

# PRODUCTION OF ZrO<sub>2</sub> NANOPARTICLES FROM ZIRCON SAND OBTAINED BY SPRAY PYROLYSIS

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## ABSTRACT

Nonstabilised monoclinic nano ZrO<sub>2</sub> particles with an average particle (d<sub>50</sub>) size of 38 nm were synthesized from zircon sand using spray pyrolysis method. Zirconyl nitrate precursor has been obtained from raw zircon sand employing alkali fusion followed by acid extraction. The obtained zirconyl nitrate precursor was used as a starting material for nano ZrO<sub>2</sub> production. A comprehensive characterisation on nano ZrO<sub>2</sub> particles have been made using X-diffraction (XRD), Energy dispersive spectrum (EDS), Fourier transform infrared spectra (FTIR), Particle size analysis, BET surface area measurement, Scanning electron microscope (SEM) and transmission electron microscope (TEM) studies. The observed results confirm that the synthesized nano ZrO<sub>2</sub> particles have non-agglomerated spherical morphology with a specific surface area of 20 m<sup>2</sup> g<sup>-1</sup>.

## OBJECTIVES

- Extraction of zirconyl nitrate precursor from raw zircon sand
- Production of nano ZrO<sub>2</sub> particles employing spray pyrolysis using zirconyl nitrate precursor
- Characterisation of as produced ZrO<sub>2</sub> particles suited for industrial applications
- To promote nanotechnology in ceramics

## Production of nano ZrO<sub>2</sub> particles

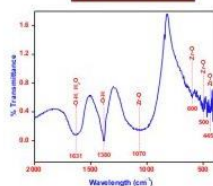
### Experimental set-up

- Atomiser - Converts the starting solution into droplets
- Automated anti-blocking unit
- Tubular electric furnace with hot air blower
- Two-fluid nozzle with compressed air inlet and sample feeding port
- Feed pump - Facilitates the flow rate of precursor
- Reaction chamber
- Cyclonic sample collectors
- Purification system

## Spray pyrolysis

- Homogeneous zirconyl nitrate solution is a starting phase
- Spray atomisation process employing compressed air
- Pyrolysis of nano sized zirconyl nitrate entities
- Decomposition reaction takes place at atomised zirconyl nitrate droplets at 723 K
- Ultra fine zirconia particles collected by cyclonic process

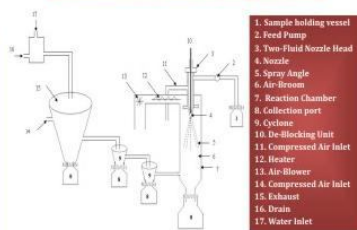
## FTIR SPECTRA



## SALIENT FEATURES

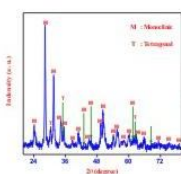
- Eco-friendly inexpensive method
- Continuous process - mass production
- Comparable chemical purity
- Controllable particles size
- High spherical morphology

## Schematic diagram of spray pyrolyser

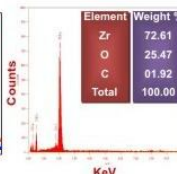


## RESULTS AND DISCUSSIONS

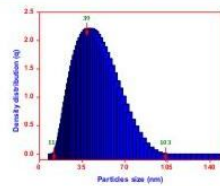
### XRD PATTERN



### EDS SPECTRUM

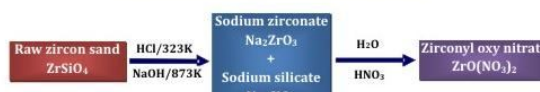


## PARTICLE SIZE DISTRIBUTION (PSD)

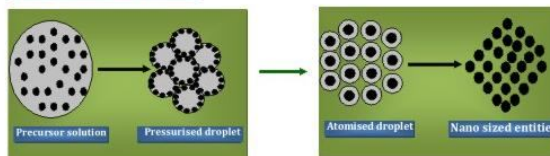


## EXPERIMENTAL

### Synthesis of zirconyl nitrate precursor



### Spray atomisation process



### XRD CHARACTERISATION

- Peak positions were recognised by JCPDS (File No. 81-1314 and 80-2155)
- Crystalline phase: 96% Monoclinic and 4% Tetragonal
- Average grain size: Monoclinic - 18.5 nm and Tetragonal - 25.9 nm

### EDS CHARACTERISATION

- The produced sample consists of 72.61 wt. % zirconium and 25.47 wt. % oxygen with the presence of residual carbon.
- ZrO<sub>2</sub> - 98.08 wt. %
- C - 1.92 wt. %

### FTIR CHARACTERISATION

- Bands at 600 cm<sup>-1</sup>, 445 cm<sup>-1</sup> and 500 cm<sup>-1</sup>, 1070 cm<sup>-1</sup> are the stretching frequencies of Zr-O-Zr vibrational bands
- Peaks at 1380 cm<sup>-1</sup> and 1631 cm<sup>-1</sup> are the O-H stretching and bending vibrations of ZrO<sub>2</sub>.xH<sub>2</sub>O and H<sub>2</sub>O molecules.
- 1631 cm<sup>-1</sup>: H<sub>2</sub>O & 1380 cm<sup>-1</sup>: ZrO<sub>2</sub>.xH<sub>2</sub>O

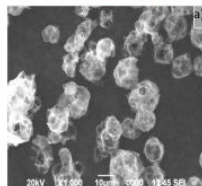
### PARTICLE SIZE DISTRIBUTION

- Finest particle (d<sub>min</sub>) = 11 nm
- Mean diameter (d<sub>50</sub>) = 39 nm
- Coarsest particle (d<sub>max</sub>) = 103 nm

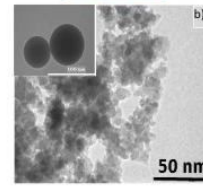
### SEM AND TEM CHARACTERISATION

- Spherical morphology
- Weak surface agglomeration
- 50 nm average particles size

## SEM IMAGE



## TEM IMAGE



## RESULTS SUMMARY

Sample characteristics	Crystalline phase	Crystallite size	Crystallite size distribution	Particles size	Particles size distribution	BET surface area	Pore volume	Morphology
Observation	96% Monoclinic 4% Tetragonal	Monoclinic - 18.5 nm	11 - 31 nm	d <sub>PSD</sub> = 39 nm d <sub>BET</sub> = 55 nm d <sub>TEM</sub> = 50 nm	11- 103 nm	20 m <sup>2</sup> g <sup>-1</sup>	0.0109 cm <sup>3</sup> g <sup>-1</sup>	Spherical

## CONCLUSIONS

An inexpensive and eco-friendly method for production of ZrO<sub>2</sub> nano powder has been developed using raw zircon sand directly. The precursors used in this process are zirconyl nitrate which is spray pyrolysed to obtain the nano ZrO<sub>2</sub> particles. The size characterisation was performed with XRD, PSA, TEM and BET studies. Monoclinic nano ZrO<sub>2</sub> powder with an average particle size of 39.59 nm with a specific surface area of 20 m<sup>2</sup> g<sup>-1</sup> through spray pyrolyser was demonstrated. Spray Pyrolysis is an effective method for bulk production ZrO<sub>2</sub> nano powder with high spherical morphology. The obtained nano ZrO<sub>2</sub> particles has many advantages when compared to other conventional processing routes such as low cost, simple process, continuous production and easy scaling for mass production of nano ZrO<sub>2</sub>.

## ACKNOWLEDGEMENT

The authors are very much thankful to Department of Science and Technology, New Delhi for the financial support to carry out this research project under Grant No. SR/S5/NM-40/2005 dt. 26.06.07.

