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Synthesis and characterization of nanosized zinc stannate for thermal application

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Nano sized bimetallic oxide materials have been extensively studied worldwide because of their unique properties and applications. One of the methods studied is the facial approach to preparing well dispersed nanocrystals of zinc stanate ($ZnSnO_3$) using Zn and Sn oxalate as precursors. Self-propagating high temperature synthesis method has been adopted using polymer fuel. Combustion processed $ZnSnO_3$ nanomaterials have been confirmed by various characterization tools. X-ray diffraction (XRD) tool is used to study the structure of the prepared bimetallic oxide nanomaterials sample. Morphological study of prepared sample is carried out by scanning electron microscope (SEM) analysis. Bonding nature of the sample has been studied by Fourier transform infrared (FT-IR) tool and presence of the Zn and Sn in the prepared sample has been identified by EDX analysis respectively. Absorption study of the prepared sample has been characterized by UV-Vis spectroscopy. Thermal behaviour of the bimetallic oxide sample has been analyzed by thermo gravimetric analysis (TGA), Raman spectroscopic (RS) study has been undertaken to view its structural organization. Dynamic light scattering (DLS) study has been implemented to know the size of the sample. Cyclic voltammetry (CV) study has also been used to know the electrolytic behaviour of the zinc stannate sample.

Keywords: Bimet¯allic oxide, $ZnSnO_3$ XRD, SEM, FT-IR, UV-Vis, DLS, RS, TGA

Perovskite-type metal oxides have been studied for many researchers because of their special properties and applications[1-3]. Doped perovskite oxides with metals have been studied extensively for their possibility of hydrogen storage applications[4,5]. Materials like $ZnSnO_3$ are known for proton conductivity in hydrogen and steam atmospheres at high temperatures[6]. In addition, this oxide material has higher hydrogen solubility than other metal oxides[7,8]. They are considered to be possible sensors for hydrogen or humidity and new class of materials for hydrogen storage[9]. $ZnSnO_3$ is a most important ternary bimetallic oxide material, which has attracted much in the field of materials science due to its unique properties such as high sensitivity, large specific area, non-toxic nature and good compatibility[10-12]. Different synthetic methods for synthesis of $ZnSnO_3$bimetallic oxide materials are listed in the literature, in which self-propagating high temperature combustion synthesis finds its importance because of its simple experimentation and easy scale up[13-15]. Tin-based mono metal oxides, bimetallic oxides, composite oxides such as SnO_2, Zn_2SnO_4, Mn_3O_4/Zn_2SnO_4, and $BSnO_3$ (B = Ca, Zn, and Co) are particularly used as an alternatives to traditional graphite anode in the next-generation LIBs, owing to their abundance and high theoretical capacities[16,17]. Among bimetallic oxides nanostructured $ZnSnO_3$ have attracted researcher's interest owing to their application in various fields such as chemical sensitivity, wide energy band gap, high transmittance percentage, electron mobility and non-toxicity. Further, $ZnSnO_3$ possesses remarkable morphological properties, making it an attractive material with diverse energy and biological applications[18,19]. The synthetic techniques employed greatly influence the structural and morphological characteristics of $ZnSnO_3$ which defines application oriented properties. Various factors, such as the capping agent, surfactant, reaction temperature, annealing temperature, concentration of metal precursors and reaction time play a major role in the development of different synthetic processes[20,21]. The different crystal structures of $ZnSnO_3$ have been extensively investigated for their special properties and applications[22]. Current research of zinc stannate is focused on the advancements in $ZnSnO_3$ nanostructures for thermal applications[23,24]. The growth mechanisms of $ZnSnO_3$ nanostructures

throughout the reaction period play an important role in the synthesis. Fabrication of $ZnSnO_3$ nano wire and the metastable phase adds complexity to the material. It can be observed that, the increasing in reaction time and overall available energy, the development of nanostructures and their corresponding significant phases[25]. The chemical stability of $ZnSnO_3$ material in RT signifies the material application in different fields. It is one of the photovoltaic materials and can find application in batteries, solar cells, sensors and catalysts have been reviewed by many researchers. In continuation, the energy storage capacity of $ZnSnO_3$ highlighted for the development of major application in energy storage systems[26,27].

The present work reports that, synthesis of nanosized zinc stannate by combustion method using polyvinyl-alcohol (PVA) as a fuel. Synthesized zinc stannate oxide sample was well characterized by various characterization tools. Thermal study of the sample is undertaken to view the thermal behaviour.

Experimental Section

Materials and methods

The chemicals used in the synthetic experimentation were of the analytical reagent (AR) grade and purified solvents are used in the preparation of solutions. Self-propagating high temperature combustion method was used for synthesis of nanosized zinc stannate. Self-propagating combustion reaction was progressed by PVA fuel.

Synthesis of zinc oxide and tin oxide

Zinc chloride mixed thoroughly with PVA in the weight ratio 1:5 and grounded well in a pestle and mortar to form complex mixing. Transfer this reaction mixture into a china dish and ignited in open air atmosphere until the complete evolution of carbonaceous products. Further, it is transferred into silica crucible and ignited in muffle furnace at 1000°C for 5 hours leads final zinc oxide as a residual product. This reaction occurs with the evolution of lot of gases and ignites automatically with self-propagation reaction. Similar procedure was used for the synthesis of tin oxide using its salt.

Synthesis of $ZnSnO_3$

The prepared zinc oxide and stannus oxide preliminary products were mixed thoroughly with PVA in the weight ratio 1:1:5 and ground well using pestle and mortar for homogeneous reaction mixture. This mixture was transferred into a china dish and was ignited initially for completion of preliminary combustion able products. Further, this pre product was transferred to a silica crucible and was ignited in a muffle furnace for its final phase. It is observed during the reaction that, initially PVA melted, then frothed and finally ignited at 1400°C for 8 hours for final $ZnSnO_3$ product. On cooling to RT no trace of carbon impurities was observed in the final oxide residue[28,29]. The ash colored final product of zinc stannate is as shown in Fig. 1 and its synthetic scheme is shown in Scheme 1. The possible chemical reaction involved in the complete combustion process is given below.

$$ZnO + SnO_2 \rightarrow ZnSnO_3$$

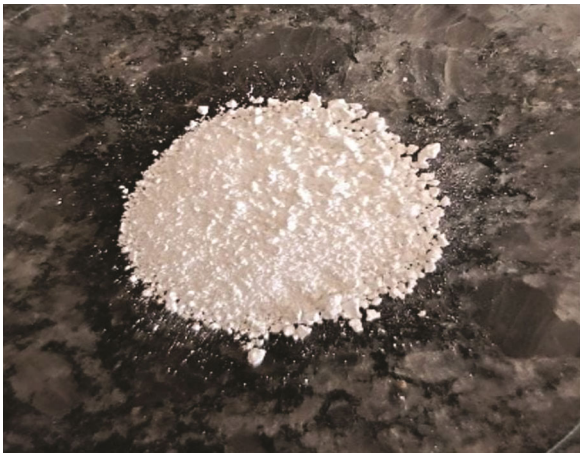
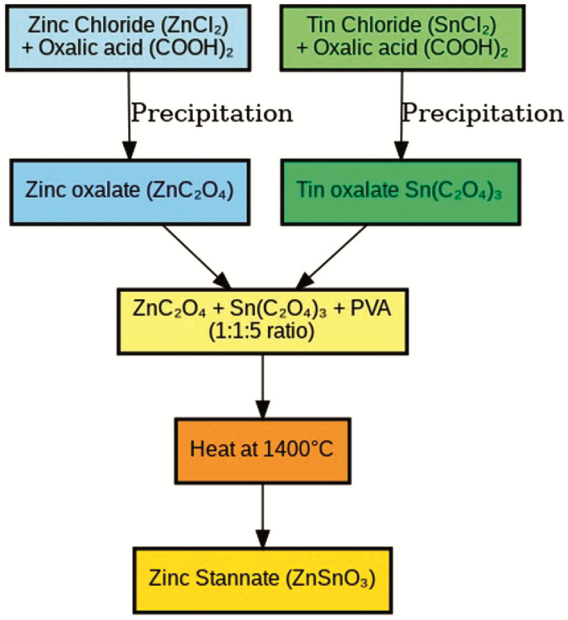


Fig. 1 — Optical image of $ZnSnO_3$ nanomaterials



Scheme 1 — Synthesis of $ZnSnO_3$ nanomaterials

Results and Discussion

Infrared studies

As formed zinc stannate sample was confirmed by knowing its bonding nature, which was analyzed by FT-IR instrumentation. Absorbed reflection of the sample Nano powders were done using KBr method at RT and is shown in Fig. 2. The broad absorption band appeared at 3450 cm^{-1} is attributed to the stretching vibration of O-H bond of moisture. Further, peaks appeared at 1550 and below 1000 cm^{-1} are attributed to the metal-oxygen (Zn-O, Sn-O) vibration mode. Metal-matal (Zn-Sn) vibration can also be viewed at lower frequency at 575cm^{-1} confirms the formation of the sample[30].

UV-Vis studies

Initial characterization of $ZnSnO_3$ nanomaterials sample was carried out using UV–Visible spectroscopic technique. Fig. 3 shows the UV-vis spectrum of combustion derived $ZnSnO_3$ nanomaterial sample. During combustion reaction the sample frames the additional bonding and will be confirmed by UV absorption of the sample product. Fig. 3 showed UV-visible spectrum of $ZnSnO_3$ nanomaterials to view and confirms the sample formation route. It showed an absorbance band at 370nm indicated that, the particles were polydispersity. It is also reflecting the characteristics of surface plasmon resonance of $ZnSnO_3$ nanomaterials. The good developments of the band indicate that the nanoparticles were successfully formed[31].

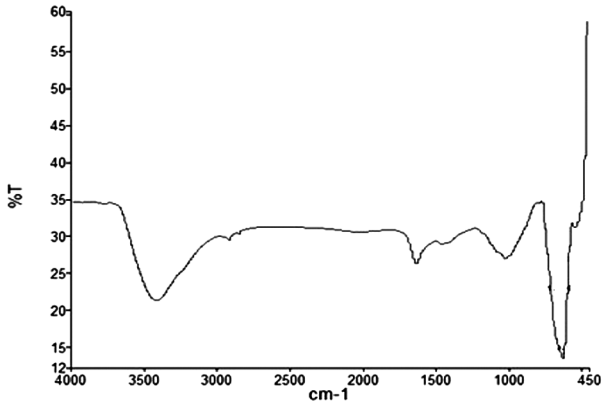


Fig. 2 — FT-IR spectra of $ZnSnO_3$ nanomaterials

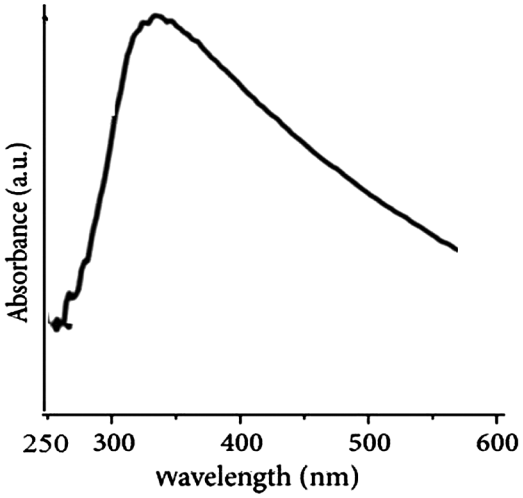


Fig. 3 — UV-Vis studies of $ZnSnO_3$ nanomaterials

SEM studies

Fig. 4 shows the SEM image of $ZnSnO_3$ Nano powder prepared by combustion reaction method. The sample shows most of the particles are nearly spherical in nature with size within nano range and formation of larger grains surrounded by small interconnected grains. The image also indicating the high crystalline nature of the sample with irregular morphology and some part shows open porosity.

EDX study

The EDX spectra obtained for $ZnSnO_3$ nanopowder is reported in Fig. 5. From the data, it is found that the specified elements were present in accordance with the literature data. It is observed in the pattern that, the presence of Zn, Sn and O atom signals at respective binding energy confirms the formation of $ZnSnO_3$ sample. No other elemental reflections other than the above observed indicates the purity of the prepared samples.

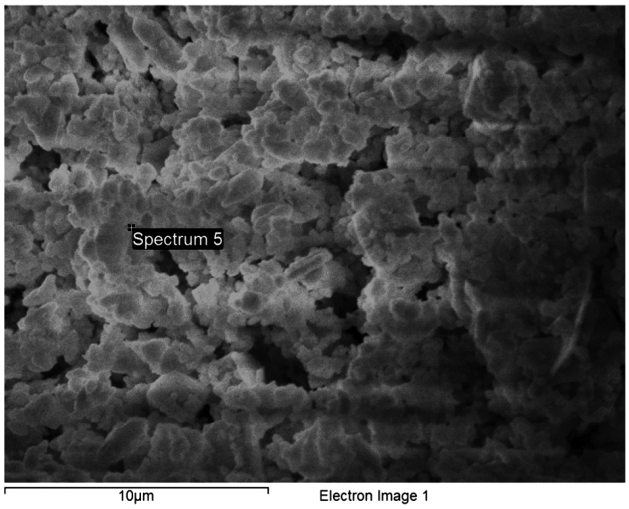


Fig. 4 — SEM image of $ZnSnO_3$ nanomaterials

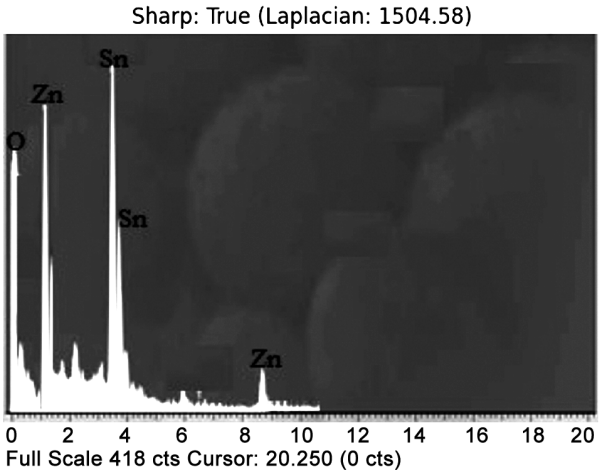


Fig. 5 — EDX pattern of $ZnSnO_3$nanomaterials

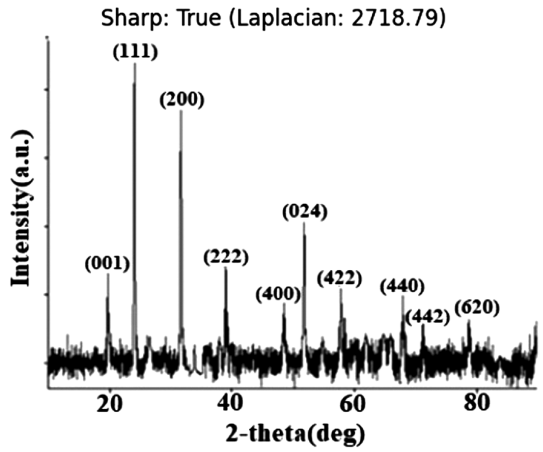


Fig. 6 — XRD studies of $ZnSnO_3$ nanomaterials

XRD study

Structural confirmation of the fabricated $ZnSnO_3$nanomaterial sample was carried out by X-ray diffraction method. Fig. 6 shows the indexed XRD pattern of as prepared $ZnSnO_3$ nanomaterial sample. The existence of (001) (111) (200) (222) (400) (024) (422) (440) and (620) Bragg's reflections revels the high crystalline nature of the sample. The absence of other reflections reveals the purity of the prepared sample.

The obtained 2Θ values of the sample are compared with 2Θ values of standard $ZnSnO_3$ nanomaterials with reference to JCPDS file 11-0274 of $ZnSnO_3$ nanomaterials. This comparison shows that, the well matching of diffraction data of sample with that of standard JCPDS data of $ZnSnO_3$nanomaterials confirms the sample. Further, obtained XRD data indicating that, the $ZnSnO_3$ sample prepared in the present work has cubic

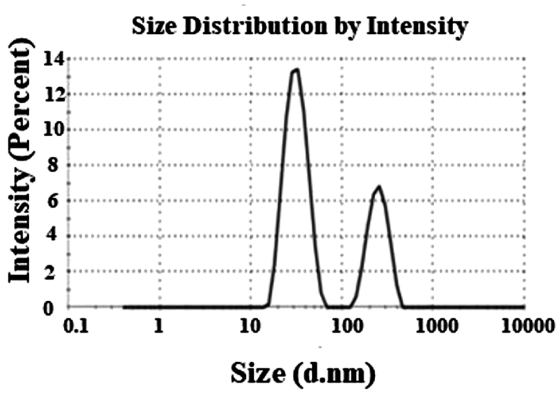


Fig. 7 — DLS study of $ZnSnO_3$ nanomaterials

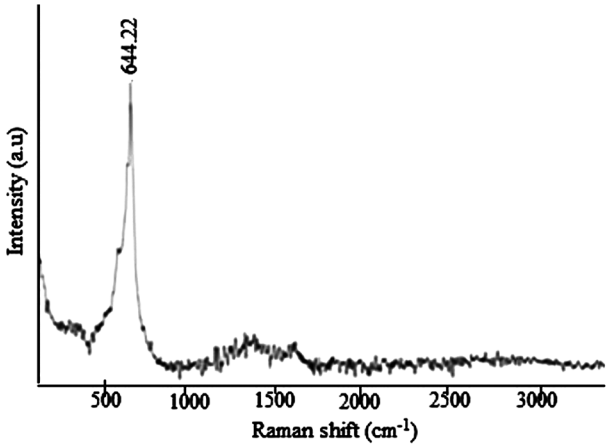


Fig. 8 — Raman spectra of $ZnSnO_3$ nanomaterial

structure calcined at high temperature. It is also shows an increase in peak intensities due to high crystallinity of the sample[32].

DLS study

The particle size distribution curves obtained for $ZnSnO_3$ sample prepared by combustion route are shown in Fig. 7. From the figures it was understood that the average particle size of $ZnSnO_3$ powder is found to be around the range of 60 to 80 nm. The presence of higher particle size may be due to the agglomeration of particles at high-temperature treatment.

Raman spectroscopic study

Raman spectroscopy is an important technique which can be used to study the chemical identification, characterization of molecular structures, effects of bonding environment and stress of the compound. In Raman spectrum of $ZnSnO_3$ nanomaterials derived with high temperature route, it is observed strong Raman shift peak at 644 $cm^{-1}$is an evident peak of the sample and is as shown in Fig. 8. No additional

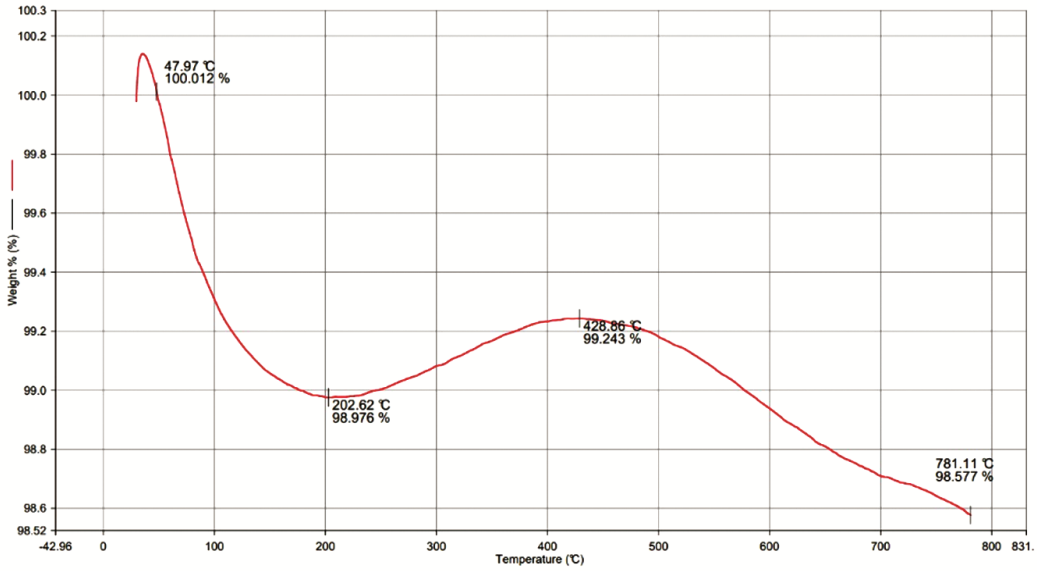


Fig. 9 — TGA trace of $ZnSnO_3$ nanomaterials

secondary phase of the sample is observed. Annealing at a high temperature induces dissolution of oxygen in the sample. The presence of all this peak with high intensity indicates the good crystallinity of the zinc stagnate. The bands can be described to the Zn-O on Sn-O vibrations for sub-surface species and O-O mode for adsorbed molecular oxygen[32].

TGA study

The TGA curves of the sample $ZnSnO_3$ was conducted from RT to1200°C and obtained trace is as shown in the Fig. 9. The thermal trace shows three main reason weight losses at different elevated temperature. Initial weight loss region temperature up to 47.97°C to 202.62°C occurs due to the removal of physical and chemical linked water moiety to the sample. In the second stage significant weight loss in the temperature range from 202.62°C–428.86°C can be attributed to the decomposition of the sample thermally. In the third region, the weight loss up to 781.11°C and it is expected that the only material changes that occur at this stage are in the crystal structure.

Conclusions

Combustion synthesis in the mode of self-propagating high temperature synthesis [SPHTS] takes advantage of the self-substituting merit from highly exothermic reaction and has the potential of energy and time savings. The nanosized $ZnSnO_3$ powder has been successfully synthesized for its phase formation by combustion route. This method offers advantages such as simplicity and relatively low processing temperature. The obtained samples exhibited desirable properties demonstrating the effectiveness of the combustion synthesis route, hence may be used for the synthesis of other bimetallic oxide materials. Further characterization and testing could provide additional insights into their potential uses. The structural analysis of the powder sample prepared by this method has shown desired structure. Thermal analysis of the sample concluding the high thermal stability and may be applicable for other bimetallic oxides.

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Conflicts of Interest

The authors declare no conflict of interest.

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