Calcium Titanate Ceramics Obtained by Combustion Synthesis and Two-Step Sintering

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Abstract:
Calcium titanate powder was successfully prepared from duck eggshell and anatase titanium dioxide with a magnesium inductant via combustion synthesis in argon. As-combusted products were leached with diluted HCl. In XRD analysis, as-leached powders exhibited a major phase of CaTiO₃ with a perovskite structure. The particle size, observed by SEM, was approximately 240 nm. As-leached powders were densified by single-step (SST) and two-step sintering (TSS) to produce calcium titanate ceramics. The first step of all TSS conditions was fixed at 1350 °C and holding times (t₁) at this temperature were varied up to 120 min. Calcium titanate ceramic obtained from holding for 120 min had a grain size of 2.18 µm, relative density of 86.68% and a dielectric constant of 92. Two-step sintered ceramic had the highest density (95.73%) and best dielectric properties (dielectric constant = 110, dielectric loss = 0.02) when the holding temperature (T₂) was 1250 °C. Calcium titanate ceramics processed by two-step sintering had denser microstructures and higher dielectric constants than single-step sintered ceramic due to grain boundary diffusion and the simplicity of relaxation polarization.

Keywords: Calcium titanate; Combustion synthesis; Two-step sintering.

1. Introduction

Calcium titanate (CaTiO₃) with a perovskite structure is an advanced ceramic material often exploited in the fabrication of high frequency electronic devices[1-3]. It was synthesized by the extended heating of titanium dioxide (TiO₂) with one of the calcium compounds powders: calcium oxide (CaO), calcium carbonate (CaCO₃), calcium nitrate hydrate (Ca(NO₃)₂·4H₂O), or calcium chloride dehydrate (CaCl₂·H₂O) at a high temperature. [4-10]. Mechano-chemical synthesis of CaTiO₃ has been carried out with various mixtures of CaCO₃, calcium hydroxide (Ca(OH)₂), CaO and TiO₂ under an extended period of mechanical activation [5, 11-15]. Calcium titanate has also been produced using soft chemistry such as sol-gel, hydrothermal and co-precipitation methods [16-20]. However, solid-state and mechano-chemical synthesis methods consume a lot of energy to produce the high temperature necessary and processing times of several hours. Soft chemistry techniques meanwhile require complex starting materials and processes.

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Combustion synthesis is a method currently used to synthesize advanced or engineering ceramics. The advantage of this method is its simplicity, energy-efficiency and short processing time [21]. Moreover, except for certain commercial grade chemical components, the reactants used in combustion synthesis can be sourced from waste materials from activities such as agriculture, food production and from mineral tailings. Interestingly, calcium titanate was recently prepared by combustion synthesis using reactants from waste materials [22-24]. Self-propagating high-temperature synthesis (SHS) is one of combustion reaction modes mostly found to synthesize numerous advanced materials at temperatures above 4000 K. At room temperature, only small external ignition energy is required to initiate the reaction and then, the combustion generated extremely exothermic heat for the reaction to be self-sustaining. The fast combustion wave traveled rapidly through the reactant compact in a second of time. After SHS reaction, fast cooling rate permitted the formation of solid materials with very finegrains and metastable compositions [25].

Monolithic ceramics can be densified by sintering, which is a technique that influences the microstructures and properties of materials. The general concept of sintering is an interface elimination process by atomic transport that reduces high surface energy. In this way, the total free energy of the system was reduced [26]. Two-step sintering (TSS) is a simple and economical approach to achieve high density ceramics without applying external pressure. The process involved two steps. The sample was first heated from room temperature to a high temperature (T_1) which was reduced at a high cooling rate to a lower temperature (T_2) and kept at T_2, limiting grain growth, until maximum density was achieved [27]. Previous studies reported that two-step sintering produced materials with higher relative density and smaller grain size than so-called conventional or single-step sintering (SST), which applies a constant heating rate cycle with a holding time at the sintering temperature [28-31].

In this work, calcium titanate powder was prepared by combustion synthesis in an argon atmosphere from reactant powders of duck eggshells and anatase titanium dioxide using magnesium as an inductant. The as-prepared ceramic powder was characterized in terms of morphology and phase composition. Subsequently, as-prepared calcium titanate powder was processed by single- and two-step sintering to produce dense calcium titanate ceramics. The microstructures, densities and dielectric properties of the calcium titanate ceramics were compared and analyzed as-sintered.

2. Materials and Experimental Procedures
2.1 Materials and chemicals

| Compound | Content (wt%) |
|----------|---------------|
| Al_2O_3  | 0.04          |
| CaO      | 53.13         |
| Fe_2O_3  | 0.03          |
| K_2O     | 0.02          |
| MgO      | 0.13          |
| Na_2O    | 0.08          |
| P_2O_5   | 0.31          |
| SiO_2    | 0.18          |
| SO_3     | 0.44          |
| SrO      | 0.03          |

XRD analysis in our previous study [23] confirmed that the as-received duck eggshell was composed entirely of calcium carbonate. XRF data of the duck eggshell after heating
(Table I) showed a major compound of calcium oxide (CaO) from the decomposition of CaCO₃ to form CaO and CO₂ gas. Raw powder reactants were anatase titanium dioxide (A-TiO₂, 99.5%) from Degussa AG and magnesium (Mg, 99%) from Riedel-de Haen. Aluminium powder (Al, 93%) from Himedia Laboratories and iron (III) oxide (Fe₂O₃, 97%) powder from Riedel-de Haen were combined to create a thermite ignitor that provided the extreme exothermic heat in the reaction. Hydrochloric acid (HCl, conc. 37%), used in the leaching process, was purchased from Merck-KGaA and polyvinyl alcohol (PVA), used as a binder in the sintering process, was from Metech.

2.2 Preparation of calcium titanate powders by combustion synthesis

Cleaned and dried duck eggshells were ground with a mortar into a friable powder and sieved through a 325 mesh to give particles less than 45 µm. Powdered duck eggshell and A-TiO₂ were high-energy milled with Si₃N₄ balls in a jar (Fritsch GMBH, Pulverisette 6, Germany) for 30 min at 400 rpm. This procedure was adapted from reference works [23, 24]. The high-energy milled powder mixtures were then mixed with magnesium powder by ball milling for 120 min using zirconia (ZrO₂) balls and a nylon vial. Using a hydraulic press (HuatSeng, 1939-15T, Thailand), 20 g of the obtained reactant powder mixture were uniaxially pressed at 0.05 MPa into a pre-hardened tooled steel mold to form a cylindrical compact of 25.4 mm in diameter, with a green body density of 50-60% of the theoretical density. A compact of 5 g of thermite from a mixture of iron (III) oxide and aluminium powders with a molar ratio of 1 : 2 was placed on top of the green reactant compact. The reaction mechanism between Fe₂O₃ and Al was described in a previous study [21]. The green reactant together with the ignitor compact (Fig. 1(a)) was placed on a movable refractory plate and loaded into an SHS reactor. The chamber of the SHS reactor was evacuated with a vacuum pressure of 70 mmHg and filled with argon gas to a pressure of 0.5 MPa.

![Fig. 1](image)

Digital photographs of a typical (a) green reactant pellet with an ignitor, and (b) as-combusted product.

This procedure was repeated at least twice to ensure an inert environment during the propagation of the combustion reaction. Components of the SHS reactor included a controlled atmospheric reaction chamber and a tungsten filament connected to a power source through a current controller, which provided the energy required for the ignition of the reaction. The schematic details of the experimental setup were presented in our previous study [21]. The heat from the tungsten filament ignited the Fe₂O₃ and Al, which melted homogeneously. This melted compound transferred its high heat to the initial part of the green reactant compact underneath it and a combustion wave immediately moved through the compact in a self-propagating mode which reached completion at the bottom of the reactant compact in about 8 - 10 sec. The layered and porous-as-combusted product (Fig. 1(b)) was allowed to cool to room temperature. The by-product of Fe + Al₂O₃ obtained from the thermite reaction (Fe₂O₃ + Al) could be broken away from the as-combusted product by hand. However, there was no
penetration of melted Fe₂O₃ + Al into the combustion product during heat transfer and the solid compound could be collected intact after it had cooled down.

The as-combusted product (Fig. 1(b)) was mechanically pestled into friable powder in a Al₂O₃ mortar. To eliminate by-products of magnesium oxide (MgO), as-combusted powders were leached using a 2 M HCl solution for 120 min under moderate magnetic stirring. The ratio of powdered product to leaching agent was 10 g : 100 mL. After leaching, the as-leached sediment was filtered through no. 41 filter paper coupled with a pump inlet and washed with deionized (DI) water several times in order to adjust pH to 7.0. The as-leached calcium titanate powder was dried at 100 °C for 24 hr.

2.3 Densification of calcium titanate ceramic by different sintering conditions

Using a hydraulic press (HuatSeng, 1939-15T, Thailand) at a pressure of 0.05 MPa, 1.2 g of as-prepared calcium titanate powder mixed with 3 % PVA solution binder were consolidated to form a cylindrical green pellet 15 mm in diameter. Compacted pellets were sintered by SST and TSS in an air atmosphere in a muffle furnace (Lenton, UAF16, USA). To avoid thermal mismatch stress due to changes in temperature, the green pellets were placed in an alumina crucible and covered with alumina powder.

Following our previous research [23], conventional, single-step sintering (SST) was performed at a heating rate of 10 °C/min with a holding time of 180 min at 1350 °C. In the case of two-step sintering, green pellets were first heated to 500 °C at a heating rate of 5 °C/min, and held at this temperature for 60 min to completely remove the organic binder. The temperature was then raised to T₁ of 1350 °C at a heating rate of 10 °C/min and held for a holding time (t₁) of 30, 60 or 120 min. The products of these sintering conditions were labeled TSS1, TSS2 and TSS3, respectively. The second holding temperature, T₂, was set at 1000 °C, reached at a cooling rate of 20 °C/min, and the holding time (t₂) at T₂ was 180 min in every case. However, one specimen (TSS4), for comparison with the other TSS samples, was sintered at T₂ of 1250 °C and also soaked for 180 min.

2.4 Characterizations

The morphologies of the as-combusted powders, as-leached powders and as-sintered calcium titanate ceramics were characterized by scanning electron microscope (SEM, Quanta, FEI 400, USA) and elemental microanalysis using energy dispersive X-ray spectrometry (EDS, Oxford, ISIS 300, USA). The average grain size of as-sintered calcium titanate ceramics, obtained from SEM, was calculated by the mean linear intercept method using the ImageJ program version 1.52v. To identify phase compositions, X-ray diffractometry (XRD, PANalytical, Empyrean, the Netherlands) was carried out at 40 kV and 30 mA using CuKα radiation (0.15406 nm).

2.5 Density measurement

The sintered density and apparent porosity of the calcium titanate ceramics were measured according to Archimedes’ principal, and were calculated using equation (1) [23],

\[ \text{D}_{\text{sintered}} = \frac{W_0}{W_2 - W_1} \times \rho_{\text{water}} \quad (1) \]

where \( D_{\text{sintered}} \) is the sintered density, \( P \) is the apparent density, \( W_0, W_1, W_2 \) are dry weight, weight in water of the water-saturated specimen, and weight in air of the water-saturated
specimen, respectively, obtained from a precision electronic balance set (Sartorius, BSA series, Thailand). In addition, $\rho_{\text{water}}$ is the density of water (1 g/cm$^3$).

### 2.6 Dielectric properties

Sintered calcium titanate ceramic pellets were abraded to a thickness of about 1 mm using silicon carbide grinding paper on both flat surfaces. Disc capacitors were created from the pellets by painting silver (Ag) paste electrodes (MG chemical, USA) to both polished surfaces which were dried at 50 °C for 12 h. Room temperature capacitance was measured using an LCR meter (GW INSTEK, LCR-821, USA) within a frequency range from 0 to 200 kHz. The dielectric constant was calculated according to equation (2) [10],

$$\varepsilon_r = \frac{Cd}{\varepsilon_0 A}$$  \hspace{1cm} (2)

where $\varepsilon_r$ is the dielectric constant, $C$ is the equivalent parallel capacitance obtained from the measurement data, $d$ is the thickness of the sintered ceramics, $A$ is the combined surface electrode area of the electrode discs, and $\varepsilon_0$ is the permittivity of vacuum ($8.85 \times 10^{-12}$ F/m). The dielectric loss obtained from the value of the dissipation factor was calculated from equation (3) [32],

$$\varepsilon_i = D \varepsilon_r$$  \hspace{1cm} (3)

where $\varepsilon_i$ is the dielectric loss, $D$ is the dissipation factor obtained from measurement data and $\varepsilon_r$ is obtained from Equation (2).

### 3. Results and Discussion

#### 3.1 Characteristics of calcium titanate powder obtained from combustion synthesis

![Fig. 2. XRD patterns of the product synthesized by combustion synthesis in argon: (a) the as-combusted product and (b) the as-leached powder.](image)

The XRD pattern of the as-combusted product (Fig. 2(a)) showed phases of CaTiO$_3$ (ICDD no. 01-074-8732), unreacted anatase TiO$_2$ (ICDD no. 03-065-5714) and a by-product
of MgO (ICDD no. 01-089-4248). However, in the pattern of the as-leached powder (Fig. 2(b)), the MgO by-product was completely eliminated and a major CaTiO$_3$ phase was present along with trace anatase TiO$_2$.

Fig. 3. SEM micrographs of the as-combusted (a) and as-leached (b) product particles.

In an SEM image of the as-combusted product (Fig. 3(a)), agglomerated particles (about 5 µm) presented distinct phases. This morphology was due to the covering of MgO on the main product particles. On the other hand, after leaching out MgO, the as-leached powder (Fig. 3(b)) showed rounded, fine particles of about 240 nm. The approximately round shape of the product particles achieved in this work was consistent with the morphology of calcium titanate powders produced in previous works [3, 4, 23]. However the particles produced in the present work were smaller in size.

3.2 Characteristics and properties of calcium titanate ceramics fabricated by two-step sintering

Fig. 4. XRD patterns of calcium titanate ceramics sintered in different conditions (a) SST, (b) TSS1, (c) TSS2, (d) TSS3 and (e) TSS4.

The as-leached powder was used to produce calcium titanate ceramics by SST in a single condition and TSS in varied conditions. XRD patterns revealed the phase formation of
calcium titanate ceramics prepared by all the processes. All sintering conditions exhibited phases of CaTiO$_3$ and trace anatase TiO$_2$ (Fig. 4(a)-(e)). This result confirmed that no phase change occurred during any of the sintering processes. These results were consistent with the elemental analysis data (Fig. 5), which displayed a spectrum of Ca, Ti and O.

![Fig. 5. SEM-EDS microanalysis of a typical sintered calcium titanate ceramic.](image)

| Sintering condition | Sintered density (g/cm$^3$) | Relative density (%) | Average grain size ($\mu$m) |
|--------------------|-----------------------------|----------------------|-----------------------------|
| SST                | 3.60                        | 90.45                | 2.23                        |
| TSS1 (T$_1$=1350, t$_1$=180) | 3.25                        | 81.66                | 1.77                        |
| TSS2 (T$_1$=1350, t$_1$=30, T$_2$=1000,t$_2$=180) | 3.27                        | 82.16                | 2.17                        |
| TSS3 (T$_1$=1350, t$_1$=60, T$_2$=1000,t$_2$=180) | 3.45                        | 86.68                | 2.18                        |
| TSS4 (T$_1$=1350, t$_1$=120, T$_2$=1250,t$_2$=180) | 3.81                        | 95.73                | 2.70                        |

The microstructures of calcium titanate ceramics produced at different sintering conditions were observed by SEM. Images of top and fracture surfaces of samples of TSS1, TSS2 and TSS3 ceramics (Fig. 6(b)-(d)) showed that increasing the holding time (t$_1$) at T$_1$ resulted in larger average grain sizes, which increased from 1.77 to 2.18 $\mu$m (Table II). The results were attributed to both grain boundary diffusion and grain boundary migration in these cases [33]. Furthermore, there were fewer pores in the fracture surfaces of samples TSS2, TSS2 and TSS3 than in the SST sample. The relative density of the products of TSS increased from 81.66% (TSS1) to 86.68% (TSS3) (Table II). However, by comparison with the SST sample, the grain size obtained from two-step sintering was typically smaller. Othman et.al [34] proposed that grain growth was suppressed by heating to a high T$_1$ and then grain boundary migration was restrained by cooling to a lower T$_2$. 
Fig. 6. SEM images show the microstructures of top and fracture surfaces of calcium titanate ceramics sintered in different conditions: (a) SST, (b) TSS1, (c) TSS2, (d) TSS3 and (e) TSS4.
If we consider sample TSS4, T₂ was 1250 °C and a relative density of 95.73% was achieved with an average grain size of 2.70 µm. These were the highest values obtained for these parameters in this study. If the driving force of grain boundary diffusion was adequately maintained, further increases in density would occur whereas, if grain boundary energy was reduced, grain boundary migration was not sufficiently driven and further densification was achieved [33, 35, 36]. Also, the larger grains in TSS4 increased density by closing up the pores present between the smaller grains in other samples [37].

Fig. 7. Dielectric constant (a) and dielectric loss (b) of calcium titanate ceramics from different sintering conditions as a function of frequency.

The room temperature dielectric constant of all the sintered calcium titanate ceramics was determined across a range of frequencies from 0 to 200 kHz. At the highest frequency, the TSS1 sample showed the lowest dielectric constant (~70). This behavior was due to the low density of the sample’s microstructure. For comparison, the dielectric constants of TSS2 and TSS3 at 200 kHz were 86 and 92. The increment of the dielectric constant was mainly due to the greater densification of ceramics sintered at longer holding times (t₁) (Table II). However, the dielectric constants of TSS1, TSS2 and TSS3 samples were lower than the εᵣ of SST (~96). The dielectric constant of the TSS4 sample, produced at T₂ of 1250 °C, was the highest at 110. The dielectric constant of the TSS4 sample at 200 kHz was greater than the εᵣ (~83) of a similar ceramic reported in other researches [23, 38]. The improved dielectric
properties of the sintered ceramics were attributed to increased densification and reductions in porosity [39] (see Fig. 6(a-e)). These results were consistent with previous reports [22, 31, 38, 40] that the charge polarization (or relaxation polarization) of simply processed structurally dense materials with fewer pores produced a larger dielectric constant. In addition, at 200 kHz, the dielectric loss of calcium titanate ceramics sintered in different sintering conditions was low, ranging from 0.02 to 0.06 (Fig. 7(b)).

4. Conclusion

Calcium titanate powder was successfully synthesized by combustion synthesis under 0.5 MPa of argon from reactants of duck eggshell/anatase titanium dioxide (A-TiO$_2$) using magnesium as an inductant. After leaching with HCl, the powder comprised a CaTiO$_3$ major phase and a trace amount of A-TiO$_2$. The crystallized calcium titanate powders consisted of rounded, fine particles with an average size of approximately 240 nm. The grain size, density and dielectric properties of the ceramics were influenced by the sintering method (single or two-step) and the conditions of two-step sintering. Increased first step holding time ($t_1$) from 30 to 120 min, produced two-step sintered ceramics of greater grain size, density and dielectric constant. When first step holding time was 30 min, grain size, density and dielectric constant were 1.77 $\mu$m, 81.66% and ~70, respectively: at 60 min, they were 2.17 $\mu$m, 82.16% and 86; and at 120 min, 2.18 $\mu$m, 86.68% and 92. Of all the samples studied, a two-step sintered sample, soaked at a higher $T_2$ temperature than the other two-step sintered ceramics, showed the highest dielectric constant (110) and the lowest dielectric loss (0.02). This behavior was a result of the material’s high density (95.73%), which facilitated polarization.

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5. References

1. O. Jongprateep, N. Sato, R. Techapiesancharoenkij, K. Surawathanawises, Adv. Mater. Sci. Eng., 2019 (2019) 1.
2. P. Ctober, J. Kotlan, Z. Pala, J. Sedlacek, Z. Hajkova, T. M. Grygar, Mater. Res. Bull., 72 (2015) 123.
3. P. Julphunthong, B. Phengraek, A. Laowanidwatana, T. Bongkarn, Integr. Ferroelectr., 150 (2014) 107.
4. M. Keshavarzi, H. Rahmani, A. Nemati, M. Hashemi, Appl. Phys. A-Mater., 124:145 (2015) 1.
5. G. Mi, F. Sailo, S. Suzuki, Y. Waseda, Powder Technol., 97 (1998) 178.
6. L. Wang, J. Li, M. Feng, L. Min, J. Yang, S. Yu, Y. Zhang, X. Hu, Z. Yang, Biosens.Bioelectron., 96 (2017) 220.
7. G. Gralik, C. Zanelli, F. Raupp-Pereira, M. Dondi, J. A. Labrincha, D. Hotza, 21 Congresso Brasileiro de Engenharia e Ciencia dos Materiais, Armação de Buzios, RJ, Brazil, 2014.
8. M. Sh. Khali, F. F. Hammad, Egyptian J. Sol., 25 (2002) 175.
9. G. Gralik, A. Thomsen, C. Moraes, F. Raupp-Pereira, D. Hotza, Process. Appl. Ceram., 8 (2014) 53.
10. S. J. Mousavi, Dig. J. Nanomater. Bios., 9 (2014) 1059.
11. K. Wieczorek-Ciurowa, P. Dulian, A. Nosal, J. Domagała, J. Therm. Anal. Calorim., 101 (2010) 471.
12. K. H. Park, H. G. Kim, J. Korean Phys. Soc., 56 (2010) 648.
13. S. Palaniandy, N. H. Jamil, J. Alloys Compd., 476 (2009) 894.
14. V. Berbenni, A. Marini, J. Mater. Sci., 39 (2004) 5279.
15. G. Mi, Y. Murakami, D. Shindo, F. Saito, Powder Technol., 105 (1999) 162.
16. P. K. Mallik, G. Biswal, S. C. Patnaik, S. K. Senapati, IOP Conf. Ser. Mater. Sci. Eng., 75 (2015) 1.
17. S. Holliday, A. Stanishevsky, Surf. Coat. Technol., 188-189 (2004) 741.
18. D. Wang, Z. Guo, Y. Chen, J. Hao, W. Liu, Inorg. Chem., 46 (2007) 7707.
19. W. Dong, G. Zhao, Q. Bao, X. Gu, Mater. Sci-Medzg., 21 (2015) 583.
20. S. K. Durrani, Y. Khan, N. Ahmed, M. Ahmad, M. A. Hussain, J. Iran. Chem. Soc., 8 (2011) 562.
21. T. Chanadee, Met. Mater. Int., 23 (2017) 1188.
22. A. Maddu, L. Permatasari, A. Arif, J. Ceram. Process Res., 18 (2017) 146.
23. S. Cherdrohm, T. Rattanapan, T. Chanadee, Adv. Mater. Sci. Eng., 2019 (2019) 1.
24. S. Rongsawat, W. Bunma, T. Chanadee, Mater. Sci. Forum., 982 (2020) 20.
25. T. Chanadee, S. Singsarothai, Combust. Explos. Shock Waves., 55 (2019) 97.
26. D. Gouve, R. H R. Castro, Appl. Surf. Sci., 217 (2003) 194.
27. X. Lei, B. Xu, B. Yang, B. Xu, X. Guo, J. Alloys Compd., 690 (2017) 916.
28. K. Maca, V. Pouchly, P. Zalud, J. Eur. Ceram. Soc., 30 (2010) 583.
29. N. J. Löh, L. Simião, C. A. Faller, A. De Noni, O. R. K. Montedo, Ceram. Int., 42 (2016) 12556.
30. G. S. Kathait, N. S. Panwar, P. Thapliyal, D. Biswas, V. Rohilla, S. Singh, Sci. Sintering., 51 (2019) 421.
31. G. S. Kathait, M. K. Panda, N. S. Panwar, Sci. Sintering., 52 (2020) 97.
32. K. Omar, M. D. Johan Ooi, M. M. Hassin, Mod. Appl. Sci., 3 (2009) 110.
33. W. Meng, R. Zuo, S. Su, X. Wang, L. Li, J. Mater. Sci.: Mater. Electron., 22 (2011) 1841.
34. N. W. Othman, A. Ramli, N. Osman, S. S. C Abdullah, M. E. A. Rahman, Mater. Sci. Forum., 819 (2015) 129.
35. W. min, A. xing, Z. jun, Comput. Mater. Sci., 90 (2014) 16.
36. U. Sutharsini, M. Thanhaichelvan, R. Singh, Two-Step Sintering of Ceramics, in: Shishkovsky I. (Eds.), Sintering of Functional Materials, IntechOpen, London, 2018, p. 3-21.
37. J. Wu, Y. Wang, Dalton Trans., 43 (2014) 12836.
38. X. Lu, Q. Li, D. Yang, J. Electroceramics., 14 (2005) 59.
39. Z. Tian, X. Wang, S. Lee, K. H. Hur, L. Li, J. Am. Ceram. Soc., 94 (2011) 1119.
40. T. Luo, B. Li, Q. Zhao, J. Zhou, Int. J. Antennas Propag., 2015 (2015) 1.
добијена двостепеним синтеровањем имала је гушћу микроструктуру и већу диелектричну константу од керамике добијене једностепеним синтеровањем услед дифузије на граници зрна и релаксационе поларизације.

Кључне речи: калцијум титанат; синтеза сагоревањем; двостепено синтеровање.

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