Improved Performance of Natural Dye-Sensitized Solar Cells (NDSSCS) Using ZnO Doped TiO_2 Nanoparticles by Sol-Gel Method

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Abstract: ***Natural dye-sensitized solar cells (NDSSCs) have gained considerable attention in the field of solar energy due to their simple fabrication, good efficiency, and low production cost. In this study, a photoelectrode using ZnO doped TiO_2 nanoparticles was prepared by sol-gel method. The technology adopted in this paper is that the dye molecules absorb light, and produce excited electrons which in turn generate current in the output terminals of the cell. In this paper we investigate the optical absorption, functional group, surface morphology and elementary composition of pure TiO_2, ZnO doped TiO_2 nanoparticles and curcuma longa dye extract by using UV-Visible spectroscopy, PL-Studies, FT-IR, FE-SEM and EDS analysis. Finally photocurrent-voltaic characterization of nanocrystaline natural dye solar cell using I-V studies. It was found that the levels of short-circuit current (J_{sc}), open-circuit voltage (V_{oc}), fill factor (FF) and overall conversion efficiency (η) of various parts of curcuma longa dye extract. Further suggestions to improve the efficiency of NDSSC are discussed***

Keywords: ZnO doped TiO_2 electrode, sol-gel method and curcuma longa dye.

1. Introduction

The energy and fuel crisis is the prime concern worldwide. Fossils fuels are facing rapid resource depletion, but the demand for energy is growing day by day and many countries around the world have no alternative but to increase domestic oil process. So there is an urgent need of sustainable energy resources, such as the solar energy, which is considered as environment friendly, novel alternative and promising candidate to address this problem.

However, the solar energy has a limited application that directly related to its high cost of the per watt electricity generated. In the present time, technology of solar cells based on crystalline silicon is facing a problem of silicon-based raw materials. So, low cost alternatives and hence new types of low cost solar cells is an urgent issue. Dye-sensitized solar cells (DSSCs), a new type of solar cells, have attracted considerable attention due to their environmental friendliness and low cost of production [1]. A DSSC is composed of a nanocrystalline porous semiconductor electrode-absorbed dye, a counter electrode, and an electrolyte containing iodide and triiodide ions. In DSSCs, the dye as a sensitizer plays a key role in absorbing sunlight and transforming solar energy into electric energy [2].Over the past decade; nanomaterials have been the subject of enormous interest. TiO_2 nanomaterials are one of the potential candidates for solar energy application due to TiO_2's unique optoelectronic and photochemical properties especially, as a photovoltaic performance to efficient energy conversion for solar irradiations; TiO_2 nanomaterials have been receiving a great deal of attention. Recently, various techniques have been employed to modify the structure of the working electrode to improve the performance of the DSSC [3 & 4].

A typical DSSC cell consists of two FTO, with a nanoporous TiO_2 layer covered with dye solution in the middle of the cell. Below it are the liquid electrolyte and the counter electrode that is usually fabricated with Pt. The mechanics of DSSC is quite different to the conventional silicon solar cell. The idea was initially inspired from the photosynthesis of the leaves. TiO_2, especially its anatase phase, has attracted much attention for its potential application in degradation of various environmental pollutants, both gaseous and liquid however; its short comings include a large band gap (~3.2 eV) which causes most of the solar spectrum unutilized [5-7]. To extend the optical absorption of TiO_2 to the visible region, various dopants have been added to the oxide to improve its solar efficiency [8-9]. In this work we describe first the preparation of ZnO doped TiO_2 nanostructure, followed by the process of dye extraction from various parts of curcuma longa. In the second part we describe the solar cell preparation using four types of curcuma extracts namely parent round turmeric, parent finger turmeric, daughter round turmeric, and daughter finger turmeric. In the third part we examine the optical absorption, functional group, optical band gap, surface morphology and elementary composition of pure TiO_2, modified TiO_2 nanoparticles and curcuma longa dye extract by using UV-Visible, FT-IR, PL, FE-SEM & EDS analyses. Finally overall conversion efficiency for various parts of curcuma longa dye extracts are determined using photovoltaic characterization [10]

2. Materials and Methods

2.1. Chemicals Used

Most of the Chemicals used in the research are standard chemicals that are normally available in the laboratory. Special materials for DSSC are mostly bought from Solaronix. The chemicals used in this study were titanium tetrachloride, zinc acetate dehydrate, benzyl alcohol, absolute ethanol, acetyl acetone, Methanol, Isopropanol, DFM solvent, Triton X-100, Polyethylene glycol, Idolyte TG 50 and diethyl ether were bought from Sigma-Aldrich. All the chemicals were used without further purification.

2.2. Preparation of ZnO/ TiO_2 nanocomposites

Metal oxide nanoparticles attract great attention in recent years on account of their special electronic and chemical properties. Among the metal oxide semiconductors, TiO_2 and ZnO have been investigated extensively due to their chemical stability and efficient photo catalytic properties. In this paper, Zn-doped TiO_2 nanoparticles with high photo catalytic activity were synthesized by sol-gel method. The sol-gel method is a versatile process used for synthesizing various oxide materials because it allows very simple control of particle size and the experimental process [11]. The major advantages of this method are high purity of precursors and homogeneity of the sol-gel products with a high purity of physical, chemical, and surface morphological properties (Pierre et al.1998). The precursors are used for obtaining composites with molar ratio of 9:1 (TiO_2: ZnO).The reactions were performed in a regular glass beaker at a controlled heating. For the preparation of nano powder, one gram zinc acetate dehydrates was dissolved in 5 ml absolute ethanol and fed into 75 ml benzyl alcohol. Thus, to a beaker containing the obtained mixture of 4 ml $TiCl_4$ was slowly added under constant stirring at room temperature. Initially, the reaction mixture was coloured in orange-red and contained some white fluffy precipitates that are completely dissolved on mixing. The container was covered with a Petri dish and the reaction mixture was stirred continuously at 100°C for 10 hours. Thus obtained materials were left at 30°C for 10 days. The resulting white thick suspensions were centrifuged at 5000 rpm for 30 minutes. The white precipitates were then washed two times with absolute ethanol and diethyl ether .After every step of washing, the solvent was separated by centrifugation. The collected materials were dried in air overnight and then grinded into a fine powder. The obtained powders were annealed at 600°C for 5 hours. The combination of these methods fosters the generation of a stable structure between the TiO_2 and ZnO. For most of the photo catalytic decomposition processes, photonic efficiency is relatively low, and furthermore, photo catalytic reactions on TiO_2 nanoparticles can usually be induced only by ultraviolet light, which limits the application of TiO_2 as a photo catalyst with visible light [12-14]

2.3. Substrate Cleaning

Coated glass with highly F-doped Transparent Conducting Oxide (TCO) usually serves as a support for the dye-sensitized oxide. It allows lightly transmission while providing good conductivity for current collection. The Substrates are first dipped into acetone with ultrasonic bath for 20 minutes to dissolved unwanted organic materials and to remove dust and contamination material that are left on the substrates post manufacture. Another 20 minutes of ultrasonic bath in methanol is followed in order to remove the acetone and materials that are not cleansed or dissolved by acetone. Finally, a 30 minutes ultrasonic bath in isopropanol was needed to further remove the residual particles on the substrates.

2.4. ZnO/TiO_2 Photoanode on FTO Glass Plate

It is very important to work with a fingerprint free F-doped Transparent Conducting Oxide (TCO), always gloves were used and FTO was cleaned with alcohol prior to use FTO was heated to 50°C at the beginning of the process to increase the adhesion and the scotch 3M adhesive tapes were applied on the edge of the conductive side of the FTO glass plate. The reason for applying tapes was preparing a mould such that nonsintered ZnO doped TiO_2 has always same area and thickness for all samples. A certain proportions of ZnO doped TiO_2 powder with ethanol, acetyl acetone; polyethylene glycol and triton(X-100) were mixing for 30 minutes in agate mortar. Then ZnO doped TiO_2 colloidal was dropped on the conductive side of the FTO after the conductive side of the FTO was checked by the multimeter. Then, the ZnO doped TiO_2 paste was uniformly distributed over the FTO by Doctor Blade method. Doctor blade means a film smoothing method using any steel, rubber, plastic, or other type of blade used to apply or remove a liquid substance from another surface. The term "doctor blade" is derived from the name of a blade used in conjunction with the doctor roll on the letter press. The term "doctor blade" eventually mutates into the term "doctor blade" [15]

2.5 Heat Treatment for Photo anode

The scotch 3M adhesive tapes were removed from the ZnO doped TiO_2 coated FTO glass plate and plates were sintered at 450°C for 30 min in air. The colour of ZnO doped TiO_2 becomes brown in the middle of the sintering process and then its colour changes to the brownish-white. This colour remained till the end of the sintering process. This is to ensure that the polymer or macromolecules in ZnO doped TiO_2 colloid such as acetyl acetone can be removed, leaving tiny holes in nano layers, resulting in better dye absorption and better contact between ZnO doped TiO_2 particles. In consequence, it optimizes the chances of electrons being excited by the photons and increases the amount of excited electrons entering into the ZnO doped TiO_2 conduction band.

2.6. Preparation of dye sensitizer solutions

The various parts of fresh curcuma longa were collected from Uthangarai taluk, Krishnagiri district. The skin surface of curcuma was cleaned carefully as shown fig 1(a).

A

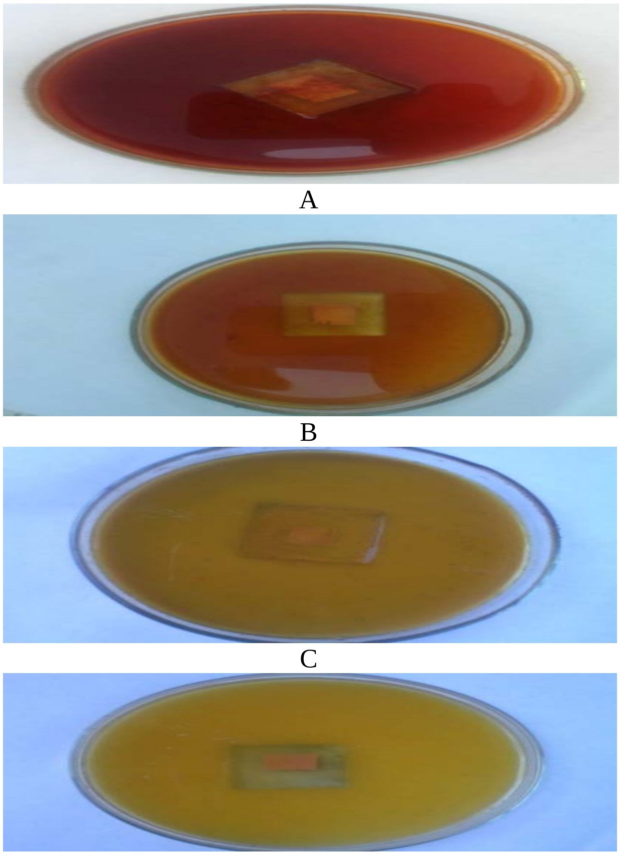
B

C

D

Figure 1: (a) Photographs of samples A, B, C, & D (A- parent round turmeric, B-parent finger turmeric, C-daughter round turmeric, and D- daughter finger turmeric).

Cleaned samples are placed separately in polythene bags. They were dried in a clean shade environment with cover to avoid the contamination for 20 days and then they are fine powdered by using mortar**.** Approximately 0.30 g of the sample was dissolved in 50 ml of acetic acid. Then after the pure solution were filtered out from solid residues by extraction process. Further purification of the extract was avoided to achieve efficient sensitization using simple extraction procedures. The extractants were properly stored, protected from sunlight and used further as sensitizers in DSSCs [16, 17].The photograph of dye dipped FTO glass plate as shown in fig 1(b)



A

B

C

D

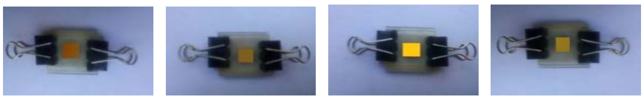
Figure 1(b) Photograph of dye dipped FTO glass (A- parent round turmeric, B-parent finger turmeric and C- daughter round turmeric, and D- daughter finger turmeric).

2.7. Preparation of Counter Electrode

The platinization procedure given by solaronix was applied because the material was taken from solaronix [18]. Actually, this method is simply called thermal decomposition which is most widely used platinization procedure. Plastitol was applied on the surface by using a brush. All FTO glasses were sintered at 450°C for 15 minutes for decomposition which was the minimum required calcinations condition according to the procedure [19]

2.8. Nano crystalline Dye Sensitized Solar Cell Assembly

Sensitized ZnO doped TiO_2 photo-anode and the counter electrode was stacked together face to face and the liquid electrolyte, Idolyte TG 50 solution drop penetrated into the working space and counter electrode via capillary action. The two electrodes were held with binder clips are shown in figure 1(c) [20].



A **B** **C** **D**

Figure 1(c): Schematic diagram of dye sensitized solar cells (A- parent round turmeric, B-parent finger turmeric and C- daughter round turmeric, and D- daughter finger turmeric).

3. Results and Discussion

3.1 UV-Visible spectroscopy

The UV-Visible spectrum of pure TiO_2 and ZnO doped TiO_2 nanopowder as shown in figure 2(a) & 2(b). The UV -Visible spectra of the ZnO-TiO_2 nanoparticles were obtained to determine the relation between the solar energy conversion efficiency and spectroscopy properties. In case of pure TiO_2 nanoparticles it exhibits the shorter wavelength in the UV region but light absorption by the sensitized ZnO doped TiO_2 nanoparticles shift from UV region to visible region. It was evident that the absorption of the ZnO doped TiO_2 nanoparticle exhibited the largest red shift.

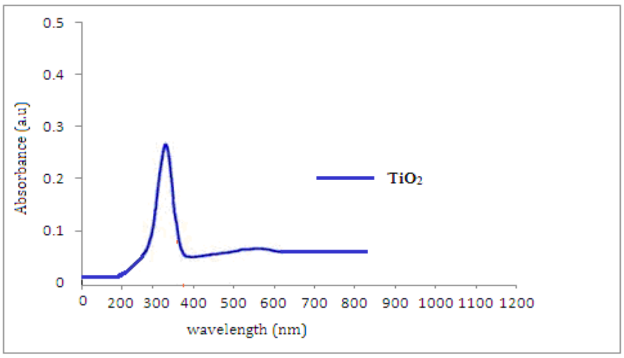


Figure 2: (a) UV –Visible absorbance spectrum of pure TiO_2

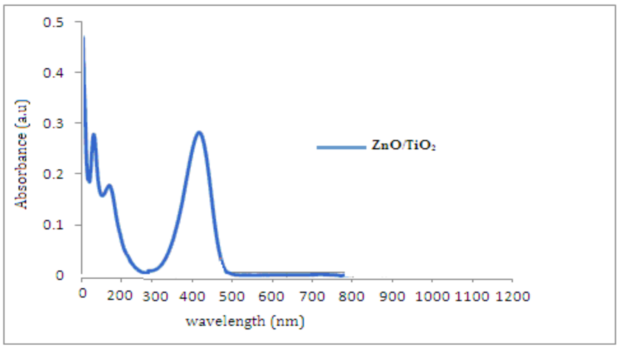


Figure 2: (b) UV –Visible absorbance spectrum of doped TiO_2

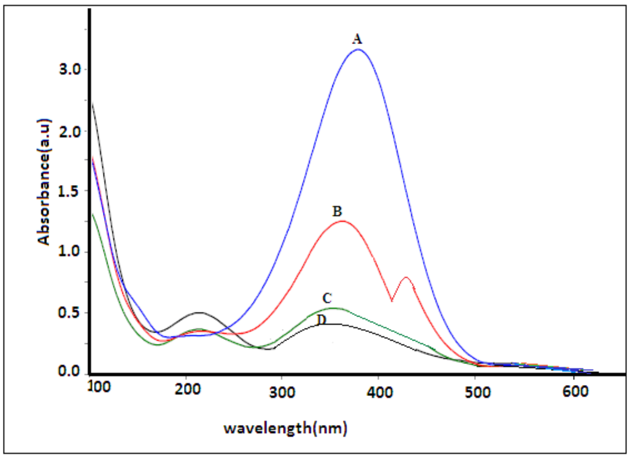


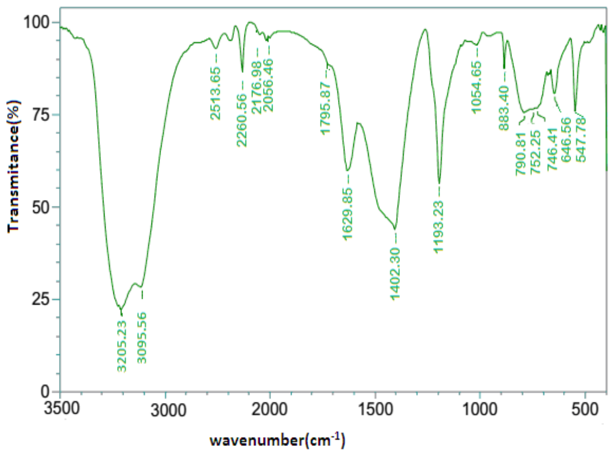
Figure 2: (c)

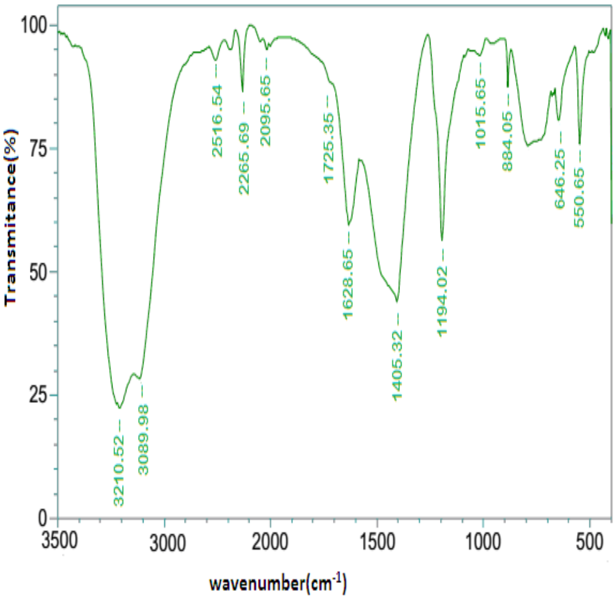
Figure 2 (c): UV –Visible absorbance spectrum of pure curcuma longa dye extract (SAMPLES A, B, C &D)

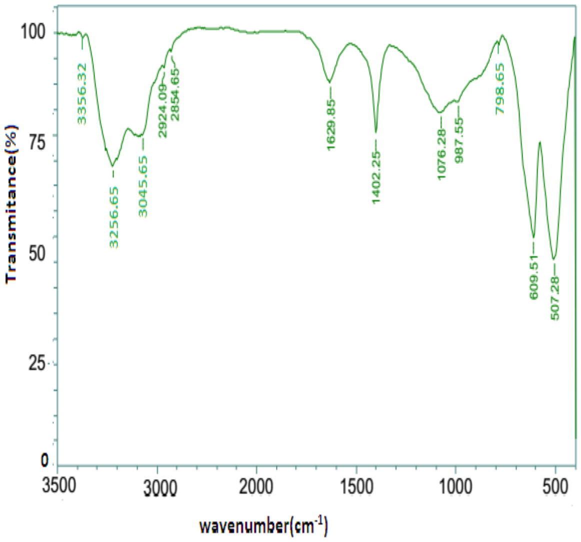
The fundamental absorption which corresponds to electron excitation from the valance band to conduction band can be used to determine the value of the optical band gap. The cut-off wavelength of pure TiO_2 and ZnO doped TiO_2 are 385.71 nm and 471.42 nm respectively and its corresponding optical band gap values are found to be 3.220 eV and 2.634 eV. The optical absorption measurement of the natural dye extracted was determined in the wavelength range 100-600 nm using UV -Visible spectrometer shown in figure 2(c). The broad absorption and sharp intense peak observed at the region of 300 nm to 500 nm [21]. From the UV -Visible spectra of sample A obtains high absorbance and longer wavelength while comparing to other samples.

3.2 Fourier Transform Infrared (FT-IR) Analysis

The Fourier Transform Infrared (FTIR) spectra of various parts of curcuma longa dye extracts were recorded on Perkin Elmer spectrometer using KBr pellet technique in the range of 3500-500 cm^{-1}.The recorded FTIR spectra of various parts of curcuma longa dye extract as shown in figure 3 (a), (b), (c) and (d). The different bands and functional groups observed at different wavelengths, an infrared spectrum is used to determine the structure of organic molecules. The area from 3500 cm^{-1} to 1300 cm^{-1} is called the functional group region. The bands in this region are particularly useful in determining the type of functional groups present in the molecule. In the high frequency region sharp intense peaks observed at 3095, 3089, 3045 and 3056 cm^{-1} are attributed to C-H stretching vibrational modes of alkanes in the compound type hydrocarbons presence of sample A, B, C and D respectively. The O-H stretching of Curcuma is observed at 3205, 3210, 3356 and 3256 cm^{-1} in the compound type of alcohols and phenols due to its hydrogen bonding interaction with the neighboring oxygen these are present in A, B &C samples. The O-H stretching is observed at 2513 and 2516 cm^{-1} in the compound type carboxylic acids present in only sample A & B. The sharp intense peaks observed at 2260 $cm^{-1}$are attributed to C≡C stretching vibrational modes of Alkynes in the compound type hydrocarbons presence of sample A only.







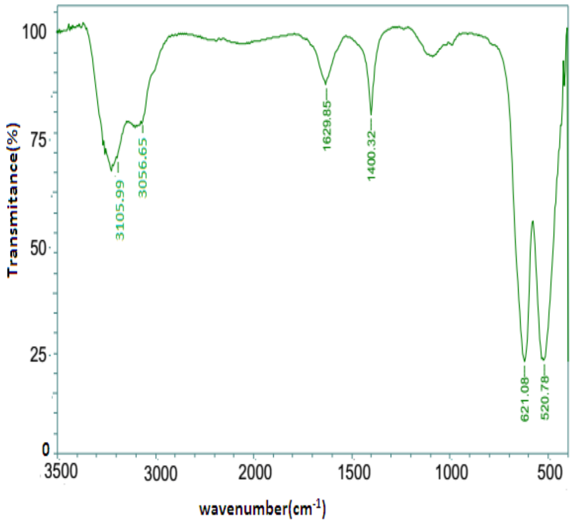


Figure 3: (a, b, c &d) FTIR spectral analysis of curcuma longa dye extracts for samples A, B, C & D

A sharp peak at 2176cm^{-1} is probably C≡C stretching for alkenes in hydrocarbons which are present in sample A. C=C stretching for this alkenes appears at 2056cm^{-1} which is also present sample A only. A sharp peak at 2265 and 2095 cm^{-1} is probably N-H bending for amino acids in hydrocarbons, these are present in sample B only. A strong peak at 2924, 2854 cm^{-1} is probably O-H stretching at carboxylic acid which is also present in C samples. The C-O stretching is observed at 1054, 1193, 1015, 1194 and 1076 cm^{-1} in the compound type of alcohols present in both samples. The O-H bending is observed at 1402, 1405 and 1400 cm^{-1} in the compound type of alcohols present in both samples. The N-H bending for these amides appears at 1628cm^{-1} and a sharp peak at 1725 cm^{-1} is probably C=O stretching for ketons which is present in C sample only. Many of these bands of due to hydrocarbon with the C-H out-of-plane bending of the alkenes at 883, 790, 746, 884, 987 and 798 cm^{-1} which are present in A, B and C samples. The C=O stretching is observed at 646, 609, and 621 cm^{-1} in the compound type carboxylic acids present in both samples. The C-X stretching is observed at 547, 550, 507 and 520 cm^{-1} in the compound type of organic halogen present in both samples [22]

3.3 Photo Luminescent (PL) studies

An investigation on the photo luminescent (PL) studies of pure TiO_2, ZnO doped TiO_2 and natural dye extracted as shown in figure 4 (a) ,(b) and (c) respectively. It can provide valuable information on the quality and purity of the material technologies. Typical photo luminescent (PL) emission spectrum for zinc oxide doped TiO_2 exhibits a sharp emission peak in the near UV region and a broad band in the visible region. A strong peak seen at 490 nm corresponds to the near band edge (NBE) of the doped TiO_2 material. The intensity of the NBE emission of TiO_2 films is dependent on the microcrystalline structure and stoichiometry. The intensity of the NBE peak of ZnO doped TiO_2 level higher than that of pure TiO_2. A pure TiO_2 gives slightly broad and maximum emission peak appeared at around 380 nm and its band gap value is around 3.26 eV. The peak at 381.5nm corresponds to the band edge emission and its band gap value is 3.259 eV. A ZnO doped TiO_2 gives broad and maximum emission peak appeared at around 490 nm and its band gap value is around 2.58 eV.

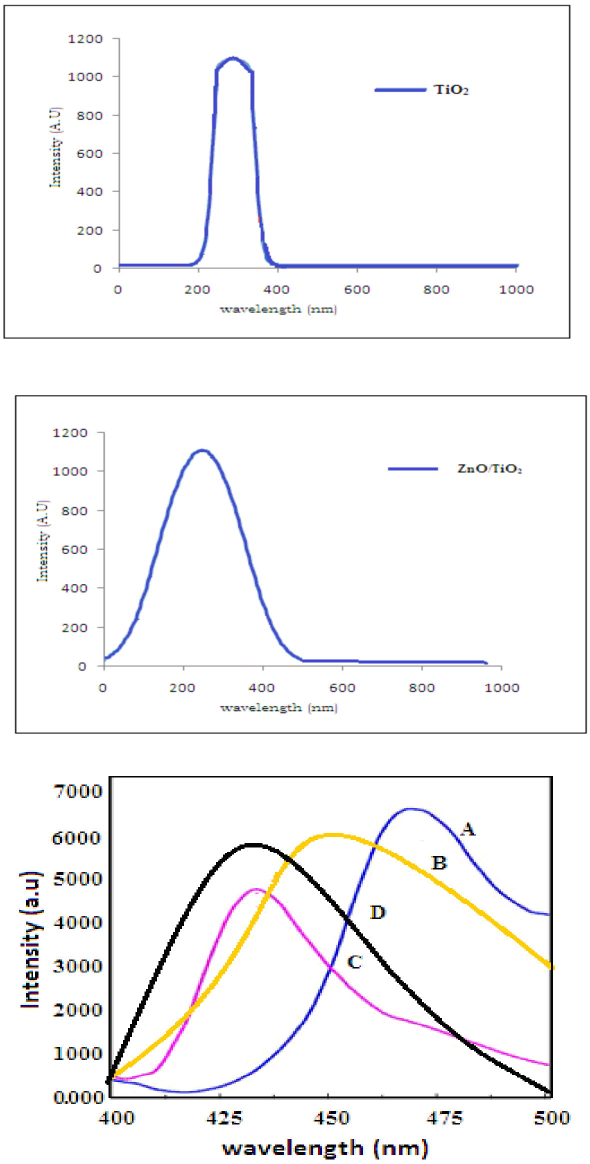


Figure 4 (a, b & c): PL study of pure TiO_2 , ZnO doped TiO_2 & after dye dipped FTO glass coated with ZnO doped TiO_2 nanopaste

The peak at 482 nm corresponds to the band edge emission and its band gap value is 2.576 eV. The peak at 480nm is due to artifact. The peak at 505 nm arises from the oxygen vacancy of ZnO materials because of recombination of a Photo generated hole in valence band with an electron in conduction band.The PL measurement of the natural dye extracted was determined in the wavelength range 400-500 nm. The broad absorption and sharp intense peak observed at in the region of 425 nm to 475 nm. In sample A obtained at high absorbance at longer wavelength compare to other samples. In the present study when increase the peak intensity of the film the second harmonic spectra were obtain as intensity also become as minimum and vice versa. From the PL spectra, it is concluded that the ZnO doped TiO_2 has better quality with lesser defect density which make the film a promising candidate for UV-Visible optical devices.

3.4 Field Emission Scanning Electron Microscopy (FE-SEM) Analysis

The morphologies of the pure and ZnO doped TiO_2 nanoparticles were studied by using FE-SEM analysis and micrographs are shown in figure 5 (a) & (b).It shows the synthesized TiO_2 nanoparticles have aggregated with spherical shape with smooth surface compare pure TiO_2. The surface smoothness can be attributed the particles are formed by uniform arrangement of the lattice points. The average particle size of pure and synthesized nanoparticle is measured as 90 nm and 40 nm. It shows the size of the synthesized particles are decreased compared to pure TiO_2.The morphology of the pure TiO_2 powder is loosely dispersed, where as for the ZnO doped TiO_2 particles are arranged uniformly. From the FE-SEM images it is revealed that the zinc oxide doped TiO_2 nanopaste coated on conducting side of FTO glass after dye dipped dye solution as shown in figure 5 (c), (d), (e) and (f). The film deposited from the sample A has larger grain size rather than of other sample due to its higher grain size it exhibits lesser receptivity and also higher transparency which are important for TCO applications. It is clear that the prepared sample A has regular spherical shape and uniform size, good packing density compare to other samples.

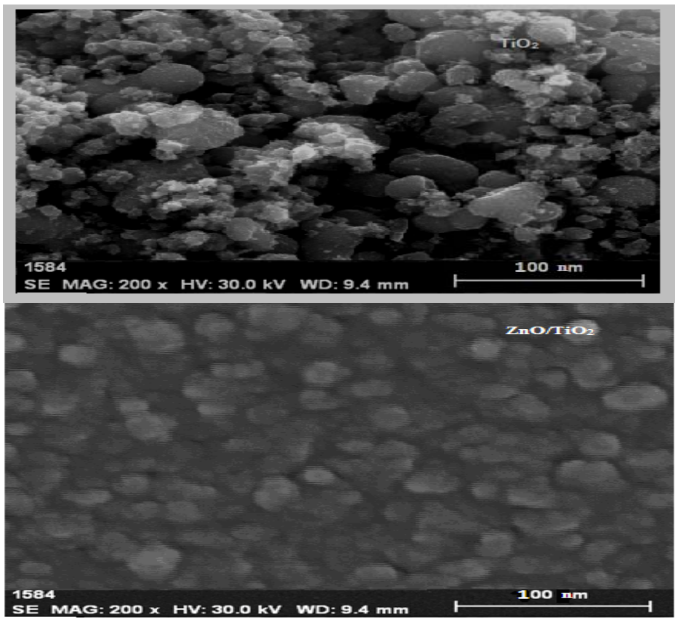
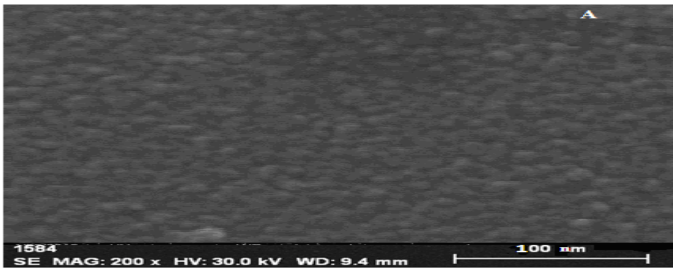


Figure 5: (a) FE-SEM image of pure TiO_2 & ZnO doped TiO_2



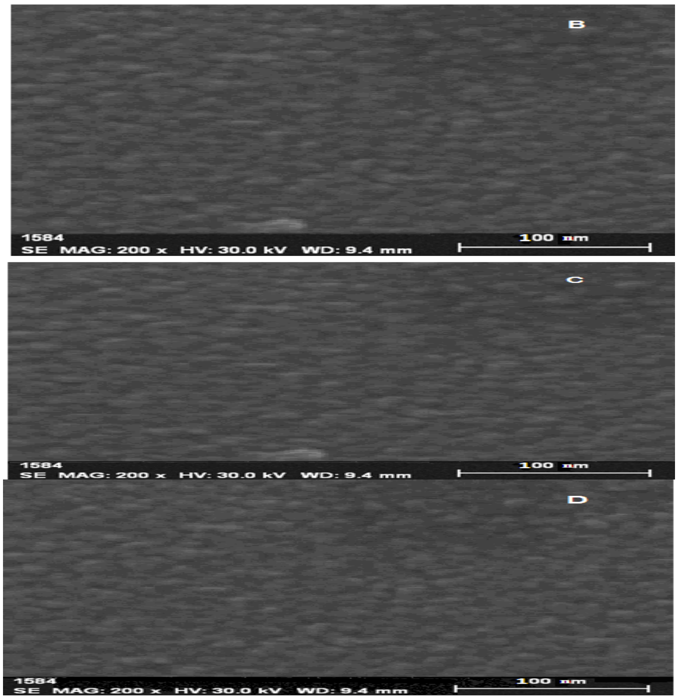
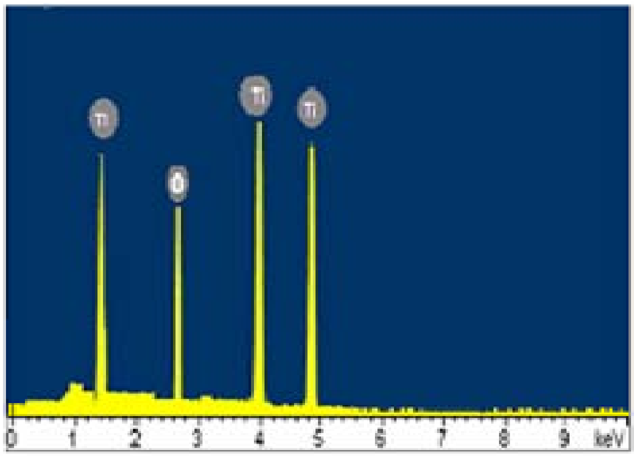


Figure 5: (c, d, e &f): FE-SEM images of after dye dipped FTO glass coated with ZnO doped TiO_2 nanopaste

3.5 EDS Analysis

The semi quantification of elemental analyses to identify the weight percentage of major and minor elements present in the samples were done using Energy Dispersive X-ray Spectrometer (EDS), JEOL model, JSD-5610 LV with an accelerating voltage of 20 KV. The result of energy dispersive X-ray spectroscopic (EDS) analysis of pure TiO_2 and ZnO doped TiO_2 nanopowders are shown in fig 6 (a&b).

Trace elements are estimated by determining the percentage abundance of elements such as Ti, O & Zn present in the sample. In pure TiO_2 the concentration of titanium is 89.20% and the concentration of oxygen is 10.80% this result gives to the titanium and oxygen presents in sample.



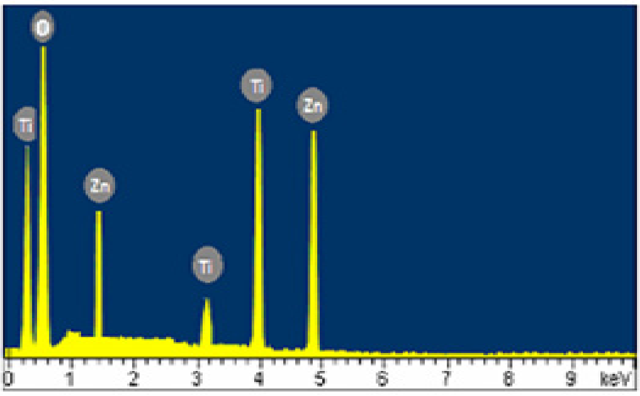


Figure 6: (a & b) EDS analysis of pure & doped TiO_2

The ZnO doped TiO_2 nanopowder as the concentration of titanium is 85.50%, the concentration of oxygen is 11.60% and the concentration of Zn is 2.81%. It shows that ZnO is present in the evidence of ZnO doped TiO_2 nano powder.

3.6. Photocurrent-Voltage Characteristic

Photovoltaic measurements were carried out using a halogen light source that was focused to one sun at Air Mass (AM) 1.5, at the surface of the cells. The spectral output of the lamp was matched in the region 350 and 800nm with the aid of a Schott KG-5 sunlight filter so as to reduce the mismatch between the stimulated and the true solar spectrum to less than 2%. The current-voltage characteristics of the cells were determined by biasing the cells externally and measured the photocurrents is shown in figure 7. The overall photo conversion efficiency (η) is calculated from the integral photocurrent density (J_{sc}), the open circuit photocurrent (V_{oc}), the fill factor (FF) of the cell, and the intensity of the incident light (P_{in}). From the figure 7 & table 1 the conversion efficiencies of DSSCs fabricated using curcuma extracted from the different parts of curcuma longa treated with acetic acid. The open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (FF) and conversion efficiency (η) of the Sample-A was, V_{oc}=0.88 V, J_{sc}=1.29 mA/cm^2, FF=0.7737 and (η) is 0.8784 %, the Sample-B was V_{oc}=0.87 V, J_{sc}=1.05 mA/cm^2, FF=0.7088, and (η) is 0.6475 %, the Sample-C was V_{oc}=0.85 V, J_{sc}=0.58 mA/cm^2, FF=0.7091 and (η) is 0.3496 %, and the Sample-D was V_{oc}=0.83 V, J_{sc}=0.26 mA/cm^2, FF=0.7590 and (η) is 0.1638 % respectively.

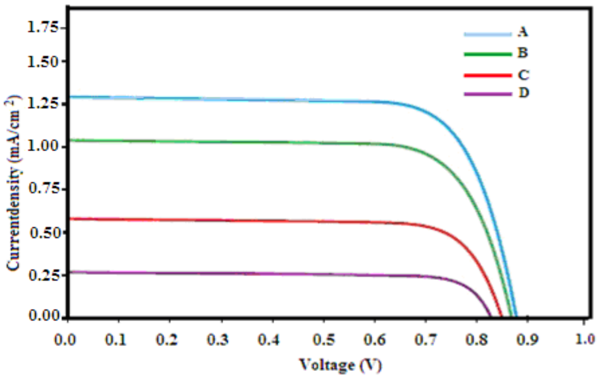


Figure 7: Photocurrent-voltage characterization of various parts of curcuma longa dye extracts (samples -A, B, C & D)

The energy conversion efficiency (η) of the samples A, B, C and D are 0.8784 %, 0.6475 %, 0.3496 %, and 0.1638 % respectively. In the present study, sample A has higher conversion efficiency compared to the other samples

Table.1: Photovoltaic parameters of various parts of curcuma longa dye extract

sample	V_{max} (V)	J_{max} (mA/cm^2)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (-)	η (%)
A	0.88	1.29	0.72	1.22	0.7737	0.8784
B	0.87	1.05	0.74	0.875	0.7088	0.6475
C	0.85	0.58	0.76	0.46	0.7091	0.3496
D	0.83	0.26	0.78	0.21	0.7590	0.1638

3.7 Conclusion and Future Work

In the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes. Nature has gifted us more than 500 dye-yielding plant species. The ZnO doped TiO_2 nanopowders were prepared by using sol-gel method to enhance the solar energy conversion efficiency. The optical absorption spectrum reveals that the absorbance is more than one unit between 300 and 1100 nm .The light absorption by the sensitized ZnO doped TiO_2 nanoparticles shift from UV region to visible region. FTIR study confirms the presence of all functional groups. In PL study of the pure and ZnO doped TiO_2 gives the broad and maximum emission peak appeared at 381.5 and 490 nm and corresponding band gap values are 3.259 and 2.58 eV. The FESEM images confirms that the morphology of film is loosely dispersed in pure TiO_2 , smooth and well defined spherical shape with grain size 20-50 nm of ZnO doped TiO_2. Also analyses the morphology of coated glass plate then conclude the sample A has regular arrangement, spherical shape, uniform size, and good packing density. The titanium, oxygen and zinc are identified from EDX spectrum, in which the spectrum confirms the elemental compositions of presented samples. In the Photovoltaic measurements were carried out to determine the open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (FF) and overall energy conversion efficiency (η) of the samples A, B, C & D. The round parent turmeric (Sample-A) showed the excellent solar energy conversion efficiency compared to that of other sample. In future various metal oxide doped TiO_2 nanopastes are prepare via sol gel method then compare overall conversion efficiency of various parts of curcuma longa dye extract.

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