Properties of Stannum-Based Li-NASICON-Structured Solid Electrolytes for Potential Application in Electrochemical Devices

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The aim of this study was to synthesize NASICON-structured LiSn2P3O12 solid electrolytes by the citric acid assisted sol-gel method upon sintering for 48 hours instead of 24 hours, as reported in our previous study. X-ray diffraction analysis confirmed the formation of a rhombohedral phase of a NASICON-type structure upon sintering at 600 and 650 °C for 48 hours. By sintering a sample of LiSn2P3O12 at a temperature of 600 °C, a conductivity of 1.38 ×10⁻⁵ Scm⁻¹ at 500 °C was obtained. Meanwhile, a lower conductivity of 1.03 x 10⁻⁵ Scm⁻¹ at 500 °C was obtained when the LiSn2P3O12 sample was sintered at a temperature of 650 °C. The decomposition voltage reached 4.8 V for the highest conducting LiSn2P3O12 sample sintered at 600 °C. Thus, the current results show that LiSn2P3O12 is a promising candidate for applications as a solid electrolyte in elevated temperature electrochemical devices.

Keywords: Stannum; NASICON-structured; Solid electrolyte; Ionic conductivity

1. INTRODUCTION

Increasing demands for high-energy power sources have led to the development of power sources technology. Due to their high ionic conductivity, solid electrolytes have proven to be viable for use in power sources industries, and are generally used as components in innovative electrochemical appliances such as batteries, fuel cells, gas separation membranes, chemical sensors and ionic switches [1]. On the other hand, compared to solid electrolytes, liquid electrolytes that are used commercially possess a number of disadvantages, some of which are that they only function within a limited range of temperatures; the electrolyte solution may corrode the electrode and cause the device to malfunction;
and they are subject to leakage [2]. In view of these shortcomings, a suitable and ideal solid electrolyte would be one that possesses a high ionic conductivity (< $10^{-4}$ S cm$^{-1}$) at a wide range of operating temperatures, a low electronic conductivity, as well as good electrochemical stability window with regard to electrodes (> 4.5 V). Moreover, solid electrolytes come in simple designs, can provide a natural seal, are able to withstand shocks and vibrations, as well as changes in pressure and temperature, have greater electrochemical stability, and are much safer [3].

Among the many solid electrolytes available, NASICON (Sodium Super Ionic Conductor) has been extensively tested for use in power sources. NASICON is better than other electrolyte materials in terms of its high ionic conductivity, its stable phosphate units and its high melting point (> 1650 °C) [5]. It was first discovered by Goodenough et al. (1976) and its general formula is Na$_{1+x}$Zr$_2$Si$_3$P$_3$O$_{12}$. The NASICON compound basically has a rhombohedral (R$\overline{3}$c) symmetrical structure comprised of a three-dimensional framework, with the corners being made up of ZrO$_6$ octahedra and PO$_4$ tetrahedra. In all NASICON compounds, the Li$^+$ ion conductor, LiSn$_2$P$_3$O$_{12}$ has been the least studied compared to LiTi$_2$P$_3$O$_{12}$ [6-10]. The main challenge with regard to this compound is to increase its ionic conductivity, as previous researchers have reported that it has a low ionic conductivity of between $10^{-7}$ S cm$^{-1}$ and $10^{-10}$ S cm$^{-1}$ [8; 11 – 15]. Alternatively, a simple and economical sol-gel method may be used to prepare LiSn$_2$P$_3$O$_{12}$ NASICON-structured materials as this method can result in a lower synthesis temperature that is favourable for obtaining a higher ionic conductivity in highly homogeneous materials [16 - 17]. In previous work, NASICON rhombohedral-structured LiSn$_2$P$_3$O$_{12}$ solid electrolytes were successfully obtained via the citric acid assisted sol-gel method upon sintering at 600 °C for 24 hours with minor traces of SnP$_2$O$_7$ impurity [18]. The conductivity of the prepared sample at 500 °C was $1.40 \times 10^{-5}$ S cm$^{-1}$, which is one order of magnitude higher than that reported in an earlier work by Norhaniza et al. (2010), and four orders of magnitude higher than that reported by Martinez-Juarez et al. (1997) and Lazarraga et al. (2004).

In the present study, LiSn$_2$P$_3$O$_{12}$ samples were synthesized by means of the citric acid assisted sol-gel method, but the sintering process was carried out for a longer period of time, that is 48 hours. The longer sintering time was expected to lead to the decomposition of the minor traces of SnP$_2$O$_7$ impurity that was found in the previous study. On the other hand, the sintering temperatures of 600 and 650 °C were chosen based on the thermal test in the previous study. Next, X-ray diffraction (XRD), Fourier transform infrared (FTIR), scanning electron microscopy (SEM), particle size analysis, energy dispersive X-ray (EDX), impedance spectroscopy (IS), linear sweep voltammetry (LSV) and transference number measurements were employed to examine the structural, thermal, electrical and electrochemical properties of the LiSn$_2$P$_3$O$_{12}$ samples.

2. EXPERIMENTAL PROCEDURES

2.1 Synthesis of LiSn$_2$P$_3$O$_{12}$ Samples

The LiSn$_2$P$_3$O$_{12}$ samples were prepared by means of the citric acid assisted sol-gel method. All the chemicals were of analytical grade and were directly used as received without further purification. The starting materials used were lithium acetate (CH$_3$COOLi), stannum (IV) chloride pentahydrate (SnCl$_4$.5H$_2$O) and ammonium phosphate (H$_{12}$N$_3$O$_4$P), while the chelating agent was citric acid,
(C₆H₈O₇) and the solvent was distilled water. Firstly, CH₃COOLi, SnCl₄·5H₂O and H₁₂N₃O₄P in the molar ratio of 1:2:3 were dissolved in distilled water under magnetic stirring. Citric acid (C₆H₈O₇), polyethylene glycol (C₂H₆O₂) and ammonium hydroxide (NH₄OH) were then mixed together into the previously prepared solution under magnetic stirring. C₂H₆O₆ and NH₄OH were added to promote polyesterification and polycondensation. The molar ratio of C₂H₆O₆·NH₄OH was 1:1. Next, the solution was shifted into a reflux system and was stirred constantly for 24 hours to form a homogenous solution. The solution was then removed and vaporized under magnetic stirring for approximately 4 hours at a temperature of 80 °C. Then, the gel that was produced was dried in an oven for 24 hours at 150 °C so as to get rid of water particles, resistant organic groups and also to prevent ceramic cracks. The resulting precursor powder was then ground finely for 30 minutes before being subjected to an annealing process at two different temperatures of 600 and 650 °C for 48 hours. The final product was then ground again for another 30 minutes before a Specac hydraulic press under a pressure of 5 tons was used to form the powder into pellets with a 13 mm-diameter and thickness of between 1.00 – 2.00 mm.

2.2 Characterization techniques

The samples were subjected to structural analysis by means of XRD using a Panalytical – X’pert³ x-ray diffractometer with Cu-Kα radiation of wavelength of 1.5406 Å. The 2θ ranged from 10 ° to 45 ° and was 0.026 ° with regard to step size. A Zeiss-Evo MA10 scanning electron microscope was used to conduct a morphological analysis of the LiSn₂P₃O₁₂ powders, while energy dispersive X-ray using an Oxford Aztec X-Act EDX spectrometer attached to the scanning electron microscope was used to perform an elemental analysis of the powders. A FRITSCH Laser Particle Sizer Analysette 22 NanoTec was used to derive information on the particle size of the electrolytes at room temperature. The impedance of the sintered pellets was measured by AC impedance spectroscopy using a Solatron 1260 impedance analyser over a frequency range of between 1 to 10 MHz. The applied voltage was set at 200 mV and all the measurements were taken at temperatures of between 30 to 500 °C. The electrochemical stability window of the electrolytes system was investigated by using the Wonatech ZIVE MP2 multichannel electrochemical workstation. LSV measurement was conducted at room temperature at a scan rate of 5 mV s⁻¹. Wagner’s D.C. polarization method was employed to assess the ionic transference number measurement. The sample was inserted between two stainless steel blocking electrodes and a potential current of 0.5 V was applied in order to polarize it. The current was then monitored as a function of time until it reached a steady state condition.

3. RESULTS AND DISCUSSIONS

3.1 X-ray Diffraction Analysis

The X-ray diffraction patterns of the LiSn₂P₃O₁₂ samples sintered at (a) 600 °C and (b) 650 °C are shown in Figure 1. A rhombohedral NASICON-type symmetrical structure (R₃c) was successfully obtained upon sintering at 600 and 650 °C for 48 hours in the presence of unreacted SnO₂ [9] compared...
to the sintering of the LiSn$_2$P$_3$O$_{12}$ samples for 24 hours, whereby there still remained slight traces of SnP$_2$O$_7$ impurity peaks [18], thus implying that SnP$_2$O$_7$ takes a longer time to decompose.

Figure 1. X-ray diffractograms LiSn$_2$P$_3$O$_{12}$ samples sintered at (a) 600 and (b) 650 °C upon sintering for 48 hours.

All the peaks observed in the LiSn$_2$P$_3$O$_{12}$ XRD pattern matched those reported in our previous work, thus, clearly indicating that the sol-gel method can be used to produce less impurity compound compared to other methods, such as the mechanical milling method that was used by Norhaniza et al. (2010). Besides that, unlike our earlier work where the double, rhombohedral and triclinic phases were found to coexist in the LiSn$_2$P$_3$O$_{12}$ samples sintered at 650 °C, in the current work only one phase, namely the rhombohedral phase, was detected in the samples sintered at the same temperature [18]. The following formula was then used to calculate the lattice parameters of the samples:

$$\frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \tag{1}$$

The crystallite size was determined by applying Scherrer’s equation as given below:

$$D = \frac{k\lambda}{\beta\cos\theta} \tag{2}$$

where $k$ denotes a Scherer constant value (0.94), $\lambda$ denotes the wavelength of the source (1.5406 Å), $\beta$ denotes the FWHM (in radians), and $\theta$ denotes the Bragg angle (in radians).
Table 1. Lattice parameters, unit cell volume and density of LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C

<table>
<thead>
<tr>
<th>Sintering Temperature (°C)</th>
<th>a [Å]</th>
<th>c [Å]</th>
<th>V [Å$^3$]</th>
<th>Crystallite size [Å]</th>
<th>Density [g cm$^{-3}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>8.3880</td>
<td>22.0148</td>
<td>1341.38</td>
<td>137.4</td>
<td>3.51</td>
</tr>
<tr>
<td>650</td>
<td>8.4071</td>
<td>21.9985</td>
<td>1346.50</td>
<td>138.3</td>
<td>3.49</td>
</tr>
</tbody>
</table>

The lattice parameters, unit cell volume and density of both the sintered electrolyte samples are presented in Table 1. From the table, it can be seen that the values of $a$, $c$ and $V$ for the LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 °C were 8.3880 Å, 22.0148 Å and 1341.38 Å$^3$, respectively, while for the samples sintered at 650 °C, these values were 8.4071 Å, 21.9985 Å and 1346.50 Å$^3$, respectively. At the same time, an increase in the sintering temperature from 600 to 650 °C was accompanied by an increase in the crystallite size, as evidenced by the narrowing of the peak, which indicates bigger grains [19]. Meanwhile, the densities of both the sintered electrolyte samples were more than 90 % of its theoretical density of 3.79 g cm$^{-3}$, with the highest density being that of the LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 °C, which is attributed to the compactness of the tiniest crystallites in the system [1] in accordance with the same pattern in our earlier work.

3.2 Scanning electron microscopy, energy dispersive X-ray spectroscopy and particle size distribution analysis

![Figure 2](image_url)

**Figure 2.** SEM micrographs of LiSn$_2$P$_3$O$_{12}$ samples sintered at (a) 600 and (b) 650 °C.

The SEM micrographs of the LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C are shown in Figure 2. From the SEM micrographs it can be seen that the powders in both samples are slightly agglomerated. Meanwhile, EDX spectroscopy was performed on both the LiSn$_2$P$_3$O$_{12}$ samples to
analyse their elements. The results of the EDX analysis of the samples sintered at 600 and 650 °C are presented in Table 2.

**Table 2.** EDX stoichiometric atomic ratios for LiSn₂P₃O₁₂ samples sintered at 600 and 650 °C

<table>
<thead>
<tr>
<th>Sintering Temperature (°C)</th>
<th>Composition</th>
<th>Stoichiometric atomic ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Starting mixture</td>
<td>Sn</td>
</tr>
<tr>
<td>600</td>
<td>EDX analysis</td>
<td>2.00</td>
</tr>
<tr>
<td>650</td>
<td>EDX analysis</td>
<td>2.00</td>
</tr>
</tbody>
</table>

Only the presence of Sn, P and O appear in the EDX spectra, while the light atomic weight of lithium prevents its emitted radiation from being detected by EDX. As such, the general idea of charge neutrality can be applied for this case [13], whereby the EDX ratios of the elements in all the samples correspond with the stoichiometric ratios of the starting materials.

**Figure 3.** Particle size distributions of LiSn₂P₃O₁₂ samples sintered at (a) 600 and (b) 650 °C.

The particle size distributions of the LiSn₂P₃O₁₂ samples sintered at (a) 600 and (b) 650 °C are shown in Figure 3. From the graphs it can be clearly seen that the size of the particles in LiSn₂P₃O₁₂ samples increased together with the sintering temperature. As the sintering temperature was raised from 600 °C to 650 °C, the average size of the particles in the LiSn₂P₃O₁₂ samples grew from 22.8 µm to 26.8 µm. On correlating these results with the X-ray diffraction analysis, it was found that as the sintering temperature increased, so did the crystallite size of the prepared samples. The size of the particles in both the sintered samples was also slightly higher than that of LiSn₂P₃O₁₂ samples sintered for 24 hours in our previous work [18].
3.4 Impedance Analysis

Figure 4. Cole-Cole plot of LiSn$_2$P$_3$O$_{12}$ sample at 30 °C sintered at (a) 600 and (b) 650 °C.
The Cole-Cole plots of the LiSn$_2$P$_3$O$_{12}$ sample at 30 °C are depicted in Fig. 4, with Fig. 4(a) and Fig. 4(b) representing the plots of the samples sintered at 600 °C and at 650 °C, respectively. Just like the pattern in our earlier work, both plots are comprised of two semicircles that overlap each other, with the region of low frequency being denoted by a spike [18]. The bulk response is represented by the high frequency semicircle, while its intersection with the X-axis represents the bulk resistance, $R_b$. Meanwhile, the grain boundary response is represented by the middle frequency semicircle, with its intersection with the X-axis being the grain boundary resistance, $R_{gb}$ [13]. The spike that can be clearly seen at the low frequency region of the LiSn$_2$P$_3$O$_{12}$ sample sintered at 600 °C may indicate the effects of electrode polarization as a result of the accumulation of ions between the electrode and the sample [20].

![diagram](image)

**Figure 5.** Equivalent circuit of LiSn$_2$P$_3$O$_{12}$ samples based on the impedance analysis of the samples at room temperature.

In addition, Figure 5 shows that the complex impedance data obtained experimentally for the LiSn$_2$P$_3$O$_{12}$ samples at room temperature may be approximately denoted by the impedance of an equivalent circuit comprised of bulk and grain boundary resistance $R_b$ and $R_{gb}$, respectively, and the bulk and grain boundary capacitance $C_b$ (CPE) and $C_{gb}$ (CPE), respectively, and the CPE blocking electrode with a constant phase element (CPE) behaviour [21]. The general expression of the CPE is [22-23]

$$Z_{CPE} = \frac{1}{C (j\omega)^n} \quad (3)$$

where $C$ is the ideal capacitance when $n = 1$, $j = (-1)^{1/2}$ and $\omega$ is the angular frequency, where $\omega = 2\pi f$.

**Table 3.** Ionic conductivity at 30 and 500 °C for LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C

<table>
<thead>
<tr>
<th>Sintering Temperature (°C)</th>
<th>$\sigma_{b,500}$ (S cm$^{-1}$)</th>
<th>$\sigma_{b,30}$ (S cm$^{-1}$)</th>
<th>$\sigma_{gb,30}$ (S cm$^{-1}$)</th>
<th>$\sigma_{t,500}$ (S cm$^{-1}$)</th>
<th>$\sigma_{t,30}$ (S cm$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>$1.38 \times 10^{-5}$</td>
<td>$7.22 \times 10^{-6}$</td>
<td>$2.99 \times 10^{-4}$</td>
<td>$1.38 \times 10^{-5}$</td>
<td>$2.87 \times 10^{-4}$</td>
</tr>
<tr>
<td>650</td>
<td>$1.03 \times 10^{-5}$</td>
<td>$3.28 \times 10^{-6}$</td>
<td>$1.79 \times 10^{-4}$</td>
<td>$1.03 \times 10^{-5}$</td>
<td>$1.78 \times 10^{-4}$</td>
</tr>
<tr>
<td>600 [18]</td>
<td>$1.40 \times 10^{-5}$</td>
<td>$1.05 \times 10^{-6}$</td>
<td>$4.24 \times 10^{-4}$</td>
<td>$1.40 \times 10^{-5}$</td>
<td>$4.08 \times 10^{-4}$</td>
</tr>
<tr>
<td>650 [18]</td>
<td>$3.64 \times 10^{-3}$</td>
<td>$1.15 \times 10^{-6}$</td>
<td>$5.79 \times 10^{-8}$</td>
<td>$3.64 \times 10^{-3}$</td>
<td>$5.51 \times 10^{-8}$</td>
</tr>
</tbody>
</table>
The bulk and grain boundary conductivities, $\sigma_b$ and $\sigma_{gb}$, respectively can then be computed from the Cole-Cole plot by means of the following equations:

\[ \sigma_b = \frac{d}{AR_b} \quad \text{and} \quad \sigma_{gb} = \frac{d}{AR_{gb}} \] (4)

where $d$ is the thickness of the sample, $A$ is the area of the cross-section of the sample, $R_b$ is the bulk resistance and $R_{gb}$ is the grain boundary resistance. Table 3 compares the values of both $\sigma_b$ and $\sigma_{gb}$ at 30 and 500 °C for the LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C with the values obtained for the samples in the earlier study. For the LiSn$_2$P$_3$O$_{12}$ sample sintered at 600 °C, the bulk conductivity, $\sigma_b$ increased from $7.22 \times 10^{-6}$ S cm$^{-1}$ at 30 °C to $1.38 \times 10^{-5}$ S cm$^{-1}$ at 500 °C, while for the LiSn$_2$P$_3$O$_{12}$ sample sintered at 650 °C, the bulk conductivity, $\sigma_b$ at 30 °C and 500 °C were $3.28 \times 10^{-6}$ S cm$^{-1}$ and $1.03 \times 10^{-5}$ S cm$^{-1}$, respectively. The highest total conductivity, $\sigma_t$ was $1.38 \times 10^{-5}$ Scm$^{-1}$ at 500 °C for the LiSn$_2$P$_3$O$_{12}$ sample sintered at 600 °C, which is one order of magnitude above that reported by Norhaniza et al. (2010) and is of the same order of magnitude as reported in our earlier work [18]. By sintering the sample at 600 °C, high density pellet with smaller grains was produced, thus resulting in a higher ionic conductivity value compared to that of the other samples. Furthermore, the LiSn$_2$P$_3$O$_{12}$ sample sintered at 600 °C in this work possessed a higher ionic conductivity compared to that of the sample in a previous work by Norhaniza et al. (2010), where traces of impurity still remained. However, the highest total conductivity, $\sigma_t$ found in the LiSn$_2$P$_3$O$_{12}$ sample sintered at 600 °C in this work was slightly lower than that obtained in our previous work, although it was still in the same order of magnitude. The impurity that was present in the samples in our previous work might have contributed to the total electronic conductivity.
Figure 6. Arrhenius plot showing (a) bulk and (b) grain boundary conductivity of LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C.

Figure 6 presents the Arrhenius plot of the (a) bulk and (b) grain boundary conductivity of the LiSn$_2$P$_3$O$_{12}$ samples sintered at 600 and 650 °C. The Arrhenius equation below was used to derive the activation energy, $E_a$ for the Li$^+$ migration:

$$\sigma = \sigma_0 \exp\left(\frac{-E_a}{kT}\right)$$

where $\sigma$ is the conductivity, $T$ is the absolute temperature, $\sigma_0$ is the pre-exponential factor, $E_a$ is the activation energy for the Li$^+$ ion migration and $k$ is the Boltzmann’s constant. Similar to our previously reported samples [18], two regions can be seen in the Arrhenius plots. The first region, region I can be associated with low temperatures, while region II can be associated with high temperatures. Table 4 gives the values of the bulk and grain boundary activation energies for both regions. The bulk activation energy for the sample sintered at 600 and 650 °C for region I was 0.005 and 0.010 eV, respectively, while the bulk activation energy for the sample sintered at 600 and 650 °C for region II was 0.250 and 0.330 eV, respectively. The same trend applied for the grain boundary activation energy for both samples. Furthermore, the activation energy reported here was much lower than that obtained in other studies as well as in our previous work [8, 11, and 18]. This low activation energy value is typical of a fast ion conductor, and is attributed to the uniform diffusion channels in the NASICON structure [19].
3.6 Electrochemical Stability Window Analysis

![Graph showing linear sweep voltammogram of LiSn$_2$P$_3$O$_{12}$ samples sintered for 48 hours at 600 °C and 650 °C.]

**Figure 7.** Linear sweep voltammogram of LiSn$_2$P$_3$O$_{12}$ samples sintered for 48 hours at (a) 600 and (b) 650 °C.

A linear sweep voltammogram was employed in this work to examine the decomposition voltage [24] of the LiSn$_2$P$_3$O$_{12}$ electrolyte samples. The linear sweep voltammogram of the LiSn$_2$P$_3$O$_{12}$ sample sintered at (a) 600 and (b) 650 °C at room temperature is shown in Figure 7. It can be seen from the figure that the LiSn$_2$P$_3$O$_{12}$ sample that was sintered at 600 °C began to decompose at 4.8 V, while the sample that was sintered at 650 °C began to decompose at a lower voltage of 4.6 V. The
value obtained was higher compared to that of the LiSn_2P_3O_12 samples sintered at 600 °C for 24 hours, which was only 4.4 V [18]. This shows that LiSn_2P_3O_12 samples with less impurity have a wider electrochemical stability window compared to those samples that contain impurity.

4. CONCLUSIONS

The citric acid assisted sol-gel method was used to successfully synthesize rhombohedral NASICON-structured LiSn_2P_3O_12 samples with a longer sintering time of 48 hours. The formation of the rhombohedral (R_3c) phase of the LiSn_2P_3O_12 samples sintered at 600 and 650 °C was verified by XRD and EDX analysis since the longer sintering time resulted in the decomposition of the SnP_2O_7 impurity. The crystallite size produced by the LiSn_2P_3O_12 samples sintered at 600 °C was smaller than those produced by the samples sintered at 650 °C. At the same time, at 500 °C the sample sintered at 600 °C had the highest bulk conductivity of 10^{-6} S cm^{-1} at 30 °C and 10^{-5} S cm^{-1} at 500 °C in comparison to the sample sintered at 650 °C. The bulk activation energy was 0.005 eV for the sample with the highest conductivity value for region I (low temperature) and 0.250 eV for region II (high temperature). Besides that, the voltage stability window for both the sintered samples was relatively high at room temperature, reaching 4.8 V and 4.6 V for the samples sintered at 600 °C and 650 °C, respectively. Thus, the current results indicate that LiSn_2P_3O_12 has a good potential to be employed as a solid electrolyte in electrochemical appliances such as lithium batteries.

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