

TWO STAGE CaTiO₃ SYNTHESIS IN DIFFERENT CRYSTALLINITY TiO₂ MIXTURES

Ričardas Kleinauskas¹, Anatolijus Eisinas¹

¹Kaunas University of Technology, Faculty of Chemical Technology, Kaunas, Lithuania
ricardas.kleinauskas@ktu.lt

Due to high caustic resistance, high thermal stability and low production cost, CaTiO₃-based materials are being actively developed for nuclear waste treatment [1], dielectric [2] and photocatalytic applications [3]. A growing number of research groups are focusing on optimizing the production of CaTiO₃ by modifying raw materials or combining various synthesis techniques. This work aims to develop a two stage calcium titanate synthesis using titanium dioxide with different crystallinity via hydrothermal and solid-state methods.

In this work, amorphous TiO₂ was prepared in accordance with our earlier experiments [4]. Samples were prepared by calcinating amorphous TiO₂ in 25, 130, 330 and 530 °C and mixing it and CaO equal to molar ratio of 1. Hydrothermal treatment was performed under saturated steam pressure of 15 bars at a temperature of 200 °C for 1-96 hours. The samples were filtered, dried and sieved (<80 μm) then sintered at a temperature of 900 °C for 1 hour in an air atmosphere. Synthesis products were characterized by XRD, FT-IR and STA instrumental methods.

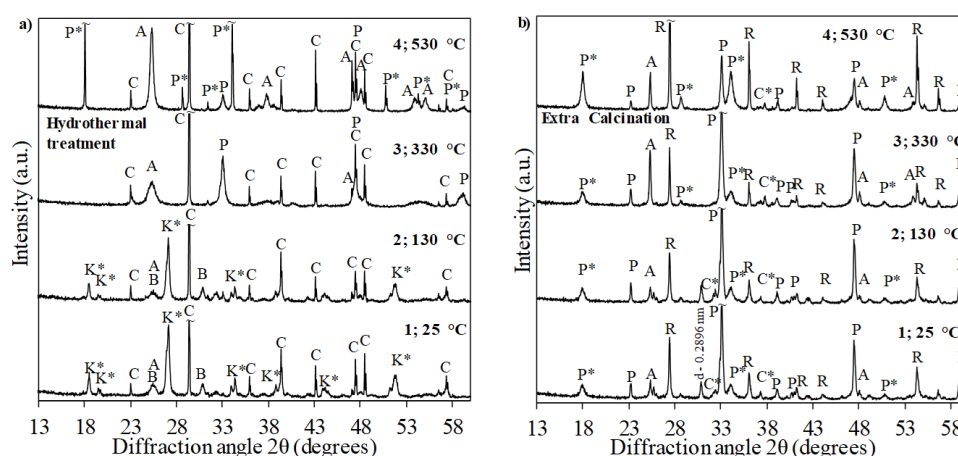


Fig. 1. XRD patterns of the synthesis products (CaO/TiO₂=1) after 48 h of hydrothermal treatment at 200 °C (a) and extra calcination at 900 °C (b) when the amorphous TiO₂ was calcinated, °C: 1 - 25; 2 - 130; 3 - 330; 4 - 530. Indexes: C - CaCO₃; P - CaTiO₃; P* - Ca(OH)₂; A - anatase; B - brookite; K* - kassite CaTi₂O₅(OH)₂

It was determined that amorphous TiO₂ treated at low temperatures (25-130 °C) enhances the formation of kassite during hydrothermal treatment (I stage). (**Fig. 1 a; 1, 2**). Meanwhile, TiO₂ calcinated at higher temperatures (330-530 °C) promotes formation of CaTiO₃ (**Fig. 1 a; 3, 4**). The most intense CaTiO₃ diffraction maximum was identified in the samples with TiO₂ calcinated at 330 °C (**Fig. 1. a; 3**). When hydrothermal synthesis products were calcinated at 900 °C (II stage), the highest CaTiO₃ diffraction peaks were identified in samples with lower TiO₂ crystallinity (**Fig. 1 b; 1, 2**). By comparison, more intense diffraction peaks attributed to rutile and portlandite suggest a limited reaction between CaO and crystalline TiO₂ (**Fig. 1 b; 3, 4**).

The results suggest that the crystallinity of TiO₂ has an influence on the formation of CaTiO₂ during hydrothermal and solid-state synthesis.

- [1] N. Bensemme et al., "Radiation stability of Nd-doped perovskite ceramics for trivalent actinide waste (An+3) storage," Nuclear Instruments and Methods in Physics Research Section B Beam Interactions With Materials and Atoms, vol. 550, p. 165306, Mar. 2024.
- [2] B. Fang et al., "Achieving excellent thermoelectric properties in CaTiO₃-based materials via entropy engineering and recrystallization," Chemical Engineering Journal, vol. 509, p. 161480, Mar. 2025.
- [3] LV. Hua et al., "Multishelled hollow CaTiO₃ cubes decorated with Co_{0.2}Cd_{0.8}S nanoparticles for efficient photocatalytic H₂ evolution via S-scheme charge transfer," Separation and Purification Technology, vol. 345, p. 127429, 2024.
- [4] J. Doneliene, E. Fataraitė-Urbonienė, M. Rudzikas, S. Pakalka, N. Dančova, and J. Ulbikas, "Effect of precursor nature and Sol-Gel synthesis conditions on TiO₂ aerogel's structure," Molecules, vol. 26, no. 16, p. 5090, Aug. 2021.