SYNTHESIS OF TIN OXIDE NANOPARTICLES IN ORDER TO STUDY ITS PROPERTIES

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Pure tin oxide nanoparticles ware synthesized via Co-precipitation method. The as-synthesized nanoparticles were characterized by Fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), X-ray diffraction (XRD) and diffuse reflectance spectroscopy (DRS). X-ray diffraction shows that tetrahedral shaped nanoparticles of crystallite size 47.35nm were prepared successfully. The crystallinity was established due the reduction in distortion ratio and dislocation density. The value of strain showed that the nanoparticles fabricated were of high stability. The direct and indirect optical band gap of as-synthesized SnO_2 nanoparticles were determined from the reflectance spectra, which were 3eV and 2.65eV respectively.

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1. Introduction

Nanoscience is the study of matter's properties at a nanoscale. It is based on the special size dependent properties of solid state materials [1]. Nanoscience deals with objects of size having the smallest dimension ranging from a few nanometers to less than 100 nanometers. In the field of physics and electrical engineering nanoscience is most often related with quantum behavior, the behavior of electrons and photons in nanoparticles [2]. The word "nanotechnology" was the first time used by Norio Taniguchi and defines nanotechnology as ""Nano-technology" mainly consists of the processing of separation, consolidation, and deformation of materials by one atom or one molecule"[1]. Nanotechnology is the technology which works on level of nanometer (nm), one billionth of a meter. In the nano region the elements change properties unpredictably. It is generally related to mechanical engineering on a molecular scale, but it is a confusing term. It is sometimes also related to today's applied nanotechnology because of its numerous uses in cosmetics or industrial coating [3]. The term nanomaterial stands for the nano sized structures in which at least one of its phases has one or more dimensions, and they include nanometer sized particles ranging from 1nm to 100nm [4]. The properties of nanomaterial change due to its size, shape, and morphology. The structure of nanomaterials depends on the method of preparation, nature of solvent, concentration, strength of reducing agent, and temperature [5]. The nanomaterials are classified as zero dimensional (0-D), one dimensional (1-D), two dimensional (2-D), and three dimensional (3-D).

Nanomaterials have wide ranging applications in many areas of studies. Some of them are narrated below:

a. Nanomaterials are used for the elimination of dense metallic ions, and dyes from waste water organic, and inorganic pollutants [6].

b. Nanomaterials are used in agriculture, and crop production [7].

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c. Nanomaterials are used to improve molecular diagnostics such as gene detection, sensitivity, and practicality [8].

d. They are used to improve the properties of transistors [9].

e. Some nanomaterials such as solid lipid nanoparticles are used for skin hydration, and also have the ability to block UV-Rays [10].

f. Nano catalysts are used in the chemical, and pharmaceutical industries for enhancing the efficiency of energy in connection with green technology with the production of very less amount of chemical waste production.

g. Mesoporous silica shell nanospheres are widely used in hydrogenation catalysis, and magnetic acid catalysis[11]. They are also used for the enhancement of chemical reactions, and separation processes, and for simplification of the processes.

h. Metallic nanoparticles, quantum dots, and Nano bubbles have also been widely used in catalysis [12].

i. Consolidated nanocomposites, and nanostructures are used for making very high strength material with tough structure, tough pliable cements, and interestingly new magnets[13].

j. Nanostructured electrodes can enhance the efficiency, and capability of Li-ion batteries[14].

k. Nanotubes can be used as electro chemical actuators because of its super capacitance [15].

Amongst various nanomaterials being researched and used world over, Tin nanoparticles are also gaining attention of different research studies owing to its wide ranging applications in different fields. The research on Tin nanoparticles is extending with passage of time and with the rapidly changing advancements in different fields including, construction industry. Tin nanoparticles have a very broad and wide research area. The main reason is that Tin nanoparticles may be easily modified by changing their chemical properties.

Out of the many advantages of Tin nanoparticles, one key advantage is that their properties vary from the majority of the properties of the same composition. Tin nanoparticles may be coated onto other metals for corrosion prevention. Tin nanomaterials are widely used for many purposes, such as, enhancing the activities of super capacitors by providing high energy density to the capacitor [16], enhancing the gas sensing ability due to high sensitivity, response, and low-priced [17], enhancing the electrochemical properties of other materials such as improving the storage capacity of a battery [18], enhancement of photocatalytic activities of the materials owing to their good optical transparency, good intrinsic vacancies, and high density [19], acting as charge transport layer to increase the charge transformation, and for reducing hysteresis [20], cosmetics materials for protecting skin from UV-rays, and other rays effecting the skin [21].

Tin oxide (SnO_2) nanoparticles have been prepared by many methods by many researchers in recent years. These researchers use different types of precursors, and reagents for their work. Tin oxides prepared by these methods have many applications in different fields.

Zhu and co-authors (2000) used sonochemical method for synthesizing SnO_2 nanoparticles. They used $SnCl_4$ aqueous solution, and azodicarbonamide under ambient air in their work, and obtained Tin oxide nanoparticles of the size 3 to 5nm. The particles were spherical in shape, and had band gap of 4.05ev which is greater than the value of band gap (3.7ev) of SnO_2 bulk material. The prepared Tin oxide was used as in lithium ion batteries as reversible insertion electrode for enhancing the performance of battery, and increasing battery life [22].

Song and Kang (2000) used homogenous precipitation method for the preparation of SnO_2 powders. They used tin chloride pentahydrate, and urea in their work. The particles prepared were in powder form, and had large surface area. The particles prepared had size ranging from 3 to 5nm. Because of the large surface area of the SnO_2 powders prepared by this method, it was used in gas sensors materials, and for enhancing the sensing properties of devices [23].

Qui and co-authors (2004) prepared tin nanoparticles using polyol process by sonochemical method. They used tin chloride and ethylene glycol for their work. The particles prepared were rod shaped having size in range of 50 to 100nm, and length in range of 1 to 3μm. the prepared Tin oxide nanoparticles were used in lithium ion batteries as electrode material and for enhancing the performance of batteries [24].

Chiu and Yeh (2007) used hydrothermal method for the preparation of SnO_2 nanoparticles. They used tin chloride 2-propanol, and distilled water as solvent for their work. The prepared nanoparticles were tetragonal shaped having size of 3nm. The band gap of the particles was 3.6eV, and were used as sensing materials [25].

Bagheri-Mohgheghi and co-authors (2008) prepared SnO_2 nanoparticles using sol gel method. They used hydro-alcoholic solution, ethylene glycol as polymerizing agent, and tin chloride, and citric acid as complexing agent. The particles prepared were tetragonal shaped of size ranging from 5 to 25nm with a greater band gap from 4.05 to 4.11eV. The prepared nanoparticles were used for the agglomeration of particles [26].

Liu and co-authors (2009) used gel combustion method for the preparation of SnO_2 nanoparticles. They used Tin Chloride, and polyacrylamide gel in their work. SnO_2 nanoparticles prepared were sphere shaped, and had size in range of 8.1 to 27.9nm. The nanoparticles obtained by this process were used for the detection purposes of gases [27].

Krishnakumar and co-authors (2009) used microwave technique for the synthesis of SnO_2 nanoparticles. They used tin hydroxyl solution with hydrazine hydrate in a precursor solution for obtaining Tin Oxide nanoparticles. The prepared SnO_2 nanoparticles were having spherical shape, and size in range of 10 to 11nm [3].

Xu and co-authors (2010) also used the homogenous precipitation method for the preparation of SnO_2 nanoparticles. They used tin chloride, urea (carbamide), and aluminum oxide rod for their work. Tetragonal shaped SnO_2 nanosheets of size 10nm were prepared. The particles prepared indicated enhancement in high performance of sensing devices [28].

Gondal and co-authors (2010) prepared tin oxide nanoparticles using highly powerful laser beams. They used tin metal as target material placed in distilled water, and highly powerful laser in their work. The tin oxide nanoparticles prepared were tetragonal shaped of size 3nm, and a band gap of 4.4eV [29].

Parthibavarman and co-authors (2011) prepared SnO_2 nanoparticles using microwave irradiation method. They used tin chloride and $NH_3.H_2O$ in their work. The prepared SnO_2 nanoparticles were spherical shaped with size in range of 20 to 25nm with a band gap of 3.7eV. The acquired nanoparticles were used for highly susceptible environmental controlling and in electronic devices for controlling humidity[30] .

Patil and co-authors (2012) also used hydrothermal method for the synthesis of SnO_2 nanoparticles. They used hydrazine hydrate as a mediator for this process. The obtained SnO_2 nanoparticles were of tetragonal shape. The obtained particles were having size 22.4nm, and a band gap of 3.6ev. SnO_2 prepared by this method can be used as gas detectors in many gas detecting devices [31].

Honghao and co-authors (2013) prepared SnO_2 nanopowder through gas phase detonation reaction. They used stannic chloride as precursor, and mixture of hydrogen, and oxygen gas. The SnO_2 nanopowders prepared were having size of 1 to 10nm, and have spherical morphology. Further details were not discussed as gas phase reaction was not announced by this method. Although, prepared SnO_2 nanopowders were having high sensitivity [32].

Naje and co-authors (2013) used chemical precipitation method for the preparation of SnO_2 nanoparticles. They used tin chloride and ammonia solution in their work. The prepared SnO_2 nanoparticles were in powder form having tetragonal shape, and size was 8 to 10nm with a band gap of 4.3eV higher than that of the band gap of bulk SnO_2 [33].

Nayef and Hadi (2014) used laser ablation technique for the synthesis of SnO_2 nanoparticles. They used tin, and methanol for this process. The prepared particles were spherical shaped, and their size varied according to different experimental conditions. The band gap of the particles was greater than that of bulk SnO_2, and was observed at different laser concentration in range of 4.16 to 4.26eV [34].

Srivastava and Mukhopadhyay (2014) used biological method for the synthesis of SnO_2 nanoparticles. They used *Erwinia herbicola* (Gram-negative bacteria), and tin chloride for their work. The SnO_2 nanoparticles prepared were tetragonal shaped having size in range of 15 to 40nm. The prepared SnO_2 nanoparticles showed good photocatalytic behavior, and were used for the photodegradation of many organic dyes [35].

Elango and co-authors (2015) used green synthesis for the preparation of SnO_2 nanoparticles by green synthesis technique. They used plant's (Persia Americana) seed extract, and stannous chloride as precursor for their work. The SnO_2 nanoparticles they obtained were tetragonal shaped with size in range of 4nm, which were used for the degradation of dyes [36].

Bhagwat and co-authors (2015) also used sol-gel method for preparing SnO_2 nanoparticles. They used tin chloride, ethylene glycol, and liquid ammonia as precursors, and double distilled water as solvent. The prepared particles were having size in range of 350 to 800nm, and a band gap of 3.78ev [37].

Azade Esmaeili-Bafghi-Karimabad and co-authors (2015) prepared SnO_2 nanoparticles using simple chemical reaction method. They used tin chloride, and dimethylamine as capping agent. The particles prepared were tetragonal shaped having size 40nm with a greater band gap of 3.82eV. These prepared particles were used for enhancing the thermal stability and reducing the flammability of materials [38].

Nagirnyak and co-authors (2016) prepared SnO_2 nanoparticles by using chemical deposition method. They used tin chloride, and ammonium oxalate as precursors, and prepared two samples of SnO_2 nanoparticles of size in range of 40 to 200nm, and hexagonal shape. The band gap was 3.69eV for one sample, and 3.78eV for other. The prepared materials were used as sensing materials, and for enhancing the properties of gas sensors [39].

Kurniawan and Rahmi (2017) prepared SnO_2 nanoparticles using electrochemical method (electrolysis) at a large potential. They used tin bare as electrodes (both anode, and cathode), and hydrochloric acid (HCl) as the electrolytic solution. The particles produced by this method showed tetragonal shape, and size in range of 25 to 150nm [40].

Oliveira and co-authors (2018) prepared SnO_2 nanoparticles using solvothermal method using indium chloride tetrahydrate, and tin chloride pentahydrate as precursors, and ethylene glycol as solvent. Spherical shaped particles of size in range of 3.4 to 8.2nm with a band gap of 3 to 3.4ev were obtained [41].

Kim and co-authors (2018) used the liquid phase plasma method for the preparation of SnO_2 nanoparticles. They used tin chloride as a precursor, cetyl trimethyl ammonium bromide, and water as solvent. The SnO_2 nanoparticles were of spherical shape with size in range of 5 to 10nm. These particles were used for enhancing the storing capability, and life time of lithium ion batteries [42].

Diaz and co-authors (2018) prepared SnO_2 nanoparticles by using two step solid state synthesis method. $SnCl_2$.Chitson at different ratio of 1:1, 1:5, and 1:10 with dichloromethane (CH_2Cl_2) as solvent were used for this process. Rod like nanostructures of SnO_2 of size 2.2 ± 0.2nm using precursors at 1:5. They used the SnO_2 prepared by this method in optoelectronic devices, and solid state sensor devices for enhancing the mechanical properties of these materials, and upgrading their performance [43].

2. Materials and methods

All analytical grade reagents were used. The solutions were prepared in distilled water (E.C. 2.8µS/cm) which was obtained from the distillation apparatus (Model WSB/4, Hamilton Laboratory Glass Ltd.). Tin chloride dihydrate ($SnO_2.2H_2O$) (Merck) was used as a precursor. Ethanol (Merck) was used as a solvent.

2.1. Synthesis of tin nanoparticles

Co-precipitation method was used for Tin oxide nanoparticles synthesis. Two solutions were mixed forming a precipitate, which was then washed, annealed to obtain the desired product. Two approaches using ethanol as solvent, and using ethanol and water as solvent, were adopted using the same method, to prepare two samples of SnO_2 nanoparticles. Detailed procedure is as under:

a. 1.12825g of tin chloride, and 50ml ethanol were taken in a beaker, and mixed with the help of a magnetic stirrer for about 10 to 15minutes to obtain a complete soluble, and transparent solution.

b. 0.4g of sodium hydroxide was dissolved in 100ml of deionized water with the help of a magnetic stirrer, and sodium hydroxide solution was obtained (0.1M solution)

c. The sodium hydroxide solution was dropwise added to the prepared tin chloride solution at constant stirring as precipitating agent, and controlling pH of the solution. With the addition of sodium hydroxide solution, precipitate was formed in the solution, and the pH value reached 8 with the addition of 109ml of sodium hydroxide solution

d. As the pH of the solution was maintained, further stirring was stopped, and the precipitate was allowed to settle down by keeping the solution still for 12hrs

e. After 12hrs the precipitate got concentrated at the bottom, and the ethanol above the precipitate was decanted.

f. Ethanol was added to the precipitate for washing, and then again kept for 12hrs to settle down. After 12hrs the ethanol was again removed. This process was repeated three times, and at last a pure precipitate was obtained.

g. The precipitation was dried on heating at 90°C for 12 hours.

h. After heating, a solid matter was obtained which was grinded into powder and annealed at 800°C for 3 hours.

i. After annealing we obtained our desired product which was further investigated by different characterization techniques.

3. Results and discussions

3.1. FTIR

In the FTIR spectroscopy graph the band from the 3394 to 3409 cm^{-1}shows the streching vibration of O-H bond .This band is due to the OH groups and the adsorbed water bound at the SnO_2 surface. The band at 1620 to 1630 cm^{-1} matched to the bending vibration of water molecules, caught in the SnO_2 sample. The peak at 521cm^{-1} matching with the stretching vibrations of the terminal Sn-OH and at the peak 660 to 600cm^{-1} region shows the stretching modes of the Sn-O-Sn. Due to increase in temperature the peak is also broadened (Fig. 1).

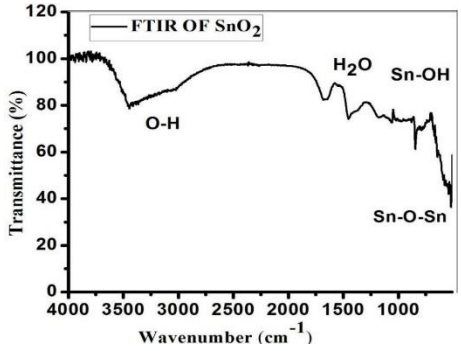

Fig. 1. FTIR analysis of SnO_2 nanoparticles.

3.2. TGA (thermogravemetric analysis)

The TGA analysis was carried out and it was observed that at 50 to 120 degrees increase in weight occurred because of oxidation of Sn^{+2}, because of this oxidation the temperature of the furnace increased and the TGA curve was distorted. The constant weight condition was reached near 300 C° after which the weight goes on decreasing slowly as the temperature increased. At 425° to 600° the weight loss further increased because of evaporation of OH^{-} and water molecules and above 600° the weight loss suddenly increased as shown in the figure.

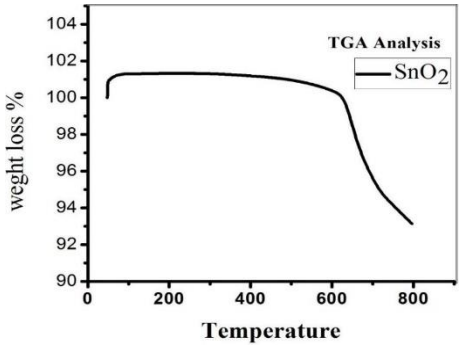

Fig. 2. TGA OF SnO_2.

3.3. XRD

The XRD pattern of SnO_2 nanoparticles synthesized by co-precipitation method is given below. All of the peaks were matched with diffraction data of the tetragonal structure of tin oxide (JCPDS 01-077-0447). Position of the main peak is 2θ= 26.68 and the width of the peak is 0.933 nm.

The average crystallite size (D) of the nanoparticles was estimated using the Debye Scherrer formula as follows:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad \text{(i)}$$

where, λ, β, θ are the X-ray wavelength of radiation used. The averge crystallite size (D) determined from SnO_2 different plane by the Scherrer formula is about 47.35 nm and tetrahedral in shaped.

The average dislocaton density of SnO_2 particles is determined to be $7.28\times10^{14}/m^2$ by the equation.

$$\text{Dislocation density} = 1/ D^2 \quad \text{(ii)}$$

The average strain was determined to be 0.206 by the equation.

$$\text{Strain} = \beta/4 \tan\theta \quad \text{(iii)}$$

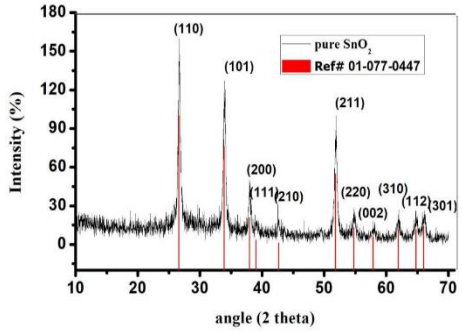

Fig. 3. XRD analysis of SnO_2.

Table 1. The detailed analysis of SnO_2 by XRD.

No.	$2\theta_B$	θ_B	$2\Theta_1$	$2\Theta_2$	$\beta=\frac{2\theta 2-2\theta 1}{2}$ radian	Interplanar Spacing d (A°)	$D=\frac{0.9\lambda}{\beta \cos\theta B}$ (nm)	Miller Indices (hkl)
1	26.6883	13.3441°	26.314°	27.247°	0.4665	3.3409	30.5	(110)
2	33.9608	16.9804°	33.541°	34.473°	0.466	2.6397	31.1	(101)
3	38.0033	19.0016°	37.737°	38.436°	0.3495	2.6677	41.9	(200)
4	42.6432	21.3216°	42.516°	42.749°	0.1165	2.1207	127.7	(111)
5	51.8903	25.9451°	51.374°	52.540°	0.583	1.7621	26.4	(210)
6	54.8388	27.4194°	54.521°	55.104°	0.2915	1.6741	53.3	(211)
7	57.9546	28.9773°	57.668°	58.367°	0.3495	1.5913	45.3	(220)
8	61.9659	30.9829°	61.631°	62.564°	0.4665	1.4976	34.6	(002)
9	64.8092	32.4046°	64.312°	65.244°	0.466	1.4386	35.2	(310)
10	66.1391	33.0695°	65.827°	66.526°	0.3495	1.4116	47.3	(112)

Average particle size = 47.35nm

Structure = Tetrahedral

JCPDS Card # = 01-077-0447

$$\text{Strain} = \beta / 4 \tan\Theta$$

Average strain = 0.206

Dislocation density = $1 / D^2$

Average Dislocation density= 7.28×10^{14}/m

3.4. Optical properties

The optical properties were studied by using diffuse reflectance spectroscopy (DRS). The reflectance spectra of the pure SnO2 nanoparticles are as shown in the figure 4. From the spectra, it is observed that he quantum confinement effect increases with increase of reflectance. The decrease in reflectance near the 300nm is due to the optical transition happening in the optical bandgap. The synthesized SnO2 showed no reflection up to 290nm and washighly reflective above the region of 320 nm.

The optical bandgap was determined by kubelka-munk relation as shown below, which is commonly used to study diffuse reflectance spectroscopy.

$$F(R) = \frac{(1-R)2}{2R} = \frac{K}{S} \quad \text{(iv)}$$

where F(R) is the kubelka-munk function which relates to the absorbance, R is the reflectance, k is the absorbance co-efficient and S is the scattering co-efficient. The optical transition in semiconductors are of two types, the direct and indirect transitions. Therefore direct and indirect bandgap determined by this relation as shown the figure 5 are 3eV and 2.65eV respectively.

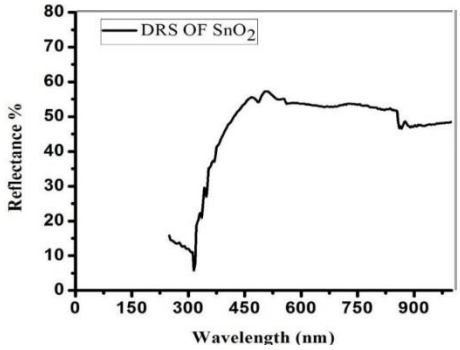

Fig. 4. Reflectance spectra of SnO_2.

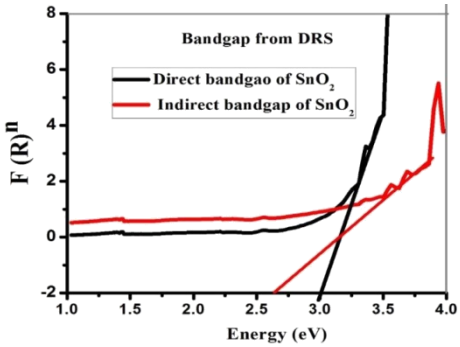

Fig. 5. Bandgap of SnO_2 from reflectance spectra.

4. Conclusions

SnO2 nanoparticles have been effectively prepared by Co-precipitation method using tin chloride (SnCl) and sodium hydroxide (NaOH) at 800°C. The Fourier transform spectroscopy (FTIR) illustrates that chlorine impurity was entirely removed by washing process. The TGA analysis showed that sudden mass change occurred at 630°C, showing the breakdown of tin oxide. The structural and optical properties were examined by X-Ray diffraction (XRD) and diffuse reflectance spectrum (DRS). X-Ray diffraction (XRD) analysis showed that samples were well crystalline and tetrahedral in shape having average crystallite size of 47.35nm. The direct and indirect band gap was found to be 3eV and 2.65eV respectively.

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