ANL/NE-15/12

Irreversible Wash Aid Additive for Cesium Mitigation

Small-Scale Demonstration and Lessons Learned

Nuclear Engineering Division

Acknowledgment

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by
Michael Kaminski
Nuclear Engineering Division, Argonne National Laboratory

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TABLE OF CONTENTS

Executive Summary 1
1. Introduction and Purpose 3
2. Methods 5
3. Results 6
   3.1 Small Scale LAKOS Testing 6
   3.2 Small-Scale Separmatic Filters Testing 8
   3.3 Small-Scale Demonstration 13
4. Conclusions and Lessons Learned 15
Acknowledgment 16

EXECUTIVE SUMMARY

The Irreversible Wash Aid Additive process has been under development by the U.S. Environmental Protection Agency (EPA) and Argonne National Laboratory (Argonne). This process for radioactive cesium mitigation consists of a solution to wash down contaminated structures, roadways, and vehicles and a sequestering agent to bind the radionuclides from the wash water and render them environmentally immobile. The purpose of this process is to restore functionality to basic services and immediately reduce the consequences of a radiologically-contaminated urban environment. Research and development have resulted in a down-selection of technologies for integration and demonstration at the pilot-scale level as part of the Wide Area Recovery and Resiliency Program (WARRP) under the Department of Homeland Security and the Denver Urban Area Security Initiative. As part of developing the methods for performing a pilot-scale demonstration at the WARRP conference in Denver in 2012, Argonne conducted small-scale field experiments at Separmatic Systems. The main purpose of these experiments was to refine the wash water collection and separations systems and demonstrate key unit operations to help in planning for the large scale demonstration in Denver. Since the purpose of these tests was to demonstrate the operations of the system, we used no radioactive materials. After a brief set of experiments with the LAKOS unit to familiarize ourselves with its operation, two experiments were completed on two separate dates with the Separmatic systems.

In the first experiment, we tested the Separmatic pressure (S-1) and vacuum filter systems. These were designed to produce potable water by employing diatomaceous earth (SiO_2) as a filter aid. Specifically, fabric filters are pre-coated with a thin layer of diatomaceous earth. The active clarification of the raw feed is provided by the diatomaceous earth while the filter bags provide only support to the earth. This remarkably simple system provides high volumes of potable water with very low capital costs and operating costs. Our tests showed that the diatomaceous earth can be substituted for the vermiculite clay without affecting the function of the filter bags or the hydraulics of the filter units. We pumped a series of vermiculite clay slurry through the Separmatic systems with and without a diatomaceous earth pre-filter and found no trace of the vermiculite clay in the product stream.

Summary-1. The pressurized filter system skid module by Separmatic Systems® that we tested. The feed tank is on the right and the pressurized filter tank is on the left.

In the second experiment, the LAKOS centrifugal separator and the Separmatic filters were connected in series. The goal was to familiarize ourselves with the operation of the LAKOS separator (5–7 gallons per minute) and to qualitatively assess the effectiveness of the LAKOS as a pre-filter for high-concentration slurry feeds. We prepared up to a 10% slurry feed (vermiculite in 0.5 molarity potassium chloride). The LAKOS removed a significant fraction of the vermiculite and the Separmatic filtered the remaining amount, producing a completely clarified product stream. We noted from these tests that the LAKOS purge valve system was difficult to monitor and control. Under normal deployment, LAKOS provides an automated purge control so that may solve some of the problems we had, but we need to engage the vendor to discuss the limitations and possibilities. We also noted a problem in maintaining a homogeneous slurry in the slurry feed reservoir. Instead of maintaining a homogeneous slurry, the clay settled quickly to the bottom of the tank and we had to modify the stir paddle depth to promote its suspension.

1. Introduction and Purpose

The Irreversible Wash Aid Additive process has been under development by the U.S. Environmental Protection Agency (EPA) and Argonne National Laboratory (Argonne). This process for radioactive cesium mitigation consists of a solution to wash down contaminated structures, roadways, and vehicles and a sequestering agent to bind the radionuclides from the wash water and render them environmentally immobile. The purpose of this process is to restore functionality to basic services and immediately reduce the consequences of a radiologically-contaminated urban environment. Research and development have resulted in a down-selection of technologies for integration and demonstration at the pilot-scale level as part of the Wide Area Recovery and Resiliency Program (WARRP) under the Department of Homeland Security and the Denver Urban Area Security Initiative. As part of developing the methods for performing a pilot-scale demonstration of the wash aid system at the WARRP conference in Denver, Argonne conducted small-scale field experiments at Separmatic Systems. The main purpose of these experiments was to refine the wash water collection and separations systems and demonstrate key unit operations to help in planning for the large scale demonstration in Denver. Since the purpose of these tests was to demonstrate the operations of the system, we used no radioactive materials. After a brief set of experiments with the LAKOS unit to familiarize ourselves with its operation, two experiments were completed on two separate dates.

In the first experiment, we tested the Separmatic filter systems. We tested both their pressure and vacuum filter systems. These were designed for producing potable water by employing diatomaceous earth (SiO_2) as a filter aid. Specifically, the fabric filters are pre-coated with a thin layer of diatomaceous earth. The active clarification of the raw feed is provided by the diatomaceous earth while the filter bags provide only support to the earth. This remarkably simple system provides high volumes of potable water with very low capital costs and operating costs.

Both systems operate as combined settling tanks and fabric filters. In the pressure filter system (Fig. 1), the raw feed can be treated in prior mixing tanks, and then is fed into a closed, pressurized tank. The larger particles will settle to the bottom of the tank while the feed is forced through annular filters coated with the diatomaceous earth contained within the tank. In the vacuum design (Fig. 2), the raw feed can be treated in prior mixing tanks and enters the filter tank, where a vacuum pump draws the fluid through the fabric and discharges the clean effluent. The vacuum tank design can be scaled more easily than the pressurized filter design and costs significantly less.

We prepared slurries of the vermiculite clay in potassium chloride and passed these through the Separmatic filter system to determine the filter's efficiency in different filter modes. We then used these tests to design the second set of experiments.

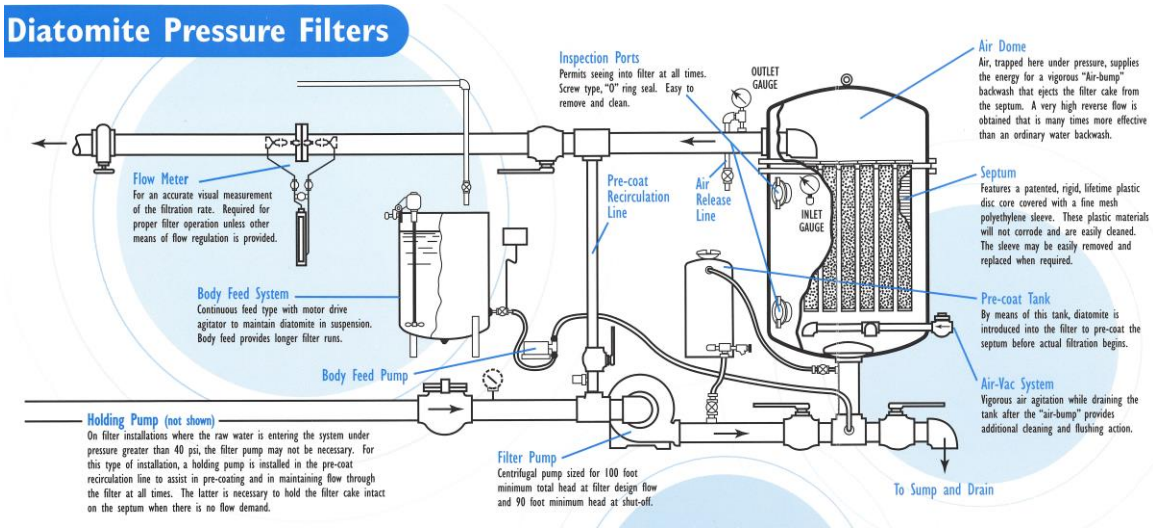


Figure 1. Schematic of the pressurized filter system (top) by Separmatic Systems®.

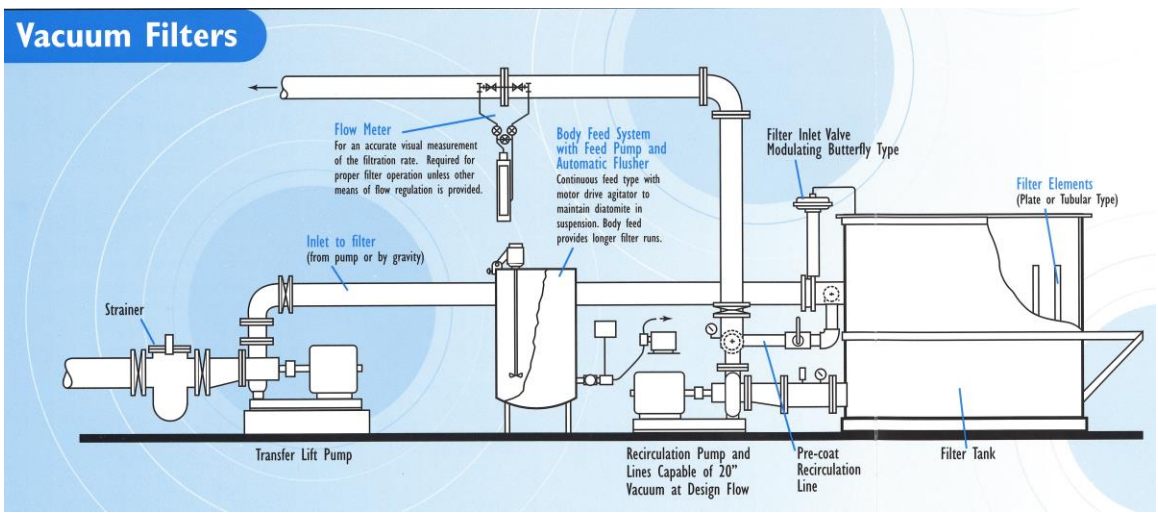


Figure 2. General schematic of the vacuum filter system by Separmatic Systems®.

In the second experiment, the LAKOS centrifugal separator and the Separmatic filters were connected in series. The goal here was to familiarize ourselves with the operation of the LAKOS separator and to qualitatively assess the effectiveness of the LAKOS as a prefilter for high-concentration slurry feeds.

2. METHODS

Grace Co. shipped 50 lb. buckets of VCX 205 vermiculite clay. We obtained the potassium chloride salt from a local home improvement store (Table 1) and diatomaceous earth from Celite Corporation (Celite®545, 100% flux calcined diatomaceous earth, <4% quartz, 2.3 g/cm^3 true density).

Table 1. Reported Chemical Analysis of Nature's Own Potassium Chloride Water Softener Crystals (99.7% Confidence, U.S. Mesh 4 pellets)

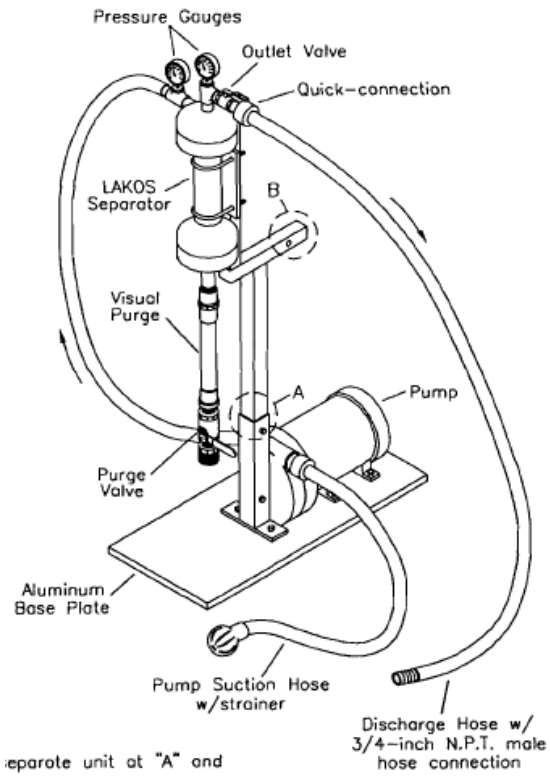
Chemical	Typical (%)	Range (%)
Potassium Chloride KCl	98.90	98.45 – 99.35
Sodium Chloride NaCl	0.90	0.60 – 1.20
Calcium Sulfate $CaSO_4$	0.06	0.01 – 0.11
Calcium Chloride $CaCl_2$	0.01	0.00 – 0.04
Magnesium Chloride $MgCl_2$	0.04	0.00 – 0.09
Moisture	0.10	0.04 – 0.16
Water Insolubles	100 ppm	70 – 130 ppm

The feed was prepared by adding the potassium salt to local tap water and then adding the vermiculite clay. The mass of salt needed to prepare a 0.5 M solution was: 0.565 kg KCl for four gallons of tap water, 0.705 kg for five gallons of tap water, and 10.575 kg KCl in 75 gallons of tap water. The slurry concentration was prepared by adding vermiculite (as-received from the vendor) slowly into the test vessel as specified for each experiment (either 5 gallon bucket or 75 gallon feed tank) while stirring the vessel contents to maintain a suspension.

3. RESULTS

3.1 Small Scale LAKOS Testing

For the small-scale demonstration we ordered the smallest LAKOS separator available—the LAKOS Portable Test Rig that includes 3/8" LAKOS Separator (IL-0037B, 3–6 gpm nominal) operated by a self-priming pump (Dayton® ½ HP, 7000 rpm, 25 GPM maximum, Fig. 2).



SPECIFICATIONS

Separator: LAKOS Model ILB-0037

Flow Rate: 5–6 U.S. gpm (1.4 m^3/hr)

System Weight: 45 lbs (20.5 kg)

Pressure Output: 33 psi (2.3 bar)

Pump: Teel Water Systems, Model 2P110A Centrifugal, 1/2 H.P.

Pump Lift: 10 feet (3 meters)

Pump Housing: Cast Aluminum

Impeller: Valox 420

Shaft & Volute Seal: Buna N

Power Cord: 8 ft (2.4m), 18–3 SJTO

Motor: 1/2 H.P., Single Phase, 50–60 Hz, 115V 8,000 R.P.M. Series

Motor Shaft: Cold Rolled Steel

Motor Bearings: Permanently Lubricated

Maximum Amps: 8.0

Maximum Fluid Temperature: 100°F (38°C)

Figure 2. Labeled schematic of the LAKOS portable test rig (left) and actual unit (right). The technical specifications are provided.

To start, a five-gallon bucket was filled with tap water. The suction hose of the LAKOS unit was placed into the bucket and the pump was activated for several minutes to flush the system. Then, the purge and separator were drained. Next, we mixed into the five-gallon bucket 10% vermiculite in salt-water (0.5 M KCl) to a volume of 4 gallons. The slurry suspension was maintained by a stirrer (Coframo) operating at 900 rpm. The suction hose was placed in the bucket and the pump was activated. Samples of the feed and separated fractions were collected into plastic tubes. The general operating characteristics were noted as well as the volume of solids in the settled slurry for each sample.

We allowed ~3.5 gallons of the feed to run through the unit in a single run. We collected a sample from the 10% slurry feed, samples from the post-filter discharge tube, and a sample from the purge. On the first run, the purge valve was initially closed when the pump was activated and kept closed for several seconds before opening. We noticed that the visual purge was filling very quickly with clay (overcapacity) which led to a higher-than-expected slurry concentration in the discharge. After the run, the purge contents and discharge contents were added back to the five-gallon feed bucket and the contents were stirred.

On the second run, the purge valve was partially opened immediately after starting the pump to permit the purge contents to dump and prevent overflow of the purge capacity. A post-filter sample was collected where we noted that more than 10% of the original solids content was passing into the discharge tube. After the run, the purge and discharge contents and post-filter sample were added back to the five -gallon feed bucket and the contents were stirred.

On the third run, we repeated the second run and collected a post-filter sample from the discharge tube and a sample from the purge tube (Fig. 3). Once the feed bucket was emptied, the slurry collected from the post-filter discharge tube was used as the feed for the next set. This was only a small volume of material (<1 gallon). When we ran this post-filter discharge volume through the LAKOS, we noticed that not much material was passing through to the discharge tube and we only collected 100–200 mL. We suspect that hold up within the system was the likely cause.

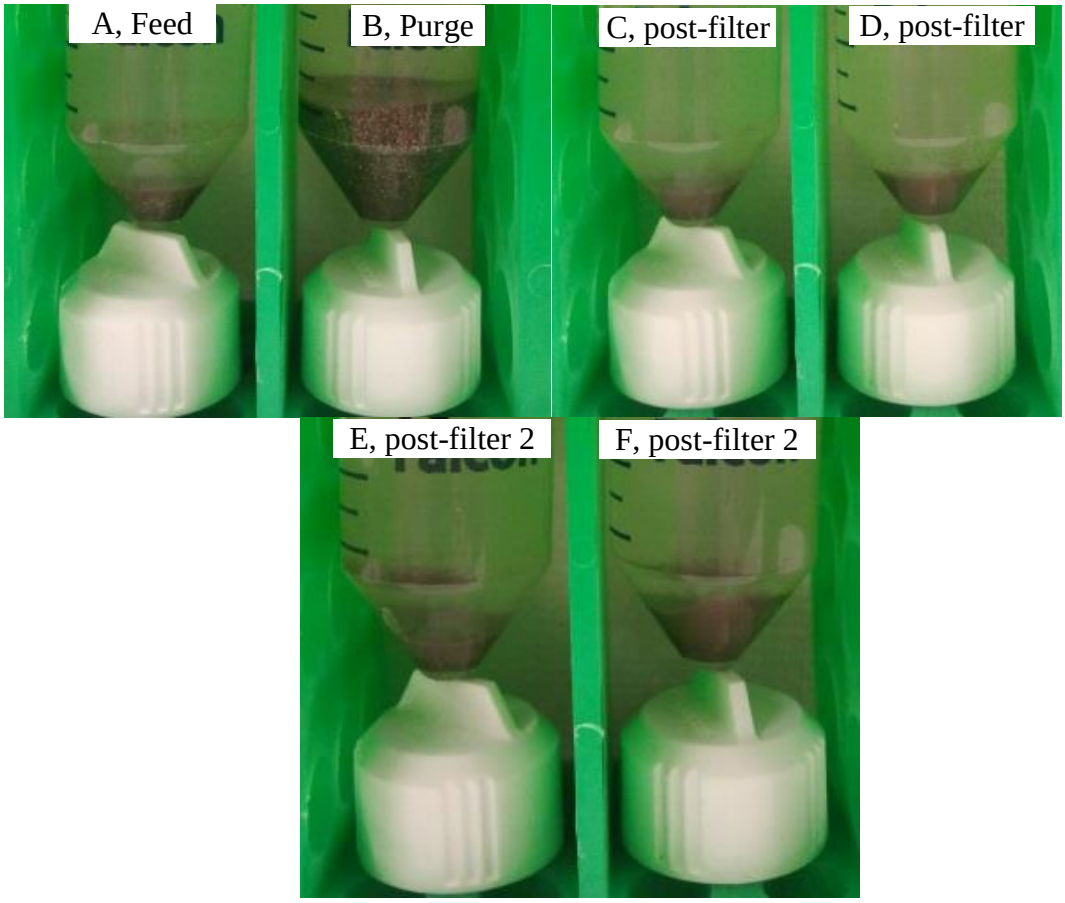


Figure 3. View of the relative slurry concentrations within the original feed (10% slurry) (A), the purge content (B), the post-filter discharge contents (C and D), and the post-filter discharge contents using C and D as the feed (E and F).

From these tests, we identified two significant problems. One was the inadequate size of the feed vessel into the LAKOS unit. The largest vessel size available to us with appropriate mixers to maintain a slurry was 5 gallons. At 3–6 gpm, this permitted only ~1-min of operating time. We quantified the actual flow rate to be approximately 10 gpm and so our operating times were too short to reach the steady-state. The other problem was in controlling the purge valve to maintain the proper pressure drop within the LAKOS unit to support the centrifugal force needed to effect the separation of solids from the liquid. To conduct larger scale tests, we shipped the LAKOS unit to Separmatic Filters for use in the "Small-Scale Demonstration" (see below).

3.2 Small-Scale Separmatic Filters Testing

The first tests at Separmatic Systems was designed to familiarize ourselves with the operation of both the pressurized filter unit (closed tank system, 16 gal/min, 16 ft^2 filter surface area, 3450 rpm ½ HP centrifugal pump) and the vacuum filter unit (open tank system, 5 gal/min, 5 ft^2 filter surface area, 30 gallon) sold by Separmatic Systems.

We used a small laser to qualitatively detect solids breakthrough. Some samples were collected and brought back to Argonne for interrogation by static light scattering (Brookhaven ZetaPlus Analyzer) to detect solids in suspension.

The general procedure for operating the pressurized filter skid unit (Fig. 4) was as follows:

1) Fill the feed tank (75 gallon) with tap water, run this through the filter tank unit, and collect pre-filter and post-filter water as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
2) After adding KCl salt to tap water in feed tank (23 lbs. to 75 gallons) to produce a 0.5 M solution, run solution through unit and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
3) In the feed tank, suspend clay (0.1 wt% slurry, 0.69 lb clay) into 0.5 M KCl salt in tap water, run slurry through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
4) In the feed tank, suspend clay (1% wt% slurry, 6.9 lb clay) into 0.5 M KCl salt in tap water, run slurry through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
5) Empty filter tank using the purge valve and flush the entire system thoroughly with tap water.
6) Add diatomaceous earth (3.2 lb Celite® C545 in 75 gallons) to tap water, run tap water through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer). (Note: In the Separmatic filter systems, the diatomaceous earth serves as a critical pre-coat on the filter to remove microscopic materials including bacteria to make potable water.)
7) After adding KCl salt to feed tank water (23 lbs to 75 gallons) to produce a 0.5 M solution, run solution through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
8) In the feed tank, suspend clay (0.1 wt% slurry, 0.69 lb clay) into 0.5 M KCl salt in tap water, run slurry through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).
9) In the feed tank, suspend clay (1% wt% slurry, 6.9 lb clay) into 0.5 M KCl salt in tap water, run slurry through unit, and collect pre-filter and post-filter as procedural blanks for solids analysis (1 sample needed from each and placed in laser cuvette and interrogated on-site using laser pointer).

Samples of the feed and separated fractions were collected into plastic tubes. The general operating characteristics were noted as well as the volume of solids in the settled slurry for each sample.

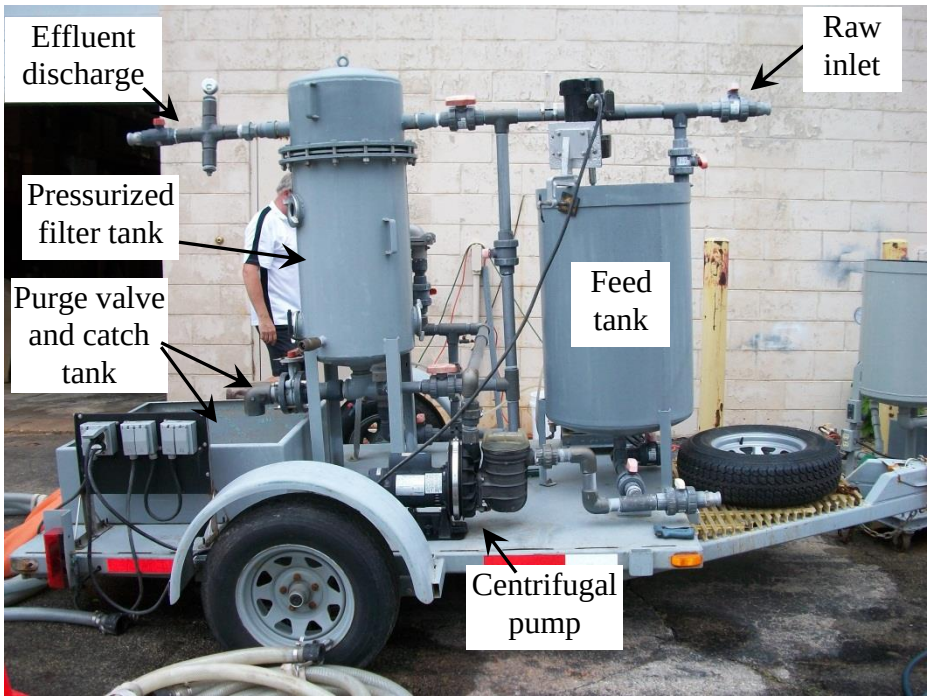


Figure 4. The small-scale Separmatic Systems® pressurized filter skid module that we tested.

Step 1 above was designed to observe any hold-up solids in the existing system before initiating our own tests. Steps 2–4 were designed to observe a breakthrough of clay material through the filters and any change in hydraulics due to filter clogging. In step 5, we were able to observe the procedure for emptying the filter tank and collecting the concentrated solids. Steps 6–9 were designed to observe a breakthrough of clay material through the filters pre-coated with diatomaceous earth and any change in hydraulics due to filter clogging.

The attached video summarizes the system. The first video shows how the system works in general (see "Separmatic demo 7 4 12 75 gal summary.mov"). The second shows the purging of the filter tank (see "Separmatic demo 7 4 12 Spent filter purge.mov").

After steps 1 and 2 above, we did not observe any pre-existing solids in the separation system. After step 3, we did not observe visually, via the diffraction of laser light through the sample vial, any indication of significant breakthrough of particles from the 0.1% clay feed[1]. After step 4, we did not observe visually, via the diffraction of laser light through the sample vial, any indication of significant breakthrough of particles from the 1.0% clay feed. In step 5, the filter tank emptied quickly (within a couple minutes). The filter could be observed through the view ports and there was no clay held up on the filter fabric after the purge. The system was flushed to remove clay held up within the conical bottom of the filter tank. After steps 6 and 7, we did not observe visually, via the diffraction of laser light through the sample vial, any indication of significant breakthrough of diatomaceous earth particles through the filter. After steps 8 and 9, we did not observe visually, via the diffraction of laser light through the sample vial, any indication of significant breakthrough of clay particles from the 0.1 and 1.0% clay feed, respectively through the filters pre-coated with diatomaceous earth.

Of note, we did not observe an increase in the pressure drop across the filter during our tests since the concentration of clay (0.1 and 1% by mass) and volume of water (75 gallons) were much lower than the unit was designed to operate.

We brought the samples back to the laboratory for interrogation by static light scattering to determine the presence of low concentrations of fines. We confirmed our results from the field. The background count rate for particles in tap water was $<5x10^3$ counts per second while the effluent from the 1% suspensions were $<6x10^3$ cps. Therefore, we conclude that there was no significant breakthrough of clay or diatomaceous earth particles using either the uncoated pressure filters or the diatomaceous earth-coated filters.

Several important observations were noted in the pressurized filter system tests. Similar to the testing at Argonne, we had difficulty controlling the purge valve to maximize the concentration of solids in the purge while minimizing the concentration of solids in the discharge to the Separmatic filter unit. We will need to discuss with LAKOS representatives in more detail how best to control this valve in the expected operating environment. Next, even though the diatomaceous earth is a critical component of the Separmatic filter design for producing potable water, there does not appear to be a need for it in our application. The existing filters effectively prevent the vermiculite clay from passing into the discharge flow. Next, we had difficulty maintaining a constant suspension of the 1% slurry due to an inadequate stirrer set-up. This was also due to the quick settling of vermiculite particles in the feed tank. Finally, we had insufficient feed tank capacity to approach a limit on the capacity of the Separmatic filter which prevented us from computing a scaling-factor for predicting the size of the Separmatic filter unit needed for every unit of slurry treated. This information is needed so we can plan how often the Separmatic filter would need to be purged and regenerated.

[1] Clay in suspension would result in bright flashes of light along the laser beam path (for larger particles) or a diffuse broadening of the laser beam (for smaller particles in suspension).

We next tested the vacuum filter system by Separmatic Filters (Fig. 5). This is an open tank system that pulls a vacuum on the discharge side to draw fluid through the filter. We added 11.5 ounces of vermiculite clay into the 30 gallon filter tank filled with tap water while the unit was recirculating water from the discharge pipe to the filter tank. The clay flocculated at the top of the tank and periodically floccs would descend to the bottom of the tank. This action differs markedly from when diatomaceous earth is added to the filter tank. With diatomaceous earth, the particles descend into the tank and are drawn to the filter membrane by the flow and coat the filter surface. We did not observe clay particles in the discharge pipe as measured by an in-line turbidity meter.

The open tank design of the vacuum filter design is much easier and cheaper to scale than the pressurized filter system and is a concept worth pursuing as we continue with larger scale demonstrations and method development. To purge or backwash the vacuum system, the valves are switched so that the pump that feeds the filter tank is diverted to the drain pipe at the bottom of the filter tank. As the water level in the filter tank drops below the fabric filters, the particulates on the filter surface fall freely and are expelled out the drain. Spray bars located just below the top of the filters remotely spray any residual material off the filters so the tank is ready for another run. Backwashing takes approximately 5 minutes, similar to that of the pressurized filter system.

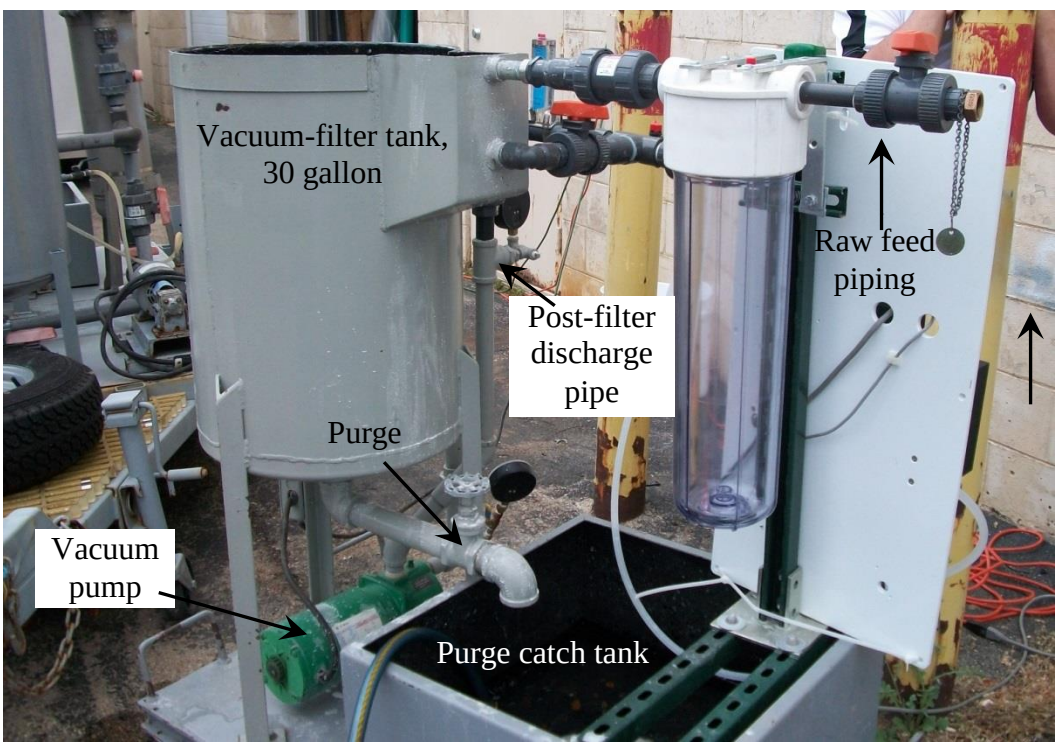


Figure 5. Vacuum filter separations test skid by Separmatic.

3.3 Small-Scale Demonstration

The LAKOS portable Test Rig was shipped to Separmatic Systems for integration into their pressurized filter system. The system was integrated with the LAKOS unit as the primary filter with the discharge fed directly into the pressurized filter tank of the Separmatic unit, bypassing the Separmatic feed tank (Fig. 6). The discharge from the Separmatic filter would be free of suspended material. The same 75 gallon feed tank was used as in the previous tests at Separmatic. In these tests, we sought to determine the filter efficiency and ensure no breakthrough of clay particles as well as become familiar with the operation of the filter system.

The tests were divided into two runs. In the first run, we filled the feed tank (75 gallons) with tap water. Using only the LAKOS ½ HP pump, we passed the entire contents of the feed tank through the LAKOS and Separmatic filters to determine the flow rates through the system. Shortly after system start-up, we measured a discharge flow rate of 7.5 gpm (5 gallons/40 second).

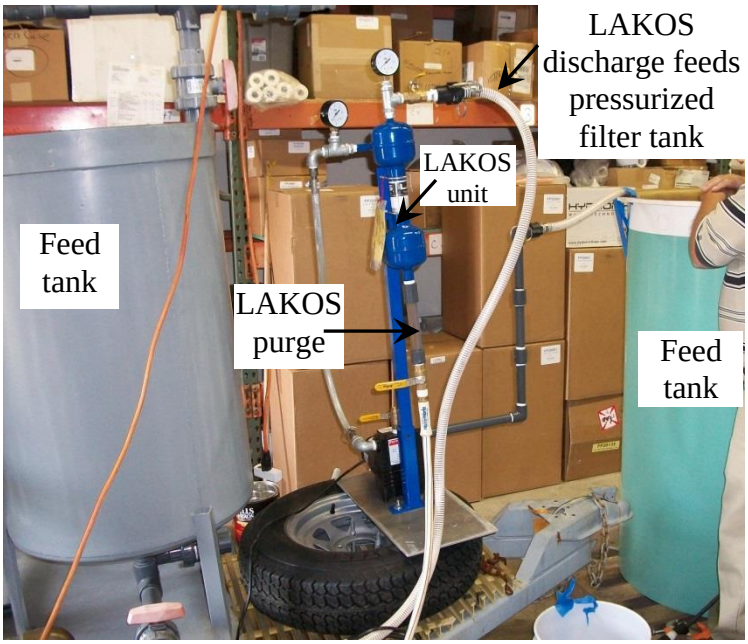


Figure 6. Small-scale demonstration skid unit containing the LAKOS unit bypassing the feed tank and feeding the pressurized filter tank of the Separmatic unit (not shown).

In run 2, we refilled the feed tank with tap water and dissolved KCl (23.3 lb.) into it. Then, we added 69 lbs. of vermiculite clay (10% slurry) and stirred for 10 min. The clay settled to the tank and was difficult to suspend. We changed the single stir paddle to a dual blade mixer that improved the mixing, although we likely did not attain a homogenous suspension, since we could still feel clay settling on one side of the tank as we adjusted the position of the mixer. Once we were satisfied with the mixer position, we started the LAKOS pump and collected samples. We estimated the flow rate from the

LAKOS purge piping to be 2.6–4.0 gpm (5 gallons/74–115 seconds). We noted that the solids content in the LAKOS discharge pipe (the feed to the Separmatic filter unit) contained low solids and less than what we had observed while running the LAKOS unit at Argonne using the 5-gallon feed volumes. The effluent from the Separmatic unit was very clean with no observable particles. Optical pictures of the various samples are shown in Fig. 7. We note that the LAKOS discharge solids concentration is lower than the feed and the purge is much more concentrated than the feed. However, we would expect the LAKOS to remove closer to 90% of the solids, but clearly, this is not occurring. The separation efficiency appears to be closer to 50%. This may be due to the very high solids content and the difficulty in controlling the purge valve on the LAKOS unit.

The attached video shows the operation of the combined filter systems. The video shows the operation of this small-scale demonstration unit containing the LAKOS centrifugal separator in series with the Separmatic pressurized filter (see "Separmatic demo 8 10 2012 Description of system.mov" and "Separmatic demo 8 10 2012 Description of running system.mov").

Figure 7. Sample from the feed (10% slurry, A), LAKOS discharge (B), the Separmatic filter discharge (C), and the purge from the LAKOS separator (D).

4. Conclusions and Lessons Learned

These tests allowed us to study the operation of the down-selected separations systems. From these tests, we could affirm that the separations systems we selected were simple to operate (see below for additional comment), required very little prior training, were effective in performing separations, and would be suitable for the pilot-scale demonstration in Denver.

There were important lessons learned from these tests. The first was the issue of maintaining a constant slurry concentration in the feed. The LAKOS centrifugal separator is designed for solids content generally less than 10% and preferably less than 1% solids. When the solids content is higher than 1%, the company prescribes a dual separator design where two LAKOS units are connected in series so that the first unit reduces the solids content to below 1% for the second unit. Our tests showed that at higher slurry content, the vermiculite clay does not separate to >90% which would be suggested by the density of the material. In practice, the suction of slurry from a reservoir will provide a feed that will vary greatly depending on the position of the intake and the degree of mixing of the slurry. Related to this, we noted that the LAKOS purge valve system was difficult to monitor and control. In regular deployment, LAKOS provides an automated purge control valve that regulates the dumping of purge material so that the proper pressure drop is maintained across the unit for optimal centrifugal forces and separation of material. Manual control may have led to less than expected separation efficiency (~50%). For these issues, we will engage the vendor to discuss the limitations and possibilities.

Our goal is to be able to run the system for prolonged periods without the need for user intervention, so that the dose to the worker can be limited. Witnessing the entire system in the small-scale demonstration allowed us to think more clearly in terms of remote operation. In our small-scale demonstration we noted the need for an automated purge valve on the LAKOS unit; not only to control the flow, but also to limit the dose to the worker. An automated purge is also required on the Separmatic system and is provided by the manufacturer. In addition, we noted the rapid settling of vermiculite clay in water. As we draw water into the LAKOS, this rapid settling is an asset because it should permit the LAKOS unit to operate efficiently. The remaining suspended material will travel to the Separmatic filters. The design of the Separmatic filters is fortuitous because the filter tank can act as a settling tank. Therefore, we will wish to take advantage of the settling capacity of the filter tanks to reduce the number of backwashes and purges needed to regenerate the Separmatic filters. That is, the vermiculite will tend to settle at the bottom of the tank instead of coating and clogging the filters. With this in mind, the vacuum filter design by Separmatic might be the better choice because we have much more versatility in integrating a large capacity settling tank with elevated filters without the cost associated with a pressure tank design. The larger tank and automated purging and/or backwashing will speed the operations and limit the dose to the workers.

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Nuclear Engineering Division
Argonne National Laboratory
9700 South Cass Avenue, Bldg. 205
Argonne, IL 60439-4854

www.anl.gov

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