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Control of Phase of Iron Oxide

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CONTROL OF PHASE OF IRON OXIDE

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Outline

- Phases of Iron Oxide
- Common Synthesis Methods
- Experimental
- Characterization
- Future Work

Phases of Iron Oxide

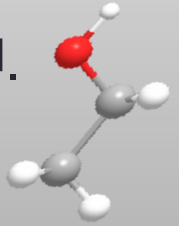
- Three primary oxidation states of Iron Oxide
- Iron(II) oxide
  - Wüstite - FeO
- Iron (III) oxide
  - Hematite - α-Fe_2O_3
  - Maghemite - γ-Fe_2O_3
- Iron (II,III) oxide
  - Magnetite - Fe_3O_4
- Iron Oxide has a number of applications
  - Magnetic properties
  - Photocatalysis
  - Energy applications

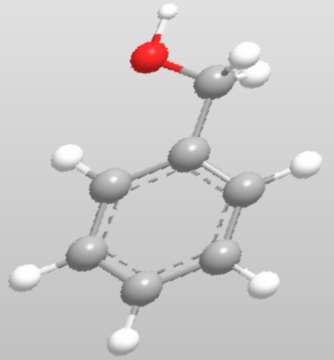
Common Synthesis of Iron Oxides

- Early methods employ aqueous co-precipitation methods
  - pH > 11
  - Metal halide salt precursor
  - Surfactants to control size and morphology

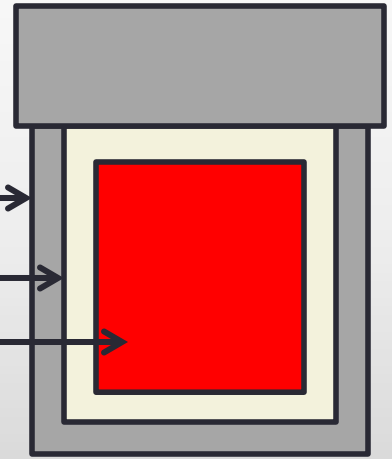
- Pinna *et al.* (2005) developed a surfactant free one step solvothermal synthesis of Fe_3O_4 & γ-Fe_2O_3[1].
  - Iron(III)Acetylacetonate and benzyl alcohol

- Qian *et al*. (2012) hybridized Fe_3O_4 on graphene[2].
  - Iron(III)Acetylacetonate and ethanol





Solvothermal Synthesis



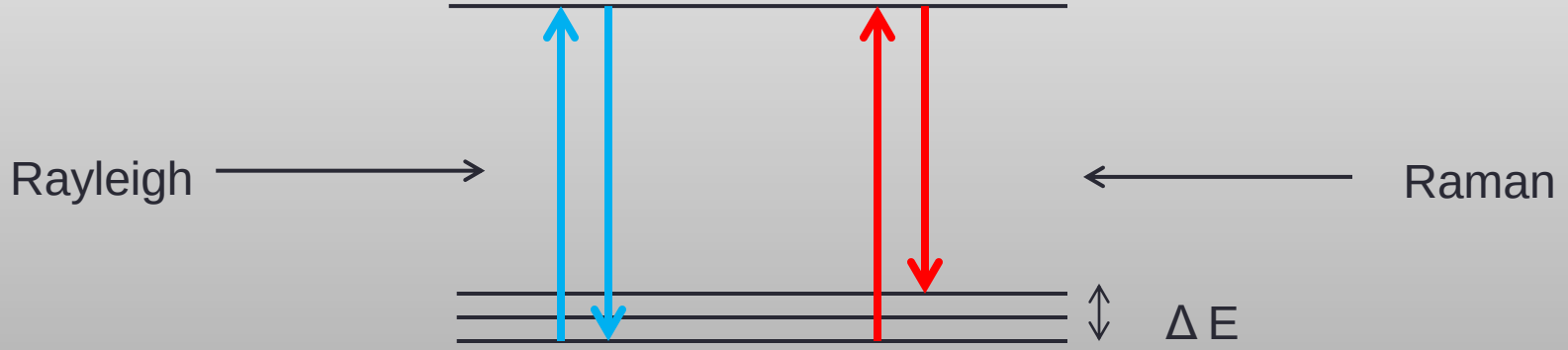
- Method of nanoparticle production
  - Stainless steel autoclave
  - Teflon liner
  - Solvent/Precursor solution
- Creates internal pressure within the autoclave
  - Pressure arises solely from heating solvent past the boiling point
- Variables of solvothermal synthesis
  - Temperature
  - Solvent/precursor solution
  - Synthesis time

Preparation of Nanoparticles

- 5 mM solution of $Fe(Acac)_3$ and solvent (ethanol or acetone)
- 1 mg of few layer graphene
- Mixture transferred to a Teflon liner and stainless steel autoclave
- Heated for 24 hours at varied temperatures
  - 70°C & 180°C
- Precipitate centrifuged at 14800 rpm for 15 minutes

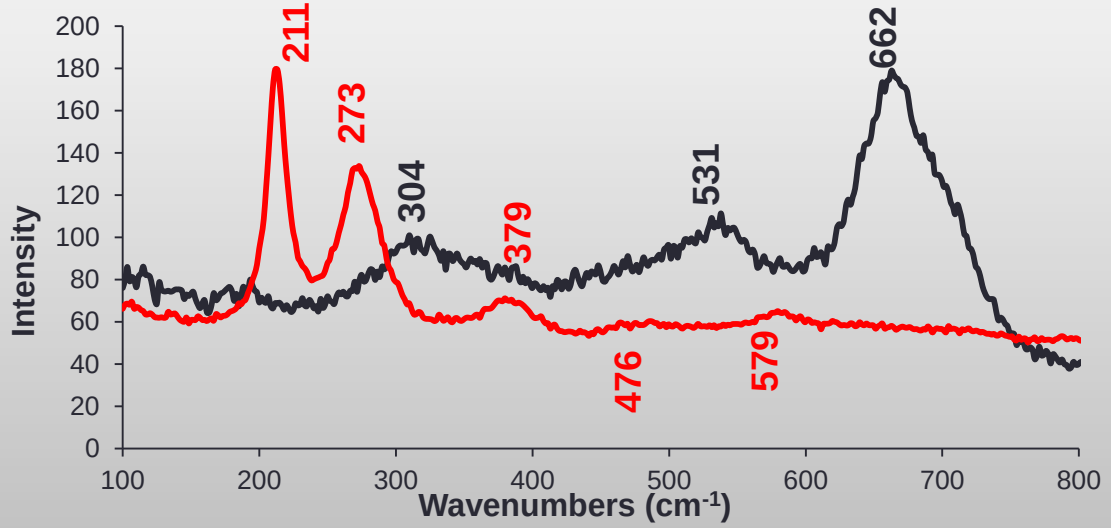
Raman Spectroscopy

- Raman is a scattering spectroscopy arising from a change in polarizability of a molecule
- Generally light scattering is elastic
  - Rayleigh scattering- no change in energy and frequency
- A small amount of light will result in inelastic scattering
  - Raman scattering- change in energy and frequency

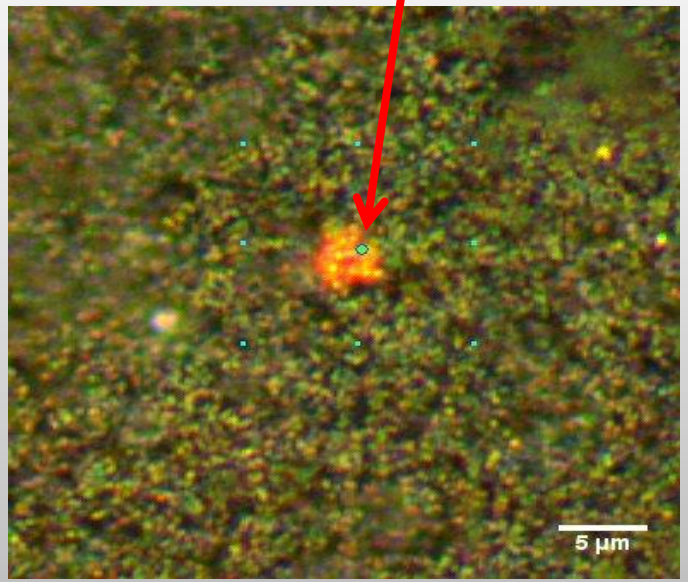


Effect of Laser on Fe_3O_4

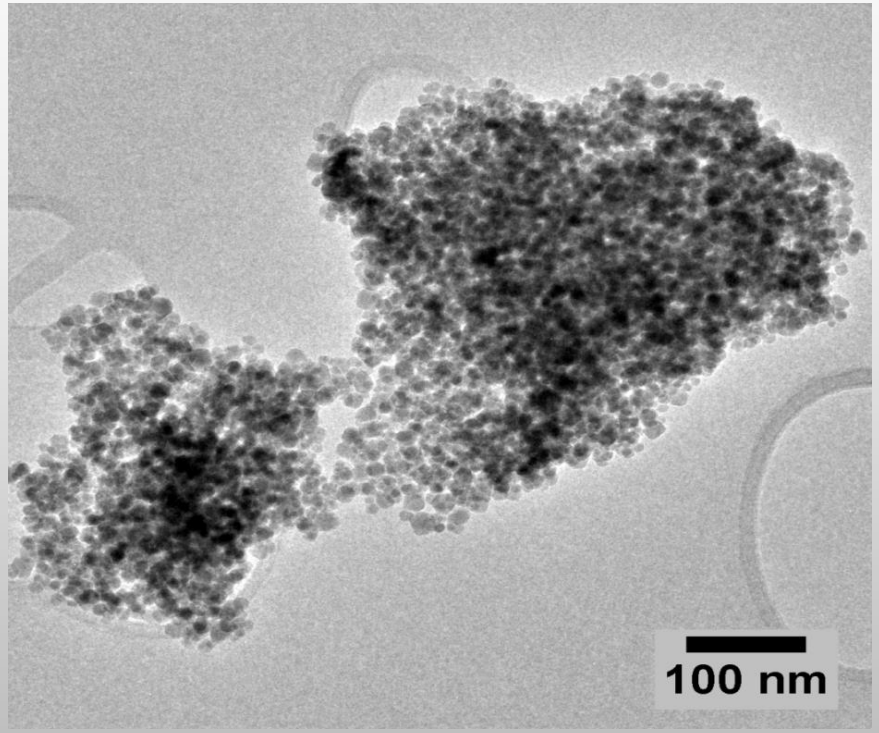
Raman laser power < 1 mW,
Raman laser power > 1 mW

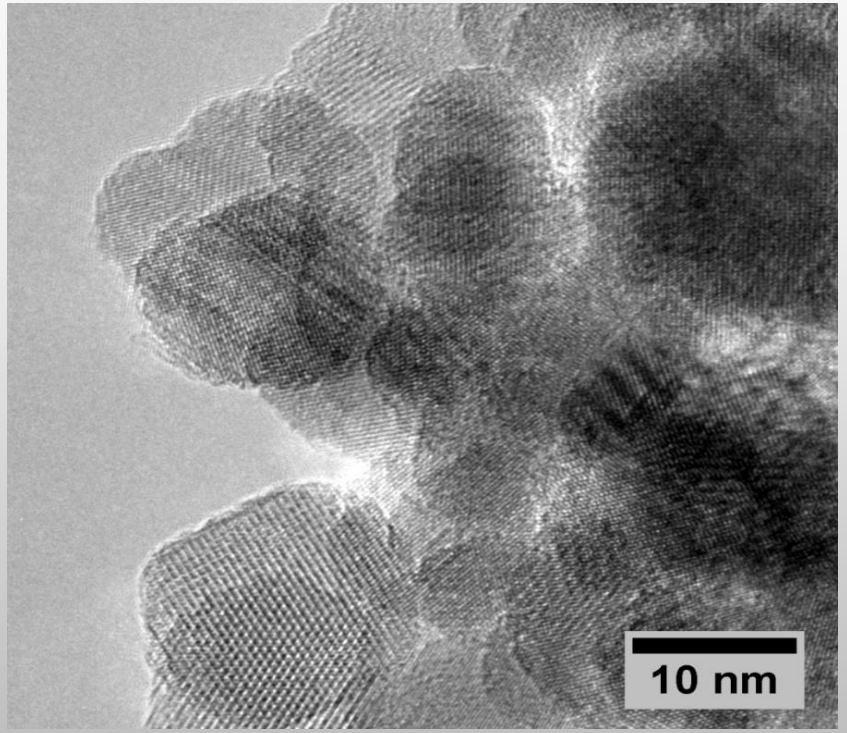


Area exposed to laser



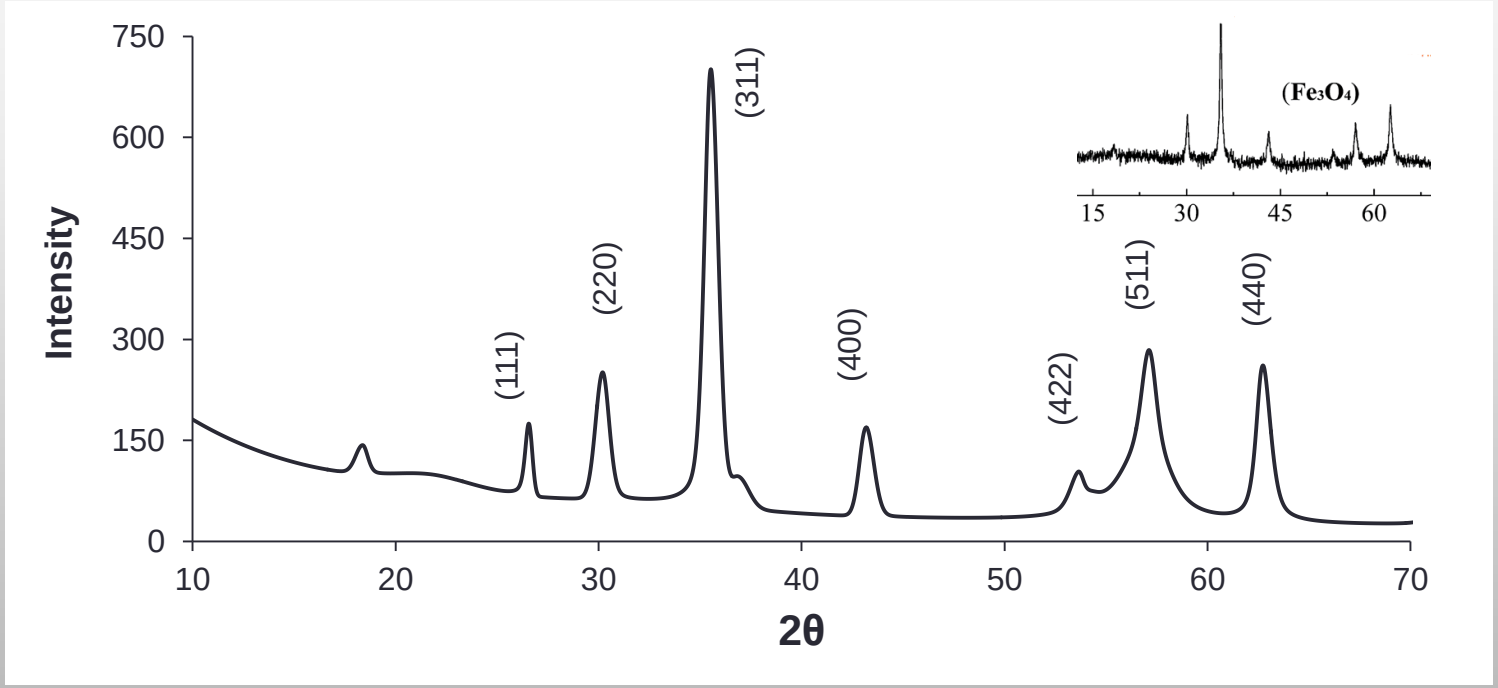
TEM Fe_3O_4





TEM of nanoparticles, reaction in ethanol at 180°C for 24 hours

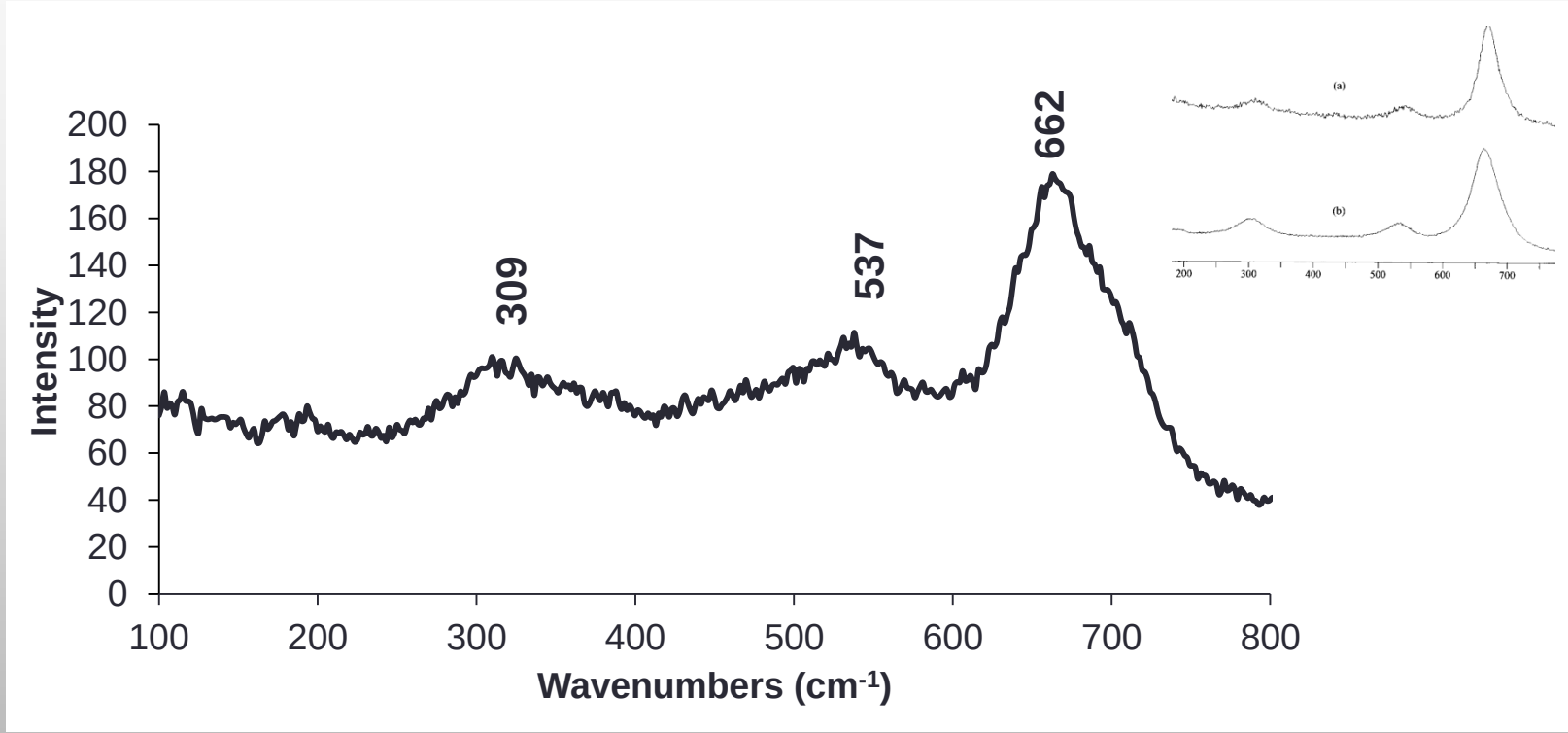
X-Ray Diffraction of Iron Oxide



- θ
  - 26.5° (111)
  - 30.1° (220)
  - 35.5° (311)
  - 43.0° (422)
  - 53.3° (511)
  - 57.0° (511)
  - 62.6° (440)

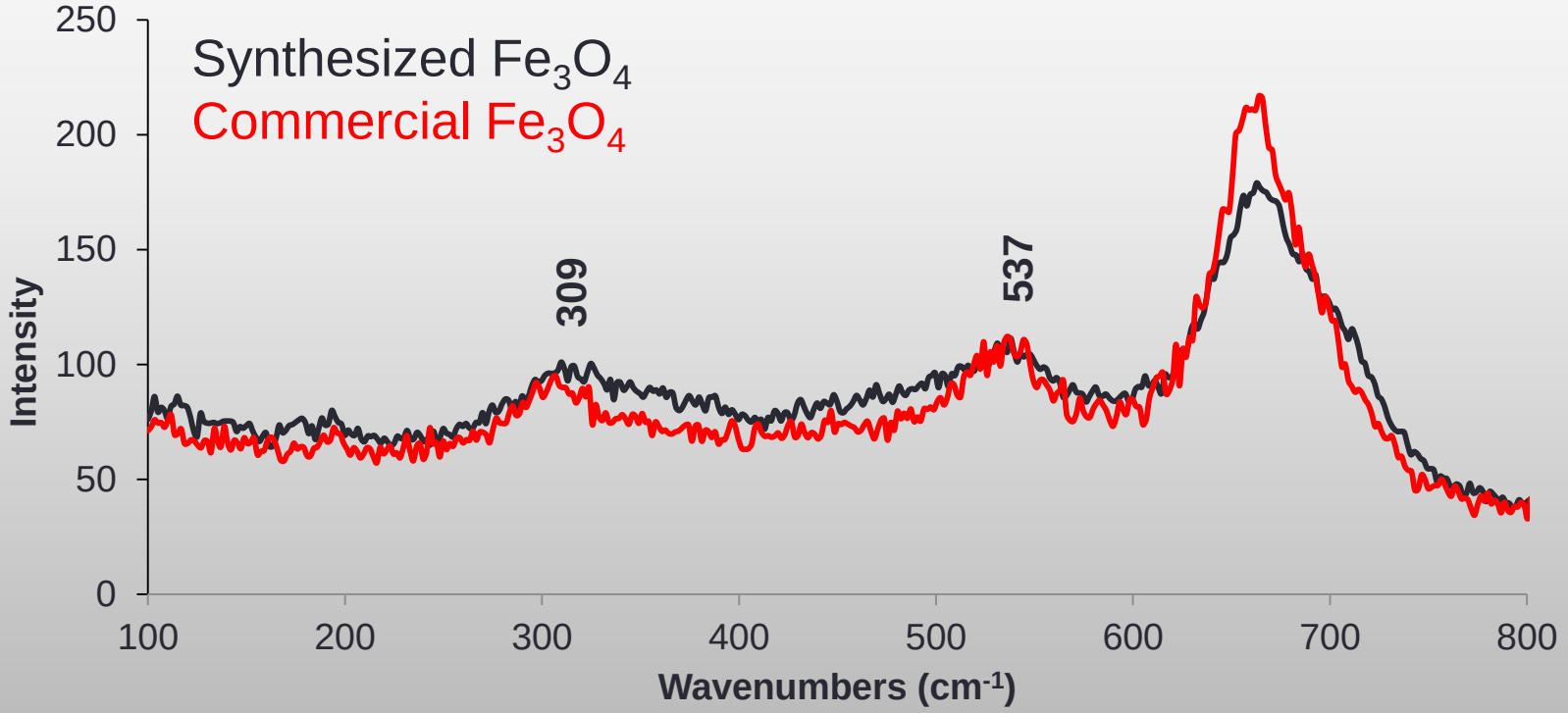
XRD spectra characteristic of the spinel structure[3]

Ethanol Reaction at 180°C

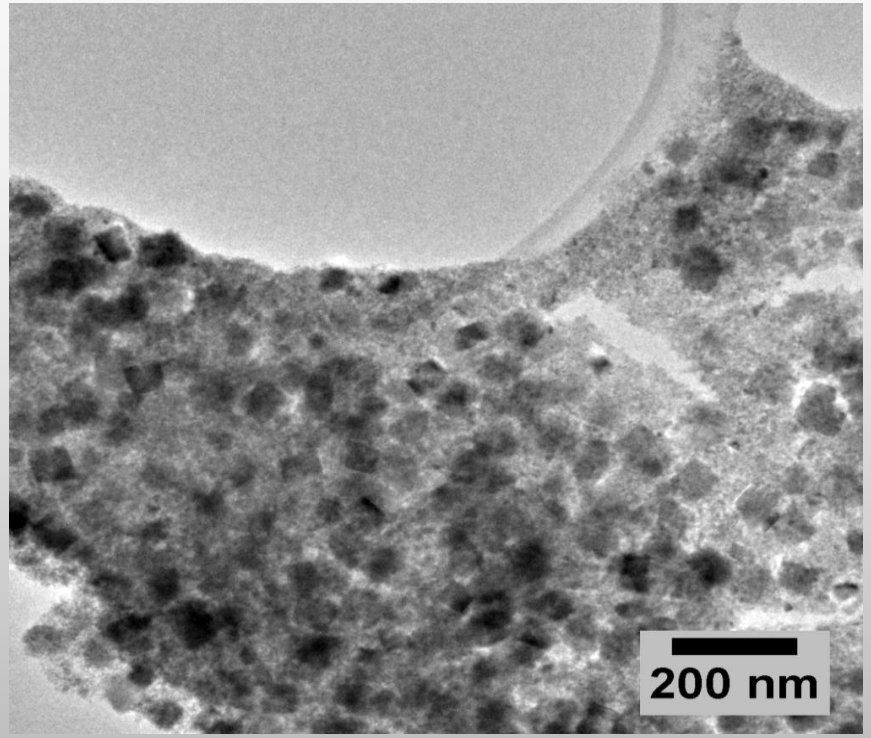


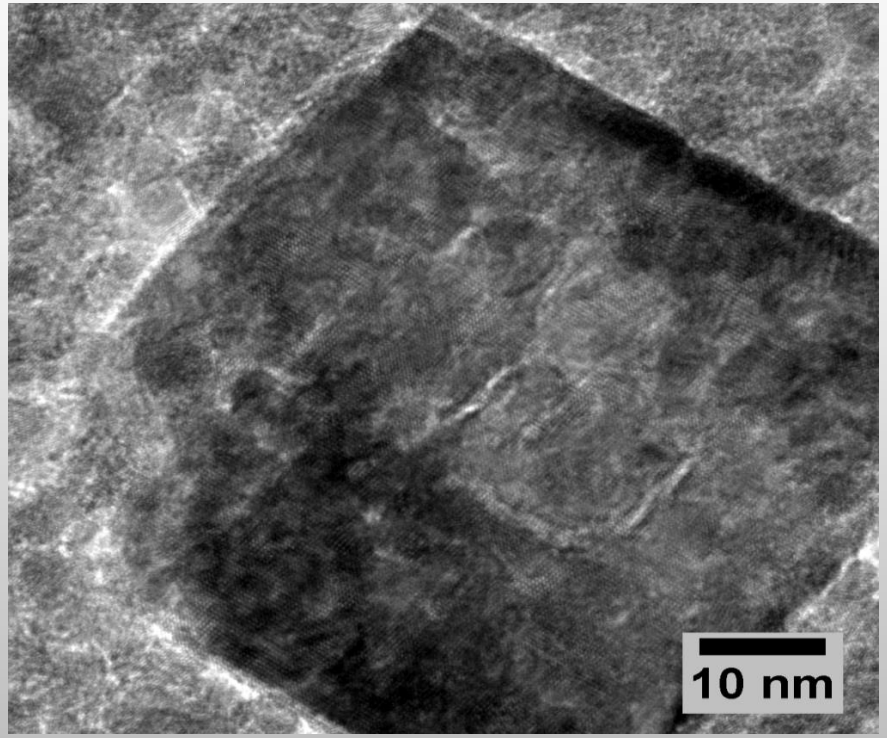
Raman spectra characteristic of Fe_3O_4[4]

Commercial vs. Synthesized



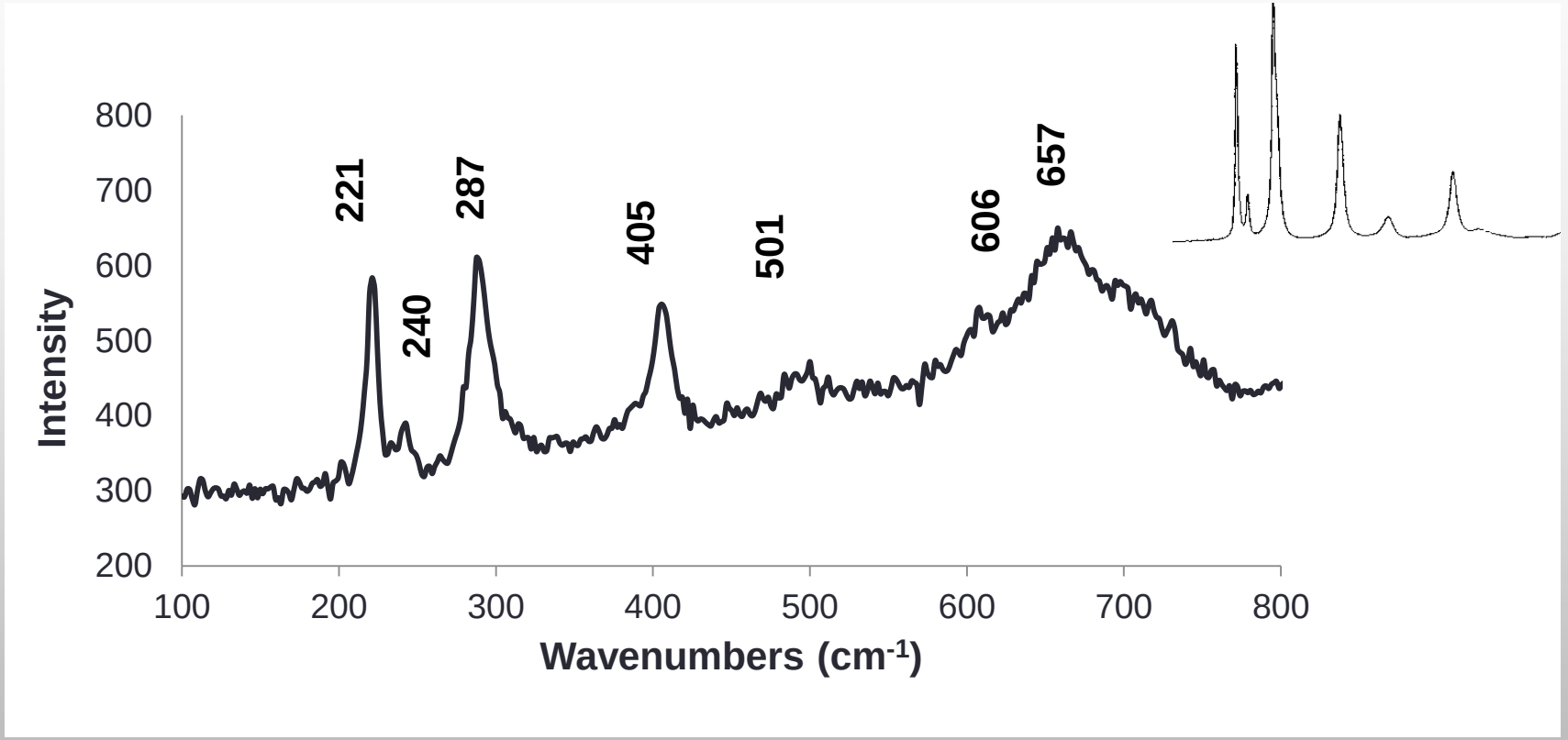
TEM α-Fe_2O_3





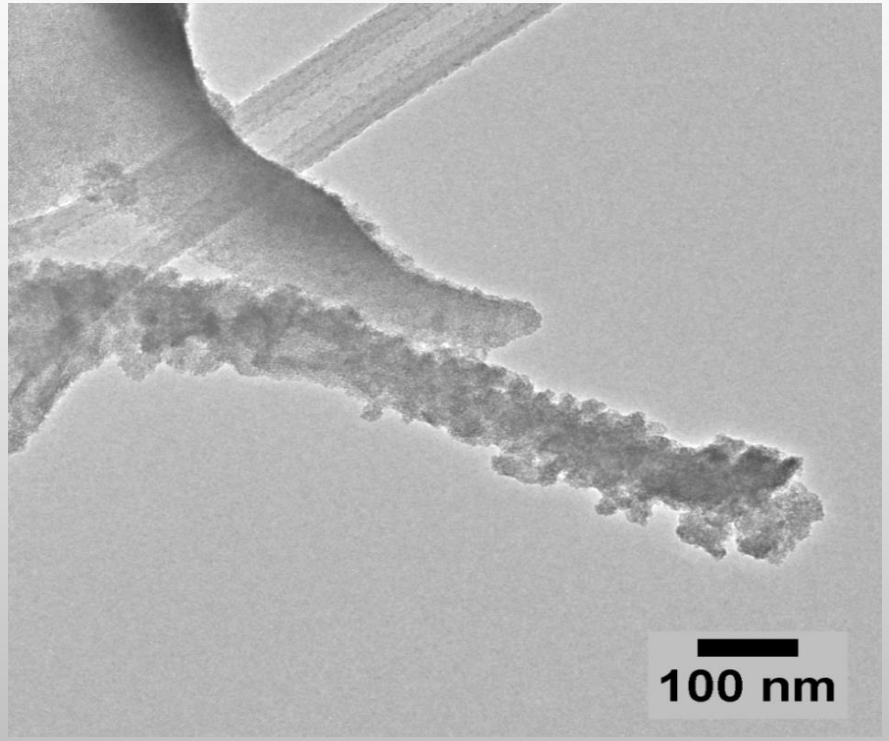
TEM of nanoparticles, reaction in acetone at 180°C for 24 hours

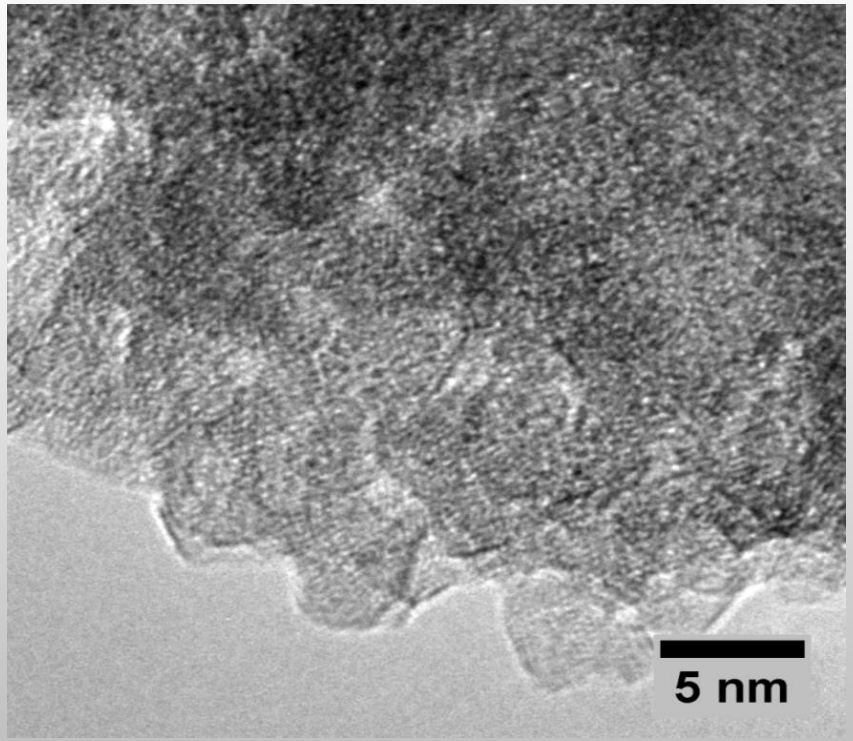
Acetone reaction at 180°C



Raman spectra characteristic of α-Fe_2O_3[4]

TEM α-Fe_2O_3





- TEM of nanoparticles, reaction in acetone at 70°C for 24 hours

Future Work

- Obtain XRD spectrum of α-Fe_2O_3
- Extend reaction time of acetone solvothermal to 48 hours
- >200°C temperature synthesis in both solvents

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Citations

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