Synthesis of $La_{.5}Sr_{.25}Ca_{.25}MnO_3$ nanoparticles

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Given Task

- Create $La_{.5}Sr_{.25}Ca_{.25}MnO_3$ (Lanthanum Strontium Calcium Manganite or LSCMO) nanoparticles

Outline

1. Reading Materials (Introduction)
2. Making Samples (Solid State)
3. Sol-Gel Method
4. XRD (X-ray Diffraction) and SEM (Scanning Electron Microscope) of nanoparticles
5. Conclusion

Reading Materials: Main points

- Ceramics fired at high temperatures calcify, creating microcrystals
- Difficult to make nanoparticles from these microcrystals
- Best manner of producing nanoparticles would be a solution-based reaction

Microcrystal

Nanoparticle

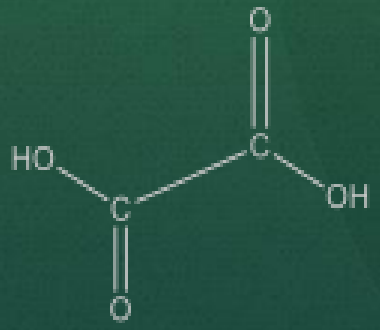
Making Samples (solid-state): Summary

- Took powders (La_2O_3, $SrCO_3$, $CaCO_3$ and MnO_3) and pressed pellets
- $4La_2O_3 + 5SrCO_3 + 5CaCO_3 + 20MnO_3 \rightarrow 20La_{.4}Sr_{.25}Ca_{.25}MnO_3 + 10CO_2 + O_2$
- Heated at various temperatures (up to 1500°C)

Sol-Gel Method

Basic Premise

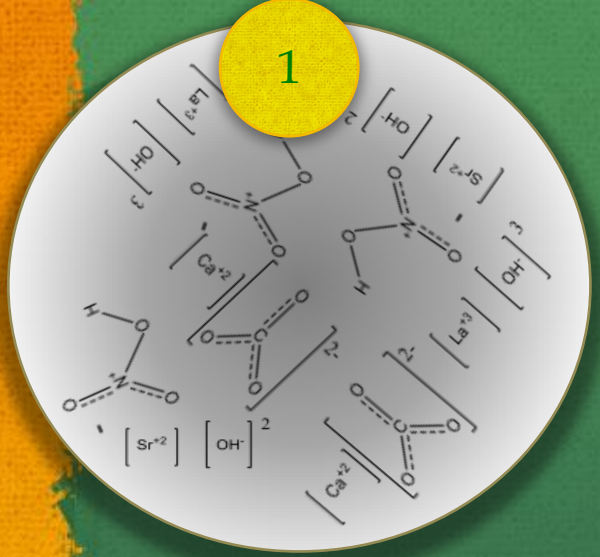
- Form nitrates and mix with coordinating compound
- Utilize coordinating compound to heat at low temperatures
- Low temperature heating means we get nanoparticles instead of microcrystals
- Coordinating compound: Oxalic Acid

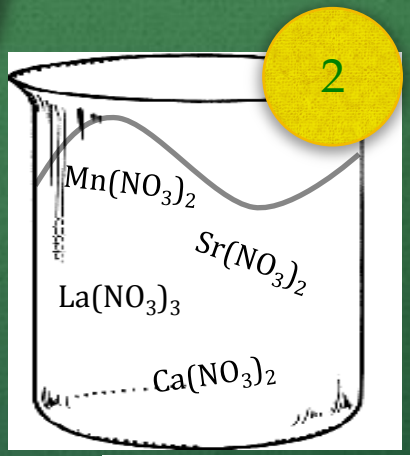


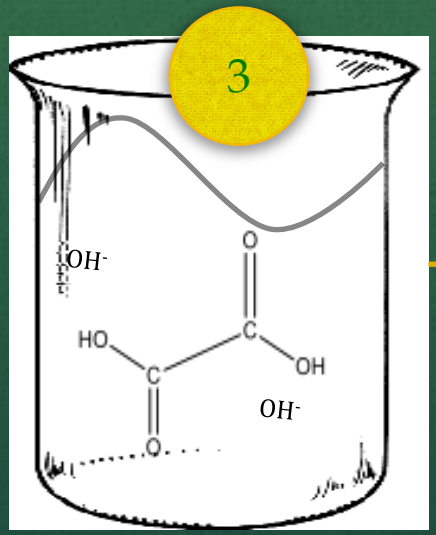
(1) Dissolve powders

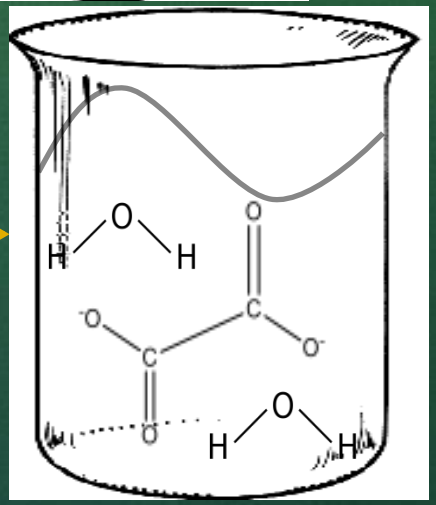
(2) Mix together nitrates (add $Mn(NO_3)$)

(3) Dissolve oxalic acid in 20 mL of DI water

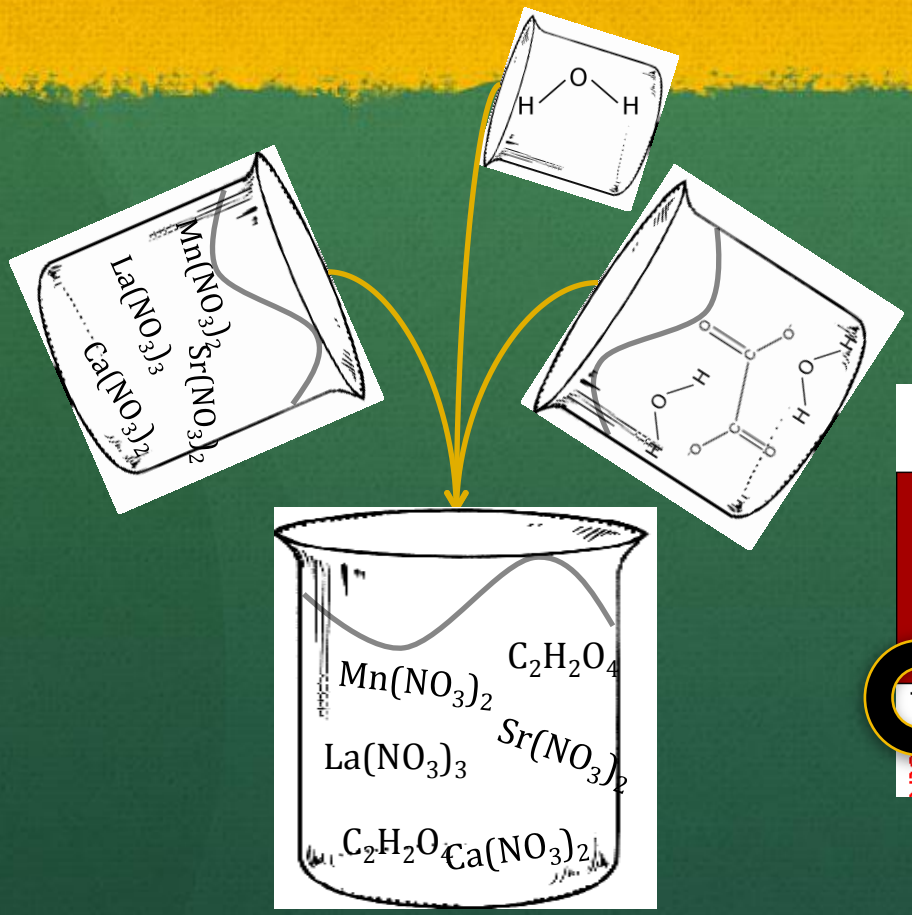






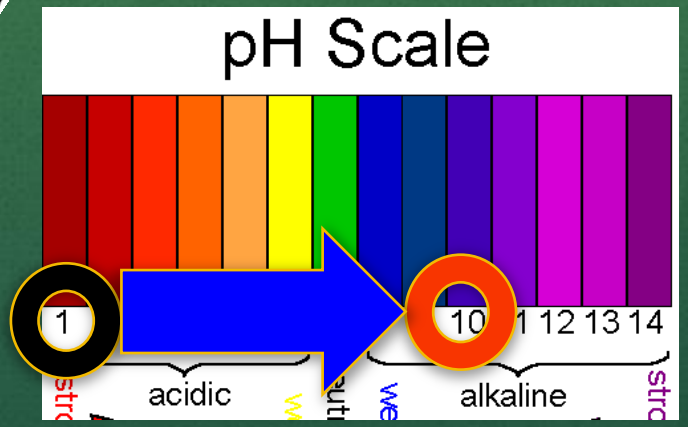


(4) Mix together the two solutions



- Add 20mL water DI water to dilute

(5) Adjust the pH using Ammonium Hydroxide



- Go from pH 1 to pH 10

(6) Sonicate for 20 minutes at 30°C

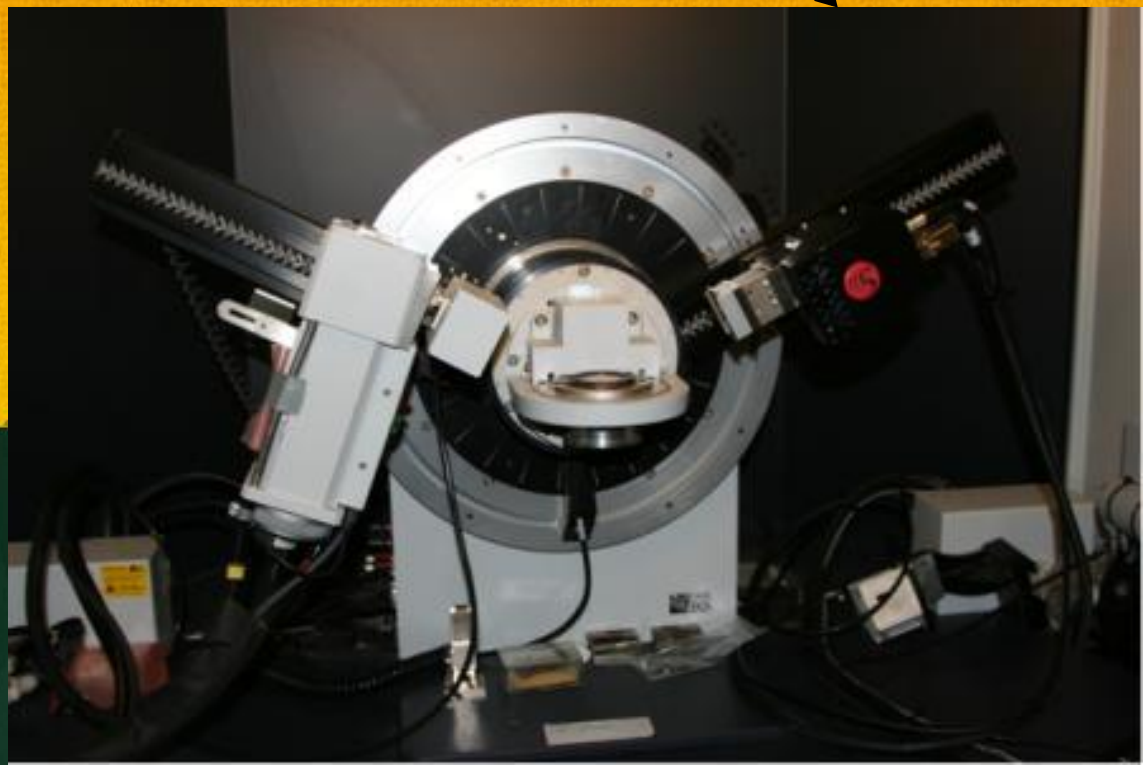
(7) Rotor evaporate for about an hour (until dry)

(8) Heat in vacuum oven overnight

(9) Heat in vented furnace

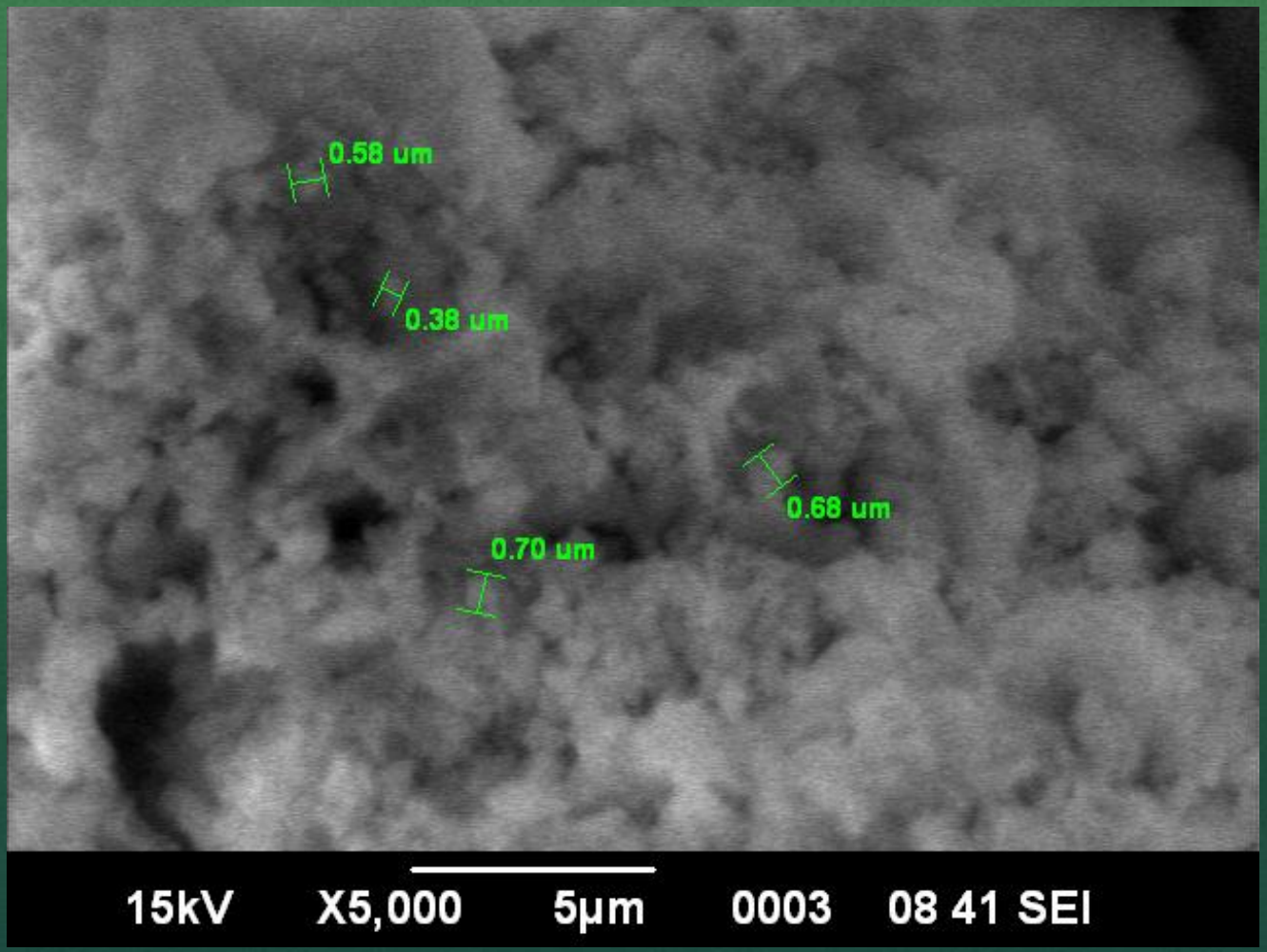
- Place the gels in the furnace hot at 600°C, for 12 hours

SEM and XRD of nanoparticles

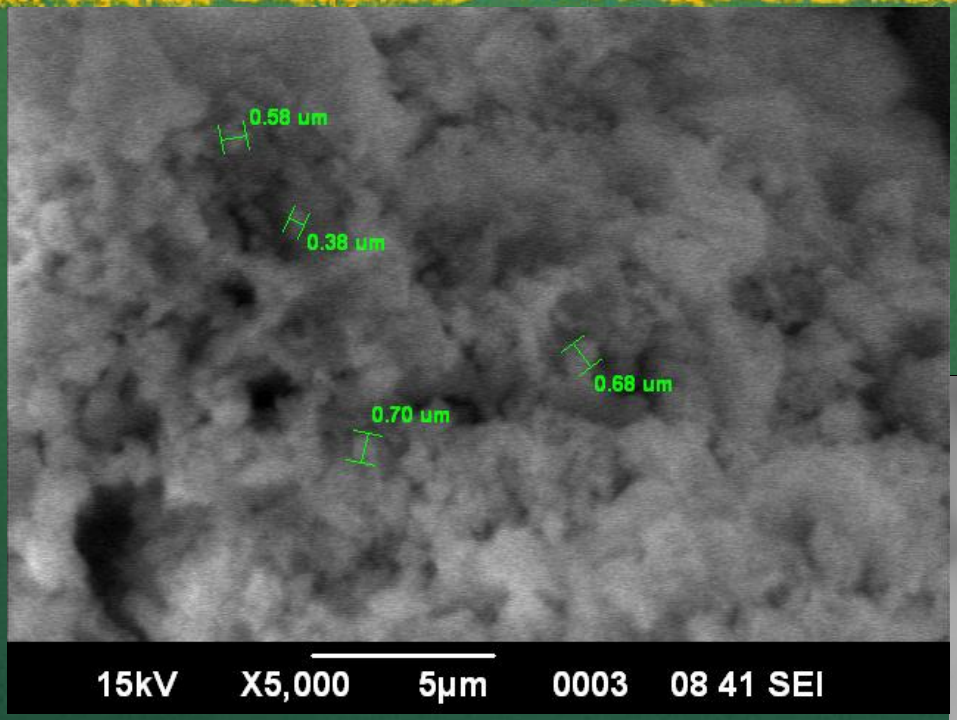


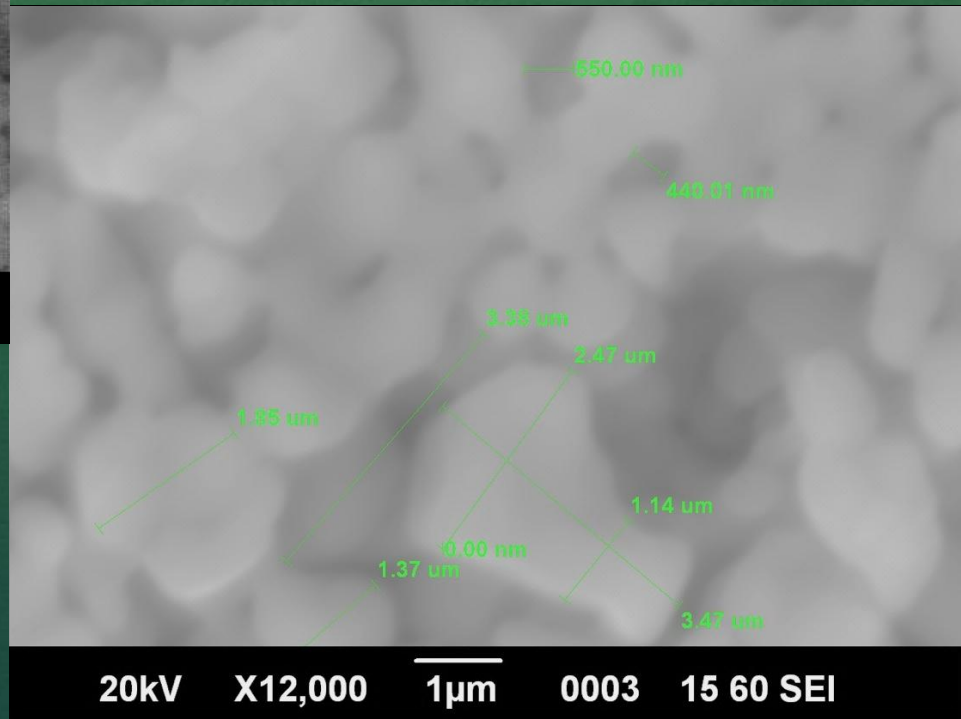
Scanning Electron Microscope (SEM) of sol-gel method

- Flowering suggests existence of nanoparticles



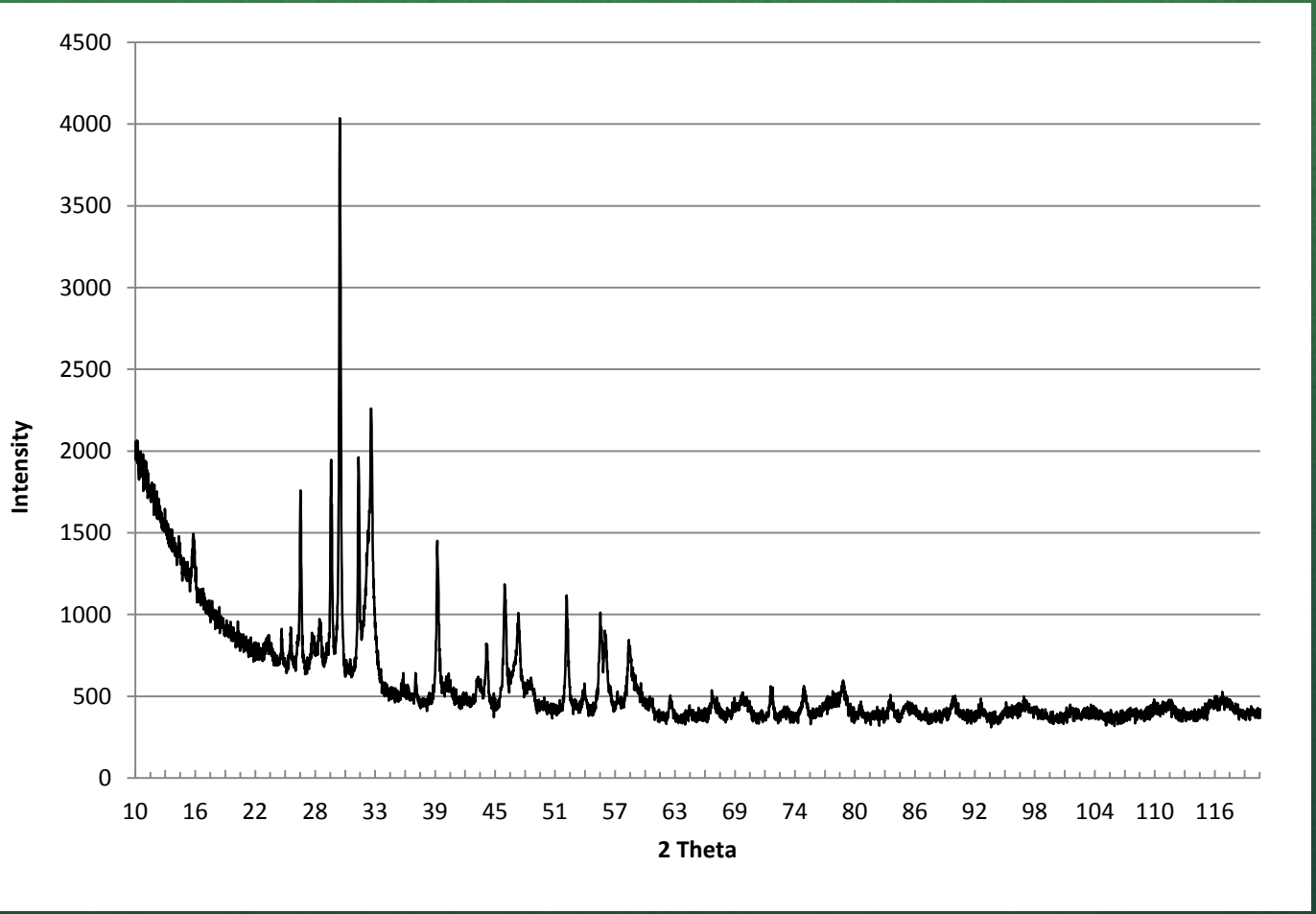
SEM of sol-gel sample and microcrystals (solid-state sample)





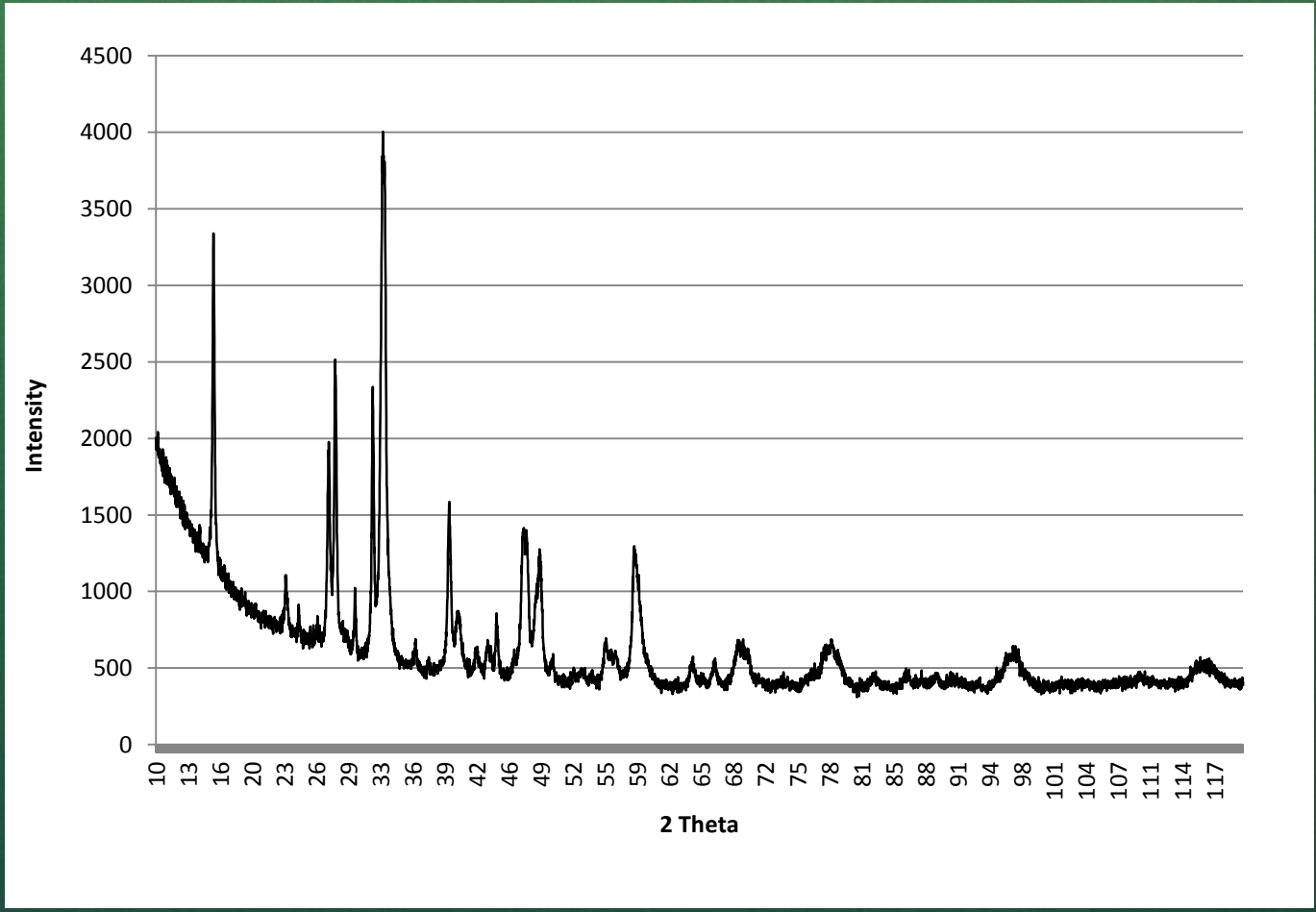
X-ray Diffraction (XRD) after heating sol-gel at 600°C

- Very wide peaks indicate nanoparticles
- Large quantity of peaks shows phase impurity
- More heat treatment!

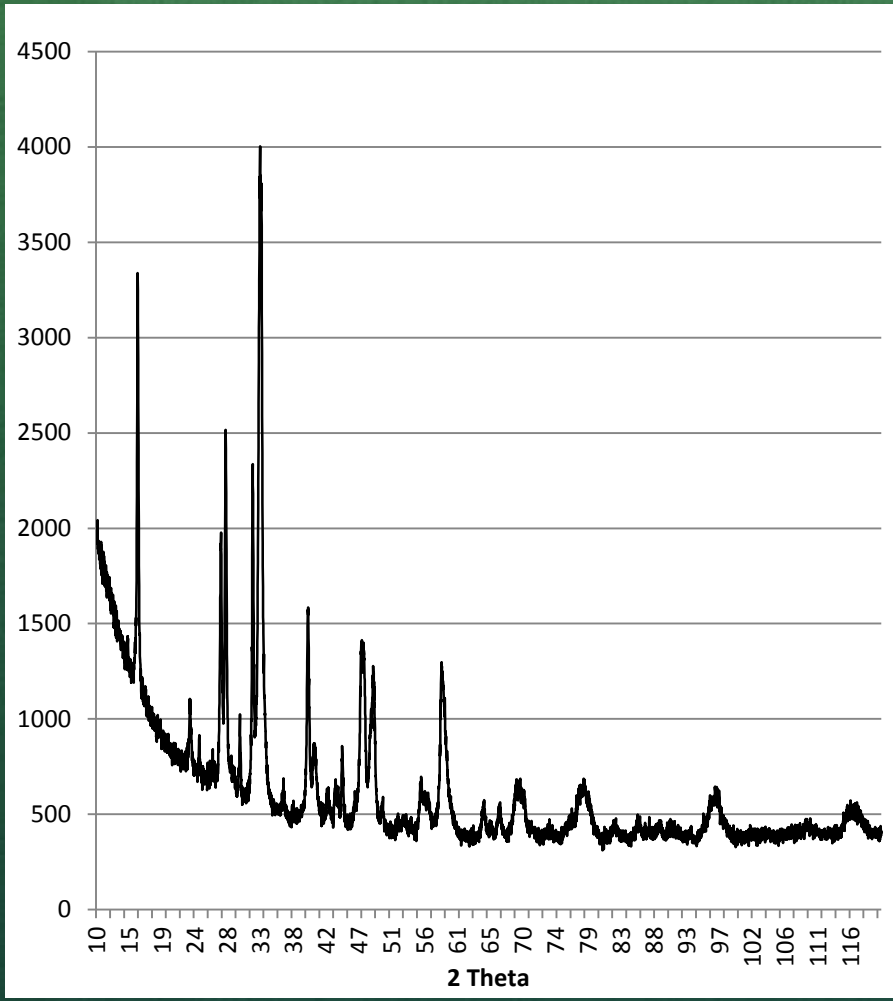


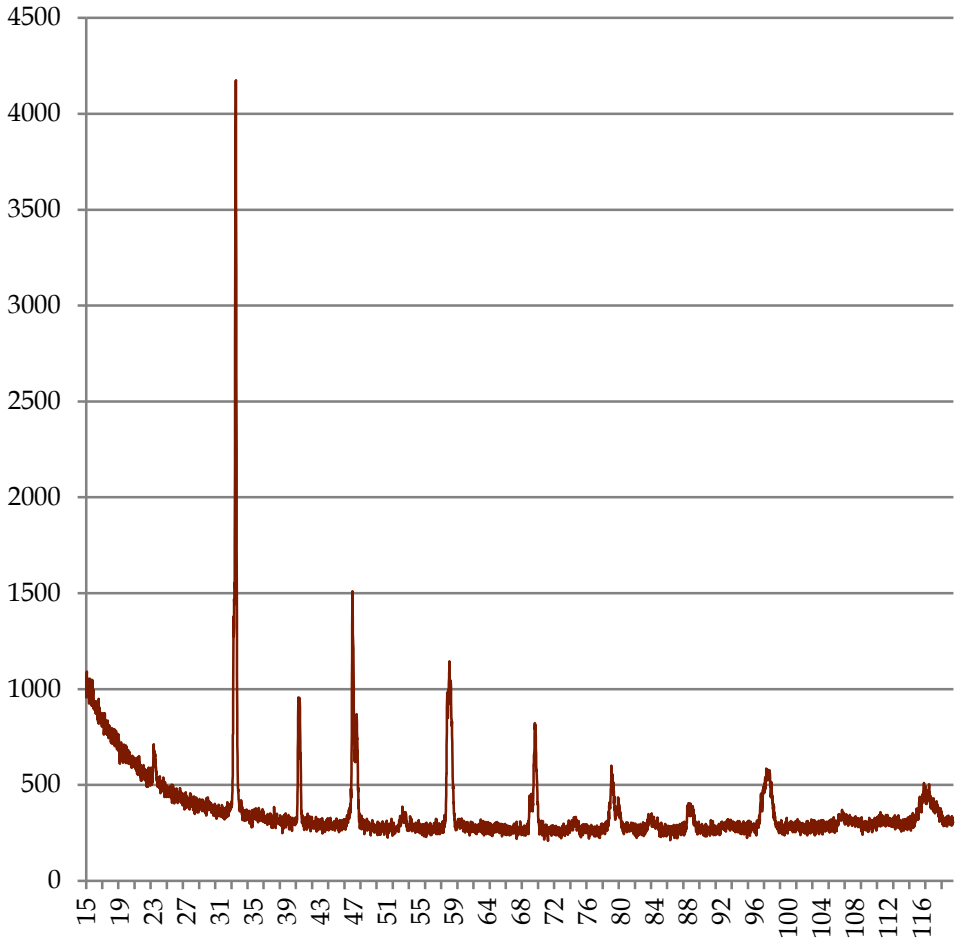
XRD after heating at 900°C under O_2 partial pressure

- Still nanoparticles (notice peak width)
- More phase pure but may still require more heat treatment



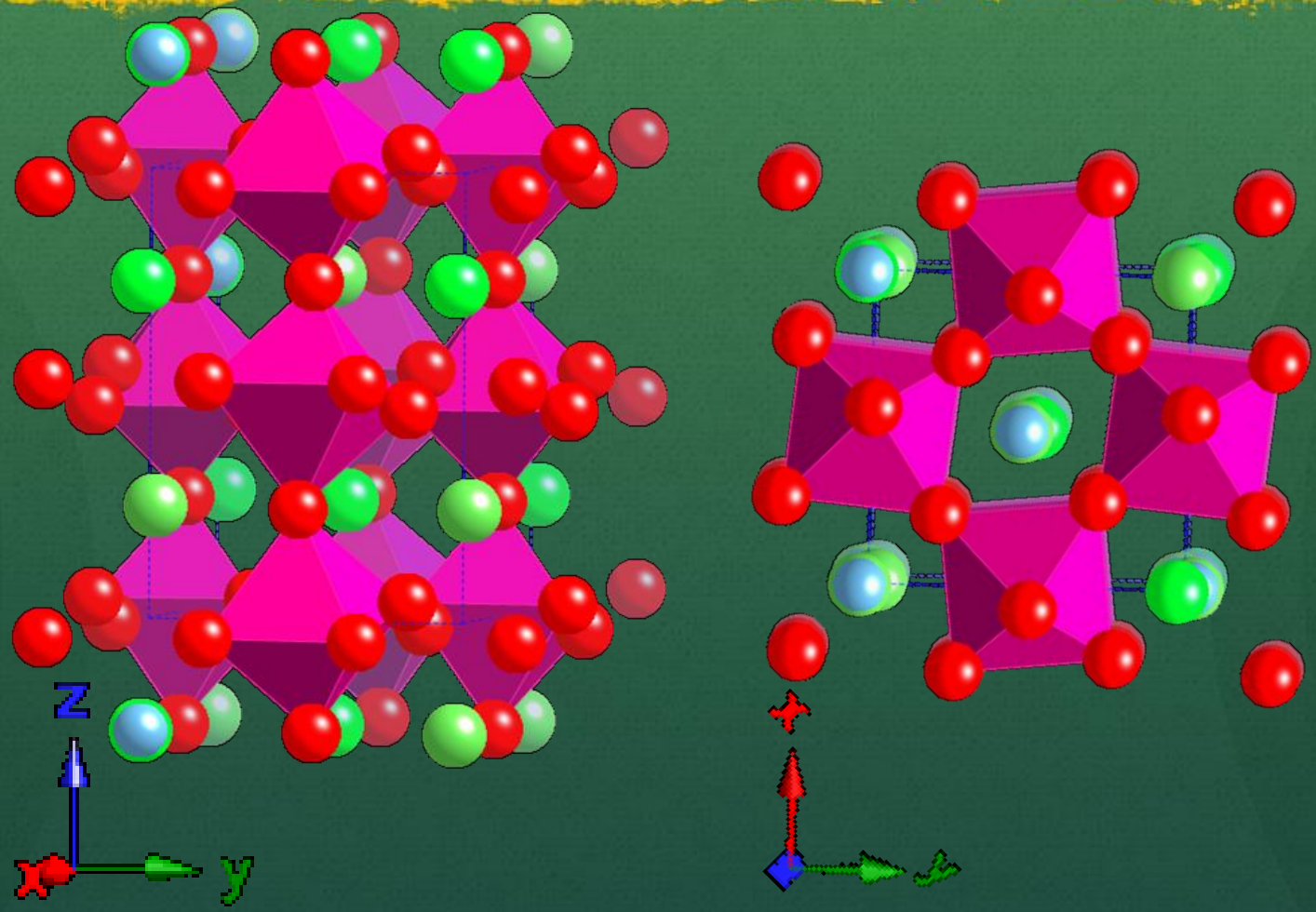
XRD of nanoparticles (left) and microcrystals (right)





Crystal Structure

- Red= O
- Magenta= Mn
- Green and Blue = La, Sr, and Ca



Conclusion

Outcomes

- Lots of pellets (most of them from the solid-state method)
  - Pellets ready for characterization tests (i.e. CMR tests)
- Two methods of synthesis
- An understanding of the crystal structure
- A whole lot of learning

Next step(s)

- Perform the characterization tests
  - Using a SQUID (Superconducting quantum interference device), MPMS (Magnetic property measurement system) or some electrochemical measuring device
- Refine the synthesis process
  - Use FTIR (Fourier transform infrared) to check for functional groups and forward reactions (helps with pH adjustment)
  - Use AAS (Atomic absorption spectrum) and XRF (X-ray Fluorescence) to identify the exact stoichiometry
  - Use TEM (Tunneling electron microscope) to measure particle size
  - Perform more heat treatments under O_2 partial pressure

Thanks for listening

Big thanks to Dr. Scott Wicker and Dr. L. L. Henry, as well as to the entire LA-SiGMA program and everyone here at Southern University!

Any questions?

Sources

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