Hydrothermal Synthesis of Sodium Tantalate Nanocubes

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Abstract
Experiments were conducted to optimize the growth parameters of perovskite structure of alkali tantalate in energy efficient environment friendly hydrothermal process. Here we are presenting Sodium tantalates out of Lithium, Sodium, and Potassium Tantalates perovskite which were grown at 140°C for 15 hours in high alkaline environment. Sodium tantalates contains monoclinic crystal phase of perovskite structure with an average size range of 70 nm. The morphological, compositional, structural, and thermal properties of as synthesized nanocubes were characterized by using scanning electron microscope (SEM), x-ray powder diffraction (XRD), and thermal gravimetric analysis (TGA) techniques.

Introduction
Alkali tantalates are perovskite compounds of group I element bonded with tantalum and oxygen atoms. A material that obeys the crystallographic structure of calcium titanate (CaTiO₃) is usually known as perovskite material. The perovskite structure simply consists of a large cation A with 12-fold coordination at the center of a cubic lattice. The corners of the cube is relatively smaller cation B with 6-fold coordination, and the midpoint of each edge are occupied by smaller anions C (halides or oxides). Alternately, cations A are at corners, cation B is at the center of the cube, and anions C/O⁻ are located at the middle of each face as shown in figure 1. The majority of perovskite compounds are oxides but halides and cyanides also exist such as MnCNO₃ (M = Al, Mg, Zn). MAPbX₃ (M = CH₃NH₃, HX₃ = halides), and M(TaO₅) (M = Li, K, Na). They possess properties of semiconductor, ferroelectric, piezoelectric, and superconducting. Perovskite oxides of type AB₂O₃, however, are fascinating functional materials which exhibit range of stoichiometries and crystal structures. The filled and unfilled 3d shells of transition metal give dielectric, electric, and magnetic behavior of these materials. The functionalities of these materials can be utilized in catalysis, fuel cells, and electrochemical sensing [1–5]. Tantalate based perovskite such as NaTaO₃ exhibits fairly high activity for the photocatalytic decomposition of water under ultraviolet irradiation [6]. The flexible structure of oxide perovskites with different A and B ions lead to the large number of known compounds. Most perovskites are distorted and do not have ideal cubic structure. Therefore, they are fascinating to be explored to exploit their special properties. Another relevant subject is to develop an environmental friendly chemical process to synthesize perovskite compounds.

Main Objectives
1. To develop an environmental friendly chemical process to synthesize nanoparticles.
2. To optimize growth parameters such as temperature, duration, and chemical concentrations to synthesize alkali nanocubes at low temperature hydrothermal process.
3. To understand how the oxygen stoichiometry and lattice distortion are introduced as a result of doping another type of cation with different valence state.

Materials and Methods
Among various methods such as mechanochemical synthesis, gas phase synthesis, and wet chemical synthesis (sol-gel process, and hydrothermal process) to synthesize tantalates perovskite [5]. Hydrothermal process is one of the most suitable, energy efficient, and environmentally friendly chemical process. We have used this process to optimize growth parameters of NaTaO₃ nanocubes and control their size range. Size and chemical compositions of oxides type perovskite in HTs process can be controlled by adjusting the concentration of precursors, reaction time and temperature. NaTaO₃ nanocubes were synthesized by reacting a Tantalate powder Ta₂O₅ as a suitable precursor in high alkaline NaOH environment under hydrothermal conditions. The reaction mechanism is given as 2NaOH + Ta₂O₅ ⇌ 2NaTaO₃ + H₂O. We have dissolved 0.44 g of Ta₂O₅ powder in 0.75 M of NaOH and 50 mL of this solution was kept in 100 mL Tellon lined autoclave and heated for 15h at 140°C. The milky-white products were centrifuged and washed with water and ethanol many times and dried at 80°C for 6 hours after reaction time is complete.

Results & Discussion
The average size of nanocubes are 70 nm as measured from full width at half maximum (FWHM) value of prominent XRD peaks using Scherrer’s formula. TGA curve was obtained to determine thermal stability and to monitor decomposition behavior of synthesized nanocubes. Continuous weight loss after 380°C may indicate no thermal stability in this experimental temperature range.

Conclusions
We have synthesized Sodium Tantalates nanocubes at 140°C for 15 hours of growth process in high alkaline atmosphere by hydrothermal process. This compound contains monoclinic crystal phase of perovskite structure. The nanocubes were about 70 nm in size and have shown phase transition state between 250°C to 600°C, without any thermal stability within our experimental range of TGA study.

References

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