One-step synthesis of lead zirconate titanate particles using a solid-state reaction method

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ABSTRACT

This study aims to determine the one-step synthesis of lead zirconate titanate (PbZr(1-x)Ti x O 3; PZT) particles, using a solid-state reaction method. These particles were synthesized at x of 0.48, using a solid-state reaction (SSR) method at various operating temperatures of 700–1000 °C. Before the reaction in a muffle furnace, the precursor powder was shaker milled for 2 h. The structural analysis of the diffraction patterns showed the formation of the PZT perovskite particles. It also showed the evolution from the tetragonal phase (at 800 and 900 °C) to the coexistence of tetragonal and rhombohedral stages in samples synthesized at 1000 °C. Furthermore, the crystallite particle size synthesized and commercialized at 1000 °C were 36.00 and 47.98 nm, respectively. These showed the formation of monodispersed size distribution, with an average of approximately 4.7 μm. Therefore, the PZT particles were successfully synthesized through a one-step synthesis, using the SSR method with a pre-mixed shaker milling.

1. Introduction

The study and development of piezoelectric mechanisms within the field of sensors and actuators are being intensively carried out as multifunctional tools, for measuring various control processes in many industries. This indicates that piezoelectric materials are principally used as sensors when mechanically deformed, to generate a detectable voltage. Furthermore, perovskite piezoelectric materials have widely been used in transducer devices, where high constants are essential for device performances [1]. Lead zirconate titanate Pb(Zr,Ti)O 3 (PZT) is also known to be one of the most applied materials in electromechanical devices, such as an ultrasonic transducer active element, due to their excellent piezoelectricity properties [1, 2, 3, 4, 5]. This was observed in a recent study, which reported that the piezoelectricity properties were applied as a piezocatalytic application, for the degradation of various environmental pollutants [6].

PZT has a perovskite structure, with Zr and Ti ions randomly placed on the edge of cell boundary sites. When used at 50% mol PbTiO 3 , this structure is referred to as the morphotropic phase boundary (MPB) [7], whose condition between the tetragonal and rhombohedral phases was explained by the composition–temperature diagram [8, 9]. The coexistence of the tetragonal-rhombohedral phases in the PZT ceramic disk showed that the piezoelectric properties were higher when close to the boundary sites [10]. Also, infrared spectroscopy measurements were performed to determine the monoclinic-tetragonal phase transition in PZT ceramics. As a function of temperature, behavioural tracking is found to be very possible [11]. Furthermore, the electrical properties of PZT were tailored by incorporating appropriate dopants, to meet the technical demands of user-based applications [12].

Several methods have been developed in recent years to synthesize nanocrystalline PZT ceramics, such as mechanochemical, chemical, and physical techniques [3, 4, 12, 13, 14, 15]. However, the mechanochemical methods involve an energy-intensive milling and a long duration of approximately 80 h, facilitated by a high-energy planetary ball mill [16]. These synthetic processes are likely to destroy large particles and refine the grains of the commercial PZT powder [4]. Besides the high-energy mechanochemical methods, a wide variety of preparation techniques are found to be available and employed to produce PZT nanoparticles, such as hydrothermal [13, 17, 18, 19] acid acetic-based sol-gel [15, 20, 21], molten salt and polymeric precursor models [22, 23], as well as pyrolysis route. Also, the bandgap of the synthesized PZT powder was reportedly found to decrease with an increase in synthetic temperature, using the sol-gel method [24].
Based on solid precursors, the solid-state reaction (SSR) has been applied in the synthesis of PZT materials due to its simplicity. The temperature of this process also determined the characteristics of the synthesized PZT, such as crystallinity and particle sizes [9, 16]. This involves a long duration, approximately 12–48 h, of the mechanical pre-mixing precursor process, such as a wet/dry ball-milling, to obtain more homogenous and finer particles [25, 26]. In the SSR process, the mixing technique becomes crucial due to these factors enhancing the synthetic procedure [25, 26]. Therefore, the development of a PZT one-step SSR synthesis with a shorter pre-mixing duration is essential, to enhance the efficiency of the reaction.

This study reports the synthesis of PZT (PbZr\(_{1-x}\)Ti\(_{x}\)O\(_3\) with \(x = 0.48\)) powder through the application of a solid-state reaction method with a shorter duration of shaker milling precursor treatment. Subsequently, this study aims to obtain a more time and energy-efficient PZT synthesis, by shortening the pre-mix duration facilitated by a shaker milling process. The temperature-dependent formation of PZT crystal powder is further discussed in this study. The comparison of the crystallite size of synthesized and commercial PZT powders is also being reported. By employing these methods, the efficiencies of the synthetic process are enhanced.

2. Experimental details

2.1. Particle synthesis

The chemical composition of synthesized PZT supported the compound formula as PbZr\(_{1-x}\)Ti\(_{x}\)O\(_3\) with \(x = 0.48\). The precursors also included the reagents produced by Sigma Aldrich, such as PbO (99.90%), ZrO\(_2\) (5 μm, 99%), and TiO\(_2\) (99.8%). The raw precursor materials were approximately weighed at a total weight of 40 mg and uniformly mixed according to their stoichiometric ratio.

The precursor mixture was then placed in a 100-ml stainless steel tube vial containing three 5-mm diameter zirconia balls. This was subsequently mixed by a shaker milling machine (manufactured by PPFUG; SM20140200-A/B LIPI-Indonesia), operated at a rate of 1500 rpm for 30 min, leading to the production of a finer and more homogenous precursor mixture. Afterwards, the shaker milled mixture was placed in a closed ceramic crucible and calcined at 700, 800, 900, and 1000 °C, with a temperature ramp rate and reaction time of 10 °C/min and 2.5 h in an electrical furnace, respectively. After the duration of the process, the reactor was naturally cooled to room temperature.

2.2. Characterizations and analyses

The crystal structure of the synthesized PZT particles was analyzed using X-ray diffraction (XRD, PANalytical), and compared to the commercial type. The 2-theta scan ranged from 10–99.9° of the synthesized particles, employed an open-source and general-purpose peak fitting software, Fityk 1.3.2 (Copyright 2001–2015 Marcin Wojdyr). The MAUD (Material Analysis Using Diffraction) program was then used for the calculations of crystallite sizes (\(d_c\)) and Rietveld refinement of the XRD patterns, for the phase identification of the prepared particles (Copyright © Luca Lutterotti).

The morphological and chemical composition analyses of the synthesized powder were carried out using a scanning electron microscope (SEM; JEOL type JSM 6560) at an acceleration voltage of 20 kV, with a magnification of 1500 and 1000 times. This was further equipped with energy-dispersive X-ray spectroscopy (EDS). The size distributions (\(d_h\)) of the synthesized particles were measured using an analyzer, based on a scattering light intensity (Horiba, SZ-100). Also, the atomic M-O bonding (with M are Ti, Zr) of the synthesized perovskite PZT were estimated using a Fourier transform infrared (FTIR) spectroscopy (Thermo Scientific Nicolet iS5 Thermo, Agilent).

3. Results and discussions

3.1. Structural analyses of as-synthesized PZT powder

The calcined temperature is known to often determine the crystallinity of the synthesized PZT particles [27], with several studies reporting the application values of 400–1200 °C in the synthetic process [19, 23]. To identify the crystallinity of the particles including the crystallographic structure, crystallite size, and preferred orientation, the XRD patterns of the synthesized and commercial PZT at 700, 800, 900, and 1000 °C were measured (Figure 1). These synthesized compounds were identified by comparing the diffraction data with a database of known reference materials. This was used to track phase changes as a function of variables, such as temperature.

Based on Figure 1, the XRD patterns of the synthesized particles were shown, strongly indicating the formation of crystallized compounds through the appearances of diffraction peaks, at an angle 2θ ranging between 10–70°. Afterwards, the reference peaks of PbTiO\(_3\) were observed in all synthesized particles. The formation of tetragonal PbTiO\(_3\) (◆ mark) was also observed in the XRD pattern of particles synthesized at 700 °C. However, some amounts of ZrO\(_2\) (● mark) were detected in the sample, as indicated by the diffraction peaks of ZrO\(_2\) (● mark), PbZrO\(_3\) (● mark), and PZT (▲ mark), respectively. This was because the reaction time and temperature were the most important parameters in the formation of PZT, through a solid-state reaction method. Meanwhile, insufficient reaction time and temperature caused an incomplete formation of the PZT perovskite phase [15]. To observe the effect of calcination, the temperature was increased to 800, 900, and 1000 °C. As observed in Figure 1, the diffraction peaks of PbZrO\(_3\) were apparent and did not detect evidence for the formation of other phases. This strongly indicated...
that the calcination temperatures of 800 and 900 °C dominantly produced rhombohedral PbZrO₃. With the calcination temperature of 1000 °C, the diffraction patterns exhibited the formation of coexistence tetragonal and rhombohedral PZT. The diffraction pattern of particles synthesized at 1000 °C was closely similar to that of the commercial compounds, as indicated by the peaks of PZT and ZrO₂. This result qualitatively implied higher and lower formations of the synthesized PZT perovskite phase and PbTiO₃, as temperature increased. The peaks of ZrO₂ were then reduced and eliminated as the calcination temperature increased, indicating the complete decomposed formation of ZrO₂, PbO, and TiO₂ precursors. The coexistence of tetragonal and rhombohedral phases was also understandable due to the molar ratio of Zr/Ti (52/48) being closely similar to the MPB (Morphotropic Phase Boundary) diagram of PZT [19].

The diffraction peaks in the 2θ range of 30–33° were investigated to elaborate the crystal phase formation of the synthesized particles. This was because the dominant characteristic peaks of the phases were included in this range (Figure 2). According to Figure 2, the PZT perovskite phase with the coexistence of the tetragonal and rhombohedral stages corresponded to the commercial compound, as small impurities was observed in all samples. In the XRD pattern of the powder calcined at 700 °C, the dominant tetragonal phase of PbTiO₃ (■ mark) was observed at the (101) and (110) planes [28]. Based on the thermal conditions in the precursor calcination, the impurity peaks were also observed in the XRD pattern obtained from the incomplete reaction. When the temperature was increased to 800, 900, and 1000 °C, the coexistence of the tetragonal PbTiO₃ and rhombohedral PbZrO₃ phases in the XRD patterns were observed [28, 29]. This suggested that there was a reaction to the formation of the PZT perovskite phase [30]. Moreover, the samples calcined at 800 and 900 °C showed similar XRD patterns, implying the formation of identical phases. The PZT sample with a calcination temperature of 1000 °C for 4 h was then considered to be the optimal thermal parameter for this study, due to its XRD pattern being similar to a commercial compound. By increasing the calcination temperature, the structure of synthesized PZT powder was comparable with the commercial samples. In addition, the PZT structure was characterized by a shift in the (101) and (110) axes, which was becoming narrower with a decrease of the ZrO₂ phase along the (111) axis.

As the calcination temperature increases, the Zr ions were observed to replace Ti in PbTiO₃, causing a structural change from tetragonal to rhombohedral phase in PbZrO₃. The splitting of the PbTiO₃ (100) reflection into (101) and (110) in PbZrO₃ and PZT provided clear evidence of the tetragonal and rhombohedral coexisting phases [31]. These results were reasonable because the formation of the Zr-rich crystal growth was more energetic when the calcination temperature increased [31].

Based on this study, the heating profile also affected the crystal growth of the PZT sample. To analyze the quality of the synthesized samples, peak fitting processes were performed according to the Rietveld refinement method, for the quantitative identification of the phases, compared to the commercial PZT powder. Furthermore, Figure 3 showed the Rietveld quantitative analysis of the synthesized and commercial samples, refined with the reference phases of PZT, PbTiO₃, PbZrO₃, ZrO₂, and TiO₂. The intensity of the diffraction peak was also compared to the crystal quantity, as regards the -phase or -interplanar spacing. According to Figure 3, many quantities of ZrO₂ with a small amount of PZT, PbTiO₃, and PbZrO₃ phases were detected in the 700 °C synthesized particles. Meanwhile, the quantity of the PZT phase increased at the synthesized temperatures of 800 and 900 °C, as indicated by the higher reflection peaks of (110) and (101). When the calcination temperature increased to 1000 °C, the quantitative analysis showed the similarities between the commercial and synthesized phases, especially the phases of PZT (110, 101) and ZrO₂. However, there was no PbTiO₃ detected in both samples, indicating that the quantities of the PZT and ZrO₂ phases increased and decreased with the greater calcination temperature, respectively. This ZrO₂ phase was detected in both the commercial and synthesized samples. Therefore, these results strongly indicated that the 1000 °C-synthesized PZT was relatively similar to the commercial sample.

Figure 2. The XRD patterns of (a) commercial powder and synthesized by solid-state calcination, at temperatures of (b) 1000, (c) 900, (d) 800 and (e) 700 °C, in a 2θ range of 30–33°.
Based on the higher temperature, the increase in the quantity of the PZT phase was attributed to the crystal growth. This was because the temperature had an important role in the growth of crystalline substances. In this study, Table 1 indicated that the calculated crystallite size of the PZT phase enlarged between 31.63-45.87 nm, when the calcination temperature increased from 700-1000 °C, respectively. One interesting result was the decrease in the crystallite size of the ZrO₂ phase as temperature increased (Table 1). This phase decreased from 57.13 to 16.28 nm as temperature increased, implying more completion of the PZT synthesis reaction. Therefore, this result showed that the PZT crystallinity was enhanced with the increase of the calcination temperature.

In comparison to the PZT synthesized at 1000 °C, the commercial sample showed a higher crystal quantity as indicated by the diffraction intensity. This was approximately two and half times larger than that of the synthesized PZT sample. Also, the width of the peaks was inversely proportional to the crystallite size, as described by the Scherrer equation. The diffraction peak of the synthesized sample showed broader width than that of a commercial sample. This indicated that the thinner peak of the commercial sample corresponded to the crystallite size, such as 53.44 nm. However, the size of the synthesized PZT sample at 1000 °C was 45.87 nm (Table 1). A broader width for the diffraction peak of the synthesized sample indicated that a defect in the crystalline structure was likely to be found or more amorphous (a solid imperfect crystallinity). From Table 1, both the synthesized and commercial samples were found to contain PZT, PbTiO₃, and ZrO₂ phases. Meanwhile, the TiO₂ and ZrO₂ phases only occurred in the synthesized PZT sample as an impurity.

### 3.2. Morphological and elemental analyses

SEM images were produced based on the type of detector used, to define a sample at the micro to the nano-scale. However, the EDS appendix should be used to measure the relative abundances of different elements, whose identities were not only determined by the SEM.

Several studies were found to have reported that the synthesized temperature determined the characteristics of the resultant particles. Oliveira et al. (2014), demonstrated that synthesized particles had spherical morphologies, agglomerated, and larger sizes due to higher temperature, through the polymeric precursor method. To investigate the effects of synthesized temperature on the morphologies, the SEM micrograph was evaluated.

Figure 4 shows the SEM micrographs of the synthesized PZT particles, where the compounds were non-spherical with irregular shapes. Also, the sizes increased with the higher calcination temperature. These sizes were based on monodispersed distribution, as described with a single peak

![Figure 3](image-url) The quantitative analysis using a Rietveld refinement of (a) commercial, and synthesized PZT powder, through the SSR method at temperatures of (b) 1000 °C, (c) 900 °C, (d) 800 °C and (e) 700 °C.

Based on the higher temperature, the increase in the quantity of the PZT phase was attributed to the crystal growth. This was because the temperature had an important role in the growth of crystalline

### Table 1. The average crystallite sizes of as-synthesized and commercial PZT samples.

| Temperature (°C) | Phase | FWHM (°) | 2θ Average (°) | Average Crystallite Size (nm) |
|-----------------|-------|----------|----------------|-------------------------------|
| 700             | PZT [101, 110] | 0.21, 0.38 | 30.55, 30.96 | 31.63                        |
|                 | ZrO₂ [111]       | 0.15      | 31.42          | 57.13                        |
|                 | PbTiO₃ [110]      | 0.16      | 32.4           | 54.04                        |
|                 | PbZrO₃ [122]      | 0.144     | 30.3           | 59.74                        |
| 800             | PZT [101, 110] | 0.25, 0.31 | 30.76, 31.00  | 31.19                        |
|                 | ZrO₂ [111]       | 0.17      | 31.46          | 49.58                        |
|                 | PbTiO₃ [110]      | 0.15      | 32.55          | 56.17                        |
| 900             | PZT [101, 110] | 0.232, 0.294 | 30.79, 30.99  | 33.22                        |
|                 | ZrO₂ [111]       | 0.257     | 31.44          | 33.57                        |
|                 | PbTiO₃ [110]      | 0.351     | 24.63          | 24.63                        |
| 1000            | PZT [101, 110] | 0.17, 0.21 | 30.97, 31.14  | 45.87                        |
|                 | ZrO₂ [111]       | 0.53      | 31.43          | 16.28                        |
| Comm.           | PZT [101, 110] | 0.14, 0.19 | 30.87, 31.01  | 53.44                        |
|                 | ZrO₂ [111]       | 0.18      | 31.31          | 47.91                        |
appearance in the Figure insets. Furthermore, the average particle size, \( d_p \), increased at 803.5, 5135.5, 3805.2, and 4686.6 nm, through the calcination temperature of 700, 800, 900, and 1000 °C, respectively. Subsequently, the particles synthesized at 1000 °C were hardly agglomerated (Figure 4d), due to the sintering during the higher temperature synthesis. The formation of larger particles due to the increasing reaction temperature was also supported by the crystallite size enlargement, as an effect of the calcination temperature. Based on Table 1, the PZT crystallite sizes increased from 31.63 to 45.67 nm when the calcination temperature increased between 700 to 1000 °C.

Higher calcination temperature also caused the formation of larger particles produced through grain coarsening. This enlargement was initiated by the crystal growth caused by a reduction in the total interfacial energy of the particles [27]. As shown in Figure 4d, the hard-agglomerated particles showed the formation of larger particles due to sintering. To investigate the effects of the synthetic temperature on the Zr/Ti ratio, the EDS spectra of the particles synthesized at 700, 800, 900, and 1000 °C, were measured (Figure 5). This ratio was calculated according to the measured elemental mass percentage from EDS spectra measurement i.e., Pb, Zr, and Ti. Based on Table 2, the calculated Zr/Ti ratio of the synthesized particles were summarized, showing a decrease of 2.90, 2.77, 1.02, and 0.69 at 700, 800, 900, and 1000 °C, respectively.

The synthesized sample at 700 °C exhibited a Zr-rich substance, as indicated by the value of Zr–Ti ratio at 2.99 (Figure 5a). The Zr-rich elements detected in this sample were associated with the formation of the ZrO2 compound, as indicated by the XRD results in Figure 1. Furthermore, the Zr–Ti ratios of 800 and 900 °C samples were 2.77 and 1.02, respectively (Figure 5b and c), indicating that the samples were also Zr-rich. However, they indicated the formation of PbZrO3, as calcination temperature increased to 800 and 900 °C. In comparison, the 1000 °C sample exhibited Ti-rich substances as indicated by the Zr–Ti ratio of 0.69, therefore, suggesting the formation of PZT compound (Figure 5d). The Ti-rich tetragonal PZT was also reportedly beneficial in stabilizing the rhombohedral lead-based ferroelectrics, through their addition into the material [32].

According to Figure 5d, the elemental composition analysis of the synthesized particles indicated the formation of a PZT compound at 1000 °C. This was due to the detection of the Pb, Zr, Ti, and O element peaks. However, a small number of other compounds (TiO2, ZrO2, and PbO) were detected. This indicated that the synthesized PZT particles were free from impurities, as no other chemical elements (instead of Pb, Zr, Ti, and O) were detected.

Based on Figures 1 and 5, the evolution of Zr into a Ti-rich substance was observed when the calcination temperature increased from 700 to 1000 °C. This indicated that 700 °C was insufficient to accommodate complete calcination, to form PZT. The replacement of Zr ions for Ti in PbTiO3 was also observed at 800 or 900 °C calcination temperature, while the formation of PZT compounds occurred at 1000 °C. This result showed that the reaction temperature determined the control of the Zr/Ti ratio.

Infrared spectroscopy was carried out by detecting the stretching vibration of M-O ions in the samples, to investigate the loading of metal reactors (Ti, Pb, Zr) into the A and B sites of the perovskite structure (ABO3). Figure 6 shows the FTIR spectra of 1000°C-synthesized and commercial PZT particles, where a flat band was observed between 1000–500 cm⁻¹. This indicated that no other elemental bonds (especially organic bonds) were observed in the synthesized and commercial samples. Also, a sharp band at ca. 584 cm⁻¹ was observed in all samples, indicating
its correspondence to the M-O bonding (M = Ti, Pb, Zr). In the 700 °C samples, a band at 755 cm\(^{-1}\) was found to be associated with the stretching vibration of Zr–O, strongly indicating the existence of the ZrO\(_2\) phase [15, 33]. This was attributed to the incomplete reaction due to a low calcination temperature, leading to a fraction of ZrO\(_2\) precursor remaining in the samples. As the calcination temperature increased (800, 900, and 1000 °C), the band of 755 cm\(^{-1}\) reduced and disappeared, indicating that the ZrO\(_2\) precursor experienced a complete decomposition reaction with PbO and TiO\(_2\), to form PZT. This was in line with the XRD results presented in Figure 1, indicating the formation of PZT in the as-synthesized samples. It also suggested the substitution of Zr ions for Ti in the structure.

Table 2. The elemental analysis (EDS) of the main elements of the synthesized PZT powder.

| Temperature (°C) | Zr (wt%) | Ti (wt%) | Zr/Ti |
|-----------------|----------|----------|-------|
| 700             | 31.32    | 10.77    | 2.90  |
| 800             | 31.38    | 11.34    | 2.77  |
| 900             | 13.28    | 13.03    | 1.02  |
| 1000            | 8.46     | 12.21    | 0.69  |

Figure 5. The EDS Spectrum of particles synthesized at a temperature of (a) 700 °C, (b) 800 °C, (c) 900 °C, and (d) 1000 °C.

Figure 6. The FTIR spectra of commercial- and synthesized- PZT particles in a wavenumber range of 500–1000 nm.
4. Conclusions

The synthesis of PZT through a one-step method was successfully demonstrated at the calcination temperatures of 700–1000 °C, by applying the processes of an SSR and a pre-mixed shaker milling, respectively. This sample was successfully synthesized by applying a shorter time of mechanical pre-mixing at 30 min. Also, the effects of synthetic temperature on the particle’s crystallinity, morphology, and Zr/Ti ratio were studied, as characterizations and analyses showed the formation of tetragonal-rhombohedral phases. Furthermore, the analysis of the synthetic temperature on the particle’s crystallinity, morphology, and Zr/Ti ratio were similar to the commercial samples. Therefore, PZT particles were successfully synthesized by applying a one-step SSR process with a shorter duration compared to the conventional method at various conditions, EPJ Web Conf 29 (2012) 1–7.

Declarations

Author contribution statement

Darmawan Hidayat: Conceived and designed the experiments; Performed the experiments; Wrote the paper.
Mohammad Taufik: Contributed reagents, materials, analysis tools or data; Wrote the paper.
Setianto Setianto: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

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Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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