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A COMPREHENSIVE REVIEW OF PREPARATION METHODS AND THEIR IMPACT ON BARIUM TITANATE PROPERTIES

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ABSTRACT

The present research investigates various techniques for producing barium titanate ($BaTiO_3$) in depth, highlighting how each affects the material's morphology and crystallography and consequently, its electrical and piezoelectric properties. Among the preparation methods taken into consideration were chemical-mechanical, hydrothermal, solid-state reaction, sol-gel, and microwave processes. The study demonstrated that by altering the preparation method, different characteristics and properties can be achieved. Furthermore, the study successfully proved the enhancement of the properties of nanomaterials through the synthesis of $BaTiO_3$ using advanced techniques, aiming to achieve better results in the future and increase its applicability in modern electronic devices.

KEYWORDS

Barium titanate (BaTiO3), Dielectric Properties, Piezoelectric Properties, Synthesis Methods, Morphology.

1. INTRODUCTION

Barium titanite is a ferroelectric material that is broadly utilized in various electronic fields like capacitors, sensors, or power transformers due to its insulating and piezoelectric properties. Advanced materials research has shown that the performance of $BaTiO_3$ is highly in need of on its processing conditions and manufacturing methods resulting in variations in crystal structure and grain size, and ultimately changing material properties throughout the application process. There are several ways to synthesis barium titanate, including solid-state processes (Dash S., et al.2018), sol-gel methods (C.J. Brinker, G.W. Scherer 2013; L.C. Klein 2013, hydrothermal methods, chemical synthesis and microwave assistance. These different methods create distinct properties in barium titanate that ultimately determine its properties (Sobha A., et al. 2017). Wu et al. 2013 synthesized barium titanate using the sol-gel method, and through this method they were able to achieve the purity of the crystals. Moreover, the dielectric constant can reach the highest level because this method provides a homogeneous and small particle size distribution while impurities are missing and the crystals are uniform and this will affect the piezoelectric properties of the material (Wu, X., et al., 2013) It will be important to add numbers to your statements. Kim et al. 2010 conducted a research; the method that was successfully used in this particular study involved solid-state reaction for making $BaTiO_3$. C.J. Brinker, et al. 2013 In this particular study, it was found that the particles of nanoparticles that were synthesized by this method had a larger size. However, the process was heavily affected by impurities and thermal effect leading to decrease in dielectric constants and piezoelectric properties although (Kim, H. S., et al. 2010). Zhang et al. (2018) synthesized Barium titanate through a process called mechanochemical synthesis and high-energy ball milling. In the materials they produced, L.C. Klein, 2013 such processes resulted in nanoscale grain sizes and increased surface area. M. Selvaraj, et al. 2015 This leads to better piezoelectricity due to the fine-tuned increased surface area unlike the earlier instances where piezoelectricity was based on bulk properties. Moreover, it’s worth noting that while there was enhancement of piezoelectric properties by precisely controlled increase in surface area; unfavorable mechanical strains could result into structural disorder and impurities causing changes in electrical conduction characteristics (Zhang, Y., et al. 2018). Inada et al. 2015 used the method of hydrothermal processing. It is worth noting that the method used by Inada and his team results in barium titanate crystals of high purity and low grain sizes vis-à-vis better dielectric constants and more effective piezoelectric properties as a consequence of size and purity control processes (Inada, M., et al. 2015). Lee et al. 2020 produced barium titanate using the microwave-assisted method. According to the study, E.K. Nyutu, et al. 2008 it reduces time and utilizes little energy in comparison with other processes instead of obtaining nanoceramics with low grain size and high purity able to enhance dielectric constants and piezoelectric performance, H. Zarkoob, et al. 2012 although in order to prevent nonuniform heating, it is imperative that this approach be adequately overseen (Lee, J. H., et al., 2020). To effectively apply barium titanate in high-technology applications, it is necessary to investigate different synthesis processes for their effects on its dielectric, structural and piezoelectric properties. E.K. Nyutu, et al. 2008 This paper delves into these strategies with an adjacent focus on the morphology features formed as well as their consequences on the behavior of $BaTiO_3$.

2. SYNTHESIS METHODS

2.1. Solid-State Reaction

Barium carbonate ($BaCO_3$) and titanium dioxide (TiO_2) were combined to create barium titanate ($BaTiO_3$) powder through a solid-state reaction technique. The ratio of $BaCO_3$ to TiO_2 was maintained at 1:1. The equimolar quantities of dry $BaCO_3$ and TiO_2 were carefully weighed and mixed using an agate mortar and pestle in acetone for two hours. The mixture was dried and maintained at 60°C before being calcined at 900°C for three hours, with a controlled heating and cooling rate of 3°C per minute. The calcined powder was subsequently pulverized to produce a fine powder, where the reaction $BaCO_3 + TiO_2 \rightarrow BaTiO_3 + CO_2$ was observed during heating. The preparation process, illustrated in Fig. 1, adheres to the conventional solid-state synthesis technique (Seong, Hyeok, Choi et al. 2023).

The solid-state synthetic doctrine was also used to enhance the degree of crystallinity of the $BaTiO_3$ powder during the synthesis of $BaTiO_3$. In order to maintain a homogeneous composition, a high crystalline nature, and a reduced particle size of the $BaTiO_3$ dielectric ceramic powder a fine nucleating agent was designed before its preparation. Also, to aid in the uniform synthesis of the precursors, 25 weight percent of 50nm $BaTiO_3$ was added shown in Fig.1 (Monika, Singh et al. 2017). This is consistent with the strategies that it has been observed that dramatic changes in the process can be achieved by altering the solid-state reaction kinetics (Brzozowski & Castro, 2000). In the range of the research already performed there are also findings concerning changes in the ways of performing the synthesis of $BaTiO_3$. For example, Yuh et al. 2005 where electrospinning nanofibers of bario titanate were synthesized, showing the possibility of nanostructured forms. Ashiri et al. 2011 developed an improved way relative to the existing technique for $BaTiO_3$ nanoparticles so as to improve on the purification and morphology. O'Brien et al. 2001 considered the case of $BaTiO_3$ nanocrystals of very narrow dispersion, following the example of the general strategy for preparing nanoparticles of oxides.

All these relate to the improvements made in the order and symmetry of dimensions and shapes of objects as controlled solid reactions in the course of the process (Kambale, Kulkarni, & Venkataramani, 2014).

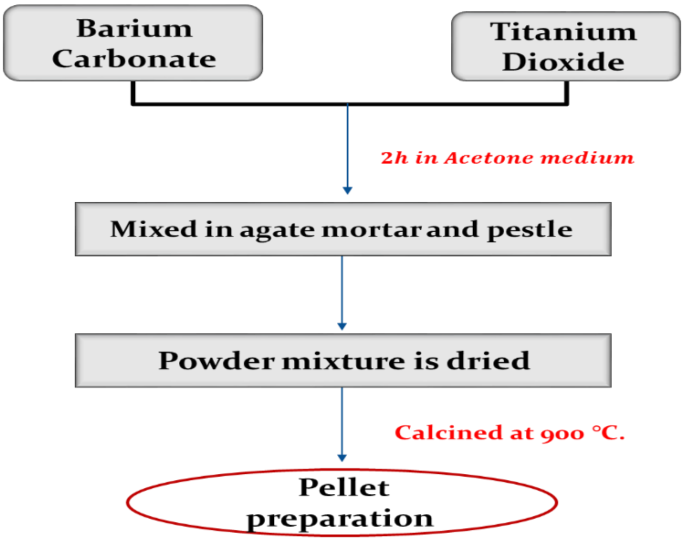

Fig. 1. Flow diagram of Solid-State Method for the preparation of BaTiO3

2.2. Sol-Gel Method

Barium titanate powder ($BaTiO_3$), was prepared using the sol-gel method as described by (Sherlin, Vinita et al. 2023) and (Chen et al. 2008). The starting raw materials included titanium tetra isopropoxide and barium acetate powders, while 2-methoxy ethanol was used as a dispersing medium. Two separate solutions were prepared by stirring 6.939 ml titanium isopropoxide and 5.476 g barium acetate in 2-methoxy ethanol for two hours. These two powders were then mixed to prepare a stock suspension, which after stirring for a further two hours resulted in a sol of $BaTiO_3$ (Lemoine et al. 1994). The sol was then subjected to infrared (I.R.) irradiation to assist in the gelation process. The gel formed was kept in the oven for drying at 80°C and then $BaTiO_3$ powder was obtained after annealing it in a controlled electric furnace for two hours at 650°C. The annealed product was additionally processed in a mortar to create a very fine crystalline powder (Kareiva et al. 1999). This observation was found to be in accord with previous reports on the sol gel processing technique that have been successful in producing high quality and purity barium titanate powder with a controllable particle size distribution (Pfaff, 1992). In Fig. 2, a flowchart represents the process of the fabrication of $BaTiO_3$ powder.

2.3. Hydrothermal Synthesis

$BaTiO_3$ was synthesized using the Ba-Ti-OH precursor through a precipitation process followed by a standard hydrothermal (C-H) technique. Initially, a 1 M solution of $BaCl_2$ was mixed with 15 ml of water, while a 2 M solution of $TiCl_4$ was dissolved in 5 ml of water. These solutions were combined at room temperature. Subsequently, the precipitation of Ba-Ti-OH occurred upon the addition of 10 ml of 10 M NaOH aqueous solution to the Ba-Ti mixture. Hydrothermal treatment was performed by adding 20 ml of deionized water or 5 ml of ethylene glycol (EG) followed by an additional 15 ml of deionized water, resulting in a total slurry volume of 50 ml. The use of 10 vol% EG promoted the formation of tetragonal $BaTiO_3$ rods, while higher EG concentrations, such as 10 ml (20 vol%), inhibited the formation of $BaTiO_3$ products (Dutta & Gregg, 1992, Chen & Chen, 2003). The precursor slurries were processed in a Teflon-lined autoclave using the C-H method. Post-treatment, the solid material was separated by centrifugation, washed multiple times with deionized water, and dried at 50°C. Hydrothermal synthesis using water alone at 200°C resulted in cubic $BaTiO_3$ nanocrystals, while the use of 10% ethylene glycol at the same temperature facilitated the production of tetragonal $BaTiO_3$ nanorods. This behavior is attributed to ethylene glycol preventing the crystallization of cubic $BaTiO_3$ by dissolution-reprecipitation mechanisms during hydrothermal treatment, favoring the formation of tetragonal $BaTiO_3$ (Eckert Jr et al. 1996, Reverón et al. 2005, Wang et al. 2023). The process highlights the role of ethylene glycol in modifying crystallization dynamics and particle morphology, consistent with earlier studies exploring the influence of synthesis parameters on $BaTiO_3$ properties (O'Brien et al., 2001). These findings align with strategies aimed at controlling particle size and phase uniformity in oxide nanoparticle synthesis.

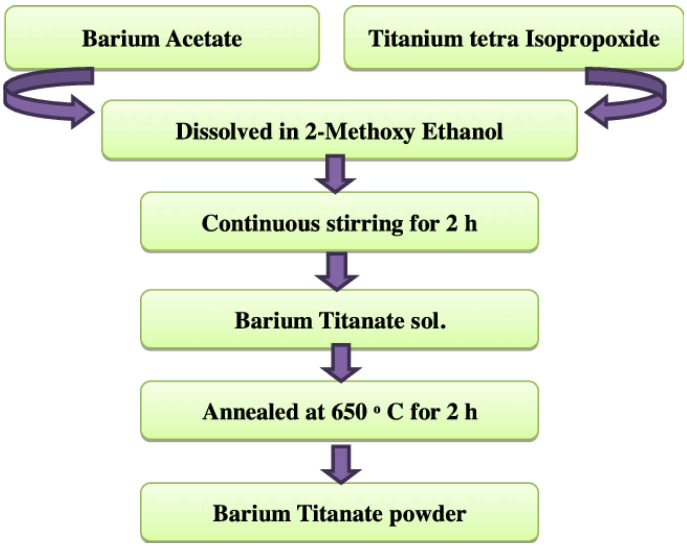

Fig. 2. Sol-gel synthesis schematic for barium titanate powder (V., Sherlin, Vinita., et al. 2023)

2.4. Mechanochemical Synthesis Using Ball Mill

To achieve a homogeneous structure in $BaTiO_3$, the oxides used in its synthesis were initially thermally treated to remove absorbed moisture, ensuring optimal reaction conditions. These oxides were subsequently combined in stoichiometric amounts and thoroughly mixed using an agate mortar. The blended mixture underwent mechanical processing in a high-energy planetary ball mill (Activator 2S). The milling process was performed at room temperature in air, utilizing a 250 mL Cr-Ni steel reaction vessel containing 10 mm diameter steel balls. The vessel was operated at a rotation speed of 1100 rpm for 1.5 hours, with a ball-to-powder weight ratio (BPR) of 40:1. This method ensured the fine dispersion and uniformity of the reactants, critical for the successful formation of $BaTiO_3$ (Stojanovic et al. 2005, Miclea et al. 2007). High-energy ball milling has been demonstrated as an effective mechanochemical approach for synthesizing $BaTiO_3$, offering advantages in controlling particle size and enhancing material homogeneity. Additionally, this process minimizes the presence of impurities and promotes the desired crystallographic phase in the final product. Studies have also shown that such mechanochemical treatments influence the microstructure and improve the properties of the resulting $BaTiO_3$ ceramics (Idehenre et al. 2018). This synthesis approach is widely adopted in the preparation of advanced ferroelectric materials due to its efficiency and reliability.

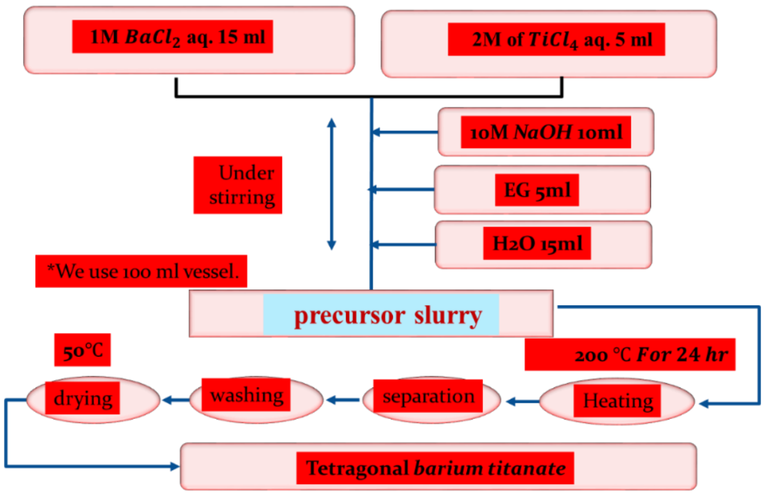

Fig.3. Flowchart of synthesis of Barium titanate powder by Hydrothermal method

2.5. Microwave-Assisted Synthesis

The process for synthesizing $BaTiO_3$ powder using microwave synthesis involves several steps. First, $BaCO_3$ and TiO_2 are separately subjected to high-energy milling for 4 hours. The milled powders are then dried. Alanine is added to the dried materials, and the mixture undergoes additional milling for 2 hours. The milled mixture is then spray dried. SiC microspheres are incorporated into the spray-dried mixture. This mixture is then subjected to microwave heating, facilitating the reaction to form $BaTiO_3$. Finally, the synthesized powder is mechanically sieved to obtain nanosized $BaTiO_3$ particles (see Fig. 5) (Reverón, H., et al. 2005, de Conti, M., et al. 2022).

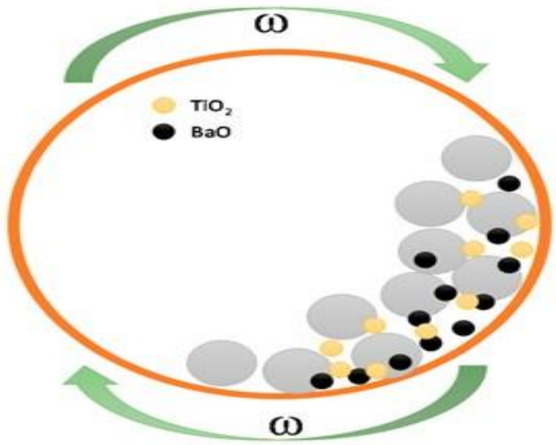

Fig.4. Flowchart of synthesis of Barium titanate powder by mechanochemical method using ball mill. (Sonia, Kudłacik-Kramarczyk., et al. 2020).

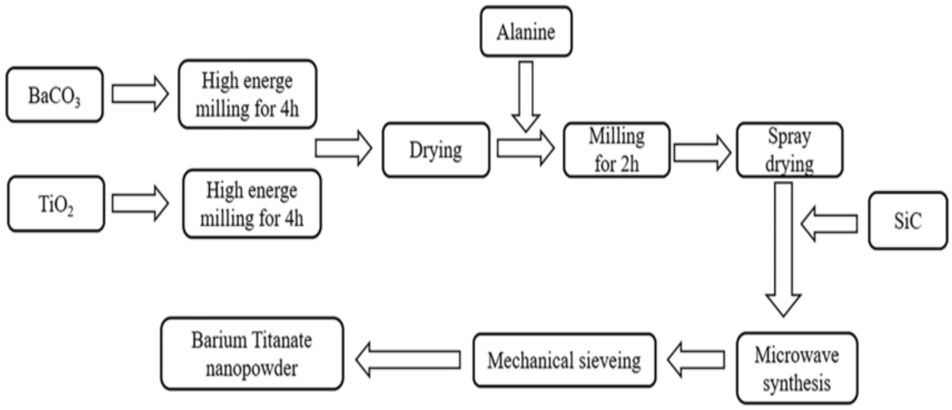

Fig. 5. Flow chart of microwave synthesis of $BaTiO_3$ powder (Rao, K. J., et al. 1997).

3. RESULTS AND DISCUSSIONS

3.1. Characterization

The manufacturing process affects the properties of barium titanate material, the crystalline structure, grain size, and defects that may be affected by each of these various methods, thus affecting the general properties of the material, as the uniformity of the size of grains and crystals in barium titanite is necessary for the dielectric behavior of the material and its electrical properties. The production of pure $BaTiO_3$ phases is confirmed by X-ray diffraction (XRD), while homogeneous grain distribution is revealed by scanning electron microscopy (SEM). While FTIR can determine functional group in structure (Haoyu, Qian., et al. 2020, Zhao, Z., et al. 2011). $BaTiO_3$ formed by the sol-gel procedure involves distinct molecular interactions found in Fourier transform infrared (FTIR) spectroscopy and well-defined crystalline phases demonstrated in the XRD spectrum. The dielectric properties of barium titanite depend on a homogeneous nanostructure, as revealed by SEM images (Kwon, S. Y., et al. 2015, Uchino, K. 2000). The hydrothermal method can produce cubic and tetragonal BaTiO3 phases depending on conditions. XRD and transmission electron microscopy (TEM) analyzes confirm the presence of uniform crystalline phases and fine nanosized (Ding, X., et al. 2008, Yoshimura, M., et al. 2008). During mechanochemical synthesis, high-energy ball milling produces $BaTiO_3$ with fine particle sizes and phases. As the molecular network of the material improved significantly, it led to reconfiguration according to the results of XRD and SEM images. Therefore, this is one of the best ways to stimulate specific properties in barium titanate for various products (Hernández, J. C., et al. 2006). Microwave-assisted synthesis provides quick heating and homogeneous temperature distribution, resulting in $BaTiO_3$ with excellent purity and tiny particle size. The addition of SiC microspheres to microwave conductors improves heating uniformity. XRD study reveals the creation of pure $BaTiO_3$ nanostructure, whereas SEM images indicated well-dispersed nanoparticles (Clark, D. E., et al. 1996, Perera, S. D., et al. 2014). Fig.6 shows the morphologies of barium titanate produced by different techniques, grain dispersal, and evenness by method, which will influence the electrical and piezoelectric properties of barium titanate.

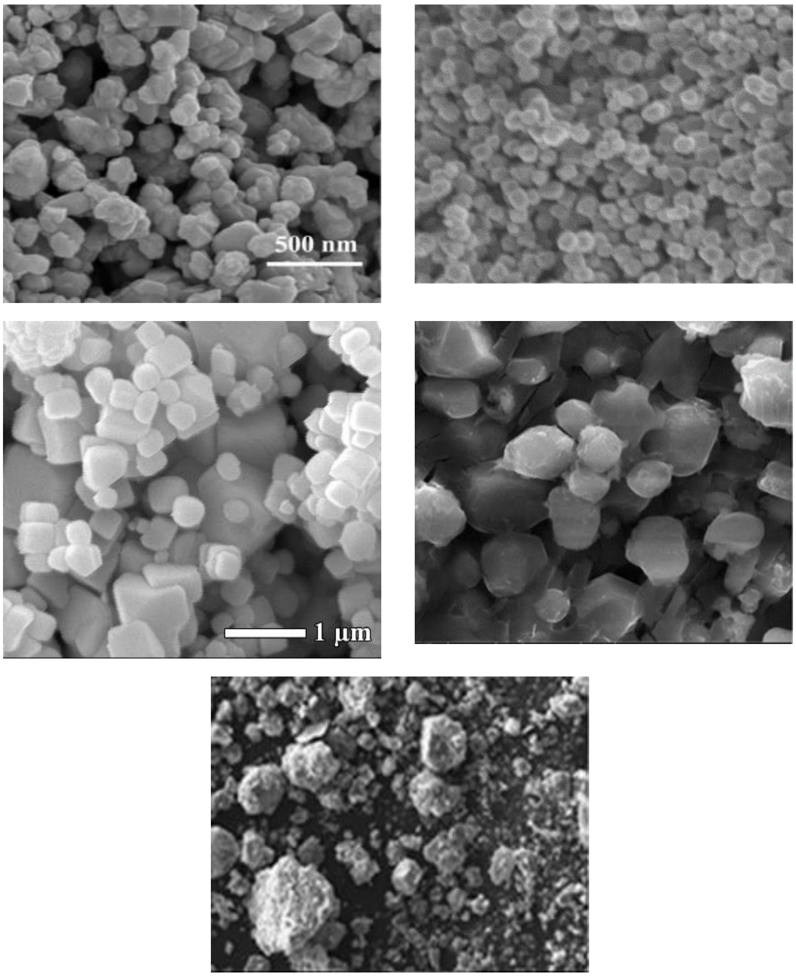

Fig.6. SEM Images of (a) Solid State method (b) Sol-Gel method (c) Hydrothermal process (d) Mechanochemical, and (e) Microwave-assisted Synthesis technique (Sonia, Kudłacik-Kramarczyk., et al. 2020), (Wan L, et al. 2022, Yu P, Liu W, et al. 2022, Özen M, et al. 2018, Qian H, et al. 2020).

3.2. Physical and Chemical Properties of Barium Titanate (BaTiO)

Barium titanate ($BaTiO_3$) is a ceramic material renowned for its distinctive physical and chemical properties, making it highly suitable for various electronic applications. Below is a summary of its key quantitative properties (Ertuğ, B. 2013, Kinoshita, K., et al. 1976):

Table 1: Physical and Chemical Properties data of Barium Titanate

Property	Value
Molar Mass	233.193 g/mol
Density	6.00 g/cm³
Melting Point	1626°C
Dielectric Constant	Up to 1700 at 1 kHz
Hardness	5
Young's Modulus	67 GPa
Refractive Index	2.4
Band Gap	3.2 eV
Curie Temperature	Approximately 120°C
Spontaneous Polarization	0.15–0.26 C/m² at room temperature
Piezoelectric Coefficient (d_{33})	>190 pC/N (pure $BaTiO_3$)
Electromechanical Coupling Factor (k_{33})	0.49

These properties render barium titanate ideal for use in ceramic capacitors, piezoelectric transducers, and a multitude of other electronic devices (Uchino, K. 2000).

3.3. Impact on Electrical and Piezoelectric Properties

Manufacturing technology greatly affects the dielectric and piezoelectric properties of BaTiO3 (see Table 2). Successful optimization of these parameters can only be achieved if a high degree of crystallinity and uniform grain size are obtained. For example, Haertling, G.H. 1999 sol-gel derived BaTiO3 has superior dielectric features due to its uniform nanostructure. Likewise, Suzuki, K., et al. 2005 BaTiO3 made by hydrothermal method has better piezoelectric performance because it forms a tetragonal phase. Nanoceramics are prepared using a solid-state reaction technique where appropriate materials are combined before being calcined under extreme temperatures. This method was found in practice to always give high-quality $BaTiO_3$ in its crystalline form while the dimensions were also correct. Waugh, M.D. 2010 In addition, this method requires a longer time than usual and very high manufacturing temperatures, leading to increased energy expenditures and potential pollution, which is why it remains the least preferred among the current alternatives. These impurities have an impact on the electrical properties of $BaTiO_3$, causing nanoceramics made with alternative methods to have lower dielectric constants and piezoelectric coefficients **(Seong Hyeok Choi, et al. 2023,** Xu, Y. 1991, **Monika Singh, et al. 2017)**. The sol-gel method, which involves converting a solution system from a sol phase to a gel phase, is a versatile, low-temperature method for manufacturing $BaTiO_3$. This technique allows for excellent control over the chemical stoichiometry and homogeneity of the final product. $BaTiO_3$ synthesized using sol-gel has high purity and small, homogenous grain sizes, which improves its dielectric and piezoelectric properties. Minimizing processing temperatures can also help to save energy and avoid undesirable phase formations. The intricacy of the sol-gel process and its sensitivity to processing conditions may influence reproducibility (V. Sherlin Vinita, et al. 2020, J. Thomas Karpagalakshmi, et al. 2012). To produce crystalline $BaTiO_3$, hydrothermal synthesis involves reacting precursor components in an aqueous solution at high temperatures and pressures. This process has been shown to create $BaTiO_3$ with tiny, well-defined grain sizes and excellent crystallinity. Because of its perfect microstructure and great purity, the resultant BT typically has excellent dielectric and piezoelectric characteristics. Furthermore, the hydrothermal approach allows for lower synthesis temperatures than solid-state procedures, conserving the material's inherent properties. Nonetheless, the requirement for particular high-pressure apparatus may be a restriction (Abd Elmoneim M. S. Elbasset, et al. 2019, Hiroyuki Takenaka, et al. 2012, Tsutomu Homma, et al. 2009). It is a process of intense grinding that mechanochemical synthesis. The generated mechanical energy of this method allows for the production of $BaTiO_3$ powders with nano-sized grains and high reactivity. Because of its finer grain structure and larger surface area, BT produced by mechanochemical processes frequently exhibits enhanced piezoelectric qualities. This procedure may be carried out at room temperature and is also energy-efficient. On the other hand, excessive mechanical stress can result in imperfections and contaminants that deteriorate electrical qualities. (Steven J. Kitchen, et al. 2015, A. Zuo, et al. 2020). By using Microwave-assisted synthesis to quickly and evenly heat the reaction mixture, microwave-assisted synthesis produces BT. This approach has the advantages of quick processing speeds and low energy use. The great purity and compact, homogeneous grains of $BaTiO_3$ generated by microwave-assisted synthesis are responsible for its exceptional dielectric and piezoelectric qualities. Furthermore, phase purity and improved reaction kinetics can be achieved by microwave heating. Nevertheless, the initial investment in microwave equipment can be substantial, and the technique necessitates meticulous regulation of reaction conditions to prevent uneven heating. (Dongxu Zhang, et al., 2011, Xiaoqing Guo, et al., 2011, Yanping Chen, et al., 2012).

Table 2: illustrates the effect of each manufacturing process on the morphology, characteristics, and properties of barium titanate

Synthesis Method	Effect on Structure	Effect on Morphology	Electrical Properties	Piezoelectric Properties	References
Solid-State Reaction	Formation of traditional cubic or tetragonal crystal structure	Large particle size, non-uniform distribution	High dielectric constants but lower compared to other methods	Lower piezoelectric constant compared to other methods	(Zhang, S., et al., 2004), (Xu, Y. 1991)
Sol-Gel Method	Pure nanocrystals with high uniformity	Homogeneous nanoparticles, uniform distribution	Higher dielectric constants due to uniform particle distribution	Higher piezoelectric constant due to nanostructure	(Chieng, B. W., et al., 2012), (Jalil, A. A., et al., 2009)
Hydrothermal Synthesis	High-purity crystals with good structural control	Homogeneous nanoparticles, less aggregation	High dielectric constants with improved conductivity	High piezoelectric constant due to crystal purity	(Hwang, Y. H., et al., 2000), (Kakihana, M. 1996)
Mechanochemical Synthesis	Fine crystals with rapid reaction	Small particles with homogeneous size, irregular shape	Moderate dielectric constants, some crystal defects	Good piezoelectric constant but lower than hydrothermal synthesis	(Boldyrev, V. V. 2006), (Baláž, P. 2003)
Microwave-Assisted Synthesis	Pure crystals with rapid and efficient reaction	Nanoparticles with good distribution, less aggregation	High dielectric constants with improved conductivity	High piezoelectric constant due to rapid reaction and crystal purity	(Roy, R., et al.,1999), (Oghbaei, M., et al., 2010)

4. CONCLUSION AND FUTURE WORK

This review highlights the influence of various synthesis methods on the structural, dielectric, and piezoelectric properties of Barium Titanate. Each method offers distinct advantages and has specific impacts on the microstructure and properties of $BaTiO_3$. The solid-state reaction and sol-gel methods are particularly effective in producing high-crystallinity $BaTiO_3$ with excellent dielectric properties. Hydrothermal and microwave-assisted synthesis methods are promising for achieving fine nanostructures and enhanced piezoelectric properties. Future research should focus on optimizing these synthesis methods to fully harness the potential of $BaTiO_3$ in advanced electronic and technological applications.

The preparation of barium titanite ($BaTiO_3$) is considered a vital field in materials science. For further innovation, the following future technologies can be regarded as that may revolutionize the preparation of barium titanite:

1. 3D printing manufacturing: 3D printing technology can change the rules of barium titanite preparation. By creating advanced ink materials that form the core of these materials (Ba and Ti) create highly complex nanostructures by improving printing resolution.
2. Processing using high pressure and temperature: The means employing responses using high temperatures and pressures can improve the crystallinity and density of the resulting materials which in return optimizes the thermal stability and electrical characteristics of barium titanate.
3. Plasma-assisted reaction: The utilize of plasma in the preparation reaction can assistance reduce the reaction temperature and increase the reaction speed. This method may produce barium titanate with developed purity and improved governor of crystal formation.
4. Ultrasound-facilitated synthesis of chemicals: The ultrasonic method supports the chemical reaction and accelerates molecules formation in the ultrasonically aided synthesis. The application of this method in the production of barium titanate can help improve the nanoscale uniformity of the particle distribution and enhance the quality of the resulting material.
5. Atomic layer deposition (ALD) chemical technique:

The use of atomic layer chemical deposition technology allows precise control of the thickness and composition of the deposited layers. This technique can be used to produce nanolayers of barium titanite with a homogeneous composition and enhanced properties.

5. BIO-PREPARATION:

Biopreparation methods involving bacteria or microorganisms can form nanostructures of barium titanite. This method may be more environmentally friendly and reduce the need for harmful chemicals.

These future technologies could revolutionize the preparation of barium titanite and enable the production of materials with improved properties and broad applications in electronics, renewable energy, and nanotechnology.

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