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PNNL-35843

Advanced Mineral Extraction and Water Processing

Application Development of Mesofluidic Separation Technology

April 2024

R Shane Addleman
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Michael Minette
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U.S. DEPARTMENT OF ENERGY

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Abstract

This project explored the development of mesofluidic devices and methods to provide a range of new capabilities with a focus on selective mineral extraction and cost-effective water treatment systems. Mineral separation and water treatment technologies are typically slow, bulky, expensive, time and energy intensive processes. This leads to very large, expensive processing plants that are often inefficient and produce low quality products and large volumes of waste. Technologies used in mineral extraction and water treatment have changed little in the last 50-100 years. The new technology, based upon mesofluidic separation devices, could disruptively change the state of the art and alter long established economics. The new technologies explored will provide systems with much smaller footprints, higher throughput, modular components easily integrated into existing industrial processes and plants, lower cost, and novel separation capabilities. The results from this effort contributed to a wide range of new capabilities and numerous inventions disclosures.

Summary

This project explored the development of mesofluidic devices and methods to provide new capabilities for selective mineral extraction and cost-effective water treatment systems. This effort has relevance to wide range of priority Department of Energy (DOE) mission areas including:

- Nuclear waste (slurry processing)
- Environmental remediation (water cleanup and treatment)
- Advanced manufacturing (required for the construction of new devices)
- Wastewater/process water treatment (relevant to fossil energy, geothermal, desalination)
- Green energy and critical materials (improved methods for domestic production of critical materials have been identified as a national priority by both the Executive Branch as well as DOE and the Department of Defense; further, green energy requires significant new supplies of critical materials such as Li, rare earths, etc.)

Mineral separation and water treatment technologies are typically slow, bulky, expensive, time, and energy intensive processes. This leads to very large, expensive processing plants that are often inefficient and produce low quality products and large volumes of waste. Technologies used in mineral extraction and water treatment have changed little in the last 50-100 years. The new technology explored in this effort is based upon mesofluidic separation devices that will provide:

- *much smaller relatively physical footprints with higher chemical throughput,*
- *modular components that are easily integrated into existing industrial processes and plants,*
- *significantly lower cost (both capital and operating), and*
- *provide novel separation capabilities.*

The new technologies can leverage the old proven methods and process chemistries while providing the economic and performance improvements delineated above. While it is too early to make conclusive statements about these technologies, they could disruptively change the state of the art and improved the economic opportunity, energy efficiency, and reduce water usage in a range of industries/applications that are of strategic DOE interest (outlined above).

In this effort, numerous novel prototype devices were designed, constructed and used to demonstrate the core operations for the new technology. Methods to combine the different mesofluidic devices and process to improve and expand performance were explored and demonstrated. Methods for bulk and trace solution treatment were explored and successfully demonstrated.

The results from this commercialization LDRD were very productive with many novel device concepts and processes explored and reduced to practice. To date (more invention anticipated) the invention disclosures filed resulting from this effort include:

- *Methods and Devices for Enhanced Industrial Flow Processing and the Recovery and Separation of Minerals*
- *Enabling Improved Dynamic Operation of Geothermal Plant*
- *Dynamic Particle Separation and Analytical Characterization*
- *Avalanche Boycott Separator*
- *Integrated Modular In-Pipe Chemical Processing Invention*
- *Improved Chemical Processing using Chemically Active Particles and Mesofluidic Separation Devices*

Additional IP is being developed. Manuscripts are in composition and will be submitted when the Intellectual Property (IP) is finalized and filed.

Acknowledgments

The research described in this report was conducted under the Strategic Investment Laboratory Directed Research and Development (LDRD) Program at the Pacific Northwest National Laboratory (PNNL), a multiprogram national laboratory operated by Battelle for the U.S. Department of Energy. PNNL is a multi-program national laboratory operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute under Contract No. DE-AC05-76RL01830.

Acronyms and Abbreviations

DHS	United State Department of Homeland Security
DOD	United States Department of Defense
DOE	United States Department of Energy
IDR	Invention disclosure reports

Contents

1.0 Introduction to Mesofluidic Devices for Separation

This project explored the development of mesofluidic devices and methods [1-9] to provide new capabilities for selective mineral extraction and cost-effective water treatment systems. Mineral separation and water treatment technologies are typically slow, bulky, expensive, time and energy intensive processes. This leads to very large, expensive processing plants that are often inefficient and produce low quality products and large volumes of waste. Technologies used in mineral extraction and water treatment have changed little in the last 50-100 years.

The new mesofluidic separation devices, created with state-of the art new 3D additive manufacturing methods, will enable new separation processes that will disruptively change industrial processes and alter long established economics. The new technologies explored in this effort will provide systems with much smaller relatively footprints, higher throughput, modular components easily integrated into existing industrial processes and plants, lower cost and novel separation capabilities. This effort is focused water treatment and domestic critical mineral supply applications. Enhanced water treatment technology benefits numerous industries of relevance to DOE and related industries (fossil energy, manufacturing, food processing, etc.) and has many environmental processing applications. Improved methods for critical materials (rare earths, Li, etc.) processing has been identified as a national priority by the Executive branch as well as the DOE, DoD, DHS and other government agencies.

An example of the new mesofluidic technology, recently invented at PNNL, is shown graphically below in Figure 1. This technology, sometimes referred to as the Micropillar Enhanced Particle Separator (MEPS) technology, utilizes the hydrodynamic forces on particles flowing through ordered array of micropillars to provide rapid efficient separation of particles from solution. For larger the particles, the larger the fluid forces on the particles making size selectivity possible.

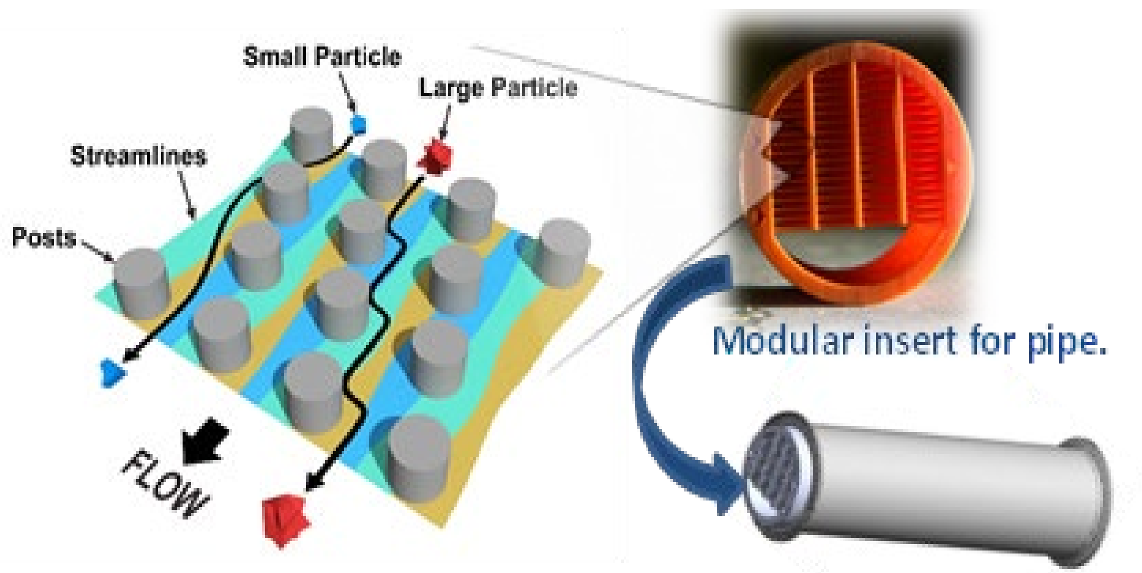

Figure 1. A graphical illustration showing MEPS technology from the microscale to the macro scale. Hydrodynamic forces on particles flowing through the carefully structures array of micropillars result in rapid efficient filtration of the solution. The MEPS devices can be custom designed for specific applications of interest and provides a wide range of novel particle separation capabilities. This project demonstrated MEPS technology can be effective for separation of particles from <50 μm to >2 cm.

The integrated graphic in Figure 1 shows MEPS technology that spans the microstructure that drives the separation of small particles to the macrostructures than can be inserted into large industrial piping systems (engineered structures with spans micrometers to millimeters—a 1000x dimensional range). Recent advanced in 3D printing now allow MEPS devices to be economically manufactured in large modular formats than can be directly inserted into industrial tubing. Modules are stacked to achieve the desired separation performance. Modules can be removed for cleaning/replacement. Modules can be printed from cheap plastics as well as durable ceramics and corrosion resistant metals as needed.

MEPS technology can provide rapid efficient, selective removal of particles from solution without the use of filters or large settling tanks. This provides significant economic advantages, both with operational and capital expenses, over conventional industrial methods. The new technology can easily work with proven old methods and chemical processes. Compared to existing industrial particle filtration methods, MEPS technology is:

- resistant to mineral scale and plugging,
- capable of operating in chemically thermally and radiologically extreme conditions,
- can operate over large dynamic flow ranges,
- has lower backpressure,
- compact and relatively inexpensive to manufacture,
- reduced operational and capital expenses.

The MEPS devices can be custom designed for specific applications of interest and provide a wide range of novel particle separation capabilities. This effort focused upon the development of MEPS, and other mesofluidic devices, for the advantageous applications to the significant challenges in critical mineral recovery and water processing. In this effort a range of novel mesofluidic devices were designed, constructed, and tested.

2.0 Technical Efforts

This project explored the development of a range of novel mesofluidic separations technology in support of improved mineral extraction and water treatment processes. Novel high throughput, compact modular devices were developed and demonstrated for specific applications of interest. Selected results are presented below.

2.1 MEPS Device Development and Innovation

This effort was undertaken on the very promising performance of mesofluidic devices, also referred to as Micropillar Enhanced Particle Separator (MEPS) technology, invented by PNNL. Prior efforts with MEPS suggested the potential for fast efficient particle separation systems that are resistant to plugging, capable of operation over large flow ranges, and are much cheaper than conventional industrial precipitation management systems. This effort developed new capabilities for MEPS while exploring and demonstrating the technology for applications in critical minerals recovery and water treatment. Graphics of the MEPS technology are shown above in Figure 1 and below in Figures 2 and 3.

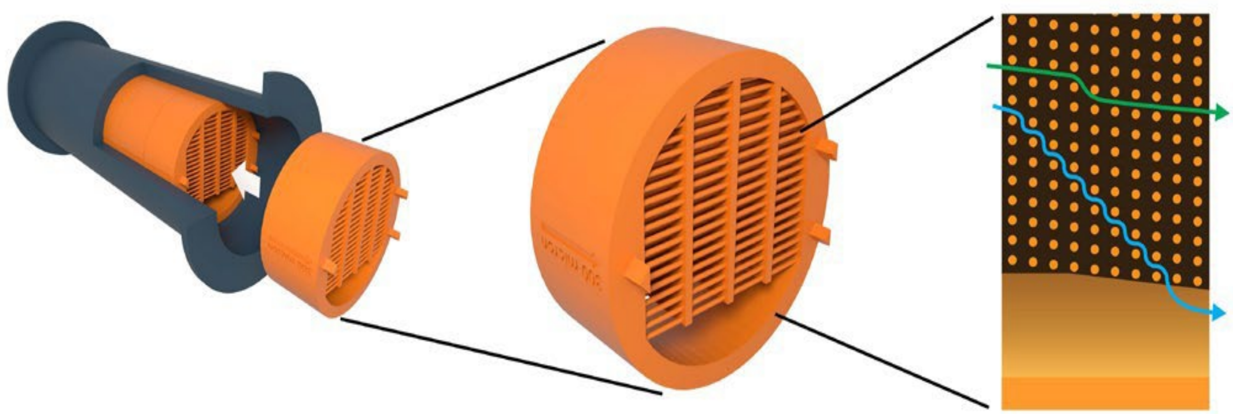

Figure 2. A graphical illustration showing MEPS modules and how they can be assembled at the industrial scale and then provide separation of particles (ranging from cm to microns in size). The blue arrow represents the characteristic path of large particles be separated and the green arrow represents smaller particles that simply pass through the devices. The MEPS modules are custom designed for provide specific particle separation capabilities. Modules are stacked to achieve the desired system separation performance. Modules can be removed for cleaning/replacement. Modules can be printed from cheap plastics as well as durable ceramics and corrosion resistant metals as needed. Recent advances in 3D printing allows MEPS devices to be printed in large modular formats than can be directly inserted into industrial tubing.

A graphical representation (upper) and engineering design (lower) of a functioning MEPS device is shown below in Figure 3. Hydrodynamic forces on particles flowing through the carefully structured array of micropillars result in rapid efficient filtration of the solution. The key to this technology is that the hydrodynamic forces on the particles moving through the device(s) are based solely on particle size and not density (resulting from careful design of the pillar array). This provides a novel separation method independent of density and standard filtration mechanisms.

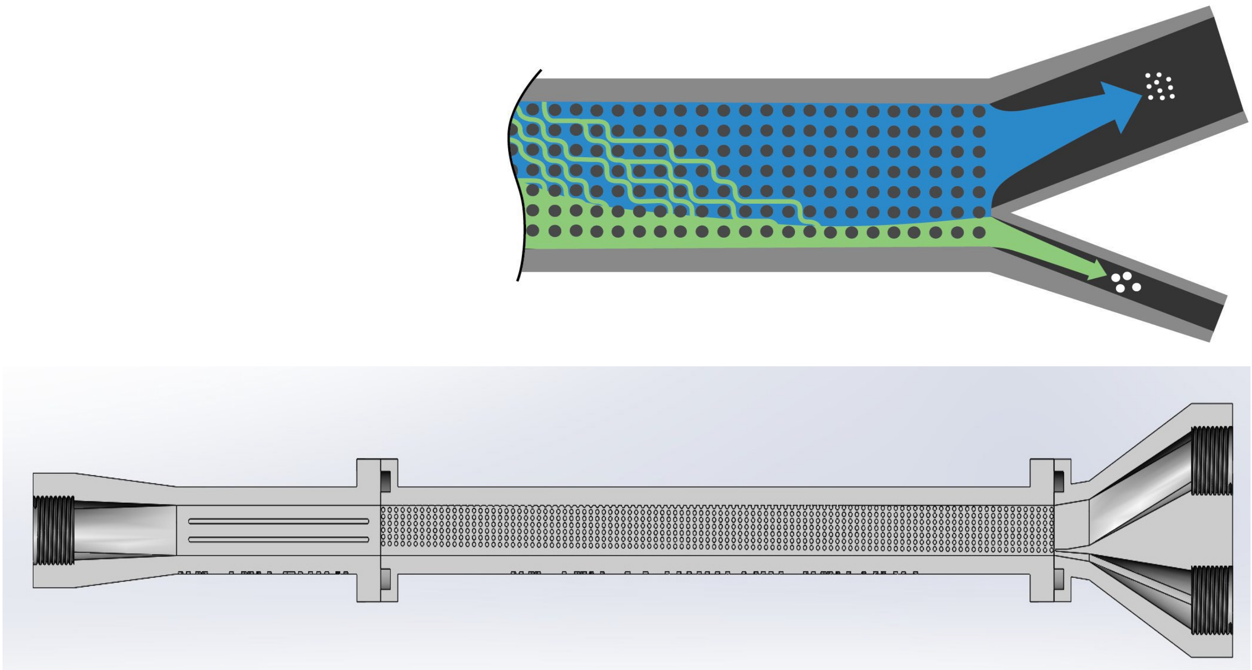

Figure 3. The upper figure is a graphical illustration showing particles flowing through a MEPS device and then provide separation of particles (ranging from cm to microns in size). The green represents the flow and separation of the large particle being separate. The blue represents smaller particles that mostly pass through the device with the majority of the process flow. Pillar spacing, size and device length determine the separation performance. The lower graphic is an engineering diagram of a functioning MEPS device similar to the upper graphic. The large number of micropillars inside the flow tube is easily observable. Some device configurations require many thousands of precisely placed micropillars. These devices can only be constructed with state-of the-art 3D manufacturing methods.

As shown in Figures 2 and 3, for these devices, small particles weave around arrays of posts. In contrast, large particles bump consecutively to one side of posts with each row of posts offset by a fixed amount. In this manner, small particles simply pass from the entrance to the exit, but large particle migrate toward one side and are separated from the main flow. Separation performance may be improved by extending the number of row or placing mesofluidic separators in series. Unfortunately increasing the length of the device(s) also increases the back pressure which may become significant. Flow rates may be improved with increasing pipe size or manifolding mesofluidic separators in parallel.

To support this project and the target a wide range of factors were investigated including:
- Improved flow models.
- Configurations that function is chemically and physically harsh conditions.
- Flow dynamics and mixing.
- Device design modeling for new configurations.
- Manufacturing methods and limits for MEPS devices
- Methods to combine different mesofluidic devices to improve and expand performance were explored and demonstrated.
- Extension of particle separation size range (for both larger and smaller are particles).

The MEPS test results of have shown effective mineral particle separation in the following ways:

- Hanes 282 alloy devices have endured heavy laboratory operations for several years.,
- Effective separation of different mineral particles has been demonstrated for low density activated carbons, to moderate density silica, to high density iron oxides,
- Efficient separation of particles over a 5x flow rate change (both laminar and turbulent conditions),
- Effective particle separation from both Newtonian and non-Newtonian slurries,
- Demonstrated effective separation of particles from <50 μm to >2 cm, and
- Operations up to 100 GPM in sand and complex slurries with 97.6 +/- 2.1% removal of particles [6].

Project efforts produced and demonstrated performance for target applications, new capabilities that can be applied to future effort, draft manuscripts, and new IP. Foundational MEPS IP was in place at the beginning of the project. During the course of this effort, we have developed MEPS devices, as well as new methods of use and applications. Project efforts in this area have significantly contributed invention disclosure reports (IDR) including the IDRs:

- Methods and Devices for Enhanced Industrial Flow Processing and the Recovery and Separation of Mineral
- Enabling Improved Dynamic Operation of Geothermal Plant
- Dynamic Particle Separation and Analytical Characterization

Additional IDR submissions are under discussion and development. Manuscripts are being assembled and will await IP filing before submission.

2.2 Invention of the Boycott Separator

As noted above MEPS technology clearly provides new valuable capabilities in particle separation. However, MEPS devices are created using 3D printing to create the large arrays of micropillars which:

a. ultimately limit the size of particles that can be removed,
b. can create significant back pressure, and
c. result in large surface area devices upon which that mineral scaling and biofouling may occur.

Consequently, we explored novel particle separation concepts not dependent upon micropillar configurations. The result was a surprisingly effective, simple, and compact separator device based on the Boycott effect. Over a century ago, Arthur Boycott reported in seminal entries in Nature [11-13] for with the correct geometry particles can be removed from solution with faster than standard gravity separations if the vessel holding the solution had the tilted sides and the correct geometry. The effect has been termed the Boycott effect and has been utilized in everything from toys to biomedical devices for blood separation. Effective configurations allow separations over 100 times faster [14] than simple gravity settling for controlled conditions. The Boycott effect has been used industrially to separate particles by placing inclined plates in large settling tanks [15-17]. *However, in this effort we explored whether particles can be efficiently and rapidly separated from larger, industrially scalable, flows streams with compact devices using the Boycott effect—and we found that they can!* A graphic of the device is shown in Figure 4. Presently the device is referred to as the Avalanche Boycott Separator since it works on the Boycott effect but the particles separate from solution so fast it appears like an avalanche of particles crashing out of the fluid flow.

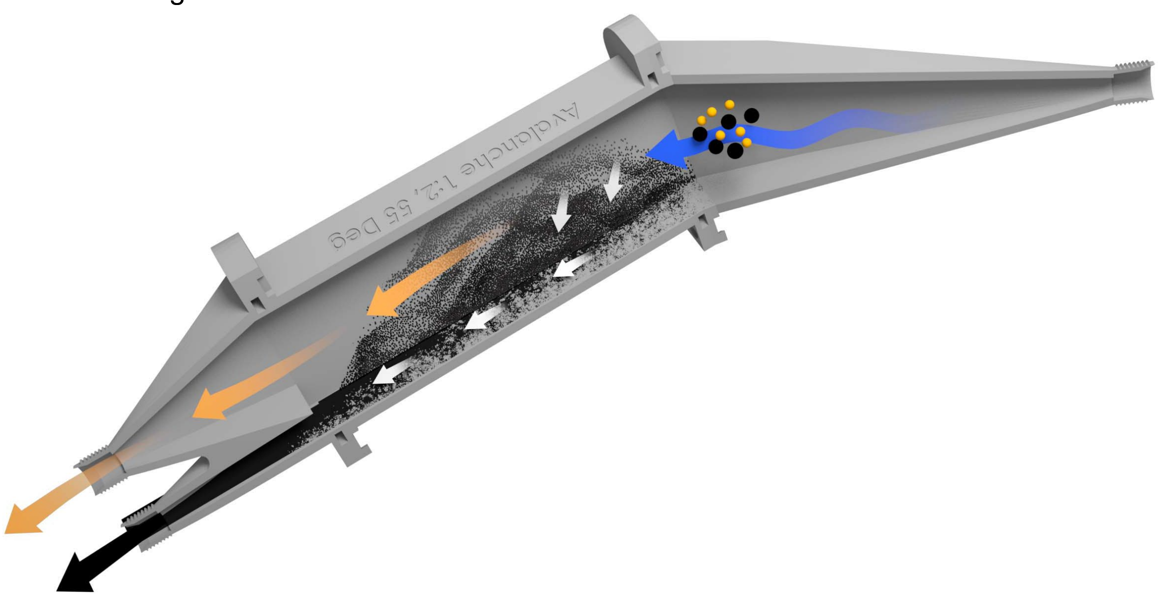

Figure 4. A graphical example of a prototype Avalanche Boycott separator showing fluid and particle flow. The fluid enters from right with a mix of large (black) and small (orange) particles. The geometry of the separator chamber rapidly directs particle out of the main flow allowing them to be removed (lower exit flow on left). Some smaller particles may not be separated and leave with the main flow (upper flow exit on left). Device geometries that provide efficient and size-controlled particle separation for continuous flow conditions ("in-pipe" processing) are under continued investigation and development.

The separator shown in Figure 4 decouples the Boycott induced flows from the bulk fluid flow with a geometry that results in the bulk flow perpendicular to the plane in which the Boycott flows occur. This orthogonal decoupling of the flows is very different than prior Boycott separators that operated in either batch mode or that coupled the flows in continuous mode. This is a fundamental new device that is relatively easy to manufacture, has very low back pressure, and provides efficient separation of both large and small particles. Key device parameters were explored, and viable configurations developed. The first invention disclosure report (IDR) based on this work has been submitted and selected for patenting "Avalanche Boycott Separator". Additional IDR submissions are under discussion and development. Manuscripts are being assembled and will await IP filing before submission.

2.3 In-Pipe Processing

For well over a century traditional chemical manufacturing, mineral extraction, and water treatment plants have used large tanks to provide residence times for crystallization, precipitation, reaction chemistries, particle separation through settling, and many other processes. For industrial scale processing the tanks are often large with volumes in excess of 10,000 gallons to open ponds containing millions of gallons. Multiple tanks/containers for the various process stages are almost always required. The ponds and small lakes normally seen around large mineral processing facilities and municipal water processing plants are easily observable examples of this. The use of batch processing in each tank slows down the processing cycle. Long residence times ranging from hours to days are not uncommon. The main drivers of longer residence times are the slow reaction rates, slow crystal growth rates, and slow settling/separation rates. These problems become much more significant as solution concentrations fall into the trace level, which is the case for many mineral processing applications and water clean-up processes.

A solution to the problems described above would be devices that allow for rapid separations and chemical reactions directly in industrial plant piping to minimize the need for large tanks and storage facilities. *This effort developed and demonstrated that mesofluidic separators are a novel compact cost effective solution to these problems.* Figure 5 is a graphic showing the conceptual process of for replacement large vessels in a chemical processing plant (utilizing for settling, clarification, chemical reactions, separations, etc.) with compact modular mesofluidic devices that can be integrated into industrial piping flows.

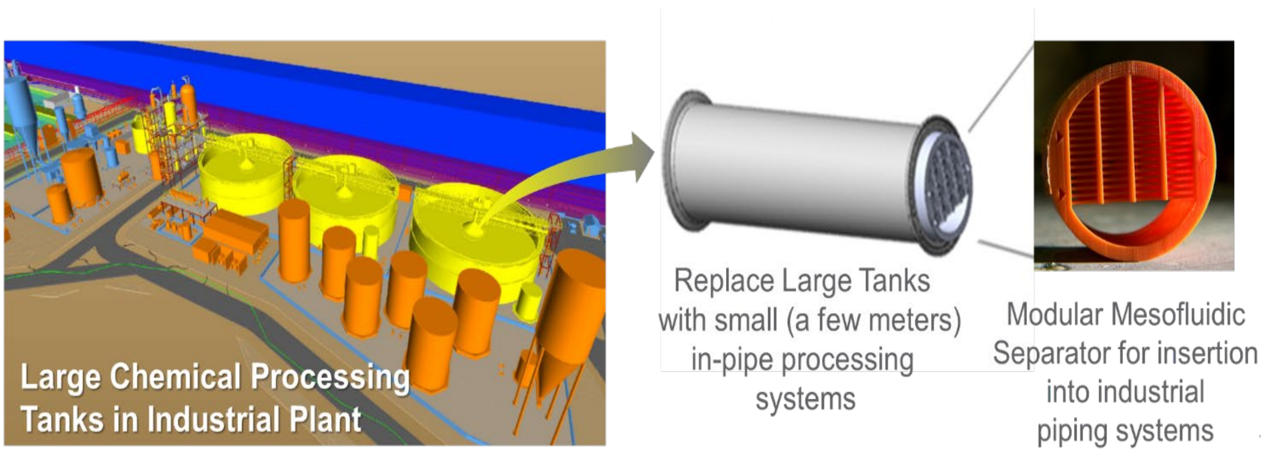

Figure 5. Conceptual Graphic for replacement of large vessels in a chemical processing plant (utilizing for settling, clarification, chemical reactions, separations, etc.) with compact mesofluidic devices that can be integrated into standard industrial piping and flows. The modular mesofluidic devices are designed for the specific application. The mesofluidic devices can be arranged in series to allow a sequence of chemical operations to be achieved as the liquid flows through the pipe. Advances in 3D printing enables cost effective and rapid production of the devices with application specific configurations.

The in-pipe processing concept focuses on conducting all, or most, of the chemical processing or mineral extraction steps directly in the flow streams within the plant piping to minimize the need for high volume solution storage. Elimination of holding pond and/or large tanks (frequent stainless steel and over a million gallons) results in large cost savings (capital and operating).

Customized modular mesofluidic devices can be assembled to allow different process stages to be easily integrated into industrial plant solution flow(s). Efficient rapid removal of particle from the process flow can significantly improve product quality and reduce waste (additional economic advantages). Elimination of the large tanks and physical footprint make this technology advantageous for remote operations such as those used at many locations in the fossil fuel extraction and mining (critical minerals) industrial sectors. For many solutions-based-nuclear-waste-processing efforts the ability to do chemistry in pipe (eliminating large tanks) results in decreased shielding cost, reduced waste, reduced air flow treatment needs, and increased safety. These factors are estimated to reduce the cost (construction and operation) of nuclear waste processing by ~35% to ~90%.

During the course of this effort the team explored a range of capabilities improvements for in-pipe process using mesofluidic devices including:

- Concept development modeling for modular processing with mesofluidic devices.
- Demonstrated mesofluidic devices in sequentially and parallel configurations.
- Demonstrated improve separation efficacy with mesofluidic devices.
- Explored the economics and technical limits regarding device manufacturing.
- Development and demonstration of new mesofluidic devices (as details previously)
- Development and demonstration of mesofluidic devices for mineral and water processing.

A major breakthrough for this effort was the development of a mesofluidic separator that is scalable to work at industrial flow rates and still separate particles by size range in turbulent (rapid) flow streams. The mesofluidic devices were demonstrated for in-pipe/flow configurations for removal of trace toxic metals from water and well as were demonstrated recovery and concentration of valuable and critical minerals. New devices were designed and developed with flow rates up to ~7 gpm demonstrated.

A comprehensive invention disclosure report (IDR) based on this work has been submitted "*Integrated Modular In-Pipe Chemical Processing Invention*". Additional IDR submissions are under discussion and development. Manuscripts are being assembled and will await IP filing before submission.

3.0 Large Particle Separation Testing

Proof of concept testing was conducted on an early Boycott Avalanche device with the positive results leading to the IP invention disclosure filing for the Boycott Avalanche Separator. Publication and conference presentations are being finalize and will be included in the IP filings.

Proof of principle testing was completed for large particle separations using the mesofluidic separator as presented in "*Recovery of Oxpure 612C-50 Coconut Carbon Particles using Bump Arrays*" [18]. Specifically, these tests evaluated the ability to recover large OxPure 612C-50 coconut carbon particles using a mesofluidic separator. This mesofluidic device was configured with a full pin array in the permeate area to improve recovery performance as is visible in Fig. 6. The mesofluidic device had one inlet and two outlets. Larger particles, namely the the OxPure 612C-50 coconut carbon particles, migrate towards the express lane by moving around the posts. Samples were extracted at multiple flow rates evaluated without sample addition to replace that from removed samples.

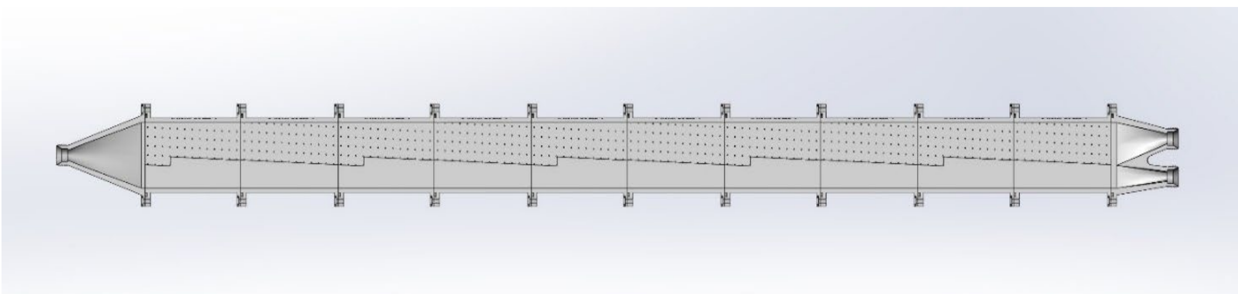

Figure 6. Large particle mesofluidic separator for recovery of carbon particles.

Tests were conducted at three flow rates. All of the measured solids' mass in all three test runs were collected from the express lane and the permeate lane. The recovery of the OxPure 612C-50 coconut carbon particles was determined. A vortex formed at the entrance to the mesofluidic device. Extending the conical entrance prior to the mesofluidic unit may prevent vortex formation in future testing.

The testing shows that at 3 gpm nearly "all of the large particles passing through the mesofluidic separator flowed to the express lane. At the higher 6 gpm flow rate 8.76 grams (99.2%) from the total of 8.82 grams of large carbon particle solids flowed out through the express lane. The slurry mass splits leaving in the express lane flows (prior to drying the liquids) were 49.5% at 3 gpm and 41.5% at 6 gpm" [18].

"Given the high particle separations rates, the large carbon particles can be quickly removed from the slurry mixtures. Having three mesofluidic units in series can reduce the water portion of the slurry to just 12.5% while we would expect over 99% large solids recovery through the express lane flows" [18].

"Cyanide is used to leach gold from crushed ore (solid matrix) into a gold cyanide solution. The gold is extracted from the cyanide solution by adsorption onto activated carbon. The gold extraction process occurs when the activated coconut carbon is placed into tanks that contain the gold cyanide solution either in a batch process or into a continuous flow circuit. The coconut carbon is then removed from the solution for gold recovery" [18].

The testing showed that mesofluidic separation can reduce waste. "In the coconut carbon recovery process used at many gold mines in Nevada, the gold is attached to carbon particles that are still in the cyanide liquor. The mesofluidic system can rapidly remove the gold bearing carbon particles from the cyanide liquor quickly. The benefits include:

- Reduced operational costs and improved performance (99.2+% recovery of carbon particles) when compared to filtration and hydro-cyclones.
- Reduced acid usage for the elution gold stripping phase as only 25% of the fluids will follow the carbon particles to the final express lane when two mesofluidic systems are used in series.

Therefore, this alternative particle removal technique would be advantageous" [18].

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