

## Synthesis of Barium Titanium Oxide Nanoparticles

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### ABSTRACT :

A new synthesis route to obtain high-purity barium titanium oxide nanoparticles, Ba<sub>2</sub>TiO<sub>4</sub>, using the microwave assisted solvothermal reaction of Barium acetate and Titanium tetrachloride solution in an oxygen atmosphere has been developed. The synthesized Ba<sub>2</sub>TiO<sub>4</sub> nanoparticles are nearly spherical in shape. Their grain sizes are determined by the Scherrer's formula. The microstructure and composition of the as-synthesized samples were investigated by X-ray diffraction (XRD).

**Keywords :** Ba<sub>2</sub>TiO<sub>4</sub>, microwave, titanium tetrachloride, barium acetate, solvothermal, XRD.

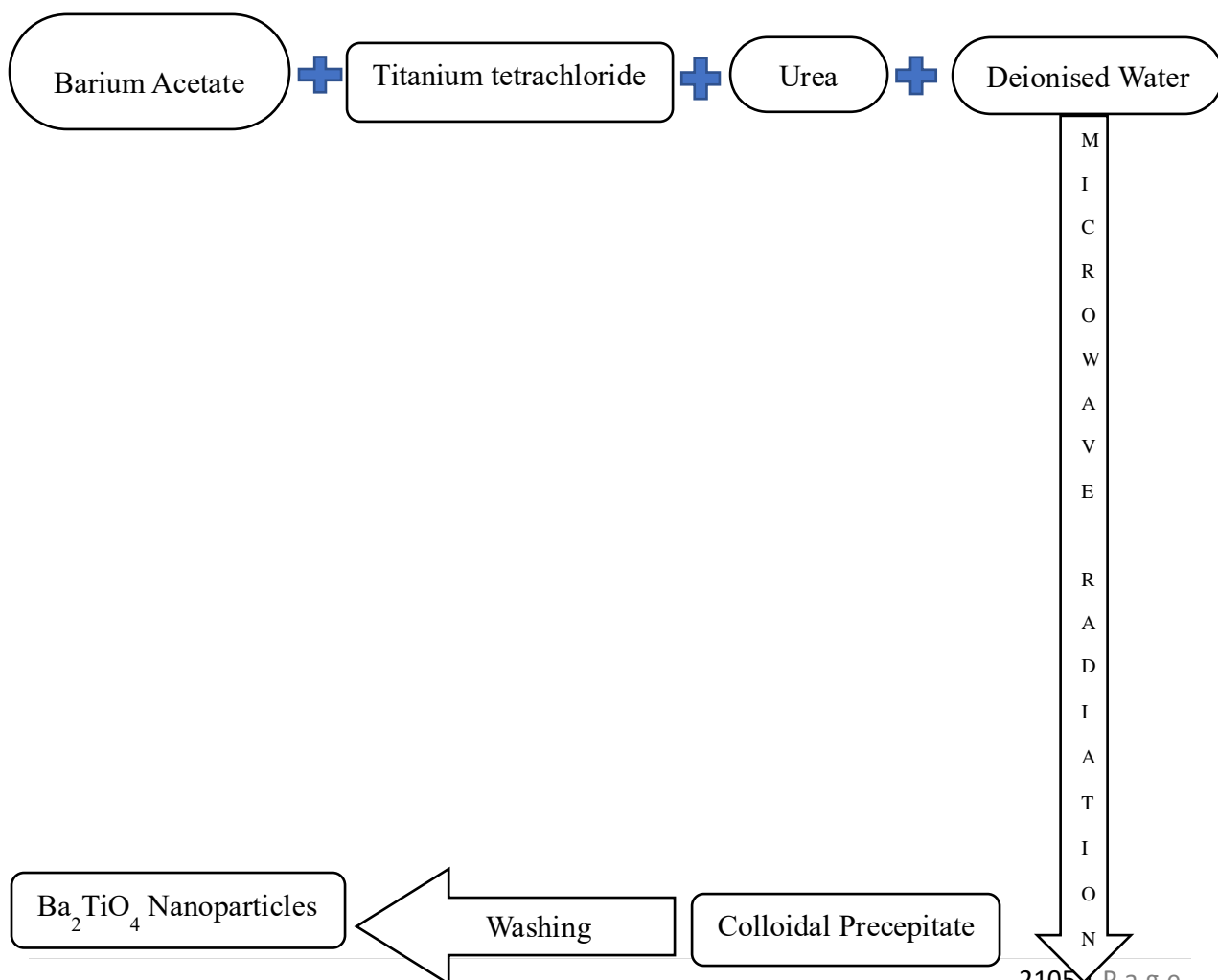
### I. Introduction :

Nanoscience is meant to deal and manipulate the materials particularly, at the molecular level wherein the property modifications may suppose to be emerged. The application potential of nanomaterials is significantly rich in wide variety of fields like biomedical, engineering, agriculture, automobiles, military and defence, buildings and infrastructure, etc [1,2]. The present work is interested in addressing and discussing the size dependent physical properties of nanosize particles with the help of a wide variety of characteristic tools. The properties taken

up to explore belong to structural, optical, morphological, crystalline aspects. Barium acetate ( $Ba(C_2H_3O_2)_2$ ) is the salt of barium(II) and acetic acid. Titanium tetrachloride is the inorganic compound with the formula  $TiCl_4$ . It is an important intermediate in the production of titanium metal and the white pigment titanium dioxide. Barium titanium oxide is an inorganic compound with the chemical formula  $Ba_2TiO_4$ . It is a colourless solid that is of interest because of its relationship to barium titanate, a useful electro ceramic [3]. It is also known as Barium orthotitanate. Barium titanium oxide ( $Ba_2TiO_4$ ) is barium rich and stable. This material can be prepared by various methods such as conventional high-temperature solid reaction, alkoxide precursor synthesis, solvo thermal method, sol-gel method [4], etc. The solid has two known phases: a low-temperature ( $\beta$ ) phase with  $P2_1/n$  symmetry and a high-temperature ( $\alpha'$ ) phase with  $P2_1nb$  symmetry [5].

**2. Materials and Methods :**

In the present work an attempt is made to obtain controlled  $Ba_2TiO_4$  nanoparticles with distilled water as solvent. However, to the best of available knowledge, the preparation of  $Ba_2TiO_4$  nanoparticles by this method is not reported so far.



M I C R O W A V E R A D I A T I O N

The required amount of substance =  $MXV/1000$  (In gram units)

Where,

M - is the Molecular weight of the substance

X - is the Concentration of Molar units

V - is required Volume of solvent

The washed final products were collected and then dried in atmospheric air. The as prepared sample was annealed at 400°C for 1 hour and finally, the products were collected as the yield and the yield percentage was calculated using the relation.

$$\text{Yield percentage (\%)} = \frac{\text{product mass}}{\text{sum of masses of reactant}} \times 100$$

### 3. RESULT AND DISCUSSION

#### 3.1 TG- DTA Analysis

The sample was white in asprepared and pale white in after annealed. Thermal analysis can be defined as the technique in which a property of a sample is monitored against supplied external temperature in a specified atmosphere. In TGA, weight of the sample is measured as a function of temperature or time. The result thus obtained is known as TG curve in which % weight loss is plotted against measurement temperature. Differential thermo gravimetric (DTG) curve is obtained by differentiating TGA plot with respect to temperature. The resulting curve may be attributed to the energy changes associated with absorption or release of heat as a function of temperature [6].

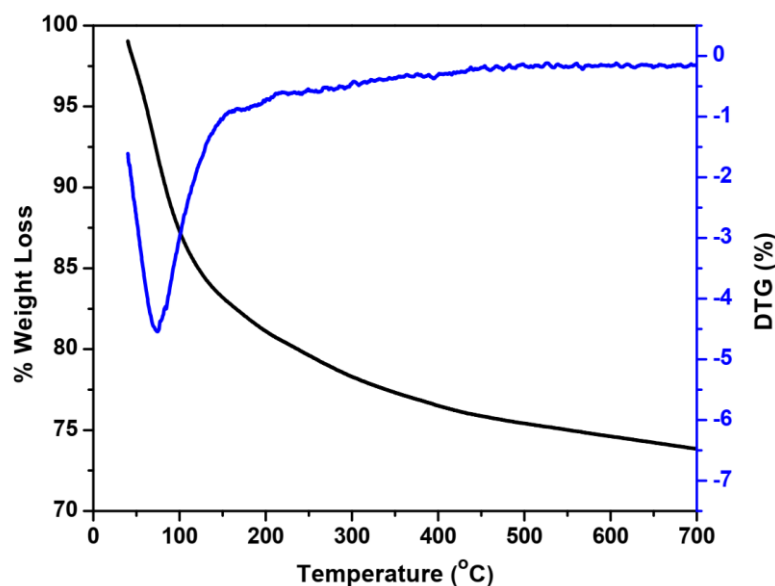


Fig. TGA/ DTG curves of as-prepared Ba<sub>2</sub>TiO<sub>4</sub> nanoparticles

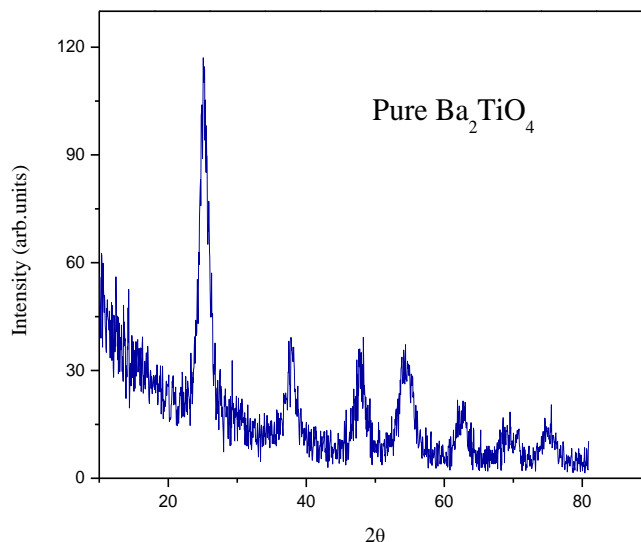
. Thermograph of the as-prepared pure Ba<sub>2</sub>TiO<sub>4</sub> nanoparticles is shown in the above Fig. As the measurement temperature increases, a sudden weight loss associated with an endothermic peak (~ 100 °C) is observed. It may be attributed to the surface adsorbed water molecules that got clung during the preparation of ultra-fine particles via microwave assisted solvothermal process [7].

### 3.2 PXRD Analysis

The prepared samples were carried out Powder X-ray diffraction analysis using an X'Pert Pro-PAnalytic instrument with mono chromated CuK $\alpha$  radiation (wavelength 1.5406 Å), in the angular range 20 – 80° of 2 $\theta$ . Figure shows the XRD pattern of pure Barium Titanium Oxide nanoparticles. The observed peaks could be assigned to the monoclinic phase which is in good agreement to the crystallographic data of Ba<sub>2</sub>TiO<sub>4</sub> (JCPDS: 70-1377). The lattice parameters of the pure barium titanium oxide nanoparticles were a = 6.1458Å, b = 7.6727 Å, c = 10.5673 Å. The crystalline size was the sample is 6nm and the strain value is 0.02860. The lattice of Ba<sub>2</sub>TiO<sub>4</sub> is found to be constraint which is realized from the micro strain values [8]. Full width at half maxima ( $\beta$ ) and lattice strain ( $\epsilon$ ) are related to each other [9] as

$$\epsilon = \beta \cos\theta/4$$

According to Scherrer equation, crystallite size ( $D$ ) and  $\beta$  were inversely related. Thus, strain is found to decrease with increasing crystallite size [10].



**Fig : PXRD pattern of Ba<sub>2</sub>TiO<sub>4</sub> nanoparticles**

Wu and Brown (1973) [11] performed more precise measurements on the crystal structure of  $\beta$ - Ba<sub>2</sub>TiO<sub>4</sub> phase especially Ti-O bond distance which by previous reports lack accuracy due to twinned planes. The general features of the structure proposed by Bland (1961) [12] have been confirmed. Each unit cell contains four isolated TiO<sub>4</sub> tetrahedra linked by barium atoms. Olaf Marks and John R Gunter (1988) [13] reported that the Ba<sub>2</sub>TiO<sub>4</sub> is the only known compound containing the isolated TiO<sub>2</sub> having Ba-rich phase. Further, their results are first of its kind to report the two phases of Ba<sub>2</sub>TiO<sub>4</sub> compounds viz.,  $\beta$ - Ba<sub>2</sub>TiO<sub>4</sub> phase along with  $\alpha$ - Ba<sub>2</sub>TiO<sub>4</sub> phase which is orthorhombic in nature and a superstructure of  $\beta$ - phase.

Thus, Ba<sub>2</sub>TiO<sub>4</sub> prepared finely divided form from the ethanolates of Ba and Ti contains both orthorhombic and monoclinic modifications. The orthorhombic form is present in larger crystallites of more than 1  $\mu\text{m}$  in length, whereas the smaller particles consist of the monoclinic form only.

#### 4. CONCLUSION

We have successfully synthesized pure barium titanium oxide nanoparticles by microwave assisted solvothermal method by using microwave oven. Microwave method is one of the best method in the all other methods because of the method is very easiest method.

The success of this proposed method is the average crystalline size is 6nm. This size is the finest size in all the methods of preparation.

## 5. REFERENCE

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