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Hydrothermal Synthesis and Characterization of Barium Titanate nano particles

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ABSTRACT

In the present work the Barium Titan ate (BT) nano spheres were prepared through Low temperature Hydrothermal method. From the X-ray diffraction method we determine the cubic perovskite pattern of the sample. By the considering of the Scherrer method we also found to be the mean crystallite- size (D_p) was 22 nm. Moreover with the help of the field emission and transmission electron microscope (FESEM) we analysied the morphology of the material. Against of the Fourier-transform infrared spectroscopy (FTIR) eludicated the formation cubic structure of the material due to the exitence of the metal oxide bonds (M-O). In the extended to evaluate the optical band gap (E_{op}) was to be of 3.22 eV.

KEY WORDS: Nanoparticles; Hydrothermal Method; Diffraction; Morphology Microscopy.

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1. INTRODUCTION:

Basically the solid substances have been categorized into two branches based on the size of the particle or grain that is bulk and nano. The scientific community mainly focused on the investigations of bulk materials for prominent applications up to 18th century. Latter, the nanotechnology and nanoscience extended until 19th century by revealing the significance of nanoscale towards the variation of different properties such as structural, physical, electrical, magnetic, biomedical and optical properties of the nanoparticles. In the connection current era has been mainly concentrated on study of various nanosized substances for numerous technological applications[1] like drug delivery systems, temperature, gas, liquefied petroleum gas (LPG) and humidity sensors, photocatalysis, dielectric, ferroelectric and piezoelectric properties, nano fibers, carbon tubes, quantum dots and quantum wires. These can be used in the biomedical sciences for identifying the tumors in human body, surgeries etc. Meanwhile many scientists have been focused on the distinct properties of $BaTiO_3$, $CoTiO_3$ and $PbTiO_3$ nanoparticles[2] . Barium titanate (BT) is an oxide material with cubic perovskite structure[3]. Since last decades; the extensive investigations have been made on electrical, dielectric and ferroelectric properties of BT[3]. Generally the barium titanate (BT) gives attracactive applications such as multi layer ceramic capacitors (MLCC) by cause of reducing of the particle size[4]. By virtue of the decreasing of the particle size we can use the low temperature method. For getting of the high purity and homogeneous and ultrafine nanoparticles we approach the low temperature techniques. Many researchers synthesis the barium titanate (BT) nanoparticles under different techniques like hydrothermal[4], sol-gel[5], micro-emulsion[6], polymeric precursor[7] and microwave heating[8]. Finally the detailed literature surveybarium titanate (BT) nano particles significantly concentrated on biomedical and sensor applications. So mainly we concentrated on the investigation of the structural, morphological and optical properties of BT nanospheres.

2. EXPERIMENTAL PROCEDURE:

For the preparation of barium titanate nano particles we selected the raw materials of Ba $(NO_3)_2$ and TiO_2 (each of 99.9 % purity, Sigma-Aldrich).According to the stoichiometric ratio we amalgamated the precursors. The total mixture was taken into a glass beaker. Additionally the distilled water was added to the precursors in the ratio of 1:4 (mixed precursors (gm): distilled water (ml)) then the prepared solution was put up in to a magnetic stirrer. This stirring rate of 500 rpm is maintained in order to the stir the solution. Next we added the NaOH solution to the prepared mixture. Moreover the solution was shift into 300 ml Teflon bowl inserted in an autoclave. The sealed autoclave was placed in a hot-air oven at a maintaining temperature of 130^{o}C/6 hrs. In

addition the after completion of the 130°C/6 hrs the autoclave was gradually cooled to 30°C. The resultant solution was removed from the Teflon lined autoclave and it was cleaned with the distilled water for more than 12 times the p^H value was getting into 7.After removing of the Barium Titanate (BT) nano particles these were analyzed by different characterizations like X-ray diffraction method field emission scanning and transition electron microscopy. In addition, the FTIR, UV visible techniques are used to find the presence of metal oxide bands and the optical energy band gap.

3. RESULTS AND DISCUSSIONS:

XRD ANALYSIS:

From the Fig.1.we observe the barium titan ate (BT) nanoparticles diffraction patteren and it also reveals the cubic pervoskite pattern of relevant reflection planes these are agree with the JCPDS card number 89-2475.Here the diffraction structure having the largest peak was recorded at 31.56°c .There was no secondary peaks were observed in this barium titanate (BT) diffraction pattern. The average crystallite size 'D_p' is calculated by considering of the average full-width at half-maxima (FWHM) of reflection planes by the help of the Debye-Scherrer equation[9]

$$D_p = = \frac{k\lambda}{\beta Cos\theta} \quad \text{_____ (9)}$$

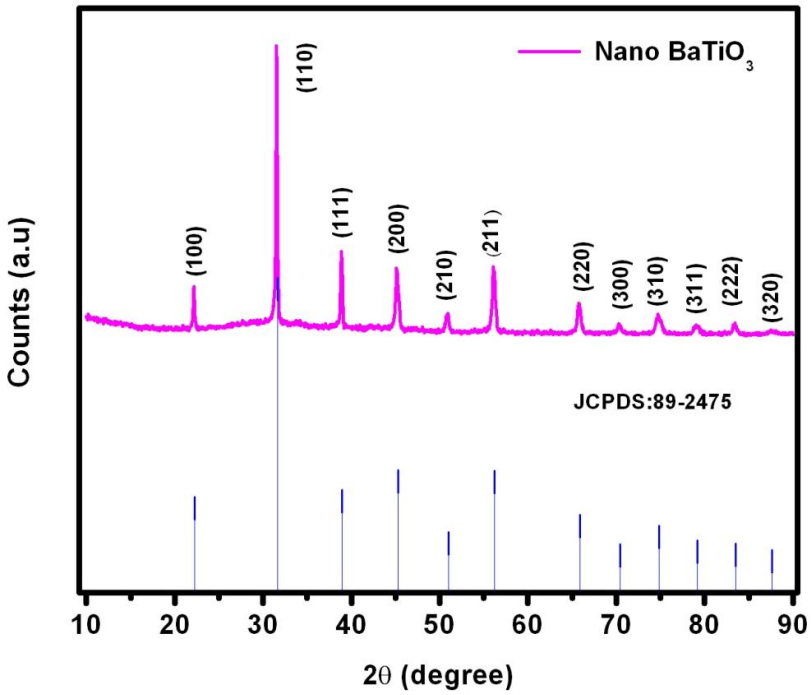
Where β is full width half maxima, λ is wave length of CuK_α radiation (0.1542 nm) and θ is diffraction angle and 'K' is a numerical constant which is equal to 0.9 for a spherical atom.

From the results the crystallite size is in the range of 4 to 44nm again we found to be the mean crystallite size is evaluated as 22nm. More over the lattice constants (a) is estimated after finding the inter-planer spacing (d) and miller indices (hkl) by help of the following formula[10]

$$a = d\,(h^2 + k^2 + l^2)^{1/2} \quad \text{_____ (10)}$$

The X-ray density (D_x) is evaluated using the formula ZM/Na^3, where 'Z' is the number of molecules per unit cell (Z=8), 'M' is the molecular weight of the composition, 'N' is Avogadro's number (6.023×10^{23}) and 'a' is the lattice parameter[10].

The synthesized barium titanate (BT) nano particle having the high homogeneity due to the high D_x value is calculated as6.022 g/cm^3 compare with that the X- ray density of bulk BT of 5.427 g/cm^3. The definite surface area (S) is a significant physical parameter for nanoparticles and is to measure with the help of the relation: $6000/D_pD_x$, where the symbols have their usual meaning [11].Finally the S value is estimates around 50 m^2/g. In the comparison of bulk materials with the surface area (S) value recorded as the minimum crystallite size. In view of maximum S value to influence the barium titanate (BT) nano particles optical and morphological and electrical properties.



1. The diffraction pattern of BT nanoparticles.

SURFACE MORPHOLOGY:

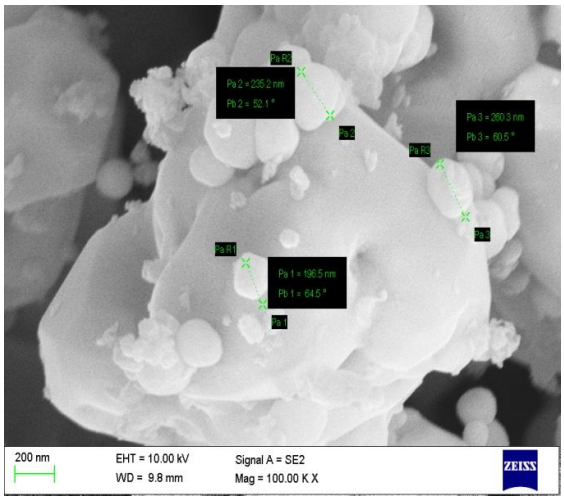
1. FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM) ANALYSIS:

Barium Titanate (BT) nano particles surface morphology study by FESEM and TEM. The BT nano-particles FE-SEM photographs of are showed in Fig.2.

From Fig.2, it is evident that BT show well defined spherical grains. The grain size (G_a) is determined using linear intercept method[12]

$$G_a = 3L / 2MN \quad \text{(4)}$$

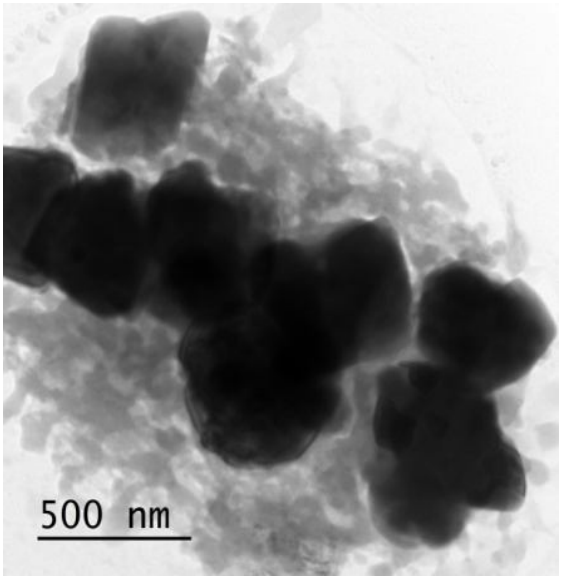
where 'L' is the line length, 'N' is the number of grains intercepting the test line' M' is the magnification. The 'G_a' is calculated to be varying between 195 to 234 nm. This explains the presence of nano spheres.



2. The FESEM photos of BT nanoparticles.

2. TRANSMISSION ELECTRON MICROSCOPY (TEM) ANALYSIS:

Conventionally the Transmission electron microscopy (TEM) illuminated overall the existence of nano particles. In view of this investigation the Barium Titanate (BT) nano particles TEM images are shown in the Fig.3.Moreover the TEM images shown a weak-agglomeration in the Barium Titanate (BT) nano particles. Here it is very clear from Fig.3. The TEM images the Barium Titanate (BT) nano particles are in Spherical shape. Mean while the particle was also estimated in between from 182 to 205nm.

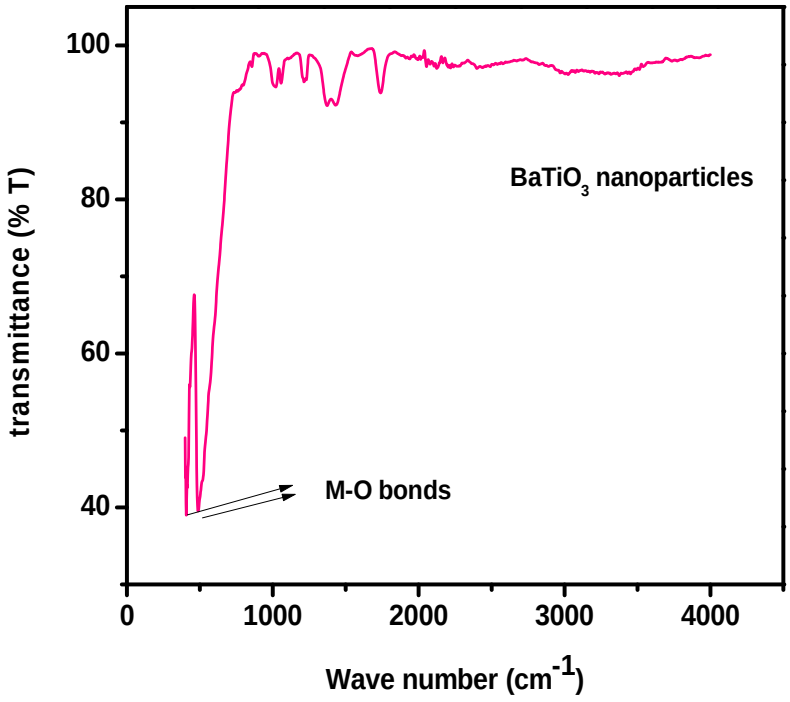


3. The TEM photos of BT nanoparticles.

OPTICAL PROPERTIES:

1. FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR) *ANALYSIS:*

The barium titanate (BT) nanoparticles FTIR absorption spectra were reported within the order of four thousand to four hundred cm^{-1} as shown in the Fig.4. Here the spectra were revealed the two types of metal oxide (M-O) stretching vibrations were found to be in the range of 400 to 520 cm^{-1}. It provides the barium titanate (BT) perovskite structure. In addition to these spectra were exhibits a small absorption band at 413 cm^{-1} meanwhile a large absorption band over 512 cm^{-1}. From the above absorption bands it attributes the existence of Ba-O and Ti-O bonds. Moreover due to the oxygen and hydrogen (O-H) some peaks were observed at 1123 cm^{-1} and 1362 cm^{-1} [11]Again the 2852 cm^{-1} absorption peak was illustrated to the intra-molecular stretching modes it causes from the definite properties of hydrogen bonding [11].



4. The FTIR spectra of BT nanoparticles.

2. UV – VISIBLE SPECTRAL ANALYSIS:

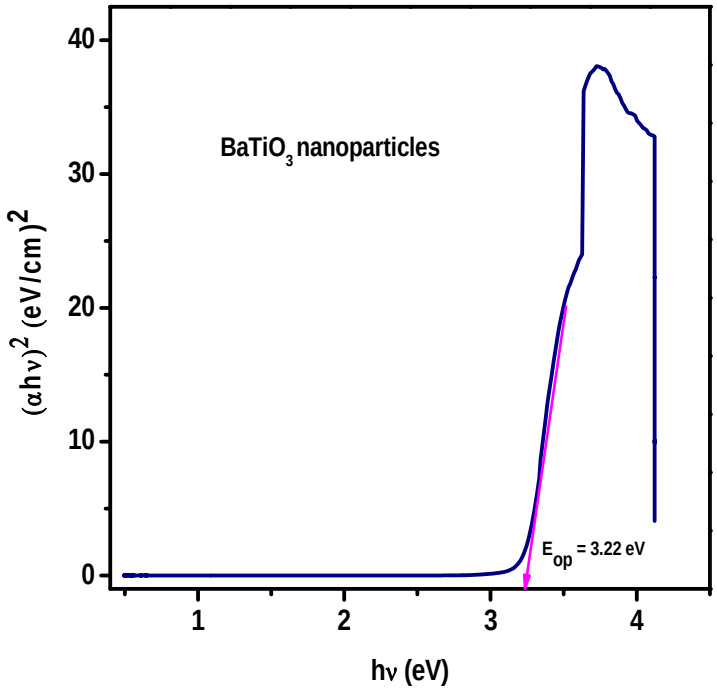
From Fig.6. The difference reflectance spectrum is recorded over a range of 500-2500 nm for calculating optical band gap of powder samples. The Kubelka –Munk function of reflectance F(r) is used to find band gap [11].

$$F(r) = (1-r)^2/2r \quad \_\_\_\_\_ (5)$$

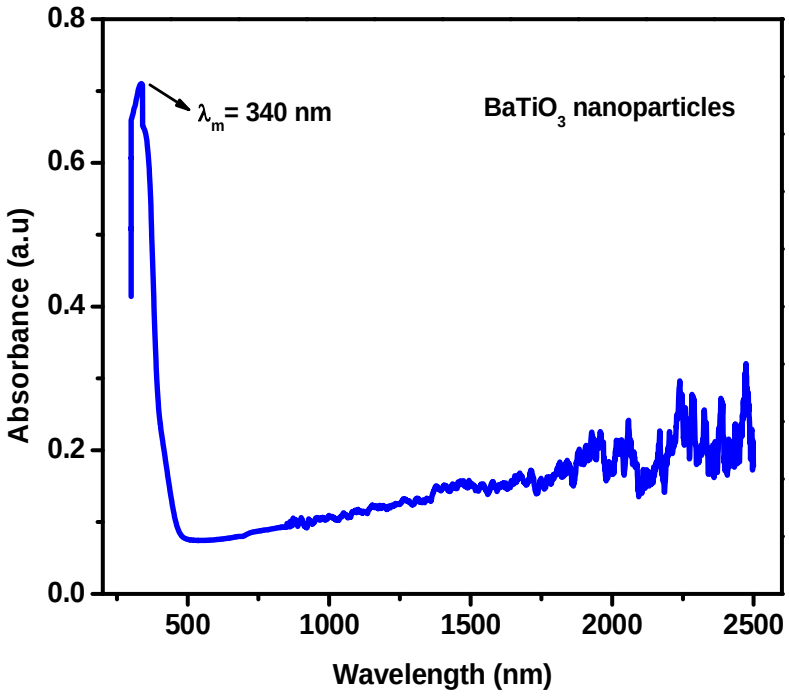
The absorption coefficient (α) is directly proportional to F(r) and hence an equation to find band gap can be reported as follows[11]

$$(\alpha h\nu)^n = m\,(h\nu - E_g) \quad \_\_\_\_\_ (6)$$

Where 'm' is energy–independent constant that depends on transition probability. E_g is optical band gap energy. N based on the kind of transition i.e., n=2 for direct transition, 2/3 for direct forbidden transition, ½ for indirect transition and 1/3 for indirect forbidden transition, and hν is photon energy. In this study n =2 is taken for the direct transition[11].The E_g value is considered as extrapolated tangent value towards x-axis for $(\alpha h\nu)^2$ versus photon energy hν (eV) plot as α tends to zero.



5. The αhν versus photon energy of BT nanoparticles.



6. The absorption spectra of BT nanoparticles.

The maximum absorption wavelength (λ_m) for barium titanate (BT) nanoparticles nano-particles is depicted in Fig .6. It is observed from figure that the λ_m value is acquired to be 340 nm for the BT nanoparticles. The optical band gap is found to be 3.22 eV and is in consistent with previously reported data[8].

CONCLUSIONS:

The Barium Titanate (BT) nano spheres were prepared through Low temperature Hydrothermal method. From the X-ray diffraction method was determine t he cubic perovskite pattern of the sample. The 'G_a' is calculated to be varying between 195 to 234 nm. Against of the Fourier-transform infrared spectroscopy (FTIR) eludicated the formation cubic structure of the material due to the exitence of the metal oxide bonds (M-O). By the help of TEM investigation we found to be the agglomerated spherical nanoparticles. In the extended to evaluate the optical band gap (E_{op}) was to be of 3.22 eV.

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