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Performance of Strelitzia reginae Flowers Dye as Sensitizer for New Dye-Sensitized Solar Cell Fabrication

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Abstract

In current study, the dye from flowers petals of *Strelitzia reginae* used for the first time to prepare natural photosensitizer for DSSC fabrication. Among five different solvents used to extract the natural dye from *S. reginae* flowers, the ethanol extract of anthocyanin dye revealed higher absorption spectrum of 0.757a.u. at wavelength of 454nm. A major effect of temperature was studied to increase the extraction yield. The results show that the optimal temperature was 70 °C and there was a sharp decrease of dye concentration from 0.827 at temperature of 70 °C to 0.521 at temperature of 90°C. The extract solution of flowers of *S. reginae* showed higher concentration in acidic media, especially at pH 4 (0.902). The C, H, N elemental analyses of natural extract showed the presence of N (10.52%), C (18.97%) and H (2.229%) contents. The presence of anthocyanin in the extract of *S. reginae* and their functional groups were determined using UV-Vis spectroscopy and Fourier-Transform Infrared Spectroscopy (FTIR). The optical properties of deposited TiO_2 thin film were studied using scanning electron microscope (SEM) and X-Ray Diffraction (XRD). In Addition, other confirmation that dye supported surface of TiO_2, inhibition of crystallinity of TiO_2 was investigated by the Energy Disperse X-ray (EDX) analysis. According to experimental results, the conversion efficiency of DSSC fabricated using anthocyanin dye obtained from *S. reginae* flowers was 0.1%, with short-circuit current (*Isc*) of 0.6 mA/cm^{-2}, open-circuit voltage (*Voc*) of 0.51 V and fill factor (*FF*) of 36.83%.

Keywords: DSSC, *Strelitzia reginae*, Natural dye, Anthocyanin, energy conversion efficiency.

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1. Introduction

Photovoltaic cells (PVC) development at the last two decades led to develop the devices that designed to expand the applications of PVC and reduce their production cost to compete with the systems of energy production [1]. Dye sensitized solar cells (DSSCs) are the devices that convert sunlight into electricity, based on the dyes work as photons harvesting and the wide bandgap semiconductors as substrate [2]. The use of dye sensitization remained limited and rather unsuccessful until 1991, when Grätzel and his coworkers developed a solar cell by the successful combination of nanostructured electrodes; efficient charge injection dyes. Hence termed this cell was dye and sensitized nanostructured solar cell [3, 4]. DSSCs have three primary parts, semiconductor film electrode (anode), electrolyte which typically coated with Platinum (Pt) catalyst and counter electrodes (cathode) [5]. The power efficiency of DSSC mainly depends on the dye (Natural or synthesis) used as sensitizer, which plays a key role in absorbing sunlight and converting photons into electrons [6]. Two important factors determine the performance of DSSC (i) the dye absorption spectrum (ii) the dye anchorage onto the surface of TiO_2 [5]. Several organic dyes and inorganic metal complexes have been synthesized and used as sensitizers such as platinum complexes, ruthenium polypyridyl complex phthalocyanine, fluorescent dyes among others [7].

When compared to other types of manufactured solar cells, the use of an organic dye as a sensitizer is unique to (DSSCs). Synthesis complexes dyes contain heavy metals that are expensive to synthesis, harmful to the environment and limited in their resources. Natural dyes are less efficient but have the advantages of being the most environmentally friendly, freely available because their sources are renewable, non-toxic, can be applied without further purification and have a low cost of production. As the light harvester in the DSSC, the natural sensitizer should have an absorption spectrum that covers a broad region from the visible light spectrum and should also have anchoring groups such as hydroxyl or carboxyl groups for binding with the TiO_2 film [8].

Natural dyes or pigments extracted from flowers, leaves, fruits, and seeds using simple procedures can be used as sensitizers in DSSCs; these dyes include anthocyanins [9], chlorophyll [10], Carotenoids [11], betalains [12]. Molecule of anthocyanin contains hydroxyl and carbonyl groups that are bound to the TiO_2 surface, which excites electron transfer from the dye to the conduction band of theporousTiO_2 film [10, 13]. Anthocyanin dye that extracted from different parts of plants provides different sensitizing conversion efficiency. Fernando & Senadeera extracted pigments of anthocyanin from tropical flowers such as *Hibiscus surattensis, Nerium oleander, Sesbania grandiflora* and *Rhododendron arboretum,* and studied their activity as sensitizers for DSSCs. The efficiencies of these fabricated DSSCs varied from 0.2% to 1.1%. [14]. Chang & Lo (2010) were used the extracted anthocyanin dyes from mulberry fruit as a natural sensitizer for DSSCs. Their experimental results showed that the power conversion efficiency of the fabricated DSSC was 0.548% [15].

In this study, the natural dye was extracted from a flower of *S. reginae* to prepare natural sensitizer for DSSC fabrication. The effect of solvent, temperature and pH on natural extract yield was investigated. The presence of anthocyanin in the extract of *S. reginae* and their functional groups were determined using UV-Vis absorption spectroscopy and Fourier-Transform Infrared Spectroscopy (FTIR). The optical properties of the deposited TiO_2 thin film were studied by scanning electron microscope (SEM) and X-Ray Diffraction (XRD). In addition, other confirmation that dye supported surface of TiO_2, inhibition of crystallinity of TiO_2 was investigated by the Energy Disperse X-ray (EDX) analysis. In order to test the anthocyanin dye as a photosensitizer, DSSC was designed using flowers of *S. reginae* extract.

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The effective light exposure window of the DSSC was 1cm^2. The main parameters of the solar cell are short circuit current (I_{SC}), open circuit voltage (V_{OC}), fill factor (*FF*), and efficiency (η) and maximum power point (P_{max}) were determined.

2. Materials and Experiments

2.1. Materials

In this study, flowers of *S. reginae* were collected from Universiti Kebangsaan Malaysia (UKM). The petals of flowers were the part of the plant used to extract anthocyanin dye. The scientific classification of *S. reginae* is: Kingdom: Plantae; Order: Zingiberales; Family: Strelitziaceae; Genus: *Strelitzia*; Species: *reginae.*

2.2. Experimental

2.2.1. Preparation of sensitizer using natural dye

The petals of *S. reginae* flowers were washed using distilled water dried at 40 ^oC for six hours. The dried petals were crushed into fine powder using grander (Mulry function disintegrator SY-04). The obtained powder used to prepare dye solution which was used to prepare a natural sensitizer for DSSC fabrication.

2.2.2 Fabrication of dye-sensitized TiO_2 photoanode

The TiO_2 paste was prepared according to the methodology described in our previous study [16]. To prepare a TiO_2 paste, 2.0 g of TiO_2 powder (Titanium Oxide Nanopowder, TiO_2, anatase, 99.5%, 15nm, supplied from US Research Nanomaterials, Inc.) was grinded with 5.0 ml of 0.1 M nitric acid using a mortar and pestle for 30 minutes. 3 ml of polyethylene glycol (PEG) (PEG, MW 10,000, supplied SIGMA-ALDRICH) was added. Five drops of Triton X-100 (Triton X-100 for electrophoresis, supplied from SIGMA- ALDRICH) were added to the mixture to ease adhesion of TiO_2 particles to a conductive surface of glass. The conductive glass coated with Fluorine doped Tin Oxide (FTO) was used as a basis for the photoelectrode. The transparent tape (Scotch, MagicTM Tape) was used to tape the conductive glass on the table and make sure that the conductive side was facing up using the multimeter probes (SANWA, YX360TRF). A TiO_2 paste was quickly spread by doctor blade technique as evenly as possible into an area of 1 cm^2 using a clean glass rod.

2.2.3 Preparation of electrolyte

The electrolyte solution of iodide/triiodide containing 0.4 M lithium iodide, 0.4 M tetrabutylammonium iodide and 0.04 M iodine (I^2) was dissolved in 0.3 M N-methylbenzimidazole in a solvent mixture of acetonitrile and 3-methoxypropionitrile with a volume ratio of 1:1. I^-/I^{3-} is a common electrolyte in an acetonitrile (organic solvents) which was used in this study. To facilitate electron transport, Lithium ion was added. This electrolyte is suitable for ion diffusion and infiltrates well into the TiO_2 film, exhibiting the highest efficiency among all DSSCs.

2.2.4 DSSC assembly

Prepared electrode was placed facing upward, and the conductive side of the counter electrode faced the TiO_2 film and sealed by gum. A DSSC was assembled by introducing liquid electrolyte into the space between the photo anode (electrode) and the counter electrode (cathode) by capillary action.

2.2.5 Characterization and measurements

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The light absorption spectrum peaks and functional groups of anthocyanin extracted from *S. reginae* flowers were assigned using UV-Vis spectroscopy (Perkin Elmer, Lambda 35) and FTIR spectra (Thermo scientific NICOLET 6700). The solar energies to electricity conversion efficiencies were measured under simulated solar light (AM 1.5,100 mW/cm^2). Based on the photo current-voltage (I–V) curve, the fill factor (FF) can be determined using the formula:

$FF= (I_{max} \times V_{max}) / (I_{sc} \times V_{oc})$

Where I_{max} and V_{max} are the photocurrent and photovoltage for P_{max} (maximum power output), I_{sc} is the short-circuit photocurrent and V_{oc} is the open-circuit photovoltage. The overall solar conversion efficiency (η) of a DSSC is defined as:
$\eta = (I_{sc} \times V_{oc}) \times FF / P_{in}$

Where P_{in} is the input power.

3. Results and Discussion

3.1 Experimental parameters

3.1.1 Absorption of natural dyes

Figure 1 shows UV-Vis absorption spectrum of the natural dye extracted. A broad main absorption peak of dye extracted from *S. reginae* in the visible region of the spectrum was found at a wavelength between around 350 and 520 nm. The absorption spectrum for the flowers extract of *S. reginae* agrees with that of anthocyanin [11, 17]. *S. reginae* flower contains abundant anthocyanin [18, 19]. The photographic image of *S. reginae* is shown as an inset in Figure 1.

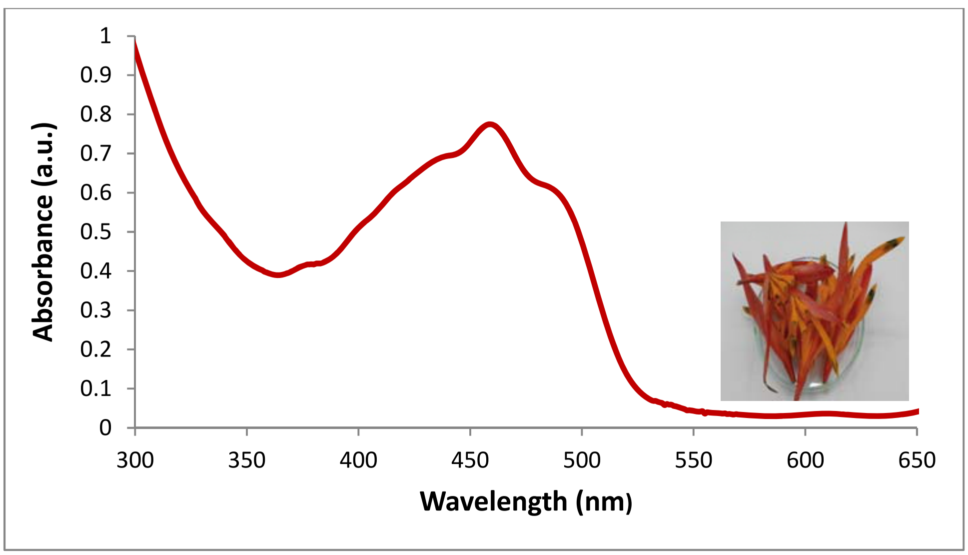

Figure (1): UV-Vis absorption spectrum of dye obtained from *S. reginae*. The inset shows the photographic image of the flowers petals

3.1.2 Structure of extracted dye

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Identification of the functional group for the chemical active compound of *S. reginae* extract was measured using FTIR spectrum based on the peak values in the region of infrared radiation. The FTIR spectrum of natural dye is shown in Figure 2. The OH group appears as the broadest peak at wavelength between 3000 cm^{-1} to 3700 cm^{-1}, which could be attributed to extraction solvent (water or ethanol). The region of the spectrum between 1500-2000cm^{-1} allows infrared absorption of C=C. Moreover, the C=O group at 1634 cm^{-1} and the peak at 1404 cm^{-1} corresponds to alkanes contained H-C-H group with slightly difference in the intensity of transmittance. The C-O groups are positioned at 1126 cm^{-1} and 1066 cm^{-1} indicating high amounts of dye. The aldehydes were found in wavelength between 900 cm^{-1} to 600 cm^{-1} emerging from the base of the natural organic dye [20-22].

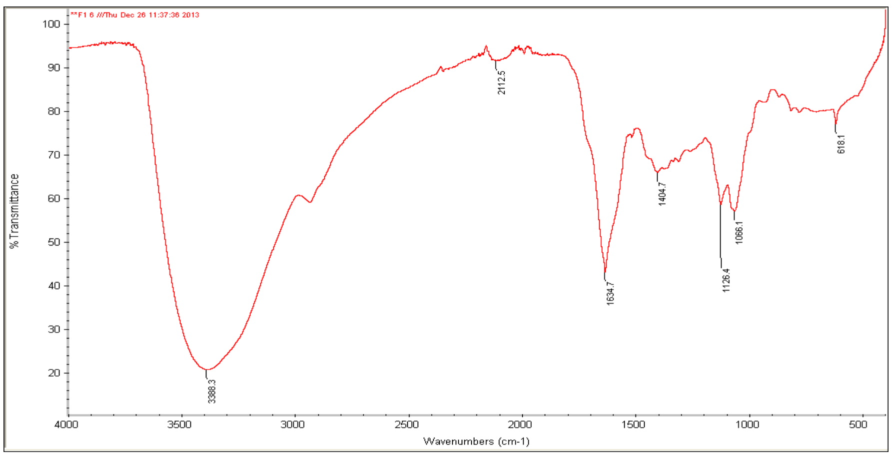

Figure (2): FTIR spectrum of dye obtained from *S. reginae* flowers

3.1.3 Effect of solvent on dye extraction

Anthocyanin was extracted from flowers petals of *S. reginae* using five different solvents: ethanol, acetonitrile, methanol, chloroform and n-Hexane at room temperature in dark condition for 24 hours. Figure 3 shows the effect of different solvent types on the extraction of natural dye from *S. reginae* flowers. Among all dyes extracts, the ethanol extract exhibited the highest absorbance of dye of 0.974a.u. followed by acetonitrile of 0.825a.u. and methanol of 0.809a.u. This result may be due to the fact that natural organic dyes were often extracted in higher concentrations using polar organic solvents such as ethanol [23]. Current result was in agreement with previous investigation of Chatha et al. (2006) who mentioned that higher extract yield from plant species of rice was obtained using high polar organic solvents. In general, dyes extraction using various solvents offered the highest absorption level with ethanol (95%) [24]. Ethanol has been extensively used to extract natural dyes from different organs of various plants [25].

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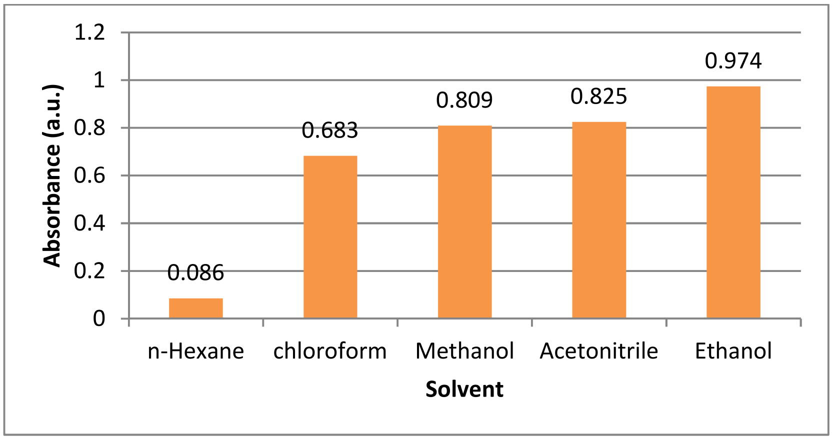

Figure (3): Effect of five different solvents on the extract absorbance of *S. reginae*

3.1.4 Effect of Temperature on dye extraction

To determine the maximum concentration of the extracted dye, the effect of temperature on the extraction of dye from flowers of *S. reginae* was studied. The effect of four different temperatures 30, 50, 70, and 90 ^{o}C on dye extraction was investigated (Figure 4). Aqueous solution of optimal solvent (ethanol:water, 70:30 v/v) used. It can be seen in figure 4, there was an increase in concentration of petal extract of *S. reginae* flowers which was stable at temperature of 30, 50, and 70 ^{o}C. An increase of dye concentration maybe caused by an increase of dye molecules diffusivity and dye solubility that related to increase of the internal energy of molecules which lead to increasing of the extract concentration [26]. A major effect of the increase of temperature was to increase the extraction yield [27]. The results show that the optimal temperature was 70 °C and there was a sharp decrease of dye concentration from 0.827 at temperature of 70 °C to 0.521 at temperature of 90 °C. Due to the high susceptibility of dyes to high temperature, the increase of temperature leads to a decrease in dyes yield. Temperature above 70 °C probably caused a decrease in the extract concentration due to degradation of chemical structure of dye. Durling *et al.* (2007) reported that an increase in extraction temperature leads to increase of extraction yield. They mentioned also, that the dye solution concentration was decreased because of extracting more inactive compounds at the higher temperature (90°C) [28]. However, further temperature increases to 90°C resulted in lower dye yield [29].

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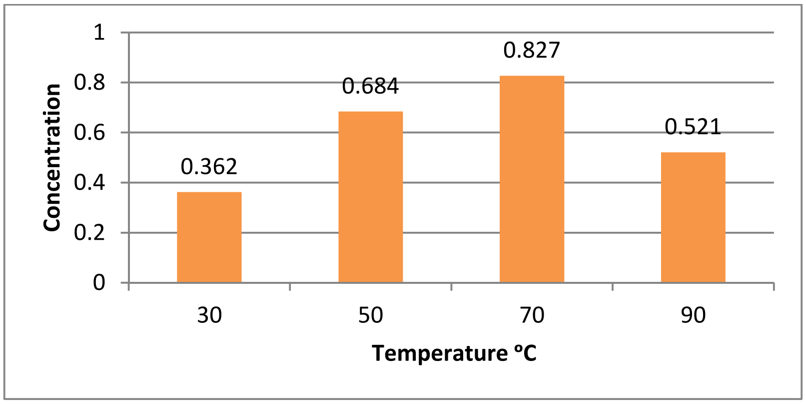

Figure (4): Effect of four different temperatures on the extract concentration of *S. reginae*

3.1.5 Effect of pH on dye extraction

The mixture solution of ethanol (ethanol: water 70:30 v/v) at temperature of 70 °C for flowers of *S. reginae* under pH graduate values from 4, 6, 8 to 10 for time of 30 min were used to select the optimal pH value to obtain higher concentration of a dye. The extract solution of flowers of *S. reginae* showed higher concentration at acidic media, especially at pH 4 (0.902). It was also found that the dyes concentration gradually increases from 0.328 at pH of 10 to 0.902 at pH of 4. To find out the reason behind this phenomenon, the C, H, N elemental analyses for extracted dye have been done. Elemental analyses of natural extract showed the presence of N (10.52%), C (18.97%) and H (2.229%) contents, the alkalinity and the solubility of these nitrogenous compounds will increase in acidic media (low pH), and this will increase the concentration of natural extracts. Devi *et al.* (2012) reported that the pH had the great effect on the stability of extracted dye [30].

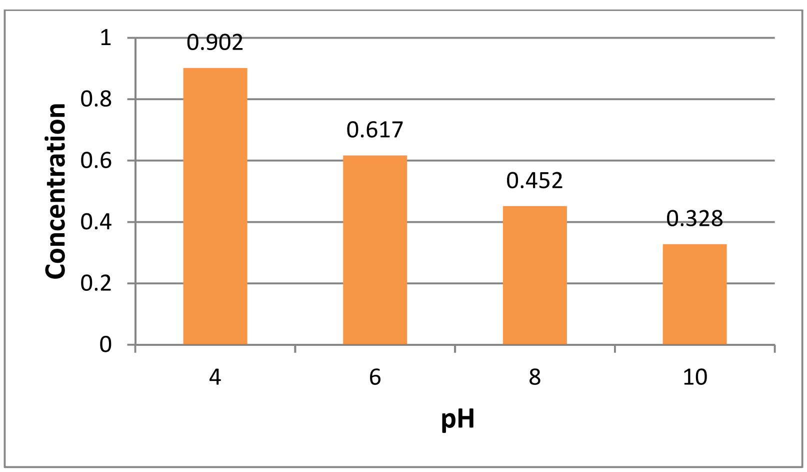

Figure (5): Effect of four different pHs on the extract concentration of *S. reginae*

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3.2 Optical and morphological properties of extracted dye and sensitizer

3.2.1 Surface morphology of TiO_2

Figure 6a shows the SEM image of pure TiO_2. This Figure revealed that TiO_2 particles aggregated to form nanoclusters and we can also observe that the TiO_2 matrix is porous and has agglomeration. Figure 6b shows the SEM image of natural dye extracted from flowers of *S. reginae* after adsorbed onto the TiO_2 surface. This figure exhibited that the aggregation of TiO_2 particles increased. It was also shown a mesoporous surface of the spherical TiO_2 nanoparticles that form nano-pores across the surface and the spherical shape of the particles slightly changed because of the adsorption of anthocyanin dye on the surface of TiO_2. These results were in agreement with previous studies of Ananth et al. (2014) [31] and Noor et al. (2011) [19].

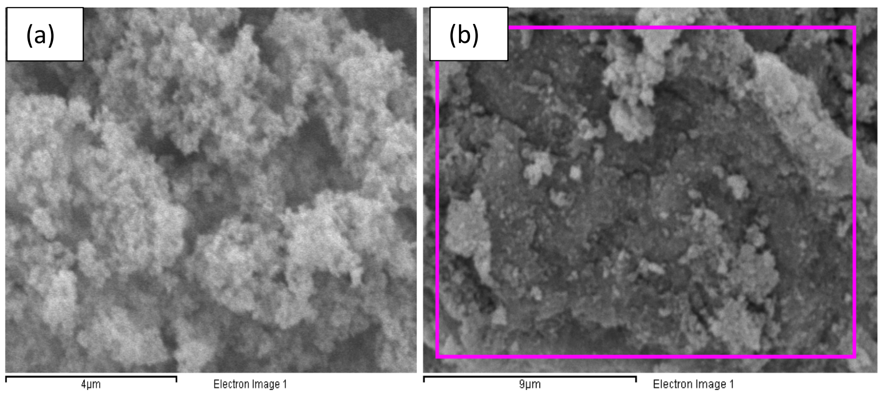

Figure (6): SEM images of (a) pure TiO_2 and (b) TiO_2– *S. reginae*

3.2.2 Energy Dispersive X-ray (EDX) Analysis

Figure 7a shows the EDX spectrum of TiO_2 particles. The EDX analysis of the TiO_2 shows the presence of titanium and oxygen. The spectrum of titanium (Ti) and oxygen (O) exhibit distinguished peaks. The EDX data showed a peak at 0.2keV and a sharp peak at 4.2keV. Another intense peak appears at 4.5keV. As shown in Table 1, the weight of Ti and O are 57.24% and 42.76%, respectively, which confirms the high purity of TiO_2 particles that will be used as photo materials with the natural dye. Figure 7b show the EDX spectrum of TiO_2 after adsorbed a dye of *S. reginae* flowers. In addition to the peaks of Ti and O, Figure 7b showed another prominent peak of the functional group of the dye. As shown in Table 1 and Figure 7b, it was clearly seen that the presence of carbon (C) from dye of *S. reginae* at the weight of 39.93%. This result indicates the presence of the functional groups of the natural dye which are important to adsorb the dye on the TiO_2 surface. This adsorption is necessary to transfer the electrons from dye to TiO_2 [4, 32].

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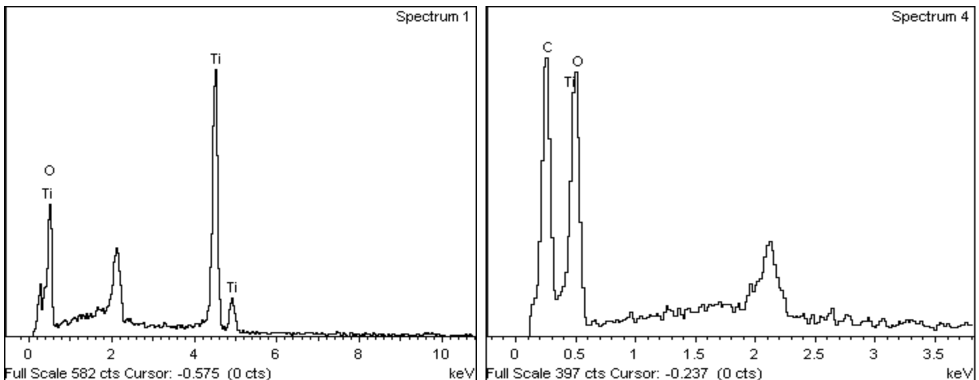
Table (1): EDX data of pure and dyed TiO_2 with extract of *S. reginae* flower

Pure TiO_2			**TiO_2/ *S. reginae***		
Element	**Wt.%**	**At. %**	**Element**	**Wt.%**	**At. %**
Ti	57.24	30.89	**Ti**	16.42	5.36
O	42.76	69.11	**O**	43.66	42.66
			C	39.93	51.98



(a) (b)

Figure (7): EDX spectra of (a) pure TiO_2 and (b) TiO_2– *S. reginae*

3.2.3 X-Ray Diffraction (XRD) analysis

Figure 8a shows the X-ray diffraction pattern of the TiO_2 nanoparticles. The well resolved diffraction peaks centered at the 2θ values of: 25.29°, 37.77°, 48.05°, 53.84°, 55.05°, 62.63°, 67.88°, 70.16°, 75.03° and 75.17°. The experimental XRD pattern agrees with the JCPDS card no. 21-1272 (anatase TiO_2) of the XRD pattern of TiO_2 nanoparticles and other literature [33]. The inhibition of TiO_2 crystallinity after adsorbing an anthocyanin dye of *S. reginae* flowers was investigated by X-ray analysis (Figure 8b). This finding confirms that the *S. reginae* dye was adsorbed on the surface of TiO_2 film.

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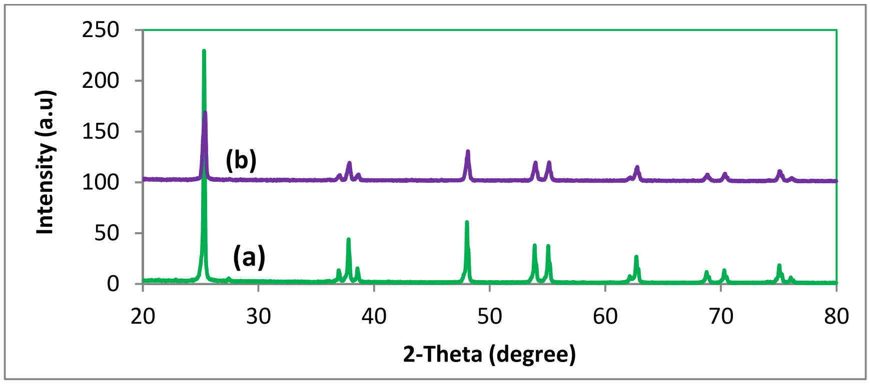

Figure (8): XRD of (a) pure TiO_2 and (b) TiO_2– *S. reginae*

3.3 Performance of DSSC sensitized with the extracted natural dye

To test the activity of extracted natural dye as sensitizer for DSSC, the cell was fabricated using anthocyanin dye extracted from *S. reginae* flowers as photosensitizer. The corresponding current-voltage (*I-V*) curve of the DSSC of extracted dye under AM 1.5 solar illumination was shown in Figure 9. The performance of anthocyanin dye as sensitizer for DSSC was evaluated by V_{oc}, I_{sc}, *FF* and energy conversion efficiency (η). The photoelectrochemical parameters of the DSSC sensitized with flower extract are listed in Table 2. FTIR spectrum of anthocyanin dye from *S. reginae* flowers show the presence of OH group appeared at wavelength between 3000 cm^{-1} to 3700 cm^{-1} and the sharp peak at wavelength of 1634 cm^{-1} which attributed to the presence of C=O group (Figure 2). Furthermore, the general chemical structure of an anthocyanin molecule contains carbonyl and hydroxyl groups which bound to the surface of the TiO_2 semiconductor to allow electron transfer from the sensitizer (anthocyanin dye molecules) to the conduction band of the porous TiO_2 film [34, 16].

In this study, the DSSC based on anthocyanin dye exhibited the conversion efficiency of 0.1% with *Voc* of 0.51 V, *Isc* of 0.6 mA/cm^{-2} and *FF* of 36.83% (Table 2). The DSSC sensitized by an extract of *S. reginae* mainly composed of anthocyanin did not offer high photo to electric conversion efficiency in DSSC (0.1%). This is because of low available bonds between the dye and TiO_2 molecules which caused low interaction between dye from flowers and TiO_2 molecules which led to deficiency of the electrons transfer from excited dye molecules of flowers extract to TiO_2 thin film [35]. These results were in agreement with Minicante et al. (2016) [36]. Calogero et al. (2008) found that the conversion efficiency of DSSC fabricated with anthocyanin extracted from red Sicilian orange juice higher than of that extracted from strawberry or blueberry juice [37]. Kimpa et al. (2012) reported that the photoelectric conversion efficiency of DSSC fabricated with anthocyanin dye extracted from Flam tree flower was 0.2% [38].

Table (2): Photoelectrochemical parameters of the DSSC using *S. reginae* extract

Dye	*Voc* (v)	*Isc* (mA)	*Jsc* (mA/cm^2)	*Vm* (v)	*Im* (mA)	*Pmax* (W)	*η* %	*FF* %
S. reginae	0.51	0.6	0.6	0.295	0.4	0.1	0.1	36.83

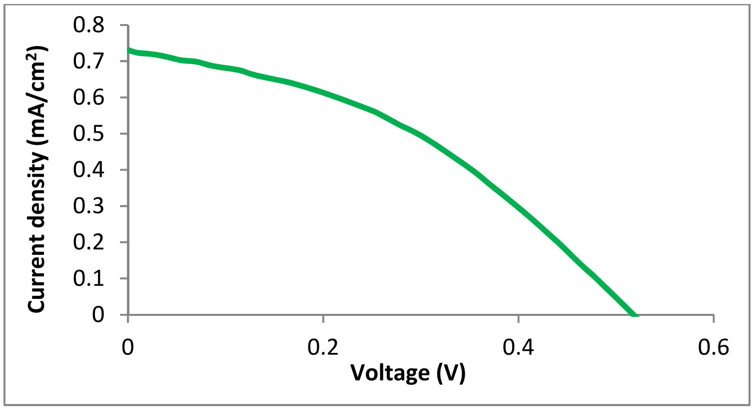

Figure (9): Photocurrent–voltage curve for DSSC sensitized with *S. reginae* extract

4. Conclusion

In this work, the performance of DSSC was evaluated. This DSSC was sensitized for the first time using anthocyanin dye extracted from flowers of *Strelitzia reginae*. The higher energy conversion efficiency (*η*) of 0.1%, fill factor (*FF*) of 36.83%, open-circuit voltage (*Voc*) of 0.51V and short-circuit current (*Isc*) of 0.6 mA/cm^{-2}, were obtained from fabricated DSSC. The effect of solvent, temperature and pH on natural extract yield was investigated. The optimum conditions of natural dye extraction were achieved using ethanol as the polar organic solvent at temperature of 70 ^{o}C which indicates the stability of dye at relatively high temperature and pH of 4. From the results of the current study we found there was a sharp decrease of dye concentration from 0.827 at temperature of 70 °C to 0.521 at temperature of 90 °C. The presence of natural dyes and their functional groups was determined using UV-Vis absorption spectroscopy and FTIR. Results of C, H, N elemental analysis indicated the presence of N which lep to increase of the extracted dye concentration in acidic media gradually from 0.328 at pH 10 to 0.902 at pH 4. Correlation between carbon contents and sensitizer performance was indicated by SEM, EDX and XRD studies.

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