

# Green Synthesis of Zirconium Dioxide (ZrO<sub>2</sub>) Nano Particles Using *Acalypha Indica* Leaf Extract

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**Abstract**— The green biogenic synthesis of nano particles using plant extracts is always eco friendly and attractive. The current study focuses on the production of ZrO<sub>2</sub> nanoparticles using the aqueous leaf extracts of *Acalypha Indica*. This green synthesis approach shows that the environmentally benign and renewable aqueous leaf extract of *Acalypha indica* can be used as a reducing agent for the synthesis of zirconium dioxide nanoparticles. The FTIR analysis played a pivotal role in displaying the important functional groups present in the ZrO<sub>2</sub> nanoparticle, which showed that the sample had strong absorbance in the range of 508 & 498 cm<sup>-1</sup>. The XRD pattern has been used to characterize the structure and size of ZrO<sub>2</sub> nanoparticles. The Scanning Electron Microscope with the Energy Dispersive X-ray studies provided the size and the elemental composition of the synthesized ZrO<sub>2</sub> nanoparticles. The average size of the nanoparticles was found to be 20-100nm. Hence the biogenic synthesis of ZrO<sub>2</sub> nanoparticles using *Acalypha Indica* can be an alternative to chemical synthesis.

**Index Terms**— Zirconium dioxide nano particle; green synthesis

## I. INTRODUCTION

The field of nanotechnology is one of the most active researches in modern material science. Nanotechnology is emerging as a rapid growing field with its applications in science and technology for the purpose of manufacturing new materials at the nano scale level. Recently, need for designing new materials with improved properties have forced fast development of nano structured materials. Thus researches have been focused on investigation of materials at the atomic, molecular and macromolecular level, with the aim to understand and manipulate the features that are substantially different from the processing of materials on micro-scale. Nanoparticles usually ranging in dimensions from 1 to 100 nm have properties unique from their bulk equivalent. With the decrease in dimensions of materials to the atomic level, their properties change. The nano particles possess unique physicochemical, optical and biological properties which can be manipulated suitably for desired applications<sup>[1]</sup>. Nanoparticles find use as delivery vehicles for drugs, genes and growth factor as well as cellular labels for imaging both *in vitro* and *in vivo*. Nanoparticles are also being studied for the use in photodynamic therapy<sup>12</sup> and hyperthermia therapy for tumour, with the goal of clinical application<sup>[2]</sup>. Plants are known to possess various therapeutic compounds which are being exploited since ancient time as a traditional medicine. Due its huge diversity plants have been explored constantly for wide range of

applications in the field of pharmaceutical, agricultural, industrial etc. Plants have been used as resources for the synthesis of different nanoparticles. Recent reports of plants towards production of nanoparticles is said to have advantages such as easily available, safe to handle and broad range of biomolecules such as alkaloids, terpenoids, phenols, flavanoids, tannins, quinines etc. are known to mediate synthesis of nanoparticles<sup>[3]</sup>.

Zirconium dioxide (ZrO<sub>2</sub>), an industrially important metal oxide with wide range of applications. In recent years, usage of Zirconium dioxide nanoparticles (nano-ZrO<sub>2</sub>) is rapidly growing in biological fields. They are widely used as drug delivery carriers for some medicines like itraconazole, penicillin, alendronate and zoledronate as gene delivery vehicles with target specificity for some tissues for improving the properties of traditional bone cements in orthopedics and some other purposes such as production of poisons like parathion or nerve agents. Although ZrO<sub>2</sub> is a neutral bioceramic material,<sup>[4]</sup> The usefulness of Zirconium oxide nanoparticles depend on their size and physical properties in addition to their chemical composition.

The aim of this work is to synthesise Zirconium dioxide nanoparticle in a green biogenetic way. The synthesized Zirconium dioxide nanoparticles were characterized by Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), Scanning Electron Microscope (SEM), and Energy-Dispersive X-ray (EDX) spectrometer, analyses.

## II. EXPERIMENT

### 2.1 Material

Zirconyl chloride octahydrate [ZrOCl<sub>2</sub>.8H<sub>2</sub>O] purchased from Fischer chemical limited- Chennai was directly used without further purification. The leaves of *A. indica* collected in the SFR college campus, Sivakasi, were washed thoroughly with double distilled water and dried for a few days at sun light.

### 2.2 Preparation of plant extract

Plant extract was prepared by the following two methods. In one method the dried leaves were powdered in a motor pestle. About 20g of the leaf powder was boiled in a 250ml beaker with 200ml of double distilled water for 30 minutes. Then the extract was filtered with Whatmann No.1 filter paper. The filtrate was kept in a refrigerator at 4°C for further work. This is the plant extract 1.

In another method Soxhlet apparatus was used. About 20g of the leaf powder were boiled at 70°C in a 500ml Soxhlet apparatus with 250ml of double distilled water for 3 hours. The condensate was cooled kept in a refrigerator at 4°C for further experiments. This is the plant extract 2.

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### 2.3 Green synthesis of ZrO<sub>2</sub> nanoparticles

Green synthesis was employed for the preparation of ZrO<sub>2</sub> nano particles .To 50ml of 0.1M aqueous solution of zirconyl nitrate octahydrate taken in a 250ml beaker was added 10ml of A.indica leaf extract(1) and stirred at 80<sup>0</sup>C for 2 hours and the solution was kept undisturbed. The particles were formed after few days.The resulting solution was evaporated in a vaccum air oven at 200<sup>0</sup>c for 2 hours to obtain the ZrO<sub>2</sub> nano particles.This was considered as sample (1).

The above procedure was repeated with A.indica extract (2) to get another sample ZrO<sub>2</sub> nano particles .This was considered as sample (1) <sup>[5]</sup>.

### 2.4 Characterization of synthesized ZrO<sub>2</sub> nano particles

#### 2.4.1 FT-IR spectrum

The synthesized samples of ZrO<sub>2</sub> nanoparticles were charaterised by taking their FT-IR spectrum using SHIMADZU model in the diffuse reflection mode.

#### 2.4.2 XRD Spectrum

To find the size of the synthesized ZrO<sub>2</sub> nanoparticles,the XRD spectrum of the samples were taken using SSD160 1D Detector (Bruker).

#### 2.4.3 SEM images

To establish the size of the synthesized ZrO<sub>2</sub> nano particles,SEM images were taken using Carl Zeiss model EVO 18.

#### 2.4.4 EDX Spectrum

Element analysis of the synthesized ZrO<sub>2</sub> nano particles were done by taking EDX spectrum.EDX spectrum were taken using Qutax 200 with X-flash-Bruker.

## III. RESULT AND DISCUSSION

### 3.1 FT-IR spectrum of synthesized ZrO<sub>2</sub> Nanoparticles

The FT-IR spectrum of the two samples of synthesized ZrO<sub>2</sub> Nanoparticles are given in Figure 1&2.The band at 766.8cm<sup>-1</sup> due to Zr-O-Zr asymentric stretching.The band at 508& 498 are due to Zr-O stretching. This conforms the formation of ZrO<sub>2</sub>.The broad peak shows that the particles have nano structure. The absorbtion broad and sharp peaks located at 3394.72 and 1627.92,1381.03,&1635.64 cm<sup>-1</sup> are associated with the -OH stretching and bending vibration of adsorbed water<sup>[6,7]</sup>.

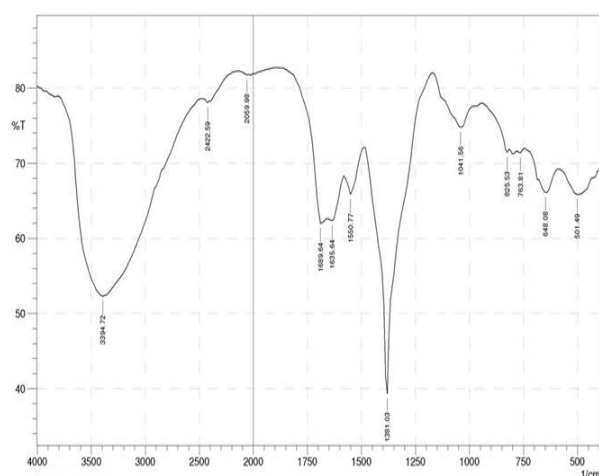


Figure1- FT-IR spectrum of the synthesized ZrO<sub>2</sub> nanoparticles Sample 1.

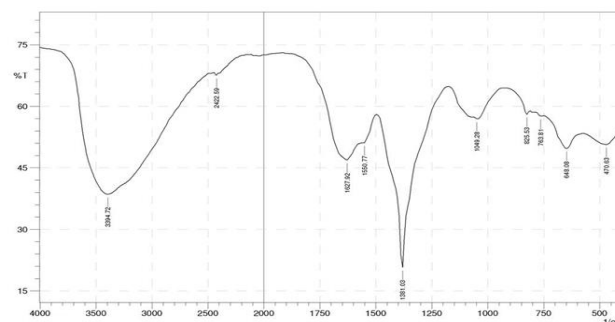


Figure2- FT-IR spectrum of the synthesized ZrO<sub>2</sub> nanoparticles Sample 2.

### 3.2 X-Ray Diffraction of synthesized ZrO<sub>2</sub> Nanoparticles

The XRD pattern of nanoparticles of ZrO<sub>2</sub> sample (1&2) is given in Figure 3&4. XRD has been used to characterize the structure of ZrO<sub>2</sub> nanoparticles. The analysis of the spectrum clearly demonstrate that, the synthesized ZrO<sub>2</sub> nanoparticle is identical and could be indexed to the standard ZrO<sub>2</sub> with monoclinic structure (JCPDS no: 37-1484).The prominent peaks have been utilized to estimate the grain size of sample with the help of Scherrer equation.

$$D = K\lambda/(\beta \cos \theta)$$

Where,

$K$  is constant(0.9),  $\lambda$  is the wavelength ( $\lambda = 1.5418 \text{ \AA}$ ) (Cu  $K\alpha$ ),  $\beta$  is the full width at the half maximum of the line( $\beta=99.218^\circ$ ),  $\theta$  is the diffraction angl[( $\theta=25.131$ (sample 1)&6.602(sample 2)]

The grain size was found to be 58 nm and72 nm for sample 1 and sample 2, Sharpness of XRD peaks indicates that particles are having in crystalline nature<sup>[8]</sup>.

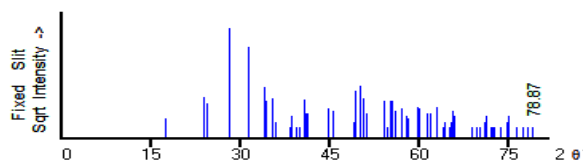


Figure3- XRD pattern of the synthesized ZrO<sub>2</sub> nanoparticles Sample 1.

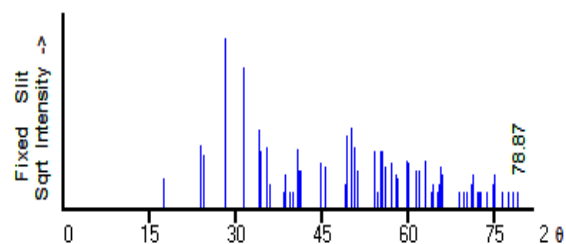


Figure4- XRD pattern of the synthenanapar2.

### 3.3 SEM images of synthesized ZrO<sub>2</sub>

Figure4- XRD pattern of the synthesized ZrO<sub>2</sub> nanoparticles Sample 2.

#### Nanoparticles

SEM images of the samples are given in Figure 5&6.This gives evidence for the formation of ZrO<sub>2</sub> in the Nanoscale in both the samples. The highly magnified SEM images substantiate the approximate cube shape of the nanoparticles and most of the particles exhibit some faceting ranging from 20-100 nm<sup>[9]</sup>.

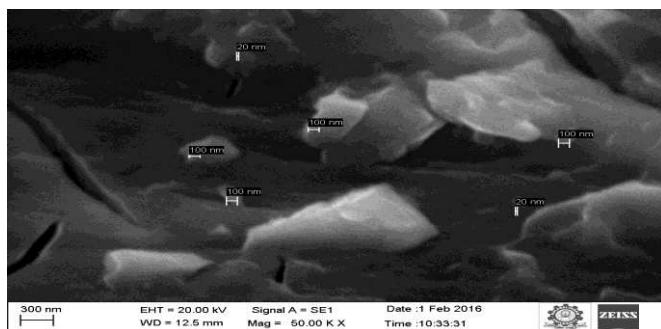


Figure5- SEM images of the synthesized  $ZrO_2$  nanoparticles Sample 1.

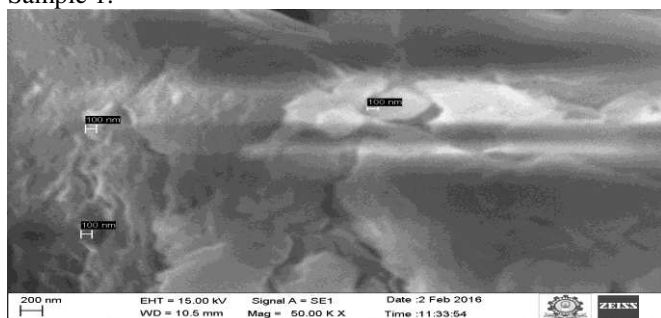


Figure6- SEM images of the synthesized  $ZrO_2$  nanoparticles Sample2.

### 3.4 EDX spectrum of synthesised $ZrO_2$ nanoparticles

EDX spectrum of the samples are given in Figure 7&8. Analysis through EDX spectrometer has confirmed the presence of elemental Zirconium and the Oxygen signal of the  $ZrO_2$  nanoparticles. The vertical axis displays the number of X-ray counts while the horizontal axis displays energy in keV. Identification lines for the major emission energies of zirconium and oxygen are displayed and these correspond to peaks in the spectrum. The formation and composition of crystalline  $ZrO_2$  nanoparticles are justified from quantitative analysis, which reveals that Zr and O are the only elementary species in the sample<sup>[10]</sup>.

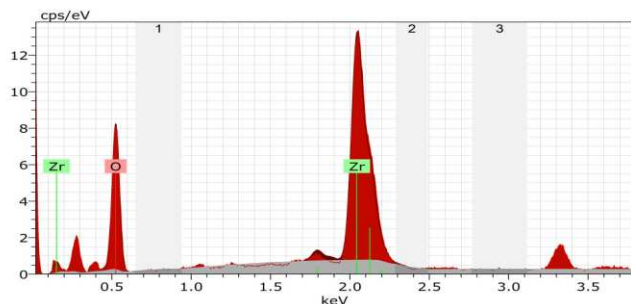


Figure7-EDAX spectrum of the synthesized  $ZrO_2$  nanoparticles Sample1.

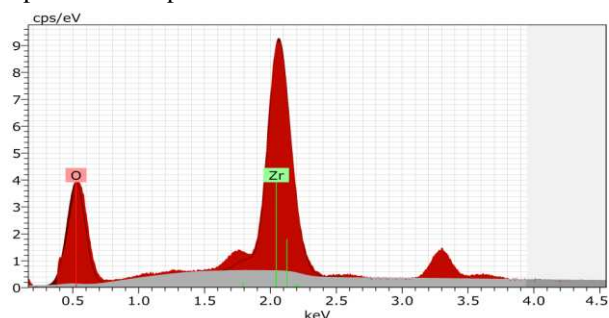


Figure8-EDAX spectrum of the synthesized  $ZrO_2$  nanoparticles Sample2.

## IV. CONCLUSION

We have developed a novel greener approach for biogenic synthesis of zirconium oxide nanoparticles. The synthesis method is faster, economical and greener since this avoids multiple reaction steps, conventional energy sources and harmful chemicals. This preparation of zirconium oxide nanoparticles using *Acalypha indica* leaf extract is being ecofriendly and can be an effective substitute for the large scale synthesis of zirconium oxide nanoparticles. FT-IR results are indicate the formation of  $ZrO_2$  nano particles. XRD pattern clearly illustrated that the  $ZrO_2$  nanoparticles formed in this present synthesis were identical and could be indexed to the standard  $ZrO_2$  with monoclinic structure (JCPDS no: 37-1484). The particle size calculated using Scerrer equation was 58 & 72 nm for sample 1 and 2. SEM study reveals that the average size. The strong intense and narrow width of Zirconium and oxygen EDX peaks indicates that the synthesized nanoparticles are of high purity with crystallinity.

## V. REFERENCE

- [1] P.Ramesh \*a, A.Rajendran a,M.Meenakshisundaram. ,(year-Feb 2014) Green Synthesis of Zinc Oxide Nanoparticles Using Flower Extract *Cassia Auriculata*, "Journal of Nano science and Nano technology", **Vol-2**, Page No-41.
- [2] S.K. KANNAN and M SUNDARAJAN, (year August 2015) Biosynthesis of Yttrium oxide nanoparticles using *Acalypha indica* leaf extract "Bull.mater.sci", vol 38,page No-945.
- [3] Kavitha K.S.1, Syed Baker1, Rakshith D.1, Kavitha H.U.1, YashwanthaRao H.C.1, Harini B.P.2 and SatishS.Herbal , (Year-June 2013), Plants as Green Source towards Synthesis of Nanoparticles "International Research Journal of Biological Science" Page No-66, **Vol-2(6)**,
- [4] AzoNano, "AZO Nano" Page No-2, **Vol-2**, (year-Apr 2013)
- [5] M.Ranjbar,M.Yousefi,M.Lahooti,A.Malekzadeh, ,(year-Dec-2012) Preparation and Characterization of Tetragonal Zirconium Oxide Nanocrystals from Isophthalic Acid-Zirconium(IV) Nanocomposite As a New Precursor "INT.J.Nanosci.Nanotechnol. **Vol-8 No.4**,Page No-194 .
- [6] K.Geethalakshmi, T. Prabhakaran and J. Hemalatha. ,(year-2012) Dielectric Studies on Nano Zirconium Dioxide Synthesized through Co-Precipitation Process, International Journal of Chemical ,Nuclear,Materials and Metallurgical Engineering" **Vol-6 No-4**, Page No-257.
- [7] MohammedRafiqHussainSiddiquia\*,AbdulaziWassila, Abdullah Mohmmmed, AlOtaibib, Refaat Mohamad Mahfouza, (year-22012) Effects of Precursor on the Morphology and Size of  $ZrO_2$  Nanoparticles, Synthesized by Sol-gel Method in Non-aqueous Medium "Material Research", **Vol-15 No-6** ,Page No-987,.
- [8] D. Gnanasangeetha and D. SaralaThambavani. (year-Nov-2013) Biogenic Production of Zinc Oxide Nanoparticles Using *Acalypha Indica* "Journal of Chemical,Biological and Physical Sciences" Page no-243, **Vol-14 No-1**,
- [9] K. Anandan and V. Rajendran. ,(year-2013). A Low-temperature Synthesis and Characterization of Tetragonal- $ZrO_2$  Nanoparticles via Simple Hydrothermal "Journal of Physical Science" Vol-17, Page No-182.
- [10] L.F.A. ANAND RAJ\* and E. JAYALAKSHMY. (year-2015), Biosynthesis and Characterization of Zinc Oxide Nanoparticles using Root Extract of *Zingiber officinale* "Oriental Journal of Chemistry **Vol-31,No-1** , Page No-55.