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QUICK AND EASY SYNTHESIS OF $BIFEO_3$ PEROVSKITE BY DIRECT COMBUSTION WITH POTENTIAL FOR PHOTOVOLTAIC APPLICATION

SÍNTESE RÁPIDA E FÁCIL DA PEROVSKITA $BiFeO_3$ POR COMBUSTÃO DIRETA COM POTENCIAL PARA APLICAÇÃO FOTOVOLTAICA

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ABSTRACT

Materials with perovskite-type structures composed of lead halides have proven to be highly promising in the field of photovoltaic cells, exhibiting high efficiency in converting solar energy into electricity. However, several studies have pointed to the need to replace lead with a non-toxic metal that maintains equivalent performance efficiency. Therefore, bismuth ferrite-based perovskites, $BiFeO_3$, have attracted the interest of the scientific community for several reasons. The objective of this study was to investigate the efficiency of the combustion reaction synthesis method in the production of $BiFeO_3$ material without the need for calcination treatment after synthesis. Thus, two $BiFeO_3$ samples were synthesized at two different reaction temperatures, namely, 200 and 500°C. The two synthesized samples were characterized by X-ray diffraction (XRD), which confirmed the formation of the $BiFeO_3$ phase; scanning electron microscopy (SEM), which assisted in determining particle size and morphological analysis; and Fourier transform infrared spectroscopy (FTIR), which demonstrated a low concentration of residual organic compounds impregnated in the samples. Furthermore, it was concluded that the direct combustion synthesis method, carried out at a reaction temperature of 500°C without the need for post-synthesis calcination, was effective for the preparation of a $BiFeO_3$ perovskite-structured material consisting of a crystalline microstructure predominantly formed by the desired phase.
Keywords: $BiFeO_3$; combustion synthesis; Perovskite, photovoltaic material.

RESUMO

Materiais com estruturas do tipo perovskitas compostas por haletos de chumbo têm se revelado materiais altamente promissores no cenário das células fotovoltaicas, apresentando elevada eficiência na conversão de energia solar em elétrica. Contudo, diversos estudos têm apontado para a necessidade da substituição do chumbo por um metal atóxico que mantenha a eficiência equivalente de desempenho. Com isso, as perovskitas à base de ferrita de bismuto, $BiFeO_3$, têm despertado o interesse da comunidade científica por vários motivos. O objetivo deste estudo foi investigar a eficiência da temperatura de pré -aquecimento do forno no método de síntese da reação de combustão na produção do material $BiFeO_3$ e sem a necessidade de realização de tratamento de calcinação após a síntese. Assim, foram sintetizadas duas amostras de $BiFeO_3$ sob dois valores distintos de temperatura reacional, a saber: 200 e 500°C. As duas amostras sintetizadas foram caracterizadas por DRX, o qual comprovou a formação da fase $BiFeO_3$, a MEV auxiliou para determinação do tamanho de partículas bem como a análise morfológica e espectroscopia FTIR que provou a baixa concentração de compostos orgânicos residuais impregnados nas amostras. Para além disto, ficou concluído que o método de síntese da combustão direta, realizado sob temperatura reacional de 500°C, sem a necessidade de calcinação posterior à síntese, foi eficiente para a preparação do material com estrutura perovskita $BiFeO_3$ constituído por microestrutura cristalina majoritariamente formada pela fase desejada.
Palavras-chave: $BiFeO_3$; material fotovoltaico; Perovskita; Síntese por combustão.

1 INTRODUCTION

The photovoltaic energy generation effect began in 1839, when French physicist Edmond Becquerel observed that immersing two brass plates in a liquid solution containing electrolytes generated electricity when the system was exposed to sunlight. This phenomenon became known as the photovoltaic effect (MACHADO and MIRANDA, 2015).

Among the advantages of photovoltaic energy is the fact that it does not emit gases such as carbon dioxide and sulfur dioxide, as well as others that contribute to the greenhouse effect and are

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harmful to human health. This type of energy generation stands out when compared to other renewable sources because it is easy to install, has low maintenance costs, does not emit noise, and can reach remote regions where the traditional energy distribution network may claim difficulty in accessing (de Oliveira, 2023).

According to Verza, 2020, due to the different construction and operating technologies of solar cells, the historical evolution of this type of electricity generation system is divided into three generations, namely: the first generation of cells, based on mono- and polycrystalline silicon, was based on the p-n junction. The second generation, based on amorphous silicon or CdTe and CuInGaSe compounds, was classified as commercial thin-film cells. The current or third generation has its architecture based on emerging thin films, organic materials, quantum dots, perovskite-type structures, among others.

Materials with perovskite-type structures have emerged as a promising photovoltaic technology (Casarin, 2024). Lead halide-based perovskites are the most widely used type of perovskite cell. However, lead is a toxic metal and can cause damage to human health and impact the environment if disposed of incorrectly (Briquezi, 2023).

The major challenge for this type of cell, for its large-scale adoption, has been combating the degradation of its microstructure, as it becomes vulnerable to this undesirable and irreversible phenomenon when in contact with moisture and high temperatures (Casarin, 2024).

Still in this scenario of third-generation solar cells, the $BiFeO_3$ material, with a perovskite structure, has emerged as a classic multiferroic material option with strong photoinduced and ferro-polarized coupling, offering a promising approach for the creation of reliable and effective photovoltaic systems in the future. Thin films based on $BiFeO_3$ are considered ideal for exploring the various properties of this material in emerging technologies (Sun et al., 2025; Calzada et al., 2025). For photovoltaic applications, bismuth ferrite typically exhibits a bandgap energy of approximately 2.7 eV; however, this value can range between ~2.1 and 2.8 eV for the pure material and decrease significantly to around ~1.3 to 2.4 eV upon doping (Wang et al., 2024).

In line with this context, the objective of this work was to investigate the efficiency of the direct combustion reaction synthesis method in the preparation of $BiFeO_3$ material, in powder form, without performing calcination treatment after obtaining the synthesis product, under relatively mild reaction

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temperature values. Part of the objectives of this work is to characterize the microstructure of the synthesized samples of $BiFeO_3$ material, which has recognized photovoltaic properties.

2 MATERIALS AND METHODS

The methodology employed in this work is an adaptation of the work by ILIĆ et al. (2022), which successfully produced $BiFeO_3$ material in powder form with improved magnetic properties using the self-combustion synthesis technique. This methodology was divided into stages, as described below:

2.1 Stoichiometry of the reaction process

Initially, the reaction equation that underpins and supports the combustion reaction synthesis process carried out in this work was established, aimed at preparing $BiFeO_3$ material in powder form. In accordance with the principles of combustion reaction, the reaction equation included oxidizing and reducing agents. Thus, as shown in the properly balanced reaction scheme in Equation 1, the oxidizing agents are bismuth III nitrates – $Bi(NO_3)_3$, 98% purity, and iron III nitrate – $Fe(NO_3)_3$, 99% purity; and the reducing agent is urea – CH_4N_2O.

Equation 1:

$$Bi(NO_3)_3 + Fe(NO_3)_3 + 5\,CH_4N_2O \rightarrow BiFeO_3 + 10H_2O + 8N_2 + 5CO_2$$

2.2 Synthesis by combustion reaction

The combustion synthesis method is widely employed in powder production due to its practicality and speed. An exothermic reaction between the precursors (metal nitrates and organic fuels) yields a powder with a high degree of crystallization at low cost and without the need for subsequent calcination (Mazouzi, 2024).

As already mentioned, this work focused on the preparation of $BiFeO_3$ material in powder form using the combustion reaction synthesis method. To this end, the reagents shown in the reaction scheme in Equation 1 were mixed together in a silica glass crucible according to stoichiometric proportions. Subsequently, the mixture was placed in a muffle furnace preheated to a temperature sufficient to promote the dissolution of the reagents, dehydration followed by

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evaporation of volatile substances released from the reaction, and ultimately, to trigger spontaneous ignition of the mixture, a phenomenon characteristic of the combustion reaction.

As one of the objectives of this work is to investigate the influence of the reaction process temperature on the microstructural characteristics of the synthesized material, this experimental synthesis procedure was carried out at two different temperatures: 200 and 500°C.

After the ignition flame extinguished, the vessel remained inside the muffle furnace for 10 minutes to eliminate any remaining volatiles. The crucible was then removed from the furnace and left to cool spontaneously in natural air until it reached room temperature. The combustion reaction synthesis procedure, at a reaction temperature of 200°C, lasted 8 minutes from the moment the crucible was placed in the furnace until the ignition flame ceased, with the ignition flame persisting 1 second. The combustion reaction synthesis procedure, at a reaction temperature of 500°C, lasted 5.5 minutes under the same evaluation criteria, with the flame persisting for 4 seconds.

The powders resulting from the reactions at 200°C and 500°C were manually deagglomerated in an agate mortar measuring 10 cm in diameter and then sieved through an ABNT 200 mesh screen with an opening of 74 μm in order to obtain a powdered material with a fine enough particle size to allow maximum interaction with the pre-established characterization techniques to explore the microstructural details of these materials. The two samples synthesized in this work were not subjected to calcination treatment after direct synthesis by combustion reaction.

2.3 Characterizations

The $BiFeO_3$ powder samples products of combustion reaction synthesis, carried out at different temperatures, as described above, were subjected to the following characterization techniques: X-ray diffraction (XRD); scanning electron microscopy (SEM); determination of average particle size; and Fourier transform infrared spectroscopy (FTIR). Brief descriptions are provided below.

The two synthesized samples were then subjected to XRD to identify the constituent phases of their microstructures, as well as details related to their degrees of crystallinity. Thus, the two samples were analyzed in a Malvern Panalytical Empyrean diffractometer, which is equipped with the following parameters: copper target with λ = 1.54 μm; voltage of 40 kV and current of 30 mA; the angle range (2θ) varying between 5 and 80° with a step of 0.02°s^-1 was used. This diffractometer

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belongs to the Materials Characterization Laboratory of the Federal University of Cariri. The diffraction peaks collected were identified using HighScore Plus software by comparing the angles of occurrence of the peaks in the ICDD/PDF-2 data library.

The morphological aspects of the constituent unit particles, as well as their agglomerates, of the two samples synthesized by combustion reaction in this work were recorded using the SEM technique. This characterization was performed using a TESCAN vegan 3 scanning electron microscope, operated at 20KV, belonging to the Materials Characterization Laboratory (LCM) of the Science and Technology Center of the Federal University of Cariri. To perform this microscopic analysis, the powdered sample was deposited on double-sided carbon tape previously placed on the equipment's sample holder.

From the images recorded using the SEM technique, relating to the morphology of the particles constituting the samples synthesized in this work, the average size of these particles was determined using ImageJ software.

The investigation of the presence of organic or molecular compounds impregnated in the samples of the inorganic material $BiFeO_3$ synthesized in this work was performed using FTIR spectroscopy. For this purpose, an Agilent Cary 630 (ATR-FTIR) spectrometer was used in the mid-infrared region 4000-650 cm^-1, belonging to the Chemistry Laboratory of the Federal Institute of Ceará – IFCE, Juazeiro do Norte Campus. The data were obtained using Agilent Microlab and Resolution Pro software.

3 RESULTS AND DISCUSSION

3.1 X-ray diffraction (XRD)

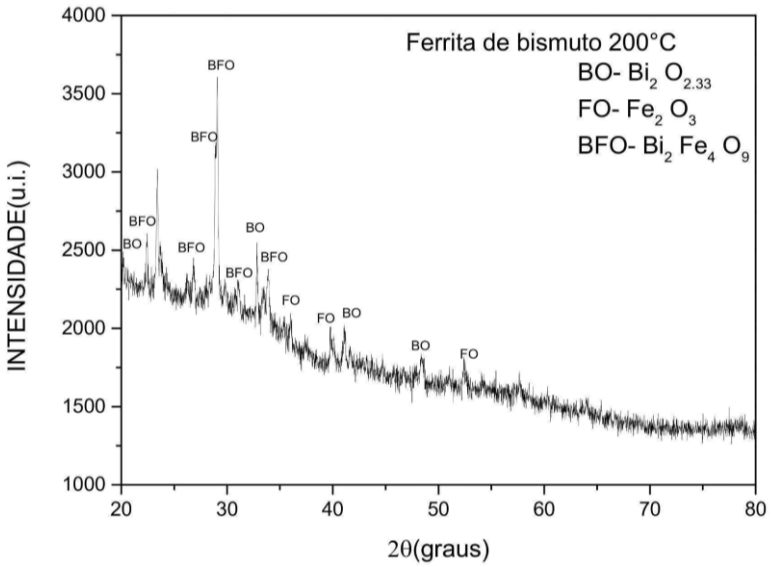
Figures 1 and 2 show the results of microstructural characterization, collected using the XRD technique, for the two samples of $BiFeO_3$ material synthesized in this work by combustion reaction, under two different reaction temperatures, namely: 200 and 500°C.

According to Benini, 2025, the X-ray diffraction (XRD) technique is a great ally for structural analysis, as it provides details about the nature and some parameters of the material's crystal lattice. Each chemical sample has a unique diffraction pattern, like a fingerprint of the material, which can be used for identification by comparison with a database (YAHIA, 2025).

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Figure 1 – Graphical profile representing the X-ray diffraction pattern of the $BiFeO_3$ material sample synthesized by combustion reaction at 200°C



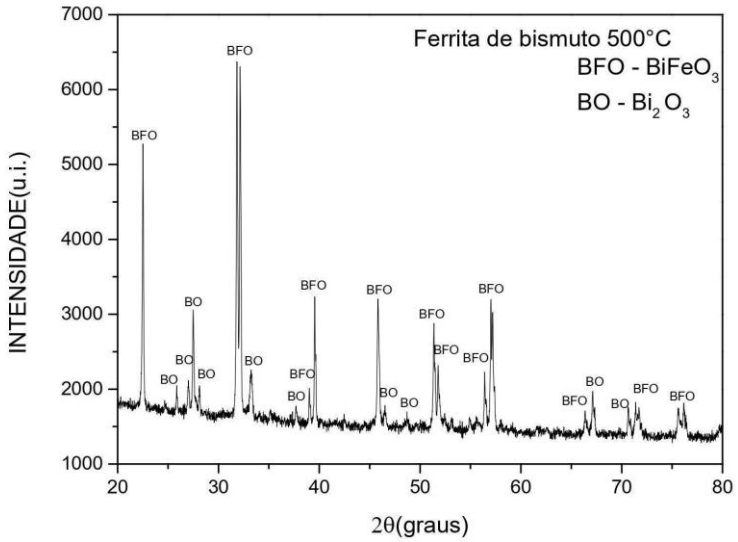
Source: The authors, 2025

With the identification of the phases present in the $BiFeO_3$ sample synthesized at 200°C via X-ray diffraction (Figure 1), it is possible to observe that the reaction at 200°C did not proceed completely, resulting in a higher amount of amorphous content and segregated secondary phases such as $Bi_2 O_{2,3}$, $Bi_2 Fe_4 O_9$, and $Fe_2 O_3$. These features can be associated with the low temperature employed, which is thermodynamically or kinetically insufficient for the full transformation of the precursors. From another perspective, the non-formation of the desired phase can be attributed not only to the low processing temperature applied in the synthesis of this sample, but also to the absence of a calcination treatment following direct synthesis by combustion reaction. In the study conducted by Lomova et al. (2025), very similar findings were achieved, which aided in the understanding of the present work.

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Figure 2 - Graphical profile representing the X-ray diffraction pattern of the $BiFeO_3$ material sample synthesized by combustion reaction at 500°C



Source: The authors, 2025

When analyzing the representative graphic profile of the diffraction data for the $BiFeO_3$ sample synthesized at a reaction temperature of 500°C, shown in Figure 2, it was found that the microstructure of this sample is significantly crystalline and consists mainly of the desired phase, $BiFeO_3$; associated with the presence of a single type of segregated phase, Bi_2O_3. When quantifying the mass fractions of these two phases detected in the HighScore Plus software, the percentage values of 66% for the majority phase, identified in the graph as BFO, and 34% for the secondary phase, identified as BO, were revealed.

In a study conducted by Zarur, 2020, it is stated that $BiFeO_3$ material is susceptible to the presence of secondary phases that tend to nucleate on grain boundaries and impurities. This is because bismuth ferrite is metastable in the atmosphere, and the main phase is impaired due to the evaporation of the Bi_2O_3 phase.

Considering that in this work calcination treatment was not performed after synthesis by combustion reaction, it is noted that direct synthesis by combustion at a reaction temperature of 500°C proved to be efficient for the formation of the desired phase, $BiFeO_3$, although this reaction temperature proved to be insufficient for the formation of a single-phase microstructure. It is worth

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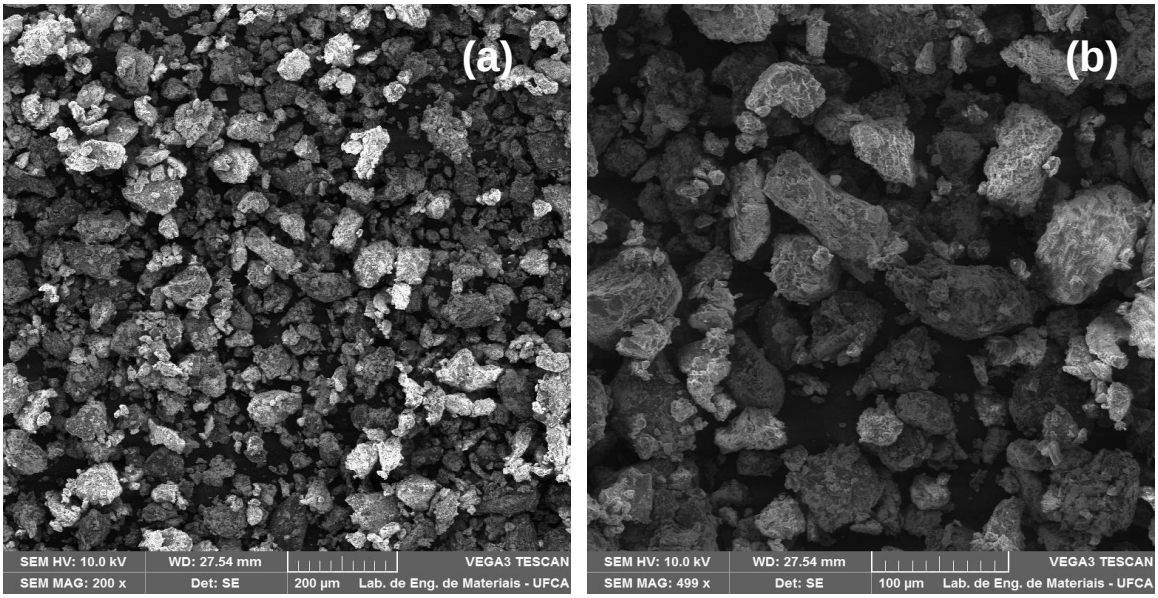
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noting that the significant percentage of the desired phase in this sample synthesized at 500°C, in direct synthesis without subsequent calcination, leads to the conclusion that obtaining a fully single-phase $BiFeO_3$ based microstructure through combustion synthesis without the need for subsequent calcination treatment is entirely possible, most likely at reaction temperatures above 500°C.

3.2 Scanning Electron Microscopy (SEM)

The images obtained by scanning electron microscopy (SEM) were captured at magnifications of 200x and 500x for each of the two samples synthesized in this work. The SEM micrographs illustrated in Figure 3 refer to the sample synthesized at a reaction temperature of 200°C.

Figure 3 - SEM micrographs of the sample synthesized by combustion reaction at 200°C with magnification of: (a) 200x and (b) 500x, respectively



Source: The authors, 2025

Analyzing the morphological aspects of the powdered sample particles synthesized at a reaction temperature of 200°C, the typical physical characteristics of unitary particles synthesized by the combustion method are evident, i.e., light and thin appearance with a leaf-like shape and an undeniable tendency to agglomerate into a visibly porous state. The strong tendency of the unit particles to agglomerate can be explained both by the ionic nature of this type of material, which is

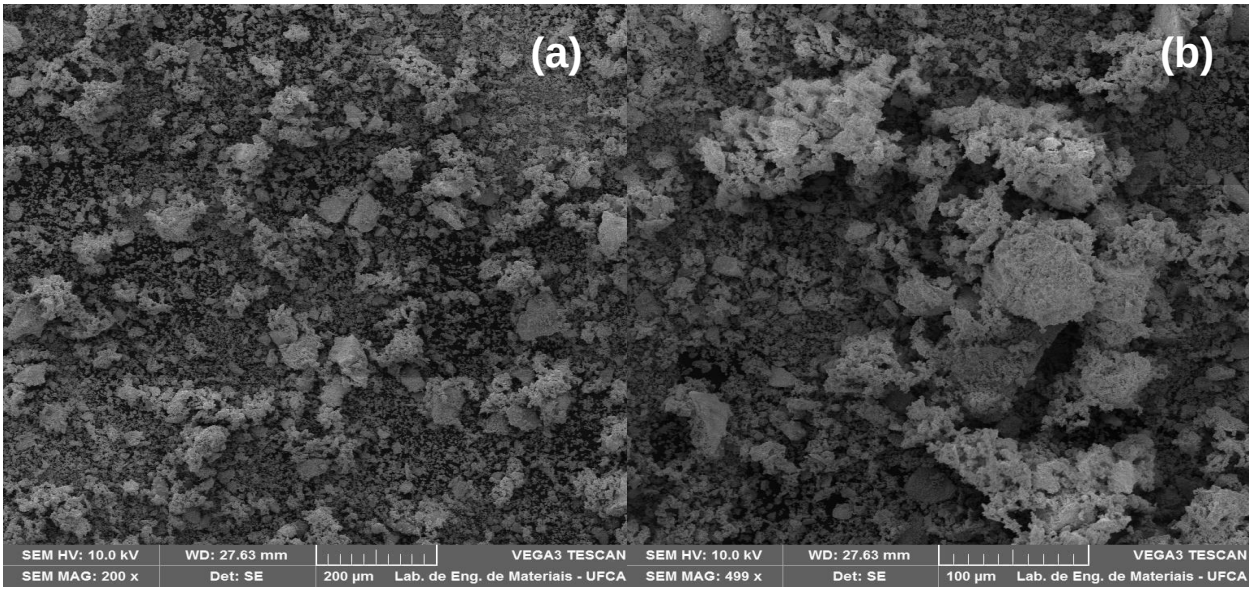
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inevitably naturally susceptible to electrostatic attraction, and by the presence of fuel, in this case urea, which can commonly leave residual residues after the ignition of the combustion process and, consequently, acts as a binding agent. Considering that in this work, direct combustion synthesis was practiced, without subsequent calcination, this assumption of the presence of binding residues gains strength. These morphological aspects characteristic of powders synthesized by combustion reaction, observed in this work, are corroborated by the results obtained by Tomina et al. (2024).

The SEM micrographs shown in Figure 4 refer to the sample synthesized at a reaction temperature of 500°C.

Figure 4 - SEM micrographs of the sample synthesized by combustion reaction at 500°C with magnification of: (a) 200x and (b) 500x, respectively



Source: The authors, 2025

The morphological aspects of the powder sample synthesized at 500°C were very similar to those of the sample synthesized at 200°C, that is, fine, light morphology, with a leaf-like appearance and prone to agglomeration. The only visible differences in a comparative analysis are in relation to the average size of the particles and, consequently, their agglomerates. The physical aspects of the particles constituting the sample synthesized at 500°C were visibly smaller, thinner, and in a more discreet state of agglomeration. This morphological or textural aspect exhibited by the sample

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synthesized at 500°C is most likely a consequence of the influence exerted by the higher reaction temperature under which this sample was synthesized, which certainly provided sufficient thermal energy to promote greater release of organic residues from the fuel used in this synthesis, resulting in the preparation of a sample containing a lower concentration of impregnated residual compounds and/or remaining binders.

Based on the images recorded in the micrographs shown in Figures 3 and 4, the average size of the constituent particles of the two samples synthesized in this work was determined using ImageJ software. Table 1 shows the estimated average sizes of the unit particles for the two samples synthesized by combustion reaction in this work.

Table 1: Average particle size of $BiFeO_3$-based samples synthesized by combustion reaction in this work.

Sample	Average Particle Size
Synthesized at 200°C	0.015 µm
Synthesized at 500°C	0.005 µm

Source: The authors, 2025

According to the data obtained and presented in Table 1, it is evident that the sample synthesized at a temperature of 200°C exhibits a larger particle size; this may be associated with the incomplete decomposition of the precursors used, which resulted in an adsorbed layer of organic volatiles and unreacted species around the inorganic cores.

In contrast, for the sample synthesized at a temperature of 500°C, there was a decrease in the average agglomerate size; this occurs due to the higher thermal input provided, which facilitates nucleation and the release of volatiles. As this release of volatile products during combustion inhibits the coalescence of primary particles and eliminates organic contaminants, it results in finer nanoparticles.

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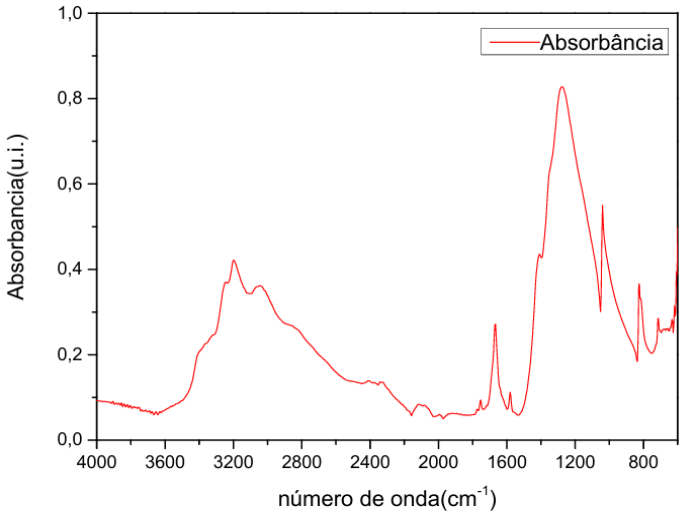
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3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The results of the characterization performed by Fourier transform infrared spectroscopy (FTIR) for the two samples synthesized in this work are shown in Figures 5 and 6, respectively, for the samples synthesized at reaction temperatures of 200 and 500°C.

To identify the absorbance peaks, data from the LibreTexts Infrared Spectroscopy Absorption Table, supported by the California State University Accessible Learning Solutions Program and Merlot (ISAT, 2020), was used.

Figure 5 - Result of FTIR analysis of the sample synthesized at 200°C



Source: The authors, 2025

The FTIR spectrum of the sample synthesized at 200°C displays distinct vibrational bands associated with organic species and surface hydroxyls, confirming incomplete thermal precursor conversion. According to previous studies, it is scientific knowledge that the broad absorption peaks between 3198 and 3042 cm^{-1} correspond to O–H stretching group vibrations, which may have originated from adsorbed atmospheric moisture and residual hydroxyl group species (Baek et al., 2024).

This made it possible to identify peaks at 2333 cm-1 and 2114 cm-1, which may be related to nitrile (C≡N) or alkene (C≡C) bonds. The range where the peaks of 1277 cm-1 and 1039 cm-1

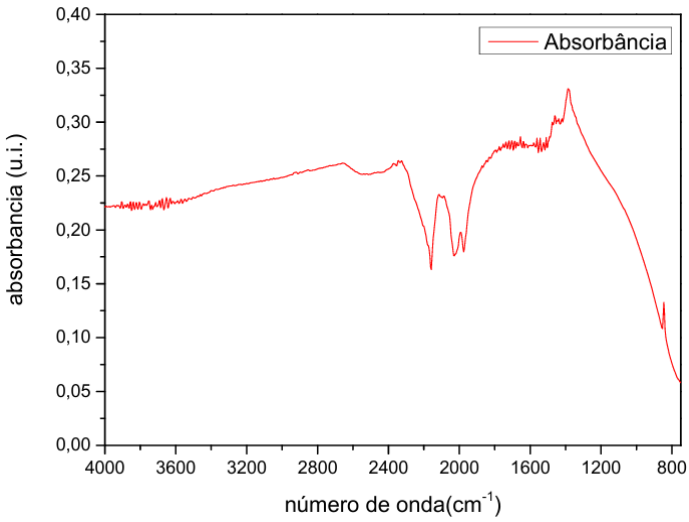
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occur characterizes the presence of CO-type bonds. Finally, the peak at 825 cm-1 may signal the presence of a CH-type bond.

Therefore, there is strong evidence that the reagents did not completely dissolve during combustion synthesis, i.e., the results of this FTIR analysis confirm the presence of residual organic compounds impregnated in the synthesized sample under a reaction temperature of 200°C. These organic residues may originate from the fuel used in the synthesis, possible contamination contained in the crucible, mortar, or sample holder, or they may simply be analytical noise.

Figure 6 - Result of FTIR analysis of the sample synthesized at 500°C



Source: The authors, 2025

Hence, the discrete peak at 3030 cm^{-1} suggests O–H group bonds. The peaks at 2343 cm^{-1} and 2114 cm^{-1} can be associated with the presence of C≡N or C≡C groups. The peak identified at 1774 cm^{-1} could correspond to the C=O group. The band at 1385 cm^{-1} is likely linked to C–H bonds.

Comparing the graphs in Figures 5 and 6, it is clear that the graph related to the sample synthesized at 500°C showed fewer occurrences of vibration peaks in the infrared region, indicating a lower concentration of residual molecular organic compounds. Once again, the influence of the reaction temperature on the final characteristics of the material obtained in powder form by combustion reaction is evident. This observation is fully consistent with the results collected in the

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XRD and SEM analyses, which clearly showed the influence of the reaction temperature on the microstructural and physical characteristics of the materials synthesized in this study.

4 CONCLUSION

According to the results achieved in this work, it was possible to conclude that the direct combustion reaction synthesis method, without the need for subsequent calcination, was feasible, efficient, and energy-efficient for the preparation of the $BiFeO_3$ perovskite structure material under a furnace preheating temperature of 500°C. At this reaction temperature, a microstructure with a significant crystalline character was obtained, based on $BiFeO_3$ as the major phase, with a mass fraction of 66%, associated with a single type of segregated phase, Bi_2O_3, with a mass fraction of 34%. Thus, it was deduced that, most likely, at a reaction temperature slightly above 500°C, it is possible to obtain a fully single-phase $BiFeO_3$-based microstructure using this quick and easy synthesis method, which took around 5.5 minutes.

In this way, it was found that the higher temperature had a significant influence on the final characteristics of the material obtained in relation to the following aspects: increased degree of crystallinity, efficient thermal energy input for the formation of the desired phase, $BiFeO_3$, reduction in the concentration of remaining organic residues impregnated in the particles and, consequently, a decrease in the tendency for the unit particles to agglomerate, as well as a decrease in the size of these particles.

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