

Study on Temperature Effect Structural and Optical Properties of Green Synthesized ZnO Nanoparticles

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Abstract— In this research, we studied the structural and the optical properties of zinc oxide thin films depend on temperature effect. Zinc oxide nanoparticles were synthesized by green method using *Phyllanthus Emblica* stem extract which was used as reducing and capping agent. The synthesized ZnO powders were fabricated films on glass substrates by the spin coating technique. The crystalline structure, surface morphology and optical properties of green synthesized ZnO films were investigated by X-ray diffractometry (XRD), scanning electron microscopy (SEM) and UV-Vis spectroscopy at 300°C, 400°C, 500°C and 600°C. From XRD analyses, the fabricated ZnO films were polycrystalline nature with wurtzite hexagonal structure and crystalline size of synthesized ZnO films were increased from 45.49 nm to 51.47 nm with increasing temperature. The surface morphology of ZnO films were study with SEM technique. In the SEM results, the average grain sizes were increased range between 130 ~ 220 nm as increasing temperature. By absorbance measurement with UV-Vis spectroscopy, the optical properties of green synthesized ZnO films varied with temperatures was studied. The band gap of ZnO films decreased with increasing temperatures but 600 °C the band gap value decrease.

Keywords— Green Synthesis, SEM, UV-Vis, XRD.

I. INTRODUCTION

Nanomaterials are particles having nanoscale dimension, and nanoparticles are very small sized particles with enhanced catalytic reactivity, thermal conductivity, non-linear optical performance and chemical steadiness owing to its large surface area to volume ratio [1]. Zinc oxide is a semiconductor with wide band gap (3.37 eV), high excitation binding energy (60meV) at room temperature has unique optical and as well as excellent thermal chemical stability [2]. Zinc Oxide (ZnO) has received considerable attention because of its unique optical, semiconducting, piezoelectric, and magnetic properties. ZnO nanostructures exhibit interesting properties including high catalytic efficiency and strong adsorption ability [3]. It is a promising material for many applications in toxic gas sensors, solar cell windows, blue and ultraviolet (UV) light emitting devices, transparent conductors, surface acoustic devices, photovoltaic devices [4]. The synthetic methods include sol-gel combustion, chemical vapor deposition, sonochemical, hydrothermal, wet polymerization, solvothermal, thermal decomposition, microwave assisted, precipitation, micro-emulsion, lyophilization and laser ablation. In addition to these standard synthesis methodologies, the green synthetic method employing biological plant extracts is one of the more extensively acknowledged routine due to its several advantages, such as

require no additional chemicals, simple, environmental friendly, inexpensive and reliable method [5]. Now a days green technology has attracted a number of interests and includes an extensive variety of procedures that lessen or take away toxic materials to restore the surroundings. In this green synthesis of nanoparticles by means of plant life is currently under improvement which is very much cost effective and eco-friendly [6].

II. MATERIALS AND METHODS

A. Preparation of Aqueous *Phyllanthus Emblica* Stem Extract

Firstly the fresh stem of *Phyllanthus Emblica* was collected and washed three times with tap water and twice with distilled water to remove unwanted impurities and they were cut into small pieces. 5g of the stem was boiled with 100 ml of distilled water at 100°C for half an hour. Finally, light brown colored solution was obtained and which was cool at room temperature. After filtering, the stem extract for further used was obtained [7]. The extraction of *Phyllanthus Emblica* stem is shown in Fig. 1.

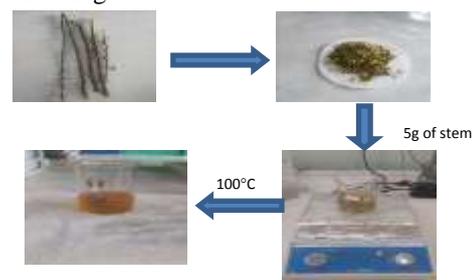


Fig. 1. Preparation of *Phyllanthus Emblica* extract

B. Synthesis of ZnO NPs by Green Method

For the synthesis of ZnO nanoparticles, 50 ml of extract was taken and boiled at 60°C. Then, 5.5g of zinc acetate was added to the solution. This mixture was stirred with magnetic stirrer until it becomes brown colored paste. This brown colored paste was calcinated at 500 °C for 3 hours, gray colored powders were obtained. Finally these gray powders were grinded with a mortar and pestle, fine powders were obtained [7]. Fig. 2 shows synthesis of ZnO NPs.

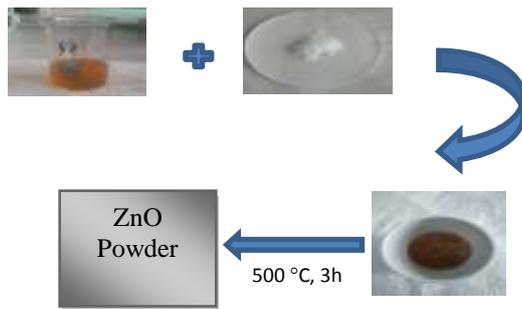


Fig. 2. Synthesis of ZnO NPs by green method

III. CHARACTERIZATION TECHNIQUE

By the spin coating technique, the synthesized ZnO powders were fabricated films on glass substrates. X-ray diffractometry (XRD), scanning electron microscopy (SEM) and UV-Vis spectroscopy were used to study the temperature effect the structural and the optical properties of the green synthesized ZnO films.

A. X-ray Diffraction (XRD) Analysis

Fig. 3(a - d) reports the XRD spectra of ZnO thin films at 300 °C, 400 °C, 500 °C and 600 °C in 2θ range 10 - 70°. All XRD profiles have 9 distinct Bragg diffractions peaks such as (100), (002), (101), (102), (110), (103), (200), (112) and (201) were observed and they were coincided with the library files. And then, no more peaks were detected that implied the synthesized materials were pure ZnO. ZnO films at different temperature were polycrystalline nature and wurtzite hexagonal structure. The crystalline size of the ZnO films were calculated by using Debye-Scherrer's formula

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

The calculated values of the crystalline size at different temperatures were listed in Table I. The crystalline sizes were increased in increasing temperature.

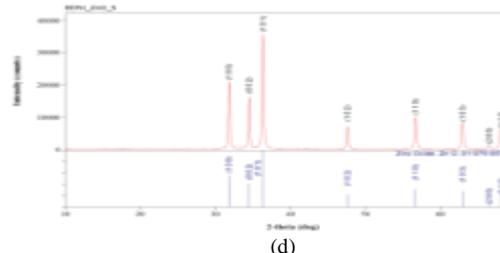
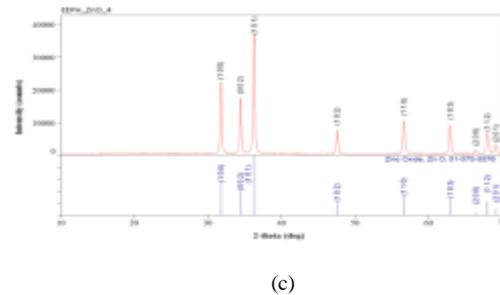
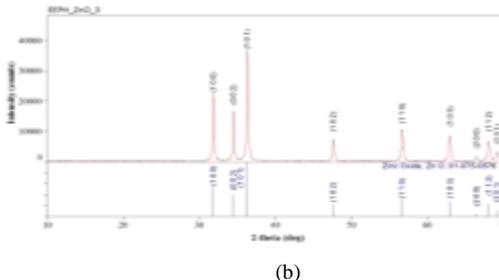
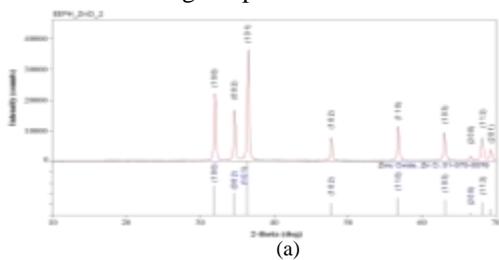


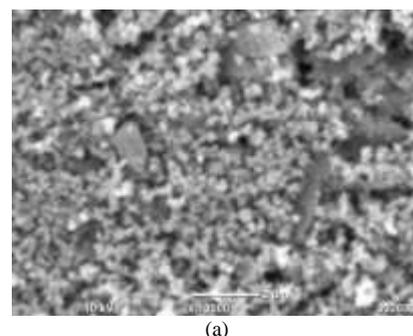
Fig. 3. The XRD spectra of ZnO thin films (a) at 300 °C (b) at 400 °C (c) at 500 °C and (d) at 600 °C

TABLE I. The crystalline size of ZnO thin films at different temperatures

Temperature (°C)	Crystalline Size (nm)
300 °C	45.49
400 °C	49.92
500 °C	50.66
600 °C	51.47

B. Scanning Electron Microscopy (SEM)

The surface morphology of green synthesized zinc oxide thin films deposited by spin coating technique were studied with SEM analysis. Fig. 4 (a) – (d) show the SEM result of ZnO films deposited at 300 °C, 400 °C, 500 °C and 600 °C respectively. The network formation of zinc oxide films were found at different temperatures. The structure of the films consists of quasi spherical in shape and uniformly distributed throughout the surface at 300 °C and 600 °C, but the particles are agglomerate at 400 °C and 500 °C. The surface morphology of the ZnO film deposited at low temperature shows a high density with small grains. With the temperature increasing up to 600 °C, the grain sizes become larger. The slightly changes of microstructure implied the effect of the temperature during the growth process of the films. The average grain size of ZnO films are listed in Table II.



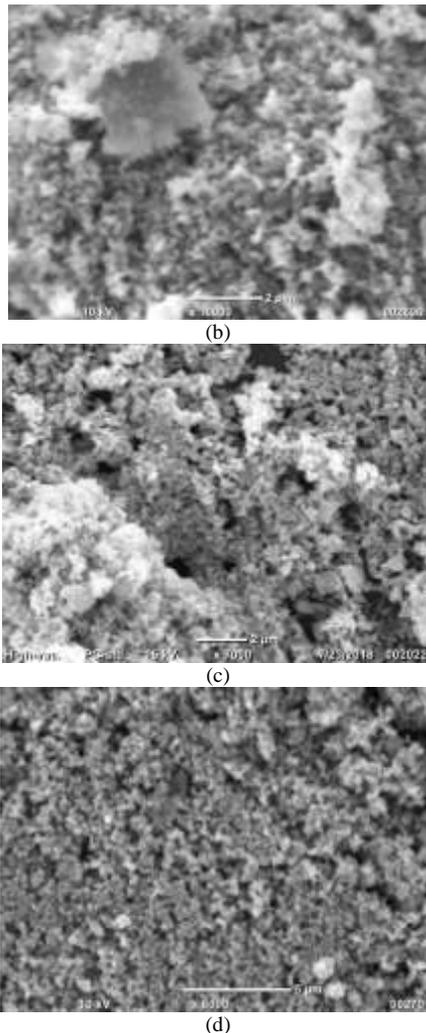


Fig. 4. The SEM image of ZnO thin films (a) at 300 °C (b) at 400 °C (c) at 500 °C and (d) at 600 °C

TABLE. II. The average grain size of ZnO thin films at different temperatures

Temperature (°C)	Grain Size (nm)
300 °C	130
400 °C	132
500 °C	168
600 °C	220

C. Effect of Temperature on the Optical Properties of ZnO Films

The optical absorbance against wavelengths of ZnO films at various temperatures are shown in Fig. 5. A rise of absorbance was observed at $\lambda \sim 270$ nm at 400 °C, 500 °C, 600 °C and 310 nm at 300 °C which indicates that the synthesized ZnO films were photosensitive in UV region. This could be ZnO nanoparticles were excited ground state to excited state.

From Fig. 5, we can see that the absorption edge shifted to shorter wavelengths for sample grown at higher temperature up to 500 °C. But at 600 °C it is shifted to longer wavelength.

The UV absorption edge is related to the optical band gap. The optical band gap energy was calculated using absorption

edge values ($\lambda_{cut\ off}$, in nm) from absorption spectra and using the following eq.

$$\text{Band Gap Energy (E)} = \frac{hc}{\lambda_{cut\ off}} \quad (\lambda = \text{Cut off wavelength})$$

From calculation, the band gap energy increased with increasing temperature up to 500 °C, but it decreased at 600 °C. From the above analysis, the increases of band gap energy with the increasing of the temperature from 300 °C – 500 °C, indicates the quality of the film. The absorption edge wavelength and the band gap energy of ZnO thin films at different temperatures is listed in Table III.

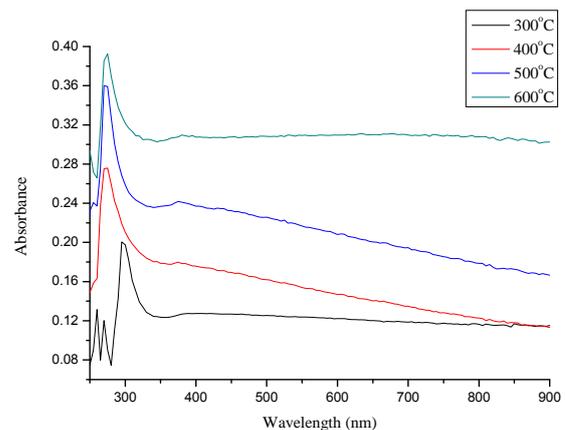


Fig. 5. The absorption graph of ZnO thin films (a) at 300 °C (b) at 400 °C (c) at 500 °C and (d) at 600 °C

TABLE. III. The absorption edge wavelength and band gap energy of ZnO thin films at different temperatures

Temperature (°C)	Absorption edge wavelength (nm)	Band gap energy (eV)
300 °C	356.3	3.46
400 °C	337.5	3.66
500 °C	325.0	3.80
600 °C	362.2	3.41

IV. CONCLUSION

We have analyzed crystallites, morphological and optical properties of green synthesized ZnO thin films on the effect of the temperature. ZnO thin films were fabricated, using the spin coating on glass substrates with 3000 rpm and annealed at different temperatures from 300 °C to 600 °C. According to XRD result, the crystalline sizes were increased from 45.49 nm to 51.47 nm due to increasing temperatures. SEM analysis showed that the surface morphology of the obtained films were uniform at 300 °C, and 600 °C but the grains were agglomerate at 400 °C and 500 °C. The grain size increases from 130 nm to 220 nm and the surface of the films become rougher with the increase of temperature up to 600 °C. The optical properties effect on the temperatures were discussed from the UV-vis spectroscopy. The optical band gap values of the films were increased when temperature increased due to the increase of the grain size of the sample, but 600 °C the band gap value decrease. This may be the decrease of the disorder in the material at 600 °C. The intensity of the UV

peaks of ZnO thin film increased with the increase of the temperature. From the above results, we concluded that these green synthesized ZnO can be used in solar cells. In the next research, we will scope on solar cell with green synthesized ZnO because of cost effective and ecofriendly.

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