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Synthesis of ZnO Nanoparticles by Aloe Barbadensis Leaf Extract via Green Method and their Characterization

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***Abstract*- Metal oxide nanoparticles prove their importance in the field of ceramics, coating, and electronics etc. Physical and Chemical methods can impart nanoparticles of desired morphology but the expansive analysis, toxicity and stability are the major obstacles in the formation of nanoparticles by these methods. In present work we focus on synthesis of ZnO by Aloe barbadensis miller (Aloevera) leaves through eco-friendly Green synthesis method. These nanoparticles were characterized by different analytical techniques. The average crystallite size (~20 nm) was measured using XRD data. The morphological and average size also confirms and supports the diffraction data. Uv-visible and FTIR result also support the synthesis of ZnO particles by Green method.**

***Keywords*- Zinc Oxide, nanoparticles, Green synthesis, Crystallite size.**

I. INTRODUCTION

In present scenario metal and metal oxide nanoparticles proved their importance in various fields e.g. agriculture, medicine, water treatment and also for clean environment [1-2]. Zinc oxide (ZnO) has a numerous roles in biosensors, cosmetics, electronics, pharmaceutical fields [3-4]. The properties of ZnO like wide direct band gap (~3.40 eV) and a large exciton binding energy of 0.060 eV at room temperature, makes it as an important tool for various applications [5]. The toxicity features, expansive analysis, stability are the main challenges in the path of formation of nanoparticles. The Green method does not only provide the better results than physical and chemical methods but also reliable, sustainable, cost effective and ecofriendly approach with no toxic byproducts [6-9]. Green approach is a method of synthesizing nanoparticles by enzymes, bacteria, fungi and plant extracts [10]. In Green synthesis mediated by plant extract, plant extract plays an important role as reducing and stabilizing agents. The phyto chemicals present in plant extract is actively participate in the reduction mechanism and this process affect the morphology of nanoparticles as well as size also such as nanosphere, nanoboxes, nanorods, hexagonal, tripods, nanoplates, nanowires, nanoflowers, nanorings, nanocages and nanotubes etc. [11, 12]

However, it is still challenging to control the crystalline structure, dispersibility, stability in solution methods (where use of water and ethanol) and complexity of nanoparticles formation mechanism, so the different synthesis routes for ZnO is trying to control the various issues [13-15].

ZnO is an important materials for the various applications in science and technology due to its tremendous properties like as wide band gap (~3.40 eV) and large exciton binding energy at room temperature [9]. Many researchers reported that ZnO nanoparticles mediated by plant extracts have better potential than reports by bacteria and fungi [7]. In this paper we synthesize the ZnO nanoparticles by Aloe barbadensis (Aloevera) leaf (powder and gel) extract, where phytochemicals (protein, carbohydrates, phenols, tannin, steroids, terpenoids and glycosides) are present, due to which the reduction mechanism of nanoparticles performed [13-14].

II. EXPERIMENTAL SECTION

Materials and methods:

A. Materials: The following materials are used for the synthesis of ZnO nanoparticles. Zinc acetate $Zn(CH_3COO)_2$ as precursor salt, Aloevera leaf extract as reducing and capping agent, NaOH pallets to maintain the pH value, Whatmann filter paper 12 for filteration and DI water as synthesis medium.

B. Preparation of Aloevera leaf (AP) and Alovera gel (AG) extract:

(a) Fresh leaves of Aloevera collected from local region wash thoroughly with tap water and again washed thoroughly with Double distilled water to remove the contamination then after removing its thorns. These Leaves cut in to fine pieces and then leave them to dry for 15 days after that make a fine powder using Grinder and then mortar pestle to mix homogenously. 200 ml D.I. water mixed with 25 gm aloevera leaves powder and stirrer for 45 minutes at 60 ^{o}C and then after filtering, successfully gets the Aloevera leaf (AP) extract.

(b) To get the extract of Aloevera gel, fresh leaves washing with tap water then double distilled water and after removing thorns, the Aloevera gel separated from the leaves and 25 gm of Aloevera gel mixed with 100 ml D.I. water and stirring for 45 minutes at 60 ^{o}C, then after filtering, get the Aloevera gel (AG) extract.

C. Synthesis of ZnO Nanoparticles: For synthesis of ZnO nanoparticles Zinc Acetate (1 gm) mixed in 200 ml D.I. water and then mixed with 16 ml Alovera leaf (AP and AG) extract. To maintain the pH value of the solution, use 2M NaOH solution (40 ml NaOH). The whole mixture was stirred at 60 ^{o}C for 60 minutes in Glove box to avoid the environment contamination and leave it for ten days. After thoroughly washing with DI water, followed by filtering and drying in an oven overnight, then the synthesized ZnO nanoparticles are formed in powder form.

III. ANALYSIS OF ZnO NANOPARTICLES

The both synthesized ZnO nanoparticles were characterized by different analytical techniques. The structural properties are determined by X-Ray Diffractometer (XRD), morphological and elemental composition studies by Field Emission Scanning Electron Microscope (FESEM) with Energy dispersive spectroscopy (EDS), Optical properties by Uv-Visible spectroscopy (Uv-Vis), and the presence of different functional groups revealed by Fourier Transform Infrared (FTIR) spectroscopy.

IV. RESULTS AND DISCUSSION

Morphology of ZnO np's: The morphology of both synthesized ZnO np's are shown in Fig. 1(a, b). The formation of nanocrystalline material shows the randomly oriented aggregates of nanoparticles with variable sizes. Zinc Oxide nanoparticles are prone to aggregate due to the large surface area and high surface energy. It is concluded from SEM results, that nanoflakes like structure with random arrangement are formed in both samples [16]. The elemental chemical analysis was carried out by EDS detector with SEM measurements and shown in Fig. 1(c, d). The result shows the presence of the elements (Zn, & O) in the nanoparticles.The calculated atomic percentage of Zinc (Zn) and oxygen (O) in both synthesized np's are shown in table I.

Table I
Atomic weight percentage of EDS spectrum of np's AP and AG extract

Element	Series	AP		AG	
		Weight (wt.%)	Atom. (at.%)	Weight (wt.%)	Atom. (at.%)
Zn (30)	K	87.31	62.73	89.65	67.94
O (8)	K	12.69	37.27	10.35	32.06

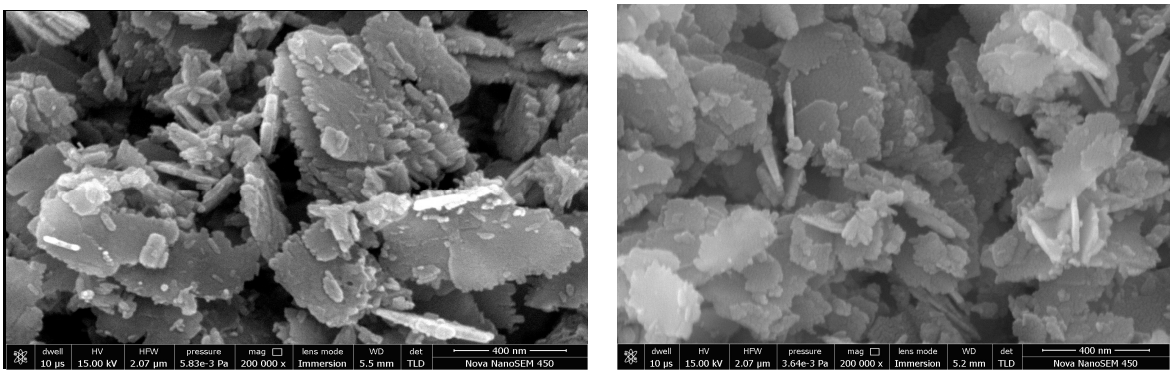

Fig.1: Micrograph of ZnO np's of (a) AP (b) AG extract

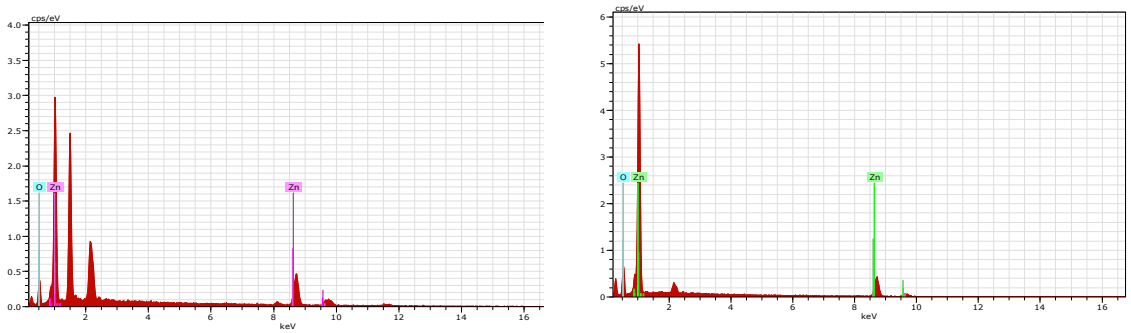

Fig. 1: Elements present in ZnO np's of (c) AP (d) AG extract

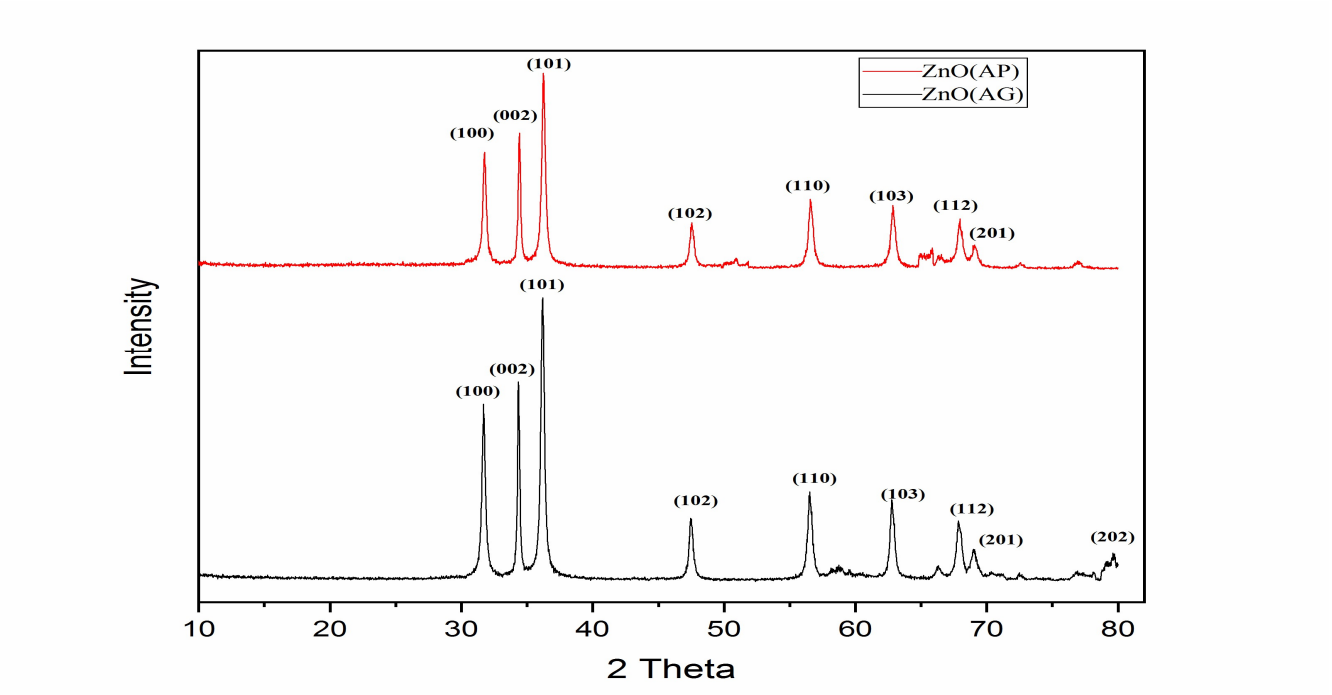

Fig. 2: X-ray Diffraction pattern of ZnO np's (AP and AG extract)

B. Structural analysis of ZnO np's: XRD is used for the crystallization structure analysis and particle size of the both (AP and AG) synthesized ZnO np's. The XRD peaks in the wide angle range of 2θ from 10 - 80° were recorded and shown in Fig.2. The pattern is showing peaks at the different angles of 2θ for ZnO np's, which corresponding indexed to diffracting planes for (100), (002), (101), (102), (110), (103), (112), (201), (202) respectively marked in the figure. The nature of the ZnO crystal lattice is cubic as per results support from the data and the standard diffraction peaks are confirmed by JCPDS files (data card no: 36-1451) [17, 18]. It is clear from the closer inspection of the diffraction spectra, that the broadening of the peaks are the indication of nanometer ranges. Average particle size was estimated by using "Debye-Scherrer" formula ($D = K\lambda / \beta\cos\theta$). Where D is the diameter of the crystallize size, K is the shape factor (typical value is 0.9), wave length of the incident X-ray is λ=1.54Å, θ is the bragg's angle and β is the broadening of the peak at the centre. The calculated average crystallite size is found to be ~ *20 nm* (average error ± 1 *nm*), which also supports the information from SEM results.

C. Absorption spectra of ZnO np's: Uv-visible spectroscopy is used for optical measurements (absorption / transmission spectra) and further quantitative analysis of the calculation of band gap of samples. The absorption of radiation in a sample follows the beers-lambert law, where the concentration of a substrate in a sample is directly proportional to the absorbance (A). The absorption spectra of the ZnO np's at room temperature are shown in Fig. 3a. Here from the spectrum, it can be seen that the absorption peak for the both synthesized sample is appeared as compared with that of bulk ZnO sample and indicates the presence of quantum confinement effect in the ZnO np's sample. The bulk ZnO materials exhibit an absorption peak of about *365 nm* but in our sample ZnO np's show almost same nature in the visible region. As the size of semiconductor particles decrease to nanoscale, the band gap of the semiconductor increases, causing a blue shift is observed in UV-Visible spectra. The Tauc plots for both samples are drawn in Fig. 3b and the calculated band gaps are 3.07 and 3.1 eV respectively.

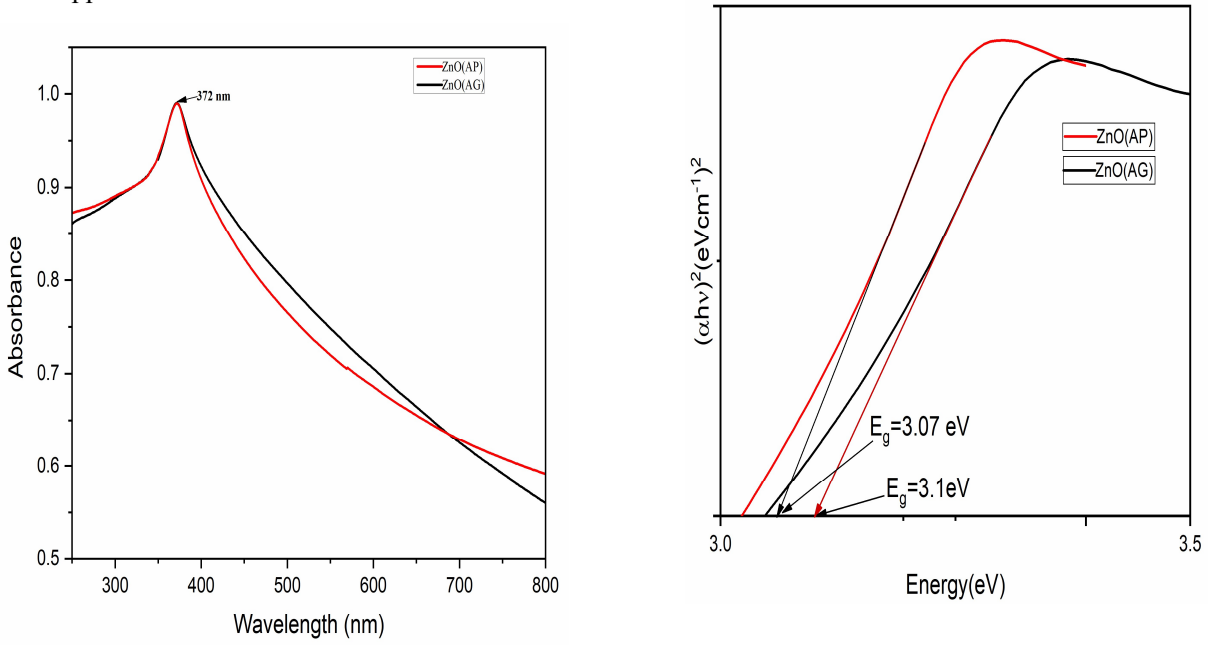

Fig. 3: Optical Spectra (a) Absorbance (b) Tauc plot for ZnO np's (AP & AG extract)

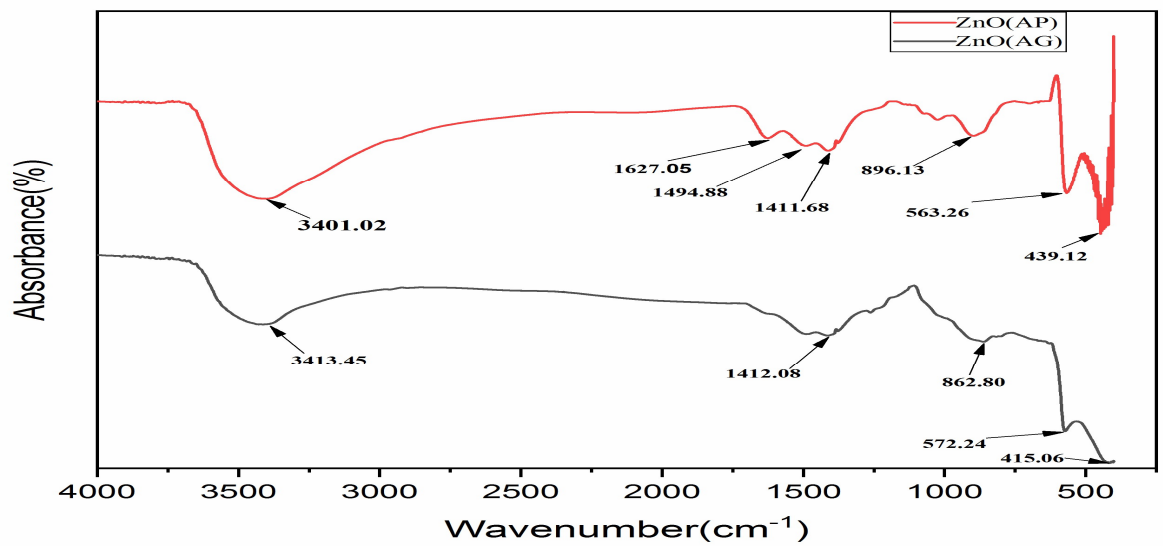

Fig 4: FTIR Spectra of ZnO np's (AP & AG extract)

D. FTIR spectroscopy: To detect the presence of adsorbed functional groups on the surface of synthesized nanoparticles during synthesis process can be revealed by Infrared spectroscopy analysis. Fig. 4 represents the FTIR spectra of both ZnO np's and the absorption peak in the range of 300–4000 cm^{-1} were observed. These were centered at 3401.02 cm^{-1} corresponds to the stretching vibration of intermolecular hydrogen bond (O–H) existing between the adsorbed water molecules and indicates the higher amount of hydroxyl group. The peaks at 1411.68, 1494.88 cm^{-1} in AP sample, while 1412.08 cm^{-1} in AG sample, shows the symmetric vibrations of O=C=O. Some reports show, the presence of sharp peak ranging from 400 to 600 cm^{-1} explains the formation of ZnO nanoparticles [15-17]. It is interesting to note that we found two sharp and intense peaks at *563.26, 439.12* cm^{-1} in AP sample and 572.24, 415.06 cm^{-1} in AG sample, which reveals the Zn-O stretching vibrations.

V. CONCLUSION

In this study, ZnO nanoparticles were successfully synthesized by an ecofriendly and cost effective approach (Green synthesis) by using zinc acetate as precursor salt and aloevera leaf (Powder and Gel) extract as reducing and capping agent. XRD analysis reveals the size of nanoparticles approximately ~ 20 (± 1) nm. SEM measurements reveal the surface morphology and EDS results show the elemental analysis of the synthesized ZnO samples. Optical measurements are used for the band gap (3.07 and 3.1 eV) calculations, which are lower for synthesized nanoparticles than their bulk samples. FTIR results show the presence of ZnO stretching in both samples. The ZnO nanoparticles are useful as an electron transport layer (ETL) for perovskite solar cells as reported by our research group [18].

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