



REGULAR ARTICLE

Influence of Calcination Temperature on Optical Properties of AlSbO₄ Alloy Nanoparticles Synthesized by Sol-Gel Method

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The influence of different calcination temperatures on the structural and optical properties, were investigated for aluminium antimonate (AlSbO₄) alloy nanoparticles synthesized using Sol-Gel method. The prepared AlSbO₄ nanoparticles are calcined at 400 °C, 600 °C, 700 °C and 800 °C. The effect of different calcinations temperature on some physical properties of the prepared sample was studied using X-ray Diffraction (XRD) and Fourier Transform Infrared (FT-IR) spectroscopy. The results showed that XRD and FT-IR confirmed the formation and the presence of AlSbO₄ nanoparticles. The investigation showed that an increase in the calcination temperature led to appreciable decrease in the crystallite size (*D*) of the synthesized nanoparticles.

Keywords: Sol-gel, Nanoparticles, AlSbO₄, Particles sizes, Calcination temperature.

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1. INTRODUCTION

Most materials are mixtures of atoms or molecules of different nature. In some cases, the constituents (atoms or molecules) of the material are soluble in any proportion and the mixtures are homogeneous. In other cases, the constituents are only partially miscible. The material then contains several phases of different composition and structure.

The combination of these phases produces different microstructures which considerably influence the properties and the technical characteristics of the materials. The concept of alloy was initially reserved for metals. Conventionally, an alloy is a material of metallic character combining one or more metals and possibly elements of non-metallic character. This concept, which is currently evolving, is gradually being extended to other materials (ceramics, polymers), and we now define an alloy as a combination of atoms, ions, or molecules to form a material whose properties differ from those of the constituents. Alloys metals oxides of Al-Sb-O have been a subjects of interesting area of research due to their potential use as catalysts of a lot of industrial processes [1-4]. The influence of calcination temperature on the physical and chemical properties of different materials has been studied previously by several authors [5-10]. Various preparation techniques, such as hydrothermal followed by calcination [11-16], the Pechini process [16], solid state routes [11], oxalate-precursor co-precipitation [17] and the spray pyrolysis method [18] are employed to prepare materials in powders form composed of nanoparticles (NPs).

Sol-gel process is one of the effective methods for synthesis nanoparticles metal oxide because it has many advantages than other methods [19-21]. The present

study aimed to investigate the effect of calcination temperature on the physical (structural and optical) properties of aluminium antimonate (AlSbO₄) alloy nanoparticles synthesized using sol-gel method.

2. EXPERIMENTAL DETAILS

Aluminium antimonate (AlSbO₄) nanoparticles were synthesized using the sol-gel method. All reagents were of analytical grade and were used without further purification. For the synthesis of this matrix, aluminium nitrate nanohydrate (Al(NO₃)₃·9H₂O; 99 % Sigma-Aldrich) and antimoine oxide (Sb₂O₃; 99 % Sigma-Aldrich) have been used as precursors, distilled water and HCl were used as a solvent.

A conventional sol-gel method was conducted to prepare the AlSbO₄ nanoparticles; in this aim, appropriate masses of aluminium nitrate nanohydrate Al(NO₃)₃·9H₂O (10 g) and antimoine oxyde Sb₂O₃ (10 g) were dissolved in 50 mL of deionized water and 100 mL of HCl.

The mixture was stirred magnetically for 6 hours at room temperature. The obtained gel was dried in the temperature range from 125 °C to 225 °C for 12 hours. Finally, the dried powders were calcined in a muffle furnace at different temperatures: 400, 600, 700 and 800 °C for 4 h.

An examination of synthesized nanoparticles involves the utilisation of various analytical methods. The structural parameters of prepared ZnO NPs were determined by employing the MiniFlex 600W powder diffractometer, utilizing CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) in the range from 3 to 90 deg. The FT-IR spectra were captured employing the Alpha Bruker FT-IR spectrometer.

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3. RESULTS AND DISCUSSION

3.1 XRD Analysis

X-ray diffraction (XRD) is a perfect technique to examine the distribution of atoms or ions in materials. XRD patterns of the synthesized aluminium antimonate nanoparticles (AlSbO_4) at 400 °C, 600 °C, 700 °C and 800 °C calcination temperatures were shown in Fig. 1. It shows diffraction peaks at 2θ values of 27.58°, 29.03°, 36.29°, 39.44°, 41.13°, 44.28°, 54.69°, 56.38°, 62.44°, 65.34°, 73.57°, 75.75° and 83.25°. These peak correspond to (110), (200), (011), (020), (111), (120), (121), (220), (002), (130), (131), (230) and (231) planes respectively. All the diffraction peaks were matched well with AlSbO_4 according to the joint committee on powder diffraction standard (JCPDS) file for AlSbO_4 (No. 98-010-8961), and confirm the tetragonal phase of AlSbO_4 with the lattice parameters $a = 4.53 \text{ \AA}$ and $c = 2.973 \text{ \AA}$; the espace group is $P_{42/mnm}$. The non identified peaks at 25.75°, 30.59°, 34.01°, 37.37°, 49.02°, 51.40° and 59.40° in Fig. 1; were attributed to the presence of some impurities and to the formation of Sb_6O_{13} oxide. It can be observed from these diffractograms that with increasing calcination temperature, the intensity of characteristic peaks increases gradually. In Fig. 2, we show the crystallographic structure of AlSbO_4 nanoparticles.

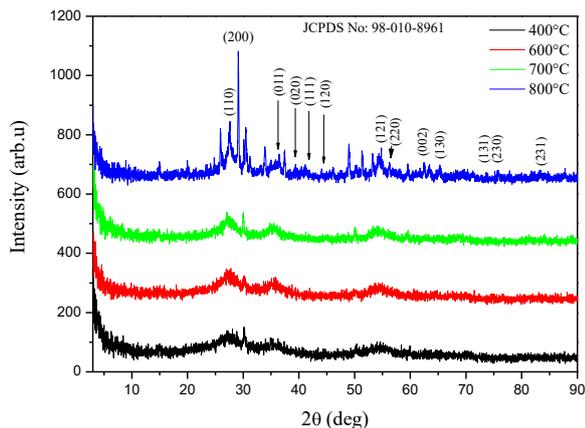


Fig. 1 – XRD patterns of AlSbO_4 nanoparticles at different calcinations temperature

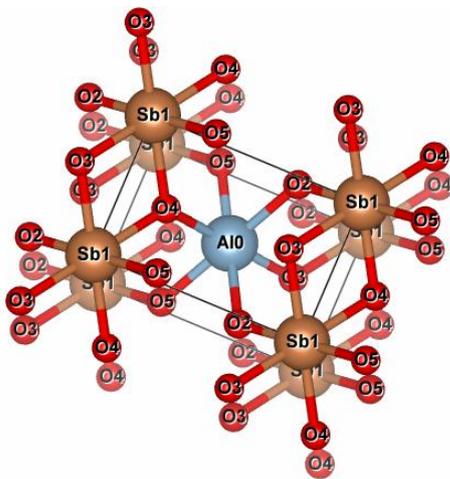


Fig. 2 – Crystallographic structure of AlSbO_4 using VESTA software

The crystallite size of the calcined powders was determined by the X-ray line broadening technique performed on the main (most intense) diffraction peak from the (200) plane using the Debye-Scherrer formula [22]; obtained values are summarized in Table 1.

$$D = \frac{0.9\lambda}{\beta \cdot \cos\theta}$$

where D is the crystallite size (nm), λ is the wavelength of $\text{CuK}\alpha$ radiation, which equals to 0.154 nm, β is the FWHM of the main diffraction peak and θ is the Bragg angle.

The dislocation density, δ was calculated using the formula [$\delta = 1/D^2$] and the results were listed in Table 1.

During the calcination process, the increase of calcination temperature is beneficial to the continuous crystallization on the surface of crystal particles, the size of these particles decreases continuously which is in accordance with XRD characterization results [23-25]. The variation of the particles size as a function of calcination temperature is shown in Fig. 3. It can be observed that the crystallite size decreases with an increase of the calcination temperature.

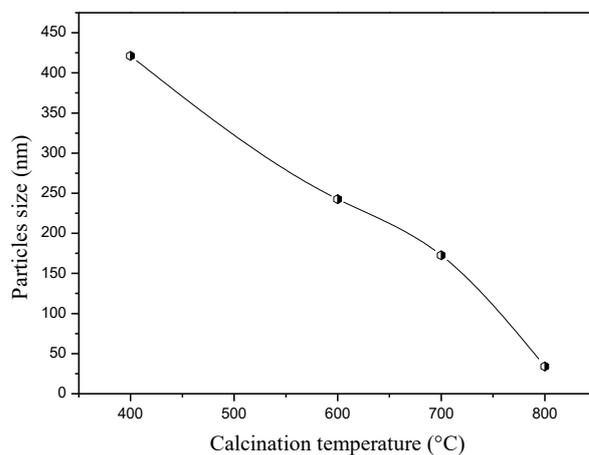


Fig. 3 – Variation of the particles size as a function of calcination temperature

Table 1 – Lattice parameters, average crystallite sizes and the dislocation

Calcination temperature (°C)	2θ (deg)	β (deg)	D (nm)	δ (line/m ²)
400	29.03	0.020	421.11	5.63×10^{12}
600	29.06	0.034	242.67	1.69×10^{13}
700	29.01	0.048	172.65	3.35×10^{13}
800	29.05	0.241	34.08	8.61×10^{14}

3.2 FT-IR Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FT-IR) was utilized to analyse the surface chemistry of the synthesized ZnO nanoparticles using an Alpha Bruker FT-IR spectrometer. This technique was applied to identify the surface functionality and vibrational modes of the inorganic core of the prepared additives. Functional groups attached to the surface of NPs were detected in the range of 4000-400 cm^{-1} .

The FT-IR spectrograms of AlSbO_4 nanoparticles prepared at different calcination temperatures are provided in

Fig. 4. The four infrared spectra are basically similar. A weak absorption peak at 1700 cm^{-1} and strong band absorption at 3500 cm^{-1} were attributed to O-H bending and stretching vibration absorption peaks in water [8]. This may be due to the absorption of water vapor in the air during the determination process. The two peaks at 747 and 970 cm^{-1} , were ascribed to the stretching vibration of Sb-O bond [26]. The appearance of two peaks at 792 cm^{-1} and 600 cm^{-1} are assigned to Al-O vibrations [27].

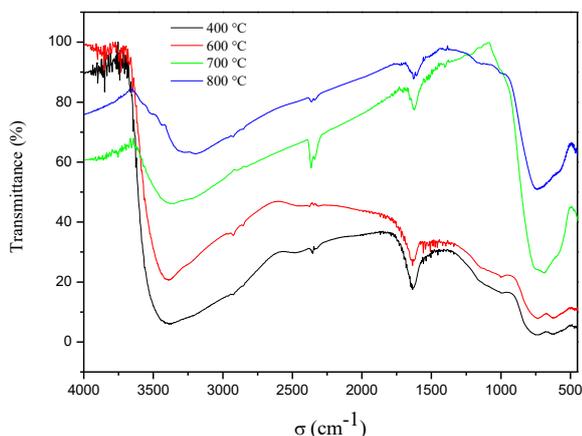


Fig. 4 – FT-IR spectra of AlSbO₄ nanoparticles prepared at different calcination temperatures

4. CONCLUSIONS

In summary, the influence of calcination temperature on the optical and structural properties of aluminium antimonate nanoparticles (AlSbO₄) has been investigated in this study. The AlSbO₄ matrix was synthesized using the sol-gel method, and then calcined at different calcination temperatures (400 °C , 600 °C , 700 °C and 800 °C). Both, the XRD and FT-IR results confirm the formation and the presence of AlSbO₄ nanoparticles. It was found that calcination temperature is an important parameter in the synthesis of our sample, and the formation of AlSbO₄ nanoparticles depends on it. The calcination temperature affected the structural properties of the synthesized material; the particles size has been decreased following the increasing of the calcination temperature.

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Вплив температури прожарювання на оптичні властивості наночастинок сплаву AlSbO_4 , синтезованих золь-гель методом

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Досліджено вплив різних температур прожарювання на структурні та оптичні властивості наночастинок сплаву антимонату алюмінію (AlSbO_4), синтезованих золь-гель методом. Підготовлені наночастинки AlSbO_4 прожарюють при 400 °C, 600 °C, 700 °C і 800 °C. Вплив різних температур прожарювання на деякі фізичні властивості підготовленого зразка вивчали за допомогою рентгенівської дифракційної (XRD) та інфрачервоної (FT-IR) спектроскопії з перетворенням Фур'є. Результати показали, що XRD та FT-IR підтвердили утворення та присутність наночастинок AlSbO_4 . Дослідження показали, що підвищення температури прожарювання призводило до помітного зменшення розміру кристалітів (D) синтезованих наночастинок.

Ключові слова: Золь-гель, Наночастинки, AlSbO_4 , Розміри частинок, Температура прожарювання.