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Synthesis and Characterization of Tin Oxide Nanoparticles by Co-precipitation Method

L.I.Nadaf[1], K.S.Venkatesh[1]
[1]*Department of Material Science, Gulbarga University, Kalaburgi,India.*

Abstract: *Present study shows synthesis of Tin Oxide (SnO_2) nanoparticles by simple precipitation method at room temperature. SnO_2 nanoparticles were synthesized by using hydrated stannic chloride ($SnCl_4..5H_2O$) as precursor and sodium carbonate as precipitating reagent. Triton X-100 and Lauryl alcohol were used as surfactants. The precipitate was calcinated at 325^0C for one hour. The size of the as prepared particles of SnO_2 with and without surfactants was estimated using X-ray diffraction(XRD). The particle sizes with surfactants are found to be less than that without surfactants.*
Keywords: *Co-precipitation Tin Oxide nanoparticles, SnO_2 with surfactant,*

I. Introduction

In recent years nanostructure metal oxides have attracted a lot of attention due to their technological applications and outstanding properties .The magnetic, optical, catalytic and electronic properties of nonmaterial depend strongly on size, structure and shape of nanoparticles. Another reason for attraction of scientists' attention towards nano size particles is that, they behave differently from bulk materials. With decreasing particle size the band structure of the semiconductors changes .The band gap increases and band edges splits into decrease energy levels.

Recently the research on tin oxide semiconductor has been growing due to the wide range of applications including gas sensors, transistors, electrodes, liquid crystal displays, catalysts, photovoltaic devices, photo sensors, antistatic coating etc.,[1-5] Tin oxide is one of the most important material [6] due to its high degree of transparency in the visible spectrum, strong physical and chemical interaction with adsorbed species, low operating temperature and strong thermal stability in air up to 500^0 C. Tin occurs in two oxidation states +2 and +4, therefore two types of oxides are possible i.e. stannous oxide (SnO) and stannic oxide(SnO_2). Among these two oxides, SnO_2 is more stable than SnO.

Literature reveals that SnO_2 nanoparticles have been synthesized by varies methods like Sol Gel [7] Micro Wave technique [8] Solvo-thermal [9], Hydro thermal [10] Sonochemical[11] Mechanochemical[12], Co-precipitation[13]etc., In the present work pure and highly crystalline nanoparticles of SnO_2 have been synthesized using Co-precipitation method. Co-precipitation method has been employed because it is simple, inexpensive and does not require high temperature and pressure. In this method the size and shape of the particle can be controlled by altering pH of the medium, concentration of the precursor and precipitating reagents. Impurities in the precipitate are easily eliminated by filtration and repeated washing.

After some time the particles undergo aggregation. The degree of aggregation depends on the nature of the particles and the conditions during their synthesis. To avoid aggregation of the particles and to reduce the size of the particles, some organic surfactants are used during the precipitation. Use of surfactants will help in tailoring the size and shape of the nanoparticles and to hinder the aggregation. Using Co-precipitation method and using surfactants SnO_2 nanoparticles of size ranging between 5nm and 23nm have been synthesized.

II. Experimental procedure

2.1 Materials

All the chemicals used were of analytical grade. Hydrated stannic chloride($Sncl_4.5H_2O$) used was obtained from Thomas Baker Mumbai, India, sodium carbonate anhydrous was obtained from Qualigens Fine Chemicals Mumbai, India and deminaralised water from Nice Chemicals (p) Ltd. Kochi, Kerala, India, and Triton X -100 from Himedia Laboratories pvt. Ltd. Mumbai and lauryl alcohol from Central Drug House New Delhi, India, are used directly without any further purification for the synthesis of SnO_2 nanoparticles. The as prepared SnO_2 nanoparticles were used for further characterization. X-ray diffractometer (Ultima IV Japan) with CuKα radiation (λ=1.5405 Å) at 40 mA and 40 kV at a scanning rate of 0.02˚ per second was used to study the crystals.

2.2 Synthesis of tin oxide nanoparticles

Solution of hydrated stannic chloride in deminaralised water and solution of anhydrous sodium carbonate in deminaralised water were prepared. The concentration of hydrated stannic chloride solution and

Sodium carbonate solution are prepared in the ratio 1:2 molar. First a known quantity of $Sncl_4.5H_2O$ solution was taken in a 1000 ml clean and dry beaker and subjected constant stirring using a magnetic stirrer at room temperature. After 5 minutes of stirring of this solution, the already prepared Sodium carbonate solution was added to this $SnCl_4$ solution under constant stirring drop by drop by taking Sodium carbonate solution in a 50ml burette and setting the rate of addition to 55-60 drops per minute till an equal quantity of sodium carbonate solution as that of $SnCl_4$ solution is added. This process is continued for about 1 hour 30minutes. During the process the solution of $SnCl_4$ turns into a white gelatinous precipitate. Starring is continued further for about 30 minutes even after total addition of the Sodium carbonate solution. The stirring is stopped and the white gelatinous precipitate is allowed for about 4 hours to settle down. The particles settled at the bottom of the beaker are separated from solution by decantation. In the present work we tried to filter the precipitate using Whatman filter paper number 41and 42 but the complete precipitate passed through the filter paper. This indicated that the particle size is much smaller than the pore size of the Whatman filter paper. Repeated washing and decantation process is used to separate the precipitate. In order to remove the impurities from the precipitate deminaralised water is added to the precipitate, stirred for 5 minutes, allowed the precipitate for about 4 hours to settle down and decanted. This process is repeated for several times. Finally the same process of removal of impurities repeated using ethyl alcohol instead of deminaralised water. This ensures complete removal of impurities form the precipitate. The beaker containing the precipitate is heated at low temperature for four hours and made it perfectly dry. The crystalline powder ground for 10 minutes with mortar pestle. The powder was taken in silica crucible and calcinated at 325^0C for 1 hour in muffle furnace. The muffle furnace was switch off and allowed it to reach the room temperature on its own. The final product was the SnO_2 nanoparticles.

2.3 Synthesis of Tin Oxide Nanoparticles with Surfactants:

A known quantity of hydrated stannic chloride solution is taken in a beaker and the surfactant Triton-X100 measuring 5% of the volume of the solution is added and Stirred it for 40 minutes using magnetic stirrer to get a homogeneous solution. There after the same procedure for synthesis and purification is followed as earlier. The final product is the SnO_2 nanoparticles with Surfactants Triton-X100.SnO_2 nanoparticles with surfactant Lauryl alcohol have also been trailed out. For this the above mentioned procedure is adopted.

Bare SnO_2 nanoparticles and SnO_2 nanoparticles with Surfactants Triton-x100and Lauryl alcohol have been synthesized and these materials were subjected to XRD measurements to determine the grain sizes.

III. Results and Discussion

Triton-X100 and Lauryl alcohol have been added as surfactants in the synthesis of SnO_2 nanaoparticles. Fig.1 shows the XRD spectrum of the bare SnO_2 nanoparticles synthesized by Co-precipitation method in aqueous medium. XRD analysis of SnO_2 nanoparticles adding surfactants Lauryl alcohol and Triton-X100 is shown in fig. 2 and fig. 3 respectively.

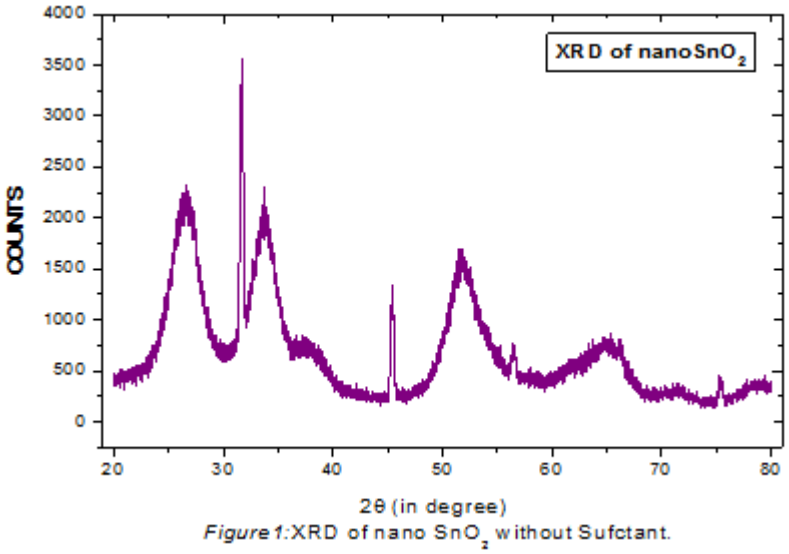

*Figure 1:*XRD of nano SnO_2 without Sufctant.

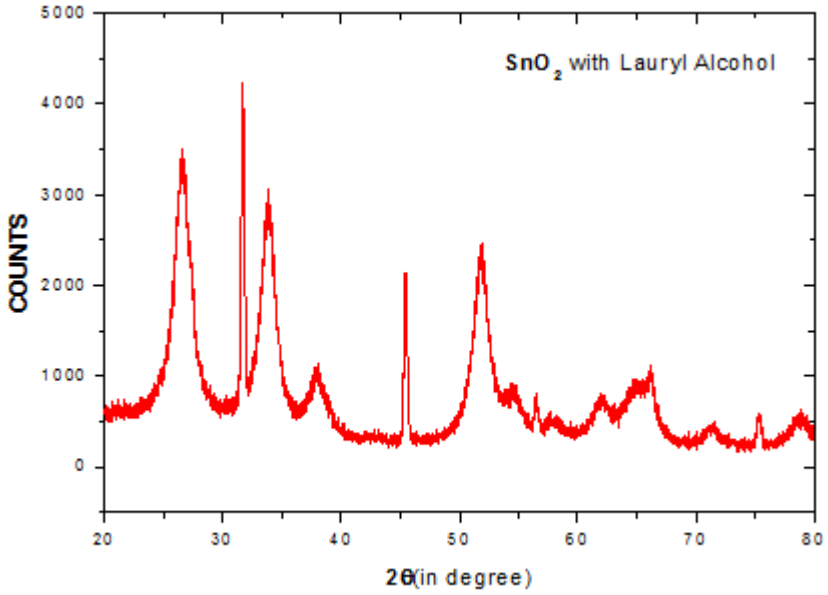

Figure2:XRD of nano SnO_2 with Sufctant Lauryl Alcohol.

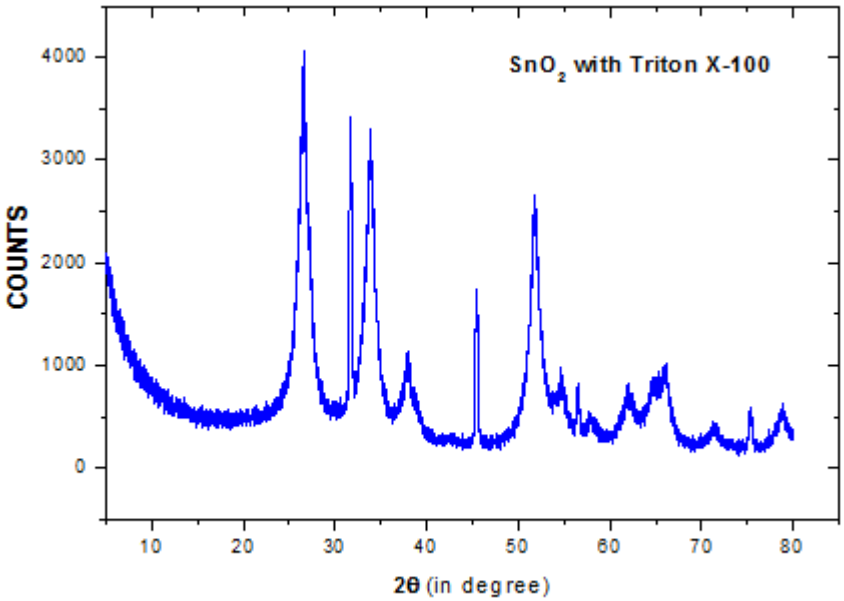

Figure3:XRD of nano SnO_2 with Sufctant Triton X-100.

The XRD patterns of all the three prepared samples are similar and reveal the crystalline nature. The average crystallite sizes of the three samples were calculated using Scherer formula and presented in Table 1.

Table 1: Comparison of the particle size of SnO_2 Naoparticles without and with surfactants.

Sample	Particle Size (nm)
SnO_2 without surfactant	23.782
SnO_2 with surfactant lauryl alcohol	21.2
SnO_2 with surfactant triton-X 100	5.819

The size of SnO_2 nanoparticles without surfactants is estimated as 23.782 nm where as the particle size with Triton-X100 and Lauryl alcohol is estimated to be 5.819 nm and 21.2 nm respectively. Hence there is a decrease in size of the particle is achieved by the use of surfactants. It is reported that particle size synthesized in organic medium is less than that synthesized in aqueous medium. Simin Tazikeh et al have reported the size of the SnO_2 nanoparticles by precipitation method in ethanol medium using Triton X100 and calcinated at 600^0C for 4hours as 23nm.[14] Kim and et al have reported the size of the SnO_2 nanoparticles ranging between 0.2 to1.3 µm sythesisized by thermal evaporation method [15]

IV. Conclusion

Co-precipitation method is found to be a very simple and suitable method for synthesis of nanoparticles that are very smaller in size. The selection of precipitating reagent, its concentration and concentration of precursor has effect on the particle size. The medium also has its impact on the size of the particles. SnO_2 nanoparticles without and with surfactants have been synthesized successfully. Use of Triton –X100 as surfactants has decreased the size of the particle from 23 nm to 5 nm. Whereas the use of Lauryl alcohol has reduced the particle size from 23 nm to 21nm.. Triton X100 is more effective as compared to Lauryl alcohol. The decrease in size of the particle is more use full in may application in photovoltaic, chemical gas sensing etc.,

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