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**INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES**Available online at: <http://www.iajps.com>**Research Article****AUTO-COMBUSTION SYNTHESIS OF NANOCRYSTALLINE
BASNO₃****Sachin Bangale**Department of Chemistry, Gopinath Mahadav Vedak College of Science Tala- Raigad
402111(M.S.) India.**Abstract:**

BaSnO₃ powder was synthesized by combustion method, having metal nitrates as precursors and urea as fuel, followed by a heat treatment 700°C for 4 hr. The powders obtained have been characterized by thermal gravimetry differential analysis, X-ray diffraction; morphological of the powder was identified by high resolution- scanning electron microscopy and electric properties studied by DC measurement. X-ray diffraction results indicate that the resultant BaSnO₃ crystallites consist of spinal phase and average particle size of the nanomaterial BaSnO₃ calculated from XRD was found in the range ~ 47 nm. Electrical conductivity of the synthesized powder was studied by DC measurement. Electrical conductivity of the nanocrystalline BaSnO₃ was increased with increasing in temperature and semiconducting nature of BaSnO₃.

Keywords: TG/DTA, XRD, SEM, Combustion Method***Corresponding Author:****Sachin Bangale,**Department of Chemistry,
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1. INTRODUCTION

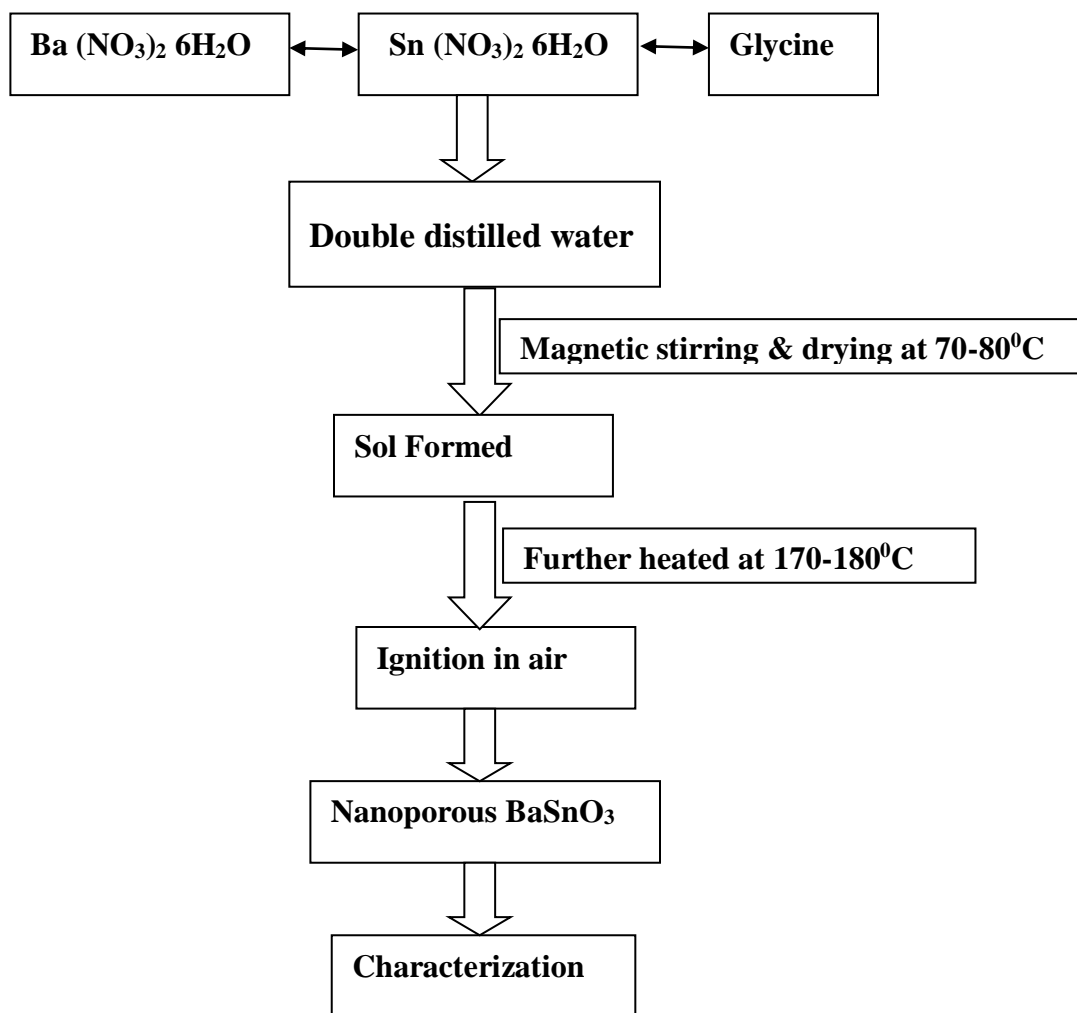
Combustion synthesis is an easy and convenient method for the preparation of a variety of advanced ceramics, catalysts and nanomaterials.[1] In this technique, based on the principles of the propellant chemistry,[2] a thermally induced redox reaction takes place between an oxidant and a fuel. Many types of combustion synthesis exist which differ mainly in the physical state of the reactants or in the combustion modality.[3-7] By combustion-based methods it is possible to produce monophasic nanopowders with homogeneous microstructure, at lower temperatures or shorter reaction times, if compared with other conventional methods like solid-state synthesis[8,9] or nitrate method[10,11]. Citrate-nitrate auto-combustion synthesis (CNA) is a very popular solution combustion method,[12,14] where the fuel is citric acid and metal nitrates are used as metal and oxidant source. CNA method shows high similarities with the very well known Pechini process [15,16] and it can be more properly described as a "sol-gel combustion method"[17]. Perovskite-type ABO_3 and related compounds have been reported to be of importance due to their wide uses in fuel cells[18], catalysts[19,20], membranes in syngas production[21], sensors[22,23] and environmental monitoring applications[24]. Gas sensing application [25, 26] etc. Among the chemical sensors $LaCoO_3$, $BaTiO_3$, $LaFeO_3$, $LaMnO_3$ etc. are perovskite-type materials of general formula ABO_3 are extensively studied owing to their notable gas sensitivity for different poisonous gases in addition to their magnetic, catalytic and other physical properties. The perovskite-type metal oxide including the d-block and rare earth elements has attracted the attention of many researchers due to their homogeneity, interesting structural, catalytic and gas sensing properties. There is an increasing interest in finding new materials in order to develop high performance solid state gas sensors.

We report here the synthesis of Oxide nanoparticles through combustion method which is a unique combination of the ignition and the chemical gelatin processes. This method has the advantages of simple preparation, cost effective and gentle chemistry route resulting in ultra fine and homogeneous powder. The ability to obtain single-phase Oxide magnetic nanoparticle with controllable particle size and distribution improves its adequacy in a wide range of technological application. $BaSnO_3$ nanoparticle are prepared by combustion method. The structural, thermal, morphological and Electrical properties are investigated.

2. EXPERIMENTAL:

2.1. Preparation of materials

For the present study, polycrystalline $BaSnO_3$ powder was prepared by combustion route using glycine as fuel. The materials used as precursors were Barium nitrate hexahydrate $Ba(NO_3)_2 \cdot 6H_2O$, $Sn(NO_3)_2 \cdot 6H_2O$ Tin nitrate hexahydrate (all these were procured from A.R. Grade of Qualligen) and Glycine (Nuclear band). Glycine possesses a high heat of combustion. It is an organic fuel and provides a platform for redox reactions during the course of combustion. Initially the Barium nitrates, Tin nitrates and Glycine are taken in the 1:1:4 stoichiometric amounts and dissolved in 250 ml beaker slowly stir with glass rod clear solution was obtained. Solution formed was evaporated on hot plate in temperature range $70^\circ C$ to $80^\circ C$ gives thick gel. The gel was kept on a hot plate for auto combustion and heated in the temperature range $170^\circ C$ to $180^\circ C$. The nanocrystalline $BaSnO_3$ powder was formed within few minutes and sintered at about $600^\circ C$ for about 4 hours got shining powder of nanocrystalline $BaSnO_3$ as shown in following sheet.



2.3 Characterization technique

The prepared BaSnO_3 samples were characterized using TG/DTA thermal analyzer (PERKIN ELMER, USA), X-ray diffract meter (RIGAKU MINIFLEX-II) using Cu-K α radiation, Scanning Electron Microscope (FEI QUANTA 200) coupled with an Energy Dispersive Spectrometer.

3. RESULTS AND DISCUSSION:

3.1 TGA Analysis

Figure 1 the first three intervals are entwined from 36°C to 350°C with broad endothermic peaks and a weight loss of 16%. These are attributed to the evaporation of residual water and burning of residual organic materials. The second from 400°C to 600°C with a rapid weight loss of 40% and a broad exothermic peak around 620°C, this is attributed to decomposition of the organic compounds. The synthesized powder was almost stable from the 600°C.

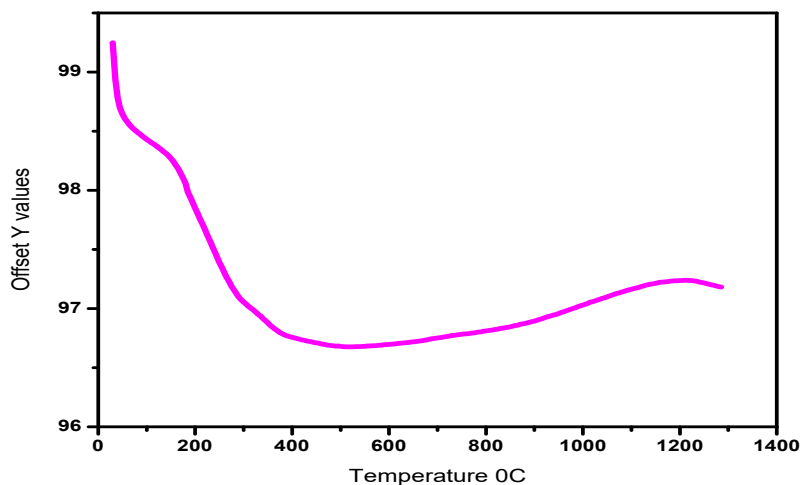


Fig. 1: Thermo gravimetric differential analysis curve of BaSnO₃ sample.

3.2 X-Ray Analysis

The X-ray diffraction pattern of BaSnO₃ powder is shown in Figure 2 the structure possesses the perovskite shape may be attributed to the different preparation method which may yield different structural defects. The crystalline size was determined from full width of half maximum (FWHM) of the most intense peak obtained by X-ray diffraction pattern. The grain size was calculated by using following Scherrer's formula.

$$d = 0.9\lambda / \beta \cos \theta$$

The crystalline size can be calculated by using Scherrer equation. Where, d is the crystalline size, λ is the X-ray wavelength of the Cu K α source ($\lambda=1.54056 \text{ \AA}$), β is the FWHM of the most predominant peak at 100 % intensity, θ is the Bragg angle at which peak is recorded. The grain size was found to be 47 nm.

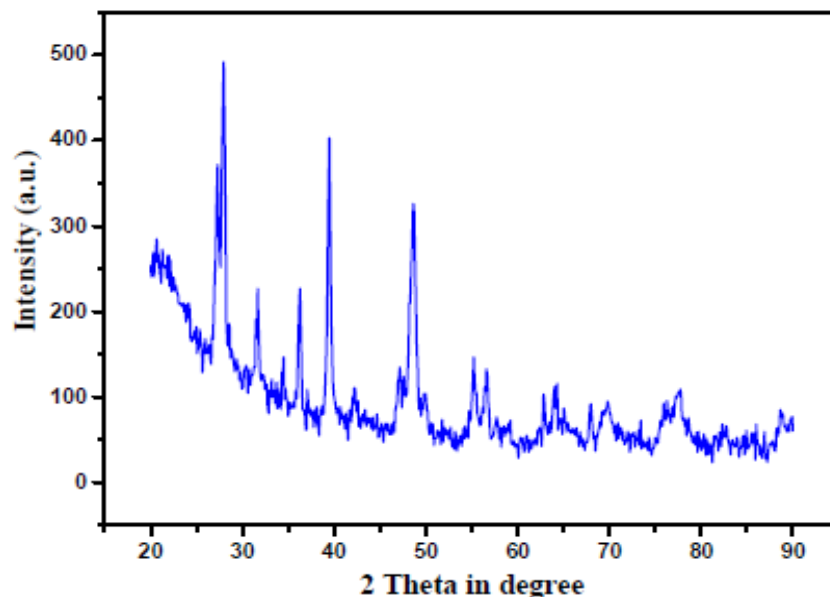


Fig 2: XRD pattern of calcined mixed precursor BaSnO₃ at 600°C, in air for 4 h.

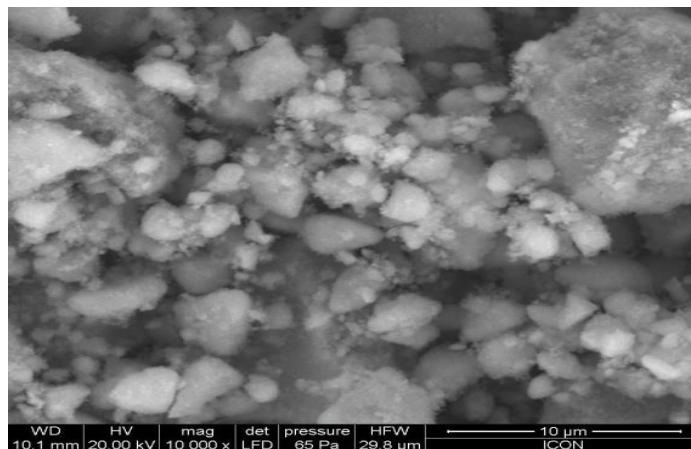


Fig. 3: SEM images of the self combustion product the powder annealed at 600°C.

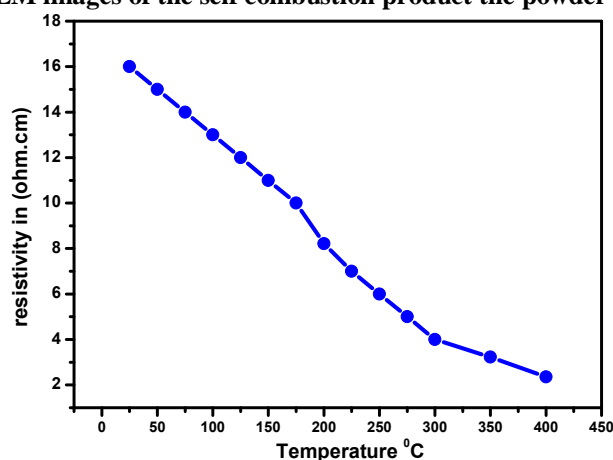


Fig. 4: Variation of log conductivity with temperature

3.3 Scanning electron micrograph analysis:

The microstructure of the sintered samples can be visualized from scanning electron microscope (SEM) tool. Figure 3 shown the particle morphology of high resolution, the particle are most irregular in shape with a Nanosize range. Some particles are found as agglomerations containing very fine particles the particles shapes are not defined porous nature and small and large core, spongy pores are seen in the micrograph.

3.4 Electrical properties of BaSnO₃

The variation of log (conductivity) with temperature. The conductivity values of sample increases with operating temperature. The increase in conductivity with increasing temperature could be attributed to negative temperature coefficient of resistance and semiconducting nature of BaSnO₃. It was observed from Figure 4 that the electrical conductivities of the BaSnO₃ films were nearly linear in the temperature

range from 50- 400°C in ambient air. The room temperature conductivity of BaSnO₃ was 3.45×10^{-2} mho/m.

CONCLUSIONS:

- ❑ Nanocrystalline BaSnO₃ has been synthesized by self combustion route. This synthesis route may be used for the synthesis of other metal oxide.
- ❑ The phase formation of the BaSnO₃ is investigated by TGA and XRD techniques. The synthesized product shows single phase of inverse perovskite type structure with an average diameter 47 nm.
- ❑ The increase in conductivity with increasing temperature could be attributed to negative temperature coefficient of resistance and semiconducting nature of BaSnO₃.

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