COPPER NANOPARTICLE SYNTHESIS IN A MICROFLUIDIC REACTOR

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Project Background

OPPORTUNITY

Copper nanoparticles are extremely small particles. They have diameters on the order of 50 nm.

Copper nanoparticles have many current applications and more potential applications

- Conductive inks and pastes
- Color for inks and paints
- Heat sink contacts
- Antibiotic coatings

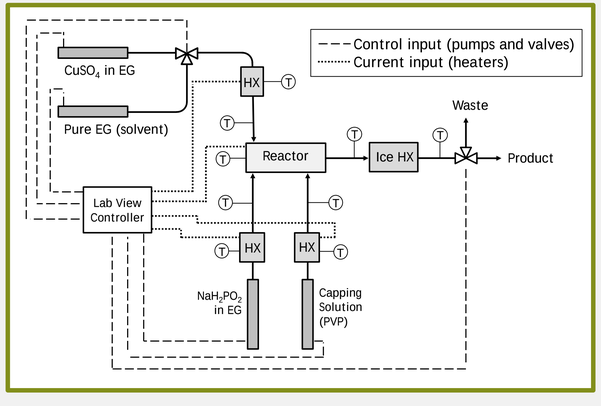
Business opportunity: produce small, uniform nanoparticles in a continuous process

- Microfluidic reactors have potential to produce smaller nanoparticles faster, cheaper, and with better uniformity
- Smaller particles result in lower melting temperatures, have processing benefits

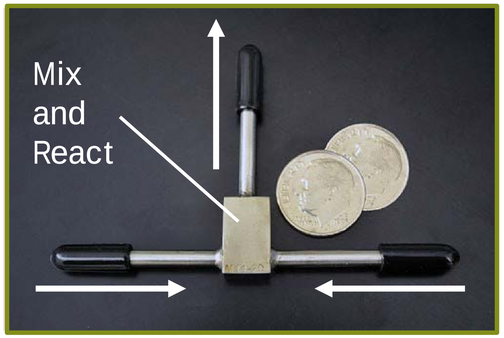
Reaction in microreactor must be characterized, optimized

MATERIALS AND METHODS

- Temperature control in LabVIEW
- Manual flow control from syringe pumps



- Samples cleaned by centrifugation at 5000 RMP for 15 minutes and dilution in isopropanol
- Scanning electron microscope(SEM) used to characterize copper particles



OUR PROJECT

Goal: Characterize the relationship between reactor conditions and properties of the produced nanoparticles

Experimental parameters:

- Temperature in reactor: 85, 100, 115 °C
- Residence time: 3, 5, 10 seconds

Important factors held constant:

- Reagent concentrations
- Reactor design

Metrics:

- Copper particle shape and diameter
- Copper particle size uniformity
- Qualitative performance

CHEMISTRY AND FORMATION

Reagents and Chemistry

- 0.05 M Copper Sulfate ($CuSO_4$) – Copper ion source
- 0.1 M Sodium Hypophosphite (NaH_2PO_2) – Reducing agent
- 0.001 M Poly(N-vinyl-2-pyrrolidone) (PVP) – Capping agent; large polymer (10 kDa)

$$Cu^{2+} + H_2PO_2^- + H_2O \rightarrow 2H^+ + H_2PO_3^- + Cu$$

NaH_2PO_2 gives electrons to convert Cu^{2+} to elemental copper. PVP does not react, but 'caps' nanoparticle growth by physically blocking atoms. Nanoparticles continue to grow and form large crystals if reaction is not stopped.

Nanoparticle Formation

During reaction, dissolved copper atoms form crystalline nanostructures.

Nucleation: Single Cu atoms → Copper Nucleus

Dissolved copper atoms must overcome an energy barrier to initially aggregate and form a 'nucleus'.

$$Nucleation\ Rate = \dot{N} \propto e^{-\frac{\Delta G^*}{kT} - \frac{E_{diff}}{kT}}$$

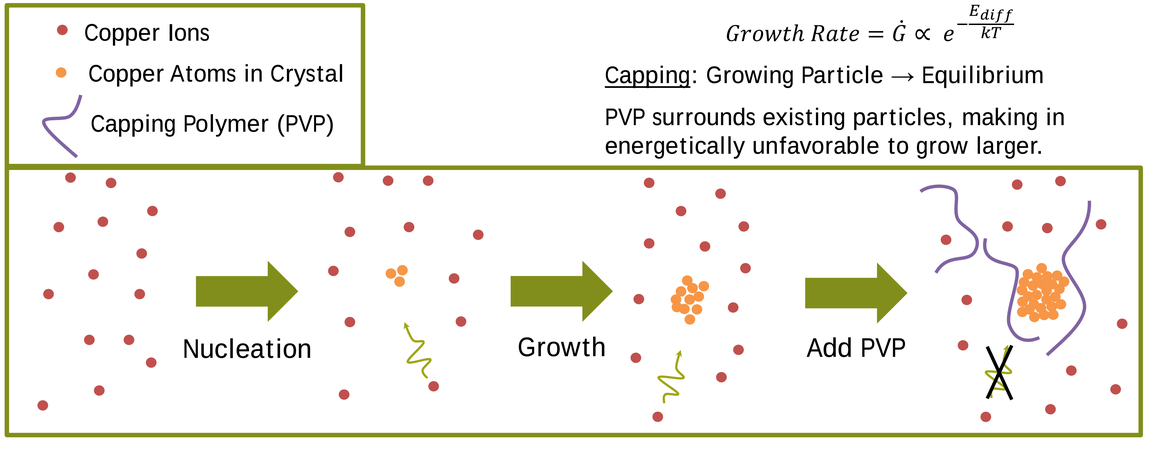
Growth: Copper Nucleus → Copper Nanoparticle

After nucleation, adding more atoms is energetically favorable, growth continues at a rate limited by diffusion.

$$Growth\ Rate = \dot{G} \propto e^{-\frac{E_{diff}}{kT}}$$

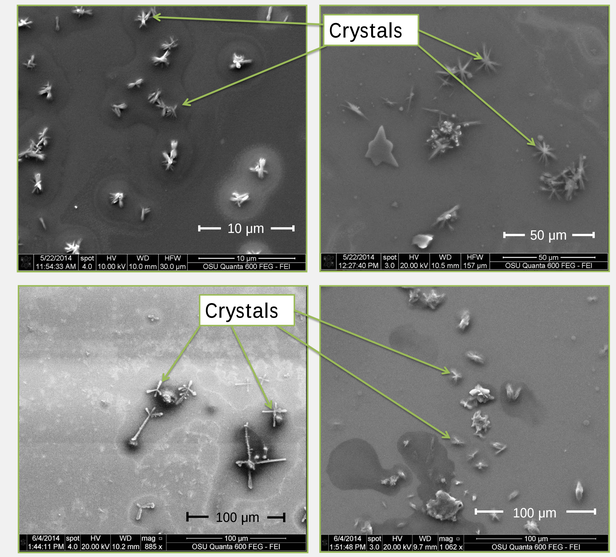
Capping: Growing Particle → Equilibrium

PVP surrounds existing particles, making in energetically unfavorable to grow larger.



Results

Copper crystals were large with dendritic (branching) structure, indicative of a diffusion-controlled process. Possible result of low reactant concentrations



Recommendations

- Focus on running experiments with higher concentrations to encourage nucleation and produce more uniform particles
- Combine PVP with NaH_2PO_2 to promote more spherical shape and slower particle growth to increase uniformity
- Investigate temperature effects at higher concentrations on types of particles formed

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