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Microwave assisted biosynthesis of rice shaped ZnO nanoparticles using *Amorphophallus konjac* tuber extract and its application in dye sensitized solar cells

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Rice shaped ZnO nanoparticles have been synthesized for the first time by a biological process using *Amorphophallus konjac* tuber extract and used as a photoanode in a dye sensitized solar cell. The glucomannan present in aqueous tuber extract acted as a reducing agent in the synthesis process, further it also acted as a template which modified and controlled the shape of the nanoparticles. The synthesized nanoparticles were dried by microwave irradiation followed by annealing at 400 °C. The FESEM and TEM images confirmed that the synthesized ZnO nanoparticles had rice shaped morphology. Furthermore, the X-ray diffraction studies revealed that the prepared ZnO nanoparticles exhibited wurtzite phase with average particle size of 17.9 nm. The UV-Vis spectroscopy studies confirmed the value of band gap energy of biosynthesized ZnO nanoparticles as 3.11 eV. The photoelectrodes for dye sensitized solar cells were prepared with the biosynthesized ZnO nanoparticles using doctor blade method. The photoelectrode was sensitized using the fruit extract of *Terminalia catappa*, flower extracts of *Callistemon citrinus* and leaf extracts of *Euphorbia pulcherrima*. The dye sensitized solar cells were fabricated using the sensitized photoelectrode and their open circuit voltages and short circuit current densities were found to be in the range of 0.45 V to 0.55 V and 5.6 mA/cm^2 to 6.8 mA/cm^2, respectively. Thus, the photovoltaic performances of all the natural dye sensitized ZnO solar cells show better conversion efficiencies due to the morphology and preparation technique.

Keywords: *green synthesis; Amorphophallus konjac tubers extract; rice shaped ZnO nanoparticle; dye sensitized solar cell; natural dye; XRD*

1. Introduction

ZnO is one of photoconductive n-type semiconductors with band gap energy of 3.37 eV with good transparency [1], high electron mobility [2] and strong room temperature luminescence [3]. Hence, it is highly preferred for variety of applications like sensors, memory devices UV-light emitting diodes, piezoelectric devices, photodiodes and photodetectors [4]. Apart from these applications, ZnO nanoparticles possess the potential to be used as photoelectrodes for energy conversion of solar energy to electricity. The dye sensitized solar cells are now becoming most efficient third generation solar cells. Remarkable conversion efficiencies have been reported with ZnO nanoparticles as photoelectrodes using different nanostructures and combinations [5–7]. In spite of these achievements cost and eco-friendly nature of these cells could be improved if the natural products were involved in construction of these cells. Cost effective eco-friendly dyes and electrolyte have been utilized in these solar cells and were successful in achieving better conversion efficiencies [8–13]. This paper presents a green approach to fabricate dye sensitized solar cells via biosynthesis of photoelectrode material.

ZnO nanoparticles are synthesized by different methods such as wet chemical method [14], vapor phase method [15], hydrothermal method [16], precipitation method [17], atomic layer deposition [18] and sonochemical method [19]. These methods involve toxic reagents, expensive equipment with tedious and also time consuming processes.

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Biosynthesis method is considered to be an alternative to the conventional methods which involve the use of plant extracts [20], fungi [21], viruses [22] and bacteria [23] in the nanoparticle synthesis process. Among the above mentioned, green synthesis process using plant extracts is relatively simple. Indeed, it does not require any special equipment and complex procedures. The phytoconstituents present in the plant extracts act as reducing and capping agents in the synthesis process, further they also act as a template which modifies and controls the shape of the nanoparticles [24]. The nanoparticles synthesized by biosynthesis method result in different morphologies such as spherical shaped [25], fibril shaped [26], hexagonal shaped [27], cubical shaped [28] and triangular shaped [29] nanoparticles. Plant extracts from *Terminalia cuneata* is used to synthesize silver nanoparticles [30], *Hibiscus sabdariffa* is used to synthesis of gold nanoparticles [31], *Magnolia kobus* leaf extracts are used to synthesis of copper nanoparticles [32], peels of *Punica granatum* are used for synthesizing ZnO nanoparticles [33]. Owing to the above said, the biosynthesis of metal oxide nanoparticles is considered in the literature as a reliable method.

In this study a novel synthesis method of ZnO nanoparticles using *Amorphophallus konjac* tubers extracts has been presented. *Amorphophallus konjac* tubers containing glucomannan, glucose, fructose, and sucrose has been reported. Every 100 g of konjac tuber contains 1.64 g protein, 0.004 g fat, 57 mg phosphorus, 4.06 mg iron, 123 mg zinc, 0.2 mg manganese, 0.25 mg chromium, 0.08 mg copper and 79.37 mg glucomannan [34]. The air dried 1 g powder of *Amorphophallus konjac* tubers contains almost 46.33 mg of flavonoid content, 12.67 mg of phenolic content [35].

The synthesis of ZnO nanoparticles involves microwave assisted drying process. Microwave drying helps in rapid and homogenous heating of the reaction mixture to the desired temperature, which saves time and energy. Various morphologies, like rod shaped, spherical shaped, flower shaped, can be obtained by microwave irradiation technique [36–38]. The phytochemicals present in the *Amorphophallus konjac* tuber extracts show good vibration response to the microwave irradiation [39]. Prepared ZnO nanoparticles were used for making a dye sensitized solar cell. Photoelectrodes for the dye sensitized solar cell were prepared by coating a thick film of prepared ZnO nanoparticles over the conductive side of FTO substrate by doctor blade method. The thick film provides more dye absorption over the nanoparticles compared with thin films and results in good photon to electron conversion efficiency [40]. The prepared photoelectrodes were sensitized using natural dyes extracted from fruits of *Terminalia catappa*, flowers of *Callistemon citrinus* and leaves of *Euphorbia pulcherrima*. The characteristics of synthesized ZnO nanoparticles and fabricated dye sensitized solar cells have been studied and analyzed.

2. Experimental

Zinc acetate dehydrate $Zn(CH_3COO)_2 \cdot 2H_2O$ (99 %) was purchased from Sigma-Aldrich and was used as received. Fresh *Amorphophallus konjac* tubers were purchased from local market in Coimbatore, Tamilnadu.

2.1. Preparation of tuber extract

Fresh *Amorphophallus konjac* tubers were cleaned with double distilled water. The outer skin of the tubers was peeled out and the flesh alone was chopped into tiny pieces. 10 g of chopped tubers were taken in a glass beaker with 200 mL of double distilled water and heated at 70 °C for 30 min until the color of the solution changed from watery to milky. Then, the content was centrifuged to remove insoluble parts and the supernatant was stored in a glass vial.

2.2. Preparation of ZnO nanoparticles

0.2 M of zinc acetate dihydrate precursor was dissolved in 100 mL of water using a magnetic stirrer at 70 °C for 30 min. Then, 20 mL of prepared tuber extract was added to the zinc acetate dihydrate solution and was stirred at 80 °C for 1 h resulting in the formation of pale white precipitate. The solution was decanted and the precipitate was dried in microwave oven for 5 minutes. Finally, a pale white powder obtained was annealed at 400 °C for 1 h.

2.3. Preparation of ZnO photoelectrode

The prepared ZnO nanoparticles were coated onto the conducting side of FTO substrate by doctor blade method. Few drops of very dilute acetic acid were added to 1 g of prepared ZnO nanoparticles and grinded in a mortar with a pestle until a colloidal suspension with a smooth consistency was obtained. Then 2 or 3 drops of the ZnO suspension was dropped on the conductive side of FTO substrate and spread out evenly on the surface of the FTO with a glass rod. Finally, the substrate was dried at 200 °C for 30 min and naturally cooled down to room temperature.

2.4. Extraction of natural dye sensitizers

The dyes used to sensitize the ZnO photoelectrode have been extracted from fruits of *Terminalia catappa*, flowers of *Callistemon citrinus* and leaves of *Euphorbia pulcherrima.* The flesh from the fruits of *Terminalia catappa* along with the skin was allowed to dry in shade. After drying it completely, the dried skins of the fruits were powdered in a blender. The powder was soaked in 100 mL of ethanol at room temperature for 24 h and then the solid residues were filtered out. The filtrate was washed with hexane several times to remove any oil residues. Then, it was directly used as the dye solution for sensitizing the prepared ZnO photoelectrodes.

The dye from *Callistemon citrinus* was prepared from its spike shaped flowers. The flowers were cleaned and dried in shade. After drying, they were soaked in 100 mL of ethanol at room temperature for 24 h and then the discolored white spikes were filtered out. The filtrate was used as the dye solution for sensitizing ZnO photoelectrodes.

The dye from *Euphorbia pulcherrima* was prepared from its red colored leaves. The leaves were collected, cleaned, cut into small pieces and dried in shade. After drying, they were soaked in 100 mL of ethanol at room temperature for 24 h and then the discolored leaves were filtered out. The filtrate was washed with hexane several times to remove any oil and chlorophyll residues. The resultant red colored dye was directly used as the dye solution for sensitizing ZnO photoelectrodes.

2.5. Sensitization of ZnO photoelectrode

The prepared photoelectrode has to be sensitized by a light absorbing dye in order to inject electrons to the electrode by photoexcitation process. Each ZnO photoelectrode was immersed into a separate beaker containing the dye extracted from the fruits of *Terminalia catappa*, flowers of *Callistemon citrinus* and leaves of *Euphorbia pulcherrima.* The sensitization was carried out in a dark environment at room temperature for about 10 h. After sensitization the electrodes were rinsed with ethanol to remove the excess of dye present in the electrode and then the electrode was dried. Thus, the prepared ZnO photoelectrode was sensitized with natural dyes.

2.6. Dye sensitized solar cell assembly

The fabrication of dye sensitized solar cell involves basic components, such as dye sensitized photoelectrode (working electrode), electrolyte and a counter electrode. In the present work, the dye sensitized ZnO photoelectrode acted as the working electrode. The platinized FTO glass was used as a counter electrode and it was placed on the top of the dye sensitized ZnO photoelectrode and sealed with 30 μm thick thermal adhesive film. The iodide/triiodide redox electrolyte solution was filled into the space between the photoelectrode and the counter electrode through a hole made in the counter electrode, due to capillary action. After filling the electrolyte the hole were sealed using the adhesive film. With the same procedure five solar cells were constructed with each dye and their J-V characteristics were analyzed.

2.7. Characterization

The crystalline properties of the prepared ZnO nanoparticles have been studied using X-ray diffractometer (Rigaku RINT 200 series). The surface morphology and chemical composition were studied using field emission scanning electron microscope and energy dispersive X-ray analysis, respectively (SIGMA HV-Carl Zeiss with Bruker QUANTAX 200-Z10 EDS Detector). The atomic structure of the sample was studied using high-resolution transmission electron microscope (JEOL, JEM-2100). The absorbance spectra of the nanoparticles and the dye have been recorded using a spectrophotometer (JASCO V-570). The J-V curves of DSSCs were taken using Keithley 2400 digital source meter under an irradiation of 100 mW/cm^2.

3. Results and discussions

The XRD patterns of the synthesized ZnO nanoparticles are shown in the Fig. 1a. The results show that there are nine peaks at 31.93°, 34.71°, 36.51°, 47.76°, 56.83°, 63.06°, 68.16°, 69.28°, 77.31° corresponding to (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 1), (1 1 2), (2 0 2) planes, clearly indicating that the prepared sample is of wurtzite type. No other peak related to impurities has been observed in the diffraction pattern. The interplanar spacing of synthesized ZnO nanoparticles was calculated using Debye-Scherrer equation:

$$D = 0.9\lambda/(\beta \cos\theta) \quad (1)$$

where D is the crystallite size, λ is the wavelength of X-ray used, θ is Bragg's angle and β is the full width at half maximum. The average crystallite size was determined to be 17.9 nm.

Fig. 1b shows the FESEM image of biosynthesized ZnO nanoparticles. The ZnO nanoparticles are in the form of rice-like structures with the length of 237 nm and diameter of 76 nm. The inset shows the image of rice which is similar to the morphology of the biosynthesized ZnO nanoparticles. Fig. 1c shows the EDAX spectrum of the biosynthesized nanoparticles which confirms the only presence of elemental zinc and oxygen. The weight percentage of elemental zinc and oxygen was found to be 70.44 % and 29.56 %, respectively.

The TEM image of biosynthesized ZnO nanoparticles is shown in Fig. 1d. The image confirms the rice shaped morphology of the synthesized ZnO nanoparticles. The selected area electron diffraction (SAED) pattern of the biosynthesized ZnO nanoparticles is shown in Fig. 2a. The pattern shows a regular polycrystalline ring with diffraction spots produced due to superposition of several single crystal orientations. The image also shows some weak diffraction spots. Fig. 2b shows the HRTEM image of the biosynthesized ZnO nanoparticles. The image shows the presence of lattice fringes with d-spacing of 5.86 Å.

The absorption spectra were investigated in solution state for both dye and dye sensitized nanoparticles. Fig. 3a shows the UV-Vis absorption spectra of as-synthesized ZnO nanoparticles. The absorption peak is observed at 372 nm. Fig. 3b shows the Tauc's plot of the biosynthesized ZnO nanoparticles. The graph presents $(\alpha h\upsilon)^2$ as a function of photon energy $h\upsilon$, where α is the absorption coefficient. The linear portion of the graph shows the value of band gap as 3.11 eV which is lower than that of the pure ZnO (3.3 eV). This reduced band gap energy may be due to some plant chemicals which are substituted into the lattice. This is in agreement with the previous studies on green synthesis of ZnO nanoparticles using *Plectranthus amboinicus* [41].

The absorption spectra of flower extract of *Callistemon citrinus* and ZnO photoelectrode sensitized by flower extracts of *Callistemon citrinus* in ethanolic solution is shown in Fig. 4a. The absorption spectra indicate that the light absorbance of the photoelectrode has been increased due to sensitization. The flower extracts of *Callistemon citrinus* contains cyanidin-3,5-diglucoside which provides red color to the flower. Cyanidin-3,5-diglucoside belongs to the anthocyanins which are a class of natural chemicals belonging to the flavonoids. The maximum absorption wavelength of cyanidin-3,5-diglucoside lies in the range of 520 nm to 540 nm, depending on its concentration and pH. The chemical structure of cyanidin-3,5-diglucoside is shown in Fig. 5a.

The leaves of *Euphorbia pulcherrima* are rich in germanicol which is used in sunscreen lotions since it absorbs light to give red color to its leaves.

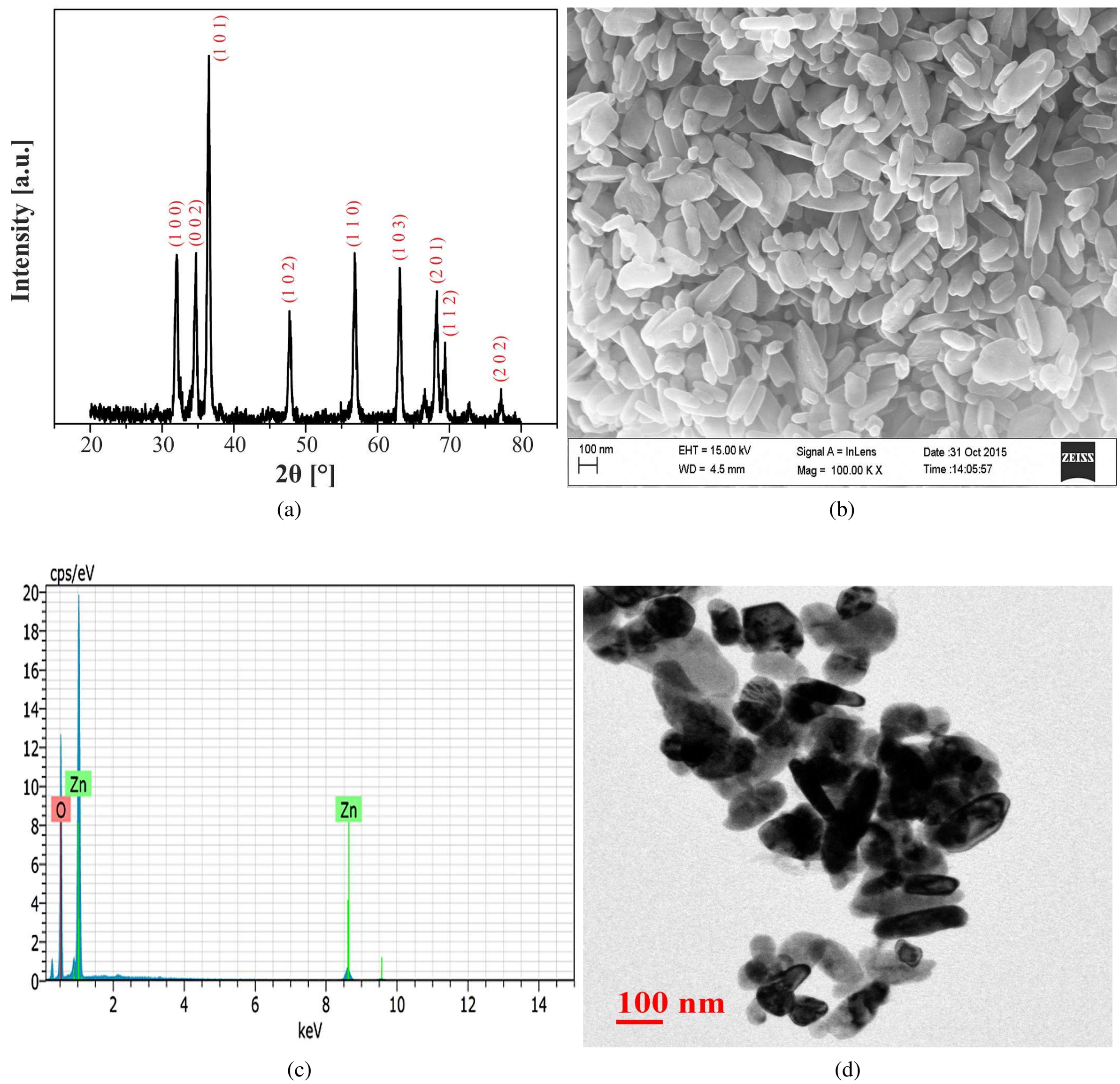

Fig. 1. Results of study of biosynthesized ZnO nanoparticles: (a) XRD pattern; (b) FESEM image; (c) EDAX and (d) TEM image.

Fig. 4b shows the absorption spectra of the extracts from leaves *Euphorbia pulcherrima* and ZnO photoelectrode sensitized with *Euphorbia pulcherrima* leaf extracts in ethanolic solution. This spectrum also shows that the absorption peak increase due to sensitization. The chemical structure of germanicol is shown in the Fig. 5b.

Fig. 4c shows the absorbance spectra of fruit extracts of *Terminalia catappa* and ZnO photoelectrode sensitized by fruit extracts of *Terminalia catappa* in ethanolic solution. The absorbance spectra of sensitized ZnO electrodes show the light absorbance which has been increased due to natural sensitizers. *Callistemon citrinus* absorbs in the wide wavelength range of 460 nm to 570 nm with an absorption peak at 536 nm. The light absorbance of ZnO photoelectrode has been improved by the sensitization of fruit extracts of *Terminalia catappa*. 100 g of the fruits of *Terminalia catappa* contains β-carotene (2.1 mg), ascorbic acid (138.6 mg) and vitamin E

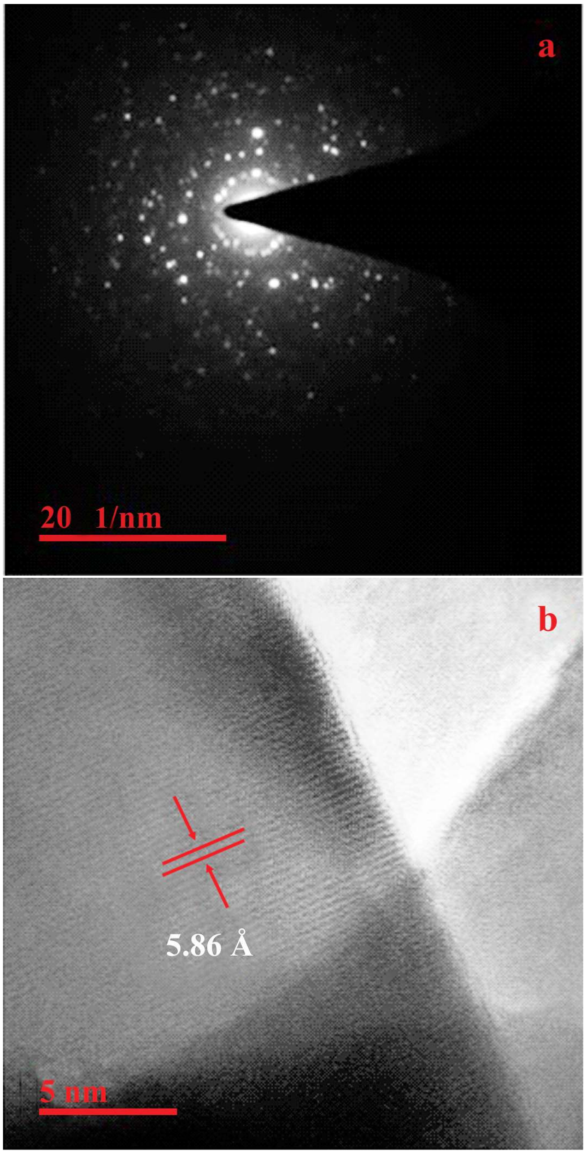

Fig. 2. (a) SAED pattern and (b) HRTEM image of biosynthesized ZnO nanoparticles.

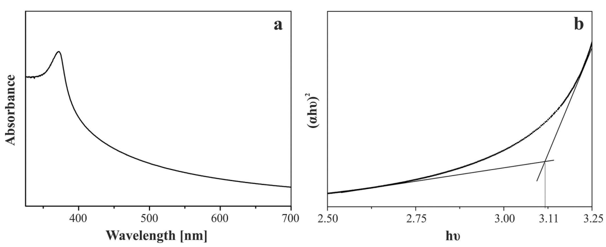

Fig. 3. (a) UV-Vis spectra and (b) Tauc's plot of biosynthesized ZnO nanoparticles.

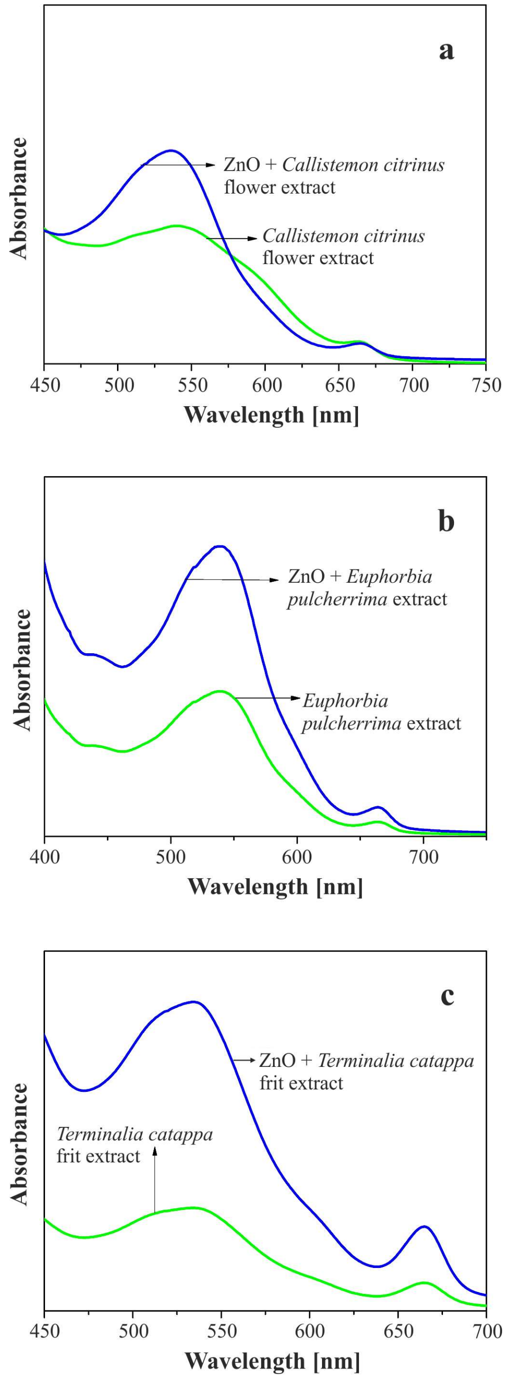

Fig. 4. Absorbance spectra of (a) flower extracts of *Callistemon citrinus* and ZnO sensitized by flower extracts of *Callistemon citrinus* (b) leaf extracts of *Euphorbia pulcherrima* and ZnO sensitized by leaf extracts of *Euphorbia pulcherrima* (c) fruit extracts of *Terminalia catappa* and ZnO sensitized by flower extracts of *Terminalia catappa.*

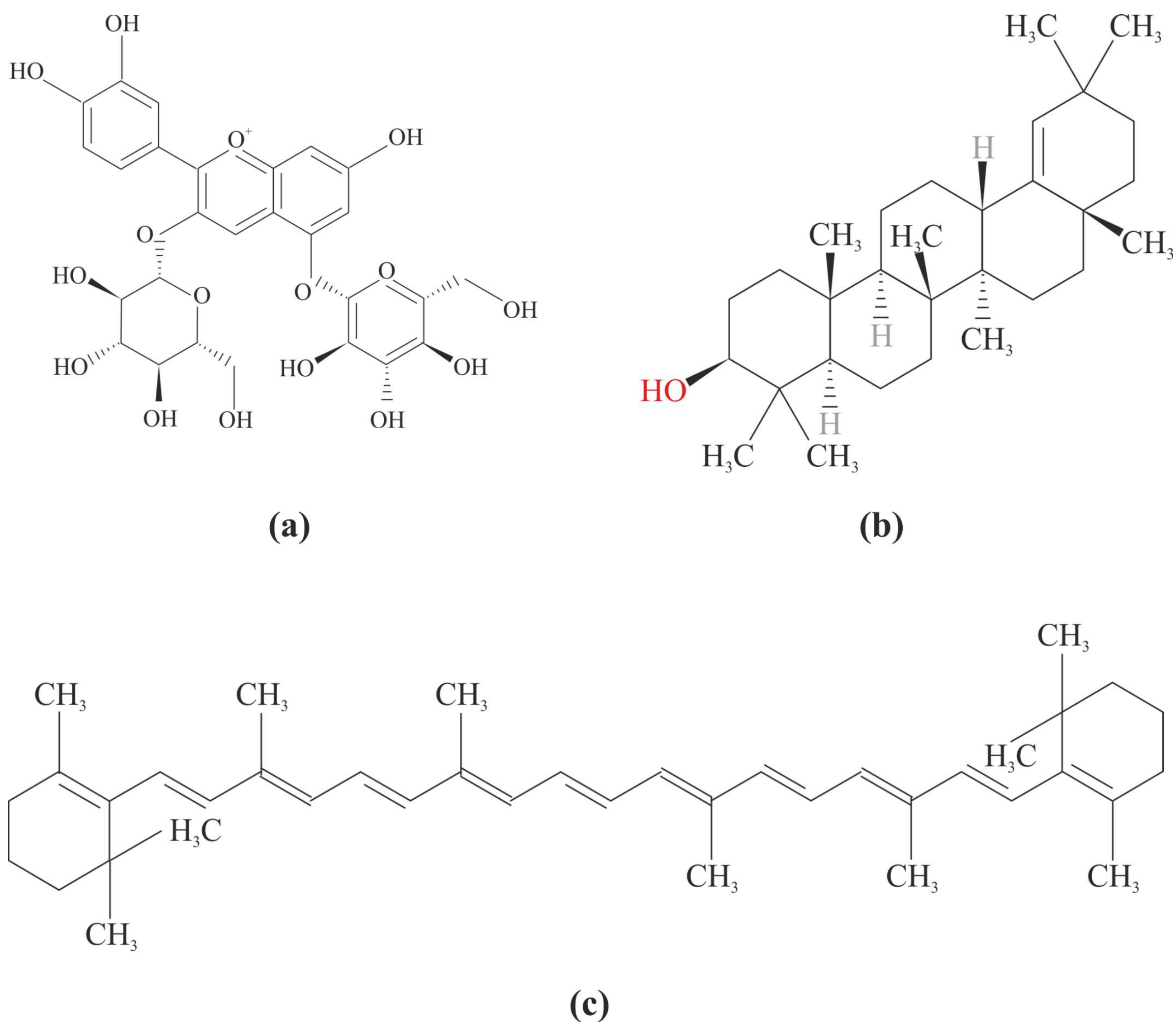

Fig. 5. Chemical structure of (a) cyanidin-3,5-diglucoside (b) germanicol (c) β-carotene.

(7.25 mg) as the major constituents [42]. The chemical structure of β-carotene is shown in Fig. 5c. The improvement in light absorption of the sensitized photoelectrode is mainly due to the presence of β-carotene in the dye [43].

Fig. 6 shows the recorded J-V characteristics of prepared solar cells and the calculated solar cell parameters, such as open circuit voltage, short circuit current density, fill factor, and efficiency. The performance of the dye sensitized solar cell depends on the morphology of the photoelectrode [44]. The parameters of the dye sensitized solar cells prepared using biosynthesized, rice shaped ZnO nanoparticles are given in Table 1. ZnO photoelectrodes sensitized with leaf extracts of *Euphorbia pulcherrima* have a power conversion efficiency of 1.66 % followed by the ZnO photoelectrode sensitized by *Terminalia catappa* fruit extracts with an efficiency of 1.63 %. The conversion efficiency of *Callistemon citrinus* flower extract sensitized photoelectrode shows a relatively lower conversion efficiency of 1.43 %. The devices remained stable for 14 day after which there was a decrease in J_{sc} values.

The result substantiates that the natural dye can be used as an effective sensitizer giving some modification of morphology of photoanode for better efficiency of the solar cell. The rice shaped nanoparticles absorb some amount of dye on their surface due to biosynthesis process involved. ZnO

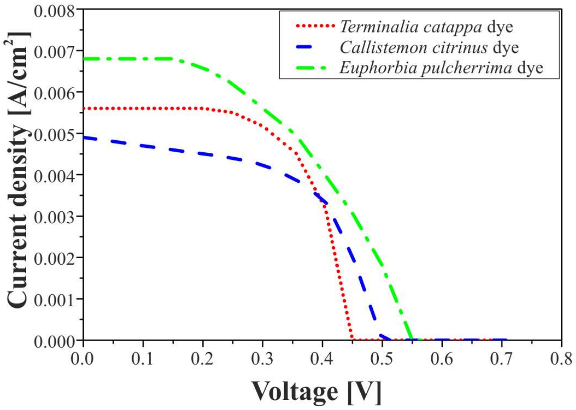

Fig. 6. J-V characteristics of biosynthesized ZnO based solar cells sensitized by natural dyes extracted from (a) flowers of C*allistemon citrinus,* (b) fruits of *Terminalia catappa*, and (c) leaves of *Euphorbia pulcherrima.*

nanoparticles have some organic compounds attached to their surface which bind the dye to the surface. The results show that the biosynthesized nanoparticles could improve the efficiency of dye sensitized solar cells, when sensitized with natural dyes.

Table 1. Solar cell parameters of fabricated ZnO based solar cells.

Dye extract/Solar cell parameters	V_{oc} [V]	J_{sc} [mA/cm^2]	Fill factor	Efficiency [%]
Callistemon citrinus	0.5	4.9	0.5857	1.43±2.4
Terminalia catappa	0.45	5.6	0.656	1.63±2.4
Euphorbia pulcherrima	0.55	6.8	0.4461	1.66±2.4

4. Conclusions

Rice shaped ZnO nanoparticles have been synthesized by microwave assisted biosynthesis method for the first time. The rice shaped morphology was obtained due to the phytochemicals present in *Amorphophallus konjac* tuber extracts. The X-ray diffraction studies and EDAX spectra confirmed that the prepared sample contain ZnO nanoparticles. The band gap energy determined from UV-Vis spectroscopy studies is 3.11 eV. The prepared ZnO nanoparticles were used to prepare photoelectrodes for dye sensitized solar cell application. ZnO photoelectrodes were sensitized using natural dyes extracted from fruits of *Terminalia catappa*, flowers of C*allistemon citrinus* and leaves of *Euphorbia pulcherrima.* The solar cell fabricated using ZnO sensitized with *Euphorbia pulcherrima* exhibited the best efficiency of 1.66 %.

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