Hydrazine-nitrate combustion synthesis of BaCeO₃ preceramic powders: structure, morphology and thermophysical properties

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Received: 9 October 2021 / Accepted: 26 December 2021 / Published online: 14 January 2022

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Abstract
In the present work, preceramic nanocrystallite barium cerate (BaCeO₃) was successfully synthesized using the hydrazine-nitrate combustion method. Using carbon-free hydrazine (N₂H₄) as fuel significantly reduced the formation of carbonate by-products. Subsequent annealing of combustion products in the air atmosphere at temperatures in the range of 500–1000 °C yielded preceramic powders based on chemically and phase-pure BaCeO₃. Characterization of the as-received powders was performed by X-ray diffraction, energy-dispersive X-ray spectroscopy (EDXS), scanning electron microscopy (SEM), simultaneous thermal analysis (DTA-TGA) and adsorption-structural analysis (N₂, 77 K). Thermophysical properties of the sample annealed at 1000 °C were investigated using laser flash analysis (LFA) in the temperature interval of 1000 °C. As a result of a comprehensive study, the sequence of chemical and phase transformations that lead to the formation of barium cerate with a rhombohedral structure (Pnma, a = 6.2145 Å, b = 8.7776 Å, c = 6.2337 Å) during the thermal processing of combustion products was investigated. It was established that the average size of the obtained nanocrystals is 38 ± 3 nm and that they form micron-sized agglomerates with a specific surface area of the powder of 4.8 m²/g. It was shown that the sintered sample of barium cerate is characterized by thermal diffusivity values of 0.28 to 0.20 mm²/s and thermal conductivity values of 0.41–0.35 W/mK, depending on temperature. These results, given the impact of porosity on the sample (~40%), show very good agreement with the thermophysical characteristics of densely sintered ceramics based on BaCeO₃—a solid oxide electrolyte SOFC. Consequently, the proposed method of hydrazine-nitrate synthesis of barium cerate presents itself as a promising approach to obtaining preceramic powders and ceramics in the area of solid oxide fuel cells.

Supplementary information
The online version contains supplementary material available at https://doi.org/10.1007/s10971-021-05717-5.
Graphical Abstract

Keywords  Solutioncombustion synthesis · Hydrazine · barium cerate · SOFC · Thermal diffusivity · Thermal conductivity

Highlights
- Novel hydrazine-nitrate combustion route was proposed to synthesize BaCeO₃ nanopowder.
- Thermal treatment of combustion products leads to chemically and phase pure BaCeO₃.
- Crystallite size and its aggregation outperform those for conventional approaches.
- Thermophysical performance of BaCeO₃ powders makes it promising for SOFC ceramics.

1 Introduction

A solid oxide fuel cell (SOFC) is a device that allows converting the energy of chemical bonds into electric energy both effectively and ecologically. According to the general classification, there exist three types of SOFC materials: high-temperature (600–1000 °C), intermediate-temperature (500–750 °C), and low-temperature (<450 °C) [1]. Among them, high-temperature SOFCs are the most effective and productive, but their use is greatly restricted by problems in the exploitation of materials and devices in such temperatures. Therefore decreasing the working temperature of SOFC to the intermediate-temperature area while maintaining their high level of effectiveness and productivity is a most relevant question. It directly translates into the need to develop new solid oxide materials that would exhibit a range of chemical, thermophysical, proton-conducting, and other properties.

The main components of SOFC are electrodes (cathode and anode) and an electrolyte. The electrolyte material must satisfy several criteria such as high ionic conductivity, low electronic conductivity, chemical and physical stability under reducing and oxidizing atmospheres at the operating temperature, and ease of preparation in the form of a dense film. At present, the key electrolyte materials include YSZ, co-doped ceria, doped lanthanum silicate apatite, molybdenum oxides (ionic conductivity), BaZr- or/baCe-based mixed oxides, lanthanum tungstates [2–4]. Typically, the active components in SOFC are complex oxides with perovskite structures that, owing to their crystalline structures, exhibit high ionic conductivity [5]. Perovskite materials are used in developing every element of SOFC – as cathodes [6], anodes [7, 8], and electrolyte materials [2, 9].

Among these materials, of particular interest is barium cerate BaCeO₃. The complex oxide perovskite BaCeO₃ has fascinated researchers for over 30 years [10]. These types of proton conductors can be used as electrolyte material for intermediate-temperature (500–750 °C) solid oxide fuel cells (IT-SOFCs). There are, however, several challenges related to the process of yielding single-phase preceramic and ceramic materials based on barium cerate in those cases when it becomes necessary to dope it with other elements. One of the most basic methods of yielding barium cerate is solid-phase synthesis. The main disadvantages of this method are low homogeneity, the complexity of size control for material particles high sintering temperatures, and long sintering time [11–13]. The second group of technologies is solution-based methods including co-precipitation, combustion methods (fuel–nitrate combustion), sol-gel Pechini methods, cryochemical and hydrothermal syntheses, and others [12, 14–20]. The main advantages of these technologies are the considerably smaller size of particles of prepared powders and their high homogeneity, which favors
the decrease in synthesis temperature and duration of phase formation of multicomponent compounds, as well as preparation of gastight ceramic products at lower sintering temperatures.

Comparison of data on preceramic barium cerate powders revealed that the size of the crystallites in the case of solid-phase synthesis is 45–47 nm, and by the method of solution combustion—240 nm and 97 nm. The specific surface area is 4.46 m²/g [21], 3.4 m²/g [22] and 3 m²/g [23]. The density in solid-phase synthesis is about 95%, but the sintering process requires a lot of energy and time. By co-precipitation of oxalates, a density of 85–90% can be obtained. The solution combustion method that involves the use of various dopants can achieve a density of >95%.

The method of solution combustion allows obtaining nanopowders of simple and complex oxides with the desired crystalline structure and size parameters via both direct synthesis and synthesis involving thermal processing of amorphous combustion products [24–26]. Advantages of this method include simplicity, short duration, and the possibility to inject large amounts of dopants while preserving the single-phasedness of the products of synthesis. This method has been widely studied [27–29] and employed in synthesizing a whole range of complex oxide systems [30–34]. Components of this method include water solutions of nitrates acting as an oxidizer and organic fuel acting as a reducing agent. The main types of fuel include urea, glycine, citric acid, and others. [10]. One of the disadvantages of this method consists in the presence of carbon residue in combustion products, which results from the incomplete oxidation of carbon-filled fuel. During barium cerate synthesis, this may lead to the formation of various carbonate by-products and require raising the temperature to stabilize the annealing and obtain a single-phase product. For this reason, hydrazine (N₂H₄), known as a constituent in rocket fuel, was purchased from Lenreactiv. All chemical reagents were used without further purification.

2.2 Synthesis

Preceramic barium cerate powder was obtained via hydrazine-nitrate combustion with subsequent annealing in air at wide temperature intervals. The sintered pellet of barium cerate was obtained from preceramic powder by pressing and sintering.

2.2.1 Solution combustion synthesis

In general, solution combustion synthesis consists of some main steps such as dissolution and mixing of reagents (including complexation of metal cations), heating and partial water evaporation (can be accompanied by the formation of a gel-like substance in the case of carbon-containing fuels, but not in the case of hydrazine), autoignition and autocombustion of reaction mixture followed by the formation of solid-phase product and abundant evolution of gaseous products (Fig. 1).

Analytically pure Ba(NO₃)₂·2H₂O, Ce(NO₃)₃·6H₂O and N₂H₄·H₂O were used as initial materials without further purification. An appropriate amount of Ba(NO₃)₂ and Ce(NO₃)₃·6H₂O (hydrazine to nitrate ratio calculated as 1.2 (stoichiometry)) were separately dissolved in distilled water. Next, the required amount of N₂H₄·H₂O was added to the barium nitrate solution under magnetic stirring. Nitric acid was added to a mixture of barium nitrate and hydrazine to reach pH = 1. After Ce(NO₃)₃·6H₂O was added to the mixture Ba(NO₃)₂·2H₂O—HNO₃. The reaction equation is shown below equation:

\[
2\text{Ba(NO₃)₂} + 2\text{Ce(NO₃)₃}·6\text{H₂O} + 12\text{N₂H₄}·\text{H₂O} \rightarrow 2\text{BaCeO₃} + 17\text{N₂} + 48\text{H₂O}
\] (1)

The resulting mixture was stirred and heated on a hot-plate at 350 °C until it became a high viscous gel. With further exposure, the gel self-ignited with the formation of a powder (Fig. 1).
2.2.2 Thermal treatment of powder and pellet quenching

The obtained powder precursor was annealed in air at 500, 600, ..., 1000 °C for 4 h. Next, the sample annealed at 1000 °C was pressed in a pellet at a pressure of 25 bar and sintered at the final temperature of 1000 °C in an electric furnace in the air by heating to the final temperature at 20 °C/min and holding there for 4 h.

2.3 Characterization

The morphology of the synthesized compositions and their chemical compositions were investigated by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDXS) using a Tescan Vega 3 SBH scanning electron microscope, equipped with an Oxford INCA X-act X-ray spectral microanalysis attachment.

X-ray phase and X-ray structural analyses were conducted using a Rigaku SmartLab 3 Powder X-ray diffractometer, and phase analysis of the compositions was performed using the PDF-2 ICDD. To exclude an instrumental error, the imaging was performed using an internal standard (SiC powder). The distribution of BaCeO₃ crystallites was measured by the fundamental parameters method. Specifying the parameters of a unit cell, X-ray density and size of crystallites was performed with the help of Rigaku SmartLab 3 software and the Rietveld method by Bourevestnik, which allows measuring the parameters of the unit cell of multiphase systems with high accuracy.

Simultaneous thermogravimetric (TG) and differential thermal analysis (DTA) measurements in air atmosphere were achieved in flowing mode with heating rates of 10 °C/min up to 1000 °C using a Netzsch STA 449 F3 Jupiter instrument.

2.4 Functional properties

Temperature conductivity $\alpha(T)$ and thermal conductivity $\lambda(T)$ were measured using the NETZSCH LFA 457 MicroFlash system in the temperature range 200–1200 °C.

Thermodynamic data about temperature dependence of relative thermal capacity of BaCeO₃ was taken from work [35]. To compare the data of this article with the data of another work [36] the experimental results were extracted from the figure of the electronic version of the article using the automatic data digitization system Web Plot Digitizer.

The specific surface of the sample was determined by the low-temperature nitrogen (N₂) sorption-desorption method. The isotherm was obtained at the temperature of liquid nitrogen (77 K) using a Micromeritics ASAP 2020 analyzer.

The pycnometric density was measured by the helium pycnometry performed using a Micromeritics Ultra Pycnometer 1000, in a 1 cm³ microcell in a regime with degassing of the specimen in a helium flow with degassing time of 10 min. The bulk density was measured using a Micromeritics GeoPyc 1365.

Porosity measurements were performed by the following formula:

$$P_{\text{open}} = \left(1 - \frac{\rho_{\text{pyc}}}{\rho_{\text{XRD}}} \right) \cdot 100$$

$$P_{\text{sum}} = \left(1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{XRD}}} \right) \cdot 100$$

$$P_{\text{close}} = P_{\text{sum}} - P_{\text{open}}$$

where $P_{\text{open}}$ stands for open porosity, $P_{\text{sum}}$ - general porosity, $P_{\text{close}}$ - closed porosity, $\rho_{\text{pyc}}$ - pycnometric density, $\rho_{\text{XRD}}$ - X-ray density, and $\rho_{\text{bulk}}$ - apparent density.

3 Results and discussion

3.1 Formation mechanism of BaCeO₃

The results of the elemental analysis of powder calcined at 1000 °C are shown in Fig. 2. According to this data, the composition of the powder contains no impurity elements, and the spectral lines correspond to barium (Ba), cerium (Ce) and oxygen (O). The presence of carbon lines is linked
to the specificities of sample preparation (using carbon substrate). Quantitative analysis of this spectrum indicates that the proportions of key elements are in correspondence with the stoichiometry of barium cerate BaCeO$_3$.

To investigate the physicochemical processes occurring during the synthesis of preceramic powders of barium cerate from the products of hydrazine-nitrate combustion, a synchronous thermal analysis (DTA-TGA) of the as-prepared sample was conducted. The results are shown in Fig. 3.

According to this data, the thermogram shows 3 main regions at the following temperature intervals: R.T. — 300 °C, 500–650 °C and 650–850 °C.

In the first region of temperatures of up to 300 °C, there occurs the removal of physically and chemically linked water.

In the second temperature range from 500 to 650 °C, barium nitrate melts. The loss of mass in the first 2 regions was approximately 10%.

In the third region of temperatures, the thermal effect at 650–800 °C was caused by the decomposition of barium nitrate [37]. The endothermic effect in the region of 800–900 °C is associated with the decomposition of barium carbonate.

It is worth noting that the exothermic effect of the formation of BaCeO$_3$ does not appear in the thermogram, which results from its overlapping with the intense endothermic effects of the decomposition of nitrates. No thermal effects or changes in the mass of the sample were observed at temperatures greater than 850 °C.

Obtaining pre-ceramic barium cerate powder in these conditions was confirmed using X-ray diffraction, which allowed to investigate the particularities of phase transformations in the examined system during heat treatment of up to 1000 °C (Fig. 4).

3.2 Structural and morphological features of BaCeO$_3$

The products of direct hydrazine-nitrate synthesis (as-prepared sample) contain phases of barium nitrate (cubic, P213, JCPDS#24-0053), cerium oxide (cubic, Fm-3m, JCPDS#34-0394) and barium carbonate (orthorhombic, Pnma, JCPDS#45-1471). During thermal processing at temperatures of up to 500 °C no phase changes occur in the sample. As a consequence of thermal processing at 600 °C several changes were observed in the composition of the sample due to the decomposition of barium nitrate, which is in line with the results of thermal analysis (Fig. 2); also there occurs cerium oxide (IV) (crystallization of amorphous cerium oxide in the range of 500-600 °C) and the decomposition of barium carbonate BaCO$_3$ [15]. The composition of the sample processed thermally at 700 °C is in close correspondence with rhombic barium cerate, however, small amounts of barium carbonate and cerium oxide (IV) are also observed and their elimination occurs only at 1000 °C. Based on these results, the temperature of 1000 °C was chosen as the optimum temperature for obtaining chemically and phase-pure preceramic barium cerate powder as well as a sintered pellet based on it.

Following the results of synchronic thermal and X-ray phase analyses, a scheme of physicochemical transformations was designed, as shown in Fig. 5.
The data of the distribution of BaCeO₃ crystallites about
the 1000 °C annealed sample is shown in the inset in Fig. 5.
The crystallites have a narrow size distribution at the
average size of (D̅) 37.6 nm and width of the distribution

Fig. 4 XRD patterns of as-prepared and annealed BaCeO₃-based samples

Fig. 5 Transformation mechanism of BaCeO₃ preceramic powder via heat treatment of hydrazine-nitrate combustion products

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Fig. 6 Rietveld refinement results of BaCeO₃-based samples annealed at 1000 °C. Inset shows the crystallite size distribution of BaCeO₃
of (FWHM) 21.8 nm. The obtained size values of crystals are noticeably lower than those of barium cerate synthesized by a solid-state method—45–47 nm replacement by [38], 50 nm [20], or citrate methods—240 nm [21],—97 nm replacement by [39].

Table 1 shows the parameters of a unit cell and the average size of BaCeO₃ crystallites refined by the Rietveld method and annealed at the temperatures of 700, 800, 900, and 1000 °C from the products of hydrazine-nitrate combustion.

The refined parameters correspond to known crystallographic data of barium cerate with rhombic structure \(a = 6.2517 \text{ Å}, b = 8.7906 \text{ Å} \) and \(c = 6.2271 \text{ Å}, V = 342.218 \text{ Å}^3, Pmna space group\) replacement by [40]. On the diffractogram of the BaCeO₃ annealed at 1000 °C reflexes of other phases were not detected. Increasing temperature causes unsystematic changes in the parameters of the unit cell - its size slightly increases with the rise of temperature, which is likely the result of the growing refinement of the crystallite structure of obtained barium cerate nanocrystals.

Morphological characteristics of the as-prepared samples annealed at 700 °C and 1000 °C were investigated through the scanning electron microscopy the results of which are presented in Fig. 7.

The above results indicate that the BaCeO₃-based sample annealed at 1000 °C was strongly agglomerated into micron- and submicron-sized structures.

The degree of agglomeration changes in as-prepared samples depending on the temperature of processing: with the rise of temperature a tendency for decreasing the size of

| Table 1 Rietveld refinement results of BaCeO₃-based samples |
|-----------------|-----------------|-----------------|-----------------|
| Temperature     | 700             | 800             | 900             | 1000            |
| a, Å            | 6.2129(19)      | 6.211(6)        | 6.2351(5)       | 6.2145(4)       |
| b, Å            | 8.8016(9)       | 8.8060(12)      | 8.7741(7)       | 8.7776(6)       |
| c, Å            | 6.209(2)        | 6.212(6)        | 6.2110(5)       | 6.2337(5)       |
| V, Å³           | 339.53          | 339.76          | 339.79          | 340.04          |
| \(\alpha = \beta = \gamma\), ° | 90              | 90              | 90              | 90              |
| \(\langle D\rangle\), nm | 35.4 ± 2.0       | 33.4 ± 2.0       | 36.9 ± 2.0       | 37.6 ± 2.0       |
| \(R_{wp}\), % | 11.52           | 10.48           | 11.77           | 10.35           |
| \(R_p\), %     | 8.64            | 8.01            | 8.57            | 8.03            |

Fig. 7 SEM images of as-prepared (a–c) and annealed at 700 °C (d–f) and at 1000 °C (g–i) BaCeO₃-based samples
agglomerates is observed, which is connected with the intense decomposition of intermediate products in the process of obtaining barium cerate. (Figs. 3 and 4).

In the original sample, the sizes of agglomerates vary in the range of 20–50 μm; in the sample annealed at 700 °C, the sizes of agglomerates do not exceed 40 μm. The sample annealed at 1000 °C, according to the XRD data, is represented by a single phase-orthorhombic barium cerate and is comprised predominantly of agglomerates not exceeding the size of 20 μm. All samples are characterized by the narrow size distribution of agglomerates.

The sample annealed at 1000 °C, following the data of quantitative and qualitative analysis, consists of barium cerate nanoparticles.

To analyze the specific surface and porosity structures of the annealed samples adsorption-structural analysis was performed. Figure 8 shows adsorption isotherms of samples annealed at temperatures from 700 °C to 1000 °C, as well as the values of corresponding specific surface areas (see inset in Fig. 8).

According to the IUPAC classification, the isotherms presented above belong to type II, and the observed hysteresis loops—to type H1. The porosity of the obtained powders varies from 11.2% in the sample synthesized at 700 °C to 4.7% in the sample synthesized at 1000 °C. The values of specific surface structures range from 4.1 m²/g to 5.8 m²/g, depending on the temperature of annealing.

The values of specific surface structure are close to those obtained in other works: 4.46 m²/g [21], 3.4 m²/g [22], 3 m²/g [23], 3 m²/g [41]. However, some works [42] reported slightly higher values of specific surface structure—6–8 m²/g.

### 3.3 Thermophysical properties

Sample annealed at 1000 °C was selected as the basis for obtaining a sintered pellet due to its high chemical and phase uniformity. To estimate the potential of this sample as a preceramic powder, its density characteristics were analyzed at the formed pellet. The results of the analysis are shown in Table 2.

According to the data presented in work [16], the density of barium cerate in the case of solid-state synthesis does not exceed 70%, while obtaining it via the nitrate method results in a density of about 80%—conditional upon prolonged annealing. Finally, using the method of coprecipitation of oxalates with subsequent annealing results in values of 85–90%. In work [42] the density of barium cerate with various dopants is characterized by values of >95%. Consequently, the nanocrystalline BaCeO₃ obtained in this work has density and porosity characteristics that correspond to known preceramic powders based on barium cerate, which lends to the conclusion that it can be used in this capacity.

Figure 9 shows the results of measurements of thermal diffusivity and thermal conductivity of the pellet sintered at 1000 °C.

At room temperature, the value of thermal diffusivity is 0.28 mm²/s, and with the rise of temperature, it decreases to 0.20 mm²/s at 1100 K. The thermal conductivity of the sintered material varies from 0.41 to 0.35 W/m·K in the same temperature interval. The temperature dependence of the thermal conductivities of BaCeO₃ shows typical phonon conduction characteristics [36], the thermal conductivities decrease with increasing temperature. Ba and Sr series perovskite compounds follow the general rule for predicting the magnitude of thermal conductivity, i.e., a material with high formula weight and with low Debye temperature exhibits low thermal conduction [43]. The data on the thermal conductivity of barium cerate in the work [36] varies from 1.33 to 1.70 W/m·K for nonporous material. The thermal conductivity values for the sintered pellet obtained in this work are noticeably lower than those for dense ceramics but the temperature dependence process is analogical. Considering that the porosity of the obtained

| Parameter | Value |
|-----------|-------|
| ρₓ₋ₓ🦆, g/cm³ | 6.36 (100%) |
| ρpyc, g/cm³ | 5.54 (87%) |
| ρbulk, g/cm³ | 3.80 (69%) |
| P_open, % | 12.90 |
| P_closed, % | 27.34 |
| P_total, % | 40.25 |

Table 2 Density characteristics of BaCeO₃-based sintered pellet

Fig. 8 N₂ adsorption-desorption isotherms of BaCeO₃-based samples annealed at 700, 800, 900, and 1000 °C. Inset shows corresponding specific surface areas.
hydrazine-nitrate combustion allows to obtain BaCeO₃ with formation of carbonate impurities. It was concluded that combustion products, which significantly lowers the formation of carbonate impurities. It was demonstrated that using hydrazine as combustion. It was demonstrated that using hydrazine as combustion products, which significantly lowers the formation of carbonate impurities.

In this work, chemically and phase-pure preceramic powder of barium cerate was obtained using hydrazine-nitrate combustion. It was demonstrated that using hydrazine as combustion products, which significantly lowers the formation of carbonate impurities. It was demonstrated that using hydrazine as combustion products, which significantly lowers the formation of carbonate impurities. It was demonstrated that using hydrazine as combustion products, which significantly lowers the formation of carbonate impurities.

4 Conclusions

In this work, chemically and phase-pure preceramic powder of barium cerate was obtained using hydrazine-nitrate combustion. It was demonstrated that using hydrazine as fuel has the advantage of the lack of carbon residue in the combustion products, which significantly lowers the formation of carbonate impurities. It was concluded that hydrazine-nitrate combustion allows to obtain BaCeO₃ with crystallite sizes smaller than in the case of solid-state and nitrate synthesis—and with narrower size distribution. It was demonstrated that the thermophysical properties of the sintered sample based on the preceramic powder is analogous to the thermophysical properties of densely sintered ceramics based on BaCeO₃, which allows expecting successfull application of this method in obtaining ceramic materials and products. Since the solution combustion method (including hydrazine-nitrate combustion) allows diverse and controlled doping of preceramic powders with d- and f-elements, further development of this method will provide the means to obtain new ceramic materials based on barium cerate for SOFC components and devices.

Acknowledgements The studies were performed using the analytical equipment of the Engineering Center of the St. Petersburg State Institute of Technology. We are especially grateful to M.I. Chebanenko, K.D. Martinson, D.P. Danilovich, and Motayo E.S. for help in characterizing the materials and D.K. Przybyla for help with translating the text of the article.

Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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