SYNTHESIS AND DISPERSION OF BARIUM STANNATE NANOPOWDERS

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This is to certify that the thesis entitled, **"SYNTHESIS AND DISPERSION OF BARIUM STANNATE NANOPOWDERS"** submitted by **Swapna Samir Shukla** in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

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CONTENTS

ABSTRACT

The present work deals with the synthesis and dispersion of Barium Stannate ($BaSnO_3$) nanopowders. $BaSnO_3$ was prepared through a novel co-precipitation route using $SnCl_4.5H_2O$ and $BaCl_2.2H_2O$ as the starting precursors and subsequent calcinations at 1100^oC. X-Ray diffraction pattern indicates the presence of single phase of $BaSnO_3$ which was supported by FTIR analysis. The lattice parameter was found to be 4.12 Å. The data obtained from BET surface area indicates that the particle size is near about 250nm at pH 13. Isoelectric point (IEP) of nanopowder suspension was determined prior to the addition of different dispersants. The isoelectric point was found to be at pH 4.95 and the electrolyte weight percentage was optimized for stable dispersions.

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CHAPTER 1

INTRODUCTION

1.1 NANOPOWDERS

Nanotechnology defines a particle as a single unit in terms of its properties and transport. Assuming a particle to be roughly spherical, classification can be done on the basis of their diameters. Fine particles are those which have sizes in the range of 100 to 2500 nanometers. Nanoparticles have sizes less than 100 nanometers. Individual molecules, even though having sizes that would normally lie within the aforementioned ranges cannot be considered as nanoparticles. Nanopowders are nanoparticles in an agglomerated form. They have nano-scale dimensions in all the three directions.

In nanomaterials, a majority of the atoms are present on the surface as opposed to conventional materials where the atoms are found to be present in the bulk as well. Owing to the presence of atoms in diverse environments, nanopowders have intrinsic properties different from those of conventional materials. Nanopowders have high surface areas, so their properties are largely augmented. The characteristic high surface area of nanopowders enhances the rate of densification. Sintering of nanopowders can thus take place at a lower temperature [1].

In the recent years nanopowders have enjoyed a great deal of attention. Perovskite nanopowders, in particular have been thoroughly investigated for their excellent dielectric properties. These powders are found to be good candidates for gas sensors. The tremendous usage of perovskite nanopowders in these fields stems from the fact that their properties can be easily modified by selecting a particular cation. These powders also have excellent thermal stability [2]. $BaSnO_3$ is one such perovskite compound which has enjoyed a fair amount of success in fabrication of multilayer capacitors, boundary layer capacitors, gas sensors, humidity and LPG sensors [2-10].

1.2 $BaSnO_3$ – STRUCTURE

Barium stannate ($BaSnO_3$) belongs to the perovskite group of compounds. These compounds posses a similar structure as that of the mineral Perovskite($CaTiO_3$). Such compounds have a structural formula which can be generalized as $A^{2+}B^{4+}O_3$ where A is an alkaline earth (group IIA) metal element or a transition metal element in +2 oxidation state and B is a transition metal element.

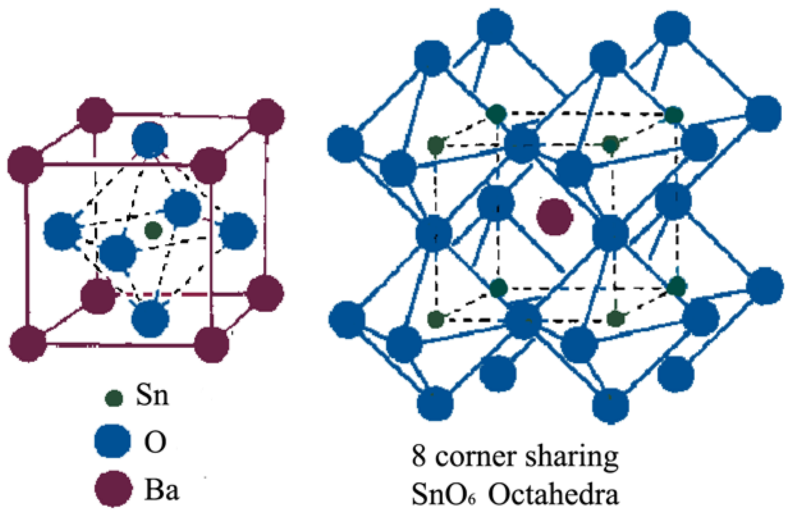

Fig 1: perovskite structure of $BaSnO_3$

The structure of $BaSnO_3$ is shown in Fig 1. $BaSnO_3$ has a cubic perovskite structure. Here Ba^{2+} ions occupy the corners of the unit cell, Sn^{4+} is present at the body center and O^{2-} ions are present at the face centers. Thus the Ba^{2+} ion is coordinated with 12 O^{2-} ions and Sn^{4+} with 6 O^{2-} ions. An O^{2-} ion on the other hand has a co-ordination number of 4 with Ba^{2+} and 2 with Sn^{4+}. The O^{2-} ion and Ba^{2+} ion form a dense cubic close packing with one-fourth of the octahedral voids filled with Sn^{4+}. Eight [SnO_6] octahedrons are linked through corners to form a three dimensional framework [3].

1.3 PROPERTIES

$BaSnO_3$ has pale yellow color. Sintering temperature is presumed to be above 1600^oC. A sample sintered at 1600^oC is still porous [3,13]. The melting point of $BaSnO_3$ is reported to be 2060^oC, however there are some disagreements as $BaSnO_3$ is found to decompose into BaO and SnO_2 at 1950^oC [3,14]. $BaSnO_3$ is paraelectrical and non ferroelectric [3].

1.4 APPLICATION

Solid state gas sensors: There are two types of gas sensors – solid state sensors and catalytic sensors. Catalytic sensors detect gases by burning them i.e. the gases react with the material of the sensor forming certain compounds. The formation of these compounds confirms the presence of a particular gas. Solid state sensors, on the other hand allow the gases to get adsorbed onto their surfaces. Thus solid state sensors have better longevity as no sensor material is consumed in the process. For better adsorption, a material with a high surface area is needed and this calls for use of perovskite Nanopowders. $BaSnO_3$ can be used to detect gases such as H_2, NO, CO, CH_4 etc [2-10].

$BaSnO_3$ can be used to fabricate **Multilayer capacitors** and **boundary layer capacitors** [2, 3, 4, 7, 8, 9, 10].

1.5 COLLOIDAL STABILITY

When particles are dispersed in a medium, they have a tendency to show Brownian motion. Due to this Brownian motion, they collide with one another. Thus, colloidal stability is influenced by particle interactions during collision. These interactions can be of two types- attractive (van der waals force) and repulsive. A colloidal system can only be stabilized if the repulsive forces outweigh the attractive forces, so that they do not agglomerate and settle down.

A colloidal system can be stabilized by: - Creating an electrical double layer (electrostatic stabilization), Steric stabilization and Electrosteric stabilization.

Ceramic particles are usually charged when present in a solution. The presence of charge on the surface of the particles changes the ion distribution in the surrounding media. Each particle develops a fixed layer of counter ions surrounded by a diffuse layer.

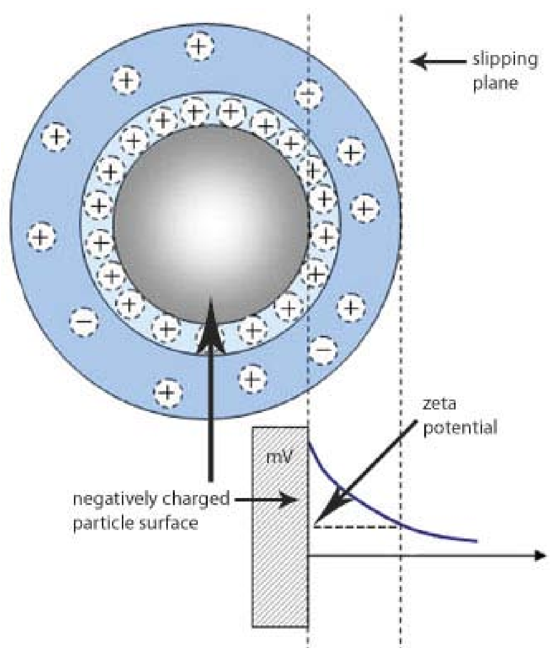

Fig 2: Schematic representation of zeta potential [12]

In between these two layers, there exists a hydrodynamic slipping plane. The ceramic particle behaves as a single unit till this place. The potential at this slipping plane is known as the zeta potential. Zeta potential has a very important role to play in deciding the stability of the dispersions. Dispersion is said to be stable if the absolute value of zeta potential is greater than 30 mV [11].

Zeta potential is dependent on the pH of a suspension [11]. This dependency can be explained in the following manner:-

Consider a particle present in a suspension having negative zeta potential

1) The particle develops a tendency for acquiring more negative charge if more alkali gets added to the suspension.
2) The charge on the particle gets neutralized on addition of acid.
3) The particle develops positive charge on further addition of acid.
4) If a graph is plotted between zeta potential and pH then, zeta potential will have a positive value at low pH and a negative value at high pH.
5) The pH at which zeta potential is zero is known as IEP (Isoelectric point). The dispersion is least stable at this pH.

CHAPTER 2 LITERATURE REVIEW

2.1 SYNTHESIS OF $BaSnO_3$ NANOPOWDERS

J.Cerda et al. successfully synthesized $BaSnO_3$ using a sol-gel route. They started with stoichiometric amounts of $K_2SnO_3.3H_2O$ and $Ba(OH)_2$. The solution was stirred vigorously for 30 minutes and the pH was maintained at 11. The sol thus obtained was dried at a temperature of 80^oC to get a xerogel. The xerogel was then calcined at temperatures of 1000^oC and 1400^oC for 8 hours. Macro Raman spectrum of the powder Calcined at 1000^oC showed the presence of a $BaCO_3$ peak. However, this peak disappeared when the powder was calcined at 1400^oC. Nanoparticles with a mean size of 200 nm were obtained through this route. Electrical characterization of $BaSnO_3$ thick films was also done as a function of temperature and gas concentration. In presence of O_2, CO and NO_2, resistance variations were measured. O_2 was found to show a maximum sensitivity at a temperature of 700^oC. A maximum sensitivity at a temperature of 600^oC was reported for CO and NO_2 [2].

Young Jung Song et al. successfully synthesized fine $BaSnO_3$ powders by oxalate co-precipitation route by using stoichiometric amounts of $BaCl_2$ and $SnCl_4$. Oxalic acid in non-stoichiometric amounts was used as the precipitating agent. The precipitates were filtered, washed by ethanol and dried at 110^oC for 24 hours. Calcination was done at a temperature of 1050^oC for 3 hours. The calcined powders had a cubic crystal structure with a lattice parameter of 4.119 Å. Grain size of the powder was 200 nm [4].

A.S. Deepa et al. investigated the synthesis of nanocrystalline $BaSnO_3$ through an auto ignited combustion technique. The synthesized $BaSnO_3$ had a cubic crystal structure with a lattice parameter of 4.109 Å and a crystallite size of 25 nm. They proposed that $BaSnO_3$ undergoes transition to an ideal cubic structure from a distorted one. They established the fact that $BaSnO_3$ is a photo luminescent material. They also determined the band gap to be 2.887 ev with the aid of UV-vis spectrum [5].

J. Cuervo Farfan et al. synthesized $BaSnO_3$ through solid state synthesis. They used $BaCO_3$ and SnO_2 in stoichiometric amounts. The mixture was made into pellets followed by calcination at 1090^oC for 12 hours. The Calcined powder however had Ba_2SnO_4 as the major phase. It is possible to get $BaSnO_3$ only after sintering. Sintering was carried out in two steps- heat treatment at 1200^oC for 20 hours followed by heating at 1400^oC for 24 hours [6]. **Azad et al.** Proposed that if $Ba(NO_3)_2$ is used instead of $BaCO_3$, then the calcination temperature can be lowered. $BaSnO_3$ could thus be formed at a temperature of 1000^oC [15].

Wensheng Lu et al. investigated the synthesis of $BaSnO_3$ nanopowders under lyothermal conditions using tin oxide hydrate and $Ba(OH)_2$ as the precursors. The tin hydroxide gel was in turn precipitated from an aqueous solution of $SnCl_4$ and NH_4OH. They made a detailed analysis of the effect of various solvents namely methanol, isopropanol, ethanol and acetone on the development of crystallinity, BET surface area, density and average particle size of the $BaSnO_3$ powders. Isopropanol showed better results. They established that agglomeration can be avoided by the addition of Genapol-X080 along with isopropanol. The surface area increased 10-fold on addition of the former. The as-synthesized powder contains $BaSn(OH)_6$ which transformed to $BaSnO_3$ at a temperature of 330^oC through an intermediate amorphous phase. FTIR studies indicate the presence of an intense peak of Sn-O in the calcined powders [8].

Wensheng Lu et al investigated the hydrothermal synthesis of nanocrystalline $BaSnO_3$. Treatment under hydrothermal conditions (250^oC for 6 hours) was done for a mixture of tin oxide hydrate sol and $Ba(OH)_2$. They also studied the pH dependence on the properties of tin oxide hydrate sol. They found out that peptization took lesser time when the pH was maintained between 8.3 and 9.8. The particle diameter distribution was also found to depend on pH. The distribution changed from (0.5-1.5) µm at pH (6.8-7.2) to less than 20 nm at pH (8.3-9.8). Calcination was done at 330^oC. The Nanopowders synthesized by this method had very high specific surface area [9].

Wensheng Lu et al investigated the synthesis of $BaSnO_3$ through metal alkoxide route. $BaSnO_3$ was prepared by crystallization of an amorphous precursor ($BaSn(OR)_6$) . They proposed that $BaSnO_3$ nucleates at 350^oC just after the decomposition of residual organic matter. Calcination was completed at 760^oC and the powders obtained had a very small average grain size [10].

2.2 DISPERSION OF $BaSnO_3$ NANOPOWDERS

Wensheng Lu studied the dispersion of $BaSnO_3$ nanopowders. He used four different surfactants namely TMAH, PEI-SC (poly ethylimine with short chains), PEI-LC (poly ethyl imine with long chains) and TEA (tri-ethyl amine) for stabilizing the dispersions. He obtained best results with TEA and therefore proceeded with the addition of 2.5 wt% TEA for stabilizing the suspension at pH 10 [3].

CHAPTER 3
EXPERIMENTAL PROCEDURE

3.1 Synthesis of Barium Stannate Nanopowders

3.1.1 Chemicals used:

- Stannic chloride pentahydrate ($SnCl_4.5H_2O$)
- Barium chloride dihydrate ($BaCl_2.2H_2O$)
- Sodium hydroxide pellets (NaOH)

3.1.2 Reaction involved:

$$BaCl_2.2H_2O + SnCl_4.5H_2O + 6\ NaOH \rightarrow BaSnO_3 + 6\ NaCl + 10\ H_2O$$

3.1.3 Procedure:

Stoichiometric amounts of $BaCl_2.2H_2O$ and $SnCl_4.5H_2O$ were taken. NaOH was used as a buffer, so excess of NaOH was taken.

Aqueous solutions of the above mentioned reagents were prepared. $BaCl_2.2H_2O$ solution was taken in a conical flask. Simultaneous heating and stirring was carried out till the temperature reached 75^oC. . This was followed by drop wise addition of NaOH. Addition of NaOH was continued till pH of the solution increased up to 11. Once the required pH was established, $SnCl_4.5H_2O$ solution was added slowly. White precipitates were formed immediately after addition of $SnCl_4.5H_2O$. Simultaneous heating and stirring was done till the temperature reached 95^oC. The solution was allowed to remain at 95^oC for 1 hour. The solution was aged overnight for precipitation.

Decantation

The precipitate was washed repeatedly with distilled water till all the chlorine gets removed. The absence of chlorine was confirmed through $AgNO_3$ test.

$NaCl + AgNO_3 \rightarrow AgCl + NaNO_3$

When milky white precipitates of $AgNO_3$ stopped forming, decantation was discontinued.

Filtration:

The solution was filtered using Whatman(41) series filter paper.

Drying:

The filtrate was taken in a petridish and dried in an oven at 100^oC for 24 hours.

Calcination:

The dried sample was calcined at 1100^oC with a soaking period of 2 hours with a heating rate of 3^oC/min.

Another batch was made by taking the same amounts of $SnCl_4.5H_2O$ and $BaCl_2.2H_2O$ but this time NaOH addition was continued till the pH was 13. Rest of the steps remained the same.

3.1.5 FLOWCHART OF $BaSnO_3$ SYNTHESIS

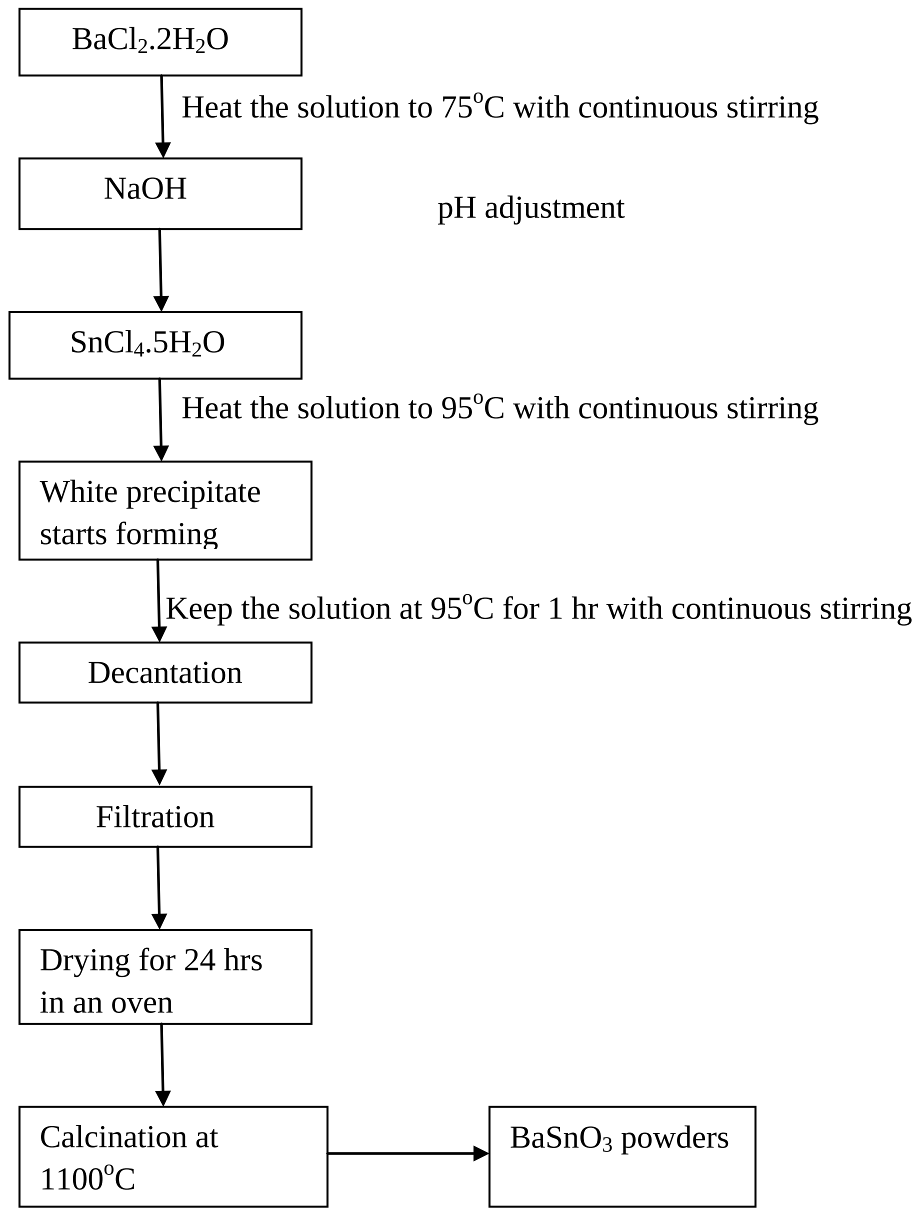

Fig 3: Flowchart for $BaSnO_3$ synthesis

3.2 CHARACTERIZATION

3.2.1 DSC/TG

DSC/TG was done by heating the sample at a rate of 10^{o}C/min (Mettler-Toledo 821)

3.2.2 X-ray diffraction

Identification of the phases, determination of lattice parameter and crystallite size of $BaSnO_3$ was done by Philips X-Ray Diffractometer(PW 1730,Netherlands). The radiation used was nickel filtered Cu Kα radiation with a wavelength of 1.5406 Å. The voltage was maintained at 40 kV and the current at 30 mA. The scan range was $(15\text{-}80)^{o}$ with a scanning rate of 0.04^{o}/sec.

From the most intense peak of XRD plot, lattice parameter and crystallite size of $BaSnO_3$ powders was determined

Lattice parameter: $a=d\sqrt{(h^2+k^2+l^2)}$

'a'- lattice parameter ; d- d spacing;

'h,k,l'- indices of the most intense plane

Crystallite size = $0.9\ \lambda/\ \beta \cos\theta$

'λ'- Cu Kα radiation(1.5406 Å) ;

'β'- FWHM of the most intense peak; θ – Bragg's angle.

3.2.3 Specific surface area by BET method

The specific surface area was measured by BET surface area analyzer (Quantachrome, USA). The amount of nitrogen adsorbed at normal atmospheric pressure and at a temperature same as that of the boiling point of liquid nitrogen vs. the partial pressure of nitrogen gives the specific surface area.

Average particle size can be calculated using the formula: 6/ ρS

ρ - Density of pure $BaSnO_3$ sample (7.24 gm/cm^3)

S is the measured specific surface area. (m^2/gm)

3.2.4 FTIR analysis

Determination of the structure of molecules was made through FTIR analysis. The Calcined powders and KBr were mixed in the ratio of 1:30 by mass. KBr was chosen as it is transparent to IR radiation. The mixture was then pressed into pellets. The machine used was the FTIR instrument of PERKIN-ELMER. The range was set in the mid IR region i.e. 4000 cm^{-1} to 400 cm^{-1}.

3.3 DISPERSION OF $BaSnO_3$ NANOPOWDERS

3.3.1 Determination of IEP

150 ml of distilled water was taken in a beaker and 0.05 grams of $BaSnO_3$ was added to it. The solution was then ultrasonicated for 10 minutes to disperse the powders. After sonication, the dispersion was maintained at pH 2 by drop wise addition of 1:5 HNO_3. Similarly dispersions at pH 4 and 6 were prepared. Dispersions at pH 8 and 10 were made by drop wise addition of 1:1 NH_4OH. Zeta potential of all these dispersions was then measured.

3.3.2 Determination of optimum electrolyte concentration for stabilization

To determine the dispersing agent and its concentration, two electrolytes were used. These are PEG-400 and DARVAN C.

3.3.2.1 Preparation of PEG-400 and DARVAN C solutions

0.14 grams of PEG-400 was dissolved in 500 ml of distilled water and the solution was stirred for 15 minutes. Similarly 0.1 grams of DARVAN C was dissolved in 500 ml of distilled water followed by stirring for 15 minutes.

3.3.2.2 Dispersion of $BaSnO_3$ powders

150 ml of distilled water was taken in a beaker and 0.05 grams of $BaSnO_3$ was added to it. 0.1 weight % of either PEG-400 or DARVAN C was added to the solution. The solution was then dispersed using the ultrasonic vibrato meter for 10 minutes. After sonication, the dispersion was maintained at pH 9 by drop wise addition of 1:1 NH_4OH

The weight percentage of both PEG-400 and DARVAN C was varied from 0.1 to 1. Dispersions were made at these weight percentages. Zeta potential of all these dispersions was then measured.

3.3.3 Measurement of zeta potential

The zeta potential of the dispersions was measured by Zetasizer Nano Series, Nano ZS of MALVERN.

CHAPTER 4
RESULTS AND DISCUSSIONS

This section deals with the thermal analysis, phase analysis, FTIR analysis and BET surface area of the powder. The IEP and variation of zeta potential with weight percentage of dispersants has also been studied.

4.1 THERMAL ANALYSIS OF THE AS-SYNTHESIZED POWDER

The DSC-TG plot of the as-synthesized $BaSnO_3$ powder has been shown in Fig 3. A total weight loss of 5.80% has been observed. An endothermic peak is observed at 256.8^{o}C. This is due to loss of water from the sample

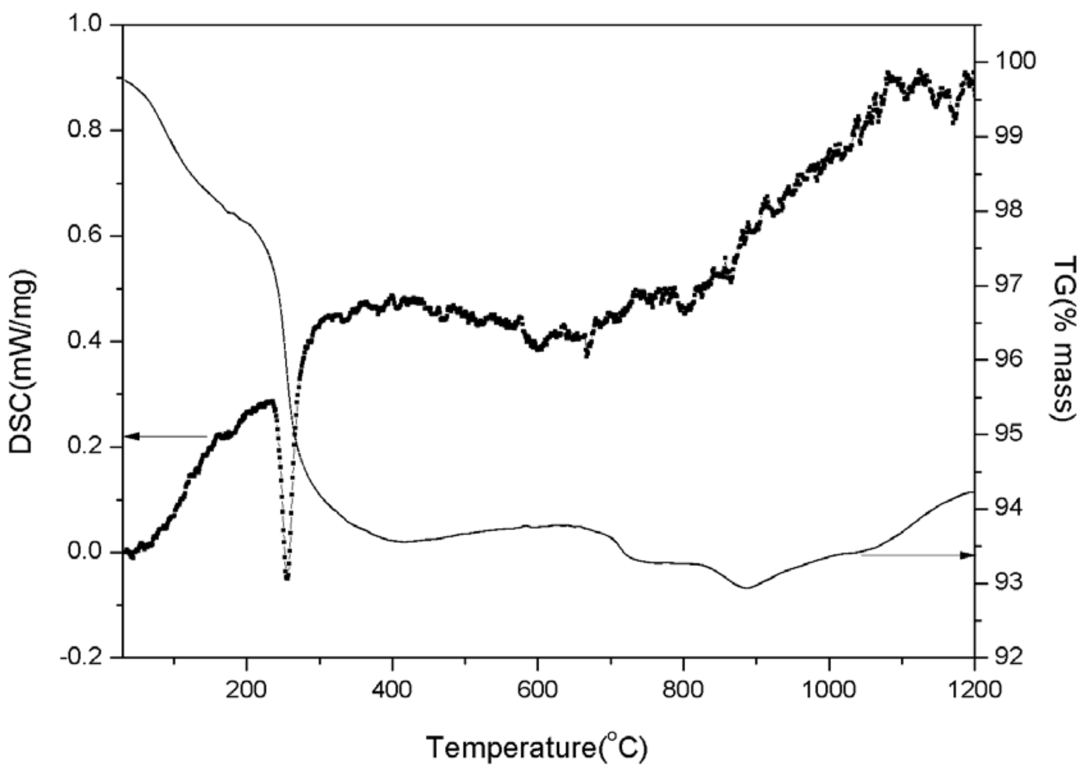

Fig 4: DSC-TG curve of as-synthesized $BaSnO_3$ powder

. 4.2 PHASE ANALYSIS OF THE CALCINED POWDERS

The XRD obtained agrees well with the XRD data contained in the JCPDS file 15-0780. The planes are properly indexed. The XRD plot confirms that $BaSnO_3$ has a cubic perovskite structure.

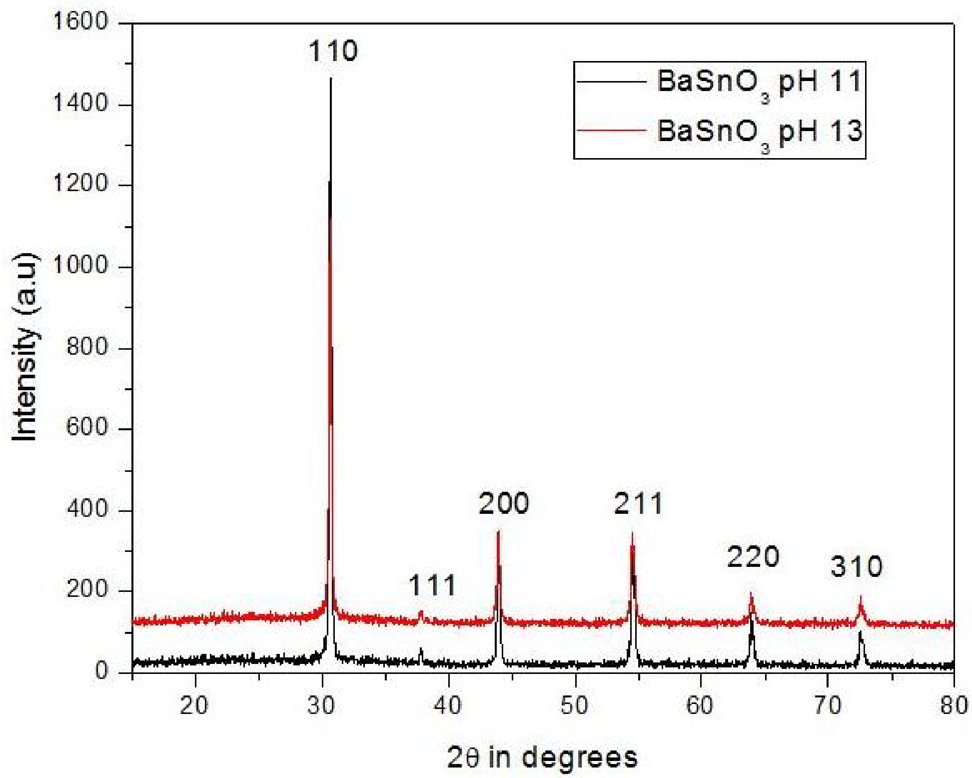

Fig 5: XRD plot of calcined $BaSnO_3$ powders

The lattice parameter in both the cases is calculated to be 4.12Å. The crystallite size however is different for the two powders. The crystallite size in case of the powder synthesized at pH 11 is 44.8 nm and 50.5 nm in case of the powder synthesized at pH 13.

4.3 SPECIFIC SURFACE AREA BY BET

Table 1: Specific surface area and particle size of the powders synthesized at different pH

POWDER TYPE	**SPECIFIC SURFACE AREA**	**PARTICLE SIZE**
Synthesized at pH 11	1.556 m^2/gm	532 nm
Synthesized at pH 13	3.254 m^2/gm	254 nm

4.4 FTIR ANALYSIS OF THE SYNTHESIZED POWDERS

FTIR analysis of the calcined sample indicates the structure of the molecules. The bonds corresponding to the labeled peaks have been identified and explained.

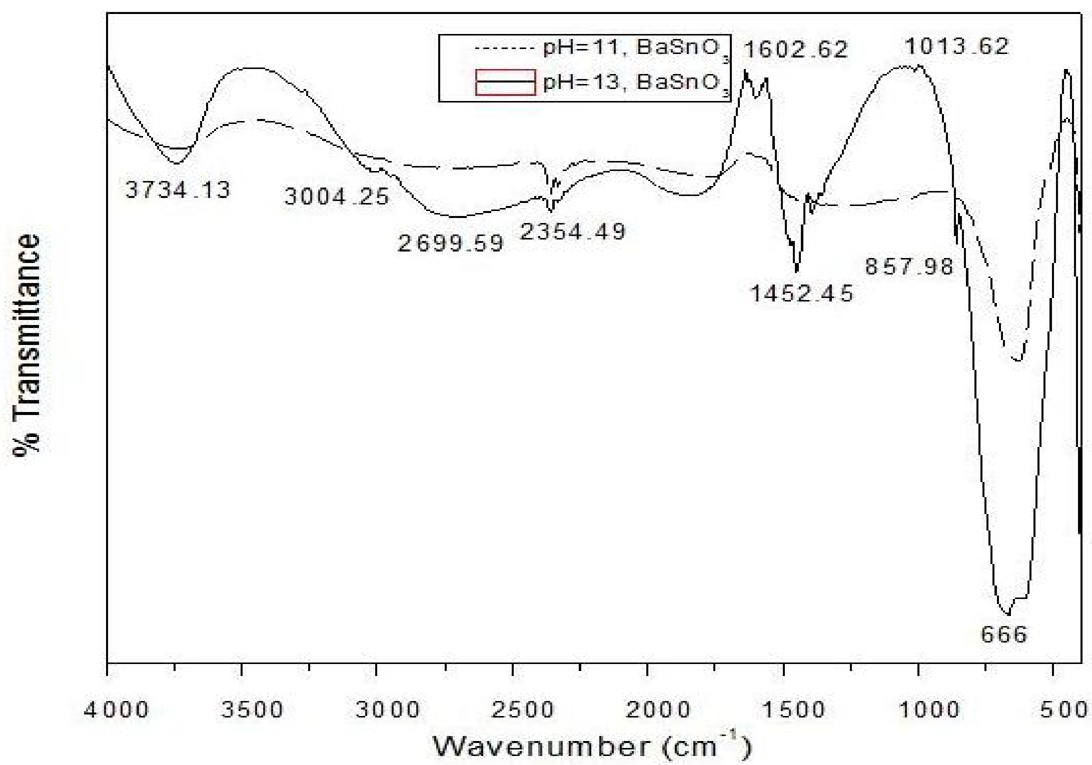

Fig 6: FTIR plot of the synthesized BaSnO3 powders.

At 666 cm^{-1} an intense peak is observed for the powder synthesized at pH 13. This corresponds to Sn-O asymmetric stretching mode of the bond. This shifts to 628.15 cm^{-1} in case of the powder synthesized at pH 11.

Now $E=hc/\lambda = hc\nu$ where E is the infrared energy required to set the bond to vibration, c is the velocity of light and $\nu=1/\lambda$ (inverse of wavelength i.e. wave number). Thus the energy requirement for stretching is more in case of the powder synthesized at pH 13. So a stronger Sn-O bond is formed at pH 13.

At 857.98 cm^{-1}, 1013.62 cm^{-1} and 1452.45 cm^{-1} three peaks are observed for the powder synthesized at pH 13. These correspond to stretching modes of CO_3^{2-}. The presence of three peaks at different wave numbers can be attributed to the coupling motion of the C-O and C=O bonds. Carbonates, basically $BaCO_3$ may form due to absorption of CO_2 from atmosphere during FTIR analysis. However, the carbonate peaks are redundant in case of the powder synthesized at pH 11.

At 1602.62 cm^{-1} and 3734.13 cm^{-1}, two peaks are observed for the powder synthesized at pH 13. These correspond to OH^-. The presence of two peaks at different wavenumbers can be attributed to the coupling motion of O-H bond. Water may be present due to adsorption by KBr pellets or by $BaSnO_3$ powders. At 3732.62 cm^{-1} a peak corresponding to O-H bond can be seen in the powders synthesized at pH 11.

At 2354.49 cm^{-1} a peak is absorbed in both the powders. This corresponds to CO_2 which has simply been adsorbed onto the surface but doesn't react to form carbonates.

4.5 ISOELECTRIC POINT

Figure 6 shows the dependence of zeta potential of $BaSnO_3$ powders on pH. The isoelectric point is found to be at a pH of 4.96.

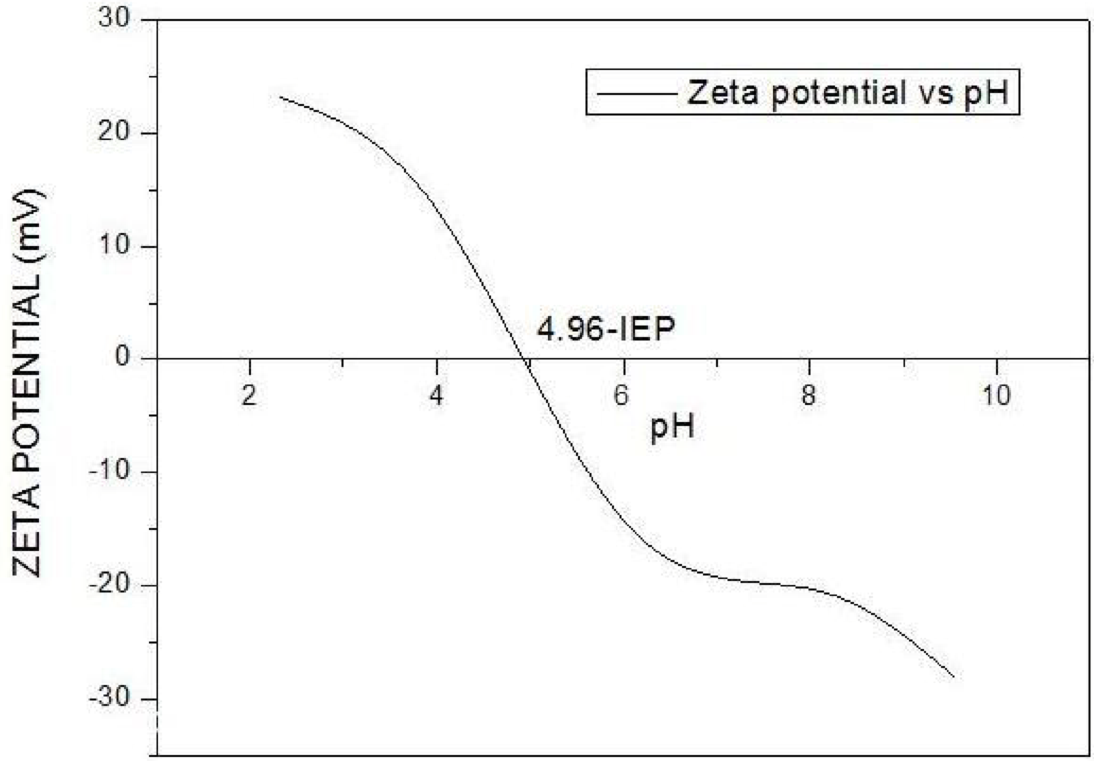

Fig 7: Dependence of zeta potential of $BaSnO_3$ powders on pH

Dispersion is said to be stable if the absolute value of zeta potential is greater than 30 mV. As seen in Fig 7, the value of zeta potential approaches to -30 mV in the pH ranges 8-10. So the dispersion of $BaSnO_3$ nanopowders can be stabilized by maintaining the pH in the range 8-10. In the present case, dispersions were made by maintaining the pH at 9 with the help of NH_4OH.

4.6 VARIATION OF ZETA POTENTIAL WITH WEIGHT % OF DISPERSANTS

Figure 8 shows the variation of zeta potential with weight % of dispersants (PEG 400 and DARVAN C) at pH 9. In case of PEG 400, the zeta potential reaches a maximum value of -33.5 mV when 1wt% is used

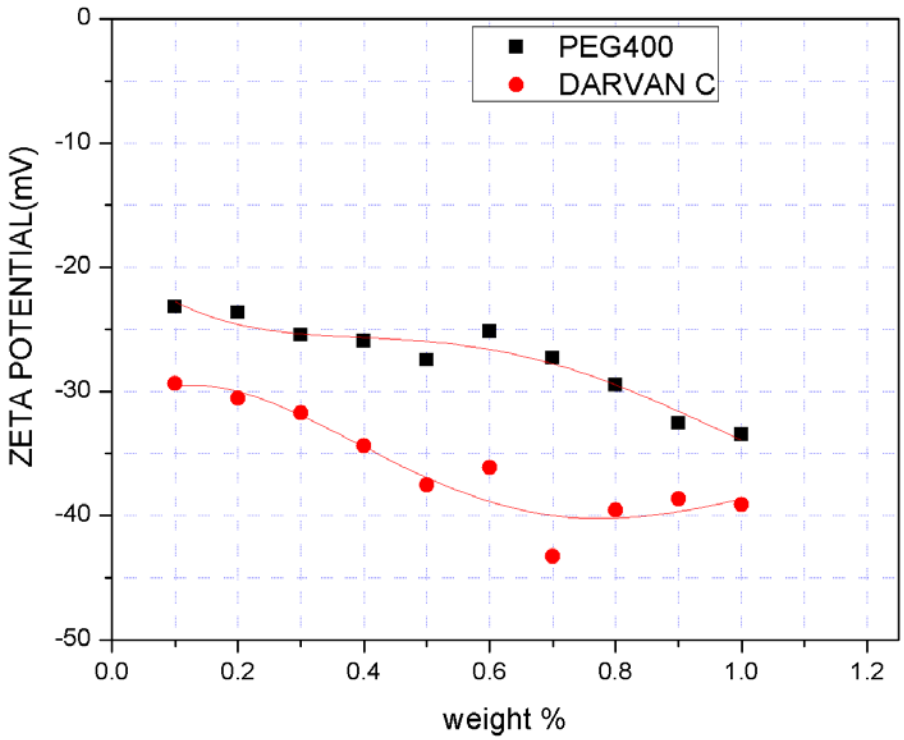

Fig 7: Zeta potential vs. weight % of dispersants at pH 9

DARVAN C on the other hand shows better results. The zeta potential attains a value of -39 mV at 0.8 wt% and remains constant. So a dispersion of $BaSnO_3$ nanopowders powders can be stabilized by maintaining the pH at 9 and by addition of 0.8 wt% DARVAN C.

CHAPTER 5

CONCLUSION

$BaSnO_3$ powders were prepared by a co-precipitation route by using stoichiometric amounts of $SnCl_4.5H_2O$ and $BaCl_2.2H_2O$. NaOH was used as a precipitating agent. During the course of synthesis the pH was maintained at two different values – 11 and 13. The powder was calcined at 1100^oC for 2 hours.

The following conclusions were drawn:-

1) The XRD studies have shown that the nanopowder is crystalline, single phase and has a cubic perovskite structure with a lattice constant a = 4.12 Å.

2) The powder synthesized at pH 13 has a higher specific surface area.

3) Effect of pH on the dispersion of $BaSnO_3$ was studied. The IEP was found to be at pH 4.96

4) A comparison of Darvan C and PEG-400 as a dispersant was done and Darvan C was found to give better results.

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