

SYNTHESIS AND CHARACTERIZATION OF ZNO POWDERS THROUGH A FACILE GREEN SYNTHETIC APPROACH.

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Abstract

Zinc oxide powders are widely researched for their numerous applications in the field of cosmetics, semiconductor devices, coating development and in medicine. A number of methods are available for the synthesis of these powders and this paper deals with the synthesis through a green approach. The synthetic method was optimized to get good quality powders and FTIR, XRD and SEM techniques were used to characterize the powders. Further studies on particle size reduction and functionalization is being done.

Keywords : Zinc oxide, coatings, green method.

1. INTRODUCTION

Metal oxide nanoparticles stand out as one of the most versatile materials, due to their diverse properties and functionalities [1]. Most preferentially, among different metal oxide powders, zinc oxide (ZnO) powders have their own importance due to their vast area of applications, e.g., gas sensor, chemical sensor, bio-sensor, cosmetics, storage, optical and electrical devices, window materials for displays, solar cells, and drug-delivery [2-4]. ZnO is an attractive material for short-wavelength optoelectronic applications owing to its wide band gap 3.37 eV, large bond strength, and large exciton binding energy (60 meV) at room temperature [5]. As a wide band gap material, ZnO is used in solid state blue to ultraviolet (UV) optoelectronics, including laser developments [6]. In addition, due to its non-centrosymmetric crystallographic phase, ZnO shows the piezoelectric property, which is highly useful for the fabrication of devices, such as electromagnetic coupled sensors and actuators. Though a number of methods[7-11] are available to synthesize ZnO powders with good purity, this paper is aimed at achieving this synthesis through a green method using plant extracts so as to make the material more bio and eco friendly.

2. MATERIALS AND METHODS

Synthesis Of Zno By Using Precipitation Method ...

Zinc nitrate was used as the precursor and NaOH as a precipitating agent to synthesize ZnO powder were purchased from Merck, India. In this work, the aqueous solution (0.2 M) of zinc nitrate [Zn(NO₃)₂.6H₂O] and the solution (0.4 M) of NaOH were prepared with ethanol 100 ml standard flask, respectively. The zinc nitrate solution is slowly added into NaOH solution at room temperature under vigorous stirring two hours, which resulted in the formation of a white suspension. The white product was centrifuged at 450 rpm for 20 min and washed three times with distilled water, and washed with absolute alcohol at last

trying. The obtained product was calcinated at the 550 °C in the air atmosphere for three hours. During drying, complete conversion of Zn (OH)₂ into ZnO takes place and ZnO obtained as a result.

Synthesis of zno powder using plant extract: Green synthesis of ZnO using Abrus precatorius

Materials:

Zinc nitrate was used as the precursor, for the synthesis NaOH as used as a precipitating agent to synthesize ZnO powder. these were purchased from Sigma-Aldrich, Pondicherry, India. Abrus precatorius leaves were collected.

Preparation of leaf extracts:

The collected Abrus precatorius leaves (80 g) were washed several times with distilled water, to remove the impurities and then boiled in 200 ml of deionised water for half an hour. The resulting extract was cooled and used as the extract solutions.

Synthesis of ZnO Powder:

(0.2 M) g of Zinc nitrate was dissolved in 100 ml water. 20 ml of the extract of Abrus precatorius was added dropwise and the resulting mixture was stirred for one hour using a magnetic stirrer. In order to adjust the pH of the solution to pH 12, NaOH (0.2 M) was added drop-wise while stirring. A light yellow precipitate was obtained. Finally, this is washed repeatedly with water and pure alcohol. Filtered and dried in an oven at 60°C to obtain the ZnO powder.

3. RESULTS AND DISCUSSION

FT-IR Analysis:

Fourier Transform Infrared Spectroscopy was used to identify the functional groups. The chemical composition of the synthesized Zinc oxide powder was studied by using FT-IR spectrometer (Perkin-Elmer LS-55- Luminescence spectrometer).

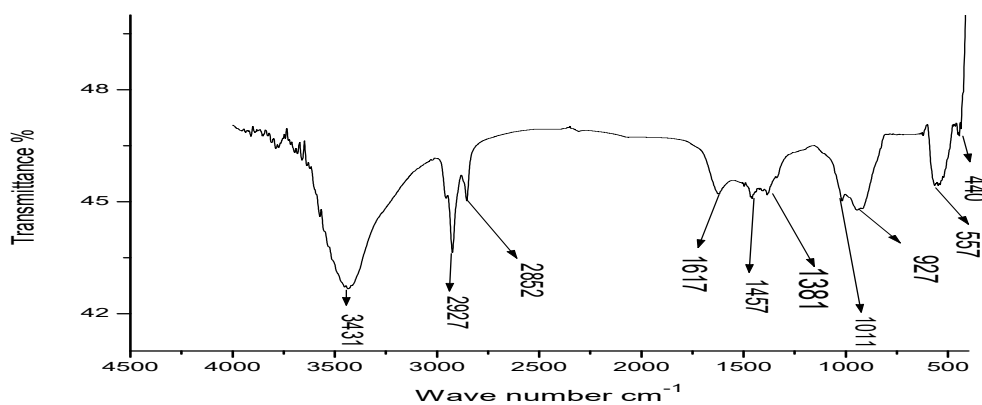


Fig : 1 FT-IR spectrum of ZnO powders using NaOH.

The solutions were dried at 75°C and the dried powders were characterized in the range 4500-500 cm⁻¹ using KBr pellet method. The result of FT-IR analysis for ZnO is depicted in Fig.1. FTIR measurement was performed in order to verify the bond structure of ZnO powder prepared. Fig.1 shows the infrared absorption spectra of ZnO powder from 4500-500 cm⁻¹ wave-number range. The bands at 3431- 2927 cm⁻¹ is due to the O-H mode of vibration. The strong asymmetric mode of vibration of C=O was observed between 1617 and 1457 cm⁻¹. The symmetric stretching occurs between 1457 and 1381 cm⁻¹ because of presence of C-O. C-O-C peak is also present. The peaks around 2852 cm⁻¹ can be attributed to the C-H stretching which may be result of presence of some organic moieties. Fig.1 shows lesser bands and the bands at 440cm⁻¹, 557cm⁻¹ and 927cm⁻¹ are indicative of Zn-O vibrations. Therefore the IR spectrum confirms the formation of ZnO powder with good purity. The presence of carbonaceous material shows that repeated washing of the precipitate was not so successful in removing the traces of chemicals used completely.

XRD Studies of ZnO Powder prepared using NaOH :

The powder X-ray diffraction studies were performed for the obtained Zinc Oxide Powder which is shown is below : The X-ray diffraction pattern of ZnO powders is shown in Fig. 2. The X-ray diffraction patterns were recorded within a range of 2 theta 10 to 90°. The crystalline nature, crystal phase and crystallite size of synthesized ZnO material were studied with the help of XRD spectrum. The obtained XRD spectrum shown in above figure matched well with the diffraction pattern of the bulk ZnO. The peaks observed at (100), (002), (101), (102), (110), (103), (200), (112) and (201) can be assigned to various crystal planes of hexagonal crystal structure of ZnO. Additionally, some minor peaks were also observed indicating excellent crystallinity of the sample.

SEM Studies of ZnO powder prepared using NaOH:

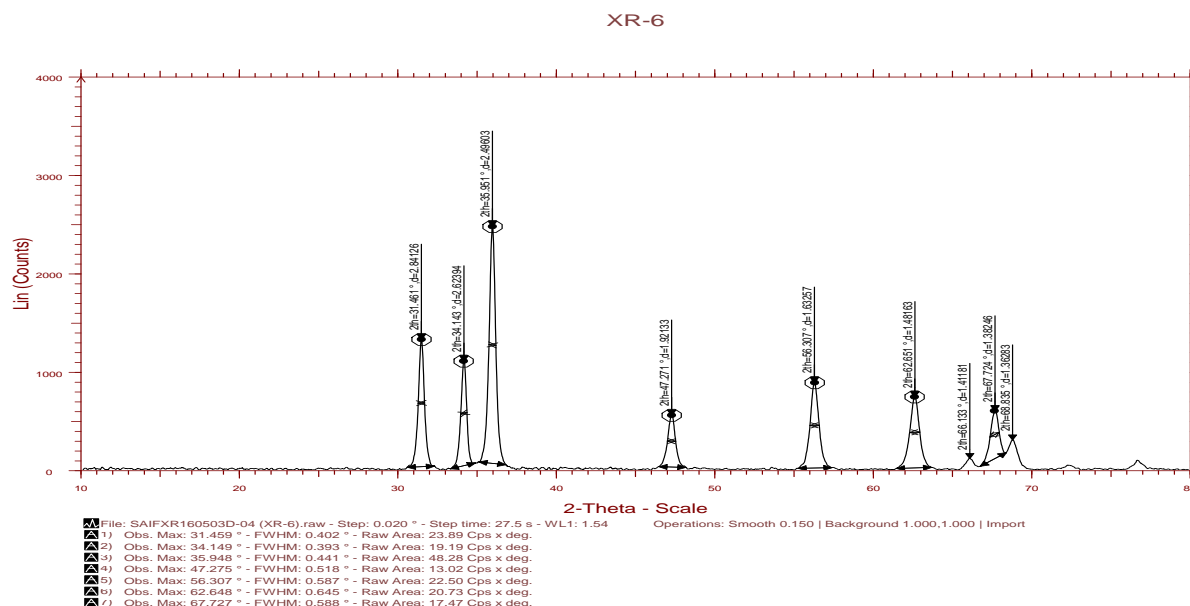


Fig.2: X-RD pattern obtained for the ZnO powder prepared using NaOH .

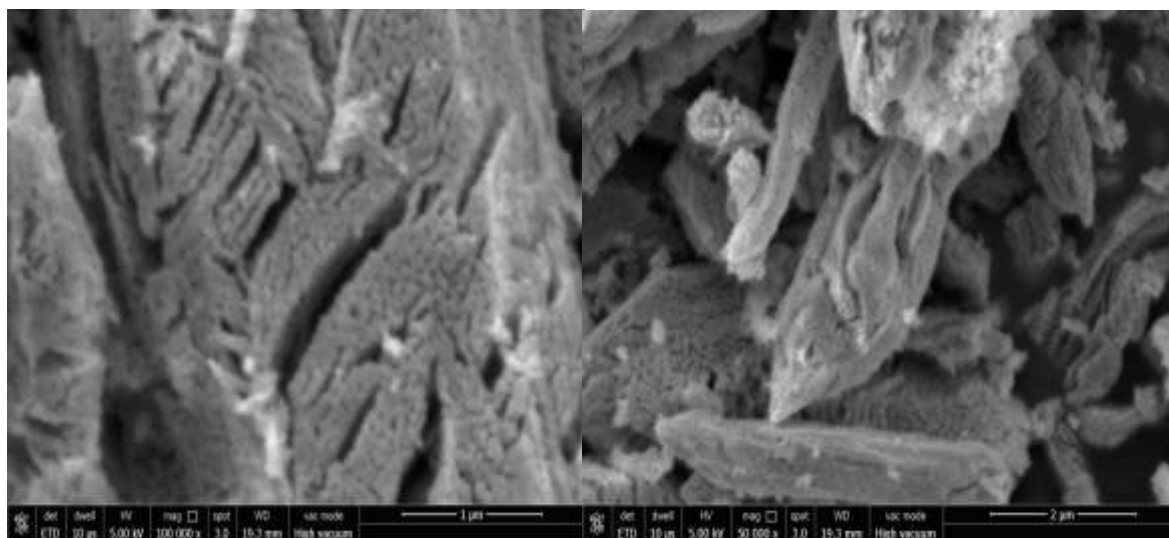


Fig. 3 : SEM image of ZnO Powder prepared using NaOH .

Scanning electron microscopic studies on the as synthesized material shed light on the morphology of the powder. The image obtained for the powder obtained with Zinc nitrate and NaOH result is shown below. From the image it can be known that there is a considerable amount of agglomeration in the products. Moreover there is elongation of particles due to the effect accelerated by the presence of excess of base.

FT-IR analysis of ZnO preparation using Abrus Precatorious:

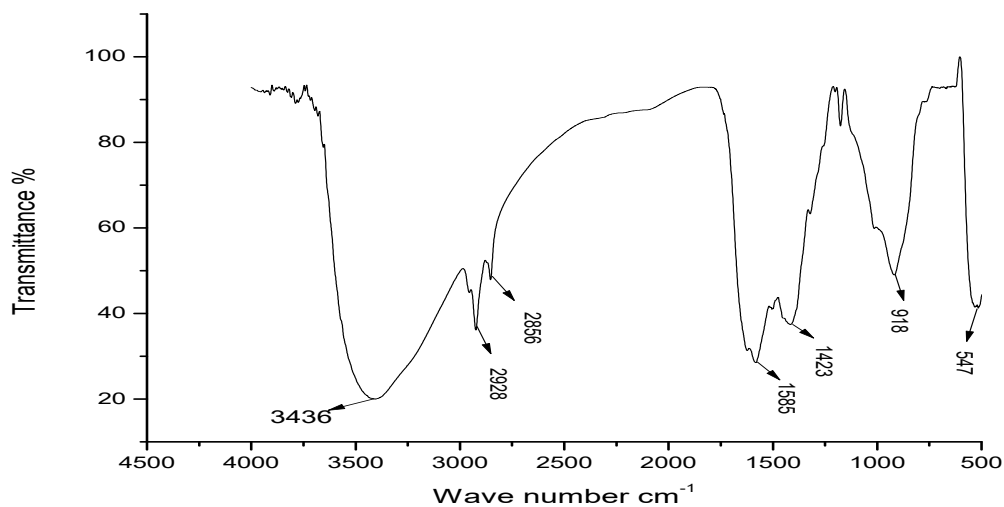


Fig. 4: FT-IR spectrum of ZnO powder prepared using Abrus Precatorious.

5.14 XRD Studies of ZnO powder preparation using Abrus Precatorious :

The powder X- ray diffraction studies were performed for the obtained ZnO Powder which is shown is below :

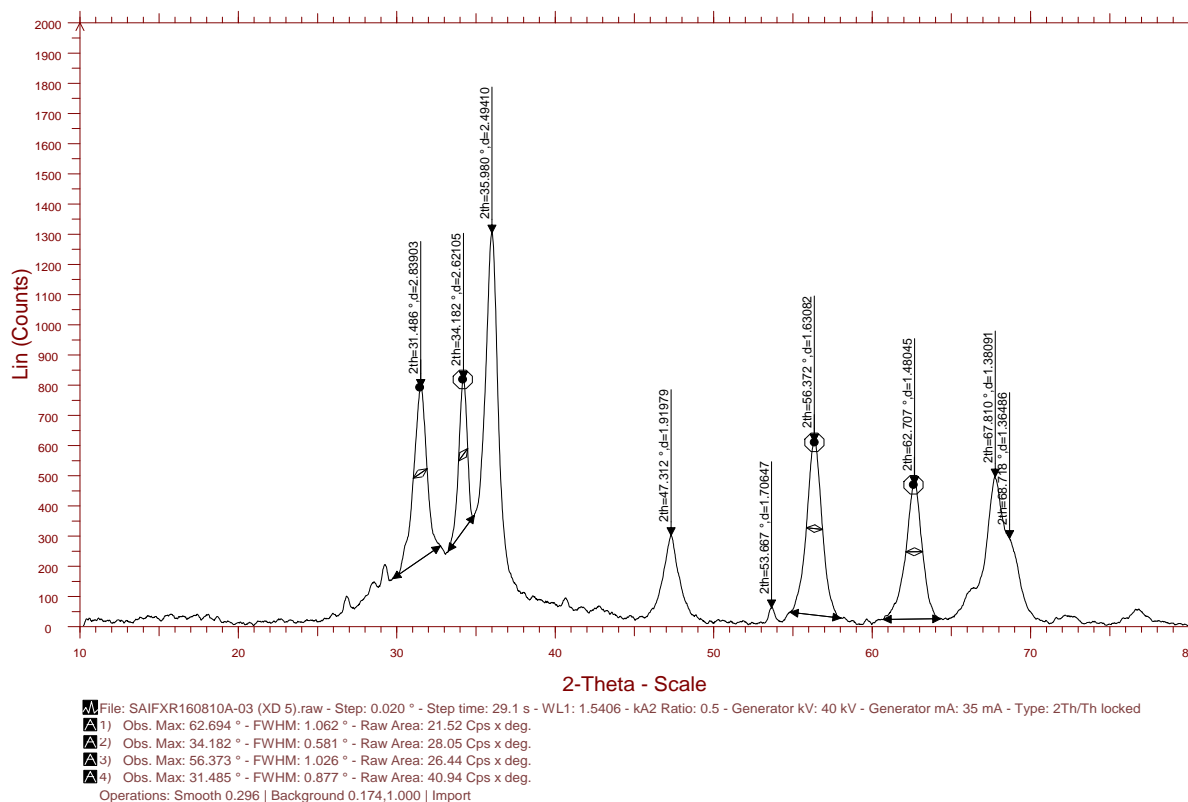


Fig. 5 X-RD pattern obtained for the ZnO powder prepared using Abrus Precatorious.

X-ray diffraction patterns were recorded within a range of 2 theta 10 to 90°. The crystalline nature, crystal phase and crystallite size of synthesized ZnO powder were studied with the help of XRD spectrum. The obtained XRD spectrum shown in **Figure. 5** matched well with the diffraction pattern of the bulk ZnO. The peaks observed at (100), (002), (101), (102), (110), (103), (200), (112) and (201) can be assigned to various crystal planes of hexagonal crystal structure of ZnO. Additionally, some minor peaks were also observed indicating excellent crystallinity of the sample.

SEM Studies of ZnO powder preparation using Abrus Precatorious :

Scanning electron microscopic studies on the as synthesized material shed light on the morphology of the powder. The image obtained for the Powder obtained with Zinc nitrate and Abrus Precatorious result is shown below.

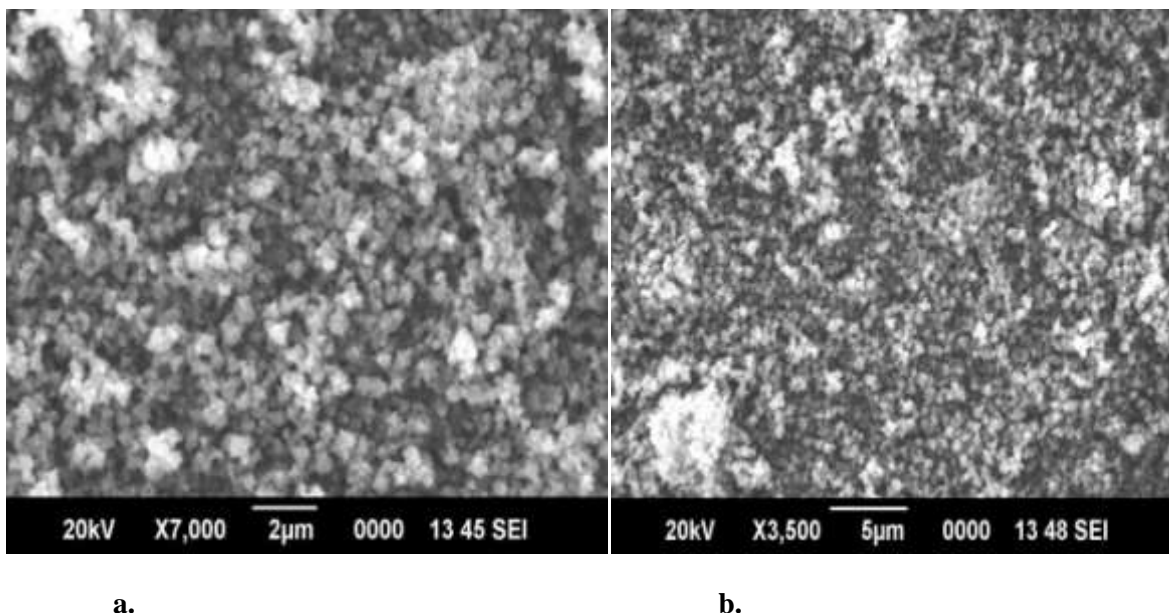


Fig. 6: SEM Images of ZnO powder prepared using Abrus Precatorious.

The SEM image of the sample is shown in Fig. 6 (a) and (b) reveals that the particles are spherical and having granular nature. In the higher resolution SEM image the agglomeration of particles is observed. Aging may have caused the agglomeration.

The SEM analysis of was used to determine the surface morphology and shape of Zinc oxide powder. In this study SEM images (Fig 6) has showed individual zinc oxide Powder as well as a number of aggregates. The closer observations, magnification at X10, 000 (Fig. 6) revealed that the surface of the product was smooth and shape of ZnO powder was found approximately spherical.

CONCLUSIONS

ZnO powders were synthesized by few common chemical methods along with novel green synthetic approach. Zinc metal oxide particle could be successfully synthesized from green methods in par with conventional methods. From the FTIR the presence of metal oxide bonds along with few peaks pertaining to the presence of organic moieties is confirmed.

The XRD patterns obtained for the metal oxide particles further confirm the phase purity and the crystallinity of the samples. The SEM images reveal that the sample contains crystallite structures with uniform morphology with a little aggregation. Hence such synthetic methods can be scaled up and used for industrially important material.

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REFERENCES

- [1] Al-Gaashani, R., Radiman, S. Tabet, N., Daud, A.R., 2011. *Materials Chemistry and Physics* 125(3), 846-852.
- [2] Belomestnykh, I. P., Voikina, N. V., 1987. *Kinetics and Catalysis* 28(3 Pt 2), 602-608.
- [3] Al-Gaashani, R., Radiman, S. Tabet, N., Daud, A.R., 2011. *Materials Chemistry and Physics* 125(3), 846-852.
- [4] Belomestnykh, I. P., Voikina, N. V., 1987. *Kinetics and Catalysis* 28(3 Pt 2), 602-608.
- [5] Jia, W., Li, T. B., 2013. *Journal of Synthetic Crystals* 42(10), 2007-2012.
- [6] Jia, W., Li, T. B., 2013. *Journal of Synthetic Crystals* 42(10), 2007-2012.
- [7] Reyes-Agüero JA, Aguirre-Rivera JR, Hernandez MH (2005) (Cactacea). *Agrociencia* 39: 395-408.
- [8] Griffith MP (2004), *Am J Bot* 91: 1915-1921.
- [9] Ahn DK (1998) *Illustrated book of Korean medicinal herbs*. Kyohak Publishing Company, Seoul, South Korea.
- [10] Zhao M, Yang N, Yang B, Jiang Y, Zhang G (2007) *Food Chemistry* 105: 1480-1486.
- [11] Hernández-Urbiola MI, Pérez-Torrero E, Rodríguez-García ME (2011) *Int J Environ Res Public Health* 8: 1287-1295.