

**Research article** 

# International Journal of Scientific Research and Reviews

## Structural and Morphological Properties of Tin Oxide (SnO<sub>2</sub>) Nanoparticles by Microwave Irradiation Method

A.Ameer baig<sup>1\*</sup>, R.Vadamalar<sup>1</sup>, K.Gnanamoorthi<sup>2</sup>, K.Mohanraj<sup>3</sup> and P.Jayanthi<sup>1</sup>

 <sup>1</sup>PG & Research Department of Physics, Muthurangam Government Arts College (Autonomous) Vellore –632 002, Tamilnadu, India.
 <sup>2</sup>PG & Research Department of Physics, Pachamuthu College of Arts and Science for Women, Dharmapuri – 636 701, Tamilnadu, India.
 <sup>3</sup>Raman Research Laboratory, PG & Research Department of Physics, Government Arts College Tiruvannamalai – 606603, Tamilnadu, India.

## ABSTRACT

In the present work  $SnO_2$  nanoparticles have been successfully prepared by a microwave irradiation method. The crystallite size and morphology of  $SnO_2$  have been investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectrum (EDS), and transmission electron microscopy (TEM) techniques. The XRD pattern of average particle sizes of  $SnO_2$  is estimated to be around 14 nm. Furthermore,  $SnO_2$  nanoparticles have the crystallite size in the range ~11-50 nm, as confirmed by TEM. Results obtained indicate that the microwave-assisted method is a promising low temperature, cheap, and fast method for the production of  $SnO_2$  nanostructures.

KEYWORDS: Structural; XRD; SEM; TEM; Microwave irradiation method

## \*Corresponding author

## A. Ameer Baig

Ph. D Research Scholar
PG & Research Department of Physics
Muthurangam Government Arts College (Autonomous)
Vellore –632 002, Tamilnadu, India
Email: <u>ameerramphysics@gmail.com</u>

#### **INTRODUCTION**

If a particle size of a semiconductor becomes comparable to the Bohr radius of the exiciton, the ratio of the surface atoms to those in the interior increases remarkably, leading to the materials<sup>1</sup>. Two tin and four oxygen atoms per unit cell the ideally stoichiometric  $SnO_2$  is an insulator, however, the real SnO<sub>2</sub> structure contains oxygen vacancies, which make this material an oxygen deficient tin (IV) oxide is n-type semiconductor with band gap 3.6 to 3.8 eV which has rutile- type Cassiterite structure. As one of the most important classes of materials metal oxide semiconductor are presenting themselves in various are of science and technology due to<sup>2-3</sup> using a nanocrystalline metal oxide as an anode materials has many advantages because of the larger surface are and high sensitivity<sup>4-5</sup>. Tin oxide enjoys a place of pride because of its very high specific capacity (>600 mAh/g). SnO<sub>2</sub> is transparent conducting oxides (TCOS). SnO<sub>2</sub> has important physical properties such as a high optical transparency in the visible-light high electrical conductivity, low electrical resistance, good chemical and thermal stability in various environmental conditions. It is fully explored due to its potential applications in catalysis<sup>6</sup>. There are two obvious approaches to improve the gas sensing sensitivity and selectivity of  $SnO_2$  one strategy is doping with some rare earth ions or noble metals ions such like Y<sup>7</sup>, La<sup>8</sup>, Ce<sup>9</sup>, Pr<sup>10</sup>, Nd<sup>11</sup> and Sm<sup>12</sup>, another approach is to increase the specific surface area because SnO<sub>2</sub> is a surface-resistance-control semiconductor material for this reason, significant efforts including Nano structured tin oxides have been synthesized by a Varity of techniques such as sol-gel<sup>13</sup>, Ultrasonic<sup>15</sup>, spray Pyrolysis<sup>16</sup>, Thermal evaporation<sup>17</sup>, coprecipitation<sup>18</sup>, hydrothermal<sup>19</sup>, solve thermal<sup>20</sup>, SnO<sub>2</sub> different morphologies have been synthesized such as nanoparticles<sup>21</sup>, Nanowires<sup>22</sup>, Nanotubes<sup>23</sup>, Nanoroads<sup>24</sup>, Nanobelts<sup>25</sup>, Nanofibers<sup>26</sup>, Grasslike nanostructures<sup>27</sup>, Core-shell nanostructures<sup>28</sup>. The studies of the micro-wave heating are even complicated by fact that the rate enhancement of chemical reactions core depends on many complex factors<sup>27</sup> and the effects of several factors The usage of energy electronic devices has been increased in recent years due to the rapid growth of smart Phones, Smart watches, Camera, Laptop and personal digital assistant (PDAS) electrochemical capacitor has stood for energy storage system.

The electrode materials such as Carbon materials, Transition metal oxides and conducting polymers play an important role in the performance of super capacitors, Ru<sup>28</sup>, MnO<sub>2</sub> <sup>29</sup>, Co<sub>3</sub>O<sub>4</sub> <sup>30</sup>, NiO <sup>31</sup>, and SnO<sub>2</sub> <sup>32</sup>, are the promising electrode materials due to the high specific capacitance. Among various metal oxide nano particles, SnO<sub>2</sub> is inexpensive and Non-toxic in nature and Gas sensors<sup>33</sup>, Dye-based solar cells<sup>34</sup>, Light emitting diodes<sup>35</sup>, Transistors<sup>36</sup> etc. Such as volume<sup>37</sup>, solvent<sup>38</sup>, size of the reaction vessel and power level in micro-wave assisted organic synthesis. SnO<sub>2</sub> nanoparticles with sizes less than 30 nm were synthesized by a micro-wave assisted solution process

in which amorphous oxy-hydroxy precipitate  $sn^{2+}$  was crystallized by micro-wave heating.  $Sn^{2+}$  oxidation otherwise prevailed in the conventional thermal heating process. The micro-wave is highly efficient and energy saving in the process, including short reaction time, tiny energy consumption and product yield. The microwave assisted method offers to larger reaction volumes , allows faster reaction time and removes the need of high demands post synthesis of annealing , huge requirement of time and large energy consumption<sup>39</sup>. Microwave assisted synthesis effects rapid processing speed, homogeneous heating and simple control of processing condition and thus attracted much attention in last four years. Synthesis SnO<sub>2</sub> nanocrystals via microwave assisted process and demonstrated that anatase nanocrystals are highly crystalline, low in Ti<sup>3+</sup> effect and free of aggregation. The Chemical synthesis in a liquid phase through microwave irradiation mainly involves dipolar polarization and ionic conduction heating mechanisms. Nature of properties of the SnO<sub>2</sub> crystals depends on different kind of defects and impurities that are present in structure of material. These defects could affect its structural, electronic, optical and/or magnetic properties <sup>40</sup>.

In this paper reports on tin oxide synthesized by micro-wave irradiation method. The effect of conventional heating treatment. In this paper we focused a microwave irradiation with 2.45 GHZ frequency to obtain SnO and SnO<sub>2</sub> after calcinations, nanocrystalline powder in rapid volumetric heating which increased reaction rates and shortened the reaction time.

## **EXPERIMENTAL**

## Material

All chemical regents were of analytical grade and used without purification. The tin Chloride was purchased from (Merck product 98% AR grade) and ethanol solution (Merck product) were used to synthesis  $SnO_2$  nanoparticles sample was prepared by using double distilled water.

## Synthesis

A pure tin (II) (SnCl<sub>2</sub>.2H<sub>2</sub>O) was used as precursor, and ethanol was used as solvent. Solution of Sncl<sub>2</sub> in ethanol was prepared by a 0.1 M of solution under continuous stirred at room temperature until the transparent and colorless sol was obtained. The pH was maintained at 7 to 8 by using ammonia solution and continuously stirred. That sol further washed with ethanol to remove NH4<sup>+</sup>ions. The resulting precipitate was irradiated with house hold micro-wave oven for 5 minutes. The radiation frequency was kept between 2.45 GHZ to 1KW between to 1KW with convection mode. The finally, a white product was annealed at various temperature.

#### CHARACTERIZATION TECHNIQUES

The microstructure of the sample was analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument and the monochromatic CuK1 wavelength of 1.5406 Å. The average crystalline size of the crystallites were evaluated using Scherrer's formula, where is the mean crystalline size, is a grain shape dependent constant (0.9), is the wavelength of the incident beam, is a Bragg reflection angle, and is the full width at half maximum (FWHM) of the main diffraction peak. The sample morphology was observed by scanning electron microscopy (SEM), using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. High resolution transmission electron microscopy (HRTEM) and selected-area electron diffraction (SAED) was recorded on a Tecnai G20-stwin using an accelerating voltage of 200 kV.

## **RESULTS AND DISCUSSION**

#### X- Ray Diffraction (XRD)

The XRD pattern of tin oxide nanoparticles after microwave treatment and different annealing (or) sintering temperature (at  $300^{\circ}$ C -  $600^{\circ}$ C) showed the formation of SnO<sub>2</sub> nanocrystals due to the microwave radiation which changed the hydroxyl group into the oxide group. It was oxidant that the different matched perfectly with the SnO<sub>2</sub> tetragonal structure. When the sample was sintered at  $300^{\circ}$ C -  $600^{\circ}$ C, the particles were oxidized at SnO<sub>2</sub>. The sintered samples at  $300^{\circ}$  C- $600^{\circ}$  C showed typical SnO<sub>2</sub> tetragonal phase which could be attributed to following miller indices (110), (101), (220), and (211).



Figure No. 1: "XRD patterns of SnO<sub>2</sub> nanoparticles."

The obtained XRD patterns of tin oxide  $(SnO_2)$  nanoparticles were in good agreement with the standard JCPDS card no 88-0287.

No significant variations were observed while increasing sintering temperature from 300°C-600°C, but the lattice parameter tended to slightly decrease. Such behavior might be attributed to the complete dehydroxylation of the material suggesting the presence of hydroxyl group in the tin oxide crystallites sintered at 300°C-600°C.



## Scanning Electron Microscopy (SEM)

Figure No. 2: "SEM images of SnO<sub>2</sub> nanoparticles."

The Morphology of the synthesized nanocomposite was analyzed by scanning electron microscope. A typical micrograph of Samples (a), (b) and (c) are shown in fig.3. SEM images also show that the synthesized samples are agglomerated and smaller crystallites joint together.

Therefore the temperature distribution is uniform and is transformed the materials inside, making a volatile effect followed by vigorous growth of the gases to form  $SnO_2$  with good polycrystalline nature. It was observed that for each annealing temperature, the synthesized nanoparticles uniform surface without any significant defects and cracks. Surface of the particles consisted spherical particles fused each other.



## Energy Dispersive Spectrum (EDS)

Figure No. 3: "EDS spectrum of SnO<sub>2</sub> nanoparticles."

The spectrum of the synthesized nanocomposite was analyzed by energy dispersive spectrum (EDS). A typical spectrum of sample (a), Sample (b) and sample (c) in shown fig. This spectrum is performed to investigate the element composition of  $SnO_2$  nanostructures. EDS analysis confirms that the presence of  $SnO_2$  nanostructures. The emission peak such as O and Sn observed in EDS Spectrum shows the presence of tin and oxygen element and confirms the Stoichiometry of nanoparticles.



Transmission Electron Microscopy (TEM)

Figure No. 4: "TEM images of SnO<sub>2</sub> nanoparticles."

The Morphologies of the tin oxide nanoparticles were analyzed using TEM micrographs (fig 3.4). The  $SnO_2$  particles showed needle and spherical shaped morphology with different magnification such as 50 and 100 nm. The estimated particle size well matched with XRD measurements. Thus the larger particles are composed of many smaller particles. After annealing the sample, it showed that the mainly spherical shaped particles with sizes in range from 50 nm-100 nm have formed (fig 3.b, c). It appears that the spherical shape breaks in to the smaller nanoparticles annealing at 600° C for 12h in an air atmosphere.

#### **CONCLUSION**

Pure  $SnO_2$  nanopowders with tetragonal phase were successfully synthesized by microwave irradiation technique and no other impurity was observed it's confirmed by XRD. SEM morphology of nanoparticles showed good agglomeration with small crystallite joint together and EDS analysis confirms that the formation of  $SnO_2$  nanostructures. TEM image reveals clearly the formation of tetragonal face nanorods when the  $SnO_2$  sample annealed at 600 °C. The obtained  $SnO_2$  product is potential material for optoelectronic device applications.

#### REFERENCES

- Asma N, Naje, Azhar S, Norry, Abdulla, Suhail M. Preparation and characterization of SnO<sub>2</sub> nanoparticles. IJIRSET. 2013; 2: 2319-8753.
- Arnim Henglein. Small-particle research: physicochemical properties of extremely small colloidal metal and semiconductor particles Acs publications. Chem. Rev. 1989; 89: 1861-1873.
- Krishna M, Komarneni S. Conventional vs. Microwave Hydrothermal Synthesis of SnO<sub>2</sub>, SnO<sub>2</sub> Nanoparticles. Journal of Advanced Ceramics. 2014; 3: 171-176.
- Shuisheng Wu and Weili Dai. Microwave-Hydrothermal Synthesis of SnO<sub>2</sub>, -CNTs Hybrid Nanocomposite with Visible Light Photo catalytic Activity Nanomaterials. 2017; 210: 379-385.
- Qing Wang, Seigo Ito, Michael Grätzel, Francisco Fabregat-Santiago, Iván Mora-Seró, Juan Bisqurt, Takeru Bessho<sup>1</sup>, and Hachiro Imai. Characteristics of High Efficiency Dye-Sensitized Solar Cells. J. Phys. Chem. B. 2006; 110: 25210-25221.
- Jouhannaud J, Rossignol, Stuerga D. Rapid Synthesis of Tin (IV) Oxide Nanoparticles by Microwave induced Thermo hydrolysis. J. Solid State. Chem. 2008; 181: 1439-1444.
- Wei BY, Hsu, Su MC, Lin PG, Wu Hi-M, Lai R-J. A novel SnO<sub>2</sub> Gas sensor Doped with carbon nanotubes operating at Room temperature. Sens. Actuator, B.chem. 2004; 101: 81-89.
- Catherine Marichy, Patricia A. Russo, Mariangela Latino, Latino MG, Karpinsky M, Yu D, Neri G. Tin dioxide-carbon heterostructures applied to gas sensing: Structure dependent properties and general sensing mechanism. The Journal of Physical Chemistry C. 2013; 117: 19729-19739.
- Thorsten Wagner, Claus-Dieter Kohl, Michael Fröba and Michael Tiemann. Gas Sensing Properties of Ordered Mesoporous SnO<sub>2</sub> Sensors (Basel). 2006; 6: 318-323.

- Elango G, Kumarn SM, Kumar SS, Muthuraja S, Roopan SM. Green Synthesis of SnO<sub>2</sub> nanoparticles and its Photo catalytic Activity of Phenolsulfonphthalein Dye, spectrochim. Acta Mol. Biomol. Spectrosc. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 2015; 145: 176-180.
- Shukla S, Patil S, Kuiry SC, Rahman Z, Du T, Ludwig L, Parish C. Seal SS Synthesis and characterization of sol-gel derived nanocrystals line tin oxide thin film as hydrogen sensor. Sensors and Actuators B 2003; 96: 343-353.
- Fang LM, Zu XT, Li ZJ. Synthesis and Characteristics of Fe<sup>3+</sup> -doped SnO<sub>2</sub> Nanoparticles via Sol-Jell-Calcinations or Sol- Jell-Hydrothermal Route. Journal of Alloys and compound, 2008; 454: 261-267.
- Valentinus Paramarta, Ardiansyah Taufik, Lusitra Munisa and Rosari Saleh. Photo catalytic Activity of SnO<sub>2</sub> nanoparticles in Methylene Blue Degradation of cationic and anionic dyes. AIP Conference Proceedings. 2017; 1788: 030125.
- BileckaI, Niederberger. Microwave-chemistry for Inorganic nonmaterial's synthesis. Nanoscale 2010; 2:1358-1374<sup>-</sup>
- During Miao M, Liu Z Z, J hang M.Z, B.X AN, Miao G.M. microwave assisted process. ICESNANO 2016 AIP Conf. Proc. 2007; 7: 0301250-30125.
- 16. Wei-Chen Chang Hung-Shuo Chen and Wan-Chin Yu Flower-shaped ZnO nanocrystalline Aggregates synthesized through a template-free aqueous solution method for dye-sensitized solar Cells Appl. Phys. Lett. 2015; 106: 013908
- Hui-Chi Chiu, Chen-Sheng Yeh. Hydrothermal Synthesis of SnO<sub>2</sub> Nanoparticles and their Gas-Sensing of Alcohol. Phys. Chem. C. 2007; 111: 7256-7259.
- Rajesh N, Kannan JC, Krishnakumar T, Leonardi SG and Neri G. Sensing behavior to ethonal of tin oxide nanoparticles prepared by microwave synthesis with different irradiation time. Sensors. Actual B-chem. 2014; 194: 96-104.
- Delong Mei, Xianxia Yuan, Zhong Ma, Ping Wei Xuebin Yu, Jun Yang and Zi-Feng Ma
   A. SnO<sub>2</sub> Based Cathode Catalyst for Lithium-Air Batteries. ACS Appl. Mater. Interfaces. 2016; 8:12804-12811.
- Parthibavarman M, Sathishkumar S, Prabhakaran S. Enhanced visible light photo catalytic Activity of tin oxide nanoparticles synthesized by different microwave. Journal of Materials Science: 2018; Materials in Electronics - Ausgabe 3 optimum conditions.
- 21. Nehru LC, Swaminathan V, Jayachandran M and Sanjeeviraja C. Nanomaterial preparations by microwave-assisted solution combustion method and material properties of SnO<sub>2</sub> powder. Materials Science Forum. 2011; 671: 69-120.

- 22. Krishnakumar T, Pinna N, Bonavita A, Micali G, Rizzo G, Neri G. Microwave-Assisted Synthesis of Metal Oxide Nanostructures for Sensing Applications Sensors and Microsystems. Lecture Notes in Electrical Engineering 2011; 91.
- Eng HD and Hossenlopp M. Combined x-ray Diffraction and Diffuse Reflectance Analysis or Nanocrystalline mixed Sn (II) and Sn (IV) oxide powders. J. phys Chem. B. 2005; 109: 66-73.
- 24. Rajesh N. Microwave synthesis of cdo and SnO<sub>2</sub> nanostructures and comparison of structural, optical and dielectric properties. International journal of engineering applied sciences and technology. 2016; 1: 2455-2143.
- 25. Ahmed AS, Shafeeq MM, Singla ML, Tabassum S, Nagvi and Azam AH. Band gap narrowing and fluorescence properties of nickel doped SnO2 nanoparticles. Journal of Luminescence. 2011; 131: 1.
- Saeideh Ebrahimiasl and Azmi Zakaria. Simultaneous Optimization of Nanocrystalline SnO<sub>2</sub> Thin Film Deposition. Using Multiple Linear Regressions Sensors (Basel). 2010; 14: 2549-2560.
- 27. Paulo G, Mendes Mario L, Moreira Sergio M, Tebcherani Marcelo O, Orland J, Andre´s Maximu S, Li Nora Diaz-Mora Jose A, Varela Elson Longo. SnO<sub>2</sub> nanocrystals synthesized by microwave-assisted hydrothermal method: towards. J. Nanopart. Res. 2012; 14: 750.
- 28. Gnanam S, Rajendran V. Luminescence properties of EG assisted SnO<sub>2</sub> nanoparticles by sol-gel process. Digest Journal of Nanomaterials and Biostructures. 2013; 3: 699-704.
- Smritimala sarmah, kumar A. Opticle properties of SnO<sub>2</sub> nanoparticles. Indian journal of Physic. 2010; 84: 1211-1221.
- Mani R, Vivekanandan K, Vallalperuman K. Synthesis of pure and cobalt (Co) doped SnO<sub>2</sub> Nanoparticles and its structural, optical and photo catalytic properties. J Mater Sci. 2017; 5:
- Farrukh BT, Heng and Adnan. Surfactant controlled aqueous synthesis of SnO<sub>2</sub> nanoparticles. Turk J. chem. 2010; 84: 537-550.
- Gaber A, Abdel Rahim MA. Influence of calcinations temperature .on the porosity of SnO<sub>2</sub> by co-precipitation method. Int. J. Electrochem Sci. 2013; 9: 81-89.
- 33. Ashok K, Singh Umash T, Nakate. Microwave synthesis, Characterization and photo catalytic properties of SnO<sub>2</sub> nanoparticles. Advances in Nanoparticles. 2013; 2: 66-70.
- 34. Mendes PG, Moreia S, Tebcherani MLM, Orlandi JMO, Andre's MS, Diaz Mora LIN, Varela JA, ELongo. SnO<sub>2</sub>. nanocrystals synthesized by microwave assisted hydrothermal

method towards relationship between structural and optical properties. J. Nanopart. 2012; 14: 750

- 35. Singh AK, Nakate UT. Microwave synthesis characterization and photo catalytic properties of SnO<sub>2</sub> nanoparticles. Adv. Nanopart. 2013; 2: 66-70.
- Hossain A, Yang GW, parameswaran M, Jennings, Wang JR. SnO<sub>2</sub> Spheres synthesis by Electrochemical Anodization and their Application in Cdse-senstized solar cells. J. phys. chem. C. 2010; 114: 21878-21884.
- 37. Mailes Area MH, Boroojerdian P, Javadi Z, Zahedi S, Morshedian M. synthesis and non linear optical characterization of SnO<sub>2</sub> quantum dots. 2012; 123: 2090-2094.
- Habibzadeh S, Beydokthi AK, Khodadadi A, Mortazavi Y, Omanovic O, Nissar MS. Stability and thermal conductivity of nano fluids of tin dioxide synthesized via microwave – induced combustion route. chem. Eng. J. 2010; 156: 471-478.
- Vidhu VK, Philip D. Photosynthesis and applications of bioactive SnO<sub>2</sub> nanoparticles. Mater. Charact. 2015; 101: 9710.
- Nanda Kumar Reddy N, Harish Sharma Akkera, Chandra Sekhar M. Zr-doped SnO<sub>2</sub> thin films synthesized by spray Pyrolysis technique for barrier in solar cells. App. Phys. A. 2017; 123: 761.