

PREPARATION OF TiO₂ PHOTO ELECTRODE FOR DYE- SENSITIZED SOLAR CELLS APPLICATION

Me Me Aung¹, Htay Htay Win², Thin Thin Kyu ³, Zin Min Myat⁴

Abstract

Green synthesis of TiO₂ nanoparticles was produced by Aloe Vera leaf. The structural properties of TiO₂ nanoparticles were characterized by X- ray diffraction (XRD). The chemical properties of TiO₂ nanoparticles were analyzed by Fourier Transform Infrared Spectroscopy (FTIR). The surface morphology and optical properties of TiO₂ nanoparticles were characterized by Scanning Electron Microscopy (SEM) and Ultraviolet Spectroscopy (UV-Vis) measurements. According to experiment result, the extraction of Aloe Vera leaf dyne solution was performed and employed as natural dyne sensitizer in DSSC.

Keywords: TiO₂ Nanoparticles, Aloe Vera leaf Green Synthesis, XRD, FTIR, FESEM, UV-Vis.

Introduction

Nanoparticles (metal and metal oxides) of various types have been widely employed via physical and chemical methods. Nanoparticles are generally defined as particulate matter with at least one dimension that is less than 100 nm. Subgroups are examples of environmentally relevant types: inorganic nanoparticles and organic nanoparticles.

Titanium dioxide is solid inorganic substance, which is a white color metal oxide. TiO₂ is poorly soluble, non-flammable, thermally stable and not classified as hazardous according to the United Nations (UN) Globally Harmonized System (GHS) of Classification and Labeling of Chemicals.[Aadarsh Mishra, 2014] TiO₂ is formed by elements Titanium of atomic number 22 from IV B group and Oxygen of atomic number 8 from VI A group. It can exhibit three different phases in nano range at the different temperature, such as Anatase, Rutile and Brookite. Among these phases, Anatase has been proved to have extraordinary chemical and physical properties for environmental remediation.[Rajneesh Mohan, et al,2013] It is also having high quality properties, such as hydrophobicity, non-wettability and large band gap. Hence, it is used in the various industrial applications: dye sensitized solar cell, photo catalysis, self-cleaning, charge spreading devices, chemical sensors, microelectronics, electrochemistry, antibacterial products and textiles.[Saowaluk Boonyod, et al,(2011)] Different types of approaches are available for synthesis of titanium dioxide nano particles, those are solution Combustion, Sol-Gel, Hydrothermal, Solvothermal, Microwave Assisted, co-precipitation, Chemical Vapor Deposition and Green synthesis. The green synthesis process is eco-friendly technique due to use of extracts of plant (leave, flower, seed and peels), bacteria, fungi and enzymes for synthesis of titanium dioxide nanoparticles instead of large quantity of chemicals.[Vijaylaxmee Mishra, et al,(2014)] Green synthesis provides more advantages over physical methods and chemical methods because it is very cost effective, easy process and scalable for large scale production. This method not required high temperatures, high pressure, costly equipment and hazards chemicals.

¹ Dr, Assistant Lecturer, Department of Physics, University of Yangon

² Lecturer, Department of Physics, University of Yangon

³ Assistant Lecturer, Department of Physics, University of Yangon

⁴ Assistant Lecturer, Department of Physics, University of Yangon

The present work green synthesis of TiO_2 nanoparticles is based on the Aloe Vera plant extract. Aloe Vera is oldest medicinal plant ever known and a most applied medicinal plant, which is succulent plant species, the species is frequently cited as being used in herbal medicine since the beginning of the first century AD. It is a stem less plant growing to 60-100 cm (24-39 in) tall and the leaves are thick and fleshy, green to grey-green, with some varieties showing white flecks on the upper and lower stem surfaces.[Yuvasree. P., etal, 2013]The role of some inorganic elements like vanadium, zinc, sodium, potassium, magnesium, aluminum, iron, nickel, cadmium, copper, cobalt and manganese in the improvement of impaired glucose tolerance and their indirect role in the management of diabetes mellitus, hypoglycemic, wound healing and anti-inflammatory effects are being used, which contain both organic and inorganic constituents.

Experimental Procedures

Preparation of TiO_2 Nanoparticles

In various leaves, Aloe Vera leaves were collected from Gyobingauk Township, Bago Division, Myanmar. The leaves of Aloe Vera were separated from plant, which were washed thoroughly with tap water for three times and cut into small pieces. The raw materials were firstly weighed by electronic balance. Took 30g of the leaves into 100 ml distilled water boiled for 3hr at 80°C . The extract was filtered using filter paper. The filtrate was stored for the synthesis of nanoparticles. To synthesis the TiO_2 nanoparticles, dissolve 1.0 g of Titanium (IV) isopropoxide (TTIP) in 100 ml of distilled water. The mixture was kept under constant stirring on a magnetic stirrer for 4 hr continuously. At that time, added leaves extract drop wise under constant stirring up to achieve pH of solution became 7. The pH was measured using a pH test paper. In this process, and then separate this nanoparticles using filter paper. Obtained materials washed with water repeatedly to remove the by products. And then, the residual parts were dried at 160°C for 12 hours. After that the powder was put in the agate mortar and it was ground for 2 hr to reduce the particle size. In order to be dried, the nanoparticles were calcined at 500°C for 4 hr. The block diagram of preparation TiO_2 nanoparticles was shown in Figure1. The photos of sample preparation of TiO_2 nanoparticles from Aloe Vera leaves extract were shown in Figure 2.

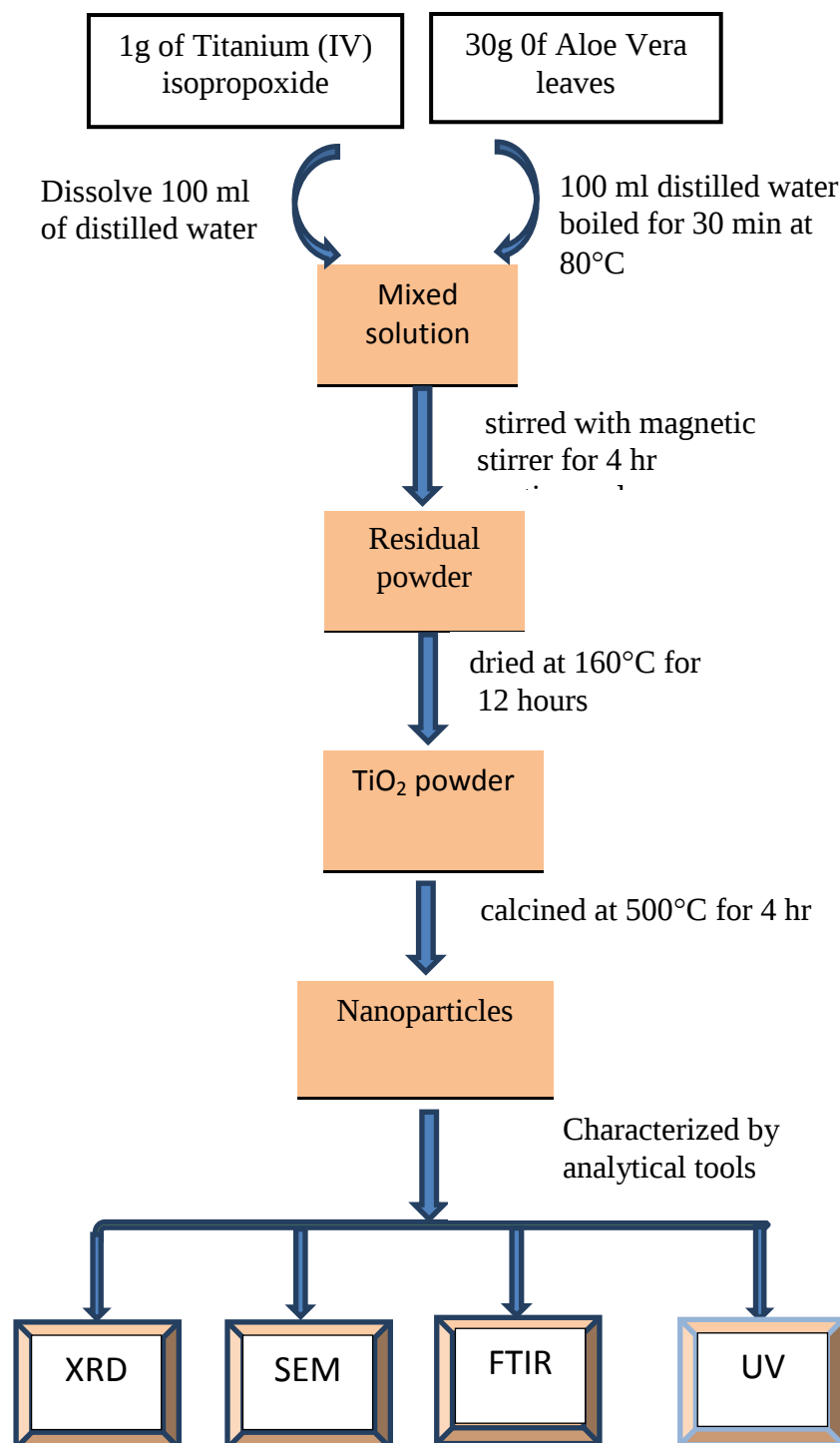


Figure 1 The block diagram of Preparation TiO_2 Nanoparticles

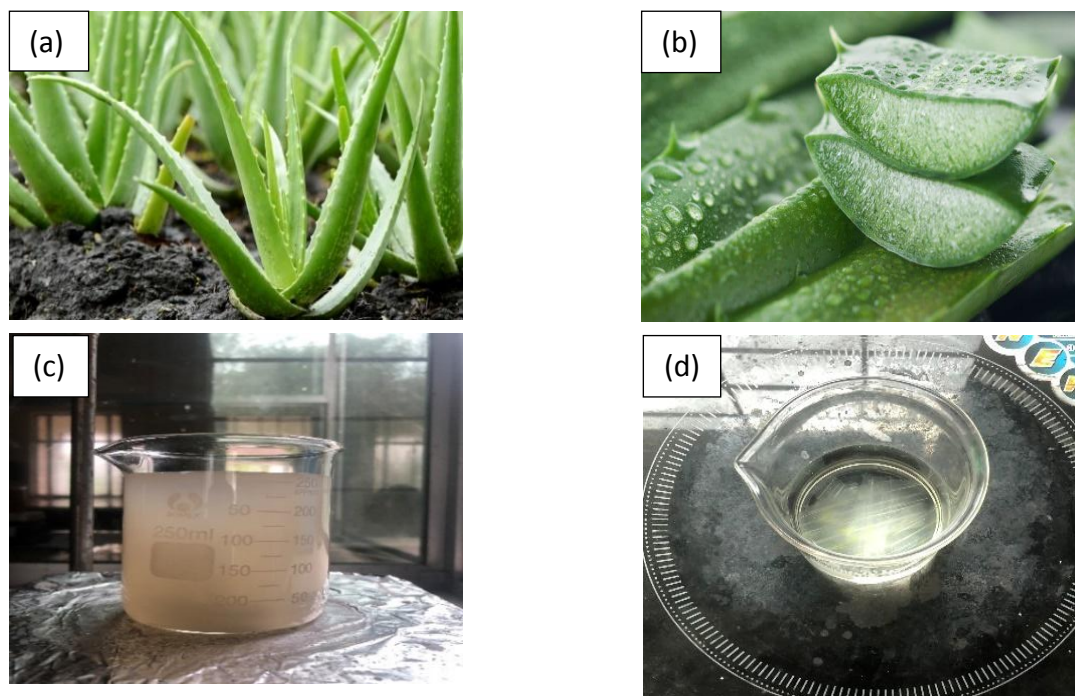


Figure 2(a) Aloe Vera plant, (b) Separated Pieces of Aloe Vera, (c) In Heating Process, (d) Juice of Leaves Extract

Characterization of TiO_2 nanoparticles

The crystal structure, lattice parameter and average crystallite size was measured by Brooker D8 X-ray diffractometer using $\text{Cu-K}\alpha$ -radiation ($\lambda=0.154 \text{ nm}$). Grain size and morphology of the nanoparticles were observed estimated by S 3400 N Scanning Electron Microscope. FTIR analysis was employed to study the functional groups present in Titanium dioxide nanoparticles by SHIMIDUZU, JAPAN. UV-visible absorption spectra of TiO_2 nanoparticles were recorded on SHIMIDUZU, JAPAN 1800 spectrophotometer.

Results and Discussion

XRD Analysis of TiO_2 Nanoparticles

The synthesized Titanium dioxide (TiO_2) nanoparticles obtained from green synthesis method at temperature 500°C is shown in Figure 3. All the peaks height and peaks position were good agreements with standard ICDD library file. On the TiO_2 XRD pattern, five peaks were clearly observed. XRD pattern of the (TiO_2) nanoparticles showed the present of anatase form which can be denoted at 2θ peaks at 25° , 38° , 48° , 55° , 62° , which are found to be (1 0 1), (1 0 3), (2 0 0), (1 0 5) and (2 1 3) respectively and confirmed the nanocrystallite nature. The (1 0 1) reflection peak becomes more intense and sharper and the crystal structure of TiO_2 was tetragonal. As the width of the peak increase size of particle size decrease, which resembles that present material in nano range. The lattice parameters were obtained $a=b=3.7857 \text{ nm}$ and $c=9.5289 \text{ nm}$. The average crystallite size was measured by Debye – Scherrer's equation as mentioned below.

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos \theta}$$

Where D is the average crystallite size of the particles, K is Debye scherrer's constant (=0.94), λ is the wavelength of the CuK α - radiation (=0.154 nm), β is the full width half maximum (FWHM) of the peak, θ is the Bragg's angle. The average crystallite size was measured as 25.74 nm using the above formula.

Table 1 The comparison of crystallite size of TiO₂ nanoparticles

International Journal Values	K.Venkateswara Rao,etal,(IJARPS), 2015 crystallite size =20 nm
	Abdul Abdul Rahuman,etal,(APJOTM) ,2014 crystallite size =32.58 nm
Observed sample value crystallite size =24.89 nm	

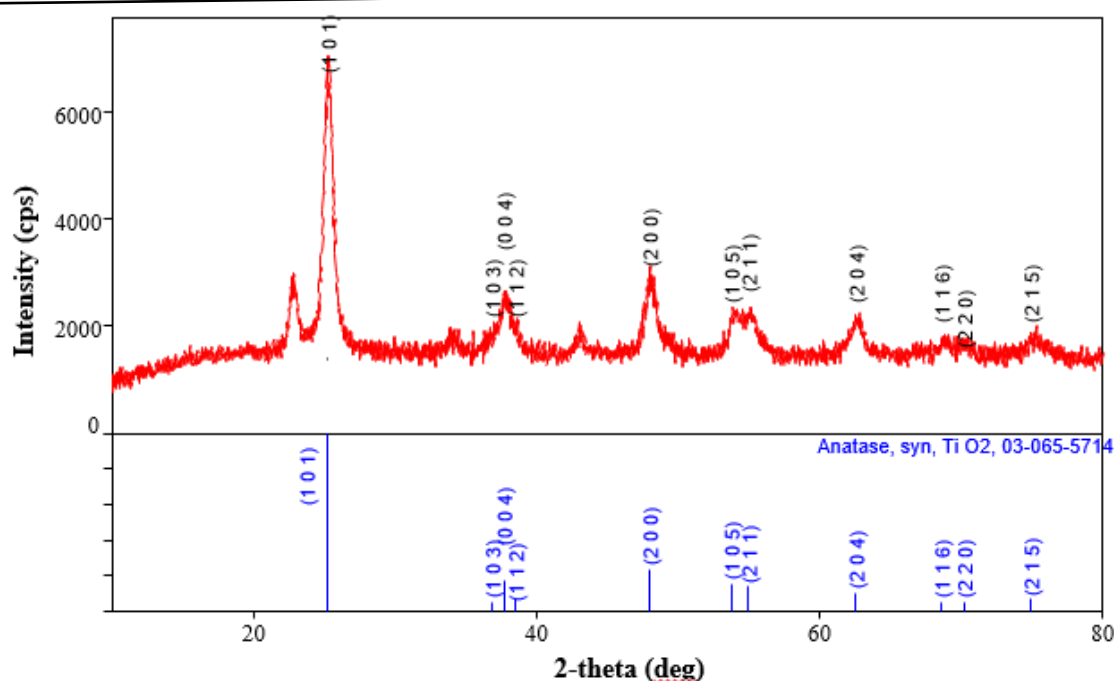


Figure 3 X – ray diffraction patterns of Synthesized TiO₂ NPs

SEM Analysis of TiO₂ Nanoparticles

The surface morphology features of synthesized TiO₂ nanoparticles were examined by Scanning Electron Microscope (SEM) shown in Figure 4. The average grain sizes were calculated by using well known bar code system with Image J software. Around the examined area, the grain size of TiO₂ was estimated to be 1.1723 μ m. In addition overall observation of TiO₂ powder indicted a good microstructure with no discontinuities in terms of microracks. This figure indicated that most of the grain size was regular structure and a few number of large grain size were found. It looks fairly dense and rough. The morphology was still observed, becoming denser and smoother. The TiO₂ nanoparticles were showing irregular particles structure.



Figure 4 FESEM image of Synthesized TiO_2 NPs

FTIR Analysis of TiO_2 Nanoparticles

FTIR spectroscopy was applied to measure the chemical properties and absorption of energy from the range of $500\text{--}4000\text{ cm}^{-1}$ by studied samples. Figure 5 represents the FTIR spectra of green synthesis derived TiO_2 . According to FTIR images in the functional group region, the outstanding wide absorption band is observed within $3500\text{--}3000\text{ cm}^{-1}$ region, corresponding to the axial OH deformation. This absorption is originated from variations of water present in samples. The samples present signal at 3438.70 and 1640.26 cm^{-1} in the spectra are due to the stretching and bending vibration of the --OH group. In the spectrum of pure TiO_2 , the peaks at 491.88 cm^{-1} show stretching vibration of Ti-O and peaks at 1425.44 cm^{-1} shows stretching vibrations of Ti-O-Ti . Peaks at 3194.23 cm^{-1} indicates the presence of amines, Peaks at 3294.53 cm^{-1} indicates the presence of Alkynes, Peaks at 1616.40 cm^{-1} indicates the presence of pyridines.

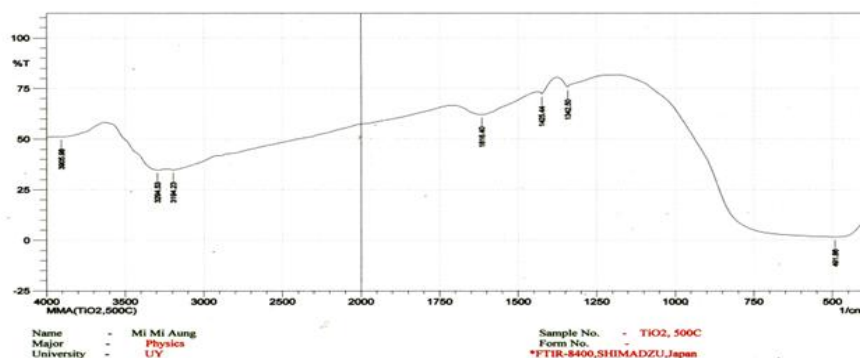


Figure 5 FTIR Analysis image of Synthesized TiO_2 NPs

UV-Vis Measurement of TiO_2 Nanoparticles

The optical band gap of the TiO_2 nanoparticles were examined using UV-Vis spectroscopy. The absorption spectrum of TiO_2 nanoparticles in the UV-Vis spectrum region ranging from 200 nm to 800 nm was shown in Figure 5. The optical bandgap is obtained by Tauc's equation, $\alpha h\nu = A (h\nu - E_g)^n$. where, A is constant, $h\nu$ is photon energy, E_g is the allowed energy band gap, n is $\frac{1}{2}$ for allowed direct transition and n is 2 for indirect transition. The extrapolation of the plot of $(\alpha h\nu)^2$ on the Y-axis as a function of $(h\nu)$ on the X-axis were provided a good estimate of the band gap of the material under investigation was shown in Figure

6. According to the absorption spectra, the energy band gap E_g for sample is 3.31 eV. The energy band gap of the standard sample is 3.2 eV and the energy band gap of prepare sample is similar to that of standard sample. It was found that the energy band gap is 3.2 eV in other research paper ("Green Synthesis of TiO_2 Nanoparticles Using Aloe Vera Extract", K. Ganapathi Rao, January 2015).

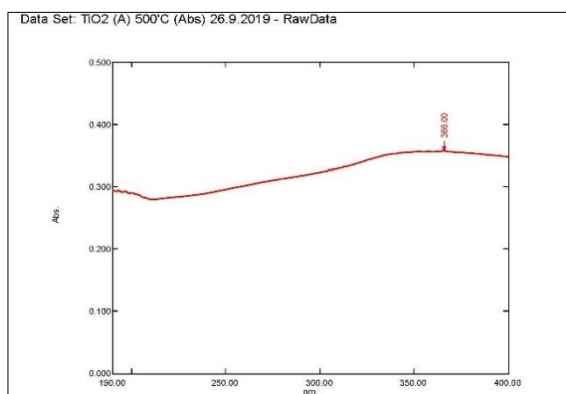


Figure 5 UV-Vis analysis of Synthesized TiO_2 NPs

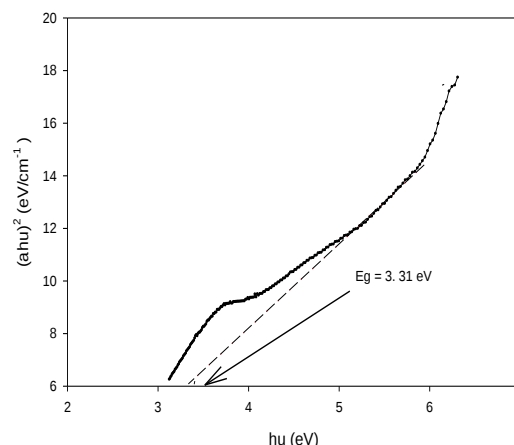


Figure 6 UV-Vis analysis of Synthesized TiO_2 NPs

Conclusions

In the research work, TiO_2 nanoparticles were successfully synthesized using green synthesis method. Then the prepared nanoparticles were characterized by modern analytical tools such as XRD, SEM, FTIR and UV-Vis. From XRD analysis average crystallite size of the sample was obtained 25.74 nm. It was observed that the tetragonal structure was formed. The tetragonal irregular particles structure was observed in SEM image. The average particle size was calculated 1.1723 μm with Image J software. The FTIR analysis confirmed the chemical composition of TiO_2 nanoparticles using Aloe Vera leaves extract corresponding to -OH group. The optical properties were studied by UV-Visible Spectroscopy it infers that the wavelength was 366 nm and energy band gap was 3.31 eV. These above results showed that as prepared TiO_2 particles were in the nano range. According to the experimental result, synthesized TiO_2 nanoparticles can use in potential application as (DSSC).

Acknowledgements

This research was totally implemented at Department of Physics, University of Yangon in Myanmar (2018-2019).

References

- Aadarsh Mishra, (2014), "Analysis Of Titanium Dioxide And Its Application In Industry", *Int. J. Mech.Eng. & Rob. Res.*, 3(3), 7pages.
- Rajneesh Mohan, etal,(2013), "Water-dispersible TiO₂ nanoparticles via a biphasic solvothermal reaction method", *Nanoscale Research Letters*, 8(1),4 pages.
- Saowaluk Boonyod,etal, (2011) , "Antibacterial Activity of TiO₂and Fe³⁺ doped TiO₂ Nanoparticles Synthesized at Low Temperature", *Advanced Materials Research*, 214, 197.
- Vijaylaxmee Mishra, etal, (2014) "A Review on Green Synthesis of Nanoparticles and Evaluation of Antimicrobial Activity",*International Journal of Green and Herbal Chemistry*, 3(1), 081.
- Yuvasree. P., etal, (2013)," Biosynthesis of Silver Nanoparticles from Aloe vera Plant Extract and its Antimicrobial Activity Against Multidrug Resistant Pathogens", *Proceedings of the International Conference on Advanced Nanomaterials &Emerging Engineering Technologies*, Pp 84-86.