

July 20, 2018

Dear Dr. Phillip Steindel,

We thank the referees and the production team for the comments expressed for our manuscript (Manuscript No: JoVE58482R1) entitled “Molten-Salt Synthesis of Complex Metal Oxide Nanoparticles” and proceed to address them as follows.

Editorial and production comments:

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.

Thank you. The manuscript has been proofread by all authors to avoid spelling or grammar issues.

2. Figure 1: Please include a space between numbers and their units (i.e., 6 h, 650 °C).

Thank you for the comment. Figure 1 has been modified as suggested in the revised manuscript.

3. Please upload Table 1 to your Editorial Manager account as an .xls or .xlsx file.

Thank you. Table 1 has been converted to .xlsx format and uploaded.

4. Keywords: Please provide at least 6 keywords or phrases.

Thank you. A sixth keyword “precursor” has been added.

5. Please use SI abbreviations for all units: L, mL, µL, h, min, s, etc.

Thank you. All units have been corrected to SI abbreviations.

6. Please include a space between all numbers and their corresponding units: 15 mL, 37 °C, 60 s; etc.

Thank you. A space has been included as suggested.

7. Please revise the protocol to contain only action items that direct the reader to do something (e.g., “Do this,” “Ensure that,” etc.). The actions should be described in the imperative tense in complete sentences wherever possible. Avoid usage of phrases such as “could be,” “should be,”

and “would be” throughout the Protocol. Any text that cannot be written in the imperative tense may be added as a “Note.” Please include all safety procedures and use of hoods, etc. Please move the discussion about the protocol to the Discussion.

Thank you. All protocol steps have been revised accordingly. All needed safety procedures have been added. In addition, all the discussion about the protocol has been moved to the Discussion.

8. 1.3.2: What is the total volume of ammonia solution added?

Thank you for the question. The total volume of ammonia solution is 200 mL. This information has been added to the revised manuscript.

9. 1.4.1: What is the pore size of the filter paper?

Thank you for the question. The filter paper used for the precursor filtration step is made of cellulose fiber suitable for gravity and vacuum filtration systems. The porosity of the filter paper is coarse (40-60 μm) with a particle retention of 20–25 μm and a flowrate 160 mL/min. The information pertaining to the pore size has been added in our manuscript “Vacuum filtration and drying of precursor” section. It reads as “1.4.1) Vacuum filter the coprecipitated solution using a filter paper with a coarse porosity (40-60 μm) (Fisherbrand, P8) to separate the solid precipitate from the supernatant.”

10. 2.1.1: Please specify the mass measured.

Thank you. The measured mass has been specified in the revised manuscript. Now it reads as:
2.1.1) Measure 30 mmol (3.033 g) of potassium nitrate (KNO_3) and 30 mmol (2.549 g) of sodium nitrate (NaNO_3).

2.1.2) Combine the measured salts with 0.35 g of the as-prepared single-source complex precursor $\text{La}(\text{OH})_3 \cdot \text{HfO}(\text{OH})_2 \cdot n\text{H}_2\text{O}$.

11. 2.1.4: Please specify the volume of acetone or ethanol added.

Thank you. The volume of acetone or ethanol added has been provided in the revised manuscript.

12. References: Please do not abbreviate journal titles. Please include volume and issue numbers for all references.

Thank you for the comment. All references have been updated as suggested.

Changes to be made by the Author(s) regarding the video:

1. Titles of the manuscript and the video do not match.

Thank you for catching the error, which has been corrected in the revised manuscript.

2. 00:32: Please change ml to mL.

Thank you for the comment. Now ml has been changed to mL in the video.

3. 01:05-02:24, 05:09-06:35: Please remove the introduction and discussion about the protocol from the protocol section. Protocol should contain only action items that direct the reader to do something.

Thank you for the comment. Both the Introduction and Discussion about the protocol have been removed from the protocol section.

4. 03:08: This specific detail “Add diluted ammonia solution to the burette” in the video is not stated in the written manuscript.

Thank you for the comment. The detail has been added to the revised manuscript. It reads as “1.3.1) Add the diluted ammonia solution prepared in the previous step into a burette, and ensure that the burette is covered at all times, since ammonia solution tends to evaporate which decreases its concentration.”

5. Please upload a revised high-resolution video here:
http://www.jove.com/files_upload.php?src=17837058

A revised high-resolution video has been added accordingly.

Video quality issues

- The videography quality does not meet our standards. Overall, there is too much camera movement. Additionally, many of the actions are out of focus and several of the shots are underexposed. This video would need to be reshot using static framing and accurate focus.

Thank you for the comments. Shots have been retaken accordingly as seen in the revised high-resolution video.

Editing issues

- 0:24, 6:36 - The audio and video are fading out while the speaker is still speaking. This should happen after they are finished.

Thank you for the comment. This issue has been solved accordingly.

Frame size/proportions issues

- 7:49-9:00 - The white backgrounds of these figures should be extended to fill the frame.

Thank you for the comment. A full-screen white background has been added to all figures.

Reviewers' comments:

Concerns regarding Reviewer #1:

1. While the manuscript/video are well made, one area which could be improved on is the discussion of common mistakes which may be made in the synthesis. This could be important if the reader/viewer is attempting to make a material not directly covered. Specifically, it would be useful if the authors discussed some pitfalls which may occur during both synthesis steps which might lead to impurities or unwanted products. (Manuscript)

We thank the reviewer for the encouraging comments and suggestion. We have modified our manuscript in the Limitation of the MSS method section accordingly and added the following: “Furthermore, common mistakes that could hinder the quality of the final nanoparticle products include the following: first is not covering the burette during the titration, which changes the concentration of the ammonium hydroxide and eventually changes the size of obtained nanoparticles. Another common mistake is not to give the coprecipitation a period of two hours to form. Adding the ammonia titrant too fast affects the coprecipitation kinetics which might render inhomogeneous complex precursor. The third pitfall is not to grind the salt(s) and precursor(s) as fine as possible, which generate impure products or inhomogeneous particles.”

2. There are sections of the video where there is a transition from the speaker to an experiment. In these sections, it is difficult to hear what the speaker is saying. It may be good to slightly re-edit the video or to add captions so that everything is clear. (Video)

We thank the reviewer for the comments. The video has been re-edited to improve the transition and clarity as suggested.

3. One section of the text lists the need to identify the crystal structure using Raman (line 239). This appears a bit out of the blue but it is discussed two paragraphs later. It would be good to mention earlier in the text that there are multiple phases of these materials and the authors are focusing on the pyrochlore phase. (Manuscript)

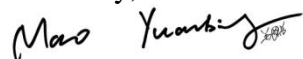
We thank the reviewer for the suggestion. We have modified the manuscript and now it reads as “The as-synthesized $\text{La}_2\text{Hf}_2\text{O}_7$ NPs could exist in the ordered pyrochlore phase. However, chemical doping, pressure, temperature could modify the phase to defect fluorite. It is possible for our material to have multiple phases but in this paper, we focus only on the pyrochlore phase for simplicity. X-ray diffraction (XRD) and Raman spectroscopy have been used to systematically characterize their phase purity, structure and phase.”

4. It would be good to discuss in more detail the role of adding solvents to the grinding process prior to the molten salt reaction. Does this change the morphology? How much should be added? etc.(Manuscript)

We thank the reviewer for the comment and questions. A new paragraph has been added to the Critical step section in the revised manuscript. It reads as “Finally, the addition of a volatile liquid such as acetone and ethanol during the mixing of the salts used and the single-source complex precursor helps ease the grinding process but is not essential. The addition of a liquid is helpful to make a homogenous mixture in shorter time and less effort, which is important to generate pure products. The added volatile liquid does not affect the characteristics of the resultant NPs since it fully evaporates by the end of the grinding process. Due to its high volatility, the amount added could range anywhere from 1 mL to 5 mL.”

Please feel free to contact me in the event of any questions or concerns.

Sincerely,



Yuanbing Mao, Ph.D.

Associate Professor

Department of Chemistry

University of Texas Rio Grande Valley

1201 West University Drive

Edinburg, TX 78539-2999

Phone: 956-665-2417

Email: yuanbing.mao@utrgv.edu

Blog: maolab.blogspot.com