INTRODUCTION

Metal metastannates, with structures closely related to the perovskite one, are of particular interest due to their unusual dielectric and semiconducting properties, leading to various applications such as thermally stable capacitors, ceramic dielectric bodies, gas and humidity sensors, and battery electrode materials. Cubic perovskite type of metal stannates (MSnO$_3$) have attracted considerable attention for potential applications. It has been observed that a partial substitution of cations at M/Sn sites results in substantial modification in their physical properties so as to make them suitable for a wide variety of industrial applications. Metal metastannates have attracted considerable attention for potential applications in ceramic dielectric bodies, gas and humidity sensors, thermally stable capacitors and battery electrode materials.

Several processes have been developed over the past decade to enhance the applications of metal metastannates prepared by a range of processes, such as a solid-state reaction, a co-precipitation method, sol-gel, a precursor route, a microwave synthesis, a polymerized complex method, a molten salt synthesis, a pulsed laser deposition, a self-heat-sustained method. The advantages of microwave synthesis are to bring a short reaction time, a small particle size and a narrow particle size distribution. Microwave energy is delivered to the surface of the material by radiant and/or convection heating, which is transferred to the bulk of the material via conduction to the material through molecular interactions with an electromagnetic field. Hydrothermal process is an efficient low temperature method that allows the formation of particles with high degree of crystallinity and easy dispersion in an aqueous medium. The use of microwave energy in hydrothermal system promotes the development of a rapid heating to the required temperature with rapid rates of crystallization.

The microwave solvothermal process is adjusted to heat the metal stannates uniformly resulting in fine particles with a controlled morphology and to fabricate the product in a green manner without the generation of solvent waste. Recently, microwave solvothermal processes have been reported the use of a facile and fast method in preparing nanocrystalline particles of metal tungstates with unique and enhanced properties. When the solvent is ethylene glycol, the reactions proceed in a sealed pressure autoclave at temperatures above boiling point of the ethylene glycol. Microwave solvothermal process using a solvent of ethylene glycol is a facile process that provides a high-qualified yield with cost-effective method in short time periods.

In this study, SrSnO$_3$ and MgSnO$_3$ nanoparticles were synthesized using a facile solvothermal route assisted by the microwave irradiation. The characteristics of the synthesized SrSnO$_3$ and MgSnO$_3$ nanoparticles are discussed in detail based on the microwave solvothermal reaction in ethylene glycol under the high sealed pressure. The synthesized SrSnO$_3$ and MgSnO$_3$ nanoparticles were characterized by X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy and transmission electron microscopy.
**EXPERIMENTAL**

Fig. 1 shows a flow chart for the synthesis of SrSnO$_3$ and MgSnO$_3$ nanoparticles by the microwave solvothermal process. SrCl$_2$·6H$_2$O, MgCl$_2$·6H$_2$O, Na$_2$SnO$_3$·3H$_2$O and ethylene glycol of analytical reagent grade (Aldrich) were used to prepare the SrSnO$_3$ compound. Each of 0.01 mol SrCl$_2$·6H$_2$O and 0.01 mol Na$_2$SnO$_3$·3H$_2$O for SrSnO$_3$ was dissolved in 30 mL ethylene glycol. In the same way, each of 0.01 mol MgCl$_2$·6H$_2$O and 0.01 mol Na$_2$SnO$_3$·3H$_2$O for MgSnO$_3$ was dissolved in 30 mL ethylene glycol. The solutions were mixed and adjusted at a pH 9.5 using NaOH. The aqueous solution was stirred at room temperature. In the sequence, the mixture was transferred into a Teflon-lined digestion vessel of 120 mL capacity. The Teflon vessel was placed into a microwave solvothermal autoclave (2.45 GHz, maximum power of 800 W). The microwave solvothermal conditions were kept at 200 °C for 0.5 h. After microwave solvothermal process, the microwave autoclave was cooled to room temperature. The resulting solutions were treated with ultrasonic radiation and washed many times with distilled hot water. The white precipitates were corrected and dried at 100 °C in a dry oven. The final products were heat-treated at 800 °C for 3 h.

![Flow chart for the synthesis of SrSnO$_3$ and MgSnO$_3$ nanoparticles](image)

**RESULTS AND DISCUSSION**

Fig. 2 shows XRD patterns of the SrSnO$_3$ nanoparticles synthesized by the microwave solvothermal process after heat-treatment at 800 °C for 3 h. All XRD peaks could be assigned to an orthorhombic structure of SrSnO$_3$ (JCPDS: 09-0086)$^{12}$. These suggest that microwave solvothermal synthesis is suitable for the growth of SrSnO$_3$ crystallites with the strongest major intensity peaks from the (210), (220) and (400) planes with some preferred orientation, respectively. Fig. 3 shows XRD patterns of the MgSnO$_3$ nanoparticles. All XRD peaks could be assigned to an orthorhombic structure of MgSnO$_3$ (JCPDS: 30-0798)$^7$. These suggest that microwave solvothermal synthesis is suitable for the growth of MgSnO$_3$ crystallites with the strongest major intensity peaks from the (104), (110) and (024) planes with some preferred orientation, respectively.

![XRD patterns of the SrSnO$_3$ nanoparticles](image)

![XRD patterns of the MgSnO$_3$ nanoparticles](image)

Fig. 4 shows FT-IR spectra of the SrSnO$_3$ and MgSnO$_3$ nanoparticles in the wavenumber range, 4000-480 cm$^{-1}$. The very strong absorbable peak at 629 cm$^{-1}$ in Fig. 4(a) reveals typical characteristics of a strong Sr-O stretching mode. The strong Sn-O stretching modes are contributed to the uniform regular SrO$_6$ octahedra of the metal stannates$^{12}$. The bands at 856 and 1050 cm$^{-1}$ are due to the presence of carbonates. The bands at 2350, 1640 and 1450 cm$^{-1}$ are assumed that the samples prepared contain a small amount of surface-adsorbed water and alcohol.$^9$ The very strong absorbable peak at 629 cm$^{-1}$ in Fig. 4(b) reveals typical characteristics of a strong Mg-O stretching mode. The strong Mg-O stretching modes are attributed to the uniform regular MgO$_6$ octahedra of the metal stannates$^{12}$. The bands at 856 cm$^{-1}$ due to the presence of carbonates.

Fig. 5 shows a SEM image (a) and a TEM image (b) of the SrSnO$_3$ nanoparticles. The SEM image of SrSnO$_3$ in Fig. 5(a) shows a well-defined and homogeneous morphology, while the TEM image of SrSnO$_3$ in Fig. 5(b) shows the particle sizes of 20-40 nm. Fig. 6 shows a SEM image (a) and a TEM image (b) of the MgSnO$_3$ nanoparticles. The SEM image of MgSnO$_3$ in Fig. 6(a) shows a well-defined and homogeneous morphology,
Fig. 4. FT-IR spectra of the (a) SrSnO$_3$ and (b) MgSnO$_3$ nanoparticles while the TEM image of MgSnO$_3$ in Fig. 6(b) shows the particle sizes of 50-200 nm. The solvothermal synthesis proceeds the reactions between MCl$_2$·6H$_2$O and Na$_2$SnO$_3$·3H$_2$O in a hot ethylene glycol solution as a polar solvent above a boiling point of 197 ºC. The microwave solvothermal process of metal metastannates occurs in accordance with the reaction:

$$\text{MCl}_2\cdot x\text{H}_2\text{O} + \text{Na}_2\text{SnO}_3\cdot 3\text{H}_2\text{O} \rightarrow \text{MSnO}_3 + 2\text{NaCl} + x\text{H}_2\text{O}$$

When the microwave radiation is supplied to the ethylene glycol under a sealed pressure above boiling point, the components dissolving in the ethylene glycol are charged and vibrated in electric field interdependently. The microwave solvothermal process is adjusted to heat the metal stannates uniformly resulting in fine particles with a controlled morphology and to fabricate the product in a green manner without the generation of solvent waste. The microwave-assisted solvothermal reaction involves the exchange of atomic/ionic species, where the driving force is the exothermic reaction in ethylene glycol accompanying the formation of NaCl with a high lattice energy.

The microwave exothermic reaction occurs so rapidly that the exothermic reaction was essentially used to heat up the metal metastannates. Well-crystallized SrSnO$_3$ and MgSnO$_3$ nanoparticles were formed after heat-treatment at 800 ºC for 3 h showing a fine and homogeneous morphology with particle sizes of 20-40 nm for SrSnO$_3$ and with particle sizes of 50-200 nm for MgSnO$_3$. The very strong absorbable peak at 629 cm$^{-1}$ was typical characteristics of a strong Sr-O and Mg-O stretching modes.

**Conclusion**

SrSnO$_3$ and MgSnO$_3$ nanoparticles were synthesized successfully by the microwave solvothermal processes between SrCl$_2$-6H$_2$O and Na$_2$SnO$_3$-3H$_2$O for SrSnO$_3$ and MgCl$_2$-6H$_2$O Na$_2$SnO$_3$-3H$_2$O for MgSnO$_3$ in a hot ethylene glycol solution as a polar solvent. The microwave solvothermal reactions occurred so rapidly that the exothermic reaction was essentially used to heat up the metal metastannates. Well-crystallized SrSnO$_3$ and MgSnO$_3$ nanoparticles were formed after heat-treatment at 800 ºC for 3 h showing a fine and homogeneous morphology with particle sizes of 20-40 nm for SrSnO$_3$ and with particle sizes of 50-200 nm for MgSnO$_3$. The very strong absorbable peak at 629 cm$^{-1}$ was typical characteristics of a strong Sr-O and Mg-O stretching modes.

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**REFERENCES**