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Comparative Study of Sol-Gel and Hydrothermal Synthesis Methods for Bismuth Ferrite Nanoparticles

Unni kisan[1], Birendra Kumar[2], Syed Asghar Husain Rizvi[3]

[1,2,3]*Department of Mechanical Engineering, Khwaja Moinuddin Chishti Language University, Lucknow*
Corresponding author's mail: krishnanswami1996@gmail.com

Abstract **- Bismuth ferrite ($BiFeO_3$, BFO), a multiferroic perovskite type material, has attractive optical, magnetic, and photocatalytic characteristics. However, phase purity, morphology, crystallite size, defect structure, and functional performance are all significantly impacted by the synthesis process. The sol gel method and the hydrothermal approach, two popular wet chemical techniques, are compared in this work with regard to BFO nanoparticles. We compare crystallinity, particle size/morphology, impurity phase formation, process parameters, and functional results (e.g., photocatalysis, magnetic/ferroelectric behavior). The main findings are sol gel often yields finer, more uniform particles at lower calcination temperatures but may require higher post annealing to remove residual organics and to obtain phase purity, hydrothermal synthesis provides good crystallinity and morphological control (e.g., rods, hollow spheres) under milder temperatures but is more sensitive to reaction time/pressure and may introduce secondary phases. The study also points out shortcomings and makes suggestions for improving the production of BFO nanoparticles.**

Keywords **– bismuth ferrite, sol-gel, hydrothermal, nanoparticles, synthesis.**

1. Introduction

Hydrothermal synthesis offers good crystallinity and morphological control (e.g., rods, hollow spheres) at milder temperatures but is more sensitive to reaction time/pressure and may introduce secondary phases [1][3]. Sol gel frequently produces finer, more uniform particles at lower calcination temperatures but may require higher post annealing to remove residual organics and obtain phase purity. At ambient temperature, bismuth ferrite ($BiFeO_3$, BFO) is a single phase multiferroic material that displays both magnetism and ferroelectricity [4].

These characteristics make it appealing for use in sensors, multiferroic devices, photocatalysis, and spintronics. Nevertheless, the volatile nature of Bi^{3+}, the limited window for phase formation, and the propensity to produce secondary phases like $Bi_2Fe_4O_4$ or Bi_2O_3 make the synthesis of pure BFO difficult [5]. As a result, the structural, morphological, and functional characteristics of BFO nanoparticles are significantly influenced by the synthesis technique selected. The hydrothermal method (reaction in a sealed autoclave under high pressure and temperature) and the sol gel method (an oxide precursor pathway, frequently followed by calcination) are two of the most used synthesis techniques. Regarding control over size, morphology, crystallinity, and scalability, each approach has benefits and drawbacks.

In this review, we compare hydrothermal and sol gel procedures specially producing BFO nanoparticles. Our goals are to: (i) explain the underlying mechanisms of each synthesis route; (ii) compare the parameters of the process and the characteristics of the resulting nanoparticles (phase purity, size, shape, and surface features); (iii) examine the ways in which these characteristics affect functional performance; and (iv) summarize research gaps and optimization strategies.

2. Theoretical Background and Synthesis Mechanisms

2.1 Sol-Gel Method

Metal alkoxide or nitrate precursors are hydrolyzed and condensed in the sol gel process to create a colloidal "sol" and ultimately a gel network that, when dried and calcined, produces oxide nanoparticles [6]. $Bi(NO_3)_3 \cdot 5H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ are common precursors for BFO that are combined in a solvent (often ethanol/water) with a chelating agent (ethylene glycol, citric acid) to create a polymeric gel. BFO crystalline phase develops after drying and calcination (e.g., at 450–600 °C) [7]. The benefits include tiny particle sizes, simplicity of doping, and good chemical uniformity at the molecular level [8]. However, eliminating organic residues and managing stoichiometry—particularly Bi losses are difficult.

2.2 Hydrothermal Method

In the process of hydrothermal synthesis, precursor solutions are heated under pressure to 150–240°C in an autoclave lined with Teflon. The BFO phase frequently requires either no additional calcination or a moderate one since it nucleates and develops directly in the aqueous media [9]. Reaction time/temperature, pH, and mineraliser (KOH, NaOH) concentration can all be used to adjust morphology (rods, hollow spheres, sheets) [10]. Better crystallinity and lower phase formation temperatures are provided by the approach, but if the process is not tuned, it may produce bigger particles, necessitate lengthy reaction periods, and be prone to secondary phases [1].

2.3 Key Issues for BFO Synthesis

Achieving phase purity (avoiding $Bi_2Fe_4O_3$, Bi_2O_3), managing Bi volatilization, reducing particle agglomeration, regulating size and morphology to increase surface area (important for photocatalysis), and maintaining multiferroic properties are all crucial synthesis concerns for BFO nanoparticles [5,11].

3. Comparative Analysis of Sol-Gel and Hydrothermal Methods

3.1 Phase Purity and Crystallinity

According to a number of studies, BFO powders generated via sol gel often have more uniform particle distribution and smaller crystallite sizes than hydrothermal ones. For example, a comparison study revealed that BFO generated from sol gel had a more homogeneous and finer particle size than hydrothermal (average grain size sol gel < hydrothermal) [1]. Another study found that single phase BFO can only be produced by hydrothermal synthesis under extremely tight temperature/time conditions; otherwise, $Bi_2Fe_4O_4$ occurs [12].

3.2 Particle Size and Morphology

Because of the molecular mixing of precursors, the sol gel method usually produces nanoparticles in the 20–50 nm range (depending on calcination) [3]. On the other hand, depending on reaction conditions, hydrothermal methods may result in bigger particles (such as 40–100 nm or rod/plate/hollow sphere morphologies) [1,13]. For instance, Kahramanmaras et al. found that the average crystallite size for BFO photocatalysts was around 39 nm for sol gel and approximately 46 nm for hydrothermal [14].

3.3 Surface Area, Porosity and Defects

Smaller particles made using the sol gel approach frequently have more active sites and a larger surface area, which is advantageous for photocatalysis. Defects, however, might impair performance if there are leftover organics or partial crystallization. Because of its controlled growing environment, the hydrothermal technique frequently produces higher crystallinity, but unless templated or hollow structures are created, surface area may be sacrificed [15].

3.4 Functional Performance – Photocatalysis, Magnetic/Ferroelectric Behavior

Studies of functional properties show that the synthesis technique influences results. greater surface area, finer particles (often sol gel) exhibit greater dye degradation rates in photocatalysis [14]. Hydrothermal synthesis with controlled development frequently yields fewer defects, resulting in improved multiferroic coupling in terms of ferroelectric/magnetic coupling [12]. However, morphology, doping, and annealing may have a significant impact on this.

3.5 Advantages & Limitations of Each Method

	Advantages	Limitations	Ref
Sol-gel	• cost-effective, • good size control, • molecular homogeneity, • lower temperature calcination possible	• requires high calcination to achieve phase purity, • risk of Bi volatile loss, • organics removal, • agglomeration.	**[6,8]**
Hydrothermal	• mild synthesis temperature, • unique morphologies (rods, hollow spheres), • good crystallinity	• longer reaction times, • autoclave equipment needed, • morphology often larger size unless templated, • sensitive to reaction conditions.	**[9,10]**

3.6 Comparative Summary Table

Method	Typical Temperature/ Process	Particle Size	Morphology Control	Phase Purity/Crystallinity	Common Application
Sol-gel	~450-600 °C post-calcination	~20-50 nm	Spherical, fine particles	Good if calcined well	Photocatalysis, sensors
Hydrothermal	~150-240 °C in autoclave	~40-100 nm or rods/spheres	Excellent shape control	Good if optimized	Multiferroics, hollow-sphere catalysts

4. Optimization Strategies and Research Gaps

There are still a number of holes in spite of several reports. Doping (e.g., Nd, La) to stabilize phase and enhance functionality [16], combining hydrothermal with templating to reduce size and increase surface area [17], using microwave assisted hydrothermal to shorten time, and using chelating agents/surfactants in sol gel to prevent agglomeration are examples of optimization strategies. Important research needs include: (i) a comprehensive side-by-side comparison of hydrothermal and sol gel for identical precursor sets in BFO; (ii) repeatability and scale-up concerns; (iii) the connection between morphology and multiferroic coupling in nanoparticles; and (iv) long-term durability of functional performance.

5. Conclusions

Both sol gel and hydrothermal techniques offer unique advantages for producing BFO nanoparticles, as this comparative research demonstrates. The sol gel technique is appealing because it produces larger surface areas and finer particle sizes, which are advantageous for sensors and photocatalysis. Although the hydrothermal process is excellent for controlling morphology and processing at lower temperatures, it may produce bigger particles and need tight adjustment to prevent impurity phases. The goal functionality (e.g., large surface area vs. flawless crystallinity) must be taken into account when choosing a synthesis for application-driven design. Future research should focus on scale-up, hybrid approaches that combine the best aspects of both methodologies, and comparative standardized studies.

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