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# Analysis of conventional and microwave synthesized tin antimony nano particles

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**ABSTRACT**

Tin antimony (SnSb) is considered to be a promising anode material used in Li ion batteries. Tin antimony nanoparticles were prepared by co-precipitation and microwave oven method. Samples prepared by both the techniques were compared with each other by structural, morphological and electrochemical analysis. Crystallite size calculated from XRD data shows the decrement of particle size from 39 nm to 37 nm. But there were no remarkable changes occur in the structural and morphological results of the SnSb and doped SnSb samples. Both the samples exhibit tetragonal crystal system. Electrochemical analysis performed in the potential window of -0.6 V to + 0.6 V show well resolved redox peaks for doped SnSb.

**KEY WORDS:** SnSb, Microwave Oven, Nano particles, anodes.

## 1. INTRODUCTION

Intercalation/ insertion electrodes play an important role in the field of li ion battery. During charging/ discharging process, li ions from the cathode / electrolytes alloy with the electrode material. At this time the structure of the electrode material changes. The conducting species entered into the intercalation electrodes show very good electrical conductivity, they gain importance in the field of electrodes. Whittinghan's work exhibits preparation of different structures of intercalation electrodes.

Among varieties of intercalation electrodes, tin antimony anodes serve as a prospective material used in li ion battery system. Tin antimony anodes possess high theoretical capacity [994 $mAhg^{-1}$]. But they undergo structural deformation when lithium ions are intercalated/ de- intercalated into the system. To avoid this different solutions are suggested like minimizing the particle size, preparing inter-metallic alloys.

Here in our present work we have made an attempt to synthesis tin antimony nano particles by microwave method which is expected to show well resolved structure. Work done by Virender Singh Kundu (20130, involves only the heat treatment by microwave oven after synthesizing the antimony tin oxide by sol-gel technique. And the application of their work was to analyze the I-V characteristics of the microwave heated nano particles. In our present work, we tend to prepare/ reduce tin antimony nano particles in microwave oven and no post heat treatment was given after synthesis.

## 2. MATERIALS AND METHODS

In the preparation of SnSb by microwave oven, initially Solution A which contained metal chlorides like tin chloride and antimony chloride and tri-sodium citrate was treated with microwaves in domestic microwave oven at 180W for 5 minutes. Then solution B which contained reducing agent $NaBH_4$ and precipitating agent NaOH was treated in microwaves at 180W for 5 minutes. Then, solution B was added into solution a drop-wise followed by the microwave treatment at 180W for 5 minutes. The obtained precipitates were washed with dil.HCl, Acetone and distilled water. Thus washed metal nano particles were dried at atmospheric temperature and collected for further analysis. Preparation of tin antimony nano particles by simple wet chemical reductive co-precipitation technique was done as reported by Nithyadharseni (2014). This product was dried at ambient temperature and used for further studies.

## 3. RESULTS AND DISCUSSION

Thus synthesized powders were tested for structural analysis [Panalytical- XPertPro with Cu-Kα radiation], morphological analysis (SEM, -JEOL-JSM 6390), functional group identification [Shimadzu, 8400S], cyclic voltammetry analysis (Bio-logic, SP-150). The results obtained are discussed in the preceding sections.

**3.1. XRD Analysis:** X-Ray diffraction analysis was done for SnSb sample synthesized by conventional and microwave oven. On comparing with the standard patterns, the crystal system possesses rhombohedral crystal structure (JCPDS card no-33-0118). All the peaks are assigned to the SnSb alloy. The absence of parental materials peak in the XRD pattern for both the samples show the better stability of them. The lattice parameters calculated for both the samples are shown in table 1. The slight decrement in crystallite size is observed for the oven substituted SnSb sample. Comparing the XRD patterns obtained for both the samples, there are no remarkable changes observed for the oven synthesized SnSb sample. The c/a ratio is same for both the samples. Hence microwave synthesis doesn't affect the crystal system of the SnSb.

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**3.2. Morphological Analysis:** SEM image was taken for SnSb samples and it is shown in Fig 2. Comparing to normal synthesized sample, microwave synthesized SnSb shows well-arranged spherical particles. Since the uniform temperature involved in the heat treatment process agglomeration is minimum in SnSb-OVEN sample. Hence it is clear that microwave method helps to improve the crystalline structure of the nano particles. Also this method is beneficial to control the important parameters like particle size and shape.

**3.3. FTIR analysis:** FTIR graph shows the functional group comparison between the SnSb particles synthesized by both methods. Comparing to the regular synthesized SnSb, microwave oven assisted sample shows strong peaks which is shown in Fig 3. This shows that bonding between two atoms is getting stronger on heat treatment by microwave oven. As there are no possibilities for composition change in both the samples, only variation in moisture content is observed.

The broad absorption peak over the range of 3000-3500 $cm^{-1}$ is ascribed to the symmetric and asymmetric OH group. And the region around 1600$cm^{-1}$ is ascribed to the stretching and bending vibrations of O-H group. Very low impurities aroused from acetone is seen from the minor peak at 2930 $cm^{-1}$. The peaks at 1430 and 1360 $cm^{-1}$ are attributed to the bonding between Sn and Sb atoms. Also peak at 1070 $cm^{-1}$ is assigned to Sn-Sb bonding. So this validates that oven treatment does not alter the crystal structure. This statement gets validated on comparison with XRD data. As explained above, changes observed only in the intensity at the functional group region which is due to the moisture content.

**3.4. Electrochemical analysis:** Electrochemical analysis was done for SnSb samples prepared by both the methods. CV graphs obtained for both the samples are shown in Fig 4a and 4b. There is no new redox species formed. But OVEN treated SnSb exhibits well resolved redox peaks. This may be due to the structural refinement obtained in oven synthesis. The peak potential is independent of the scan rate which shows the reversible nature of the system. Also, the plot between square root of the scan rate and peak current exhibits the similar reversible behavior Fig. 5a and 5b. On the whole electrochemical performance, oven treated sample shows good reversible behavior. Well resolved peaks with standard reversible behaviors are observed in the CV graph.

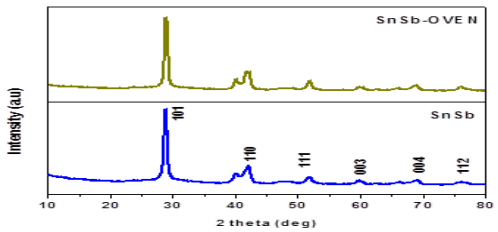


**Figure.1. XRD pattern of SnSb sample**

**Table.1.Lattice parameters calculated from XRD data for SnSb sample**

| Sample | Crystallite Size (by Scherer formula) nm | Lattice constants | | c/a Calc | c/a Exp | Dislocation density (x $10^{14}$ lines/$m^2$) | Strain | Volume of the unit cell ($Å^3$) |
|---|---|---|---|---|---|---|---|---|
| | | a | c | | | | | |
| **SnSb** | 39.57 | 4.4281 | 5.2881 | 1.1942 | 1.2361 | 6.3850 | 0.3263 | 89.7950 |
| **SnSb- Oven** | 37.03 | 4.4279 | 5.2845 | 1.1935 | | 7.2927 | 0.3856 | 89.7258 |

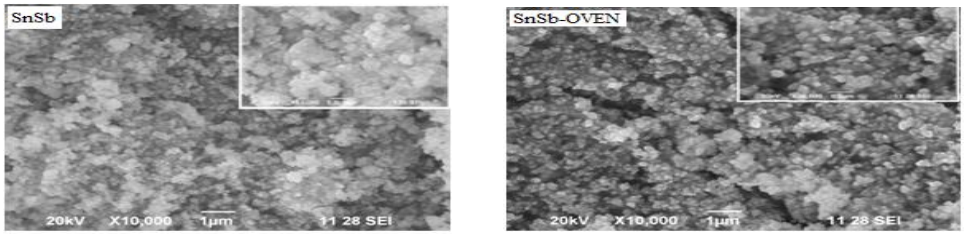


**Figure.2.SEM images of SnSb and microwave oven synthesized SnSb**

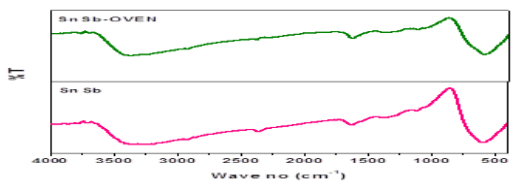


**Figure.3.FTIR spectra of SnSb and microwave oven synthesized SnSb samples**

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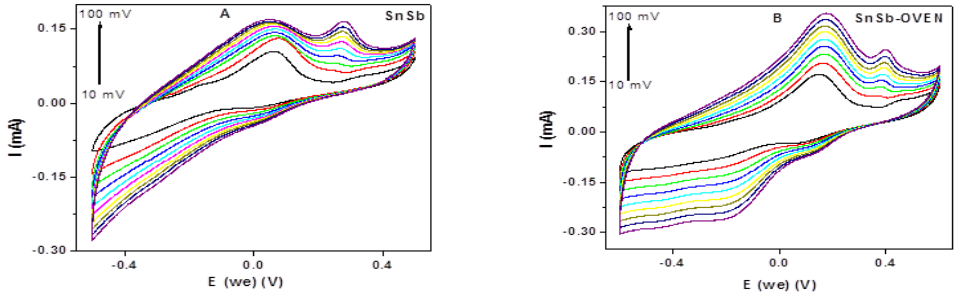


**Figure.4A and 4B.Cyclic voltammetry analysis of SnSb and SnSb-oven sample**

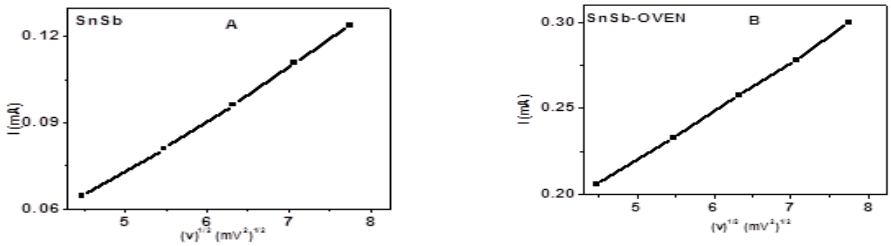


**Figure.5A and 5B. Plot between square root of scan rate and peak current values for SnSb samples**

## 4. CONCLUSION

Preparation and analysis of tin antimony nano particles by co-precipitation method and microwave oven was reported. The structural and morphological analysis reveals the well resolved structure of tin antimony nano particles by microwave synthesis. FTIR studies show the lesser moisture content for microwave oven synthesized SnSb nano particles. The CV analysis show that oven synthesized sample show the well resolved redox peaks.

## 5. ACKNOWLEDGEMENT

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