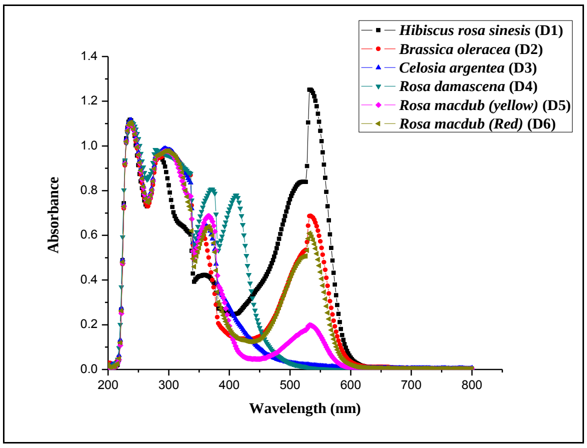

Fig. 1. UV-Vis absorption spectra of all six natural dye sensitizers (D1-D6).

Cyclic Voltammetry of Natural Dyes

The electrochemical behavior of natural dyes were investigated by means of cyclic voltammetry. The cyclic voltammograms of all six natural extracts are presented in Fig. 2. The D1 and D2 dye extracts exhibits clear oxidation peaks and proved to be more suitable for their application in DSSCs. The presence of hydroxyl and carbonyl groups in anthocyanins molecules of extracted natural dyes could bound to the surface of absorbent TiO_2 thin films which is responsible for the electron movement from the anthocyanins molecules to the conduction band of TiO_2.

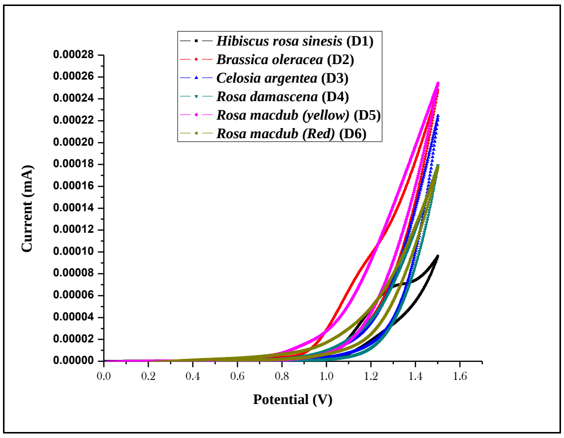

Fig. 2. Cyclic voltammograms of individual dye extracts of all six samples of flowers (D1-D6).

Photovoltaic Performance of Natural Dyes

The DSSCs fabricated with natural extracts were tested for their performance and the current-voltage curves were attained by a digital multimeter (Keithley, 2014). The photocurrent density- photovoltage (*J-V)* profiles of DSSCs fabricated with six natural dyes are shown in Fig.3. The detailed photovoltaic parameters like V_{oc}, J_{sc}, *ff* and η of DSSCs using natural dye extracts are tabulated in Table 2. In order to construct an effective and efficient DSSC, the extracted natural dye must be well-adsorbed on the top of the semiconducting layer. It is evident from Fig. 3 that J_{sc} (short-circuit current density) values of D2 = 4.69 mA/cm² and D1 = 5.48 mA/cm² are higher and in accordance with their UV results followed by D6 = 5.18 mA/cm², D4 = 3.99 mA/cm², D5 = 2.71 mA/cm² and D3 = 1.71 mA/cm².

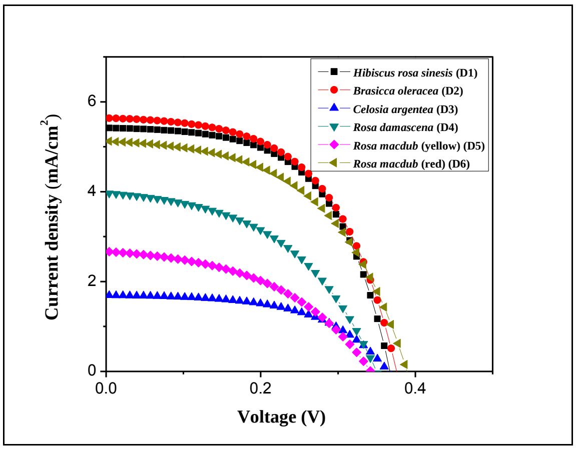

Fig. 3. *J-V* curves of the natural dyes D1-D6.

According to the results presented in Tab. 2. the conversion efficiencies of cells based on natural dyes D1–D6 are 1.02%, 0.9%, 0.2%, 0.5%, 0.3% and 0.8% respectively. The best fill factor value was achieved from the dye D1 = 0.51 with J_{sc} and V_{oc} values of 5.48 mA/cm² and 0.36V respectively with an active area of 1 cm².

Table 2. Photovoltaic parameters and power conversion efficiencies of natural dye extracts of flowers for DSSCs.

Sample Code	Sample Name	J_{sc} (mA/cm²)	V_{oc} (V)	*ff*	Efficiency (%)
D1	*Hibiscus rosa sinensis*	5.48	0.36	0.51	1.02
D2	*Brassica oleracea*	5.69	0.37	0.44	0.9
D3	*Celosia argentea*	1.71	0.36	0.40	0.2
D4	*Rosa damascena*	3.99	0.34	0.35	0.5
D5	*Rosa macdub* (yellow)	2.71	0.34	0.33	0.3
D6	*Rosa macdub* (red)	5.18	0.39	0.42	0.8

The highest sunlight to electricity conversion efficiency (η =1.02%) was obtained from the dye D1 followed by the dye D2 (η = 0.9%). Both these dyes have better UV-absorption, as there ICT band have clear peaks at 534 nm and 532 nm respectively. There short circuit current density is also higher as compared to other dyes (Table 2) which lead them higher overall power conversion efficiency (Safie *et al.*, 2017).

Hence from this study, it is clearly found that natural dyes can be used as sensitizer for the dye-sensitized solar cells. As natural dyes are easily and equally available in all over the world, so there are ample opportunities for the development and reliability on natural dyes for DSSCs.

Conclusion

Natural resources are utilized to extract six different dyes (D1-D6) from locally available flower species. These natural dyes extracts have been used as sensitizers for solar cells. Suitability of these dyes for fabrication of DSSCs was assessed by UV-Vis spectroscopy, cyclic voltammtery and finally through *J-V* curves. DSSCs using thin films of TiO_2 nanoparticles and the natural dyes were assembled. The relation between the absorbance of natural dyes and their conversion efficiency is analyzed and strongly observed. The optical absorption and *J-V* measurement of all dye sensitizers shows interesting values of J_{sc}, V_{oc} and *ff*. Out of all the extracted natural dyes (D1-D6), the D1 dye extracted from *Hibiscus rosa sinesis* presents the highest absorption peak at at ICT band 534 nm, high short circuit current density and best photoelectric conversion efficiency of 1.02%. All other natural extracts also presents considerable results. Natural dyes as light harvesting material for DSSCs are promising future candidate due to environment friendliness, low-cost of production and ease of fabrication.

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