Advantage of solution methods towards synthesis of \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \)

Jelena Bijelčič, Dalibor Tatar, Anna-Marija Milardović, Antonia Vicić, Anamarija Stanković, Pascal Cop, Sebastian Werner, Zvonko Jagličič

1Department of Chemistry, Josip Juraj Strossmayer University of Osijek, Osijek, Croatia
2Institute of Physical Chemistry, Justus Liebig University of Giessen, Giessen, Germany
3Institute of Mathematics and Physics & Mechanics, University of Ljubljana, Ljubljana, Slovenia
4Faculty of Civil and Geodetic Engineering, University of Ljubljana, Slovenia

Jelena.Bijelic@kemija.unios.hr | bijelic@chem.hr

INTRODUCTION

The properties of materials have appeared to be size- and shape-dependent which is why in this work we compare two synthesis routes: sol-gel solution synthesis and solid-state synthesis of triple \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \) perovskite. This material has been prepared in form of semi- spherical particle agglomerates using a modified aqueous sol-gel citrate route (nitrate salts dissolved in 10% solution of citric acid) and as a bulk material using solid state synthesis by means of planetary ball milling.

EXPERIMENTAL

SOLUTION METHOD

- \( \text{NH}_3 \) (w = 25%)
- Stirring
- Heating (95 °C)
- Grinding
- Calcination (600 °C)
- Calcination (1000 °C)

SOLID STATE METHOD

- Mixing carbonate and oxide reactants
- 15 min, 250 rpm
- Calcination (1350 °C)
- Calcination (1400 °C)
- Calcination (1650 °C)

RESULTS AND DISCUSSION

Table 1. Phase composition of the samples synthesized by sol-state route.

<table>
<thead>
<tr>
<th>Calcination temperature (°C)</th>
<th>Phase composition (wt. %)</th>
<th>Crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050</td>
<td>( \text{Sr}_3\text{Fe}_2\text{WO}_9 ) (48%) + ( \text{Fe}_2\text{O}_3 ) (21%) + ( \text{Fe}_3\text{O}_4 ) (13%) + ( \text{SrWO}_4 ) (17%)</td>
<td>Medium</td>
</tr>
<tr>
<td>1350</td>
<td>( \text{Sr}_3\text{Fe}_2\text{WO}_9 ) (64%) + ( \text{Fe}_2\text{O}_3 ) (36%)</td>
<td>Good</td>
</tr>
<tr>
<td>1400</td>
<td>( \text{SrFe}_2\text{WO}_9 ) (82%) + ( \text{Fe}_3\text{O}_4 ) (18%)</td>
<td>Bad</td>
</tr>
</tbody>
</table>

Figure 1. PXRD patterns of the samples synthesized by solid-state route.

Figure 2. Rietveld output plots of the phase pure \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \) prepared by sol-gel route. The observed, calculated and difference plots for the fit to the PXRD patterns are shown along with Bragg reflections of identified phases (vertical ticks).

Figure 3. Selected TEM and HRTEM images recorded at different magnifications for \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \) synthesized by sol-gel route.

Figure 4. Magnetic susceptibility versus temperature (left) and magnetization measurements (right) for the compound synthesized by sol-gel method reveal ferrimagnetic behavior with Curie temperature of 213 K. There is no "standard" phase transition with sharply defined transition temperature, but the system goes gradually from paramagnetic to ferrimagnetic state.

CONCLUSIONS

In this research comparison has been made between solution chemistry routes and solid-state routes towards the synthesis of triple perovskite \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \). It has been revealed that solid-state route produced the targeted compound but with impurities. Sol-gel solution method produced phase pure nanocrystalline \( \text{Sr}_3\text{Fe}_2\text{WO}_9 \) which presents a tetragonal lattice, space group \( \text{P}4/m \) with \( a = 5.5724(1) \) and \( c=7.9022(2) \) Å. It shows ferrimagnetic behavior and gradual rather than usual sharp phase transition which occurs due to nanocrystallinity. Advantages of sol-gel synthesis used in this reasearch are also shorter reaction time and lower reaction temperatures.