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STUDY OF THE EFFECT OF PVA INCORPORATION ON THE PARTICLE SIZE OF CERAMICS WITH NBT STRUCTURE WITHOUT STRUCTURAL LEAD

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Abstract: In this work, we report the particle size and compositional results obtained during the incorporation of polyvinyl alcohol in the regrind of an advanced ceramic with NBT (Bi0.5-xNa0.5+x) TiO3 structure, which was synthesized by high-energy mechanical milling. Mechanical milling proved to be a suitable process to synthesize this type of material by obtaining a high purity of the phase. The effect of incorporating polyvinyl alcohol (PVA) as a milling aid after the thermal process was studied. As a result, a particle radius reduction of 48 % was obtained, reaching nanometric sizes. In the study by infrared spectroscopy, it was observed that PVA did not generate bonds with the ceramic, so it is not an agent that modifies the structure of the ceramic.

Keywords: Advanced ceramics - Mechanosynthesis - Infrared spectroscopy - PVA - Particle size analysis - Infrared spectroscopy.

INTRODUCTION

The new technological needs have led to an exponential increase in the research on this type of material worldwide [1-3]. One category of the most widely used materials are the so-called "smart materials", and for energy storage perovskites are of particular interest. Perovskites have a general formula ABX3 [4-8]. The most commercial perovskite is known as PZT (lead zirconate titanate). However, it has already been shown to have harmful effects on humans when handled and also on the environment during processing. It is because of the above-mentioned that many researchers have focused on alternative ceramic systems to replace PZT while retaining their optimum properties [9-15]. In previous works we were able to synthesize an NBT ceramic with high purity [16]; the NBT ceramic is an interesting option for the substitution of PZT ceramics due to its synthesis processes and good properties. Our synthesis method is high-energy mechanical milling. This technique is mainly used for particle size reduction; however, it can also form solid solutions, chemical reactions, and crystalline phases [17]. An important aspect of advanced ceramics is the particle size, since the finer the particle size the more properties can be improved [18-21]. Looking for the size reduction of our ceramic, experiments were designed where different percentages of PVA (polyvinyl alcohol) are incorporated as an aid for the swirling of the NBT (Bi0.5-xNa0.5+x) TiO3) ceramic system.

MATERIALS AND METHODS

The precursor reagents for the synthesis were: Bi2O3 (99.9 % purity, Aldrich), Na2CO3 (99.5 % purity, J.T. Baker), and TiO2 (99.9 % purity FAGALAB). Based on the required stoichiometry the powders were weighed and treated accordingly. After this process, the powders were subjected to high-energy mechanical milling for 5 hours. Once the powders were recovered these were subjected to X-ray diffraction analysis to identify the crystalline phases, using a Bruker D8 Advance equipment, with Cu Kα1 radiation (40 kV, 40 mA), in a 2θ range from 20° to 80°. For initial particle size analyses, the powders were analyzed on a NanoBrook 90 plus equipment. For particle size reduction, the experiments will be based on the incorporation of PVA by varying the grinding time, using zirconia balls as grinding media, the grinding time was 5 hr.

RESULTS AND DISCUSSION

X-RAY DIFFRACTION

The results of the X-ray diffraction characterization are shown in Figure 1.

The results of the XRD analysis for the compositions NBT are shown in Figure 1. As a result, we can observe that at low levels of x in the composition, the ceramic has the presence of secondary phases, phases such as $Bi_4Ti_3O_{12}$ (COD: 1534182 F mmm), and Bi_2O_3 (COD: 1010004, P 1 21/c 1). On the other hand, as the value of x increases, it is observed the more significant formation of the perovskite-type structure with the phase sodium bismuth titanate $(Bi_{0.5}Na_{0.5})TiO_3$ (NBT) (COD: 2103296 R3 c: H) [22-25].

PARTICLE SIZE ANALYZING

The results of the particle size analysis are shown in Figure 2. In it, we can see that we obtain a ceramic with fine particle sizes obtaining an average particle diameter of 250 nm. A minimum size close to 50 nm and a maximum size of 1000 nm.

PARTICLE SIZE ANALYSIS AFTER PVA INCORPORATION

Once the phase quantification was obtained, new particle size analyses were carried out, analyzing the influence of PVA incorporation and establishing five different periods for the analysis. Figure 3 shows the results of the distribution of particle size.

By analyzing Figure 4, we can contrast the particle size of the ceramic without PVA (star-shaped curve) compared to the new analyses incorporating PVA into the milling after annealing. After one hour of milling, the smallest particle size is obtained, reaching an average size of 72 nm, which represents a reduction of 48 % from the original size. When 2 hours of milling are applied, 77 nm is obtained. As for the rest of the milling, times present similar behaviors, with 116 and 141 nm values.

ANÁLISIS DE ESPECTROSCOPIA INFRARROJA

Were performed infrared spectroscopy studies to verify that the incorporation of PVA does not generate bonds with our ceramic system. Figure 4 shows the spectra obtained by FTIR; the dashed lines indicated the vibrational bands attributed to the bonds present in the PVA. In the spectrum corresponding to the NBT ceramic, we have the characteristic band in the perovskites near a wavelength of 500 cm^{-1}. Based on this information, we can establish that the incorporation of PVA does not generate bonds with the ceramic; due to the spectra of the ceramic incorporating PVA, no strange bands have a presence, only traces of the characteristic bands of PVA. Table 1 shows the locations of the vibration bands of the ceramic systems studied.

CONCLUSION

Regarding particle size, the incorporation of PVA in the milling of the ceramic system reduces the average particle size by 48 %. Based on these results, we can confirm that high-energy mechanical milling is an effective process for obtaining complex ceramic powders, thus obtaining a highly reactive ceramic, significantly decreasing the synthesis times compared to different methods. Furthermore, PVA does not interact chemically with the ceramic system, only reducing the particle size in this case.

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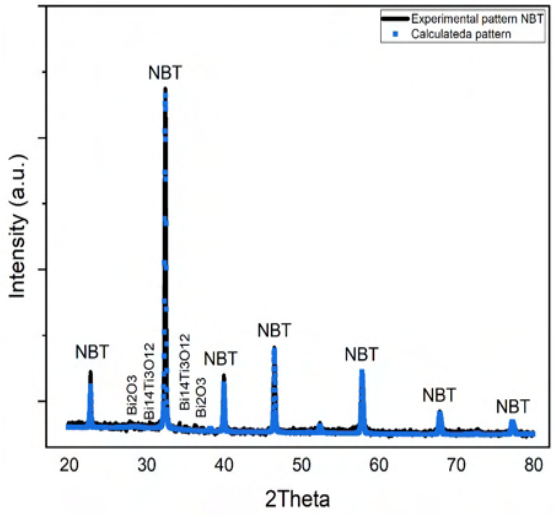

Figure 1.- Diffraction pattern of the NBT ceramic system.

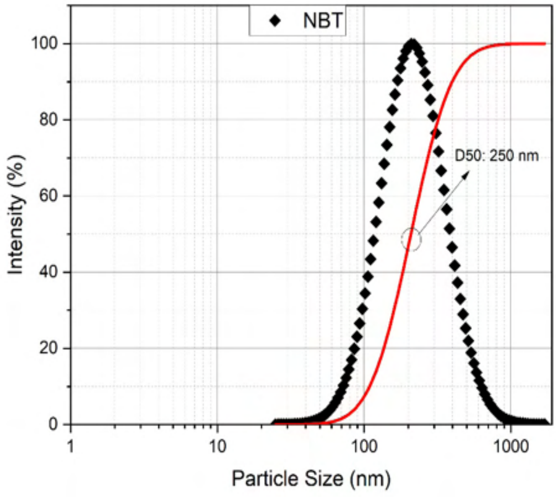

Figura 2.- Diámetro medio de radio de partícula obtenido.

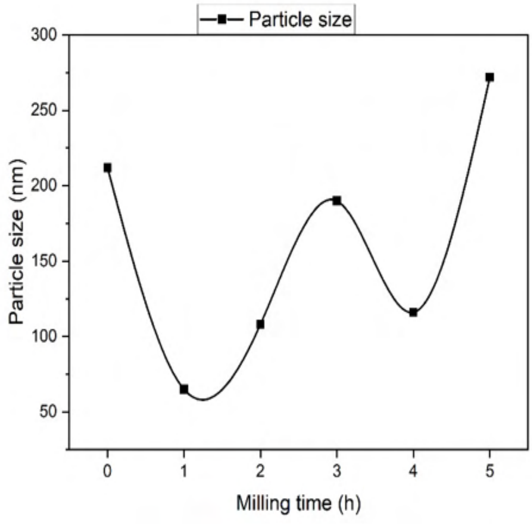

Figure 3. - Particle size populations of ceramic powders under different milling times incorporating PVA.

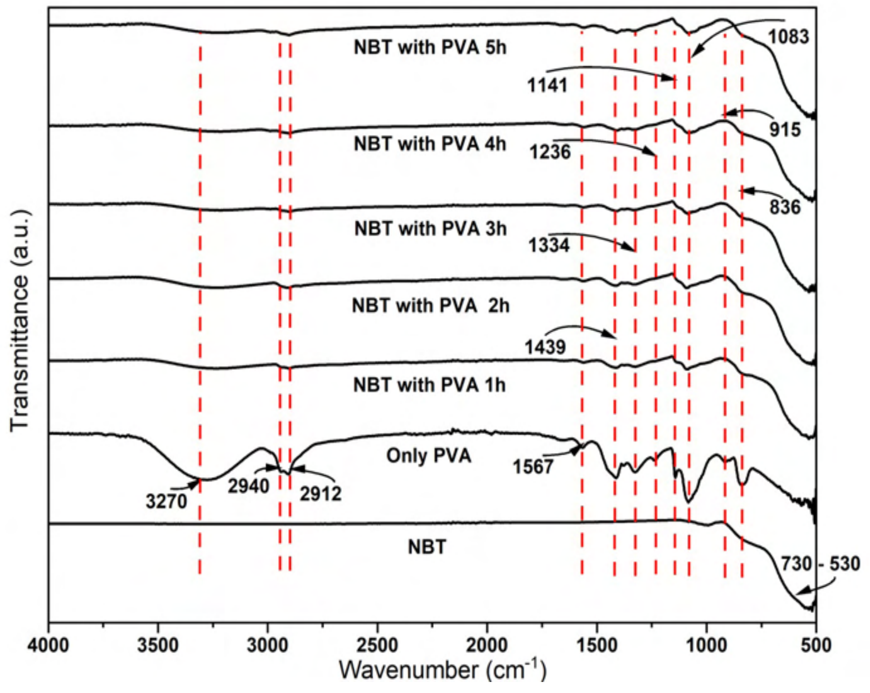

Figure 4. - FTIR spectra of the NBT ceramic system under different milling times incorporating PVA.

Position (cm^{-1})	Chemical group	Reference(s)
3270	Stretching of O-H	[25, 32]
2940	Asymmetric stretching of C-H	
2912	Asymmetric stretching of C-H_2	
1567	Vibrations C-O	
1439	Bending of CH_2	[25, 32-33]
1334	Bending of CH_2	
1236	Stretching vibrations C(O)-O	
1141	Stretching of C-O	
1083	C-O-C	[25, 32-33]
915	Rooking of CH_2	
836	Stretching of C-C	
730-530	Set of bands attributed to the NBT phase.	[34]

Table 1. - Vibration modes and band frequencies in NBT ceramics incorporating PVA.

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