Title

Field Validation of a Pilot-scale Black Liquor Membrane for Water Removal at Ahlstrom-Munksjö Paper Mill in Mosinee, WI

Authors

Earni, shankar
Pevzner, Amy
Sheaffer, Paul
et al.

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Field Validation of a Pilot-scale Black Liquor Membrane for Water Removal at Ahlstrom-Munksjö Paper Mill in Mosinee, WI

Operating and Performance Technical Report

Industrial Technology Validation

September 2024

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The Industrial Technology Validation (ITV) program is designed to address the need to identify, validate, and showcase the capabilities of new, emerging, and underutilized technologies in the industrial sector. The primary objective of ITV is to conduct robust evaluation and document performance data on these technologies to help expedite their commercialization and widespread deployment. By performing thorough validations and demonstrating the efficacy of these industrial technologies, the ITV program plays a crucial role in providing the necessary information for industry stakeholders to make informed decisions about their adoption. Each report conveys the performance results from a specific installation at a specific industrial site, following a specific methodology. Performance may vary for other installations of the same technology or if other methodologies are used to assess performance.

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For more information, contact:

John O'Neill
Technology Manager
Industrial Efficiency & Decarbonization Office
U.S. Department of Energy
john.oneill@ee.doe.gov

Authors

Prepared for the

U.S. Department of Energy's (DOE) Office of Energy Efficiency and Renewable Energy,
Industrial Efficiency and Decarbonization Office

Prepared by

Shankar Earni, Ph.D., Amy Pevzner, Paul Sheaffer, Prakash Rao, Ph.D.

Ernest Orlando Lawrence Berkeley National Laboratory
1 Cyclotron Road
Berkeley, CA 94720

Jeff Davis, P.E., Josh Weissert, P.E., Mike Christianson, CEM, CMVP, Katie Roessler

Energy 350
1033 SE Main St #1
Portland, OR 97214

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List of Acronyms

°C	Degrees Celsius
°F	Degrees Fahrenheit
ADT	Air-dried tons daily
Btu	British thermal unit
CIP	Clean-in-place
CTO	Crude tall oil
gal	Gallons
gpm	Gallons per minute
hp	Horsepower
hr	Hour
HPR	High-pressure positive displacement
ITV	Industrial Technology Validation
kW	Kilowatt
kWh	Kilowatt-hour
lbs	Pounds
M&V	Measurement and verification
MEE	Multi-effect evaporator
psi	Pounds per square inch
psig	Pounds per square inch gauge
RI	Refractive index
rpm	Rotations per minute
SBL	Strong black liquor
WBL	Weak black liquor

Executive Summary

Project Background

The Industrial Technology Validation (ITV) program aims to identify and demonstrate the performance of new, emerging, and underutilized technologies in the industrial sector to help inform decisions towards accelerating commercialization and deployment.

Pulp and paper is considered one of the most energy-intensive industries in the manufacturing sector. There are several methodologies for converting wood into pulp in the paper-making industry. The kraft process is a chemical method for producing wood pulp. The kraft process generates black liquor as a byproduct of pulp production. Traditionally, water is evaporated from the liquor by a set of multi-effect evaporators (MEEs), which concentrate weak black liquor (WBL) into strong black liquor (SBL) to support efficient combustion in a recovery boiler.

Concentrating black liquor is an energy-intensive step in recovering pulping chemicals and generating high-pressure steam from dissolved wood solids. About 7% of pulp and paper energy usage, or nearly 164 trillion Btu, is used to remove water from black liquor in U.S. kraft pulp mills per year. The U.S. DOE's IEDO is interested in the black liquor membrane technology evaluated in this report because it offers the potential for a more energy-efficient and less carbon-intensive kraft pulping process across 99 kraft pulp mills in 24 states (Agenda 2020 Technology Alliance 2016). This ITV project validates an innovative black liquor membrane technology for kraft pulp mills to understand its impact and benefits.

Via Separations is a technology vendor that developed a graphene oxide membrane system to remove water from WBL before entering the evaporator set. High-pressure positive displacement (HPR) pumps move the black liquor through membranes that separate water and create a more concentrated black liquor. The vendor claims their pre-commercial technology reduces evaporator steam consumption by dewatering WBL before entering the evaporators. They also claim their technology has non-energy benefits such as enhanced soap collection and improved pulp throughput. This evaluation focuses on validating the energy and carbon dioxide (CO_2e) emissions benefits associated with the membrane system.

Facility Description

The selected host site for testing Via Separations' membrane system is DOE Better Plants partner Ahlstrom-Munksjö's (A-M's) paper mill in Mosinee, WI. The plant was constructed in 1910 and purchased by A-M in 2018 and currently produces a variety of specialty food packaging and technical paper grades (e.g., food wrappers, microwave popcorn bags, masking tape backing, laminates, and composites for airplane wings). The site has a kraft pulp mill to process softwoods and hardwoods. The pulp mill generates up to 340 air-dry tons of pulp daily (ADT/day) and operates for 355 days annually.

Study Design and Objectives

This measurement and verification (M&V) report evaluates the performance of the Via Separations membrane system in a pilot-scale installation that was sized to process three gallons per minute (gpm) of WBL, a fraction of the paper mill's total WBL flow. The study objectives are to quantify the steam savings and electrical energy consumption associated with the membrane system along with the associated CO_2e impact. The host site did not share baseline system data due to its non-disclosure and confidentiality policies. Via Separations managed data acquisition from the pilot-scale installation over a three-week period. Given the constrained trial timeline, ensuing data errors and omissions limited the M&V analysis. Of the three-week trial data period, only two days of usable data were found. Several issues caused data collection challenges including sensor failure, clogging, and electrical outages.

Because of these challenges, the M&V results presented here have a high degree of uncertainty due to lack of complete data; hence, scaling this system to a full-size, annual model can result in a wide range of values. This report presents steam savings and pumping electricity consumption for the pilot scale system based on a very limited data set.

Project Results/Findings

The analysis found that additional pumps which feed black liquor through the membranes use 1.28 kW in lieu of the steam that can range from 5.4–6.9 pounds per hour (lb/hr) depending on the steam economies of the evaporator train. Table 1 summarizes the pump electricity requirements for the membrane in terms of power, site energy, and source energy, as well as the carbon impact.

Table 1. Membrane Pump Power, Energy, and Emissions

Pump Name	Pump Power (kW)a	Site Pump Energy (Btu/hr)b	Source Pump Energy (Btu/hr) b	Carbon Impact (lbs CO_2e per hr)c
T1 HPR Pump	0.28	965	2,703	0.23
T1 Recirc Pump	0.79	2,682	7,510	0.65
T2 HPR Pump	0.10	355	994	0.09
T2 Recirc Pump	0.11	360	1,007	0.09
Totals	**1.28**	**4,362**	**12,213**	**1.05**

[a] At mean water removal rate of 0.11 (gpm water/gpm WBL) and mean WBL flow of 0.55 gpm.
[b] Source-site ratio of 2.80 for electricity in the U.S. (Energy Star 2020). Emissions resulting from the membrane pumps use the U.S. electric grid generation mix in eGRID2020 (U.S. Environmental Protection Agency 2022).
[c] At the U.S. average output emission rate of 822.6 lbs/MWh (U.S. Environmental Protection Agency 2022).

Water removed across the membranes reduces the load on the existing evaporator and results in steam conservation. At a mean WBL flow of 0.55 gallons per minute (gpm), the water removal rate is 0.11 gpm per gpm of WBL and the energy intensity is 0.37 kilowatt-hours per gallon (kWh/gal) of permeate.

Steam savings of between 5.4–6.9 lb/hr was estimated based on a steam economy ranging from 4.2–5.4 lbs of water removed per pound of steam consumed in a typical multi-effect evaporator system (Frederick and DeMartini 2019). Table 2 shows the site steam conserved in lb/hr and Btu/hr for the pilot system, as well as the associated source fuel Btu/hr and CO_2e emissions reduction from the steam boilers. The source fuel conserved considers the weighted source-site ratio (Energy Star 2020) and weighted boiler efficiency (International Energy Agency 2010), which were developed using the national average pulp and paper boiler fuel mix (Bhander and Jozewicz 2017).

Table 2. Steam Conservation Summary

Steam Economy (lb H_2O/lb Steam)	Steam Conserved (lb/hr)[a]	Site Steam Conserved (Btu/hr)[a]	Source Fuel Conserved (Btu/hr)[b]	Decarbonization Impact (lbs CO_2e per hr)[c]
4.2	6.9	6,337	8,490	1.17
4.4	6.6	6,049	8,104	1.11
4.6	6.3	5,786	7,752	1.07
4.8	6.1	5,545	7,429	1.02
5.0	5.8	5,323	7,132	0.98
5.2	5.6	5,118	6,857	0.94
5.4	5.4	4,929	6,603	0.91

[a] Source-site ratio of 2.80 for electricity in the U.S. (Energy Star 2020). Emissions resulting from the membrane pumps use the U.S. electric grid generation mix in eGRID2020 (U.S. Environmental Protection Agency 2022).
[b] Considers the weighted source-site ratio from typical pulp and paper industrial boiler (1.01) and the weighted boiler efficiency (76%).
[c] At an assumed emission rate of 184 lbs/MMBtu.

For the limited data set at the scale of this pilot, the source pump energy consumption (12,213 Btu/hr) is higher than the source fuel conserved (6,603–8,490 Btu/hr) across the entire range of steam economies. The carbon emissions due to the added pumping loads are similar to the carbon savings at the steam boilers. With many efforts to clean up electric grid emissions across the country, the carbon impact and source energy results for this electrification technology can be expected to look more attractive in the future.

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SECTION

1

Introduction

1 Introduction

The kraft process, widely used in pulp mills, involves cooking wood chips with caustic soda and sodium sulfide to produce wood pulp. This chemical process allows for the recovery and reuse of pulping chemicals, making it a closed-loop system. An essential component of this process is the treatment of weak black liquor (WBL), which is a byproduct containing dissolved solids and chemicals. Traditionally, the concentration of WBL is achieved using multi-effect evaporators (MEEs), which utilize steam and recycled vapor to evaporate water and concentrate the liquor.

Via Separations has developed an innovative membrane technology designed to enhance the concentration of WBL before it enters the evaporator system. This new membrane system utilizes a graphene oxide membrane capable of withstanding the high temperatures and corrosive nature of black liquor. By integrating this membrane system into the pulp mill's process, Via Separations aims to reduce the reliance on evaporators, thereby decreasing steam consumption and improving overall energy efficiency.

The system operates by feeding WBL through high-pressure pumps into the membrane unit, where it is concentrated before entering the existing evaporator set. This approach reduces the amount of steam required for evaporation, translating into fuel savings for the mill's auxiliary boiler and potentially increasing electricity savings due to reduced demand on boiler support systems. Additionally, the membrane technology is expected to enhance production capacity and soap yield, providing further economic benefits.

This evaluation focuses on the performance of the Via Separations membrane system, including its impact on energy consumption. By comparing the membrane technology to traditional evaporation methods, this study aims to demonstrate the potential advantages of integrating advanced membrane systems into the kraft process, ultimately contributing to more sustainable and efficient pulp production.

SECTION 2

Description:
Facility, Technology, and Project

2 Description

2.1 The Kraft Process

The kraft process used at this mill is a chemical method for producing wood pulp using caustic soda and sodium sulfide. This process allows for the recovery and reuse of pulping chemicals, effectively making it a closed cycle. For an overview of the process, refer to Figure 1 (U.S. Environmental Protection Agency 1990). The red box outlines the evaporators, which are the area of interest for this study.

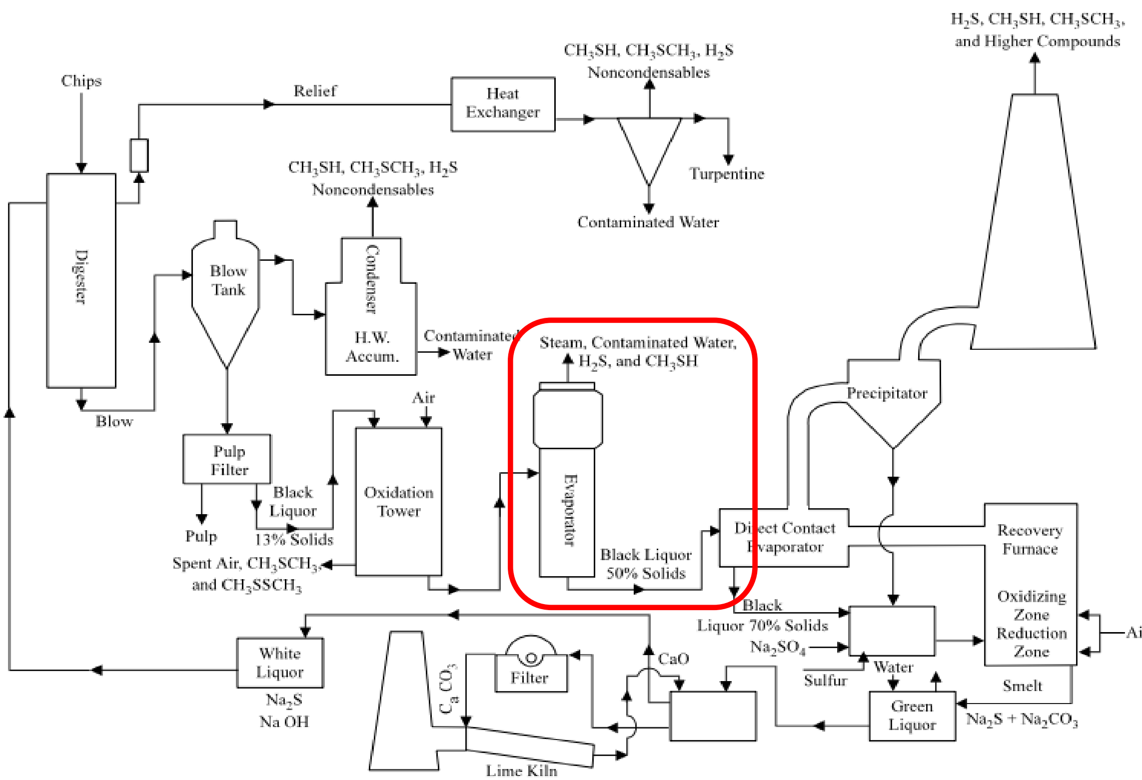

Figure 1. Kraft process overview with red box outlining the evaporators, which are the area of interest for this study

The kraft process involves cooking wood chips in a digester under high temperatures and pressure. The pulp is washed following cooking. The wash water and the spent cooking liquor combine to create a WBL with approximately 16% solids.

Traditionally, water is evaporated from the liquor by a set of evaporators in series (also referred to as multi-effect evaporators, or MEEs). Evaporators are large heat exchangers that use steam and recycled vapor to heat the liquor. In an MEE, the vapor outlet from one evaporator is used as the steam inlet for the next unit. Figure 2 shows an example schematic of an evaporator (Hajiha 2009).

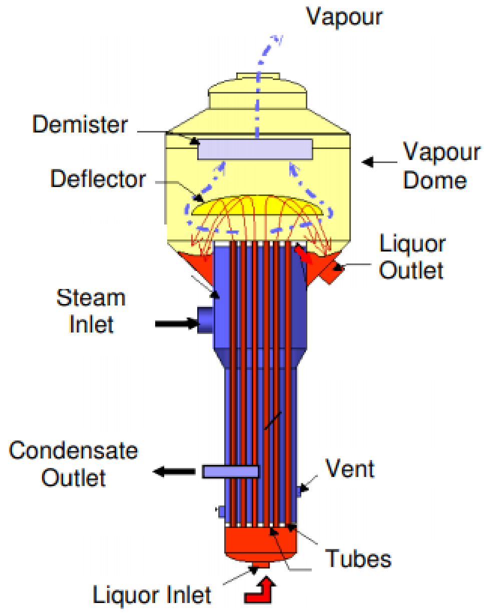

Figure 2. Illustration of a rising film evaporator

Black liquor must be concentrated to 65–85% solids to be considered SBL and support efficient combustion in the recovery boiler. The recovery boiler is essential to the chemical regeneration process and provides steam and electricity to the mill as an added benefit. The inorganic chemicals from the black liquor create a molten smelt at the bottom of the furnace. This smelt is dissolved in water to create green liquor. Green liquor is causticized using quicklime to create white liquor, which restarts the cycle. The causticizing process generates lime mud, also burned in a kiln on site.

2.2 Technology Description

Via Separations has developed a membrane system to dewater WBL before entering the evaporator set. A membrane system is an alternative to evaporation. The Via Separations design uses a graphene oxide membrane to withstand the high temperatures and corrosive properties of black liquor. High-pressure positive-displacement pumps feed the black liquor through the membrane.

A pulp mill would install the new membrane system between the WBL storage tank and the evaporators, as shown in Figure 3 (Via Separations 2020). Weak black liquor enters the system and exits as concentrated black liquor and permeate (mostly water). The existing MEE system would be “re-balanced” to continue providing 65–85% solids SBL for combustion in the recovery boiler with a higher-concentration WBL input. “Re-balancing” refers to adjusting the steam input to the MEEs since the membrane unit would offset some steam use.

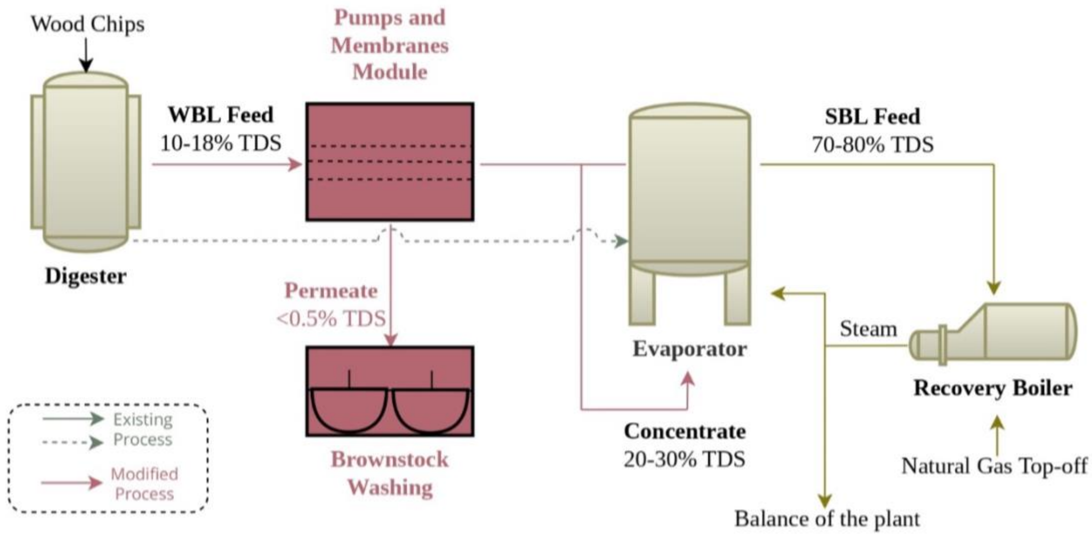

Figure 3. Via Separations' black liquor concentration system

The membrane system reduces MEE steam consumption, translating to fuel savings at the mill's auxiliary boiler.[1] An auxiliary boiler varies its firing rate to maintain steam header pressure while all other operating boilers run at the maximum firing rate. Boiler support systems such as fans and feedwater pumps may contribute additional energy savings.

Aside from energy savings, the vendor claims its technology will benefit production including additional pulp production capacity and soap yield. Soap is a mixture of sodium salts of rosin acids and fatty acids that separates from the black liquor with increasing concentration.[2] This soap, another byproduct in the kraft process, is skimmed from the black liquor as the black liquor settles. While black liquor itself is used as an energy source, the soap goes on to be used to create crude tall oil (CTO) that can be sold. This CTO is used in the production of coatings, paints, varnishes, lubricants, soaps, and other products. Limitations in steam or evaporator capacity require the mill to curtail pulp production, as weak black liquor generation increases with pulp production. Curtailment occurs when the evaporator capacity is insufficient to process an increased liquor flow to at least 65% solids. The recovery boiler will not fire efficiently if the moisture content of the liquor fuel is too high. As these constraints are highly site-dependent, this study does not quantify production throughput or the additional benefits associated with soap generation.

[1] Mills depend on additional boilers beyond the recovery boiler to satisfy steam demand; as the recovery boiler firing rate is process-dependent, other boiler fuels will be conserved.

[2] Soap solubility reduces with increasing concentration of black liquor. Minimum solubility occurs between 25–30% solids.

SECTION

3

Technology Demonstration Intent

3 Technology Demonstration Intent

3.1 Study Design and Objectives

The vendor completed a trial run of the pilot membrane unit from July 20 to September 8, 2021. The pilot unit diverted a small fraction of the overall flow of the weak black liquor flowing into a typical evaporator set.

Each pilot membrane unit includes two sets of centrifugal pumps, referred to as “stuffing pumps” and “recirculation pumps.” The stuffing pumps are 0.33 hp each. At full scale, the stuffing pumps would not be necessary, as the membrane unit would be installed in line with a weak black liquor piping header pressurized by existing pumps. The recirculation pumps are 5 hp each.

The stuffing pumps feed VFD-controlled, high-pressure, positive displacement (HPR) pumps of unspecified horsepower. These HPR pumps then feed two membrane trains, each with six membranes. Recirculation pumps recirculate liquor across the membranes whenever the train is on. A clean-in-place (CIP) system rinses the membranes. The CIP system utilizes heating elements to warm the water on the pilot unit but will not require heating elements at full scale. The vendor anticipates that each of the membranes will undergo an hour-long cleaning cycle every 24–48 hours. The CIP system was not analyzed in this M&V study because pressure data was not available for the CIP system.

The M&V analysis quantifies the MEE steam energy conserved and the electrical load required to support the pilot-scale membrane unit. The analysis is specific to the pilot-scale application at this test site and based on the dataset collected from two days of operation.

3.2 Energy and Non-Energy Savings Description

The membrane concentration system reduces evaporator steam, related boiler fuel use, and electric consumption at the boiler fans and feedwater pumps. Evaporator steam is conserved by pre-concentrating the black liquor before entering the evaporator set. Reduced steam use will translate to fuel savings at the auxiliary boiler and some electric energy savings (e.g., at the combustion fan and feedwater pump). The addition of electric pumps that feed WBL through the membranes is expected to increase electricity consumption at the site. This electric pumping energy consumption was evaluated in this report, whereas the auxiliary boiler electric energy savings were not evaluated, as they are expected to be small in comparison.

The vendor states the following additional non-energy benefits, which were not evaluated as part of this study due to a lack of relevant baseline data:

- The increase in the feed solids to the evaporator train also likely increases the concentration of the soap in the tank, and thus the efficiency of skimming and collecting soap.

- The combustibility of the liquor improves when soap is removed more effectively from the liquor (Uloth et al. 2009). An increased volume of liquor can be burned in the recovery boiler without soap, which may lead to increased pulp production if a mill's production bottleneck is at the recovery boiler.

3.3 Affected Equipment Inventory

The host site currently operates a five-effect rising film MEE with a concentrator supplied with 50 psi and 35 psi steam. WBL enters the MEE system at 16% solids, mixes with SBL to enter the pre-concentration tank at around 22% solids, and leaves the five-effect evaporator set at 52%. The liquor then enters a concentrator, raising solids to at least 65% as recovery boiler fuel. Figure 4 shows A-M's current evaporator configuration and process flow. The orange flow line demonstrates SBL recirculation to increase the incoming WBL solids to allow for soap recovery. The Via Separations membrane could reduce or eliminate this need.

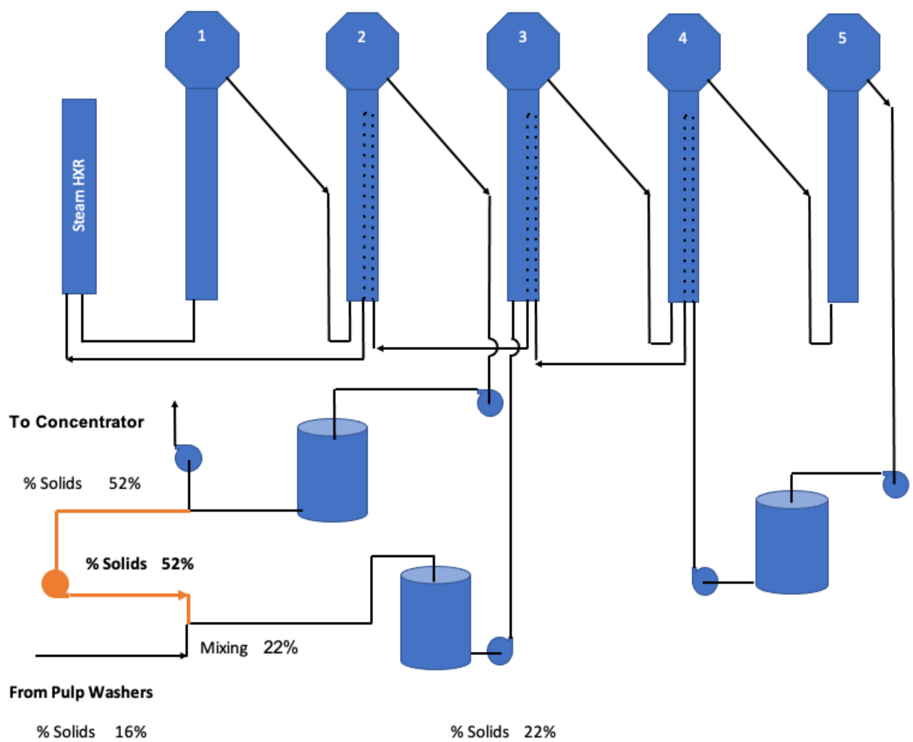

Figure 4. Simplified evaporator process flow diagram

3.4 Expectations of a Full-scale Deployment

Via Separations' design for black liquor concentration enables a modular increase in capacity and intensification of processes, enhancing the overall energy efficiency of the pulp production process. ITV did not study potential full-scale energy and environmental impacts due to the limited data set and low confidence to extrapolate.

SECTION 4

Evaluation Scope and Boundary

4 Evaluation Scope and Boundary

4.1 IPMVP Option

The evaluation methodology followed the International Performance Measurement and Verification Protocol (IPMVP), developed by the Efficiency Valuation Organization (EVO). The objective of the IPMVP is to develop a consensus approach to measuring and verifying efficiency investments and facilitate a scaled-up, global engagement on energy efficiency.

The IPMVP outlines four options depending on the purpose, scope, and objective of the project (EVO 2022). These four options are categorized into two general types: retrofit isolation and whole facility. Retrofit-isolation methods consider only the affected equipment or system independent of the rest of the facility. Whole-facility methods consider the total building or facility energy use and de-emphasize specific equipment performance. The primary difference in these approaches is where the measurement boundary is drawn. Options A and B are retrofit-isolation methods, Option C is a whole-facility method, and Option D can be used as either, but is usually applied as a whole-facility method.

This M&V effort followed a modified Option B, retrofit isolation with all parameter measurements. It was not possible to obtain baseline measurements to characterize current operations, so the baseline was developed from a model using the available data obtained from the site with information supplemented from the paper and pulp industry. The only complete measurements from the trial run are from two of the three days between August 25 and August 28, 2021. A period of nearly a full day within that timeframe was excluded because both the plant and the pilot system went offline.

4.2 Measurement Boundary Descriptions

The boundary of the affected system includes the evaporators and the membrane concentration unit, as shown in Figure 5 below. The boilers, pulp washers, and digesters are all excluded from the system boundary.

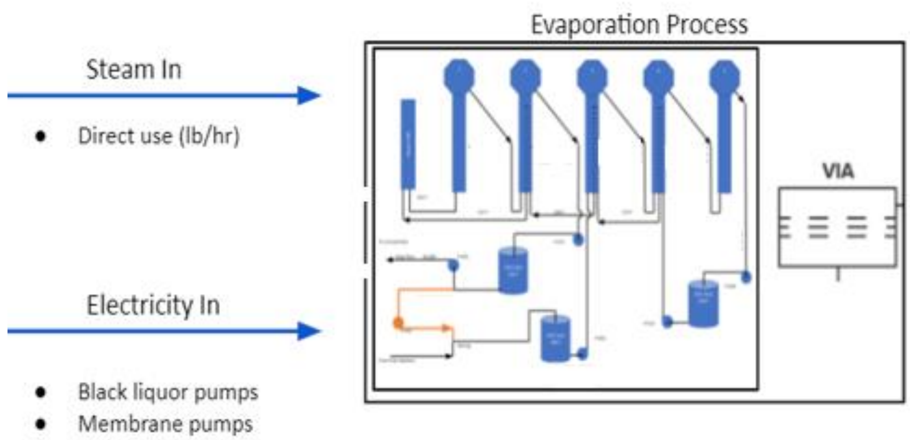

Figure 5. Measurement boundary for the evaluation

Baseline data on the MEEs could not be collected at the project site. For that reason, typical steam economies ranging from 4.2–5.4 pounds of water per pound of steam were considered in this analysis to assess the impact of the membrane systems when used in the conjunction with existing MEEs.

4.3 Interactive Effects Beyond the Measurement Boundary

The study measurement boundary only encompassed the evaporator and the membrane concentration units. Equipment outside the boundary of this equipment was not evaluated since baseline data could not be collected.

SECTION

5

Data Collection and Adjustments

5 Data Collection and Adjustments

5.1 Baseline Data

Baseline data was unavailable due to the host site's confidentiality and non-disclosure policies. For that reason, baseline steam and electricity consumption could not be determined and have not been included in the analysis. Rather than develop a baseline energy consumption from site data, the evaluation team used a range of MEE steam economies (from 4.2–5.4 pounds of water per pound of steam) to estimate steam savings.

Data collected for variables that may influence MEE steam consumption are summarized in the list below for reference:

- Mean WBL percent solids = 14.8%. (Varied between 14–16%.)
- Mean Train 1 (T1) Concentrate percent solids = 20.5%. (Varied between 17.7–21.5%.)
- WBL temperature = 163°F. (Varied between 155–170°F.)
- Mean Infeed Pressure = 9.9 psig. (Varied between 3–25 psig.)

5.2 Post-Retrofit Data

Membrane Pilot Units

The pilot system's electrical load due to additional pumping energy is quantified. The measurement boundary was constrained to the pilot-scale membrane system. Collected data that was used in the analysis includes:

- System shutoff valve position
- Train 1 (T1) and Train 2 (T2) HPR and recirc pump inlet and outlet pressures (psig)
- T1 and T2 infeed, concentrate, and permeate temperatures (°C)
- T1 and T2 infeed, concentrate, and permeate concentration (% Brix)
- T1 and T2 infeed, concentrate, and permeate flow rates (gpm).

The following section describes the data exclusions and filters applied before performing the outlier and M&V analyses.

5.3 Data Exclusions Based on Operator's Shift Log

The complete pilot-scale data set was from August 9 to August 31, 2021. Most trial period data was excluded from the M&V analysis due to one of several issues that occurred during the trial period, as described in the operator's shift log and summarized in Table 3.

Table 3. Summary of Exclusions from Trial Data Set

Time Range of Exclusion	Reason for Data Exclusion
8/9 12 AM – 8/25 12:25 PM	T2 permeate flow meter not working
8/21 3:02 PM – 8/25 1:04 PM	T1 permeate flow meter not working
8/25 7:50 PM – 8/26 8:18 AM	2–5 membrane failure ruined permeate readings
8/26 1:57 PM – 2:28 PM	System offline
8/27 1:19 AM – 9:36 AM	System offline
8/28 12:26 PM – 8/31 11:59 PM	T1 permeate flow meter not working

As shown above, data was excluded when one of the flow meters was not working, when a membrane failure resulted in invalid permeate readings, or when the entire system was off, as indicated in the operator's shift log. Those time periods all show erroneous or atypical data.

The only complete data set occurs within the time period of August 25 at 1:04 PM to August 28 at 12:15 PM, with nearly a day of that time excluded when the system went offline. The remaining time (two days) was used in the analysis, including T2 off times which is considered normal operation. T2 regularly cycled on and off, running about 43% of the time.

Before performing an outlier analysis, the raw data was aggregated from five-second intervals to one-minute averages. Figure 6 highlights when the T1 permeate flow meter was not working, showing a near-zero flow from August 21 to 25, followed by max flow (3.3 gpm) from August 28 to 31.

Figure 7 shows when the T2 permeate flow meter was not working – from August 9 to 25, most of the data period. It also shows when the system was shut down for 30 minutes on August 26 (foam overflow problem) and August 27 (low-pressure alarm).

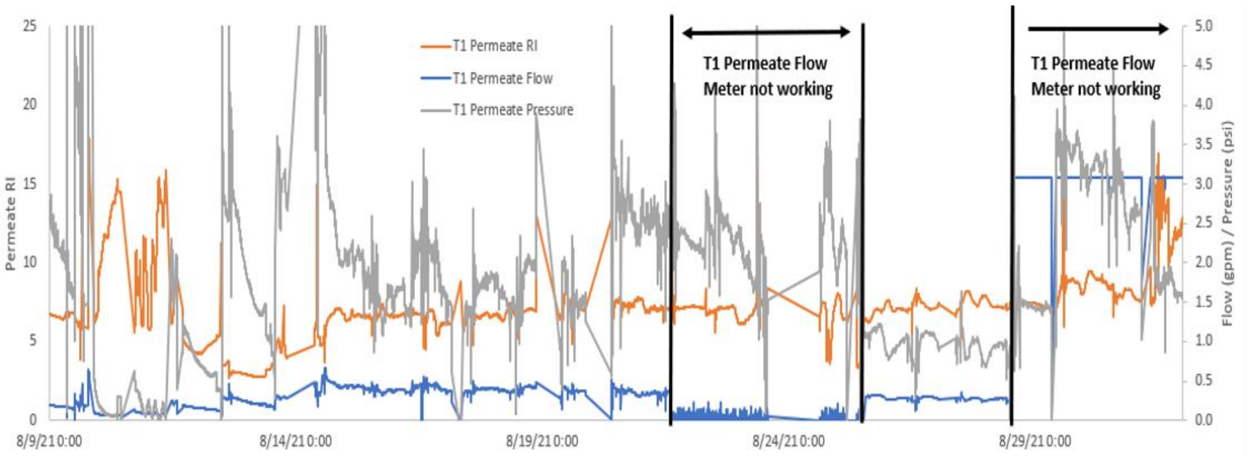

Figure 6. T1 permeate RI, flow, and pressure

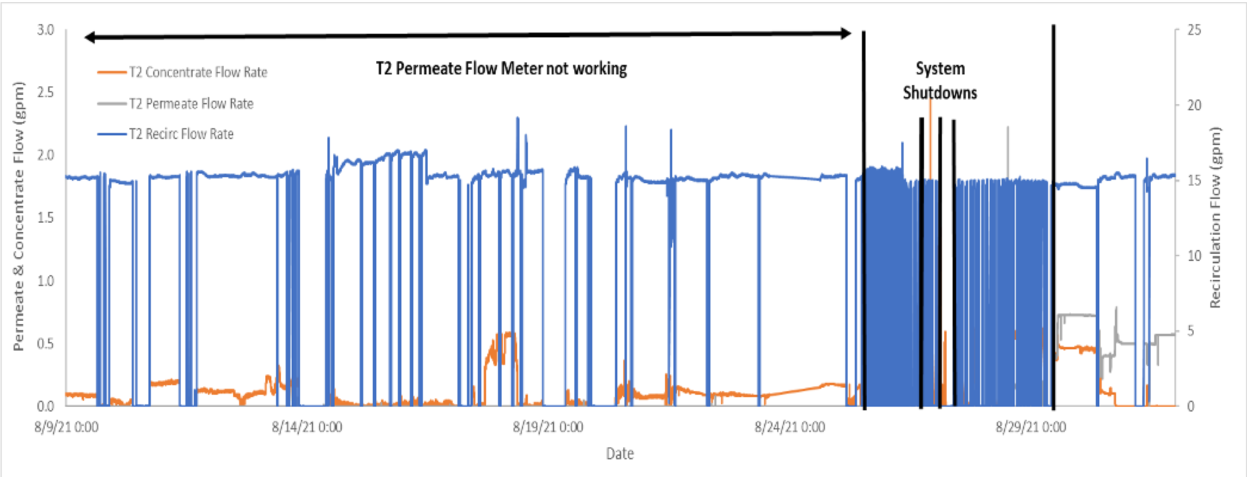

Figure 7. T2 recirculation, concentrate, and permeate flow

5.4 Outlier Analysis

Before savings could be finalized, an outlier analysis was performed to remove atypical values for trended variables that are used in the analysis. The outlier analysis considers the two days of complete data after removing the data exclusions described in Section 4.3.

The outlier analysis was performed by finding all values beyond three standard deviations for variables pertinent to the savings analysis. These potential outliers were filtered, evaluated, and eliminated where necessary on a variable-by-variable basis. Important variables with count of outlier observations include the T1 permeate flow rate (18), T1 permeate pressure (27), and the T2 concentrate flow rate (1). All 46 outliers (1.6% of the data set) were removed from the M&V analysis because they either coincided with abnormal operating conditions, such as low permeate pressure, or showed high/low values near when the system started or shut down.

SECTION 6

Calculation Methodology

6 Calculation Methodology

The analysis calculates the impact on electric and steam energy use associated with a pilot-scale membrane. More specifically, the energy calculations quantify the reduction in steam use by the MEEs and the increase in membrane pumping electric use. These calculations do not quantify any impacts on the existing MEE electrical loads, which are small by comparison.

6.1 Initial Calculations

The following list is a summary of calculations performed to prepare the analysis for steam savings and electric penalty calculations:

- All temperatures in Celsius were converted to Fahrenheit.
- The T1 infeed flow rate was calculated by adding T1 concentrate and T1 permeate flows.
- T1 and T2 infeed, concentrate, and permeate black liquor-specific gravities were calculated using the density of black liquor based on the temperature and RI, or % Brix (Frederick and DeMartini 2019).
- T1 and T2 HPR pressure differentials were calculated by subtracting pump outlet pressure from pump inlet pressure.
- T2 on/off status was determined. The system is considered “off” whenever the recirculation flow is zero.
- T2 water removal (gpm) was calculated by dividing the T2 permeate flow by the T2 permeate specific gravity (SG).

6.2 Membrane Pumping Power

Since pumping power was not measured over the trial period, it is calculated according to the equation below for the T1 and T2 high-pressure pumps (DOE 2006).

$$HPR\ Pumps\ [BHP] = \frac{Flow\ [gpm] *\ Head\ [psi] * Specific\ Gravity}{1{,}714\ *\ Pump\ Efficiency\ [\%]}$$

An 85% pumping efficiency was used for the positive displacement (HPR) pumps, based on the pilot system’s Hydra-cell pump specifications.

T1 and T2 recirculation pump power was calculated according to the following equation, where a 70% pumping efficiency was assumed for centrifugal pumps based on the optimal efficiency of the pilot system’s pump manufacturer’s specifications.

$$Recirculation\ Pumps\ [BHP] = \frac{Flow\ [gpm] * Total\ Head\ [ft] * Specific\ Gravity}{3{,}960 * Pump\ Efficiency\ [\%]}$$

Note that the recirculation pumps operated at a much lower efficiency point (~35%) during the trial period, as they were being operated at a much lower load factor. The optimal efficiency of 70% was used in this analysis to avoid skewing the results, since pumps are typically sized properly in new projects. (See Appendix for HPR and recirculation pump specifications.)

6.3 Summary of Important Assumptions

Important assumptions that impact savings figures are summarized here:

- T1 permeate feeds into T2.
- The study analyzes the impact of energy consumption with steam economies ranging from 4.2 to 5.4 for a multi-effect evaporator set (Frederick and DeMartini 2019).
- Variations in operating conditions are assumed to be within normal conditions except for data exclusions influenced by the operator's shift log and outliers, as described in Sections 4.3 and 4.4, respectively.
- The source energy and resulting CO_2e emissions associated with steam boiler savings assume a typical fuel mix for pulp and paper steam boilers: 45% wood, 28% coal, and 27% natural gas (Bhander and Jozewicz 2017).
- The CO_2e emissions associated with membrane pumping energy are based on an average U.S. electric grid output emissions rate of 822.6 pounds per megawatt-hour (U.S. Environmental Protection Agency 2022).
- Positive displacement (HPR) pump efficiency is 85%. (See Appendix for HPR specifications.)
- Centrifugal (recirculation) pump efficiency is 70%. (See Appendix for pump curve.)
- Motor efficiency is 89.5% for the 5-hp, 3,600-rpm enclosed recirculation pumps. Motor efficiency is 88.5% for the 1,800-rpm enclosed HPR pumps (U.S. Department of Energy 2014). Note that based on the operating pressure and flow of the HPR pumps, the efficiency is assumed to be the same as a 5 hp motor since the size of these motors is not included in the specifications.

SECTION

7

M&V Results

7 M&V Results

7.1 Membrane Pumping Energy

The analysis quantifies steam savings and electric pumping energy using the final data set with exclusions and outliers removed. Figure 8 shows flow data for the M&V analysis time period (August 25–28).

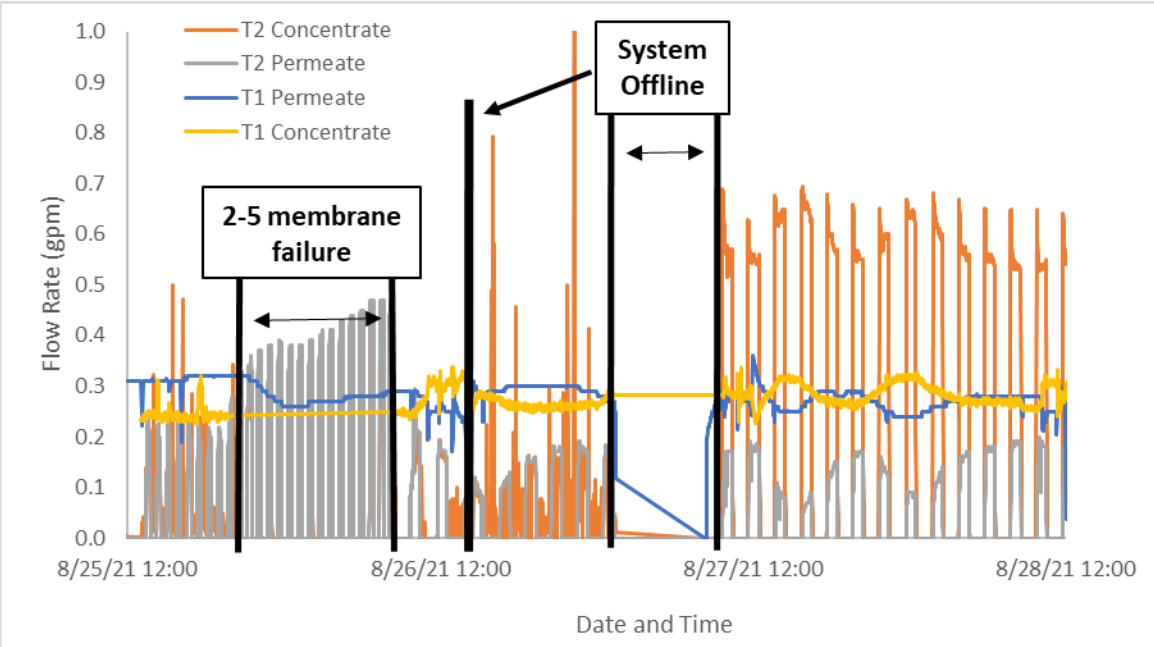

Figure 8. T1 and T2 flow rates

Note the T2 concentrate flow meter (orange line) was not working August 26–27 when the system was online. However, the T2 permeate flow meter *was* working and is the important variable used in savings calculations, so data from this period was still helpful and included in the analysis. When all flow meters are working (from August 27 at 9:36 AM to August 28 at 12:15 PM), the T1 permeate flow is roughly equal to the sum of T2 concentrate flow and T2 permeate flow.

T1 and T2 recirculation and HPR motor power are calculated from BHP values using the following equation:

$$HPR\ and\ Recirculation\ Pump\ Powers\ [kW] = \frac{0.746 * BHP}{Motor\ Efficiency}$$

Where a motor efficiency of 89.5% was used for the recirculation pumps and 88.5% was used for the HPR pumps (U.S. Department of Energy 2014). Figure 9 shows pumping power during the analysis period.

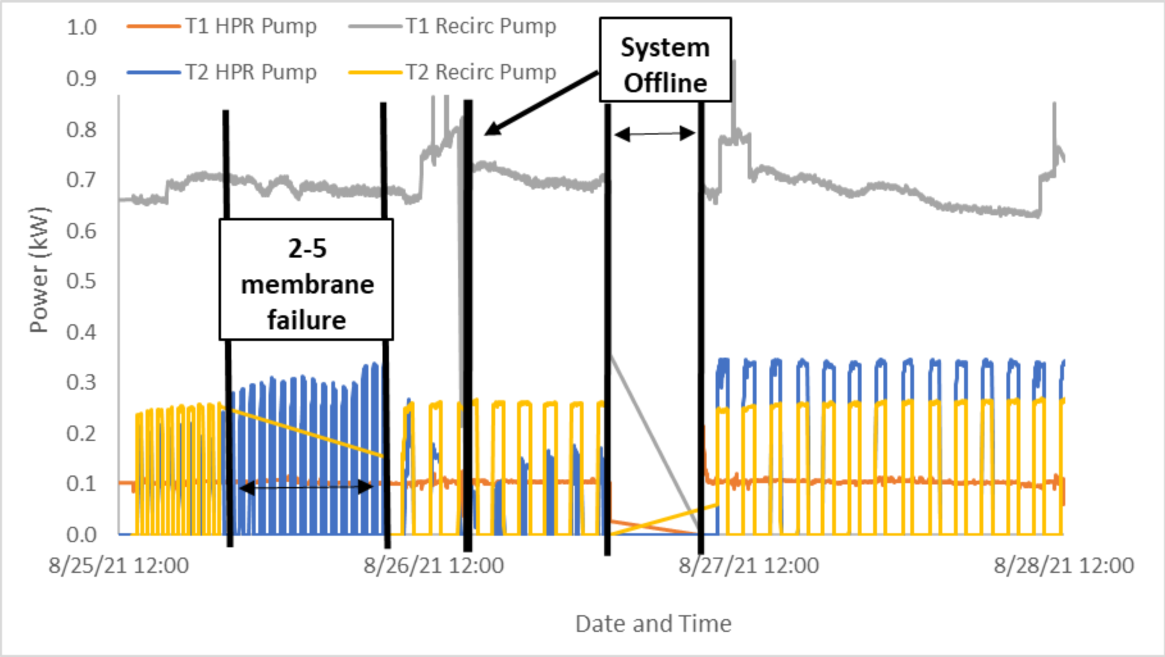

Figure 9. T1 and T2 recirculation and high-pressure pumps

T1 pumps run throughout the analysis period, whereas T2 pumps cycle on/off with Train 2. All pumps were off when the system went offline. Note that the T1 recirculation pump shows significantly higher power than the T2 recirculation pump due primarily to a much higher average pressure (176 psi vs 50 psi). Table 4 shows the average pump power and Btu/hr during the analysis period.

Table 4. T1 and T2 Recirculation and High-Pressure Pump Power

Pump Name	Pump Power (kW)[a]	Site Pump Energy (Btu/hr)[a]	Source Pump Energy (Btu/hr)[b]	Carbon Impact (lbs CO_2e per hr)[c]
T1 HPR Pump	0.28	965	2,703	0.23
T1 Recirc Pump	0.79	2,682	7,510	0.65
T2 HPR Pump	0.10	355	994	0.09
T2 Recirc Pump	0.11	360	1,007	0.09
Totals	**1.28**	**4,362**	**12,213**	**1.05**

[a] At a mean water removal rate of 0.11 (gpm water/gpm WBL) and mean WBL flow of 0.55 gpm.
[b] Source-site ratio of 2.80 for electricity in the U.S.
[c] At the U.S. average CO_2e equivalent output emission rate of 822.6 lbs/MWh.

At a mean WBL flow of 0.55 gpm, the water removal rate is 0.11 gpm per gpm of WBL and the energy intensity is 0.37 kWh/gal of permeate.

The carbon emissions for pump power (kW) considers the eGRID2020 emissions factor for the U.S., which is 822.6 lbs/MWh (U.S. Environmental Protection Agency 2022). The carbon impact of the pumps was calculated using the following equation:

$$Pump\ Carbon\ Impact\ \left(\frac{lb\ CO_2}{hour}\right) = Pump\ Power\ (kW) * 822.6\frac{lb}{MWh} * \frac{1\ MWh}{1{,}000\ kWh}$$

7.2 Train 1 and Train 2 Concentration Analysis

Train 1 and Train 2 membranes effectively concentrate their respective infeed, which is one of the main objectives of the technology. Train 2 processes the concentrate leaving Train 1 but cycles on/off. Figure 10 shows the concentrate and permeate refractive index (RI) in units of % solids in T1 and T2 throughout the three-day analysis period.

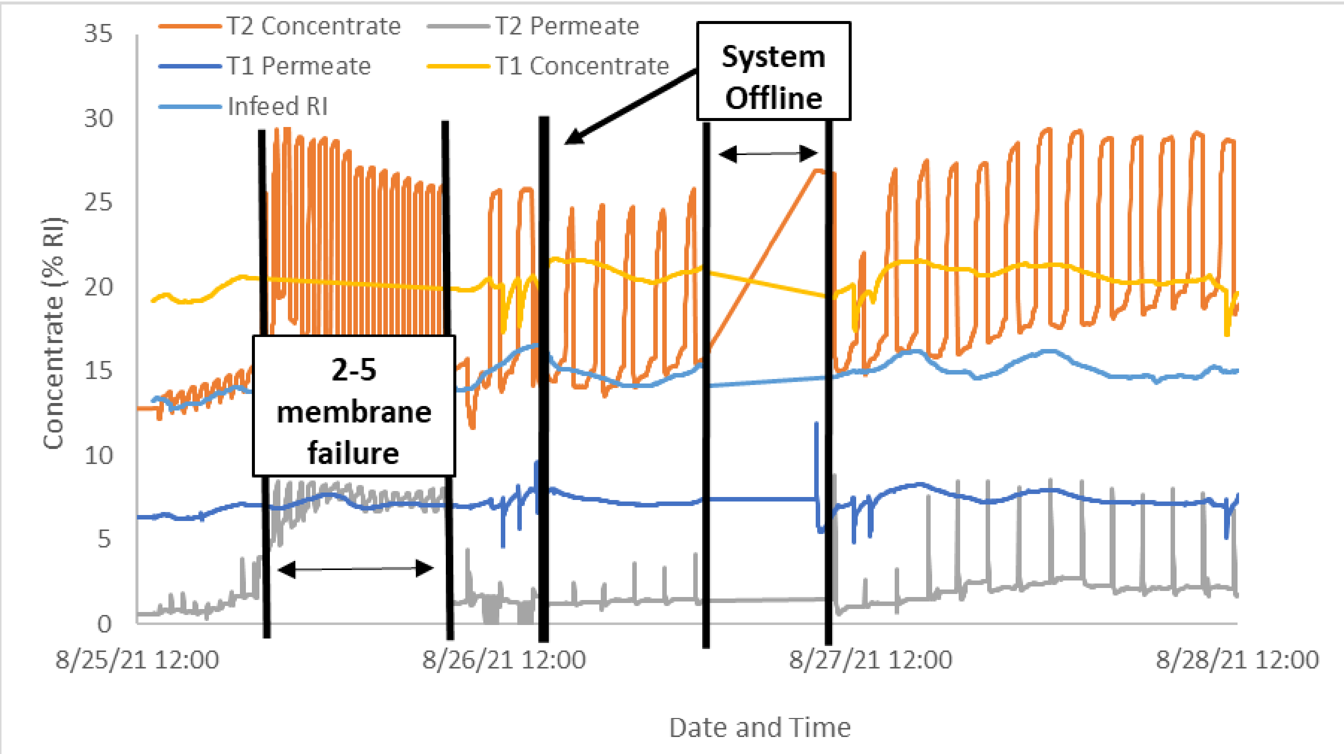

Figure 10. T1 and T2 concentrations

Observe that the WBL infeed RI (~15%) is between the T1 concentrate RI (~20%) and T1 permeate RI (~7%). The difference between T1 concentrate and T1 permeate RIs demonstrates that the Train 1 membranes concentrate the WBL. Also, notice that the T1 permeate (~7%), aka T2 infeed, is between the T2 concentrate RI (~16%) and T2 permeate RI (~2%). This shows further purification of T2 permeate, which is mostly water.

7.3 Steam Savings Analysis

The following equation was used to calculate MEE steam savings (in lb/hr):

$$Steam\ Conserved\ (pph) =$$

$$\frac{T2\ water\ removal\ \left(\frac{gpm\ water}{gpm\ WBL}\right) * WBL\ Flow\ \left(\frac{gal}{min}\right) * 8.314\left(\frac{lbs\ water}{gal\ water}\right) * 60\left(\frac{min}{hour}\right)}{Steam\ Economy\ \left(\frac{lb\ water}{lb\ steam}\right)}$$

Where averages during the final analysis data period were used for T2 water removal and WBL flow. The T2 water removal is T2 permeate flow divided by the fluid's specific gravity, and WBL flow is the T1 infeed flow.

Based on the observed water removal rates (gpm water removed/gpm ingoing WBL) and typical industry steam economies ranging from 4.2–5.4 lb water/lb steam, steam savings and associated impacts are summarized in Table 5.

Table 5. Steam Savings and Carbon Impact

Steam Economy (lb H_2O/lb Steam)	Steam Conserved (lb/hr)[a]	Site Steam Conserved (Btu/hr)[a]	Source Fuel Conserved (Btu/hr)[b]	Decarbonization Impact (lbs CO_2e per hour)[c]
4.2	6.9	6,337	8,490	1.17
4.4	6.6	6,049	8,104	1.11
4.6	6.3	5,786	7,752	1.07
4.8	6.1	5,545	7,429	1.02
5.0	5.8	5,323	7,132	0.98
5.2	5.6	5,118	6,857	0.94
5.4	5.4	4,929	6,603	0.91

[a] At a mean WBL flow of 0.55 gpm and mean water removal rate of 0.11 (gpm water / gpm WBL).
[b] Considers the weighted source-site ratio from a typical pulp-and-paper industrial boiler (1.01) and the weighted boiler efficiency (76%).
[c] At an assumed emission rate of 184 lbs/MMBtu.

Note that site steam conserved was converted from lb/hr to Btu/hr using 912 Btu/lb and the evaporated saturated steam enthalpy at 50 psig, a typical pressure for MEEs.

The source fuel conserved and decarbonization impact account for a reduced load on the steam boilers. Solving for these units considers the typical U.S. pulp and paper industry boiler fuel mix, consisting of 44% wood, 28% coal, 27% natural gas, and 1% residual oil (Bhander and Jozewicz 2017). The weighted CO_2e emissions for a boiler was calculated at 184 lbs/MMBtu based on EPA emission factors for the different fuel types as shown in Table 6 (U.S. Environmental Protection Agency 2014).

Table 6. Blended CO_2e Emissions for a U.S. Pulp and Paper Typical Steam Boiler

Boiler Fuel Mix	Fuel Type % of Total	Fuel CO_2e Emissions (lbs/ mmBtu)[a]	Weighted Emissions (lbs/ mmBtu)	Industrial Boiler Combustion Efficiency (%)[b]	Weighted Boiler Efficiency	Source-Site Ratio [c]	Weighted Source-Site Ratio[c]
Wood	44%	206	91	70%	0.31	1	0.44
Coal	28%	216	60	85%	0.24	1	0.28
Natural Gas	27%	117	32	75%	0.20	1.05	0.28
Residual Oil	1%	160	1.6	80%	0.01	1	0.01
Weighted Values Used in Analysis			**184**		**76%**		**1.01**

[a] U.S. EPA Emission Factors for Greenhouse Gas Inventories.
[b] ETSAP Industrial Combustion Boilers Actual Efficiency at full load.
[c] Energy Star Source Energy Ratios in the U.S.

The weighted emissions value (184 lbs/MMBtu) was used to find the decarbonization impact of conserved steam (Btu/hr). The decarbonization impact in pounds of CO_2e per hour was calculated using the following equation:

$$Decarbonization\ Impact\ \left(\frac{lb\ CO_2}{hr}\right) = Site\ Steam\ Conserved\left(\frac{Btu}{hr}\right) Weighted\ Emissions\left(\frac{lb\ CO_2}{MMBtu}\right) * \left(\frac{1\ MMBtu}{1{,}000{,}000\ Btu}\right)$$

The weighted boiler efficiency was calculated at 76%, based on industrial boiler combustion efficiencies for each of the different fuel types shown in Table 6 (International Energy Agency ETSAP 2010). The weighted source-site ratio was calculated at 1.01 considering that wood, coal, and residual oil all have a ratio of 1, while natural gas has a 1.05 source-site ratio accounting for transmission losses (Energy Star 2020). These values were used to find the source fuel conserved (Btu/hr) at each steam economy using the following equation:

$$Source\ Fuel\ Conserved\ \left(\frac{Btu}{hr}\right) = Site\ Steam\ Conserved\ \left(\frac{Btu}{hr}\right) * \frac{Weighted\ Source - Site\ Ratio}{Weighted\ Boiler\ Efficiency}$$

SECTION

8

Summary and Conclusions

8 Summary and Conclusions

Based on the evaluation results from a small-scale demonstration system, the analysis shows that the membrane system dewaters weak black liquor to create a more concentrated black liquor stream and support the existing thermal-based evaporator system. As a result, the membrane system saves steam energy by preconcentrating the black liquor with a membrane compared to a purely thermally operated evaporator system. This steam savings analysis is based on a range of typical steam economies associated with the MEEs due to the lack of site-specific baseline data for the evaporator and boiler systems.

At a mean WBL flow of 0.55 gpm for the small-scale system, the site steam savings are estimated to be between 5.4–6.9 pounds per hour (or 4,929–6,337 Btu/hr at site and 6,603–8,409 Btu/hr at source) based on a steam economy between 4.2–5.4 pounds of water removed per pound of steam. The steam savings are equivalent to a reduction of 0.91–1.17 pounds of CO_2e per hour, based on an assumed energy mix for the generated steam. The membrane system requires additional pumping energy of 1.28 kW (4,362 Btu/hr at site and 12,213 Btu/hr at source) to preconcentrate the black liquor, resulting in an increase of 1.05 lbs of CO_2e per hour based on the national electric grid composition. The carbon reductions associated with reduced steam are similar to the increase in carbon emissions due to the added pumping loads for the pilot scale membrane system.

The source pump energy consumption (12,213 Btu/hr) is higher than the source fuel conserved (6,603–8,490 Btu/hr) across the entire range of steam economies. With many efforts to clean up electric grid emissions across the country, the carbon impact and source energy results for this electrification technology will likely look better in the future.

Since starting this pilot-scale study, Via Separations conducted subsequent evaluations at other sites and gathered additional data. The results from those evaluations and this A-M analysis were included in a recently published TAPPI paper showing the performance and reliability of the membrane system alongside existing MEEs (Rae et al. 2023). These analyses show an improvement in energy intensity and reliability of the system and are summarized in the Addendum.

8.1 Considerations for Future Evaluations

To improve the accuracy and confidence levels associated with this evaluation, six months of at least hourly trend data would be needed for both the MEE and membrane systems. Analyses of this data would achieve higher accuracy results that can be analyzed further to assess the energy and GHG impact associated with larger black liquor systems, improving confidence. The following lists the MEE and membrane system data points recommended for complete baseline and trial system analyses:

MEE system

- Weak black liquor (WBL) solids (%)

- Strong black liquor (SBL) solids (%)
- WBL flow rate (gpm)
- SBL flow rate (gpm)
- Recirculation flow rate (gpm)
- WBL temperature (°F)
- SBL temperature (°F)
- Power, current, or pump speed for all MEE pumps (kW, amp, or Hz)
- Steam pressure to the MEEs (psig)
- Steam boiler fuel types and quantities
- Boiler control strategy (baseload boilers with an auxiliary boiler)
- Recovery boiler fuel consumption (SBL gpm)
- Steam generation – pressure (psig)
- Steam generation – temperature (F)
- Steam generation – flow (lbs/hr)
- Pulp production (ADT)
- Soap collection (tons)

Membrane system

- Shutoff valve position (open/closed)
- T1 and T2 weak black liquor (WBL) solids (%)
- T1 and T2 concentrated black liquor (SBL) solids (%)
- T1 and T2 permeate solids (%)
- T1 and T2 WBL flow rates (gpm)
- T1 and T2 SBL flow rates (gpm)
- T1 and T2 recirculation flow rates (gpm)
- T1 and T2 permeate flow rates (gpm)
- T1 and T2 WBL temperatures (°F)
- T1 and T2 SBL temperatures (°F)
- T1 and T2 permeate temperatures (°F)
- T1 and T2 tank levels (%)
- WBL pressure (psig)

- SBL pressure (psig)
- Differential pressure for all pumps (psi)
- Steam pressure to the MEEs (psig)
- HPR pump power, current, or pump speed (kW, amp, or Hz)
- Recirculation pump power, current, or pump speed (kW, amp, or Hz)
- CIP pump power, current, or pump speed (kW, amp, or Hz)
- Steam boiler fuel types and quantities
- Recovery boiler fuel consumption (SBL gpm)
- Pulp production (ADT)
- Soap collection (tons)

8.2 National Impact

This analysis projects the potential energy and carbon impact related to membrane-based systems to aid the existing steam-based evaporators to concentrate black liquor in the pulp and paper industry. This is based on ITV evaluation results, data related to national energy consumption and black liquor production, and other assumptions obtained from existing literature across the pulp and paper industry (see Appendix Table A.1).

Total energy consumption in the pulp and paper sector is estimated at 2,343 TBtu, with an annual output of 45 million tons across 99 kraft pulp mills in the U.S. (Agenda 2020 Technology Alliance 2016). With a production of 2,800 lbs of strong black liquor solids production per ton of pulp processed, the energy consumption to process black liquor stands at 3.6 MMBtu/ton, using a steam-based evaporator system for black liquor concentration. ITV evaluation results from a small-scale demonstration indicate that these membrane systems consume 0.035 kWh/gal of electricity to process 0.55 gpm to concentrate black liquor from 15% to 20% solids. The results show an increased site energy consumption of 132 Btu/gal (due to added pumping electricity) and a reduction of 168 Btu/gal (due to reduced steam consumption by the evaporators), resulting in net savings of 36 Btu/gal.

Based on this analysis, adopting the membrane system to preconcentrate WBL to support the MEEs would save 0.31 MMBtu/ton, while resulting in an increase in electricity consumption of 70 kWh/ton. Though there is not a significant carbon impact from this technology when a current U.S. grid average emissions factor is assumed, this obscures the potential impact. First, it does not consider local variations – some regions have much cleaner electricity than the national average, which would lead to a net emissions reduction today in those regions. Furthermore, with current and expected trends in grid emissions, the emissions impact of this electrification technology will likely become more significant in the future, regardless of region. Based on market projections, if the technology were to be adopted across all pulp and paper plants in a similar configuration, it would reduce net GHG emissions by 0.8 million tons in CO_2e equivalent based on 2030 grid composition projections.

SECTION 9

Appendix

9 Appendix

Figure A.1 shows HPR (positive displacement) pump specifications, while Figure A.2 shows the recirculating (centrifugal) pump curve. Note that pump manufacturer and model numbers were removed to protect Via Separations' proprietary technology.

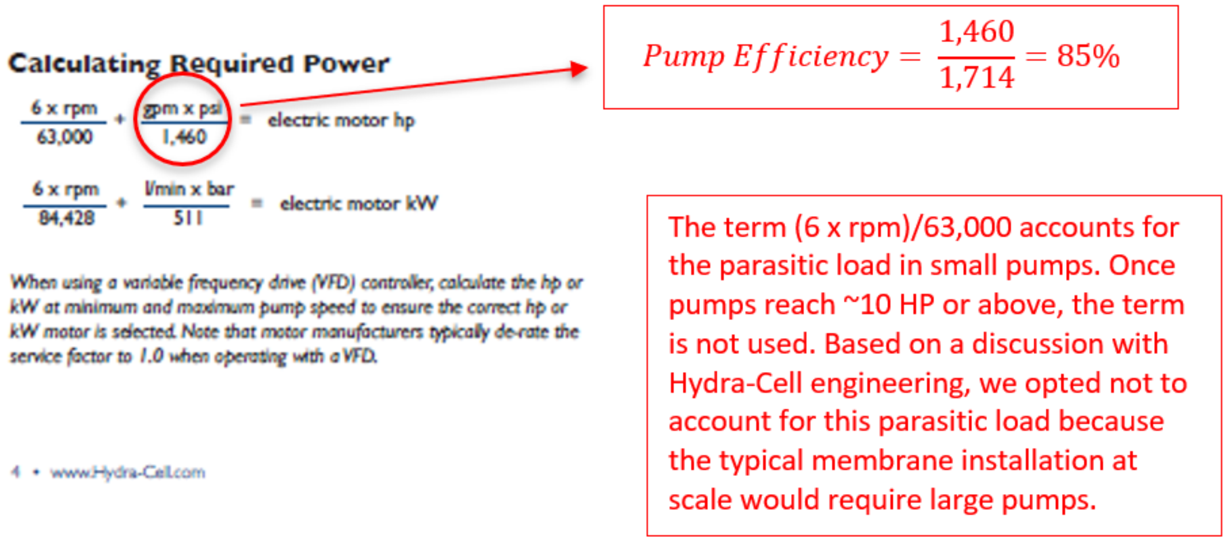

Figure A.1. HPR (positive displacement) pump specifications

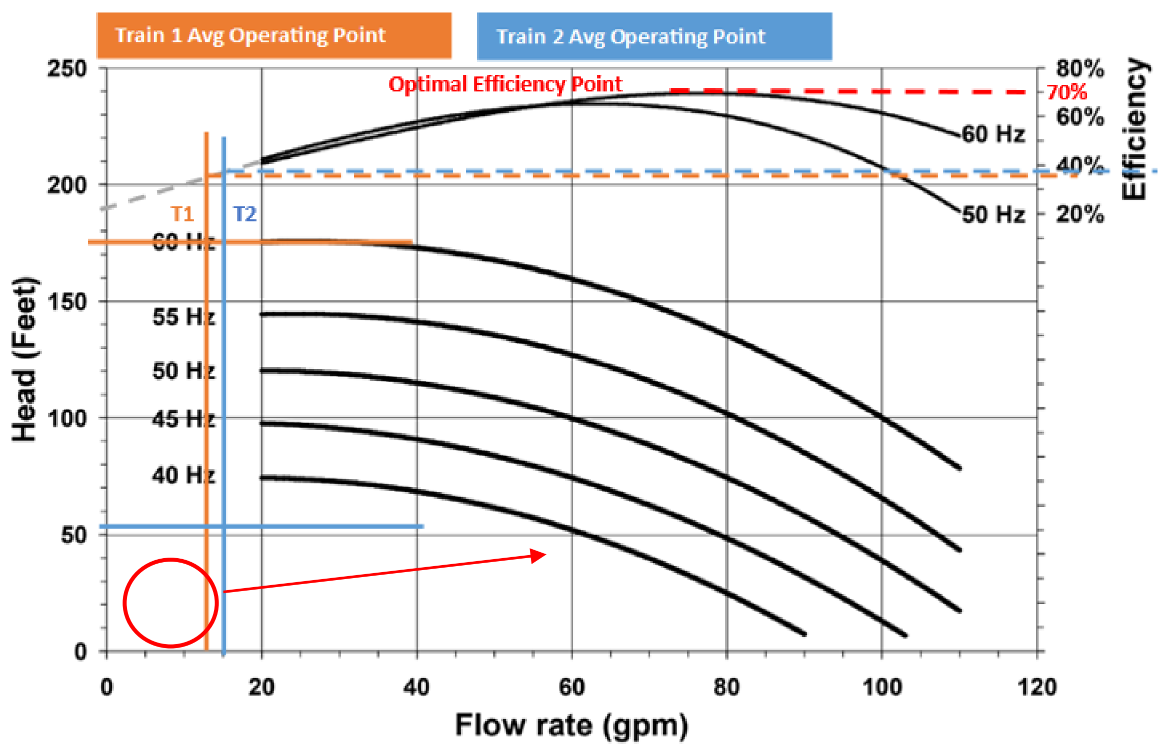

Figure A.2. Recirculating (centrifugal) pump curve

Table A.1. National Impact Energy Analysis for Membrane-Based Black Liquor Concentration Systems

Research Roadmap on Black Liquor Concentrationa	Number	Type (Units)a
Total U.S. manufacturing sector energy consumption	22,700	Energy (TBtu)
Pulp and paper sector energy consumption	2,343	Energy (TBtu)
Black liquor concentration energy consumption	164	Energy (TBtu)
Number of kraft pulp mills in U.S.	99	
Output	45	mil tons
SBL production	2,800	lbs per ton
SBL/WBL Ratio	15%	
WBL Production	18,667	lbs per ton
Total WBL Production	840,000	Mil lbs
Black liquor concentration energy consumption per unit of WBL	3.64	MMBtu/ton
Black liquor concentration energy consumption per unit of WBL	195	Btu/lb
Black liquor concentration energy consumption per unit of WBL	1,952	Btu/gal
Electricity Impact	0.07	MWh/ADT
Normalized Electricity per lb of WBL	0.00375	kWh/lbs
Normalized Electricity per gal of WBL	0.0375	kWh/gal
Normalized Electricity per gal of WBL (Site Btu)	127.95	Btu/gal
Normalized Electricity per gal of WBL (Source Btu)	358.26	Btu/gal
From ITV Via Demonstration Analysis (With Original Pump Efficiency Assumptions)[a]	**Number**	**Type (Units)[a]**
Pump power (kW)	1.15	kW
WBL Flow (gal/min)	0.55	gal/min
ITV Electricity per gal of WBL (kWh/lb)	0.00348	kWh/lbs
ITV Electricity per gal of WBL (kWh/gal)	0.035	kWh/gal
Normalized Electricity per gal of WBL (Site Btu)	118.90	Btu/gal
Normalized Electricity per gal of WBL (Source Btu)	332.93	Btu/gal
Site Steam Savings (Btu/lb) (at 4.8 SEE)	16.80	(Btu/lb)
Site Steam Savings (Btu/gal) (at 4.8 SEE at a density of 10 lbs/gal	168.03	(Btu/gal)
Thermal Savings (%)	9%	
Net savings (Site Btu) (elec +steam)	49.13	(Btu/gal)
Net Savings (%)	3%	
Total Energy Savings (USA wide)	4.13	Energy (TBtu)

[a] ADT=air-dried tons daily, Btu=British thermal units, gal=gallons, ITV=Industrial Technology Validation, kW=kilowatt, kWh=kilowatt-hour, lb=pounds, min=minute, M=million, MM=metric million, MWh=megawatt-hour, SBL=strong black liquor, WBL=weak black liquor

SECTION 10

Addendum

10 Addendum

This Operating and Performance Report focuses on Via Separations' graphene-oxide membrane energy consumption, steam conservation, and emissions observed in a limited-duration, pilot-scale field trial in 2021. Since starting this pilot-scale study, Via Separations submitted a TAPPI paper titled "Pilot Scale Black Liquor Concentration using Pressure Driven Membrane Separation" (Rae et al. 2023). The TAPPI paper describes other important considerations and results observed from field trials across multiple sites.

Four significant pilot-scale operational results are described in the TAPPI paper. First, the black liquor concentration stream (referred to in this paper as the retentate) showed a solids output increase averaging 5% and peaking at 8% over a 12-day test period. Second, performance stability was confirmed with a consistent permeate flow rate during 250 hours. Via Separations shared verbally with the ITV team that unfiltered suspended fiber solids were fouling the membranes in the A-M trail. Improved filtering of incoming WBL resulted in more sustained flow rates, as demonstrated in this paper. Third, the permeate quality was visibly clear and free of any color bodies that might interfere with reuse. Last, the steady-state pumping power required to extract clean water from WBL was calculated at 0.11 kWh/gal permeate, or 0.14 kWh/gal including CIP. For comparison, these energy intensities are roughly one-third of the first trial at A-M, which demonstrated 0.37 kWh/gal. These results show the technology has improved with more recent trials. Please see the referenced paper for more information.

Following this evaluation, Via Separations received funding from the Advanced Research Projects Agency – Energy ((ARPA-E 2022) and the DOE Office of Clean Energy Demonstrations (OCED) to conduct full-scale demonstrations using the technology (U.S. Office of Clean Energy Demonstrations n.d.).

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