



# Preparation of $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$ perovskite-structured ceramic using the glycine-nitrate combustion technique



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## ABSTRACT

$\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  perovskite-structured ceramic was produced using wet processing method namely glycine-nitrate combustion technique. The quality of powders produced through this wet processing method was strongly related to processing parameters and heat treatment. This study investigated the effect of calcination temperature and mixing time of nitrate solution on the formation of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$ . Results revealed that calcination temperature and mixing time significantly affected the purity and average crystallite size of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$ . The crystallite grew to 31.48 nm when the calcination temperature was increased, whereas small crystallites (~30 nm) were produced when the mixing time was prolonged. In terms of the purity of powders, XRD analysis indicated that secondary phases ( $\text{Sr}_3\text{Fe}_2\text{O}_7$  and  $\text{TiO}_2$ ) were retained when the calcination temperature was further increased to 1300 °C. As the mixing time was prolonged, secondary phases were removed. After 15 h of mixing, the lowest intensity of secondary peak was observed. The effects of mixing time on the production of pure and fine crystallites of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  powders were more significant than those of calcination temperature. Thus, mixing time should be prolonged until the optimum duration is reached to produce pure  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  perovskite-structured ceramic powders.

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## 1. Introduction

The perovskite-structured ceramic  $\text{ABO}_x$  is widely known for its mixed ionic-electronic conductor property and thus suitable for use in gas sensor and fuel cell applications [1,2]. Perovskite materials for gas sensor membranes and SOFC electrode applications are traditionally produced through a solid-state reaction, but powders synthesized through this method yield high impurity, contain large particles (60–500 nm), and exhibit low homogeneity [1,3,4]. Thus, wet processing techniques, such as combustion, are proposed to obtain pure powders with small particles of perovskite oxides [5].

This work aimed to produce  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  perovskite with high purity and fine crystallites through glycine-nitrate combustion (GNC). Purity and size were strongly related to calcination and processing parameters, such as mixing time. The effects of the calcination and mixing time of nitrate solution on the purity and crystallite size of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  powders were also discussed.

## 2. Experimental

Precursor powders of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  were prepared using GNC. Raw materials were purchased from Sigma-Aldrich. Metal nitrates, such as strontium nitrate,  $\text{Sr}(\text{NO}_3)_2$  and iron nitrate,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were dissolved in de-ionized water with as-prepared titanyl nitrate,  $\text{TiO}(\text{NO}_3)_2$ . Glycine,  $\text{NH}_2\text{CH}_2\text{COOH}$  was added to the solution at a stoichiometric ratio to nitrates of 0.55. The glycine/nitrate (G/N) ratio was calculated in accordance with propellant chemistry principle [6,7]. Nitrates and glycine were mixed for 1 h before the temperature of the hot plate was increased to 150 °C to allow the drying process. Auto-ignition combustion occurred when the temperature was increased to 350 °C. Dark precursor powders of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  were yielded after combustion.

The thermal decomposition of precursor powders was analyzed through thermogravimetric analysis (TGA) (Pyris Diamond TG/DTG analyzer) and Fourier transform infrared (FTIR) (PerkinElmer) spectroscopy. TGA was conducted in static air from 30 °C to 1200 °C at a heating rate of 10 °C  $\text{min}^{-1}$ . Calcination temperature ( $T_c$ ) was proposed by referring to thermal decomposition results. The proposed  $T_c$  was then applied to three additional batches of precursor powders. Each batch differed in terms of the mixing time of the nitrate solution during synthesis. After calcination was

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performed, the formation of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  powders for each batch was determined using X-ray diffractometer (XRD), a field emission electron microscopy (FESEM; JEOL JSM-6701F) and a transmission electron microscope (TEM; ZEISS LIBRA-120).

### 3. Results and discussion

Fig. 1 presents the thermal decomposition behavior of the  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  precursor powders. The TG and derivative TG curves showed the three stages of significant weight loss as the temperature increased. The downward peaks from the derivative TG curve confirmed the significant weight loss regions. In the first stage (below 200 °C), 1.16% of weight loss was recorded because of the elimination of water [8]. The slow rate of weight loss occurred continuously until it became significant as the second stage was reached. In the second stage, 2.36% of weight loss was detected from 380 °C to 520 °C. The losses in this temperature region occurred because of the decomposition of nitrates and carbons

[9]. In the final stage, a sudden decrease in weight percent was detected from 800 °C to 1000 °C. Approximately 5.35% of weight loss was recorded because of the decomposition of the remaining carbon in the powders [8]. Therefore, the decomposition of precursor powders to the desired oxide yielded 11.68% of weight loss. The weight loss stopped at approximately 1000 °C. Thus,  $T_c$  of precursor powders should be higher than 1000 °C. At the same time,  $T_c$  must not exceed 1435 °C because the  $\text{SrFeO}_{3-\delta}$  phase begins to melt at this temperature [10]. To observe the undesired phase elimination, we calcined the powders at 700 °C, 1100 °C, 1200 °C and 1300 °C. The removal of the undesired phases and the formation of the oxide phase were then observed through FTIR.

The thermal decomposition of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  was extensively examined by using an FTIR spectrometer. FTIR analysis was performed on the precursor powders and  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  powders that were calcined at 700 °C, 1100 °C, 1200 °C and 1300 °C (Fig. 2). The peak of the precursor powders was observed at wavenumbers of 1400–1600, 860 and 550  $\text{cm}^{-1}$ . The peak at 1400–1600  $\text{cm}^{-1}$  was attributed to the in-plane bending of the

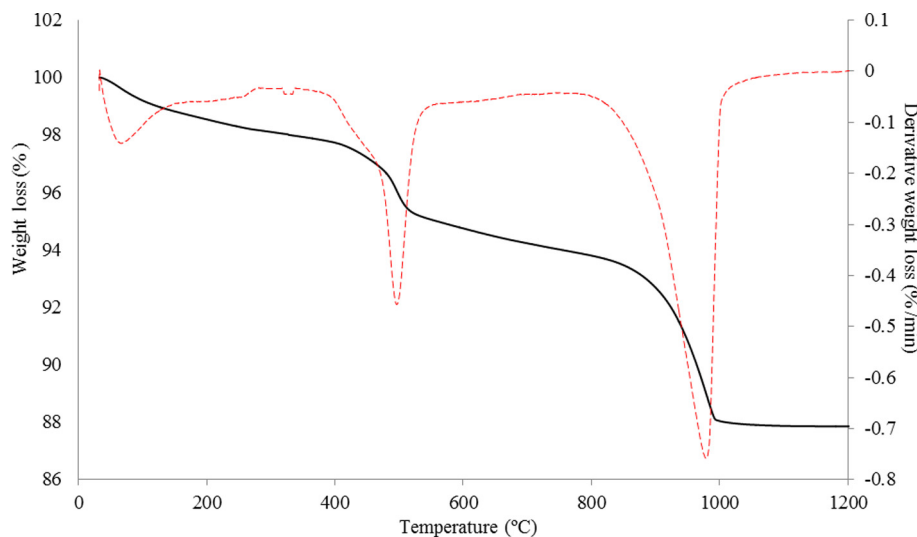


Fig. 1. Thermal decomposition behavior of  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  precursor powders.

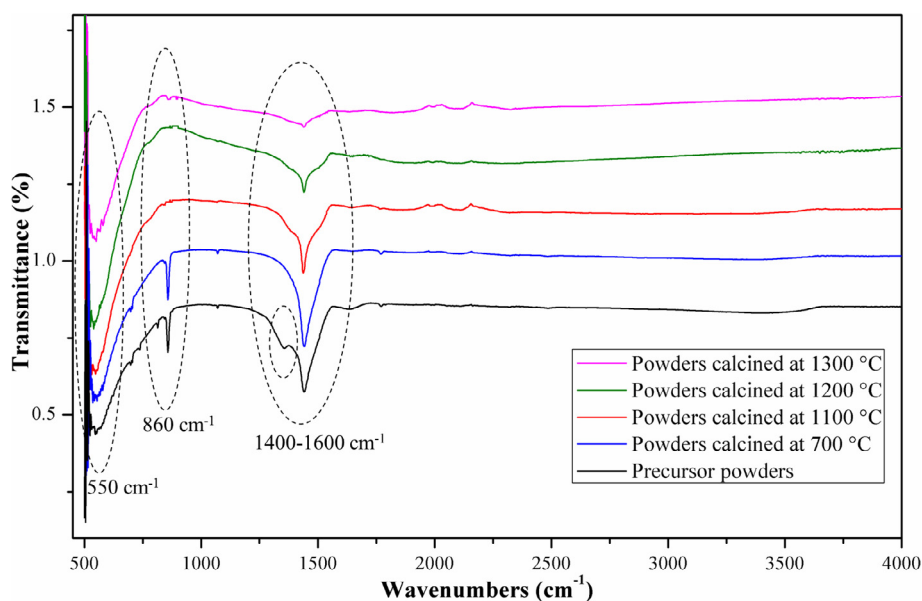


Fig. 2. FTIR results for precursor powders and calcined  $\text{SrFe}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$  powders at temperature 700 °C, 1100 °C, 1200 °C and 1300 °C.

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