An Advanced Materials Technology for Recovering Metals from Dilute Aqueous Streams

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Presentation Outline

- Introduction & Background
- Nanocomposite Particle Synthesis & Characteristics
- Continuous Flow Reactor Design & Operation
  - 3rd Generation
  - 4th Generation
- Metal Recovery via Electrowinning
- Present status/Concluding remarks
- Acknowledgements
- Questions

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Example application: metal-contaminated mine waters

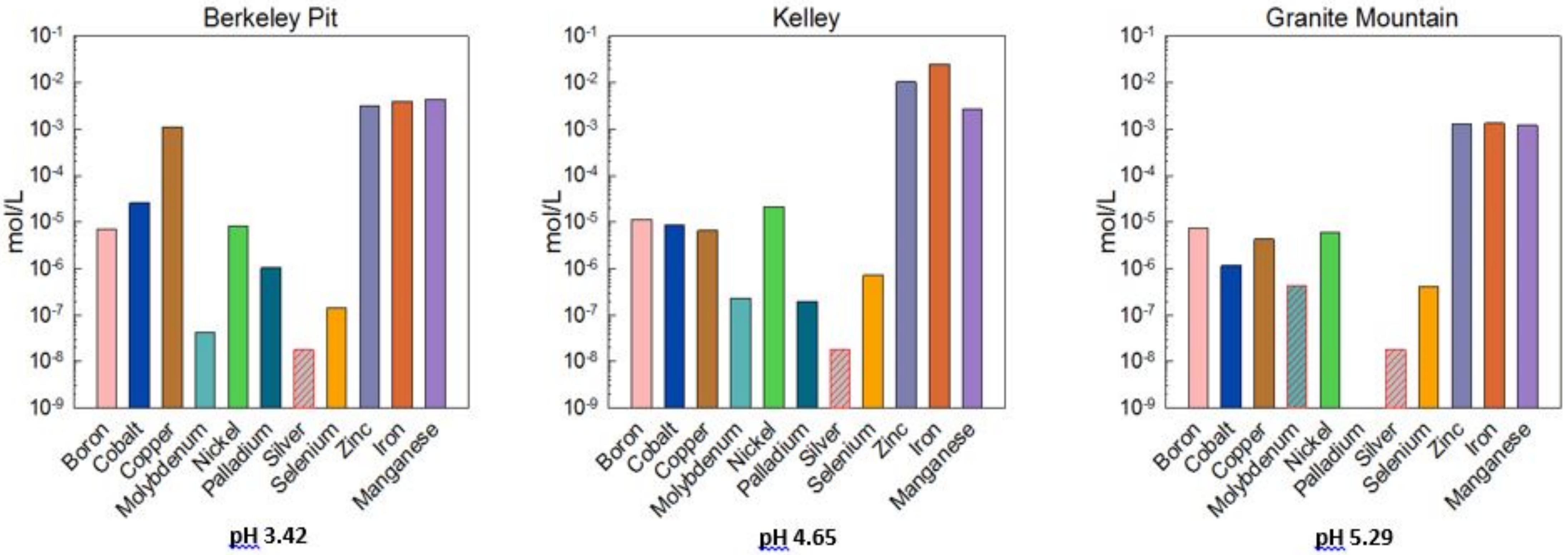


Figure 1 – Selected metal concentrations of mine site samples

Background

- Dr. Arijit Bose (University of Rhode Island) developed a concept for a continuous magnetic field reactor for special ion exchange applications.

- U.S. Patent No. 6,635,181, Continuous Hybrid Magnetic Field Gradient Rotating Wall Device for Colloidal Magnetic Affinity Separations.

- URI and Hazen Research, Inc. (Golden, Colorado) collaborated on a Phase I STTR project to design, construct, and operate a bench-scale reactor.

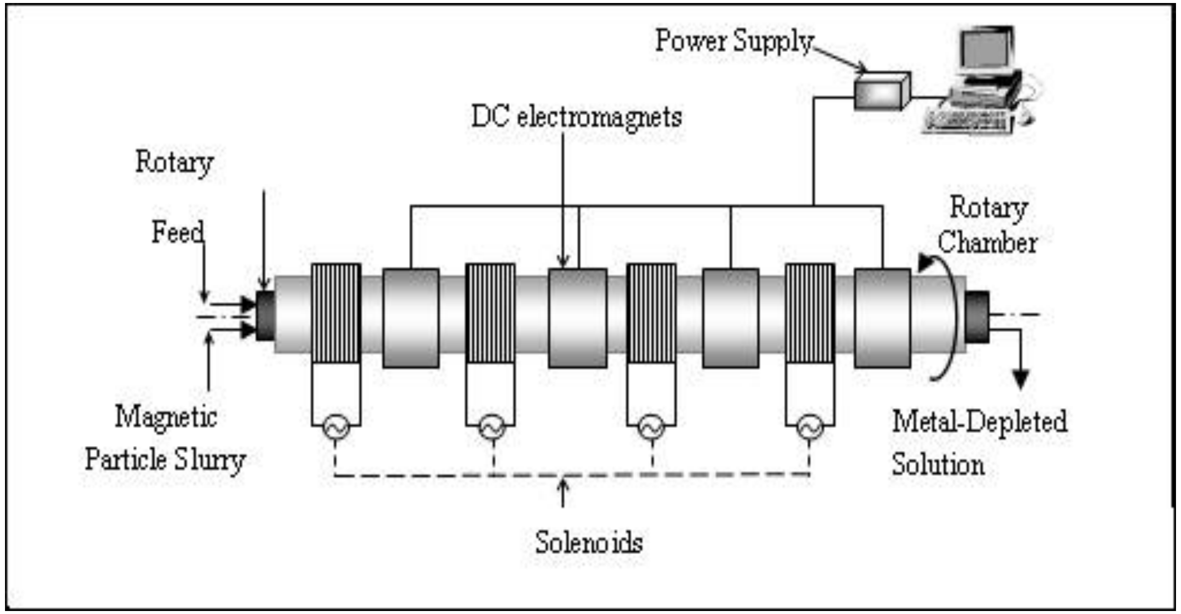


Figure 2 – 1st generation reactor

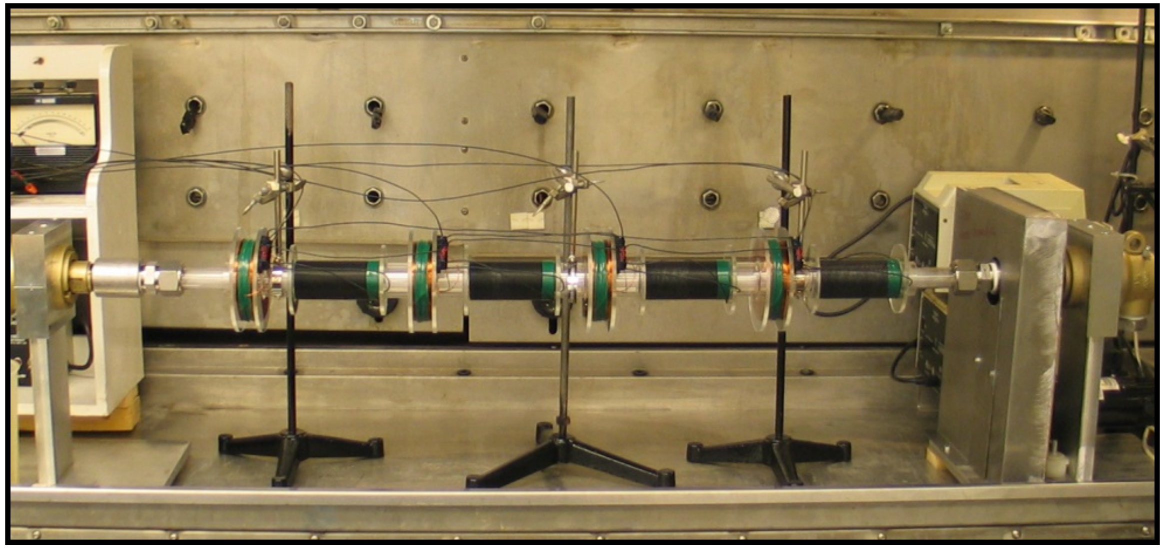


Figure 3 – 2nd generation reactor

Metal capture efficiency

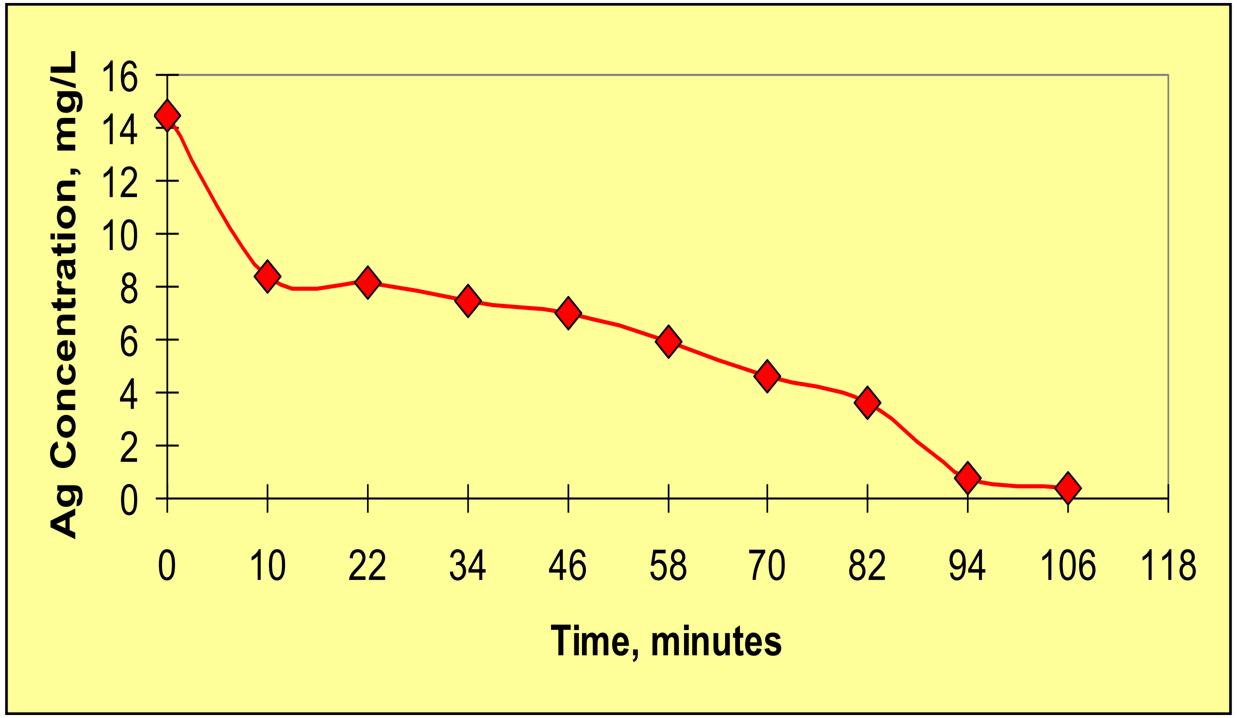


Figure 4 – Discharge solution analysis vs. time for the silver surrogate solution trial

Particle retention

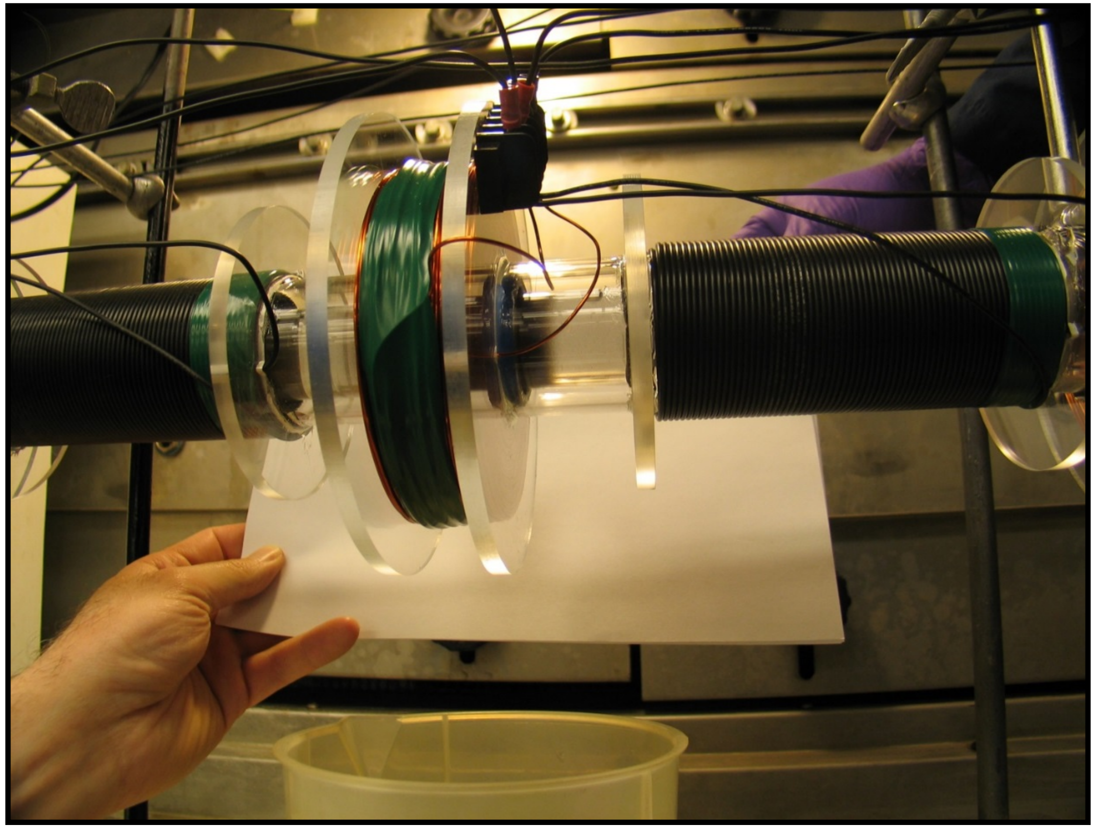


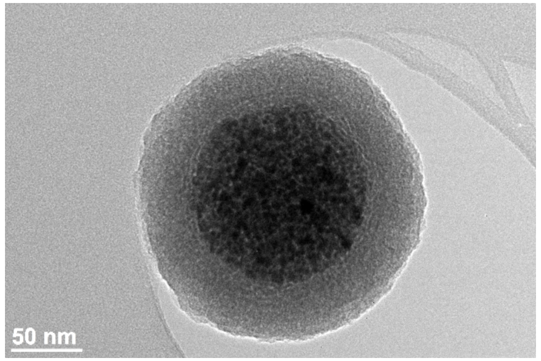
Figure 5 – Photograph showing magnetic IX particles collected by the electromagnet

Nanocomposite particle synthesis

Magnetite core (20 – 30 nm)

Synthesis of core-shell nanoparticles using PAA as the metal-capturing ligand begins with tetrethyoxysilane (TEOS) hydrolysis.

Polyallylamine (PAA) was used as the metal-capturing ligand



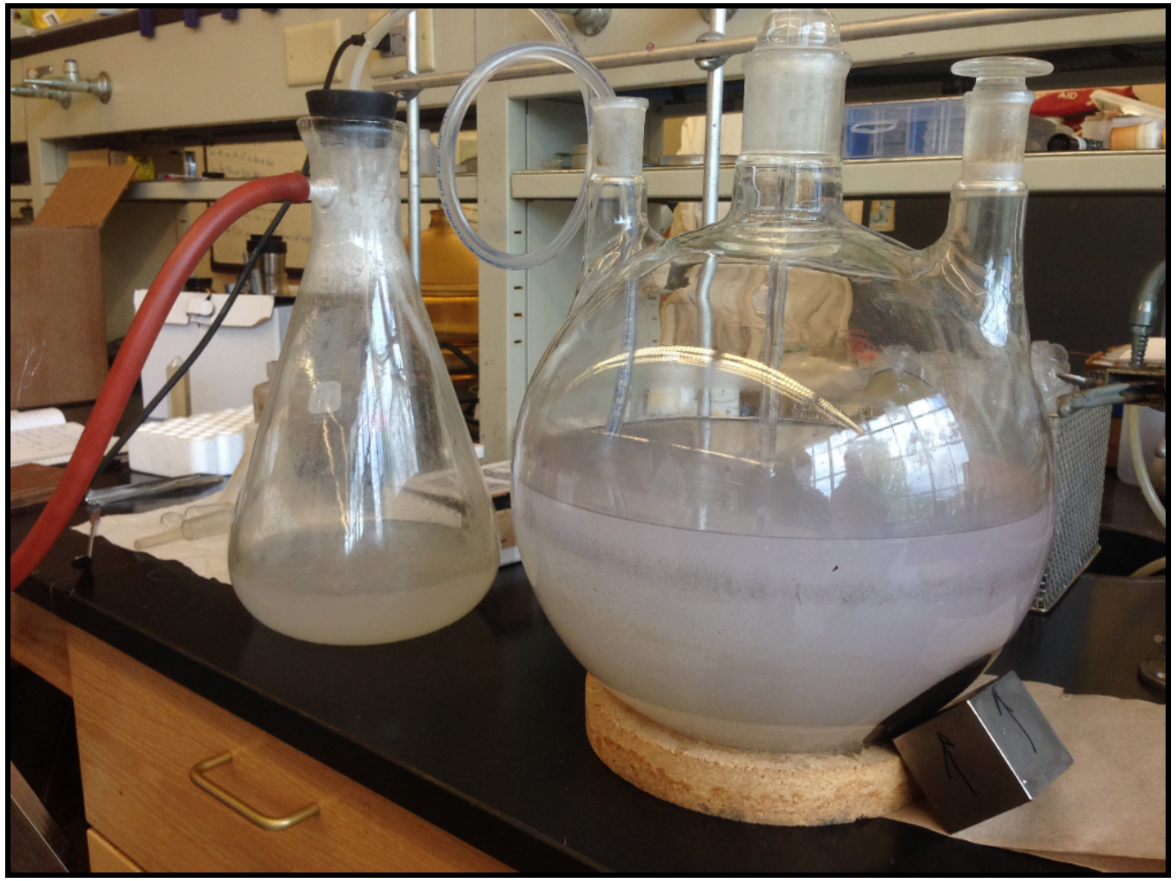


Figure 6 – magnetic particles gathered with a permanent magnet following the TEOS step.

(performed in Dr. Rosenberg's lab at UM)

Nanocomposite particle characteristics

Energy dispersive X-ray measurements (EDX) gave a percent by iron of 80-81 % and a 10 % Si, consistent with deposition of a thin silica layer.

Dynamic Light Scattering (DLS) measurements gave a Z averaged diameter (ZAD) of 162 nm.

SEM and TEM images of the core-shell nanoparticles revealed particle aggregates of about 200 nm but individual 10-12 nm core-shell nanoparticles can be clearly identified.

Copper loading capacities of 0.5 mmole/gram.

The nanoparticle loading rates are about *10 times faster* than the related micro-scale silica polyamine composites.

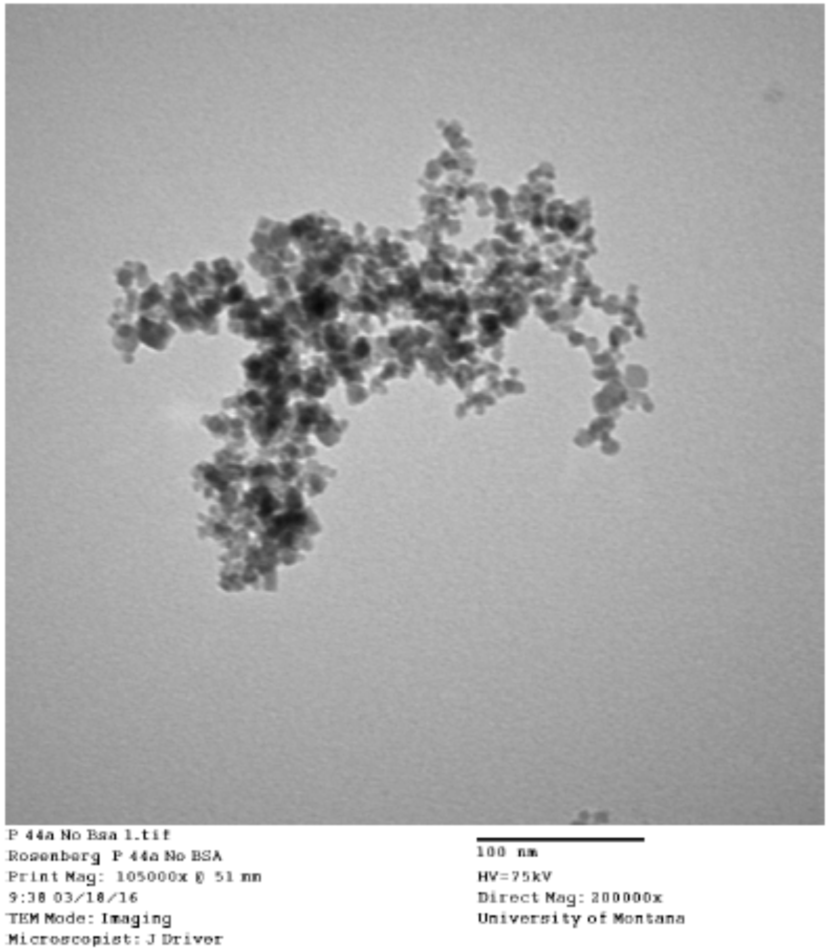


Figure 7 – TEM image of a cluster of nanocomposite particles

3rd generation continuous flow reactor

The design is much simpler and more robust than the previous prototypes:

- 100 liter total reactor volume
- Solution flow rates range from 2 to 10 liters per minute
- A single internal electromagnet replaces multiple external magnets
- Static mixers replace magnets intended to oscillate particles
- No moving parts except for pumps and solenoid valves

Figure 8 – 3rd generation continuous flow reactor system with in-line magnet module

System operation

A slurry of magnetic nanocomposite ion exchange (IX) particles is introduced to the surrogate wastewater stream at the top of the platform.

Functional groups impregnated on the surfaces of the ion exchange particles bond with metal ions as the particle-laden stream flows through the reactor.

The electromagnet captures and retains the nanocomposite particles without impeding the wastewater flow.

Valve positions are periodically changed to divert flow through a parallel magnetic module.

The electromagnet is de-energized to release the particles, which are subsequently stripped, reconditioned, and returned to service.

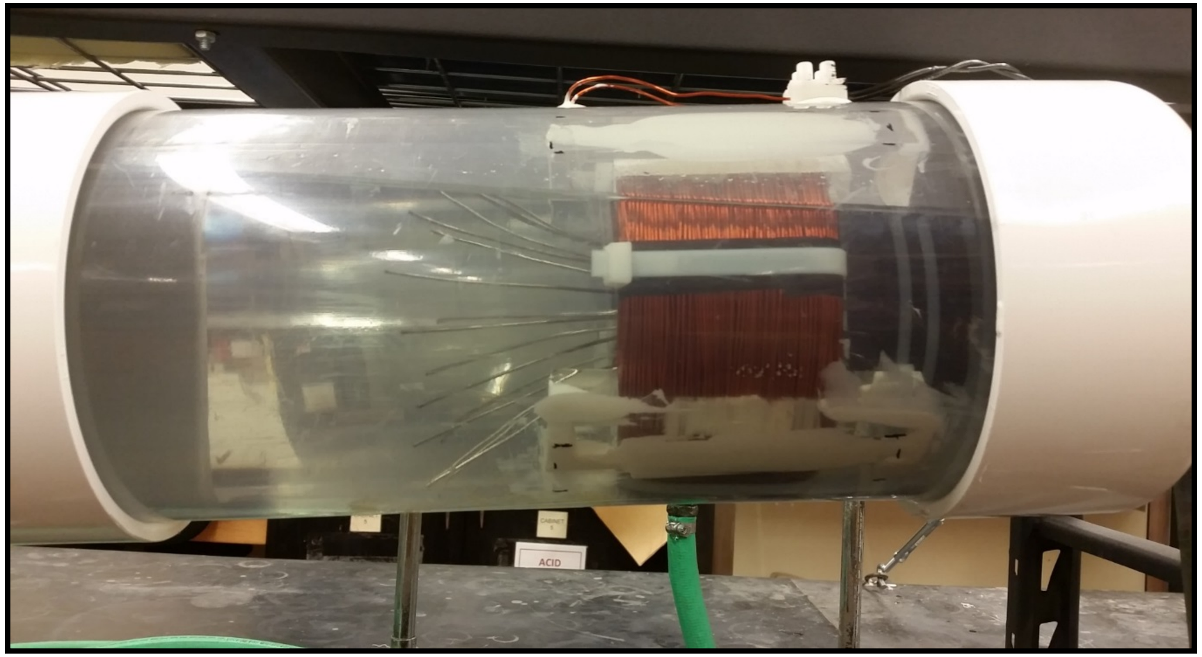


Figure 9 – The in-line electromagnet module consistently achieves magnetic particle capture efficiencies in excess of 98%.

System operation

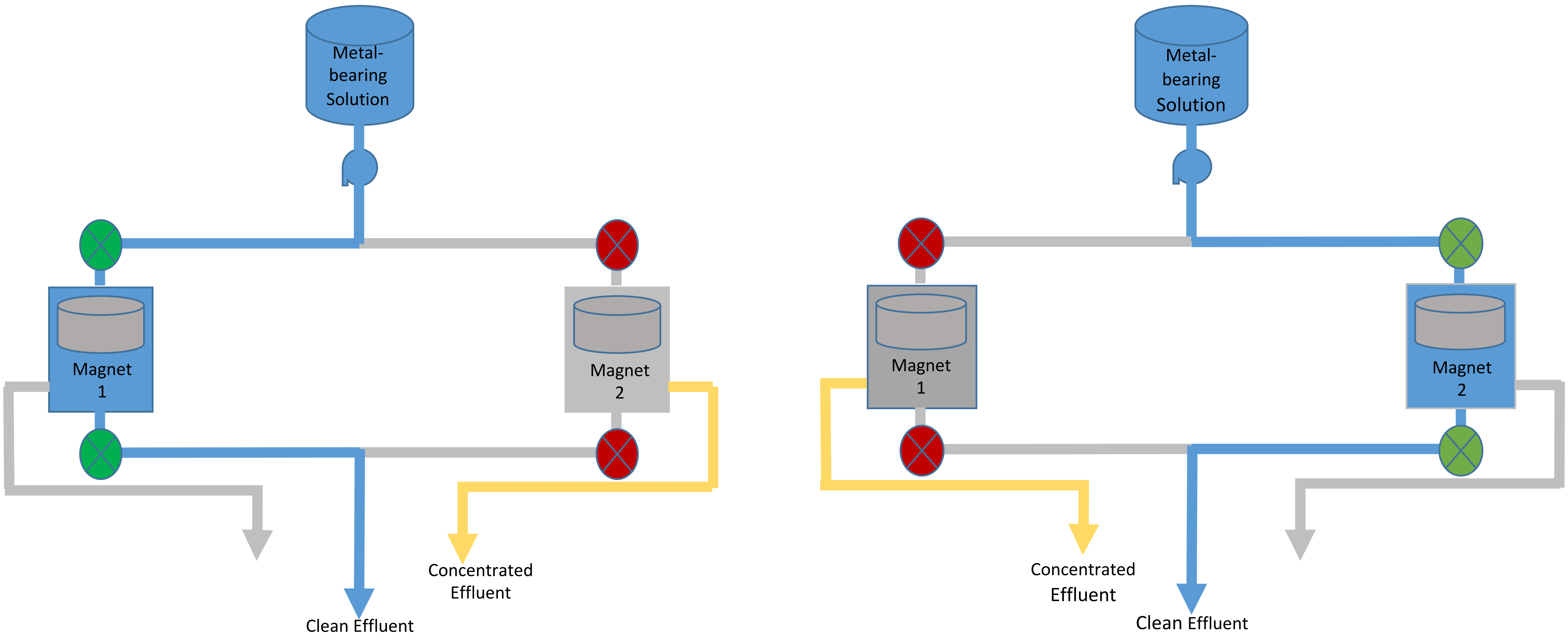


Figure 10 – Schematic diagram depicts IX system operating sequence

4^{th} generation continuous flow reactor

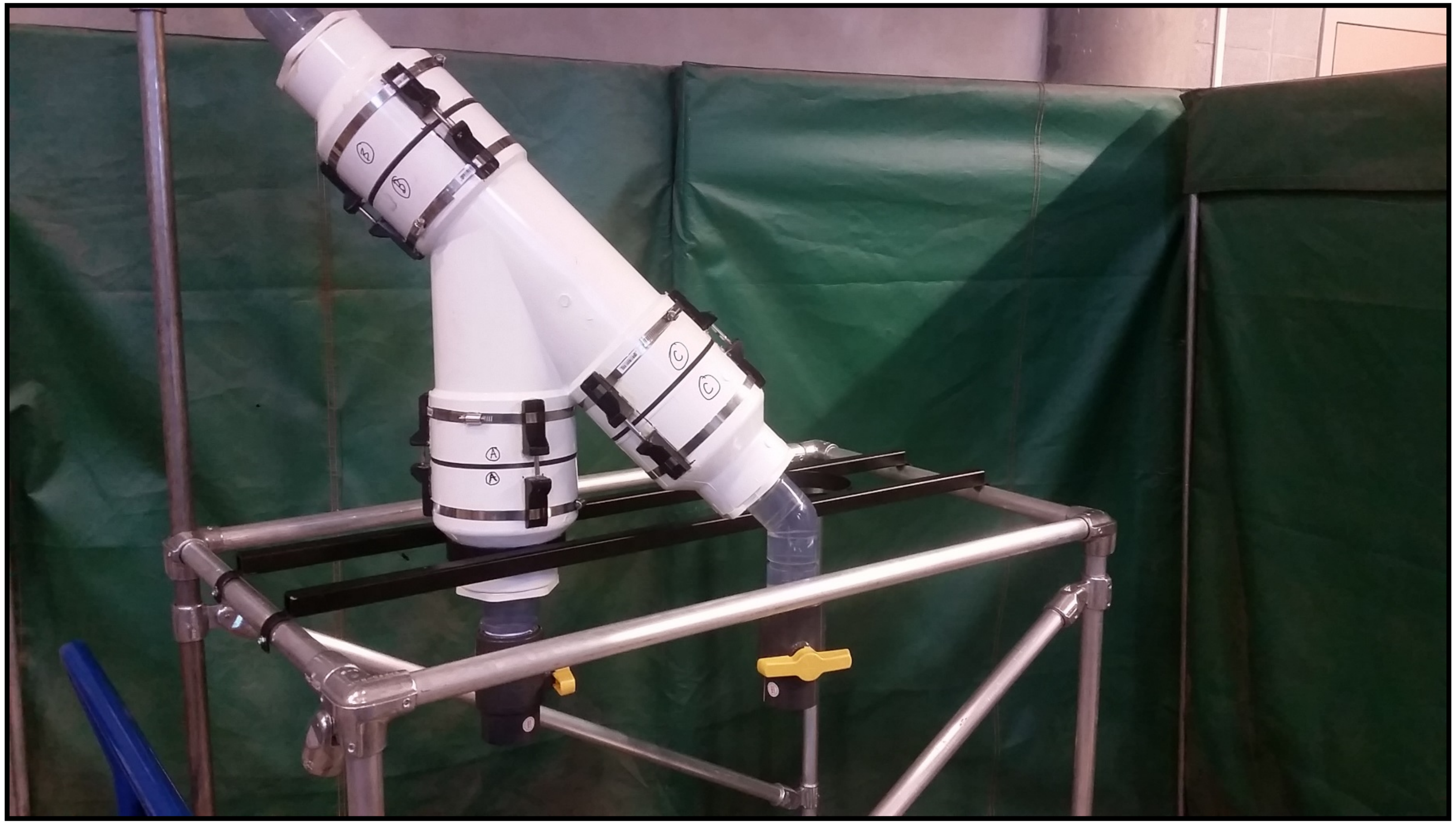


Figure 11 – the modified electromagnet configuration

Figure 12 – the 4^{th} generation continuous flow reactor (120 L)

Metal recovery

Figure 13 – the laboratory EMEW™ electrowinning circuit.

Figure 14 – copper and zinc sheets

System performance:

Copper

40-45 °C electrolyte
2.5V 11A
94% current efficiency

Zinc

30-50 °C electrolyte
5V 20A
95.5% current efficiency.

Present status

The culminating achievements of this research project include:

1. Successful development of a method for preparing magnetic nanocomposite IX particles that have proven effective in both laboratory and pilot plant evaluations

2. Two pilot-scale continuous flow reactor systems have been constructed, commissioned and successfully operated. These systems will be used to further develop and demonstrate the process.

3. Several Montana wastewater streams have been sampled and characterized; candidates for treatment via the magnetic nanocomposite/continuous flow reactor technology have been identified.

Concluding remarks

The combination of these innovative ideas represents a powerful technology for rapid continuous extraction of valuable and/or toxic metals from a broad range of contaminated waters

- continuous IX method proven capable of efficiently capturing metals and other contaminants from dilute aqueous streams
- mechanically very simple and can be configured for site-specific applications
- amenable to automated control systems
- cost effective – low low capital and operating costs

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Acknowledgements: Project Team

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