EFFECT OF SYNTHESIS TIME ON FORMATION OF FE_3O_4 NANOPARTICLES USING THE SOLVOTHERMAL PROCESS

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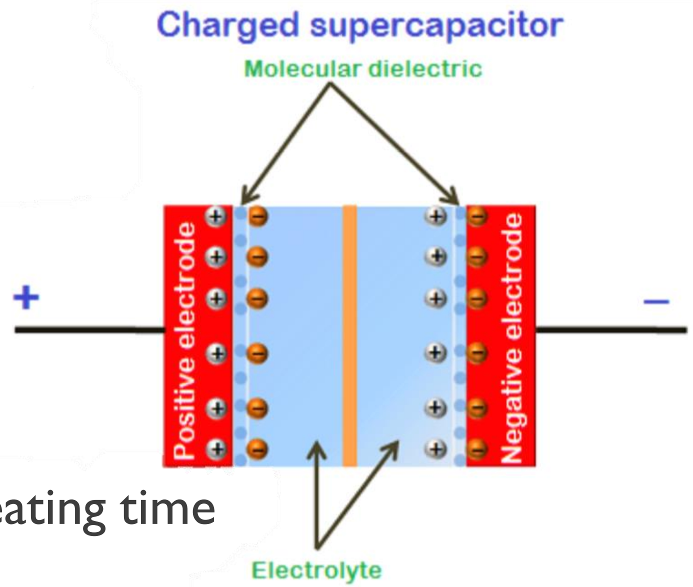
OVERVIEW

- Introduction
- Method
- Results
- Discussion
- Conclusion

INTRODUCTION

- Transition oxide nanoparticles (NPs) exhibit high magnetic signals and can be altered by external magnetic fields
  - Unique physical and chemical properties; lighter and stronger; high surface area-volume ratio
  - Magnetite (Fe_3O_4) NPs are ferrimagnetic
  - Very abundant, environmentally friendly, and can be "grown" rather than built
  - Functional groups can be used for more sensitive biolabeling, biodelivery, and higher contrast MRIs
- Hybridized graphene and iron oxide NPs have improved specific capacitance and less electrical resistance
  - Graphene has a high surface area and electrical conductivity
    - Can serve as substrate and conductive pathway for NPs

INTRODUCTION CONT.



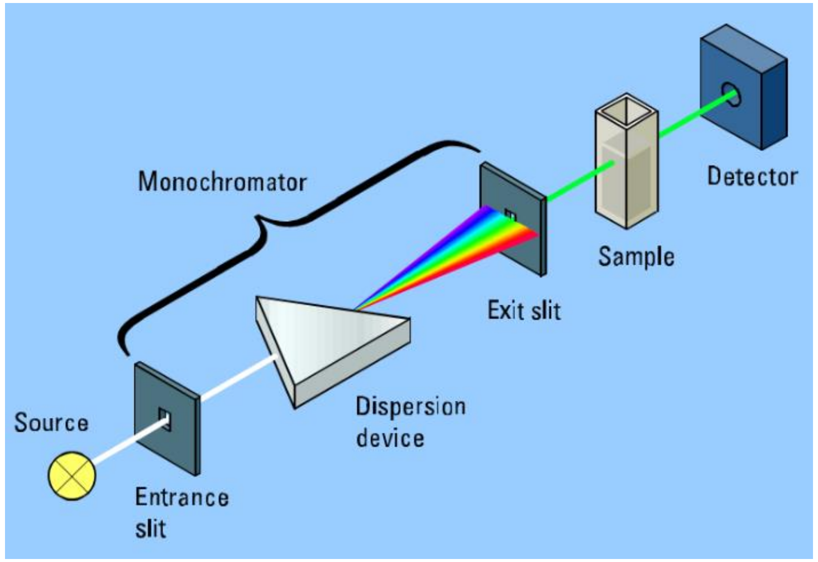
- Hybrid used as electrode material in supercapacitors/double-layer capacitors
  - More energy efficient than traditional batteries and capacitors
  - Builds up charges on two metal plates to store static electricity
  - Creates thin double-layer of charge
- Properties of NPs altered by precursor material, heating temperature, and heating time
- Size, shape, and distribution largely impact its magnetic properties
- One-step procedure produces NPs attached to the graphene surface
  - Hypothesized that nuclei are absorbed into spherical NPs through intermolecular forces
- **Purpose**: to explore the effect of synthesis time on altering the size and distribution of iron oxide nanoparticles on graphene sheets

METHOD OVERVIEW

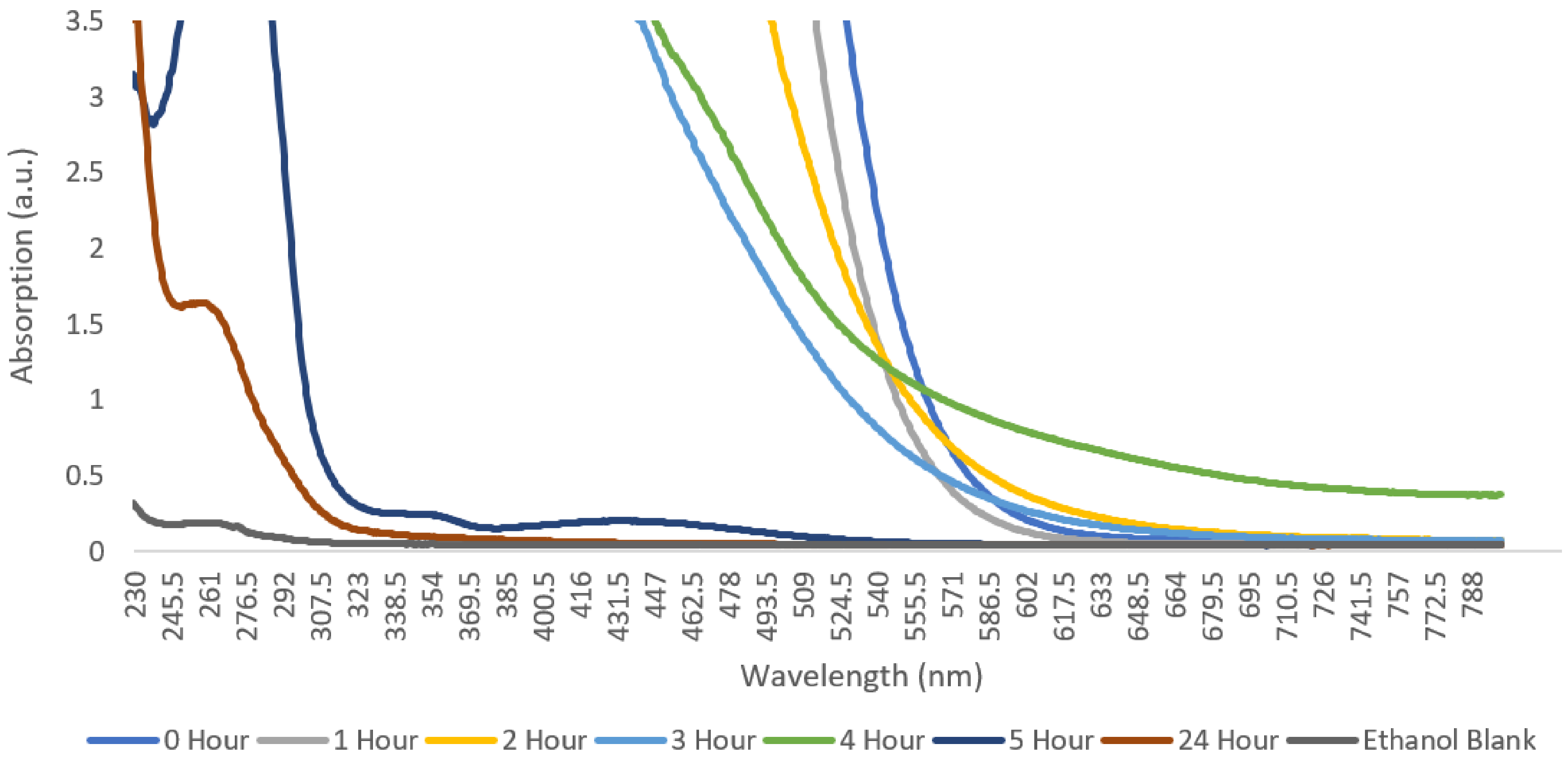
- Iron (III) acetylacetonate, expanded graphite, ethanol
- Simple solvothermal process forms graphene attached to magnetite nanoparticles, which can be used for other transition metals
- **Graphene Exfoliation**
  - Sonicate expandable graphite with NMP and centrifuge graphene out
- **Solvothermal Synthesis**
  - Place iron (III) acetylacetonate and 200 proof ethanol in an autoclave
  - Heat autoclave in a muffle furnace at 180 C for varying amounts of time
  - Put autoclave under forced air cooling and centrifuge product out
- Test various heating times to see effects on particle size and coating density

CHARACTERIZATION

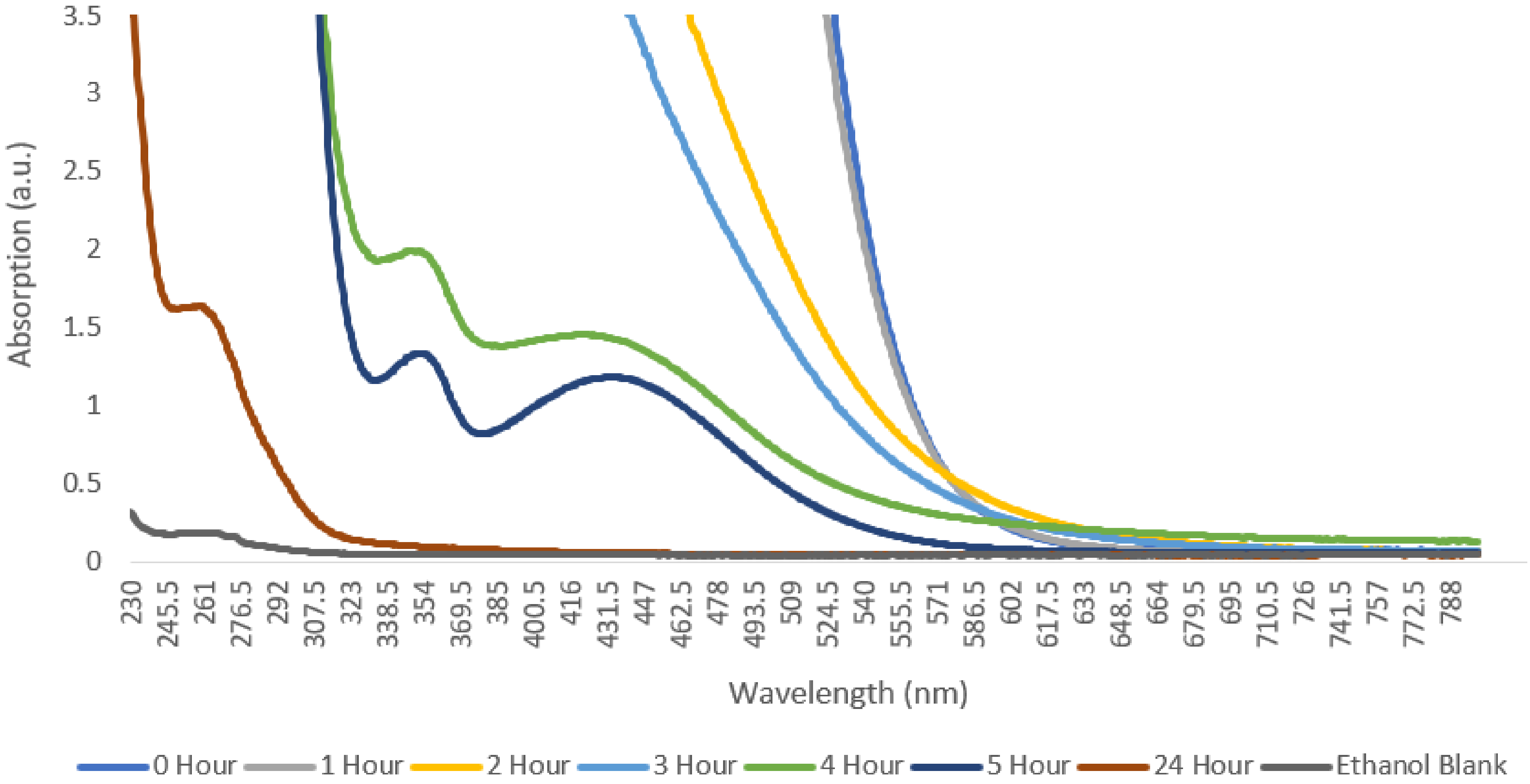
- **UV-Vis Spectroscopy**
  - Remaining solution placed into a cuvette
  - Energy is passed through and absorbed to excite electrons to higher energy orbitals
  - Detector records degree of absorption at each wavelength
  - Beer-Lambert Law states absorbance is proportional to the concentration of a solution
  - Measures change in concentration of reactants or products over time
- **Transmission Electron Microscope (TEM)**
  - Used to observe and characterize possible structures that are formed



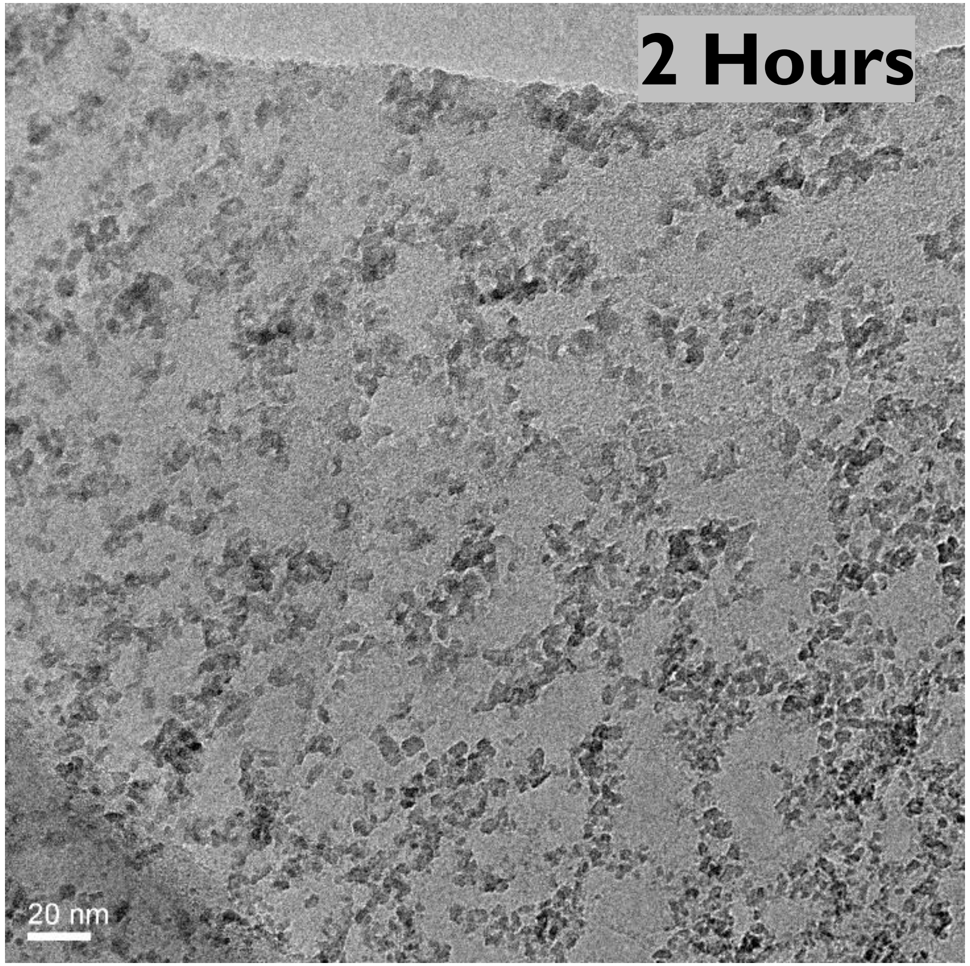
UV-VIS OF HEATING TIME VS ABSORPTION (TRIAL 1)

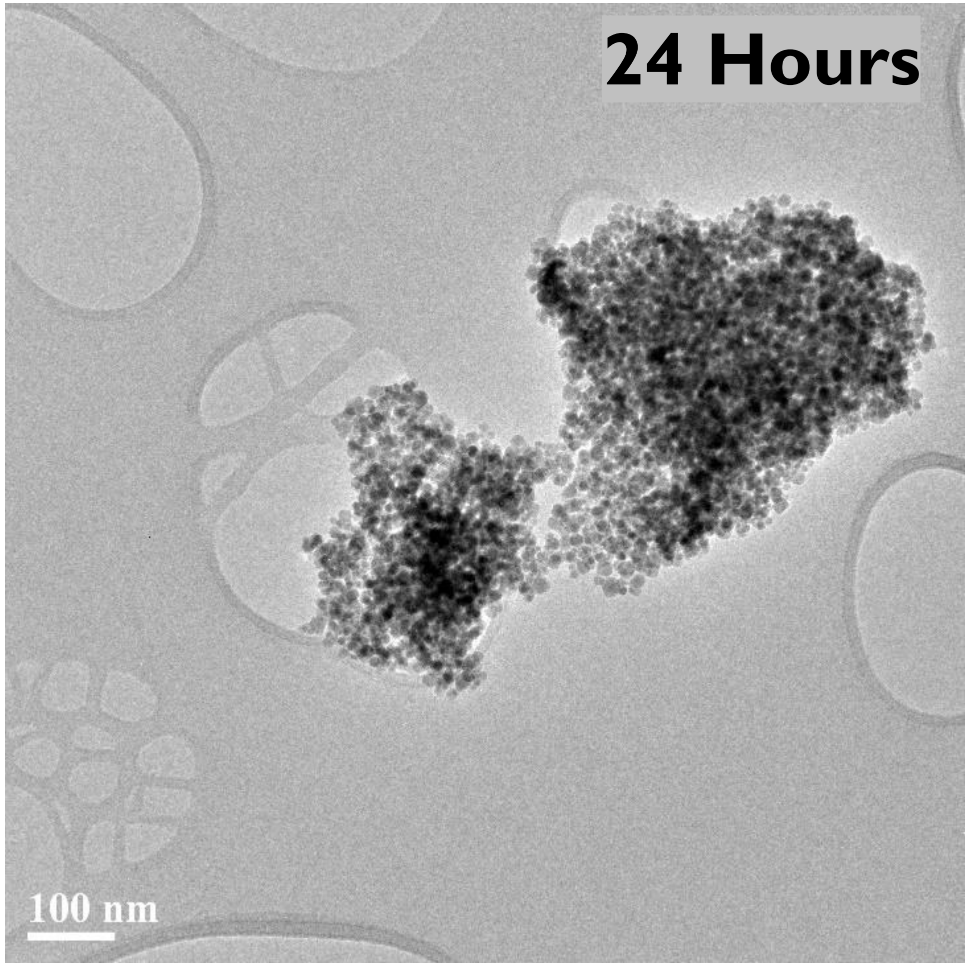


UV-VIS OF HEATING TIME VS ABSORPTION (TRIAL 2)



TEM IMAGES OF FE_3O_4 NANOPARTICLES





DISCUSSION

- **UV-Vis Spectroscopy**
  - First trial demonstrated a sudden jump between 4 and 5 hours of heating
  - Repeated a second trial due to leaks during the synthesis process and unclear results
  - Second trial showed a less dramatic jump between 3 and 4 hours of heating
  - Most of the synthesis completes within 5 hours of heating
- **Transmission Electron Microscope (TEM)**
  - Nanoparticles were being formed on the carbon support even with only 2 hours of heating
  - Synthesis is able to proceed (but not complete) within a short amount of time

CONCLUSION

- Majority of the reaction completes within 5 hours of heating time, which is much shorter than the previously hypothesized 16 hours
  - Allows for more efficiency with reduced synthesis times of iron oxide nanoparticles
- Understanding the growth pattern of the nanoparticles allows us to more accurately tailor the particle size and distribution
- Future work would center around gathering consistent data on heating times from 0 to 24 hours
- Can be used for large-scale production and more environmentally friendly devices due to its wide range of capabilities and possible applications

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