Example 3.4: Continuous Process for Treatment of Heavy Metal Liquid Waste

Start Date: January 1, 2004
End Date: December 31, 2004
FYE: December 31, 2004

Issues Addressed Related to SR&ED:

- Continuous multi-stage process
- Experimental Development zone (ED zone) using all or a Portion of Full-scale Equipment
- Full-scale Plant trials in support of shop floor SR&ED project
- Experimental Development using a Commercial Facility

Preamble:

This example has been developed for illustration of the concepts that characterize experimental development (ED) using a commercial plant. The example is intended to provide guidance on how to apply the principles contained in Chemicals Guidance Document 3 Part 1 [1] for the review of chemical sector claims.

Background:

In the present scenario Company B is the operator of a chemical processing facility. The company operations generate a total volume of 500,000 L/week of a hazardous liquid waste containing up to 500 mg/L of dissolved heavy metals in the fresh feed.

Company B was required to remove large concentrations of heavy metals (varying randomly from 50 to 500 mg/L in waste feed) to "trace" or non-detectable levels in the effluent stream, which is discharged to the environment. Proposed environmental restrictions on discharge limits for heavy metals are such that Company B will not be able to meet the new federal standards for effluent release with its existing water treatment technology. The technology consists of an integrated 5-stage plant based upon membrane technology and two thin film evaporators (see Fig. 1). The difficulty facing the company may be expressed with the following example: currently the technology is capable of reducing cadmium levels to about 10 μg/L, whereas the required standard for cadmium is about to drop from 20 μg/L to 5 μg/L.

A microfiltration (MF) system is typically used for the removal of suspended solids larger than a nominal 0.2 microns in diameter. In the envisaged application for this project, the company intends to extend the capability of the MF system, by giving it the ability to remove a large fraction of the dissolved heavy metal salts. This will be accomplished by introducing metal ion sequestrants into the waste feed. In this way the dissolved metal ions will be adsorbed into a suspended solid, making it possible for their removal by filtration through the MF system. The goal of the present project described here is to develop a process that can be implemented within the constraints of the equipment presently in place at the Waste Treatment Centre (WTC).

Company B staff examined several competing technologies currently available for heavy metals removal from solution, including ion exchange (IX) resin technology. The use of IX for the removal of metal ions in solution is well documented in the scientific and engineering literature. Moreover, IX technology has matured to the point where the resins can be judiciously chosen for the precise application, and can be matched to meet the selective removal of certain contaminants in the waste stream. However, in the past, the IX resin has always been used as a fixed bed IX exchange process, where a bed of "spent" or saturated resin is periodically regenerated with an appropriate solvent. Company B technical staff proposed that instead of using a fixed bed concept, the resin would be dispersed as fine particles (50 micron Sauter diameter), or as “seeds”, within the processing environment of a microfiltration (MF) system.

The removal efficiency of the downstream reverse osmosis stages (consisting of spiral wound and tubular reverse osmosis units) is insufficient to adequately remove the heavy metals at the levels in which they were known to be present in the feed stream from the MF system.

A total of 80,000 L of waste is processed per plant trial from which 1,600 L of MF backwash concentrate containing the used resin particles is produced. The remaining 78,400 L is sent to the membrane plant where there is a 10-fold volume reduction factor. As a result, 7,840 L of reverse osmosis concentrate (10% of MF filtrate) and another 1,600 L of MF backwash concentrate (2% of original waste feed volume) is sent to the thin-film evaporator for further volume reduction and final encapsulation with bitumen.

Company B planned to conduct lab and bench-scale tests in order to establish resin types or combinations that could be used in particulate form for the selective removal of the heavy metals to trace levels.

Results of the lab study were to be used to develop a pilot-scale study. The pilot would consist of an MF system to carry out a series of experiments to investigate the removal efficiency of heavy metals using the proposed IX resins. Some of the goals of this project would include establishing the effects of resin types and combinations, particle size, feed and crossflow velocities, and backwash frequency, on removal efficiency. In addition, the ability to encapsulate the waste particles using bitumen may turn out to be dependent upon heavy metal loading onto the resin. This potentially would have to be investigated.

The set of operating protocols that were used for the pilot-scale trials were adapted to the full-scale system.

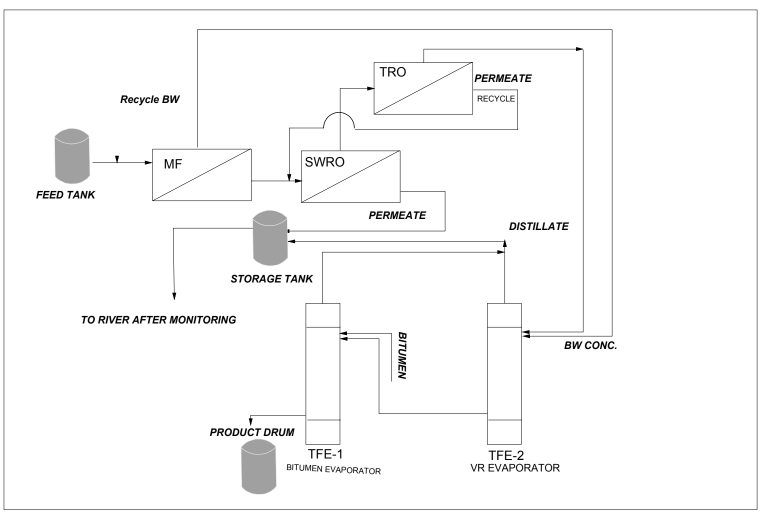

Fig. 1 : Integrated Hazardous Waste Processing Plant

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Acronyms:

Major Processes:
MF = Microfiltration system
TRO = Tubular Reverse Osmosis Membrane System
SWRO = Spiral Wound Reverse Osmosis Membrane System
TFE-1 = Thin Film Evaporator #1 (used for Solidification with Bitumen)
TFE-2 = Thin Film Evaporator #2 (used only for Volume Reduction of combined TRO concentrate and Microfiltration system backwash streams)

Waste streams:
BW Concentrate: Microfiltration Backwash Concentrate
Recycle BW: Microfiltration Backwash recycled for Further Processing with Microfiltration system
Permeate: Clean water from SWRO system
Distillate: Evaporator overheads from TFE-1 and TFE-2

1A. Scientific or Technological Objectives

The specific technological objective of this project is to augment the capacity of the microfiltration (MF) system in order to reduce the loading on the more expensive reverse osmosis downstream stages. Analysis of the new regulations suggests that an improvement of at least 50% efficiency of heavy metal removal at this stage may be sufficient to meet their requirements.

The company felt that by using fine IX resin (greater than 0.2 microns in diameter) in suspension as means of sequestering dissolved heavy metal ions from solution and thereby making them susceptible to removal by filtration, they might be able to meet the regulatory requirements.

The project involves experimental development in the fields of chemical and environmental engineering.

1B. Technology or Knowledge Base or Level

Company B knew that IX resins in a fixed bed could be used for the selective removal of heavy metal ions in solution. A drawback of the fixed bed IX system is that it is susceptible to fouling by organics and other contaminants, which leads to large pressure drops and more frequent regeneration. An advantage of incorporating the resin in the MF system is that the surface area of the resin in contact with the waste solution is maximized, leading to better mass transfer. However, a disadvantage of using the resin in a dispersed manner is that the resin cannot be regenerated.

Company B also knew that the resin would be removed with the microfiltration (MF) backwash concentrate. They anticipate enhanced removal of heavy metal ions from solution due to the increased surface area of the fine IX resins. The impact of fluid hydrodynamics on the removal efficiency would need to be investigated.

1C. Scientific or Technological Advancement

The technological advancement being sought is the development of a continuous process using fine IX resins for the treatment of heavy metal-laden wastes, to achieve very low residuals in the effluent stream, and to encapsulate used resin particles within bitumen for safe disposal.

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1D. Description of Work in the Tax Year

January to March 2004

Company B technical staff performed a series of lab and bench-scale studies with simulated waste solutions to determine what IX resins could be used for the selective removal of heavy metals. Experiments were initially done in a lab-scale stirred tank reactor. A literature study showed that there were 25 possible resins that could be used for this application. Upon further analysis, eight of these resins were selected for the lab studies. Three promising resins were identified from the lab study that were used for subsequent stages of the work. At this stage of the investigation there was no attempt made to study the range of feed and crossflow velocities that would be used for the larger-scale work. The range of pH expected during the normal course of variations for waste treated with the full-scale system was investigated as part of the lab study.

A feasibility report on the use of a seeded IX resin/MF concept was issued to senior staff on March 31, 2004.

April to June 2004:

The goal of this phase of the work was to determine if it was feasible to use a seeded IX/MF concept in combination with a gas-backwashable crossflow MF system for the selective removal of dissolved heavy metals in a liquid waste solution.

Company B carried out experiments on samples of actual liquid waste to determine if the seeded IX/MF resin principle could be demonstrated using a pilot-scale MF system and preselected IX resins (identified from the lab study). A pilot-scale MF system was purchased specifically for these experiments. Three different resins, identified from the lab-scale study, were studied at the pilot-scale. Feed flowrates (30 – 100 L/min) and MF gas backwash frequencies (5 minutes to 30 minutes at 5 minute intervals) were varied within the ranges expected for the large-scale system as part of the scope of this phase of the project. In addition, Company B contracted with a local analytical laboratory to carry out specific chemical analyses on the feed and effluent streams to verify that the removal efficiencies without the IX resin was a true match for that obtained with the existing system. A similar set of analyses was used to measure the effects of putting each of the candidate resins into the system.

Results from the pilot study revealed that a seeded IX/MF concept in combination with a gas-backwash MF system for the selective removal of dissolved heavy metals was feasible with one of the resins. The selected resin was used for further full-scale plant trials at Company B's integrated 5-stage waste treatment plant (see Fig. 1). During this period, a project team was assigned by Company B for the plant trials.

July to August 2004:

Commissioning of the full-scale MF system was carried out using the selected IX resin identified from the initial phase of this study. Flushing of the MF system was carried out to ensure that all process lines are cleaned of any residual waste streams. The system was ramped up for the planned sequence of plant trials using the actual waste and predetermined mixtures of fine resin particles in slurry. The resin concentration was varied from 10% by weight for Trial 1, to 20% for Trial 2, and 30% for Trial 3. These were chosen to be within the maximum range of resin solids that could be safely incorporated into the final bitumen end-product.

Full-scale trials were carried out to determine if the selected resin would be suitable for the selective removal of heavy metals under dynamic full-scale plant conditions. For each trial a volume of 80,000 L of waste (representing 1 batch of waste) was processed at flowrates ranging from 30 L/min to 60 L/min. A trial typically lasted for 36 hours (or 3x 12 hour operator shifts) including the initial transition period preceding each trial, as well as the time to clean and flush the system after each trial. During each trial the feed crossflow rates on the MF system were varied between 50 and 200 L/min at 50 L/min intervals, and filtrate was continuously removed from the system at 30 to 60 L /min. After sampling, filtrate was recombined with recirculated feed and returned back to the feed tank. The backwash concentrate was stored in a separate tank for eventual reprocessing through the thin film evaporator plant. The gas backwash frequency for the removal of IX resins was varied from 5 to 20 minutes in duration.

During Trials 1-3, the MF system was taken off-line from the rest of the integrated plant shown in Fig. 1 and the company planned to claim an ED zone consisting of the MF system alone. There was no commercial activity during this time since all waste would need to be reprocessed through the entire plant (including reverse osmosis and evaporator stages) after these trials were completed.

During Trial 2 (20% resin loading) results were so promising that the MF plant was connected back on-line with the remainder of the reverse osmosis plant, and all three units (microfiltration, and two reverse osmosis units) were placed into service. This was done to determine the ultimate removal efficiency using the entire membrane facility. Trial 2 was repeated in its entirety (Trial 4) to check the reproducibility of the data. The thin film evaporators were not in service for Trials 1-4.

Preliminary results demonstrated that with a 20% resin loading it was possible to remove greater than 90% of the dissolved heavy metals in the feed, and still be within the known processing constraints for maximum solids loading for the downstream bitumen thin film evaporator. The MF backwash concentrate and RO concentrate were stored for subsequent trials with the thin film evaporators.

<u>September – December 2004</u>

Work during this period was focused on the final volume reduction and encapsulation stages. It was anticipated that there would be a further 2-fold volume reduction factor using the first (non-bituminizing) thin film evaporator.

Although the seeded IX resin/MF waste treatment process seemed to be effective for the treatment of this heavy metal liquid waste based upon the results of the plant trials, it was necessary to further develop some of the individual thin-film evaporator processes at the WTC. Despite the earlier expectations the existing equipment was found to give poor encapsulation at the 20% loading rate.

To address this issue, three different rotors were designed by Company B staff for these experimental trials to determine the turbulence level needed to ensure an intimate contact between downflowing bitumen emulsion and saturated resin/waste solids particulates. Separate trials were done with each of the impellers to determine heat and mass transfer coefficients within the evaporator. Among the variables investigated during these trials for each impeller design were: process temperature (varied from 100 to 120 degrees centigrade), impeller speed (500 – 1500 rpm), bitumen flowrate (0.8 – 2.2 kg/min in 0.4 kg/min intervals), and waste feed flowrate (0.4 to 1.2 kg/min in 0.2 kg/min intervals).

For these three trials (or Trials 5-7) the ED zone claimed consisted of both thin film evaporators 1 and 2. There was no commercial activity during this time.

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One final trial was done in which all 5 stages were put into operation. The purpose of this trial was to assess if all of the stages could be operated as a whole and under continuous conditions, as opposed to the batch conditions that were used in the prior trials. Wasteform from the bitumen thin film evaporator was continuously withdrawn in 200 L product drums, and samples were taken for detailed chemical analysis and leach and integrity tests. Feed and effluent samples before and after all five stages were taken and analysed for the same chemical contaminants as were analysed in the earlier runs. That chemical analysis data was used to compare the efficiency of the process with the new technology implemented. It was found that the efficiency of the process had been sufficiently increased that the effluent quality met or surpassed the proposed new environmental standards.

For Trial 8 the ED zone claimed consisted of the entire 5-stage plant. A total of 36 hours was claimed, and the trial was replicated (Trial 9) to check reproducibility of the operations and analytical data. All operations data that was part of a standard operations run was also collected during Trial 9.

List of Contractors

CONTRACTOR NAME	ROLE & RESPONSIBILITY
Contractor A	Sampling and Chemical Analysis

1E. Supporting Information

- Description of work of Chemical Analysis contractor with project billings
- Progress Reports
- Document indicating senior managerial approval
- Experimental operating instructions and Test methods
- Detailed logs of start-up operations
- Process operators' Log books
- Updated mechanical drawings of waste treatment facility
- Bitumen wasteform leachability report and other physical data
- Company report on wasteform studies
- Experimental Trial Logs

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Table 1: Expenditures Claimed Using the Proxy Method

Total Directly Engaged Labor Cost	$385,000
Material consumed/transformed (recapture rules may apply to materials transformed) • Bitumen • IX resin • Chemicals • 10 MF Membrane elements removed for destructive autopsy (post-trial) as planned at the beginning of the project	$30,000 $ 5,000 $10,000 $ 5,000 $10,000
Contract Costs (analytical services - testing)	$400,000
Capital Expenditures – ASA SR&ED • Pilot-scale equipment • Bench scale equipment	$210,000 • $200,000 • $10,000
Total Claimed, excluding prescribed proxy amount (PPA)	$1,025,000
PPA = labour*.65	$ 250,250
Trials 1-9 Total Claimed, including PPA	$1,275,250

Analysis of Project:

The project involved the development of a 5-stage waste treatment process. As a result of this project, the company decided that it would be to their advantage to use the resin in a dispersed form rather than in a fixed bed because the cost of the resin is much less expensive in comparison with either replacing or regenerating the downstream RO membranes.

The work was planned and carried out in a systematic fashion by qualified technical personnel. The lab, bench, and pilot-scale work, as well as plant trials 1-8 meets the definition of SR&ED in Subsection 248(1) of the Income Tax Act. Plant trial 8 is eligible as a support activity to validate the overall process. None of the work carried out in plant trial 9 meets the definition of SR&ED.

The eligible work may be summarized as follows:

1. The planning done by the SR&ED team members
2. All literature search and preliminary evaluation work related to the project
3. Lab and bench studies
4. Pilot studies
5. Plant trials1-7
6. Plant trial 8 is a support activity for the SR&ED undertaken above.

The company claimed the following:

<u>January to March 2004</u>

The company claimed all the directly engaged labour during this period (steps 1,2 and 3 above). Steps 1 and 2 are support activities to the work undertaken in step 3. They also claimed the cost of the lab-scale equipment and all materials consumed (e.g. IX resin) during the experiments.

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April to June 2004:

The company claimed all the directly engaged labour during this period (step 4). They also claimed the cost of the pilot-scale equipment which was intended to be used ASA in SR&ED and all materials consumed (IX resin) for the experiments.

July to August 2004:

The company claimed all the directly engaged labour during this period (step 5). This would include the MF operators in addition to the SR&ED project team. The MF operators were supervised by technical staff, therefore they were considered to be directly engaged in SR&ED.

The claim for Trials 1 and 3 included all the directly engaged labour for operation of the MF. The ED zone was comprised of the MF system alone for Trials 1 and 3. The claim included the labour for two operators (2 operators per stage), a supervisor and a process engineer.

The claim for Trials 2 and 4 (replicate of Trial 2) included all the directly engaged labour for operation of the MF and two reverse osmosis stages. The ED zone was comprised of the MF system and the two reverse osmosis stages for Trials 2 and 4. The claim included the labour for six operators (2 operators per stage), a supervisor and a process engineer.

The claim for materials included the costs of 10 MF membrane elements sent for destructive autopsy (post-trial) as planned at the beginning of the project, IX resin and chemicals (e.g. for pH adjustment). There was no commercial activity during this time since all waste would need to be reprocessed through the entire plant (including evaporator facility) after the experimental trials were completed. Sampling and analysis of the feed, backwash, and effluent streams were carried out by the analytical laboratory. In addition, the process parameters on the MF system were taken continuously during each plant trial.

September – December 2004

The company claimed all the directly engaged labour during this period (step 6). This would include the operators for the thin-film evaporator plant in addition to the SR&ED project team.

The claim for Trials 5-7 included all the directly engaged labour for operation of the two thin-film evaporators. The ED zone was comprised of the two thin-film evaporators, but not the remainder of the plant. The claim included the labour for four operators (2 operators per stage), a supervisor and a process engineer. The claim for materials included the costs of bitumen. There was no commercial activity during this time. The same contract lab carried out sampling and analysis of the feed and concentrate streams from the volume reduction thin-film evaporator, an also measure metal leachate rates from the encapsulated materials produced during these three trials. In addition, the process parameters on the thin-film evaporators were taken continuously during each plant trial.

Trial 8 was done with all 5 stages in operation. Wasteform from the bitumen thin film evaporator was continuously withdrawn in 200 L product drums, and samples were taken for detailed chemical analysis and leach and integrity tests. Feed and effluent samples before and after all five stages were taken and analysed for a suite of target metals by the same contract lab. All operations data that was part of a standard operations run was also collected during Trial 8. For Trial 8 the ED zone claimed consisted of the entire 5-stage plant. Trial 9 was claimed as a replicate of Trial 8. All the directly engaged labour (10 operators, 1 supervisor, 2 process engineers and the SR&ED team) as well as the materials for these two trials were claimed.

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CRA Decision:

The Research and Technology Advisor (RTA) determined that all lab, bench, and pilot work, as well as, Plant trials 1-7 were found to meet the requirements of Subsection 248(1)(c) of the Income Tax Act (ITA). The identified technological objectives and the technological challenges had been resolved during Plant trials 1-8. Plant trial 8 was found to meet the requirements of Subsection 248(1)(d) of the ITA.

Plant trial 9 did not meet the requirements of Subsection 248(1) of the ITA. There was no evidence that any unresolved technological challenges remained after Trial 8.

The Financial Reviewer (FR) determined that the expenditures associated with the lab, bench, and pilot work could be verified. As well, the expenditures for Plant trials 1-8 were all found to be supported with suitable documents. Claims for materials and capital were supported by invoices. The contract agreement for the analytical services was confirmed.

Based on the RTA's report, plant trial 9 was disallowed in its entirety and the claim was amended accordingly.

Reference for Example 3.4

[1] Chemicals Guidance Document 3 Part 1 – Chemical Processes
http://www.cra-arc.gc.ca/taxcredit/sred/publications/chem3/chem3-README.html
http://www.cra-arc.gc.ca/taxcredit/sred/publications/chem3/chem3-LISEZ-MOI.html