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ZnO Nanorods Based Dye Sensitized Solar Cells Sensitized using Natural Dyes Extracted from Beetroot, Rose and Strawberry

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Dye sensitized solar cells were fabricated using natural dyes extracted from beetroot, rose and strawberry. The ZnO nanorod working electrode has been prepared by simple hydrothermal method. The crystallinity and morphology of the prepared electrode has been studied using X-ray diffraction and scanning electron microscopy techniques. The effect of natural dye extract temperature, pH of the dye and the solvent used for dye preparation on the solar cell characteristics have been studied. The efficiency of strawberry extract sensitized ZnO nanorod solar cells are found to be better than the other solar cells sensitized using beetroot and rose extracts.

Key Words : Strawberry, Beetroot, Nanorods, Natural dyes, Dye sensitized solar cells

Introduction

In recent years dye-sensitized solar cells have attracted much attention because of their advantages like low cost, less toxic nature, transparency and flexibility.[1] The dye-sensitized solar cell consists of an electrode with wide band gap semiconductor such as TiO_2 or ZnO sensitized using a suitable dye, a redox electrolyte and a counter electrode. When the light is incident on the working electrode the incident light is absorbed by the dye anchored to the semiconductor, the resulting photoelectron being transferred from the excited level of the dye into the conduction band of semiconductor, and through the electrode into the external circuit. The electrolyte facilitates the transport of the electron and the regeneration of the sensitizer, through reduction of the tri-iodide ion at the counter electrode, followed by oxidation of the iodide ion at the dye.[2] The electron transport and power conversion efficiency of the device depends upon various factors among them surface morphology of the working electrode and the dye used are important.[3-5] Compared with nanocrystalline semiconductors, hierarchical patterns like nanorod and flower like structure based dye-sensitized solar cells represent a promising alternate for the large scale conversion of solar energy into electricity.[6]

These hierarchical patterns not only exhibit high surface area it also provides better light scattering properties which promote dye loading and better light absorption. All the available titanium precursors are highly reactive with water so preparing nanorod like structure with simple equipment is more difficult, whereas the morphology of ZnO nanostructures can be easily controlled by simple chemical method.[7-9] The chemical methods are not only simple and low-cost, they also provide a large-scale route for growing ZnO nanostructures directly on the substrate.

In a dye sensitized solar cell, ruthenium based complex sensitized solar cells have exhibited maximum efficiency of 12% and they are the preferred materials for dye sensitization.[10] However, ruthenium based synthetic dyes are relatively expensive for large-scale applications in solar cells and also involve complex synthetic procedures.[11] In order to replace the expensive ruthenium compounds, many kinds of natural dyes have been investigated and tested as low-cost materials and environmentally friendly alternatives to artificial sensitizers for dye sensitized solar cells.[12-13] The synthetic dyes are not only expensive, also the synthesis procedure is difficult and complicated. On the other hand, natural dyes extracted from some fruits, flowers and leaves show various colors and it contains several pigments that can be easily extracted.[14] Carbonyl and hydroxyl groups present in the natural dye can be easily bound to the surface of the ZnO nanorods. This makes electron transfer from the dye molecule to the conduction band of ZnO a very easy process.

In recent years many natural dyes have been tried with a view to improve the efficiency of natural dye sensitized solar cells. Park *et al*., 2013; have fabricated gardenia yellow as natural photosensitizer and reported maximum power conversion efficiency of 0.35%.[15] Gomez-Ortiz *et al*., 2010; have prepared achiote seed extract sensitized ZnO nanostructured dye sensitized solar cells and reported the maximum power conversion efficiency of 0.01%.[16] Thambidurai *et al*., 2011; have fabricated Ixora coccinea, Mulberry and Beetroot extract sensitized ZnO nanorod based solar cells and have reported that the efficiencies are 0.33, 0.41 and 0.28% respectively.[17] However, a systematic study on the influence of dye extracting temperature, pH of the extract solution and the solvent used for dye extract preparation on the performance of solar cells have so far been limited. So in this work, ZnO nanorod like structures have been prepared by simple sol-gel hydrothermal method and used as a working electrode for dye sensitized solar cells. Natural dyes extracted from beetroot, rose and strawberry have been used to sensitize the working electrode and solar cells have been fabricated and the characteristics of these dye sensitized solar cells have been studied.

Experimental

Preparation of Working Electrode. In our experiment, indium doped tinoxide (ITO) glass substrate was used as substrate. All the chemicals used in the experiment were purchased from Aldrich. Initially, the ITO glass substrates were ultrasonically soaked in acetone and ethanol, and then dried at 100 °C in an oven. To synthesize ZnO nanorods, two step chemical method has been used. In the first step, ZnO seed layer has been prepared by simple sol-gel method. The detail for the preparation of seed layer is as described in one of our earlier works.[18] To prepare ZnO seed layer, 0.3 M of zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) was dissolved in a mixture of 10 mL ethanol and 0.25 mL water. ITO coated glass substrates were dipped in the prepared solution and this resulted in the formation of seed layer, these seed layer films are annealed at 300 °C for 30 minutes. In the second step, ZnO nanorods have been prepared by hydrothermal method. To prepare ZnO nanorods, an aqueous precursor solution was prepared by dissolving 0.02 M zinc nitrate ($Zn(NO_3)_2{\cdot}6H_2O$) and 0.2M hexamethylenetetramine ($(CH_2)_6N_4$) in 20 mL deionized water. The solution was transformed into teflon stainless steel autoclave and the seed layer coated substrate was vertically dipped in the aqueous solution and it was maintained at a bath temperature of 85 °C for 4 hrs. After the growth period, the substrates were removed from the solution and were thoroughly washed with deionized water to remove the residual salt from the surface of the film. Now the prepared film was annealed at 450 °C for 30 minutes and this resulted in the formation of ZnO nanorods.

Preparation of Natural Dye Sensitizer. For rose extract preparation fresh rose flower petals were put into 100 mL of ethanol. Solid dregs in the solution were filtered by filter paper to obtain a pure natural dye solution. For beetroot and strawberry extract preparation, the cleaned vegetable and fruits were cut into small pieces and put into two different beakers. Chopped vegetable and fruits were soaked in 200 ml of ethanol at different temperatures. Then the residual parts were removed by filtration and the filtrate was washed with hexane several times to remove any oil or chlorophyll present in the extract. This was directly used as dye solution for sensitizing ZnO nanorod electrodes. The strawberry extract was extracted from the ethanol solvent at different temperatures such as room temperature, 50 °C, 75 °C and 100 °C. To study the effect of pH on the performance of solar cell, the pH of the strawberry extract solution was changed by adding dilute HCl and dye solution with three different pH values 1.0, 2.0 and 3.0 have been used as sensitizer. To study the effect of extracting solvent on the performance of solar cell, the strawberry extract was also extracted by using methanol. By using methanol the natural dye was extracted at a temperature of 75 °C.

Assembling the Solar Cell. To assemble the natural dye sensitized ZnO nanorod based solar cell, the prepared ZnO nanorod electrodes were immersed in the synthesized dye solution at room temperature for 24 h, after that period the film was rinsed in anhydrous ethanol and then dried. A Pt-coated ITO electrode was then placed over the dye-adsorbed ZnO nanorod electrode, and the edges of the cell were sealed with a sealing sheet (PECHM-1, Mitsui-Dupont Polychemical) by heating with hot plate at 100 °C for 2 minutes. A redox electrolyte was prepared using 0.5 moL KI, 0.05 mol I_2, and 0.5 mol 4-*tert*-butylpyridine and a drop of electrolyte solution was injected into the drilled hole in the counter electrode and was driven into the cell. Finally, the hole was sealed using additional cello tape and the size of the electrodes used was 0.4 cm^2.

Characterization. The structure of the prepared films has been studied by X-ray diffraction studies using a PANalytical X-ray diffractometer with nickel-filtered CuKα (30 kV, 30 mA). The surface morphology of the films were studied using scanning electron microscopy (SEM; S-4100, Hitachi), and the atomic composition of the film was measured by energy dispersive X-rayanalysis (EDS; EX-250, Horiba) operated at 120 kV. UV-visible absorption spectra of extracted pigments in acidified ethanol solution and ZnO films have been recorded using a Cary 500 spectrometer. The photocurrent-voltage (*J-V*) characteristics of the devices were measured using white light from a xenon lamp (max. 150 W) using a sun 2000 solar simulator (ABE technologies). Light intensity was adjusted using a Si solar cell to ~AM-1.5. Incident light intensity and active cell area were 100 mWcm^{-2} (one sun illumination) and 0.4 cm^2 (0.5 × 0.8 cm), respectively.

Results and Discussion

Figure 1 shows the X-ray diffraction pattern of the ZnO seed layer and the ZnO nanorods. All the diffraction peaks in the pattern can be well indexed to the hexagonal wurtzite structure of ZnO with lattice constants a = 3.256 Å and c = 5.215 Å (JCPDS Cardno. 36-1451). The diffraction peaks at 2θ (degrees) of 32.7°, 35.2° and 36.8° are respectively

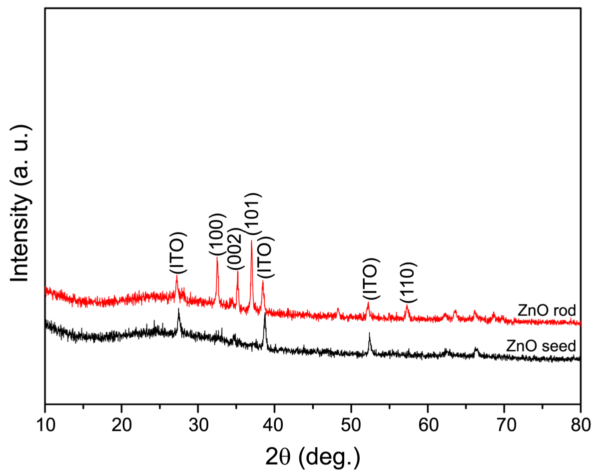

Figure 1. X-ray diffraction pattern of the ZnO seed layer and the ZnO nanorods.

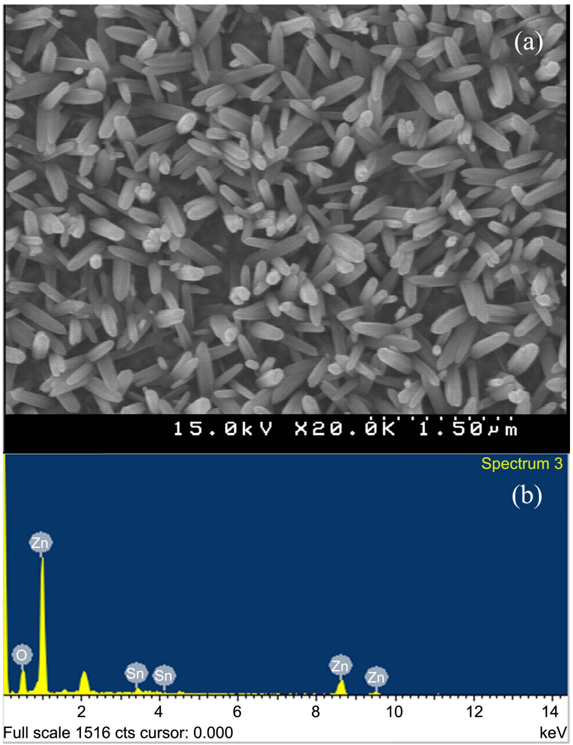

Figure 2. (a) SEM image of ZnO nanorods (b) EDXA pattern of ZnO nanorods.

indexed to (100), (002), and (101) planes of ZnO. Compared to the standard pattern of hexagonal phase ZnO, the relative intensity of the peak corresponding to (002) plane is extremely weak, which is due to the fact that the (00n) planes are absent in the ZnO nanorods.[19]

Figure 2(a) is the scanning electron microscope image of ZnO nanorods. The SEM image shows that the prepared films have rod like structure. The ZnO nanorods have been grown almost vertically from seed layer. Energy dispersive x-ray analysis (EDXA) of ZnO is shown in Figure 2(b). EDXA spectra result shows the presence of Zn and O. The unidentified peak at 2 keV is due to presence of indium. The origin of indium and tin (Sn) are due to the ITO substrate.

Figure 3(a, b, c) shows the UV-vis spectra of rose, beetroot and strawberry extracts. It is found that the absorption peaks for rose, beetroot and strawberry extracts are at about 400 nm, 485 nm and 420 nm respectively. The difference in the absorption characteristics is due to the presence of different types of flavonoids and colors in the extracts. The attachment of dye to the ZnO electrode can also be ascertained through the absorption spectra. Figure 3(d) shows the absorption spectra of ZnO electrode sensitized with three different natural dyes. It can be seen that the intensity of light absorption increases with the adsorption of natural dyes onto ZnO. Bare ZnO shows no absorption of light beyond 400 nm. The sensitization of ZnO with dye increases the absorption of light in the visible region below 550 nm. Compared with other two natural dyes strawberry sensitizer greatly increases the absorption of light by ZnO in the visible region which dominates the terrestrial solar spec-

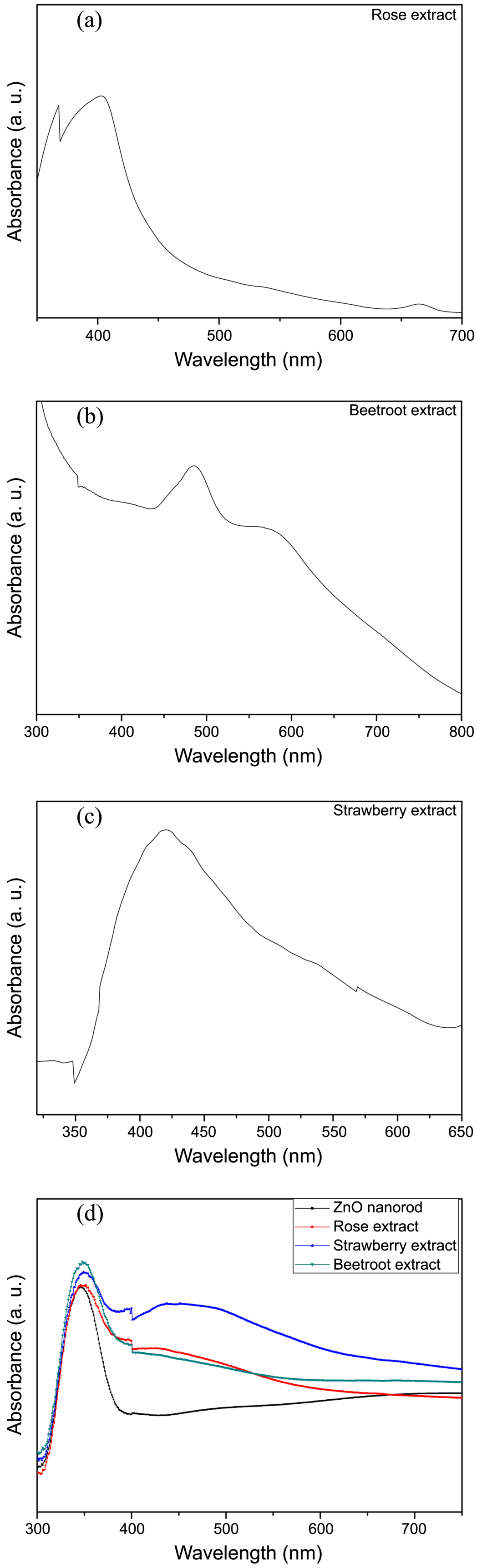

Figure 3. UV-vis spectra of (a) rose, (b) beetroot, (c) strawberry extracts and (d) ZnO working electrode sensitized with three different natural dyes.

trum. Typically ZnO has its featured absorption band in the region of 300-400 nm, while strawberry sensitizer has the featured absorption band in the 400-580 nm region. The red shift of the absorption edge of ZnO implies the absorption of more dye molecules onto ZnO. Even though beetroot extract has broad absorption range (Figure 3(b)), the adsorption on the working electrode is lesser than the strawberry extract (Figure 3(d)). The reason may be the pigments present in the strawberry (anthocyanin) and beetroot (betanin) extracts do not attach to ZnO by the same amount. The adsorption coefficient or the amount of dye molecules adsorbed on the surface of ZnO working electrodes were determined by spectroscopic measurement of the dye desorbed from the surface. The ZnO working electrodes were soaked in an ethanol solution containing dye solutions (0.1 M) at room temperature for 24 h. After sensitizing ZnO electrodes with the dye molecules the electrodes were sequentially washed with water/ethanol and dried in the air. In order to analyze the amount of dye loading in ZnO electrode, the dye was desorbed from the electrode into NaOH solution in water/ethanol (1.0 M, 50:50, v/v). A UV-vis spectrophotometer was employed to measure the dye concentration in the desorbed-dye solution. The calculated values are 9.5×10^{-8}, and 20×10^{-8} mol/cm^2 for beetroot and strawberry extracts respectively. This result indicates that the amount of dye adsorbed are remarkably different, even though the same concentration was maintained for both the dyes. This is due to the difference in the adsorption rate of the dye on the film. The low amount of adsorption of betanin is due to the weak bond between the dye molecule and the surface of ZnO film. Whereas the dye molecules present in strawberry extract (anthocyanin) easily interacts with the ZnO surface, so large amount of dye molecules are adsorbed on the surface. Due to this strawberry extract sensitized electrode shows broader absorption than the other extract sensitized electrodes.

Figure 4 shows the photocurrent density-voltage (*J-V*) characteristics of natural dye (prepared at room temperature) sensitized ZnO nanorod based solar cells. The solar cell parameters such as open circuit voltage, short circuit current, fill factor and efficiency are obtained by averaging measures from three dye sensitized solar cells. Three different similarly prepared experiments were performed to determine the statistical error of voltage and current measurements. The conversion efficiency (η) of the strawberry extract sensitized ZnO nanorod based solar cell is 0.22% with short circuit current density of 0.81 mA/cm^2, open circuit voltage of 0.48 V and fill factor of 0.56, while the conversion efficiency (η) of the beetroot extract sensitized solar cell is 0.1% with a short circuit current density of 0.39 mA/cm^2, open circuit voltage of 0.46 V and fill factor of 0.66. The conversion efficiency (η) of the rose extract sensitized solar cell is 0.08% with a short circuit current density of 0.28 mA/cm^2, open circuit voltage of 0.45 V and fill factor of 0.66. Table 1 shows the photovoltaic performance of natural dye sensitized ZnO nanorod based solar cells prepared using different natural dyes extracted at room temperature. It is observed that the solar cells prepared using strawberry extract shows maximum short-circuit current density (J_{sc}) and conversion efficiency compared to other solar cells. This is due to a more and broader range of light absorption of the strawberry extract present on ZnO nanorods and this is also clearly seen in the UV-vis absorption spectra. The higher interaction between ZnO nanorods and the anthocyanin in the strawberry extract leads to a better charge transfer. Under room temperature dye extraction the dye sensitized solar cells prepared using strawberry extract showed better performance when compared to the dye sensitized solar cells prepared using other natural dyes. Therefore strawberry extract sensitized solar cell was chosen for further study.

The effect of pH value on the performance of the solar cell was investigated. Figure 5(a) shows the photocurrent density-voltage (*J-V*) characteristics of natural dye (extracted at room temperature and at different pH values) sensitized ZnO nanorod based solar cells. The pH of the dye extract has an important effect on the performance of natural dye sensitized solar cells and it is shown in Table 2. The solar cells fabricated using ZnO sensitized using dye extract with pH values 1, 2 and 3 show efficiency values of 0.24%, 0.28% 0.25 % respectively. The result shows that the optimum pH for dye extraction is 2.

This is also confirmed by using UV-vis absorption spectra of the synthesized dye and the dye sensitized ZnO working

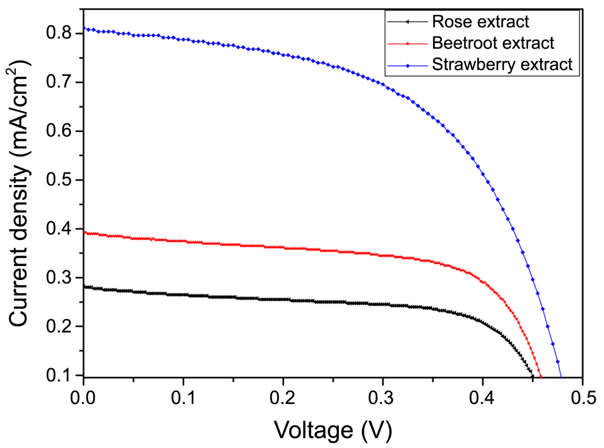

Figure 4. *J-V* characteristics of natural dye sensitized solar cells.

Table 1. Solar cell parameters of the ZnO nanorod based solar cells sensitized with different natural dyes

Dyes	V_{oc} (V)	J_{sc} (mA/cm^2)	FF	CE
Rose extract	0.45	0.28	0.65	0.08
Strawberry extract	0.48	0.81	0.56	0.22
Beetroot extract	0.46	0.39	0.66	0.12

Table 2. Solar cell parameters of the cells sensitized with strawberry dye extracted with different pH values

pH	V_{oc} (V)	J_{sc} (mA/cm^2)	FF	CE
1	0.45	0.69	0.65	0.24
2	0.50	0.99	0.87	0.28
3	0.46	0.74	0.68	0.25

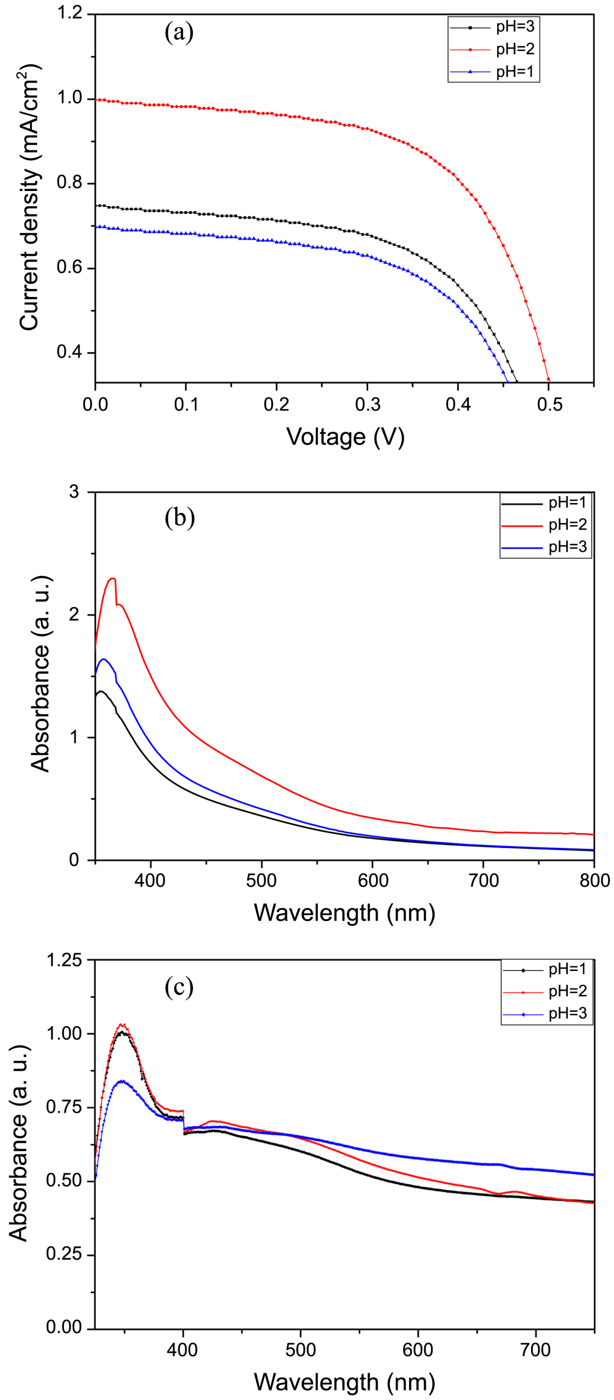

Figure 5. (a) *J-V* characteristics, absorption spectra of the (b) synthesized dye and (c) dye sensitized working electrode.

electrodes. Figure 5(b) shows the absorption spectra of the natural dyes synthesized at different pH values. The absorption spectra clearly shows that the absorption of light is found to have enhanced at pH = 2. The absorption band of the dye adsorbed ZnO working electrode having a dye with pH = 2 is found to be extended more to longer wavelength when compared to the dye sensitized films sensitized with dyes prepared at pH = 1 and 3, and is shown in Figure 5(c). The reason for the better efficiency at pH = 2 is the adsorption of more dye molecules (20×10^{-8} mol/cm^2) on the working electrode. As a result, more electrons are injected from the dye molecules, resulting in high J_{sc}. Normally, J_{sc} is directly related to amount of dye-sensitizer adsorbed on the ZnO surface because the dye sensitizer acts as a photo receiver to absorb photon and inject the electron to generate current.

Table 3. Solar cell parameters of the cells sensitized with strawberry dye extracted with different temperatures

Temperatures	V_{oc} (V)	J_{sc} (mA/cm^2)	FF	CE
Room temp	0.48	0.81	0.56	0.22
50 °C	0.45	1.80	0.87	0.46
75 °C	0.47	1.85	0.85	0.49
100 °C	0.45	1.76	0.82	0.42

The dyes synthesized at pH = 2 shows good interaction with the working electrode, the reason is at pH = 2, the anthocyanin existed as flavylium ion, which is stable form of anthocyanin; an increasing pH hydrated this ion to quinonoidal bases.[20] However, the cell deterioration by acid leaching is expected as the pH goes lower (pH = 1), which results in a lower efficiency.[21]

Figure 6(a) shows the photocurrent density-voltage (*J-V*) characteristics of strawberry extract sensitized solar cells extracted at different temperatures. The effect of dye extracting temperature on the performance of dye sensitized solar cells is shown in Table 3. Solar cell sensitized using dye extracted at 50 °C shows a power conversion efficiency of 0.46%, with V_{oc} of 0.45 V, J_{sc} of 1.80 mA/cm^2 and FF of 0.87. Solar cell sensitized using the dye extracted at 75 °C shows power conversion efficiency of 0.49%, with V_{oc} of 0.47 V, J_{sc} of 1.85 mA/cm^2 and FF of 0.85. Solar cell sensitized using dye extracted at 100 °C shows a conversion efficiency of 0.42% with V_{oc} of 0.45V, J_{sc} of 1.76 mA/cm^2 and FF of 0.82.

The solar cell sensitized using dye extracted at 75 °C shows maximum power conversion efficiency, whereas solar cell sensitized using dye extracted at higher temperature (100 °C) show lesser efficiency; this is due to the less stability of anthocyanin at higher temperatures. At high temperature, the thermal degradation of anthocyanin could be caused due to the loss of glycosyl moieties and α-diketone formation.[22]

Solar cells sensitized using the dyes extracted at lower temperatures (room temperature and 50 °C) also gives lower efficiency than that of the solar cell sensitized using the dyes extracted at 75 °C. This is due to the lighter color of the extract, which is due to restriction of anthocyanin solubility.[20] It shows that, the optimum extracting temperature is 75 °C which is in between room temperature and boiling point of water. To confirm the effect of dye synthesizing temperature on the performance of solar cell, UV-vis absorption spectra was recorded for synthesized dye and dye sensitized working electrode. Figure 6(b) shows the absorption spectra of natural dyes synthesized at different temperatures. The dye synthesized at 75 °C shows greater absorbance of light than the dye synthesized at other temperatures. The reason is the dye

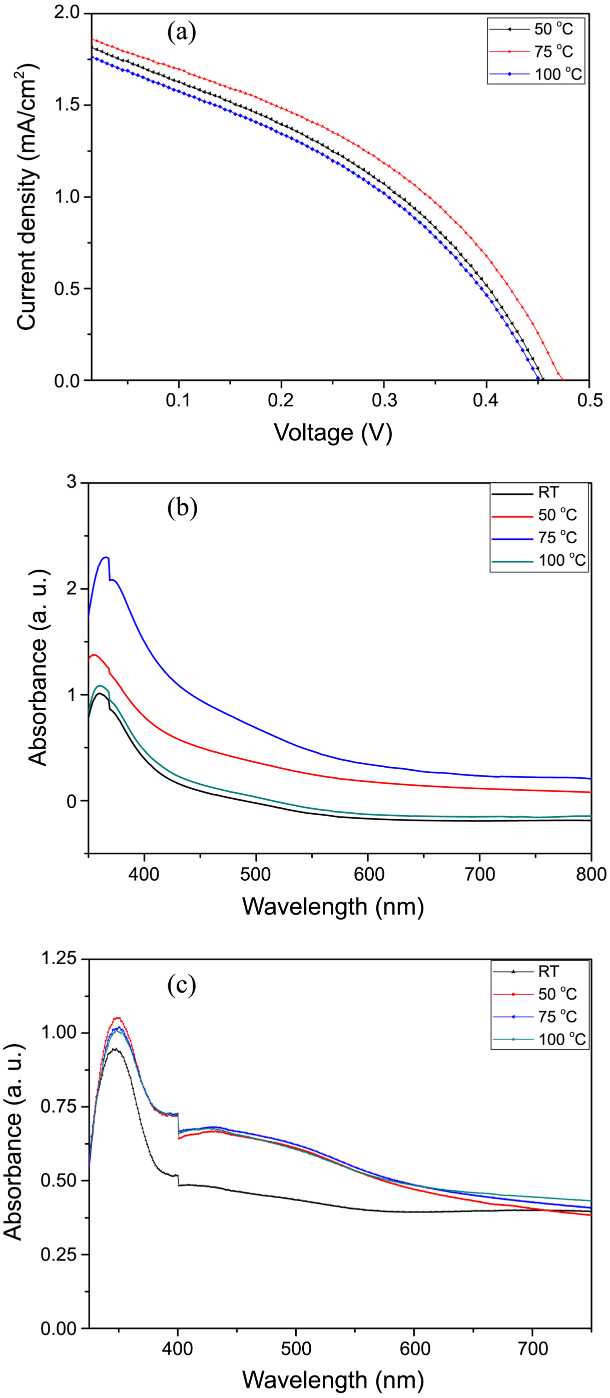

Figure 6. (a) *J-V* characteristics, absorption spectra of the (b) synthesized dye and (c) dye sensitized working electrode.

extracted at 75 °C resulted in deep colored solution. Figure 6(c) shows the absorption spectra of the natural dye (prepared at different temperatures) adsorbed ZnO working electrodes. The extracts prepared at 75 °C absorb visible light and sensitize the oxide semiconductor to low-energy irradiation. The broadening of the absorption band of the colored photo anode is related to the charge transfer interaction responsible for the binding of the dye to the oxide surface. The amount of dye molecules adsorbed on the surface of ZnO working electrodes for room temperature,

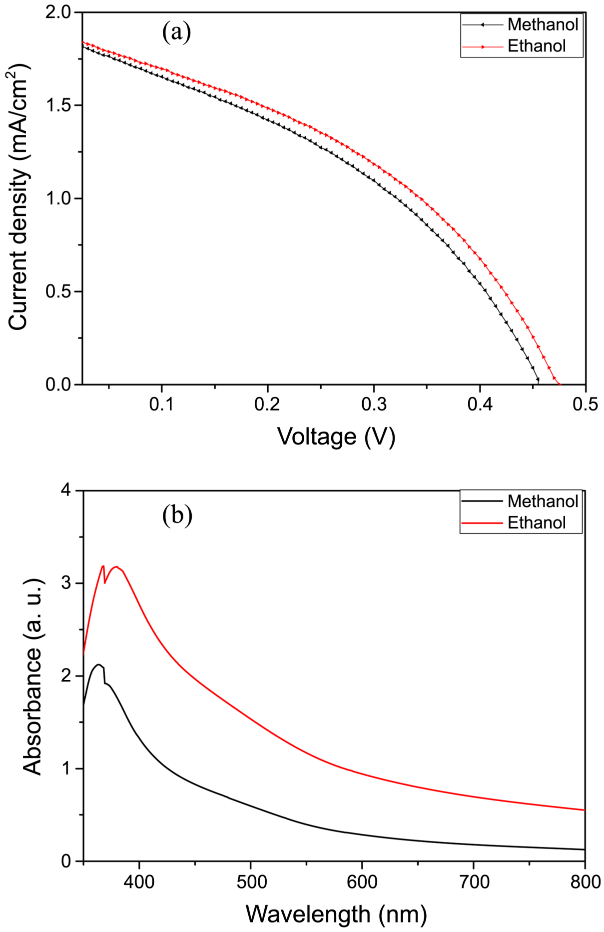

Figure 7. (a) *J-V* characteristics and (b) absorption spectra of the synthesized dye.

50, 75 and 100 °C are 20×10^{-8} mol/cm^2, 24×10^{-8} mol/cm^2, 27×10^{-8} mol/cm^2 and 22×10^{-8} mol/cm^2 respectively.

Figure 7(a) shows the photocurrent density-voltage (*J-V*) characteristics of natural dye (extracted using different solvent) sensitized ZnO nanorod based solar cells. As shown in Table 4 the solar cells prepared using natural dye extracted in ethanol shows a higher efficiency than that of solar cells prepared using natural dye extracted in methanol. This may due to the higher solubility of anthocyanin in ethanol and hence the aggregation of dye molecules is less as expected.[23] A good dispersion of dye molecules on the oxide surface could in fact improve the efficiency of the system. Due to this dyes extracted using ethanol shows good adsorption on the working electrode. The adsorption coefficient of the dye molecules on the working electrode using ethanol and methanol solvents are 27×10^{-8} mol/cm^2 and 24×10^{-8} mol/cm^2

Table 4. Solar cell parameters of the cells sensitized with strawberry dye extracted with different solvents

Solvent	V_{oc} (V)	J_{sc} (mA/cm^2)	FF	CE
Ethanol	0.47	1.85	0.85	0.49
Methanol	0.45	1.81	0.82	0.42

respectively. The absorption spectrum of the synthesized dye is shown in Figure 7(b). The figure clearly shows that the dye extracted using methanol absorbs less light compared to that of the dye extracted using ethanol.

Conclusion

The ZnO nanorod working electrodes have been prepared by simple hydrothermal method. The prepared ZnO working electrode was sensitized with natural dyes extracted from beetroot, rose and strawberry. The efficiency of strawberry extract sensitized solar cell shows better performance than the other dye sensitized solar cells. It was also found that the efficiency of the dye sensitized solar cells can be enhanced by changing the solvent used in the preparation of the dye, changing the dye extracting temperature and pH of the extract. Ethanol is found to be the suitable solvent for natural dye, the optimum dye extracting temperature is found to be 75 °C and the suitable value of pH is found to be 2.

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