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ARTICLE

Synthesis and Characterization of $ZrTiO_4$ for Bioceramic Applications

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Abstract: This study presents the synthesis and characterization of bio-ceramic powder composed of zirconium titanate ($ZrTiO_4$) using a combination of the powder method and the hydrothermal technique. The objective was to investigate the feasibility of this approach in producing high-purity $ZrTiO_4$ nanocomposites with potential applications in various fields. The raw materials, titanium dioxide (TiO_2) and zirconium dioxide (ZrO_2), were mixed in equal proportions and subjected to a series of processing steps. The resulting powder was analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS). XRD analysis confirmed the crystalline structure of the nanocomposites, while SEM revealed diverse granule shapes, resembling cotton fibers or clusters of zirconium and titanium compounds. EDS analysis confirmed the elemental composition and the absence of impurities, demonstrating the high purity of the prepared $ZrTiO_4$ nanocomposites. The findings highlight the successful synthesis of bio-ceramic $ZrTiO_4$ nanocomposites through the combined powder method and hydrothermal technique. These materials hold promise for various applications, including biomedical and electronic devices, due to their unique properties and high purity.

Keywords: Bioceramic, $ZrTiO_4$, Hydrothermal technique, X-ray diffraction, SEM, EDX, Elemental composition.

Introduction

This work focuses on synthesis and characterization only not the application. Ceramic materials are used in electronic and microwave devices, as well as in the manufacturing of heat-resistant materials, paints, and dyes. Their widespread utility has driven an industrial revolution that has caught the interest of many researchers [1, 2]. There are several methods for preparing ceramic composites, including chemical preparation, gel preparation, chemical precipitation, hydrothermal preparation, and powder method. Although powder technology is an effective method that can produce a large amount of compound powder, it requires time, equipment, and devices [3, 4]. In this study, we employed an efficient mechanical mixing technique to fabricate nanomaterials based on ceramics through a hydrothermal process. The synthesis of these nanoparticles necessitated the use of cutting-edge technologies, including high-speed electric mills with durable grinding balls and precise high-temperature sintering furnaces [5, 6]. This research holds great importance in the pursuit of creating non-toxic ceramic-based nanocomposites that seamlessly integrate with human body tissues. Recently, the scientific community has directed its efforts toward

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devising diverse ceramic systems for various biological purposes, including the fabrication of bone and dental prosthetics, cosmetic fillings, and medical lenses. In this context, the primary objective of our study is to advance ceramic-based nanocomposites through the powder method, specifically tailored for medical applications in orthopedic and dental surgery, by meticulously modifying their structural properties. Our ultimate aim is to draw the attention of researchers who are inclined towards exploring alternative materials to autologous bone and preserved cadaveric bones, given the potential adverse consequences associated with their usage in the future. Zirconium titanate is a bio-ceramic material that has attracted significant attention in recent years due to its exceptional properties and potential applications in various fields. It has a high wear factor, corrosion resistance, and biocompatibility with body tissues, which is particularly noteworthy [7-10].

Zirconium titanate exhibits excellent biocompatibility, chemical stability, and unique electrical and optical properties, making it suitable for use in diverse areas such as biomedicine, electronics, and catalysis.

In the field of biomedicine, bio-ceramic materials have gained prominence in applications such as bone tissue engineering, drug delivery systems, and dental implants [11]. $ZrTiO_4$, with its bioactivity and biocompatibility, holds great promise in these areas. Its ability to promote osteointegration, the process of bonding between bone and implant, makes it an ideal candidate for bone restoration and implantable medical devices [12]. Furthermore, $ZrTiO_4$ has shown potential in combating harmful bacteria in the digestive system. These medical applications highlight the importance of synthesizing high-quality $ZrTiO_4$ nanomaterials with precise control over their properties [13].

To date, several methods have been employed for the synthesis of $ZrTiO_4$, including solid-state reactions, sol-gel methods, and hydrothermal techniques [14]. Among these, the combination of the powder method and the hydrothermal technique has shown promise in achieving high-purity $ZrTiO_4$ nanocomposites with enhanced properties. Powder offers advantages in terms of homogeneous mixing, while the hydrothermal technique enables the formation of well-defined crystalline structures with controlled morphology. In this study, we aimed to synthesize $ZrTiO_4$ using a combination of the powder method and the hydrothermal technique. The utilization of these techniques allows for the preparation of highly pure $ZrTiO_4$ powders with tailored characteristics. The resulting nanocomposites can possess unique features, such as specific surface area, particle size distribution, and crystalline structure, which are crucial for their application in various fields. In terms of recent references, studies have reported on the synthesis and characterization of bio-ceramic nanocomposites using similar approaches. For instance, a recent publication by Li *et al.* demonstrated the fabrication of $ZrTiO_4$ nanocomposites through a combination of the powder method and hydrothermal synthesis, highlighting their enhanced properties for biomedical applications [15]. Additionally, in the vast realm of metal-organic frameworks (MOFs), Zr-based MOFs stand out due to their diverse structures, remarkable stability, and intriguing properties. They are hailed as promising materials for real-world applications, despite being in the early stages of development. Recent years have witnessed substantial progress in this field. This review focuses on advances in Zr-MOFs since 2008, exploring design, synthesis, structure, and applications. It starts with four synthesis strategies, emphasizing eco-friendly and scalable approaches. Zr-MOFs are then categorized by structural variations based on Zr-based building units and organic ligands. Furthermore, the review highlights the utility of Zr-MOFs in catalysis, molecule adsorption, drug delivery, fluorescence sensing, and their role as porous carriers. This specialized overview is poised to guide deeper investigations into MOFs for practical uses [16].

These references demonstrate the ongoing research efforts in the field and the significance of our study in contributing to the existing body of knowledge. In this study, we present a detailed synthesis and characterization of bio-ceramic $ZrTiO_4$ prepared through the combined powder method and hydrothermal technique. A comprehensive understanding of the synthesis process and the properties of the resulting nanocomposites is crucial for their successful application in various fields. By elucidating the methodology and analyzing the obtained nanocomposites, we aim to provide valuable insights for the further development and utilization of $ZrTiO_4$ in biomedical, electronic, and catalytic applications.

Materials and Methods

The materials and methods employed in this study aimed to produce and characterize the $ZrTiO_4$ nanocomposites using specific techniques. The powder synthesis involved the heat and pressure method at 800°C under standard conditions. The calcination process was carefully executed with two key conditions in mind: precise control over the presence of oxygen gas within the reaction system and a controlled temperature gradient rate of 5°C per minute. These conditions were essential to maintain the compound's atomic structure and to influence the material's properties during calcination. A highly ordered and crystalline atomic structure was achieved through calcination at 800°C for 2 hours with a gradient rate of 5°C per minute. Scanning electron microscopy (SEM) analysis was conducted using a typical SEM instrument (Sigma 300, USA). The SEM setup included an electron column, sample chamber, electronics console, and visual display monitor, with magnifications of 50.00 KX (1µm), 300.00 KX (200 nm), 100.00 KX (1 µm), and 300.00 KX (300 nm). SEM was used to observe the surface topography and granule shapes of the resulting powder. Energy dispersive spectroscopy (EDS) using X-rays was performed to identify the reactants and their proportions used in preparing the nanoparticles. Analysis of the weight and atomic ratios confirmed a close match with the ratios employed in the preparation process, thereby validating the accuracy of the synthesis.

To prepare $ZrTiO_4$, the following steps were followed: First, titanium dioxide (TiO_2) with a particle size of 1 micrometer and zirconium dioxide (ZrO_2) with a granular size of 5 micrometers were mixed in equal proportions by weight (Fig. 1). The mixture was then subjected to continuous stirring for 6 hours at a temperature of 40°C. After stirring, the mixture underwent grinding using an electric mixing device with grinding balls for 4 hours at a rotational speed of 350 revolutions per minute to obtain a homogeneous powder.

Subsequently, the powder was treated using a hydrothermal technique in a pressure and temperature system within a hydrothermal device. The system was placed in an oven at 100°C for 72 hours. The resulting mixture was filtered, washed, and dried until a homogeneous powder was obtained. Afterward, the powder was thermally treated at 800°C (calcination). Structural tests were conducted to evaluate the purity and composition of the $ZrTiO_4$.

The materials used in this study included titanium dioxide (TiO_2) and zirconium dioxide (ZrO_2), both with a purity of over 99%. Titanium dioxide had a particle size of 1 micrometer, while zirconium dioxide had a particle size of 5 micrometers. Both materials were sourced from the IPEN (Institute of Energy and Nuclear Research) in Brazil, a renowned research institution specializing in energy and nuclear technologies. The materials obtained from IPEN ensured the quality and reliability of the raw materials used in this study.

The combination of these powders proved to be a promising method for producing high-purity bio-ceramic zirconium titanate ($ZrTiO_4$) with a desirable composition (Fig. 1).

FIG. 1. Preparation of $ZrTiO_4$ nanocomposites.

Results and Discussion

Figure 2 displays the X-ray diffraction (XRD) pattern of the nanocomposites obtained through powder combination. The regularity in the crystalline nature and structure of the nanocomposites is reflected by the highest intensity peaks at 2Θ = 30.495° (hkl = 111) and 2Θ = 27.565° (hkl = 110), which is consistent with the findings in Ref. [11]. The crystal size was also determined using XRD.

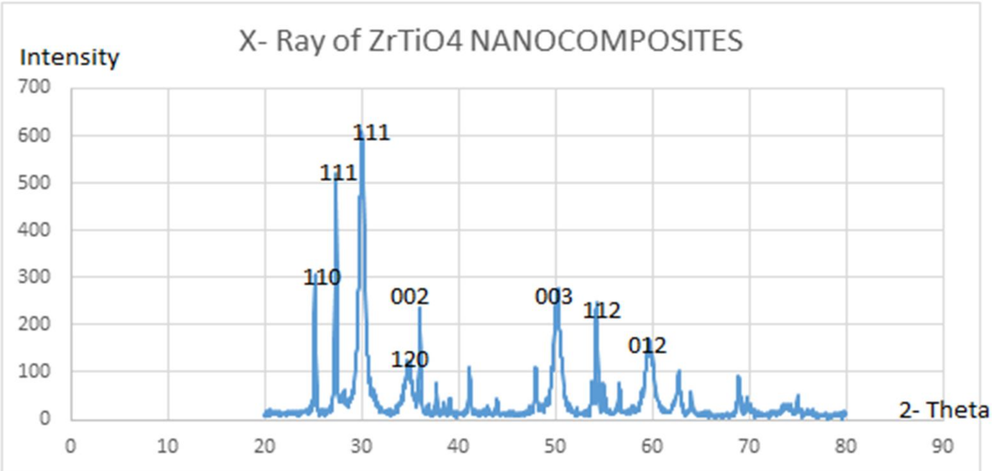
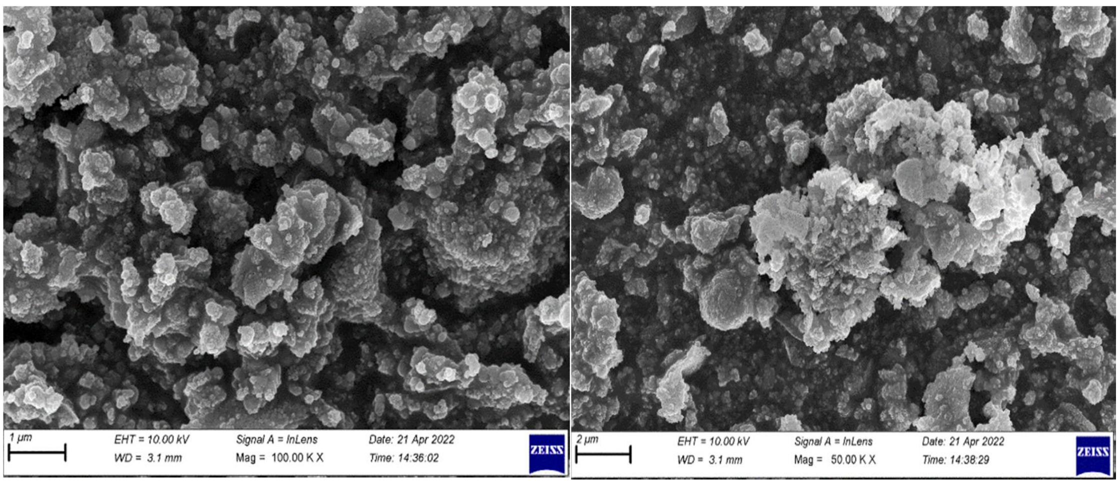


FIG. 2. X-ray diffraction patterns of the $ZrTiO_4$ bio ceramic, prepared by the hydrothermal technique and calcined at 800°C for 2 hours.

Figure 3 displays the surface topography of the powder produced through the heat and pressure method under standard conditions. The scanning electron microscope images reveal various granule shapes attached to a pattern resembling cotton fibers or a cluster pattern of zirconium and titanium compounds, which aligns with previous research [17, 18]. The magnification used in the examination aids in interpreting the granular sizes of the nanoparticles created using the hydrothermal method. These different shapes have potential medical applications, such as in bone restoration and combating harmful bacteria in the digestive system. Understanding the granular shape of the nanocomposites is crucial for such applications and distinguishes them from other methods [19, 21]. The SEM images show granule shapes resembling cotton fibers or clusters of zirconium and titanium compounds, with grain sizes ranging from 44 to 55 nm and up to 76 nm, all under 100 nm, which is consistent with previous findings. The magnification measurements facilitated the interpretation of the granular sizes obtained through the hydrothermal method. These unique granular shapes highlight the material's potential for medical applications, including bone restoration and combating harmful bacteria in the digestive system. The absence of impurities further validates the purity and precision of the preparation method. Notably, the observed zirconium-to-titanium ratios were identical to the molar ratios used during manufacturing, providing additional support for the suitability of $ZrTiO_4$ in various high-purity medical and industrial applications. SEM images of $ZrTiO_4$ show varying magnifications and grain sizes, with the smallest grains measuring less than 100 nm (Fig. 4).



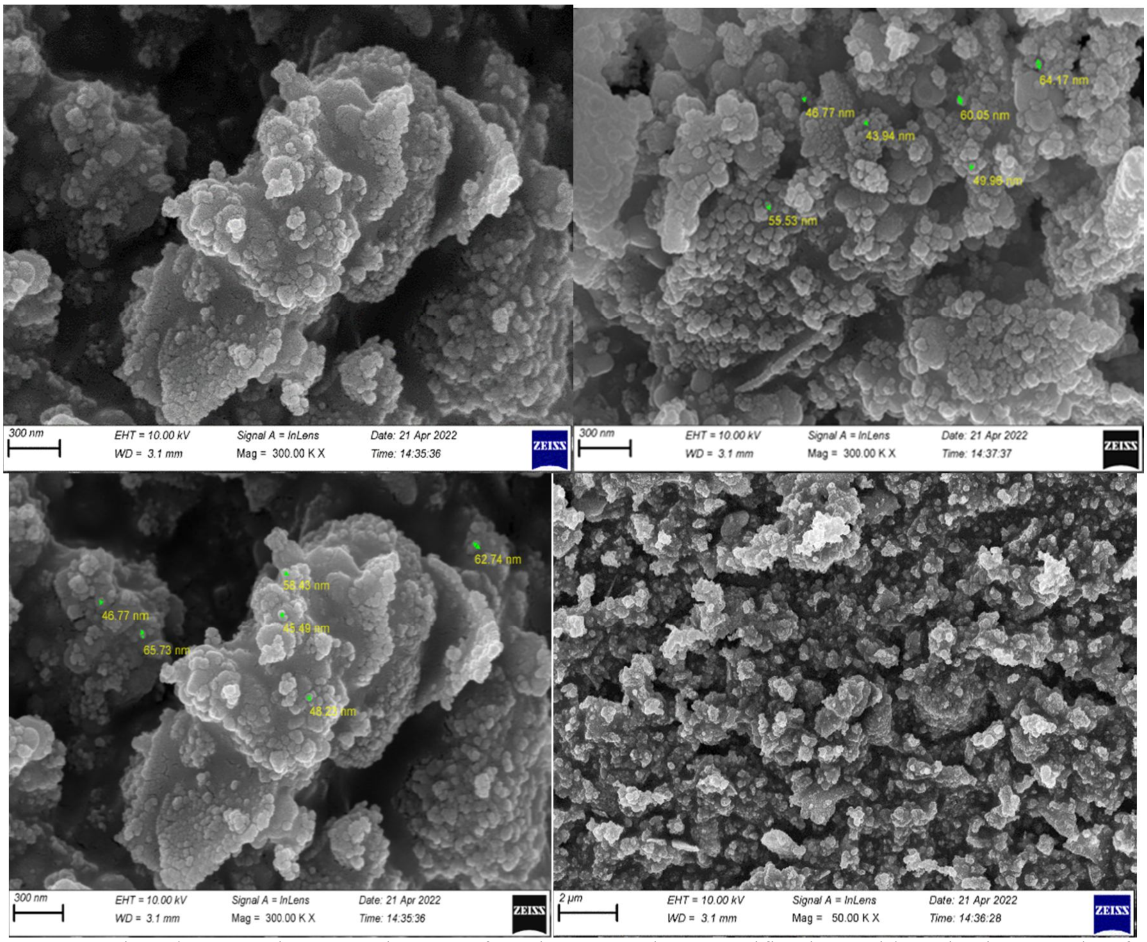


FIG. 3. Scanning electron microscope images of $ZrTiO_4$ at varying magnifications with grain sizes ranging from 44 to 55 nm and up to 76 nm, all under 100 nm.

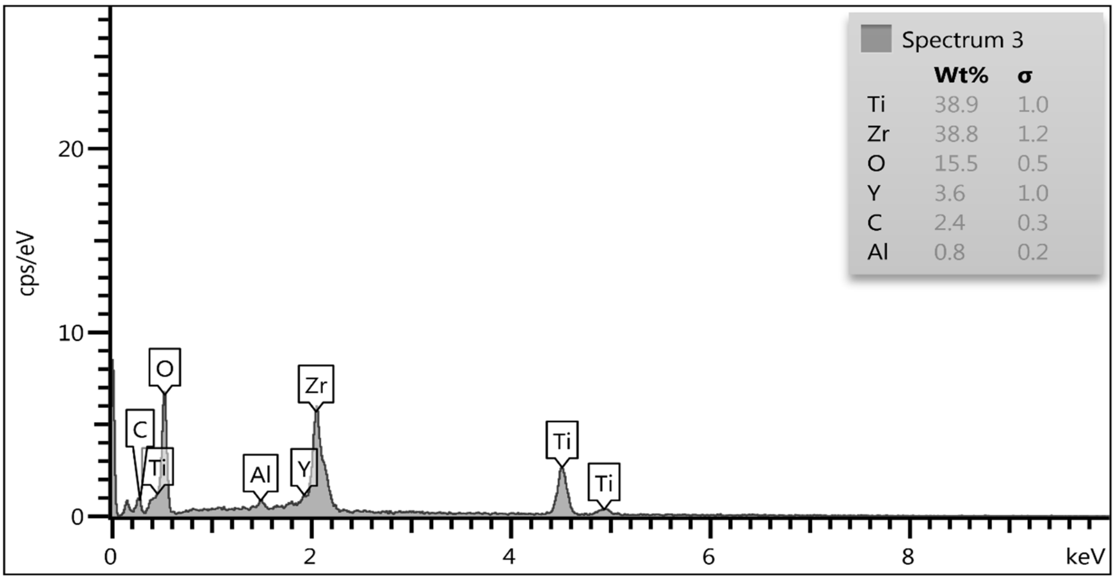


FIG. 4. EDX (energy dispersive X-ray) analysis of the $ZrTiO_4$ nanocomposites, depicted through images and a table.

Elements	Zr	Ti	O	another
Wt%	38.8	38.9	15.5	6.8

The diagram in Fig. 5 demonstrates the ability of $ZrTiO_4$ with a bio-ceramic structure to absorb and transmit infrared waves within the range of 400 to 4000 cm^{-1}, based on the energy used and the wave number. The examination results indicate that the highest permeability value for the prepared nanoparticles was observed at 1701 cm^{-1}, as depicted in Fig. 5. Additionally, the value of transmittance at 1220.2 cm^{-1} was observed. This composite has the potential for use in electronic devices that require materials with high permeability.

Table 1 provides valuable insights into the transmittance characteristics of the composite material. Notably, the highest transmittance, at 75%, occurs at a wave number of 1701.22 cm^{-1}. In contrast, the lowest transmittance is recorded at 99%, corresponding to a wave number of 2866.22 cm^{-1}. These findings indicate specific wavelength regions where the composite demonstrates its highest and lowest transparency, which is of significance for understanding its optical properties and potential applications.

TABLE 1. FTIR $ZrTiO_4$.

T%	K cm^{-1}
95%	3475.73
98%	2981.95
99%	2866.22
75%	1701.22
82%	1431.18
85%	1323.17

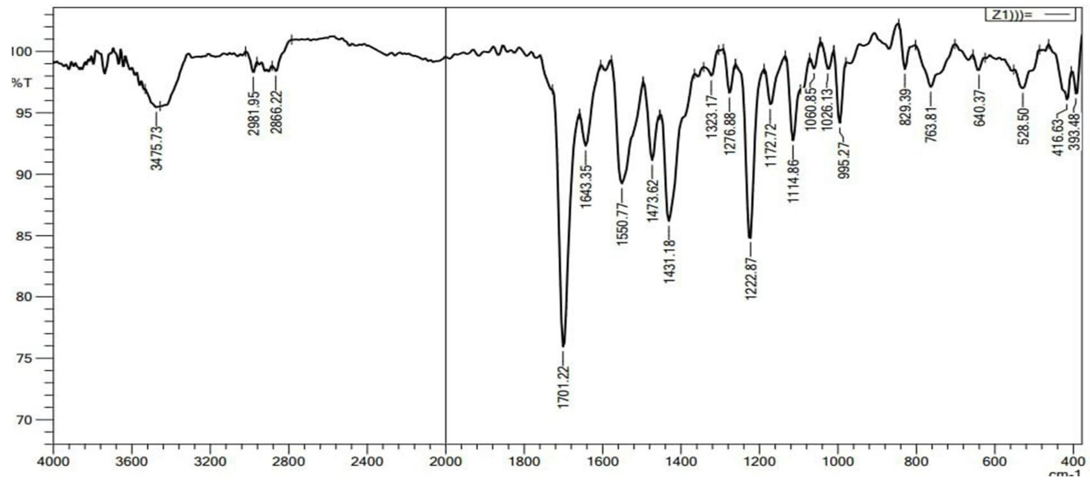


FIG. 5. The analysis of transmittance and absorbance of the $ZrTiO_4$ nanocomposites.

Conclusions

In summary, the results of this work have led us to the following conclusions:

The pressure and heat technique successfully produced ceramic nanoparticles with high purity and without any loss in the amount of material prepared. The method used in this work resulted in the formation of various forms of granules composed of zirconium and titanium that formed $ZrTiO_4$ at a temperature of 800°C. All the techniques used to interpret the atomic composition of the nanoparticles confirmed the accuracy of the preparation method. The values of the materials involved in the preparation were consistent with the values of the prepared products in the $ZrTiO_4$. These nanoparticles can be potentially used in various medical applications since they are free from any impurities that could pose risks to biological tissues.

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