INNSPUB

International Journal of Biosciences | IJB |
ISSN: 2220-6655 (Print), 2222-5234 (Online)
http://www.innspub.net
Vol. 15, No. 5, p. 600-609, 2019

RESEARCH PAPER OPEN ACCESS

Natural dye extracts of wasted fruit peels as photo-sensitizers for TiO_2 based dye-sensitized solar cells (DSSCs)

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Key words: Dye-Sensitized Solar Cells (DSSCs), Titanium Dioxide nanoparticles, Fluorine Doped Tinoxide, Open-Circuit Voltage, Short-Circuit Current Density.

http://dx.doi.org/10.12692/ijb/15.5.600-609 Article published on November 28, 2019

Abstract

Fruits and vegetable peels/skin are considered as inedible and waste material or by-products during juice processing. However, peels are excellent source of natural dye that can be used for various purposes. Six different plant species (ND1-ND6) namely *Beta vulgaris, Lycopersicum esculentum, Prunus domestica, Vitis vinefera, Punica granatum* and *Daucus carota* were selected as a source of natural dye extraction and its application for DSSCs. The ethanolic extraction was made through maceration. The photo-physical, electrochemical and photovoltaic responses of natural dyes were analysed through UV-visible absorption spectrometry, cyclic voltammetry and *J-V* curves respectively. The absorption spectra of dyes indicate two discrete bands *i.e.* $\pi - \pi^*$ and ICT band. The ND4 dye exhibits best absorption of ICT band at λ_{max} 550 nm. Cyclic voltammetric studies revealed discrete oxidation and reduction peaks in ND4 and ND5 dyes while in other dyes these peaks were unclear. The photovoltaic performance of dyes was investigated by fabricating DSSCs with thin film of TiO_2. The open circuit voltages of dyes were 0.45V, 0.31V, 0.34V, 0.38V, 0.34V, 0.33V while the short circuit current density values were 2.93mA/cm^2, 3.38 mA/cm^2, 1.65 mA/cm^2, 5.41 mA/cm^2, 2.53 mA/cm^2 and 2.35mA/cm^2 respectively. The highest light to energy conversion efficiency was of ND4=0.9% with good UV absorption and higher short circuit current density which leads to higher efficiency. Efficiency of other dyes is in the following order ND1=0.7% > ND5=0.5% > ND2=0.42% > ND6=0.4% > ND3=0.3%. Hence, Natural dye from wasted peels can be economical and eco-friendly source of dye for DSSCs.

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Introduction

Energy is considered as an essential necessity for the survival and progress of human civilization (Sthel *et al.*, 2013). There is a close linkage between development of society and its energy consumption through various resources. However, a considerable change between the energy utilization of human ancestors to modern men is reported (Ruotsalainen *et al.*, 2017). Global energy demand is escalating because of rapid growth in human population and industrial sector (Kannan and Vakeesan, 2016). Overland (2016) reported that presently, 1.4 billion people are unable to use electricity and 2.7 billion people across the globe are dependent on conventional biomass to fulfill their energy needs (Richhariya *et al.*, 2017). The widespread use of conventional energy resources *i.e.* fossil fuels because of industrial revolution resulted in severe energy crisis (Mahmood *et al.*, 2014) as well as environmental issues like air pollution which results in a number of severe incidents including photochemical smog, acid rain, ozone depletion, global warming and damage to human health.

Pakistan being a developing and overpopulated country, needs a huge and constant supply of energy to carry out its household and industrial activities (Shakeel *et al.*, 2016). Total energy consumption of the country has augmented in recent decades just like most of the emergent economies and is estimated to follow the same trend. The biggest challenge of today's world is to shift from conventional sources of energy like hydrocarbons towards cleaner, greener as well as renewable and sustainable energy resources to keep environment healthy (Naseem and Khan, 2015). Solar energy is the energy attained through the sun which can be converted into heat and electricity (Aslam *et al.*, 2015). Solar energy is now acknowledged globally and today the solar technology is undergoing an astounding growth. In Pakistan, despite of huge potential and favorable climatic conditions, solar energy technologies are in early stages of development. The two most commonly used solar technologies for the generation of electricity are solar thermal conversion and solar photovoltaic (PV). Shakeel *et al.* (2016) also reported that global capacity of solar photovoltaic amplified by 55% since past five years. Photovoltaic is an advance technique that take benefit from the sunlight (Joshua, 2002 ;Gratzel, 2004). Photovoltaic devices transforms sunlight into electric energy without creating any reported environmental issues (Ludin *et al.*, 2014). Dye sensitized solar cells (DSSCs) are the third generation photovoltaic devices (Kumara *et al.*, 2017). DSSCs have gained popularity because of their cost effectiveness, higher photon-to-current efficiency and flexibility (Hamadanian *et al.*, 2014). The working of DSSCs is based on sensitization of wide band gap semiconductor with the help of a dye sensitizer (Torchani *et al.*, 2015). Metal complexes are widely used dye sensitizers because of their high efficiency and long life but high cost and complicated preparation procedures are their major drawbacks (Prabu *et al.*, 2014).

Since dye plays a vital role in converting sunlight energy into electricity, several kinds of synthetic, organic and metal complexes (Ruthenium) have been studied for their sensitizing properties and light harvesting potential (Dinesh *et al.*, 2019). However, metals like ruthenium and platinum can make DSSCs highly expensive (Pervez *et al.*, 2018). Metal-based dyes show good results but they need very complicated synthesis and purification procedures (Ayalew and Ayele, 2016). At present, metallic dyes are replaced with natural dyes in most growing technologies worldwide due to their versatility and cost effectiveness (Rahul *et al.*, 2018). Another main advantage of using natural dyes is waste reduction and recycling of waste products with minimum consumption of resources like water and energy for dye production (Sadeghi-Kiakhani *et al.*, 2019). Most of the vegetables peels and fruit leftovers after juice extraction are inedible to humans. These parts do contain many chemicals and natural colorants like Pomegranate peels are rich in flavonoids (Marchi *et al.*, 2015). Anthocyanins can be found in many colored plant materials and they contain carboxyl and hydroxyl group for the bonding with photoanode this having advantage over chlorophyll in electron transfer towards TiO_2 surface from the dye molecules with enhanced association (Altaf *et al.*, 2017; Kim *et al.*, 2013). Betalain extracted from beetroot peels can be used as a photosensitizer for DSSCs because of good light absorption range (Bhanushali *et al.*, 2015). The current progression in the field of dye-sensitized solar cells is to study natural colorants with maximum efficiencies along with suitable cost and non-toxic nature (Simoncic and Tomsic, 2010). Carotenoids belongs to family of isoprenoids which are responsible for distinct red to orange and yellow color among many flowers and fruits. They acquire two distinguished role in photosynthesis; first as pigments in light harvesting and second as photo-protector against oxidative damage (Vargas *et al.*, 2000).

The present research work was designed to extract natural dyes from discarded peels of fruits or root bulbs of six different plant species including *Beta vulgaris, Lycopersicum esculentum, Prunus domestica, Vitis vinefera, Punica granatum* and *Daucus carota* for dye-sensitized solar cells application. Their photo-physical, electrochemical and photovoltaic studies are performed.

Materials and methods

Preparation of Natural Dye Sensitizers

Extraction of natural dyes from the plant sources was carried out via the reported procedure by Senthil *et al.*, 2014 with slight modifications. Six different types of locally available plant species namely *Beta vulgaris, Lycopersicum esculentum, Prunus domestica, Vitis vinefera, Punica granatum* and *Daucus carota* were selected. Each sample was assigned with a distinct code from ND1–ND6 respectively. 250g discarded peels of ND1, ND3, ND5 and ND6 were collected from shops and juice corners. In case of ND2 and ND4, fruits were collected from local market and thin peels were separated from rest of the fruit for further processing. Collected peels were washed and cleaned in order to remove dust. Dried and cleaned samples were chopped into pieces and soaked in 1.5L of ethanol and acidified with 2 to 3 drops of conc. nitric acid. Samples were kept in amber coloured glass bottles for 24 hours. The residues were filtered. The collected filtrate was washed with n-hexane many times in a separating funnel to confiscate all fatty acids and chlorophyll from the extract. The ND1 dye was of deep red colour, ND2 and ND3 and ND6 dyes were orange to red in colour ND4 have very bright purple colour while ND5 has brown colour. The extracts were collected and 100mL of each extract was removed through rotary evaporator. The 50mL of each extracted natural dye sample was then stored in reagent glass bottles and kept in the absence of light at a cool place.

Photophysical Characterization of Natural Dyes

The UV-visible spectroscopy was carried out to measure the light absorption properties of natural dyes. The extracted dyes were 10 times diluted with analytical grade 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.

Electrochemical Evaluation of Extracted Dyes

Cyclic voltammetry was carried out to analyze the electrochemical properties of the extracted natural dyes ND1–ND6 by means of the conventional three-electrode method in a compartment with an Eco Chemie Autolab PGSTAT 302 Potentiostat/Galvanostat drived by GPES 4.9 software (Utrecht, The Netherlands) with the help of previously described process by Kavitha *et al.*, 2017 with minor modifications.

The working electrode preferred was Platinum in addition to counter electrode and Ag/AgCl wire was used as the reference electrode. The scan rate was set as 50 mV/s. The electrodes were engrossed in a solution of supporting electrolyte that contain natural dye. All analysis was performed at room temperature. For the supporting electrolyte, 0.1 M Tetrabutylammonium Perchlorate ($TBAClO_4$) solution in distilled water was used. Degassing of all solutions was carried out with argon prior to analysis (Altaf *et al.*, 2017).

TiO_2 based Working Electrode Preparation

Fluorine doped tinoxide (FTO) glass was used as working electrode for the assembling of DSSCs. Initially, the cleaned and dried FTO glass was cut into 1x1.5 dimensions pieces. The glass pieces were washed again using detergent in an ultrasonic bath for 20 minutes and a blocking film was layered on the FTO glass pieces by submerging in 50 mM $TiCl_4$ ethanol solution for 30 min at 70 °C temperature. The FTO glass was coated with nanostructured particles of TiO_2. A suspension of TiO_2 nanoparticles was prepared by incorporating the previously adopted and reported method by Ghani *et al.* (2014). The nanoparticles suspension was prepared by mixing 2 g TiO_2 fine particles, 4 mL deionized water (DI), 0.1 mL Triton X and 0.8 mL of acetylacetone. The blend was kept for continuous stirring in an ultrasonic bath. After 2 hours, the mesoporous film of colloid was equally applied on glass slides by using doctor blade technique and sintered for 450 °C for 30 minutes. After cooling down, each of the TiO_2 anode was engrossed completely in respective concentrated natural dye extract for 24 hours at room temperature.

Assembling and Fabrication of Natural Dye-Sensitized Solar Cells (NDSSCs)

TiO_2 based photoanodes were analyzed against six natural dye extracts in order to assess their light harvesting properties. The two electrodes i.e. photoanode and counter electrode were sandwiched together confronting each other. The two electrodes were joined together by means of binder clips. The drops of Iodine/Tri Iodide (I^-/I^{-3}) electrolyte were injected in the space among photoanode (working electrode) and the graphite based counter electrode. The purpose of electrolyte is to sustain the electrical trail between both electrodes. A solar simulator (SS80AAA by Photo Emission Tech. Inc. USA) was used with solar irradiation power of 100 mW/cm² to inspect the photoelectric conversion efficiency of natural dye based DSSCs. The *J-V* curves were attained with the help of a digital electrometer connected directly to computer. The values of J_{sc} (short circuit current density) and V_{oc} (open circuit voltage) were assessed through the *J-V* curves. The *ff* (fill factor) and η (energy conversion efficiency) of the dye-sensitized solar cell (DSSCs) was calculated for every dye extract by following the 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} = Max. power point current (mA/cm²)

V_{max} = Max. power point voltage (V).

Results and discussion

UV-Visible Absorption Properties of Extracted Natural Dyes

Six natural dye extracts were assessed for their light absorption properties. The UV absorption of the natural dyes extracts was carried out after dilution with ethanol by using UV-Visible spectrophotometer. The UV-Visible absorption spectra of the respective ethanolic dye extracts of fruit peels coded as ND1–ND6 were recorded and values are enlisted in Table 1.

Table 1. Wavelength and max. Light absorption of natural dye extracts within visible region of spectrum.

Sample code	Botanical Name	Part used for dye extraction	Wavelength ICT band (nm)	Absorbance
ND1	*Beta vulgaris*	Peels/skin of root bulb	462 nm	0.717
ND2	*Lycopersicum esculentum*	Peels	532 nm	0.224
ND3	*Prunus domestica*	Peels	462 nm	0.227
ND4	*Vitis vinefera*	Peels	550 nm	0.105
ND5	*Punica granatum*	Peels	532 nm	0.742
ND6	*Daucus carota*	Peels of root bulb	528 nm	0.586

Fig. 1 illustrates the distinctive UV-Visible light absorption spectra for the natural extracts of all the selected dyes. It is apparent from the Fig. 1 that the maximum absorption of all the dye solutions (ND1–ND6) were within the UV and Visible array of electromagnetic spectrum varying between 358 nm to 534 nm. Figure 1 clearly shows that all the extracts have two individual bands ranging from 462-550 nm. Initial one is π-π* band and the second one is referred as Intermolecular Charge Transfer (ICT) band.

Natural extract of *V. vinefera* (ND4) presented the highest absorption 550 nm at ICT band followed by ND2 and ND5 with 532 nm absorption.

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 (%)
ND1	*Beta vulgaris*	2.93	0.45	0.52	0.69
ND2	*Lycopersicum esculentum*	3.38	0.31	0.4	0.42
ND3	*Prunus domestica*	1.65	0.34	0.55	0.31
ND4	*Vitis vinefera*	5.41	0.38	0.43	0.9
ND5	*Punica granatum*	2.53	0.34	0.52	0.45
ND6	*Daucus carota*	2.35	0.33	0.48	0.37

The sequence of absorption observed among all dyes was ND1>ND5>ND6>ND3>ND2 and ND1 correspondingly. The light absorption spectra of two of the extracted dyes namely *L. esculentum* (ND2) and *P. granatum* (ND5) gave absorption peaks at 532 nm. The dye extract of *D. carota* (ND6) peels has an absorption peak at 528 nm. Two of the dye extracts ND1 and ND3 came up with lowest absorption peaks *i.e.* 462 nm. As revealed in Fig. 1.

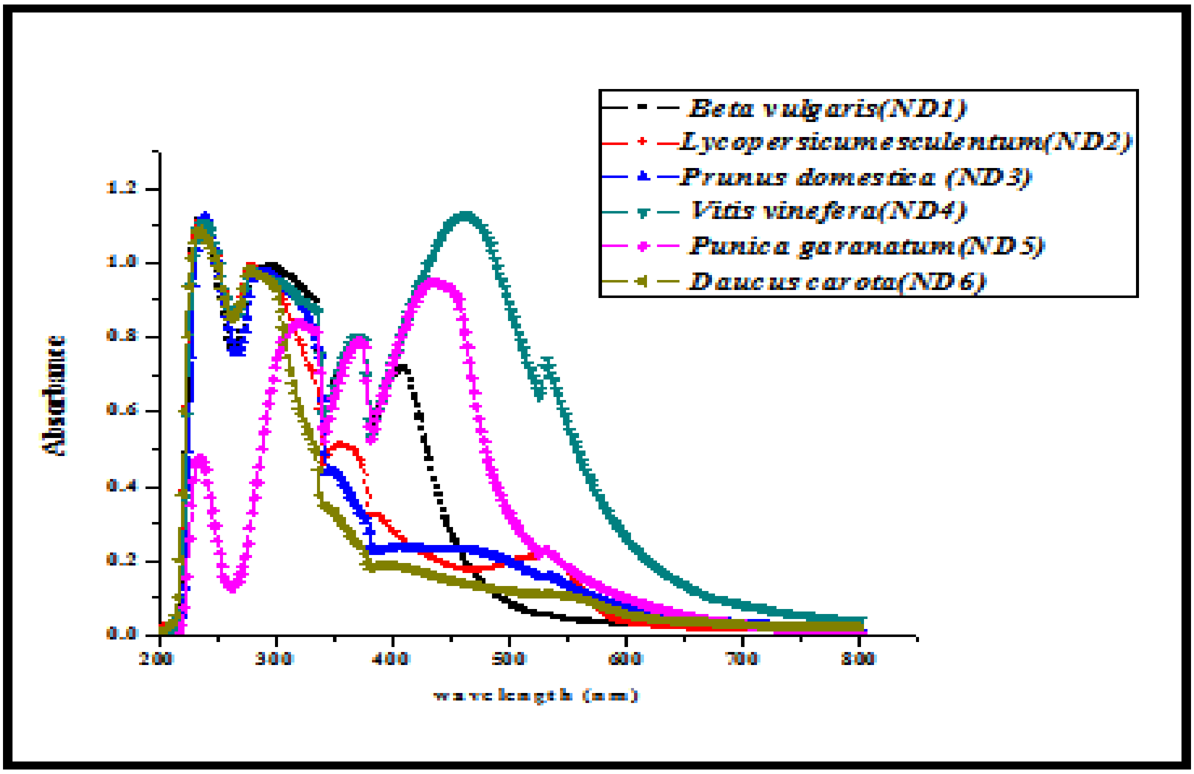

Fig. 1. UV-Vis absorption spectra of all six natural dye sensitizers (ND1-ND6).

ND4 peak is at maximum wavelength as compared to five other samples as they also have analogous absorption trends. The absorption peaks attribute the presence of light absorbing components namely anthocyanins, which are a group of natural phenolic compounds and betalains which are the main component of all natural dye extracts.

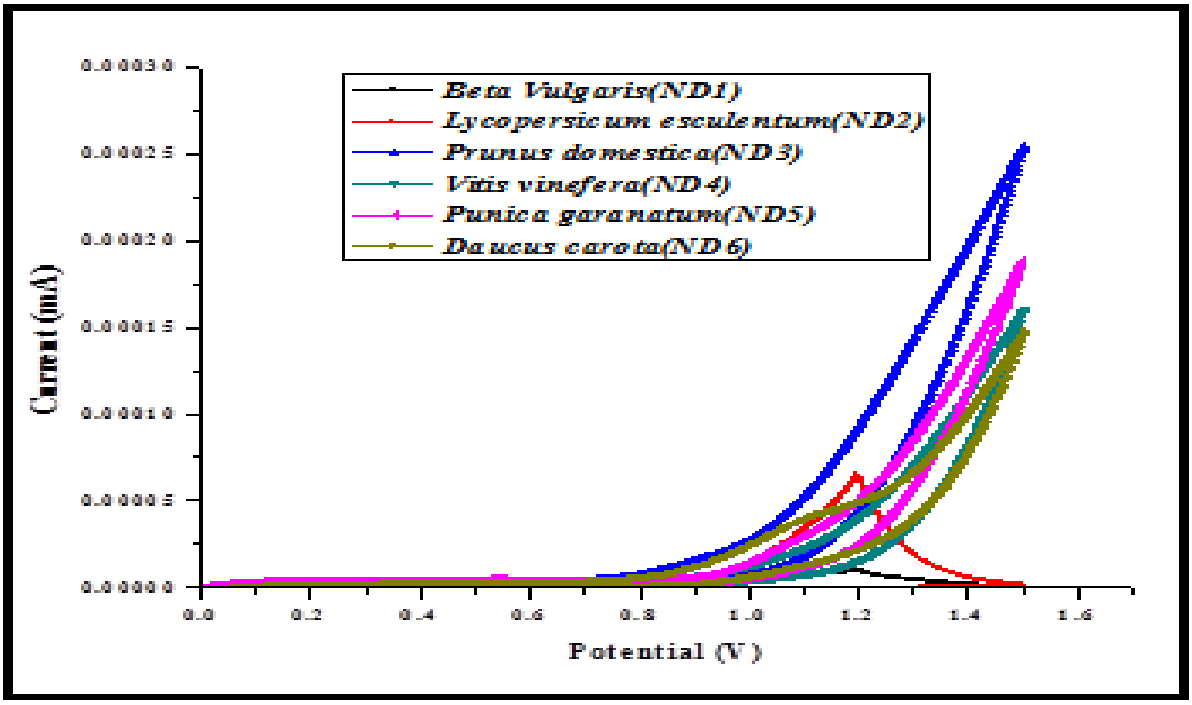

Fig. 2. Cyclic voltammograms of individual dye extracts of all six samples of flowers (ND1-ND6).

Cyclic Voltammetric Measurements of Natural Dye Extracts

The electrochemical characteristics of natural extracts of selected plants were inspected through cyclic voltammetric technique. The obtained cyclic voltammograms of each sample of natural dyes are depicted in Fig. 2. It is evident from the results that clear oxidation peaks are obtained for ND4, ND5 and ND6 dye extracts hence proving them more appropriate DSSC applications. The presence of hydroxyl and carbonyl groups in anthocyanin molecules of extracted natural dyes make them bound to the surface of absorbent TiO_2 thin films which is responsible for the flow of electrons from the anthocyanins molecules to the conduction band of TiO_2.

Photovoltaic Response of Natural Dye Sensitizers

The photo-excitation behaviours of natural dye sensitizer based solar cells were investigated under comparable irradiation setting to check the performance by a digital multimeter/*J-V* curve analyser (Keithley 2400). The current-voltage characteristic curves were prepared.

The current-voltage (*J-V*) profiles of DSSCs fabricated with six natural dyes are given in Fig.3. Table 2 shows the achieved values of photovoltaic parameters including V_{oc}, J_{sc}, *ff* and η of DSSCs using natural dye solutions. Well-adsorbed dye on the surface of the semiconducting layer of nanoparticles can result in efficient and effective DSSC. The results of optical absorption and *J-V* curves of all dye sensitizers came up with interesting values of J_{sc}, V_{oc} and *ff*.

It can be clearly seen in Fig. 3 that J_{sc} (short-circuit current density) values of *V. vinefera* (ND4) = 5.41 mA/cm² and *B. vulgaris* (ND1) = 2.93 mA/cm² are relatively high just like their UV peaks subsequently *L. esculentum* (ND2) = 3.38 mA/cm², *P. garanatum* (ND5) = 2.53 mA/cm², *D. carota* (ND6) = 2.35 mA/cm² and P. *domestica* (ND3) = 1.65 mA/cm².

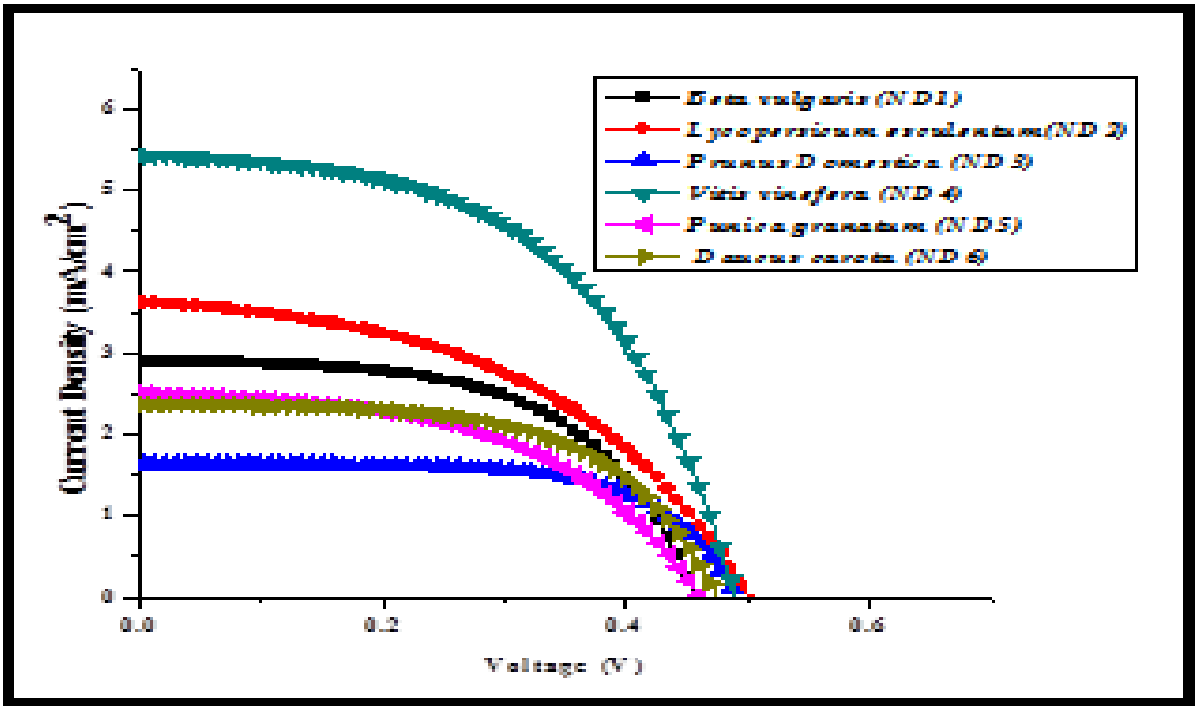

Fig. 3. *J-V* curves of the natural dyes D1-D6.

Table 2 presents the photovoltaic parameters calculated from the obtained results. It is evident that increased short circuit current density J_{sc} is responsible for a better overall conversion efficiencies of cells based on natural dyes.

The calculated values of energy conversion efficiency of the dyes ND1–ND6 are 0.69%, 0.42%, 0.31%, 0.9%, 0.45% and 0.37% respectively. So far, the best incident photon-to-current conversion efficiency value was obtained from the natural extract of *V. vinefera* (ND4) = 0.9% with fill factor 0.43.

The relevant J_{sc} and V_{oc} values are 5.41 mA/cm² and 0.38V respectively with an active area of 1 cm².

The *B. vulgaris* (ND1) dye-based DSSC ranked second for its efficiency value which is η = 0.69%. Both these dyes have better UV-absorption, as there ICT band have clear peaks at 550 nm and 462 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). The relevancy among natural dye absorption and conversion efficiency was determined and clearly observed. Therefore, it is apparent from present research that plant waste materials can be used for natural dye extraction and their application as photosensitizer in fabricating dye-sensitized solar cells which ultimately result in waste minimization and cost effectiveness due to their easy availability all around the world.

Thus, plenty of opportunities are available for the utilization and dependability on natural dyes for DSSCs.

Conclusion

From all six dye extracts (ND1-ND6), the ND4 dye extracted from *V. vinefera* exhibits the high absorption peak at ICT band 550 nm, high short circuit current density and most excellent photoelectric conversion efficiency of 0.9%. Rest of the natural dyes have also shown considerable results.

These dyes can efficiently replace conventionally used expensive and hazardous light absorbing materials. Natural extracts as light harvesting materials for DSSCs are most capable nominee for future towards green economy.

Acknowledgements

This research work was monetarily funded by Higher Education Commission (HEC) Pakistan under Indigenous PhD Scholarship Program (IPFP). The laboratory amenities were provided by Applied Chemistry Research Centre (ACRC) of Pakistan Council of Scientific and Industrial Research (PCSIR), Quaid-e-Azam University, Islamabad and Forman Christian College, Lahore.

The author would like to pay deepest gratitude to Dr. Tahira Aziz Mughal and Dr. Zafar Iqbal for their continual assistance and help throughout the research work.

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