

# Preparation and Characterization of Barium Titanate Nano Particles Using Solution Combustion

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## Abstract

Ferroelectric materials are gaining increased importance as a result of their high dielectric constants making them useful for electrical capacitors, high piezoelectric constants to make sensors, actuators, RF Filters, ferroelectric hysteresis suit making non-volatile memories, high pyroelectric properties for infra-red detectors, thermistors, and strong electro-optic effects to be used in optical switches, data storage, etc. Barium titanate undergoes change in shape from perovskite into cubic structure Curie temperature which causes polarization, or spontaneous, polarization. Many techniques were described for the synthesis of Barium titanate including solid-state reaction, sol-gel method, hydrothermal and solution combustion. Solution combustion. The last method offers good control of the properties to meet specific requirements of the products and allows the preparation of nanomaterials to suit energy saving and protection of environment made it attractive for many purposes. In the present work barium titanate is synthesized as submicro to nano sized particles using the solution combustion technique utilizing urea and glycerin as fuel / oxidant mixture. The experimental parameters were varied to suit the optimization of the process.

**Keywords:** solution combustion; nano barium titanate; glycerin urea; annealing; IR spectra; x-ray diffraction.

## 1. Introduction

Ferroelectric materials contain one or more polar axes along which a spontaneous polarization can be developed below the Curie temperature [1]. They offer a wide range of useful physical properties. They are: 1) High dielectric constants making them useful for electrical capacitors, 2) high piezoelectric constants useful in making sensors, actuators, RF Filters, 3) Ferroelectric hysteresis useful to make non-volatile memories, 4) high pyroelectric properties for infra-red detectors, thermistors, 5) strong electro-optic effects used in optical switches, data storage, etc.

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Beyond the Curie temperature (130°C) the shape of BaTiO<sub>3</sub> changes from perovskite crystal to cubic structure with Ba<sup>2+</sup> ions at the vertices, O<sup>2-</sup> ion in the face center position and Ti<sup>4+</sup> ion in a body-centered position. This causes polarization, or spontaneous polarization as a result of the unit shift of axially elongated Ti<sup>4+</sup> ion crystal and, hence, BaTiO<sub>3</sub> shows ferroelectric property [2]. The importance of BT, increased as a ferroelectric material. It is characterized by high dielectric constants, high piezoelectric constants. They are utilized in the manufacture of electrical capacitors, actuators, and radio-frequency, RF, Filters. Optically the material is useful to make infra-red detectors and optical switches [1]. Many techniques were described for the synthesis of BT including solid-state reaction, sol-gel method, hydrothermal and solution combustion. Solid-state reaction involves the milling of BaCO<sub>3</sub> or BaO and TiO<sub>2</sub> and calcination at high temperature (800° C to 1300° C) [3-5].

In sol-gel method a chemical precursor is hydrolyzed to form a sol and then a gel, which on drying (evaporation) and pyrolysis gives an amorphous oxide [6]. Barium acetate, tetrabutyl titanate, isopropyl alcohol and glacial acetic acid were starting reagents. Sol-precipitation process was developed, where no further calcination or annealing of the product is required. The single-crystal BaTiO<sub>3</sub> nanoparticles can be directly obtained at the low temperature of 80° C and a strong alkaline condition rather than amorphous gel that often formed in the standard sol-gel process. From transmission electron microscope, TEM, examination nanoparticles with an average diameter of about 20 nm were detected [7]. Boulos and his colleagues [3] synthesized BaTiO<sub>3</sub> powders by the hydrothermal method using two different titanium sources TiCl<sub>3</sub> and TiO<sub>2</sub>. The barium source was BaCl<sub>2</sub>·2H<sub>3</sub>O. Synthesis was performed at two temperatures, namely 150° C and 250° C. To yield spherical highly crystallized elementary grains with sizes in the nanometer range. Microwave, MW, heating was developed with some advantages over the conventional hydrothermal route like the cost and short time of the process starting with Ba(NO<sub>3</sub>)<sub>2</sub>, TiCl<sub>4</sub> and KOH [8, 9]. All chemical reactions were conducted in a MW digestion system to give cubic BaTiO<sub>3</sub> powders within 15 min compared with 150 min for conventional method (Guo and his colleagues [10]). The material was also prepared in a molten salt at 925° C. [11].

The BaTiO<sub>3</sub> may be doped by solid-state decarbonation at 900 °C and sintering at 1400 °C for 8 hrs restoring the tetragonal crystal phase of BaTiO<sub>3</sub> [12]. However, the introduction of Zirconium into the BT structure caused a shift of diffraction pattern to lower angle side. It also affected the structure of perovskite BT [13]. Bachina and his colleagues [14] used various glycine to nitrate mixtures to design a synthesis procedure for nanocrystalline lanthanum ortho ferrite, LaFeO<sub>3</sub>, with average crystallite size of 18 to 85 nm. The specific surface area of the nano powders based on them ranged from 8 to 33 m<sup>2</sup>/g.

The synthesis by solution combustion became a widely accepted method for making a variety of inorganic materials, ceramic and composites[27]. Good control of the properties can be insured to meet specific requirements of the products. The versatility of the method furnished the way to study the optimization of the synthesis conditions. The application of the method towards the preparation of nanomaterials to suit energy saving and protection of environment made it attractive for many purposes.

In the present work, BaTiO<sub>3</sub> micro to nano size particles are synthesized by solution combustion method. The material will be characterized by advanced chemical and physical techniques.

## 2. Materials and Methods

### 2.1 Materials and Reagents

The Ba(NO<sub>3</sub>)<sub>2</sub> and TiO<sub>2</sub> used were analytical grade chemicals. Urea was a general purpose material and glycerin was a 98% pure material. Deionized water was used for all the purposes.

### 2.2 Apparatus

The FTIR spectra of the specimens were recorded on a Shimadzu IRAffinity-1S FTIR spectrophotometer where the samples were analyzed as KBr discs. The X-ray diffraction patterns were recorded on a Diffractometer. The TEM images were also measured

### 2.3 Synthesis Procedures

Barium nitrate (26.1337 g, corresponding to 0.1 mole) was dissolved in minimum amount of deionized water and 7.987 g (0.1 mole) of TiO<sub>2</sub> were added and mixed thoroughly (Mixture A). Certain amount of urea was dissolved in a minimum amount of water (reagent B). Reagent B was added to mixture A. An aliquot of glycerin was added and water was then added to complete 100 mL. The overall composition of reaction mixtures is indicated in Table 1. The mixture was placed on a hot plate set at the maximum temperature (approximately 180° C) with continuous stirring for about 30 min. The mixture was removed from the heater surface and ground in a porcelain mortar. The mixture was divided into four portions. One portion was kept for analysis and physical measurements. The second, third and the fourth portions were placed in a Muffle furnace set at 500° C, 600, and 700° C, respectively. Heating was continued for about 4 hours and the furnace was allowed to cool overnight. The description of the samples is given in Table 1.

The samples were characterized by XRD, TEM and IR spectroscopy.

**Table 1:** Details of barium titanate reaction mixtures and heating conditions\*

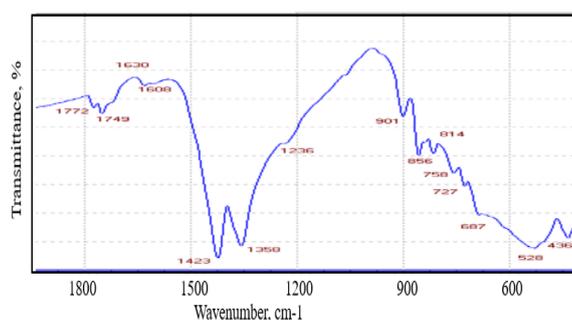
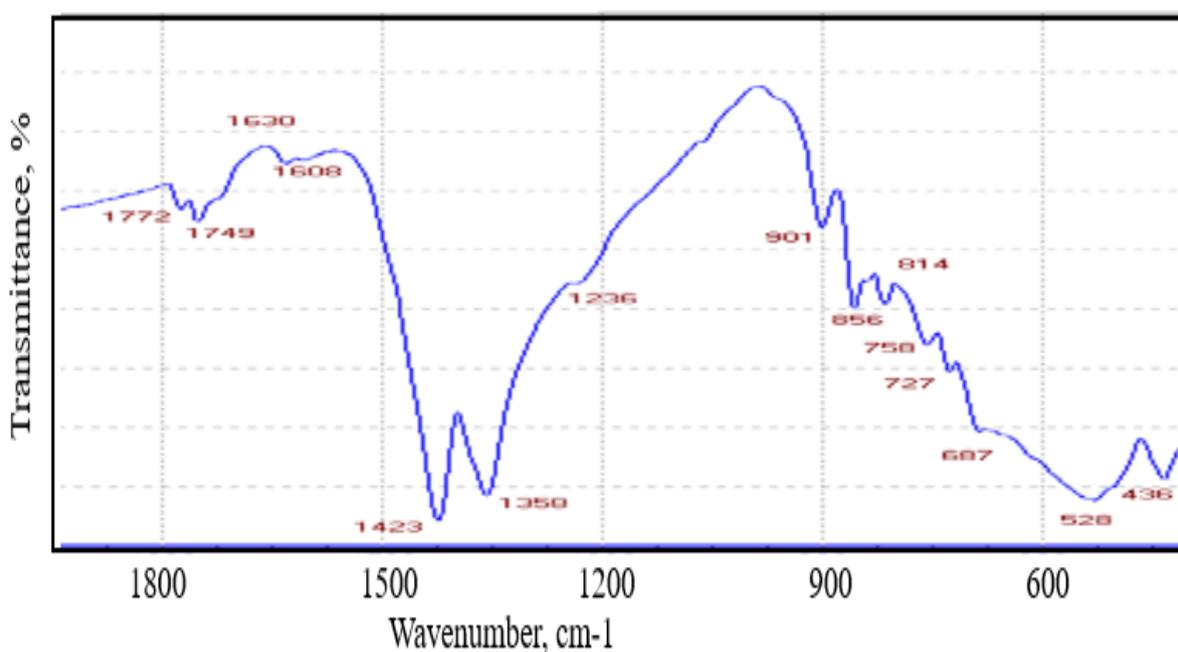
Parameter	Run No.					
	1	2	3	4	5	6
Urea, g	10.0	10.0	10.0	5.0	5.0	5.0
Glycerin, ml	5.0	5.0	5.0	2.5	2.5	2.5
Temperature, °C	500	600	700	500	600	700
Time, hr	4	4	4	4	4	4

\* Barium nitrate (26.1337 g) and 7.987 g of TiO<sub>2</sub>.

## 3. Results and discussion

FT-IR analysis is frequently employed to support the detection of functional groups in organic and inorganic

compounds [15]. Thus, it can be used to follow the progress of chemical reactions by detecting the reduction in peak intensity of peaks related to the reactants and the appearance of those of the products [16]. Ianculescu and his colleagues [17] reported that the absorption bands at wave numbers smaller than  $800\text{ cm}^{-1}$  for thin films BT are related to the Ti - O and Ti - O - Ti bands. In the present work FTIR spectra was recorded for all reaction mixtures and their solution combustion and the annealed products at the designed experiments. Typical spectra are displayed in Figures 1 and 2. A peak at  $1425 \pm 2\text{ cm}^{-1}$  characterized all the analyzed samples. A group of peaks appeared at  $858 \pm 2$ ,  $685 \pm 2$ ,  $584 \pm 2$ ,  $543 \pm 2$ , and  $433 \pm 3\text{ cm}^{-1}$  were also observed. The peaks of the high temperature annealing were sharper than those recorded for low annealing temperature. This is related to the homogeneity of the compounds and agrees with the report of Brundle and his colleagues [18]. The IR peaks at  $485\text{ cm}^{-1}$  and  $732\text{ cm}^{-1}$  which refers to the O-Ti-O bonding of anatase titanium dioxide morphology (Karakitsou and Verykios) [19] did not show up in all spectra indicating complete reaction. Tetragonal BT appeared dominant phase of the reaction product as the characteristic peak at  $584 \pm 3\text{ cm}^{-1}$  [20]. The bands at the range of  $570\text{-}400\text{ cm}^{-1}$  are designated to indicate that cubic and tetragonal  $\text{BaTiO}_3$  exist in the product according to the temperature, especially  $535$  ( $528$ ) and  $579$  ( $582$ )  $\text{cm}^{-1}$  may indicate the existence of the tetragonal  $\text{BaTiO}_3$  [21].

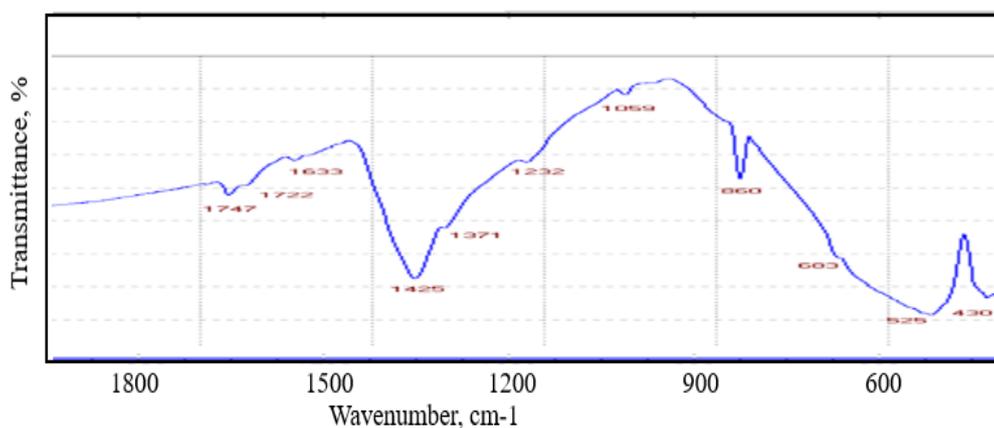


**Figure 1:** The IR spectrum of the material annealed at 500

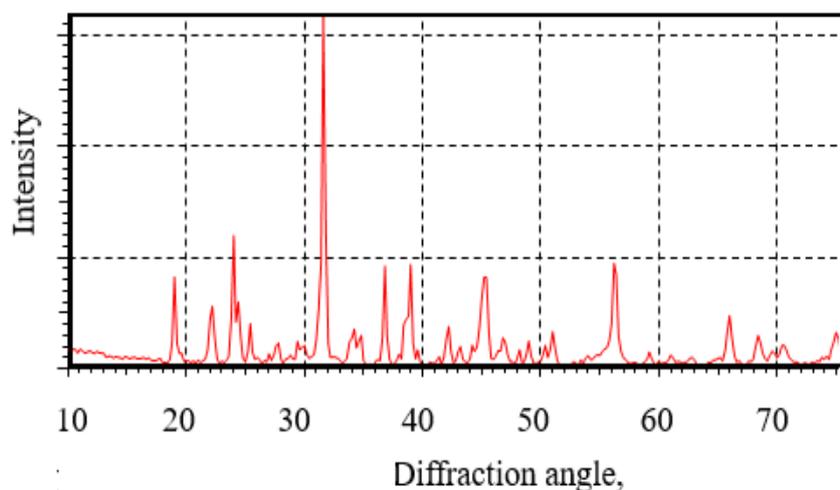
Figure 3 and the first three columns of Table 2 reveal the existence of some remaining barium oxide in the product as given by the XRD peaks at  $2\theta$  19.05 (19.65), 25.43 (26.91), 34.04 (33.619), 42.16 (43.0288), 45.21 (44.8540), 56.24 (55.9910), 60.9634, and 68.40 (68.237). The values in parenthesis refer to the barium oxide standard XRD peaks given by Bazeera and Amrina, 2017 [22].

### 3.1 X-ray diffraction, XRD, patterns

The examination of the specimens by XRD was indicative for the degree of reaction completion as well furnishing information about the structure and the crystalline nature of the products. Table 2 shows the XRD data of the products from reaction heated by a mixture of 5.0 g urea + 2.5 g glycerin.



**Figure 2:** The IR spectrum of the material annealed at 700°C

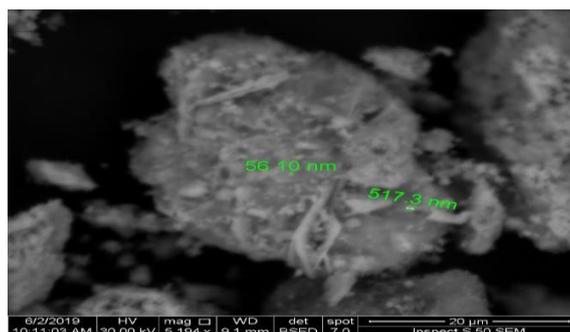


**Figure 3:** XRD pattern of the product in the presence of 5.0 g urea and 2.5 g. glycerin heated at 500°C

A large number of peaks showed up referring to the produced BT in addition to some remaining reactants and

intermediate products (BaO). The main peaks obtained were 22.48 (22.2), 31.63 (31.5), 38.97 (38.9), 45.24 (45.3), 51.17 (51), 56.23 (56.2), 65.93 (65.8), 70.47 (70.4), 74.95 (74.9) and 79.46 (79.4); the values in parenthesis refer to the BT synthesized by reaction in molten chloride matrix at 925° C [11]. The Table 2 shows complete disappearance of TiO<sub>2</sub> diffraction peaks [23] from the reaction products from all of the studied samples.

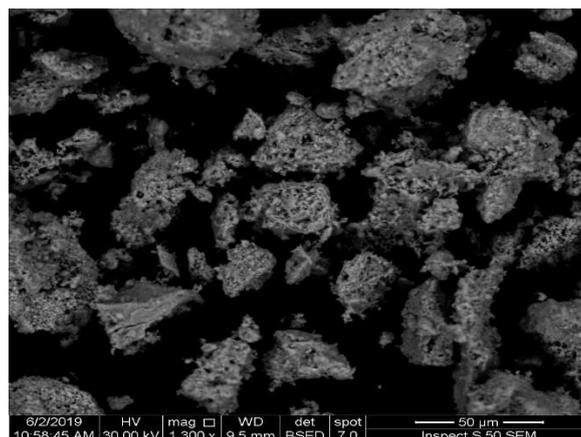
This is a solid indicator on the activity of the solution combustion to supply the required energy to incorporate TiO<sub>2</sub> in the reaction which necessitated heating at temperatures exceeding 900° C. [24]



**Figure 4:** SEM image of the product in the presence of 5.0 g urea and 2.5 g. glycerin heated at 500° C

**Table 2:** XRD data of the products from reaction heated by a mixture of 5.0 g urea + 2.5 g glycerin

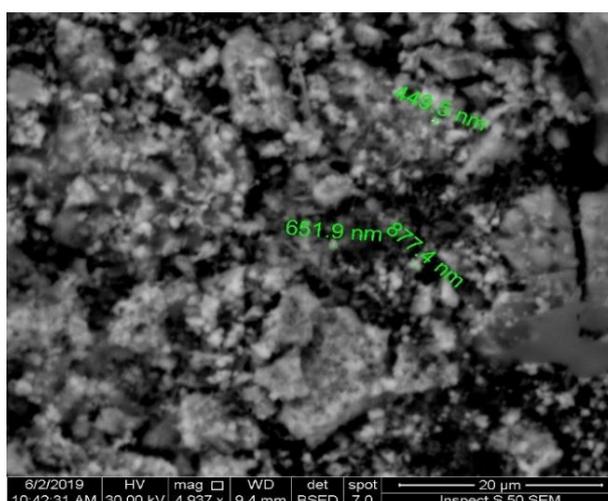
Peak No	500° C,			600° C.			700° C.		
	2θ	D space, Å	I/I1	2θ	D space, Å	I/I1	2θ	D space, Å	I/I1
1	19.05	4.65343	22						
2	22.18	4.00367	19	<b>22.36</b>	<b>3.97271</b>	<b>17</b>	<b>22.44</b>	<b>3.95878</b>	18
3	24.12	3.68744	36	<b>24.18</b>	<b>3.67651</b>	<b>13</b>	<b>24.30</b>	<b>3.65967</b>	14
4	25.43	3.49956	10						
5	31.62	2.82692	100	<b>31.72</b>	<b>2.81867</b>	<b>100</b>	<b>31.82</b>	<b>2.8100</b>	100
6	34.04	2.63175	11						
7	34.84	2.57318	8						
8	36.82	2.43901	27						
9	38.84	2.31674	28	<b>39.06</b>	<b>2.30396</b>	<b>31</b>	<b>39.16</b>	<b>2.29840</b>	25
10	42.16	2.14179	11						
11	45.21	2.00400	34	<b>45.36</b>	<b>1.99782</b>	<b>38</b>	<b>45.45</b>	<b>1.99377</b>	33
13	46.79	1.93985	8						
14	48.97	1.85847	6						
15	50.43	1.80821	6						
16	50.98	1.78979	11	<b>51.04</b>	<b>1.78796</b>	<b>11</b>	<b>51.17</b>	<b>1.78377</b>	10
17	56.24	1.63424	38	<b>56.32</b>	<b>1.63228</b>	<b>40</b>	<b>56.42</b>	<b>1.62957</b>	38
18	65.93	1.41567	16	<b>65.98</b>	<b>1.41462</b>	<b>22</b>	<b>66.07</b>	<b>1.41290</b>	19
19	68.40	1.37037	10						
20	70.50	1.33460	7	<b>70.55</b>	<b>1.33378</b>	<b>6</b>	<b>70.65</b>	<b>1.33211</b>	6
21	74.95	1.26608	12	<b>74.98</b>	<b>1.26558</b>	<b>15</b>	<b>75.06</b>	<b>1.26441</b>	12
22				<b>79.29</b>	<b>1.20728</b>	<b>7</b>	<b>79.38</b>	<b>1.20606</b>	7
23				<b>83.58</b>	<b>1.15585</b>	<b>7</b>	<b>83.67</b>	<b>1.15489</b>	6



**Figure 5:** SEM image of the product in the presence of 5.0 g urea and 2.5 g. glycerin heated at 700° C

The SEM image (Figure 4) indicated the submicron (517.3 nm) particles partially agglomerated with very few fine particles (56.1 nm). Thus, low fuel / oxidant amount coupled with low annealing temperature (500° C) allowed only partial disintegration of the particles. By employing higher annealing temperatures, (600 and 700o C), it appeared that the prepared material has fine-grains with a submicron to nanoscale in the range of 65- 310 nm (Figure 5) which is close to those obtained by Khort and Podbolotov [1]. Probably, the main reason for such morphology is that the exothermal reactions during combustion proceed while the components are in a foam like state, which, as mentioned above, originates during boiling of the liquid charge. The aggregation of flaky formations could be explained by a high temperature attained during the synthesis and a high rate of exothermal reactions.

The SEM image indicated clear crushing of the particles into almost homogeneous state but with irregular shapes and sharp edges within the submicron range of size. Thus, the increase in annealing temperature (700° C) caused the disintegration of particles.



**Figure 6:** XRD pattern of the product in the presence of 10.0 g urea and 5.0 g. glycerin heated at 500° C

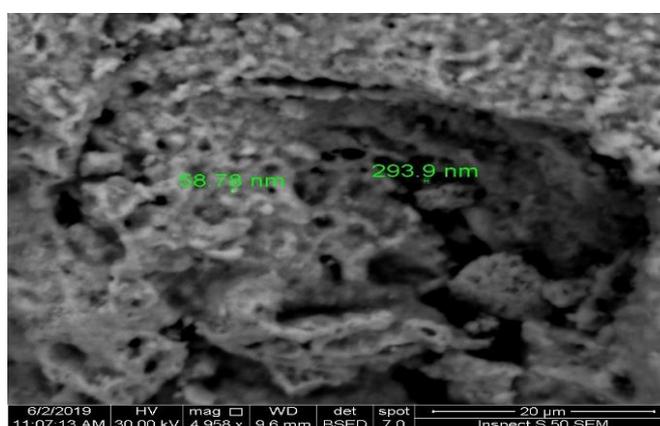
Table 3 shows the XRD data of the products from reaction heated by a mixture of 10.0 g urea + 5.0 g glycerin.

The excess Urea/glycerin content resulted in sort of explosion to yield almost homogeneous mixture of BT particles.

The SEM image (Figure 6) indicated the submicron (449.5-877.4 nm) particles partially agglomerate. Thus, the excess fuel / oxidant amount coupled with 600° C annealing temperature allowed overall crushing of the particles into such low particle sizes and homogeneous. Lee and his colleagues [25] found that the particle size can be easily tuned by adjusting the experimental parameters of the Solvothermal process while retaining the tetragonality.

**Table 3:** XRD data for the products prepared by a mixture of 10.0 g urea + 5.0 g glycerin

Peak No	500 °C.			600 °C.			700 °C.		
	2θ	D spac, Å	I/I1	2θ	D spac, Å	I/I1	2θ	D spac, Å	I/I1
100	22.48	3.95129	18	22.25	3.99217	17	22.53	3.94301	16
2	24.32	3.65676	20	24.08	3.69193	21	24.35	3.65218	22
101	31.85	2.80708	100	31.63	2.82675	100	31.91	2.80228	100
4	34.54	2.59436	8	34.29	2.61260	7	34.24	2.61684	7
111	39.21	2.29593	27	38.97	2.30894	28	39.25	2.29325	30
6	42.30	2.13464	5	42.09	2.14489	7	42.34	2.13299	6
002	45.47	1.99299	30	45.24	2.00284	28	45.51	1.99162	29
8	46.98	1.93249	5				47.07	1.92911	6
210	51.17	1.78370	9	50.99	1.78945	9	51.24	1.78137	11
10	56.46	1.62846	35	56.23	1.63445	39	56.50	1.62729	38
220	66.14	1.41168	16	65.93	1.41558	18	66.17	1.41108	20
13	70.68	1.33164	6	70.47	1.33521	6	70.76	1.33043	6
310	75.15	1.26323	10	74.95	1.26607	10	75.18	1.26267	11
15	79.46	1.20514	5	79.29	1.20731	6	79.45	1.20523	6
16	83.77	1.15370	6	83.57	1.15599	7	83.79	1.15357	7



**Figure 7:** SEM image of the product in the presence of 10.0 g urea and 5.0 g glycerin heated at 700° C

Further increase in annealing temperature up to 700° C resulted in the production of the smallest sizes particle of all specimens. The SEM image (Figure 7) indicated the presence of (58.78 to 293.9 nm) sized particles. Thus, the excess fuel / oxidant amount coupled with 700° C annealing temperature allowed the crushing of the particles to such low particle sizes. This is in agreement with Maison and his colleagues [26] results using catecholate process.

#### 4. Conclusions

Solution combustion can be successfully employed for the preparation of barium titanate utilizing the urea glycerin (fuel/oxidant) system the increase of the use of higher fuel/oxidant content helps in the reduction of annealing temperature. The oxidation process supplies the required heat energy for the reaction and acts to initiate the dissociation of the particle into smaller sized particulate in the submicro to nano scale sizes.

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