# Synthesis and Characterization of BaTiO₃ Nano particles by Organic Precursor Method

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## Abstract

$BaTiO_3$ nano powders prepared by using organic acid method. IR spectrum and EDS were used to identify the composition of the nano particles. XRD spectrum indicated the polycrystalline nature of the    particles. Surface morphology of the particle was studied by scanning electron microscope (SEM). No pits and pin holes were found in the surface.

***Keywords:*** *$BaTiO_3$,, EDS, XRD ,SEM, etc.,*

## 1. INTRODUCTION

Ferroelectric thin films are very promising for a wide range of application such as high dielectric capacitors, insulating surface layer, non volatile memories with low switching voltage, infrared sensors and electro optic devices. $BaTiO_3$ family ceramic thin films are receiving extensive investigation as the charge- storage dielectric for G-bit dynamic random access memory(DRAM), on-chip components such as de-coupling capacitors and positive temperature coefficient of resistance (PTCR) thermistors due to high dielectric constant, low dielectric loss, low leakage current, low temperature coefficient of dielectric constant and thermal stability[1].

Synthesis of $BaTiO_3$ nanopowders and thin films has attracted great attention over the  last few decades due to the desirable properties and applications. $BaTiO_3$ particles could be produced by employing various chemical methods such as sol-gel technique[2],metal-organic processing [4], co-precipitation[3], hydrothermal treatment [5,6] and mechanochemical synthesis[7]. Organic acid precursor is a promising method that offer relative low cost, uniform size, homogeneous powder and high purity of the ceramics.

This films of $BaTiO_3$ could be prepared by different technique like r.f sputtering [8], pulsed laser ablation [9] and metal- organic chemical vapour deposition [10] etc. Despite that several technique have been explored to deposit thin film of $BaTiO_3$ less attention has been devoted to thermal evaporation. In the present   study, we have reported about the $BaTiO_3$ nanopowder prepared by using organic acid precursor method using titanium dioxide $TiO_2$ as a source of titanium and oxalic acid as organic acid..

## 2. EXPERIMENTAL

### 2.1 Synthesis of BaTiO₃ nanopowder

$BaTiO_3$ nanopowders were synthesized using organic acid precursor method. The starting materials used were barium chloride $BaCl_2.2H_2O$, $TiO_2$ powder and Oxalic acid. A solution of Ba: Ti : Oxalic acid mole ratio1: 1: 1 was stirred and evaporated at $70^oC$ till a clear, viscous resin was obtained, then dried at $110^0C$ for 20 hours. The precursor formed was heated at $900^0C$ for 2 hours to form $BaTiO_3$ nanopowder.

### 2.2 Characteristics of BaTiO₃ nanopowder

FTIR and Energy Dispersive X-ray micro analyzer (EDX) were used for the identification of the chemical composition. The XRD patterns of the resulting products were obtained from X- ray powder diffraction with CuKα radiation. The micrograph of $BaTiO_3$ was examined by direct observation via scanning electron microscope (SEM).

## 3. RESULT AND DISCUSSION

Fig.1 shows infrared spectra of the prepared $BaTiO_3$ nanopowder. A strong absorption feature at $3205 cm^{-1}$ has been identified as the stretching mode of OH ions interpreted in the film and is commonly found in pervoskite films grown at low temperature by more conventional techniques.
This absorption referred to as hydroxyl defects because of the high mobility of hydrogen in pervoskite materials and is usually annealed out at temperatures near the phase transformation temperatures. The peaks at     $809 cm^{-1}$, $1290 cm^{-1}$, $1663 cm^{-1}$, $2357 cm^{-1}$, $2521 cm^{-1}$ and $2646 cm^{-1}$ are characteristics of the $CO_3^{2-}$ group. The band at $3205 cm^{-1}$ assigned to bulk hydrogen impurities [11].

It is well known that hydrogen impurities can penetrate several ternary oxides with perovskite structure as $H^+$ bonded to lattice oxygen in the form of an $OH^-$ [12]. These protons can compensate for the cation charge defect due either to reduced centers such a s $Ti^{3+}$ or to cation vacancies in nonstoichiometric samples. The weak shoulder near 2900 $cm^{-1}$, 1958 $cm^{-1}$ and 1042 $cm^{-1}$ which are characterized by the absorption of $CH_2$ groups. The broad bands at 557$cm^{-1}$ and 400$cm^{-1}$ are due to Ti-O vibrations in $BaTiO_3$. FTIR analysis suggested that defects of negligible percentage are present in the lattice of $BaTiO_3$ nano powder.
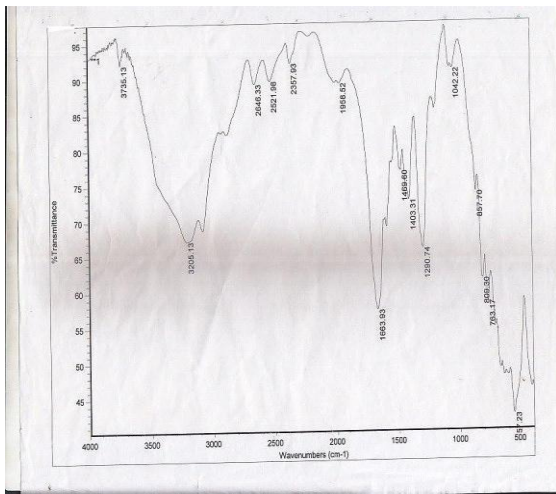


Fig. 1 FTIR spectrum of $BaTIO_3$ nano powder

Fig.2 shows the EDS spectrum of as prepared $BaTiO_3$ nano powder. Elemental composition analysis indicated the presence of Ba, Ti and O in the synthesized $BaTiO_3$ nano powder.
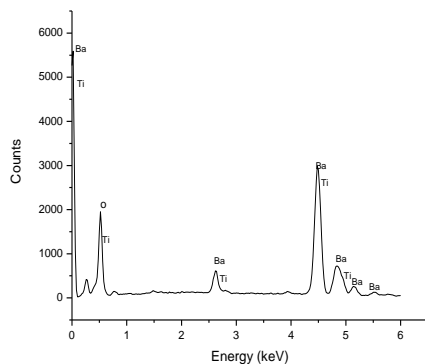


Fig.2 EDS spectrum of $BaTIO_3$ nano powder

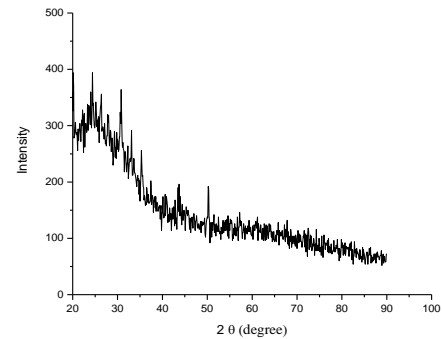A typical X-ray diffraction (XRD) pattern of $BaTiO_3$ powder as shown in the Fig.3.



Fig. 3 XRD pattern of $BaTIO_3$ nano powder.

The crystallite size of $BaTiO_3$ for the most intense peak determines from the X-ray diffraction data using the Debye-Schemer formula.

$$d_{RX} = kd/\beta \cos \theta$$

Where $d_{RX}$ is the crystallize factor to account for particle size k = 0.9 is a correction factor to account for particle shapes, F is the full width at half maximum (FWHM) of the most intense diffraction peak, $\lambda$ is the wavelength of Cu target = 1.5406 Å, and $\theta$ is the Braggs angle. The small peaks are observed at $2\theta$=24.4 Å with a d value of 3.64 Å, at $2\theta$=30.712 Å with a d value of 2.908 Å, at $2\theta$=35.34 Å with a d value of 1.81 Å. The peaks at $2\theta$ = 24.4 Å , 30.71 Å, 35.34 Å and 44 $A^0$ are respectively results from (001), (101) ,(110) and (002)/(200) planes of $BaTiO_3$. The resulting XRD and lattice parameters exactly match $BaTiO_3$ pattern (JCPDS 31-174).

A typical scanning electron micrographs of $BaTiO_3$ nano powder as shown on Fig. 4. The particulate phase in the micrograph is the $BaTiO_3$ grains and the voids seen in the micrograph. The micrograph indicates that the average grain size of $BaTiO_3$ nano powder varies from 90.68 nm to 133 nm.
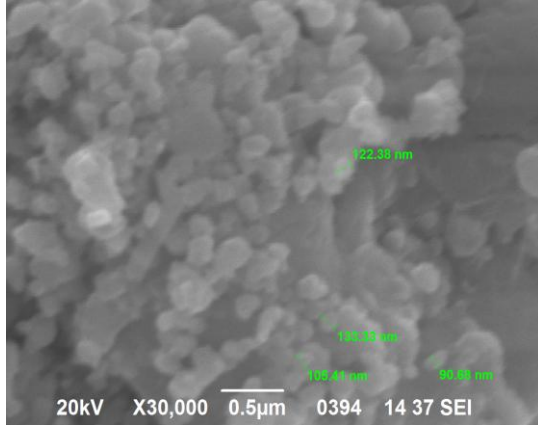
Fig.4 SEM micrograph of BaTiO$_3$ nano powder.

The grain size of vacuum evaporated BaTiO$_3$ thin films are relatively large compared to as prepared BaTiO$_3$ nano powder. Arlt.et.al [13]  have reported that BaTiO$_3$ exists predominantly in the tetragonal structure for grain size of 1.5 μm and above and that, for an average grain size below 1.5 μm, tetragonal and orthorhombic phases coexist. Therefore, it seems that BaTiO$_3$ films prepared in this study also exists as a mixed system of tetragonal and orthorhombic or pseudocubic structure. Our X-ray studies also support this conclusion, because the peaks corresponding to the (002) and (200) planes are not well resolved.

## 4.   CONCLUSION

We have successfully synthesized nano powder of BaTiO$_3$ by low cost organic acid precursor method using commercially available chemicals such as oxalic acid, TiO$_2$ and BaCl$_2$.. The composition of BaTiO$_3$ nanopowder were confirmed by IR and EDX analysis. XRD indicated the predominantly amorphous nature of the film. The presence of small peaks indicated the presence of crystallites of very low dimension in the amorphous region. No pits, pin holes and dendritic features are found on the surface in SEM micrographs.

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