Sustainable Swedish Industry by Improved Excess Heat Recovery

Low Temperature Evaporation of Black Liquor using Waste Heat

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Förord

Tack till alla ni som hjälp mig på den här resan till att idag kalla mig Civilingenjör.

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Abstract

Achieving a sustainable Swedish industry will require better utilization of resources already available and already spent. Nationally, vast amounts of heat are released into the atmosphere and not made use of. If only a fraction of this energy could be exploited, the path to net zero emission society by 2045 would be thousands of tons worth of CO_2 closer. In Sweden, 25% of excess heat delivered to district heating has its origin in the pulp- and paper industry. The importance of this, is that it can be assumed that available excess heat can be delivered through existing infrastructure as district heating. Research focused on Kraft method of pulp production, an energy intensive chemical pulping process with great potential for excess heat recovery. One of the key steps in Kraft pulp production is the chemical recovery of spent cooking chemicals, known as black liquor. The spent liquor has an initial water content of around 83 w/w% and needs to be concentrated to 15 w/w% water content, for chemicals to be recovered and burnt in the recovery boiler.

Today, evaporation is done by multiple effect evaporators at a capacity of thousands of tons black liquor per day, making it one of the world's most energy intensive processes. Here, Helios Innovations AB novel evaporation technology is studied, and it is concluded that it is possible to evaporate black liquor using temperatures considered waste heat. Pilot and laboratory scale tests conclude that packing materials, used for mass and heat transfer, can withstand relevant conditions for long-time exposure. Tests reveal performance of vapor condensation rate with an average of 15-17% lower compared to pure water. Large-scale design has potential of recovering 386 GWh/a thermal energy, saving up to 67 GWh/a of steam to power existing effects. Electricity savings of up to 42 GWh per year translate to cost savings on electricity be worth up to 14.7 MSEK/a. A Helios retrofit can potentially increase production capacity in evaporators by 6%. Potential energy savings can be translated to mitigated CO_2 emissions of 26,700 tons/a.

Sammanfattning

Att uppnå en hållbar svensk industri kommer kräva en högre utnyttjandegrad av tillgängliga resurser och bättre utnyttjande av de resurser som redan har spenderats. På nationell nivå släpps enorma mängder värme till atmosfären, utan att tas tillvara på. Om så en bråkdel av denna energimängd kunde nyttjas skulle vägen till ett netto-noll utsläpp år 2045 vara tusentals ton CO_2 närmare. En del av tillgänglig spillvärme levereras däremot redan till fjärrvärmenätet. Det uppskattas att 25% av den levererade värmen i Sverige kommer från pappersmassaindustrin. Det innebär att man kan anta att infrastruktur kring leverans av överskottsvärme redan kan levereras till befintligt fjärrvärmenät. I denna studie undersöks pappers och massatillverkning via sulfat-metoden, en energiintensiv kemisk massaprocess där det finns stor potential för ökad spillvärmeåtervinning. Ett av de absolut viktigaste stegen i processen är återvinningen av kokningskemikalier, känt som svartlut. Kemikalierester från kokeriet har en ingående vattenhalt av cirka 83%, men måste koncentreras till 15% för att kemikalierna och värmevärdet ska kunna återvinnas i sodapannan.

I dagsläget görs detta med flerstegs-indunstare med kapacitet på flera tusen ton förångad mängd vatten per dygn, vilket gör denna industri till en av världens mest energiintensiva. Helios Innovations AB:s nya indunstningsteknik studeras i denna rapport och har tekniskt validerat att tekniken kan indunsta svartlut med lågtempererad spillvärme. Tester i pilot och laboratorieskala har visat att packningsmaterialen som används i enheten, till mass- och värmeöverföring, står emot processförhållanden som råder, även vid längre exponering. Testerna visar lovande prestanda med en genomsnittlig kondenseringshastighet på 15–17% lägre hastighet, jämfört med rent vatten. En storskalig design har potentialen att tillvarata 386 GWh/år termisk energi, beräknat på förutsättningarna på bruk A. Det översätts till en besparing på 67 GWh/år färskånga som idag driver befintliga indunstare. Elektricitet kan sparas med upp till 42 GWh per år och kan översättas till kostnadsbesparingar för el på upp till 14,7 miljoner kronor/år. En retrofit med Helios teknik kan potentiellt öka produktionskapaciteten i befintliga indunstare med 6%. Energibesparingen på bruk A kan översättas till en minskning av CO_2-utsläpp på 26 700 ton/år.

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1   Introduction

To achieve a sustainable industry every country must assess their climate impact on a social, economic, and technological level. Reaching net zero carbon footprint requires not only sustainably sourced fuels, but a reduction of total energy demand. Minimizing required energy improves chances of switching to a sustainable industrial economy within one generation. Furthermore, energy efficiency improvements can drastically increase profitability in the industry and help cope with stricter regulatory standards. Heat and electricity savings can be used to either increase production capacity or increase amount of energy delivered as district heat.[1]

Vast amount of thermal energy is consumed in the pulp and paper industry. This industry account for 1.5-2 TWh of available waste heat per year, or 25% of Sweden's total available waste heat that is delivered to district heating.[2] Here, waste heat is energy that is of too low quality to be used in production processes and is usually in the span of 70-120°C. This thermal energy is equivalent to a magnitude of 110,000 electrically heated households, or 330,000 households supplied by district heating per year.[2,3] In this report, research was focused on waste heat streams in the temperature span of 40-95°C.

There are several ways of producing pulp and paper, with one of the common methods being the Kraft method. This is a chemical pulping method where the chemicals do convert the wood into chemical pulp. This process is also where most of the power consumption occurs. To solubilize the lignin and remove cellulose, an alkali solution of white liquor at high temperatures, is used to digest the raw material. Cooking chemicals in white liquor in Kraft pulping include sodium hydroxide and sodium sulphide. After the cellulose is separated, the following stream known as black liquor has a high moisture content. To recover chemicals and heating value, the solution is burnt in a furnace. To burn, it must be concentrated to dry substances of 65-85 w/w% by an intensely energy demanding process where the water content is evaporated by multiple effect evaporators. Existing processes are powered by high pressure steam, a primary source of energy.[4,5]

1.1 Aim

In this study, the main goal was to improve energy and resource efficiency at Swedish pulp and paper manufacturers. Recovering excess heat could potentially decrease demand for renewable fuels in this industry by saving steam or increasing capacity in existing operations.

Helios Innovations AB, a Swedish start-up company have developed a novel approach to evaporate liquids and make use of otherwise wasted energy. The technology utilizes low temperatures (40-95°C) as its primary source of energy. The report presents a study on whether this novel approach is suitable for evaporation of black liquor using waste heat available in Kraft pulp and paper manufacturers. If technically possible, this innovation has the potential to drastically reduce energy demand in this industry and possibly mitigating thousands of tons worth of CO_2. It is investigated if available heat can be exploited, how to best make use of it and quantify the impact. Helios technology is studied and will see if it could improve energy utilization by using heat that is presently being sent to coolers or not being exploited to a satisfactory degree.

1.2 Research questions

- Is waste heat available in the pulp and paper industry?
- Can Helios technology be used to evaporate process liquids using waste heat?
- How would a retrofit best be implemented?
- How would an implementation affect the energy consumption?
- Will a retrofit decrease the release of greenhouse gases?

1.3 Scope

The scope is to deliver a proposal of how to implement Helios technology in the Swedish pulp and paper industry. This will be done mainly by:

- Research of available energy flows with potential to be exploited.
- Data collection from Swedish pulp- and paper producers.
- Explore conventional technologies used for evaporation.
- Explore barriers of implementation of energy efficient technology.
- Reduce internal steam demand, to make use of those resources elsewhere.
- Quantify potential savings and performance increases.
- Experimental setup in small scale using black liquor.
- Packing material tests to verify that it can withstand process conditions.

1.4 Boundaries

The project will only consider the possible implementation of Helios technology using waste heat and no other competing technologies. System boundaries do not include disposal of condensed vapor in the process. It is assumed that energy supplied is generated by biofuels such as lignin and black liquor. Here, only pulp and paper manufacturers in Sweden using the Kraft chemical pulping process are studied. Analysis on cleanliness of condensed vapor is outside the scope of this report.

2   Background

2.1 Helios Innovations AB

In this report, a comparison and technical validation in chemical pulping applications of the novel technology developed by Helios Innovations AB is presented. The evaporator saw its first applications in desalination of sea water (Figure 1), to produce process water for industrial use, lowering their need for clean drinking water. Recent trials have concluded that the technology also works for evaporating liquids other than sea water, such as hazardous waste.[6]
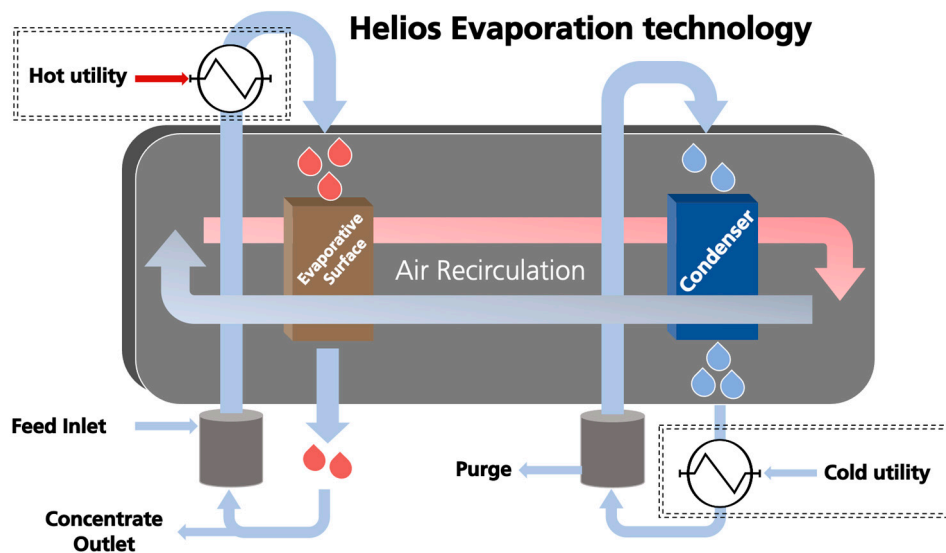

Figure 1: Schematic view of Helios innovation's dewatering technology.

Helios's technology utilizes indirect heat exchange to heat the incoming flow of liquid to be evaporated. Hot liquid is sprayed across a packing material, where air is blown cross directional to the flow of the liquid. Unsaturated air becomes saturated and heat from the liquid stream is removed as it is blown across the packing materials. In a traditional cooling tower, the heat is vented to the atmosphere. Here, the saturated air is led to a condenser and a condensate stream is collected before recirculating the air to perform another cycle. If all available excess heat could be utilized with a Helios unit, the resource efficiency of Swedish industry could be dramatically improved.

Packing material of cellulose is traditionally used by Helios technology. The material must endure conditions with process liquids at elevated temperatures for long periods of time. In previous pilot scale applications, the impregnated cellulose packing material, continuously endured process conditions with brine solutions and hazardous waste at elevated temperatures.[6] The packing material creates a large surface area for the drying agent in contact with feed to be dried. The surface acts as the interface where both mass- and heat transfer can take place. In applications like direct drying or cooling, this is one of the most critical parameters for efficient operation.[7]

Plenty of industries use evaporative processes, with fresh steam as a common primary energy source. By better exploiting these resources, a great cut of yearly carbon dioxide emissions can be lowered. Great efforts and expenses are presently allocated to achieve a net zero carbon emission by 2045.[8] Between 2018-2020, an average emission of 49.75 million tons of CO_2 was emitted, a far away from zero still. An average of 2018-2020 are presented to better represent data-set; 2020 was an abnormal year due to the COVID-19 pandemic.[9] Energy from biogenic sources such as burning lignin or black liquor, the intensity of carbon dioxide emission is estimated to 345.6 kg CO_2 per MWh energy used.[10] A rough estimate concludes that per TWh of saved excess heat, a potential save of 345.6 thousand tons of CO_2 can be achieved, every year. This is the equivalent of almost 1% of Sweden's total net CO_2-emissions, based on biogenic CO_{2e} emission factors on request of Naturvårdsverket.[9, 10]

2.2 The Pulp and Paper industry

Being one of the most energy intensive industries in the world[11], improving efficiencies in pulp- and papermaking processes can greatly contribute to sustainable developments. Annually, 12 Mtons (2020) of paper is produced each year in Sweden, of which almost 90% (2020) is exported.[12, 13] Mainly, four methods of production exist.[5] In Sweden the most abundant pulp and paper production process is the Kraft process, also known as Sulphate process using pine as raw material.[13] Another chemical way of producing pulp is the Sulphite process, this process is not reviewed in this report. [4, 14]

The raw material (Figure 2), logs of primarily pine[13], arrive at the mill and enter a washing step to get rid of unwanted dirt. Pine is also commonly referred to as softwood. Logs are fed to a debarking section before heading into the chipping stage, where they are grinded down to small chips and screened. The process of turning the wood into equally small chips is important for the next step in the process; the digester.[4]
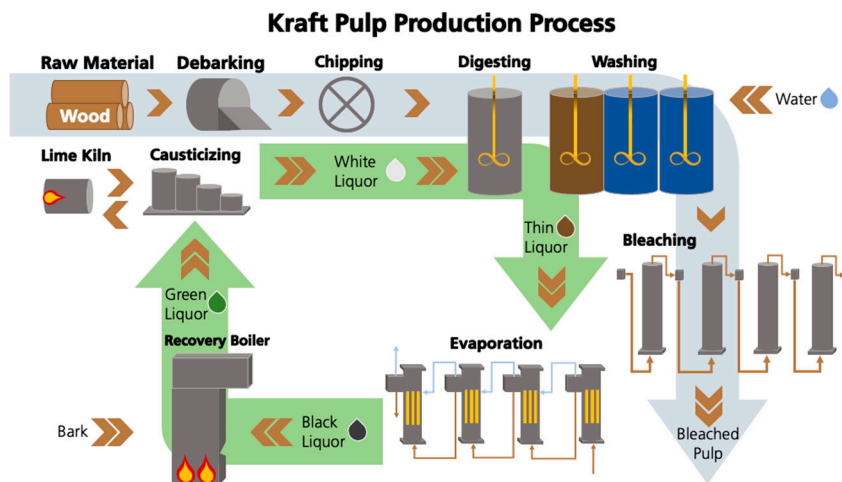

Figure 2: Overview of Kraft pulp- and paper production process. The schematic is a general description and variations may exist. Detailed steps are not included, but the main pathways are shown.

In the digester, (Figure 2) wood chips are fed to a boiler continuously or batch-wise. There, white liquor is added and heated with steam to 160-170°C at a raised pressure of 7 bar.[15] The white liquor is an aqueous solution of primarily sodium hydroxide (NaOH) and sodium sulphide (Na_2S) with a pH ranging between 13,5—14.[4, 16]

In biomass used for paper production, known as lignocellulosic biomass, the cell wall of the plant is constituted by cellulose fibers, hemicellulose, and lignin (Figure 3, Left). Cellulose fibers are layered in a regular way, with hemicellulose and lignin chemically bonded in intervals between the layers. Breaking the chemical bonds and extracting the cellulose fibers from the matrix is primarily done in the digester.[17]

The digester breaks the chemical structure of the lignocellulose and solubilizes the lignin (Figure 3, Right). The solution from the digester is washed with water and separated in two streams. The stream of solubilized lignin and residual carbohydrates, make up a stream known as thin Liquor (or weak liquor), with a solids content of about 15 w/w%.[18, 19] The second stream of mainly cellulose fibers is headed to further pulp treatment. For production of paper from pulp, bleaching is a commonly employed process, but not always used.
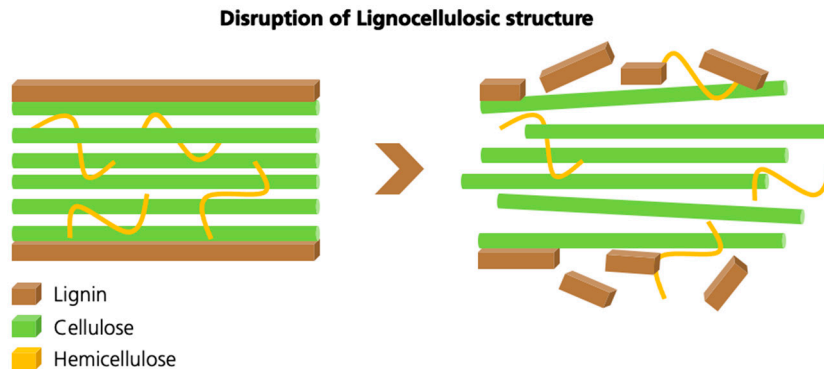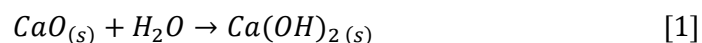
Figure 3: Disruption of Lignocellulosic structure and solubilization of lignin by white liquor in pulp production.
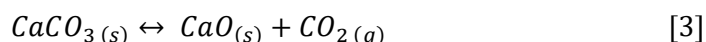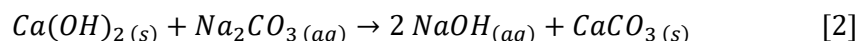
To recover chemicals and make use of heating content of dissolved lignin and carbohydrates, the solution must be concentrated. Thin liquor is concentrated to a dry weight of 65-85 w/w% in multiple effect evaporators before being combusted in the recovery boiler.[15, 19] The feed coming from evaporators that is combusted is known as black liquor (Figure 2). It is combusted together with bark from the debarking step, forming a melt of inorganic chemicals and generating steam. The superheated steam produced powers several functions of the mill. Kraft process would not be economically viable without this function, as the heating value of the organic components (mainly lignin) is of great importance, along with the chemical recovery. The heat of combustion is used to produce steam or energy in a combined heat and power (CHP, or co-generation) type process.

Reports show that precipitation of sodium sulphates (Na_2SO_4) and sodium carbonates (Na_2CO_3) in black liquor occurs at solids concentrations above 48-53 w/w%.[18] Concentrating black liquor may also produces volatile gases of both methanol and sulphur compounds. Before condensed gases are disposed of, these compounds must be treated before being disposed of. [20]

Typically, black liquor containing 65 w/w% solids have a viscosity of 200 centipoise at ~93°C. For black liquor from softwood species as pine or conifer; 200 cp is a common viscosity at 70 w/w% solids at 110°C. I.e., somewhat lower viscosity than that of hardwood black liquor.[18] As a point of reference, tomato ketchup has a viscosity of ~1000 cp at slightly elevated room temperature (30°C)[21]. A viscosity of 200 cp serves also as a practical limit for pumping systems.[18] Black liquor with a dry solids content of 75-85 w/w% behaves as a solid at room temperature and therefore need elevated temperatures of about 130-150°C for the pumps to function.[22]

The inorganic melt coming out of the recovery boiler, consists of sodium carbonate (Na_2CO_3) and sodium sulphide (Na_2S) is referred to as green liquor. This melt then heads to the Causticizing step to be reverted back to white liquor.[15, 19] The last step of chemical recovery occurs in the Lime Kiln, seen in Figure 2, where burned lime is mixed with water to form slaked lime (Ca (OH)$_2$) by the reaction [1] and [2] below.

$$CaO_{(s)} + H_2O \rightarrow Ca(OH)_{2\,(s)} \qquad\qquad [1]$$

$$Ca(OH)_{2\,(s)} + Na_2CO_{3\,(aq)} \rightarrow 2\,NaOH_{(aq)} + CaCO_{3\,(s)} \qquad [2]$$

$$CaCO_{3\,(s)} \leftrightarrow CaO_{(s)} + CO_{2\,(g)} \qquad [3]$$

Reaction [3], calcination, is an endothermic reaction and requires 850-900°C to operate, whereas Reaction [1] and [2] are slightly exothermic, occurring at 100°C. The causticizing step consumes a major part of external fuel consumed by the mill, but is necessary in the conversion of chemicals to white liquor that can be reused in the process.[19]

The chemical recovery of black liquor is a major heat producing step and the size of the boiler is determined by the production requirement of pulp. Pulp and paper manufacturers try their best to maximize the use of this heat by steam turbines, selling it to district heating or heating of production facilities and offices. As mentioned, the heat sold as district heat would in a generic case be at 70-120 °C and lower temperatures is considered waste heat in the context of this report.[23] Cooling of low-grade heat is usually done by either cooling towers, large fans or by large mass flows of water from nearby water bodies as lakes or sea. One of the treatment steps employed when handling wastewater is a biological step known as activated sludge. Microorganisms degrade matter present in the wastewater and help clean solutions that cannot be sent to recipients without treatment. Activated sludge and biological degradation basins can at a maximum be at a temperature of 45°C. Large cooling duties are supplied to keep these organisms from dying, as they are critical to the wastewater treatment on site.[24]

2.3 Technology Implementation

Adoption of energy efficient or environmental technology is a complex interaction between forces within and outside of the company. New technology can be divided into two main categories: clean- or end of pipe technology (EOP). Clean technology is defined as changes in the process that reduces pollution or waste generated per unit of production. EOP technologies are commonly retrofits that aim to handle emissions at the end of production, making pollutants easier to deal with.[25]

The mismatch between available technology that is both economically and environmentally attractive, compared to what is actually implemented is known as *energy-efficiency gap*[26]. In a clean technology article by González[25], factors determining adoption of new technologies are furthermore divided into:

Drivers for energy efficiency: In recent years, organizations have started adopting environmental preserving measures, as part of their corporate policies. However, it is argued that the main pursual for increased energy efficiency is more aimed towards coping with regulatory frameworks, rather than working towards sustainable processes.

Technological capacity: Technological capacity or competencies is regarded as the knowledge that managers within the firm possess about emerging and non-traditional technologies. EOP technologies are easier to adopt as they require less knowledge unfamiliar to the organization. Absence of competency of new cleaner technologies serve as one of the major threats against adoption of environmentally clean technologies.

Financial position: Firms and organizations with a better financial record are more prone to adopt clean technology. This can be traced back to the expensive nature of new, clean-, technologies demanding large capital investments that smaller firms may not afford.

Adoption behaviors in the pulp and paper industry are characterized by innovations that are provided and developed in collaboration with producers and technology suppliers. This industry is characterized by very large firms in a capital-intensive market and therefore the size of investments is relatively large. To penetrate the pulp and paper sector, the innovation must overcome listed obstacles to reduce the gap:[25, 26]

- High initial investment costs and long Pay-Back time.
- Constraints by both human and financial capital.
- Systemic changes to the production process.
- Satisfactory traditional technology.
- Not required to meet regulatory standards.
- Competitors.

A more recent study[1] of Sweden's pulp and paper industry suggest that more counterintuitive obstacles are considered more important and act as large barriers to implementation of new technology. Listed as the top of the rank on importance as a barrier to energy efficiency in the Swedish pulp- and paper industry ranks: risk of production and disruptions. It shows that these costly production stops are what is seen as the largest risk and obstacle when it comes to implementing new technology. More identified obstacles could be that technical managers lack influence; long decision chains hinder the process and a lack of budget funding do not create opportunities for new projects to develop.

On the other aspect, driving forces for these types of investments are said to prioritize cost reductions by lower energy usage. The lower energy usage is ranked as the most important driving force in this industry. Other driving forces include:[1]

- Electricity Certificate System (ECS)
- Long term energy strategy.
- Threat of rising energy prices.

ECS is seen as an important driver in this energy intensive industry. Many Swedish pulp and paper producers generate electricity by turbines This is driven by the fact that producers that can produce renewable electricity get a certificate for each MWh of electricity produced. Some pulp and paper producers use back pressure turbines to generate electricity and it is powered renewably through biomass on-site. In a similar study in the Finnish pulp and paper sector, these types of energy efficient investments typically had an average payback time of 2 years. Furthermore, this study presents argues that there is an energy gap in the Swedish pulp and paper industry.[1]

3   Theory

This chapter reviews basic theory of liquids in equilibrium with gaseous states, relevant for the understanding of evaporation systems.

3.1 Air-Water System

Vapor pressure is a measure of the tendency of a substance to go from one aggregation phase to another, e.g., liquid to gas. For water, the heat of vaporization and physical properties are derived from the intermolecular forces between water molecules, mostly hydrogen bonds. Due to these hydrogen bonds, the heat of vaporization is consequentially larger than for most other substances.[27] The vapor pressure for a material is directly dependent on the temperature. The vapor pressure at temperature T (°C) can be calculated by the Antoine's equation, showcased as equation (1) below. The Antoine constants A, B and C are species specific.

$$log\ P = A - \frac{B}{T + C} \tag{1}$$

To evaporate water, the liquid water needs to be above or equal to its boiling temperature (T_b). The boiling point temperature is where the vapor pressure of the liquid is equal to the surrounding pressure. For pure water at atmospheric pressure (101,325 Pa) this occurs as familiar at 100°C.[27, 28] Antoine coefficients for water are found in Table 1.

Table 1: Antoine's constants for water in the range of 1-100°C.[29]

Constant	Value
A	10.19625
B	1730.630
C	233.426

Heat of vaporization is the measure of how much energy (kJ) required to change 1 kg of liquid water into vapor and heat of vaporization for water is 2260 kJ/kg or 40.8 kJ/mol.[30]

3.1.1 Vapor-Liquid Equilibrium

To better understand the behavior of water and evaporation, it is relevant to look at the vapor-liquid equilibrium. Vapor pressure increases with temperature and water evaporates from the surface of a liquid body. However, when the pressure equals or exceeds the vapor pressure, water will start to condense. What happens is that the vapor is in thermodynamic equilibrium with the condensed state. This is known at the saturation pressure, denoted P_S.[7, 27, 28] The total pressure close to the surface can be calculated as the sum of the saturation pressure and the surrounding gas pressure, being commonly air. This relation is found in Eq. (2) below:

$$P = P_S(T) + P_g \tag{2}$$

Looking into a gas/vapor mixture, the thermodynamic properties can be determined if two or more state variables in the system is known. Commonly used in Europe is the Mollier diagram, also known as a psychrometric chart.[31]

3.1.2 Boiling Point Elevation

Chemical, and physical interactions in a mixture of components may increase or decrease the temperature at which a liquid boil for a given pressure. When i.e., inorganic salts are introduced in a binary (two component) solution with water, the vapor pressure of water will decrease, as the ionic concentration is increased. For black liquor, relations for boiling point elevation (abbreviated BPE) at increased dry solids concentrations exist. Järvinen et al.[22] derived a simple equation, based on Raoult's law for a binary solution of water and dry solids in black liquor. Experimental data provided by Järvinen et al.[22] at 50% dry solids content lies around 7-8°C. This is confirmed by Clay.[20] Furthermore, the author provides a correlation of boiling point elevation below 51%, with stated values listed in Table 2.

Table 2: Complementing list of black liquor boiling point elevation below 50% dry solids.[20]

Dry Solids content (%)	BPE (ΔT, °C)
18	1.8
21	2.2
26	2.8
34	4.1
42	5.6
51	7.8

This model was experimentally fitted to 14 different compositions and variants of black liquor, performing within 0.5°C in the range of 50-85% dry solids. In Table 3 the results from the beforementioned study are listed. Listed values are derived from a model, developed to predict BPE in black liquor solutions. The author (Järvinen et al.[22]) mentions that the model underestimates the BPE at 50% and below but predicts well within ±0.5°C at higher dry solids content, compared with experimental values.

Table 3: Model results in article by Järvinen et al.[22] for predicting boiling point elevation in black liquor at 50-85% dry solids content.[22]

Dry Solids content (%)	BPE (ΔT, °C)
50	6
60	9
70	13
80	21
85	27

3.2 Evaporation

The process of evaporating a liquid, aim to increase the percentage dry weight of a desired feedstock. Evaporation or drying technology may also be classified depending on what heat source is used, either indirect or direct heat. Types of heat sources that are relevant to discuss in the context of Helios novel technology are listed below:[28]

1. Convection drying with hot air in contact with material.
2. Conduction from a hot surface.

The basics of drying involve heating a fluid, turning it into vapor and condensing it at a desired location. For water in air, the relative amount of vapor that can be sustained in the air at a certain temperature and pressure, is decided by the saturation pressure. For vapor pressures above the saturation pressure (P_S), or temperatures below the saturation temperature (T_S) water will start to condense. This is known as the Dew point (t_d). Humidity Ratio (W) is used to describe the amount of moisture per kilogram of dry air (kg H_2O / kg Dry Air). The calculation is done according to Eq. (4), and requires that the average molecular weight of gases, and the Moisture by volume (% M_V), is known. Moisture by volume is the ratio of partial pressure of water in air (P_w) divided by the total pressure (P_T). The factor of 0.622 in Eq. (4) is the ratio of molecular weight of water vapor divided by the average molecular weight of gases present in dry air.[32]

$$\% \, M_V = \frac{P_W}{P_T} * 100 \tag{3}$$

$$Humidity \, Ratio = W = 0.622 * \frac{\% \, M_V}{(100 - \% \, M_V)} \tag{4}$$

There are two types of moisture: bound and unbound moisture. Unbound moisture is easily evaporated as it has the same or excess vapor pressure than for that of pure water or solvent at a given temperature. Bound moisture can be adsorbed either physically or chemically to a solid and exerts a vapor pressure less than that of pure water or solvent at given temperature.[33, 34]

Drying is a complex process but for a given set of conditions it can be divided in four periods (Figure 4). In the first period, the drying agent heats the unbound moisture to a point of evaporation; second, the maximum drying rate is achieved after the initial heating period and the drying rate is constant; third, as the feedstock is dried out, heat transfer is reduced, and drying rate will continue to decrease until the bound moisture of solids is evaporated. The equilibrium moisture content is the lowest amount of moisture a feedstock can retain for a given set of drying conditions.[33]
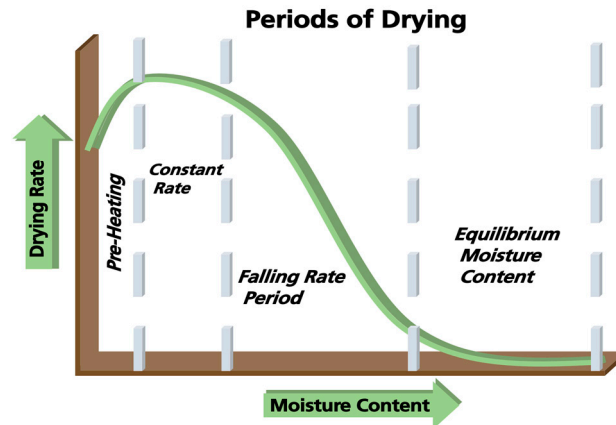
Figure 4: The different stages of drying as the surroundings gas get saturated with moisture and reaches its equilibrium.

3.2.1 Convection

There are two types of convection: natural or forced convection. Natural convection is the movement of a fluid, caused by increased kinetic energy (movement) of e.g., individual particles. The movement is induced by expansion, causing warmer particles to move and occupy a larger space. This creates local density changes, thus creating a net movement within the fluid. Forced convection is the net movement caused by an external force, i.e., an impeller or a pump.[7] Drying rate is a measure of the vapor flux escaping the surface per time unit, denoted $\dot{m}$ *(kg m^{-2}s^{-1})*. To provide heat for vaporization, a *drying agent* is used. Drying curves can be experimentally calculated by Eq. (5) and the setup may look like the schematic in Figure 5. This is known as a direct contact drying method.

$$\dot{m} = -\frac{1}{A} * \frac{\Delta M}{\Delta t} = -\frac{M_S}{A} * \frac{\Delta X}{\Delta t} \tag{5}$$

Where A is the surface area that is in contact with the air, or so called drying agent; X represent the moisture content; t represent time; M is the mass of the component of interest.[7]
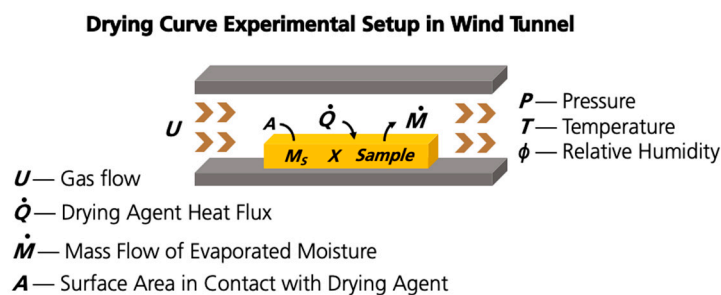
Figure 5: Experimental setup of drying curve test.[7]

3.2.2 Conduction

Conduction drying, also known as *Contact Drying,* is an indirect drying method. Simplified, this usually means that the feedstock that is dried, is separated from the heat source by a conductive material to provide heat transfer. When heat transfer is dominated by conduction, it is important to provide good mixing, forced convection, for equal distribution of heat in the fluid.[33] Falling film evaporators, are an example of an indirect drying method.

3.3 Conventional evaporators

After the digester, superheated thin liquor enters a flash separator, producing steam and increasing the dry weight of dry solids of incoming thin liquor. Two models of evaporators dominate the scene: falling film (FF) and rising film (LTV) evaporators, with the most common being the FF model. It shows more efficient heat transfer and less fouling formation than the LTV model.[18] The FF model is the only one presented in this report.

3.3.1 Flash Evaporator

A process stream of superheated liquid needs to be kept at higher pressures to remain in its liquid state. By quickly reducing the pressure, or by expanding the volume, a fraction of the superheated liquid can turn to vapor. Seen below in Figure 6 is a flash steam evaporator.

Flash Steam Separator
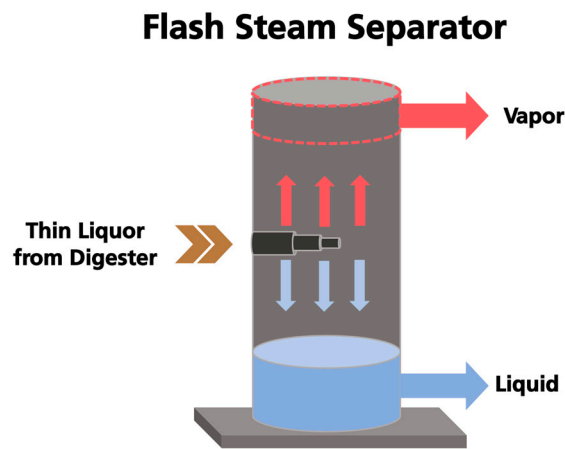
Figure 6: Flash Steam Separator.

It is a rather simple process where hot superheated liquid is sprayed into a vessel at lower or atmospheric pressure. Depending on the pressure, a fraction is turned into vapor that can be used in a system operating at a lower pressure. By Eq. (6) the mass fraction (X_{Flash}) of steam produced per kilogram thin liquor can be calculated.

$$X_{Flash} = \frac{h_u^L - h_d^L}{H_d^V - h_d^L} \tag{6}$$

Where h_u^L and h_d^L is the liquid enthalpy upstream and downstream respectively to the spray nozzle seen in Figure 6. H_d^V represent the vapor enthalpy downstream the nozzle exit.

3.3.2 Falling film evaporator

Feed enters the top of the column where it enters a tubular section where black liquor covers the inner surface of the tubular walls. The outside of the tubes is where indirect heat exchange take place, condensing steam are used as heat source for evaporation. By gravity, the film moves downwards the tubular design and eventually enter a flash chamber. Vapor and liquid are separated by a droplet separator and the evaporated steam exits the top of the flash chamber, whereas concentrated liquor exits the bottom. To achieve desired solids content in single effect operation (Figure 7), the concentrated liquor need to be recirculated.[18]
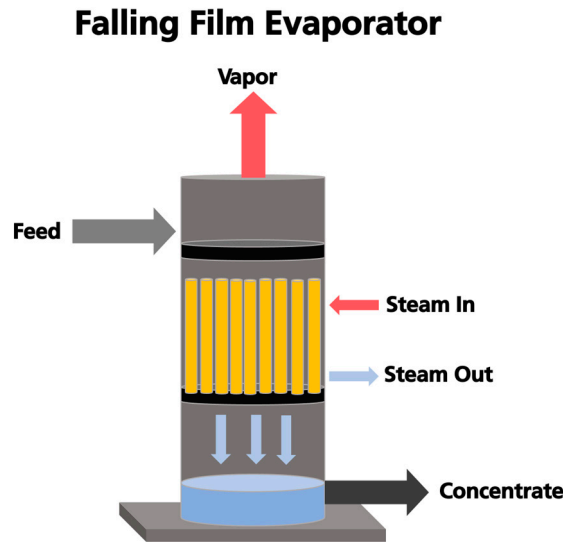
Figure 7: Single effect falling film evaporator.

To achieve desired solids content of black liquor, FF evaporators are usually coupled by several effects in sequence. This is referred to as multiple effect evaporators (MEE). In contrast to single effect operation, the evaporated steam is fed as a heating medium to the next evaporator in the sequence, evaporating more steam per mass of steam used. This is commonly referred to as steam economy, according to Eq. (7).

$$Steam\ Economy = \frac{Mass\ of\ Water\ Evaporated}{Mass\ of\ Steam\ used} \qquad (7)$$

Theoretically, in a six-effect evaporation train, a fresh steam intake of 100,000 kg could potentially evaporate 600,000 kg of water; a ratio of 1:6. A rule of thumb is that approximately 0.7-0.9 kg of vapor is produced for each effect, per 1 kg of steam condensed in the first effect. For a six-effect evaporator train: a ratio of 1:4 to 1:5.5 can be expected (steam economy between 4-5.5).[18]

4 Method

This chapter describes the methodology used for measurements and assembly of a new pilot test unit. Equipment assembly and experimental procedures are presented.

4.1 Validation

To technically verify that Helios evaporation technology works as desired, a pilot scale testing unit is designed. A small pilot scale reveal what capacity losses or increases can be expected and gives indications of problems that may need to be considered when scaling the unit to larger dimensions. Performance was measured in terms of vapor condensation rate at different process conditions. Packing material was evaluated based on degradation resistance in laboratory tests.

4.1.1 Equipment assembly

The equipment is designed and manufactured out of stainless steel 316L. Connections are made of brass and hoses made of PVC plastic. The design is created for easy cleaning and operation with drainage pipes in the bottom and a pre-built-in stainless-steel wall for fan-insertion. The design is based on the flowchart seen in Figure 8. Black liquor is pumped from a buffer tank into a heat exchanger before running across the evaporative surface where it meets the cross-current air stream. Similarly, on the right side of Figure 8, cooling water is recirculated in a condenser where it is cooled, and condensed vapor is collected.

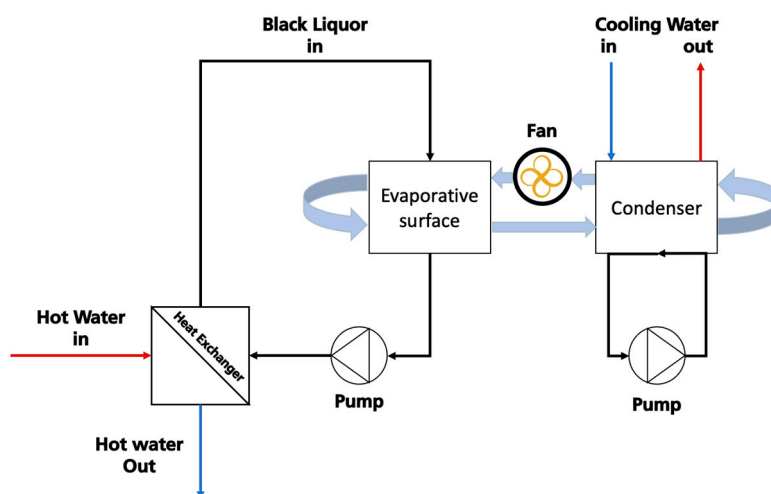## Flowchart of Experimental Setup

Figure 8: Flowchart of experimental setup. Recirculating black liquor flows across packing material and heat exchanges with hot water. Black liquor and cooling water from the condenser are recirculated from individual small buffer tanks.

Hot and cool utilities is supplied by two welded 30-plates-heat exchangers mounted on the back of the assembly (Figure 9, Right). Heat is delivered by an inhouse supply of 5 bar steam whereas cooling water is supplied by tap water outage. Liquid is heated and cooled in a countercurrent flow setup.
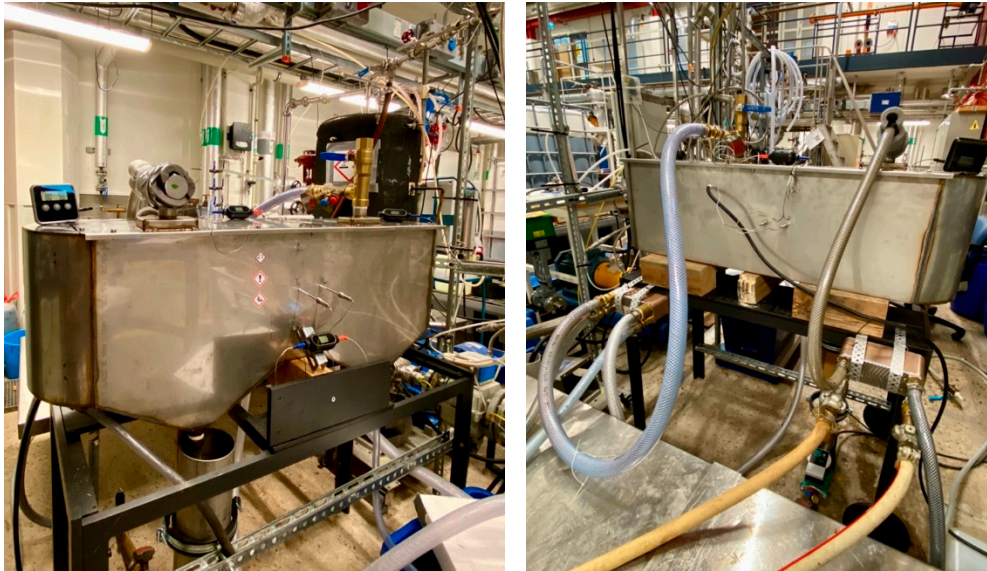
Figure 9: Final assembly of Helios pilot unit, used for tests in thesis.

Below the left outlet, a reservoir of feedstock is kept before recirculating back into the heat exchanger. Below the right outlet a container is placed on a scale to measure the amount of condensate in kilograms collected during operation. Electric pumps are placed below the pilot unit and recirculate the liquid back into the pilot unit at a controlled rate. Two probes measure the incoming liquid temperatures on hot feed side and incoming cooling water respectively. Seen in Figure 9, dual temperature probes are placed in series to determine relative humidity and air temperature. The right probe of the two pairs, are wrapped in a dishcloth and wetted. Temperature probes on the front are denoted Zone 1 (Figure 9, Left) and on the back Zone 2 (Figure 9, Right). By knowing the wet temperature in combination with dry air temperature, the humidity ratio can be calculated and monitored.

4.2 Data Collection

One of the goals with this report was to quantify the impact a potential implementation could have on in the pulp and paper industry. After collaboration with Södra and several meetings with energy managers at their facilities, a questionnaire was an appropriate way of gathering information needed for calculation. To quantify existing evaporator energy consumption the feed sent to evaporators in m^3 per hour and both heat and electricity consumption in kWh per tonne condensed vapor was of interest. In conversations it became evident that they had cooling water on site that they sent to cooling towers and both temperature and mass flow in m3 per hour was of interest to quantify available waste heat that could be used for evaporation.

Below is an English version of the questionnaire sent to pulp- and paper producers (Table 4). The response was used to study possibilities of an implementation and how it would affect energy performance. The original questionnaire in Swedish and

corresponding response is found in Appendix B and C respectively. Additionally, supplied by facility B: 400 kg of black liquor was delivered to Kemicentrum in Lund. The black liquor was used to test and performance of the pilot unit.

Table 4: Questionnaire sent to pulp and paper producers for data collection.

Facility	A	B	C	Unit
Evaporator Feed				m3 / h
Number of effects				Effects
Heat Consumption				kWh/ton
Electricity Consumption				kWh/ton
Temperature				°C
Feed to Coolers				m3 / h
Temperature				°C
Excess Heat				m3 / h
Excess Heat Temperature				°C
Solid Fuels burnt				GWh / year

4.3 Material Resistance

The material used for evaporation must be able to withstand black liquor at elevated temperatures. A series of test was be performed to evaluate resistance of degradation and scale build-up, according to Table 5.

Table 5: List of tests for the cellulose packing material in sodium hydroxide solution.

Test no.	Packing Material	Temperature (°C)	Time (hours)
1	Cellulose	50	1
2	Cellulose	80	24
3	Cellulose	80	48
4	Cellulose	80	30 [days]

Packing materials was submerged in 500 ml of sodium hydroxide solution and placed on a heating stove. A temperature probe monitors the temperature, and the experiment was performed inside a fume hood. Sodium hydroxide is dissociated in water by magnetic stirrers at room temperature. Packing material was in the size of 5x5 cm blocks as in Figure 10. During tests, the container was be sealed by a plastic film to avoid drying out.
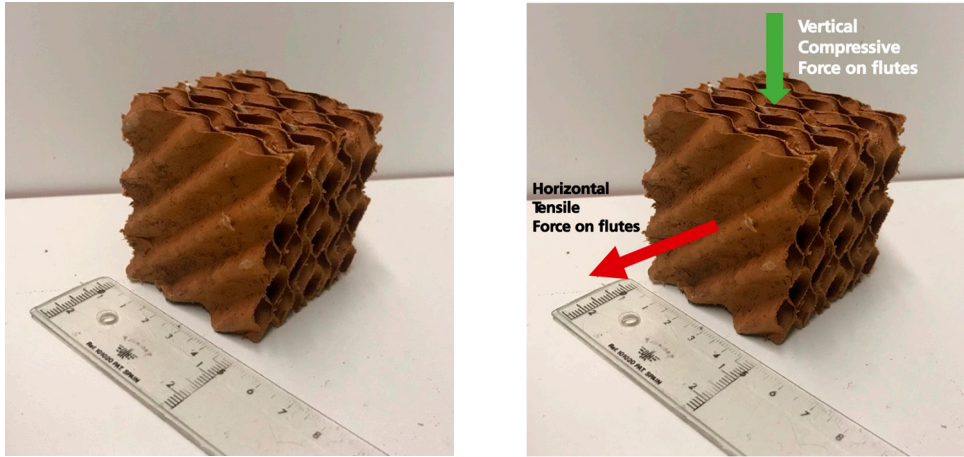
Figure 10: Cellulose blocks that undergone degradation test in sodium hydroxide solution.

In a horizontal direction (Figure 10, Right), the corrugated board is expected to remain weak. But by supporting these sides mechanically by holding the walls with a pair of pliers, the vertical compressive force on the flutes could be measured on a scale. The force can be calculated by load divided by the total upper surface area of the block according to Eq. (8). Where g is the gravitational acceleration of 9.82 m/s^2, the vertical compressive force is represented by Load and A_{block} is the upper surface area of the cellulose material. For a 5x5 cm block, this equals to a surface of 0.0025 m^2. Load applied is measured by putting the block on a scale and then applying weights on top. The load at which the structure fails to carry the load is considered the result.

$$F = \frac{g\left(\frac{m}{s^2}\right) * Load\ (kg)}{A_{block}} \tag{8}$$

Black liquor has a pH of approximately 13 with bases NaOH and Na$_2$S. To represent this environment a solution of sodium hydroxide and water is used. Sodium hydroxide is a strong base, and it dissociates completely in water, thus the pH of the solution can be calculated by the concentration of OH$^-$ ions in solution:[4, 16]

$$pOH = -log[OH^-] \tag{9}$$

For water at room temperature (25°C):

$$pH + pOH = 14 \tag{10}$$

Desired pH was 13, subtracting the right part of Eq. (10) result in a pOH = 1. Using Eq. (9) it is possible to determine the OH$^-$ concentration.

$$[OH^-] = 10^{-pOH} \tag{11}$$

Insert pOH = 1 in Eq. (11) and the molar weight of sodium hydroxide is 40 g/mol and thus calculated by Eq. (12) below. Rearranging and solving for X gives a weight of 2 grams of sodium hydroxide per 500 ml of deionized water to achieve a solution of pH≈13 and a molarity of 0.1 M.

$$\frac{\frac{X\ [g]}{40\ \left[\frac{g}{mol}\right]}}{0.5\ [L]} = 0.1\ \left[\frac{mol}{L}\right] \tag{12}$$

4.4 Pilot test: Water

As a point of reference and comparability of the performance with black liquor, the pilot unit was run with water. Intervals appear in Table 6. This method determined steady state conditions for the pilot unit. After a parameter has been changed, 5 minutes must pass before measurements can begin. Before testing began, several runs on the unit concluded that after 5 minutes the vapor condensation rate remained constant after changing a parameter. This allowes the system to enter a steady state where performance at different process parameters can be assessed.

Table 6: Pilot unit test intervals for water experiments. Percentages describe pump settings as a percentage of maximum flow.

Type	Variable	Test interval
Cooling water	Flowrate setting	63% of pump max
Pump hot side	Flowrate setting	15-100% of pump max
Temperature feed side -	Temperature in (°C)	50-80 °C
Fan		
Airflow	Velocity (% Max)	25-75%
Dry Temperature		
Gas	Zone 1 (°C)	
Gas	Zone 2 (°C)	
Wet Temperature		
Gas	Zone 1 (°C)	
Gas	Zone 2 (°C)	
Liquid temperature		
Temperature feed side	Temperature out (°C)	
Condensation block	Temperature out (°C)	
Condensate collection	% Of maximum	

Main performance figure that was reviewed in these tests was the rate at which vapor condensed in the condenser. This rate gave an indication of how the system was performing and was easy to monitor during experiments.

5   Results

This chapter presents results and insights from experimental work. This will provide the backbone for whether this technology is suited for an introduction to the pulp and paper process environments. Insights from this chapter will bridge into the next chapter, where a possible design and its potential is assessed.

5.1 Material resistance

In Table 7 the results are compiled to illustrate how packing materials withstood a sodium hydroxide solution, at 80°C for prolonged durations.

Table 7: Result of packing material tests submerged in hot sodium hydroxide solutions.

Test no.	Packing Materials	Visual Degradation	Time	Pressure applied before break (kg)
1	Cellulose	No	0	10
2	Cellulose	Yes	24 h	8
3	Cellulose	Yes	48 h	6
4	Cellulose	Yes	25 d	2.5

Test 1: Structural integrity was tested by a weight on top of the cube and placing it on a scale. Shaping of the packing material could have influenced the load performance, but it is representative of how Helios have prepared it in the past. The result compared similar to tests performed in earlier experiments by Helios[35], by submerging the cellulose in hot saltwater for prolonged durations. Results was a compressive strength of approximately 10 kilograms, or 39 kN per m^2.

Test 2: As appears on the left of Figure 11, after 24 hours, the solution is observed to have a dark yellow colour. The cellulose inserts are easily broken and separated from each other in a horizontal direction of force applied (Right, Figure 11). The cellulose was transferred to a beaker of tap-water before compressive test began, as indicated by the change in colour of solution in Figure 11.
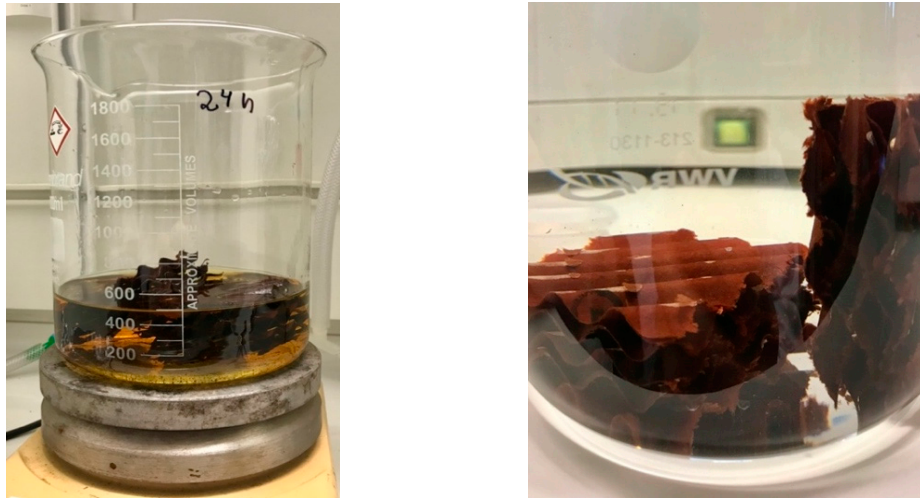
Figure 11: Cellulose Test result after 24 hours.

As expected, the resin between the corrugated paper sheet would not withstand a sodium hydroxide solution at elevated temperatures. In Figure 12 there has been some weakening of the resin between plates, but as appear by the white rings left on the paper sheets, they have not fully dissolved after 24 hours. The paper sheet retained their structural integrity comparably well. As appear in Table 7, it could resist a load of up to 8 kilograms before break, or 31 kN per m^2, when a counterforce was applied horizontally on sidewalls with a pair of clamps.

Figure 12: Cellulose test result after 24 hours. Resin dissolution between corrugated paper sheets.

Test 3: Similar to past tests, the sodium hydroxide solution has turned to a dark yellow colour (Figure 13). Compressive test resulted in a maximum load of 6 kilograms, or 24 kN per m^2. The paper sheets have softened somewhat but still maintained their structure in the vertical direction. After 48 hours, all paper sheets have separated, and no tensile stress in the horizontal direction had to be put on to facilitate the separation. This indicate that extended durations in sodium hydroxide solution dissolve the glue keeping the sheets together.
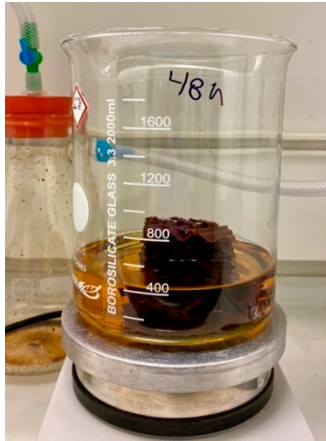
Figure 13: Cellulose test result after 48 hours.

In Figure 14 below, there are less resin-contact-points intact, compared to what was seen earlier in Figure 12, at 24 hours exposure.

Figure 14:Cellulose test result after 48 hours. Resin dissolution between corrugated paper sheets.

Test 4: a seal failure occurred during testing at 25 days of exposure. The solution went dry before test end date. Due to time constraints, the test could unfortunately not be redone. As seen in Figure 15, the part of packing material that was completely soaked in sodium hydroxide solution, have completely dissolved the resin between the flutes. Structural integrity of the material remained intact, but the material could be seen from earlier tests not withstand any tensile forces in a horizontal direction. Compressive test resulted in an approximate load of 2.5 kilograms of force applied before break or 9.8 kN per m^2.

Figure 15: Result of 25 days packing material test in sodium hydroxide solution.

5.1.1 Pilot Testing: Materials

After several days of testing in the pilot unit. The evaporative block could withstand liquid feed at stress testing up to 80°C and large mass flows. After tests with black liquor, the cellulose blocks show impressive structural integrity and remain intact as appear below (Figure 16).

Figure 16: Evaporative block after serial runs on black liquor at high temperatures and concentrations.

Tests on the pilot unit can visually conclude that the flutes retain their shape. As noted in laboratory tests, the resin between flutes is dissolved and blocks cannot withstand tensile forces in a vertical direction. In the pilot unit, blocks are compressed vertically to remain in place, this consequentially led to increased durability. Colour is darker than before testing, stained by the black liquor remains (Figure 10 compared to Figure 16). The broken flutes (Figure 16, Right) are not from testing, but are remains of shaping of the blocks. Throughout testing, the block continued to provide good mass and heat transfer for evaporation. Technically verifying that in this pilot scale, the block shows good resistance to degradation.

5.2 Pilot test: water

Seen in Appendix A; water tests on the pilot unit aim to provide a reference compared to later runs with black liquor. Variable parameters include air velocity, incoming feed temperature and mass flow. These were varied individually to determine the effect and estimate its importance.

Importance of gas flow: results indicate that little to no measurable effect were identified on the variation of air velocity after water trials. This could mean that the starting point were already set to a velocity that provided enough air to satisfy the mass transfer of vapor. Looking at a comparison of vapor condensation rate as a function of air velocity (Figure 17), no significant change in condensation is seen in this interval.
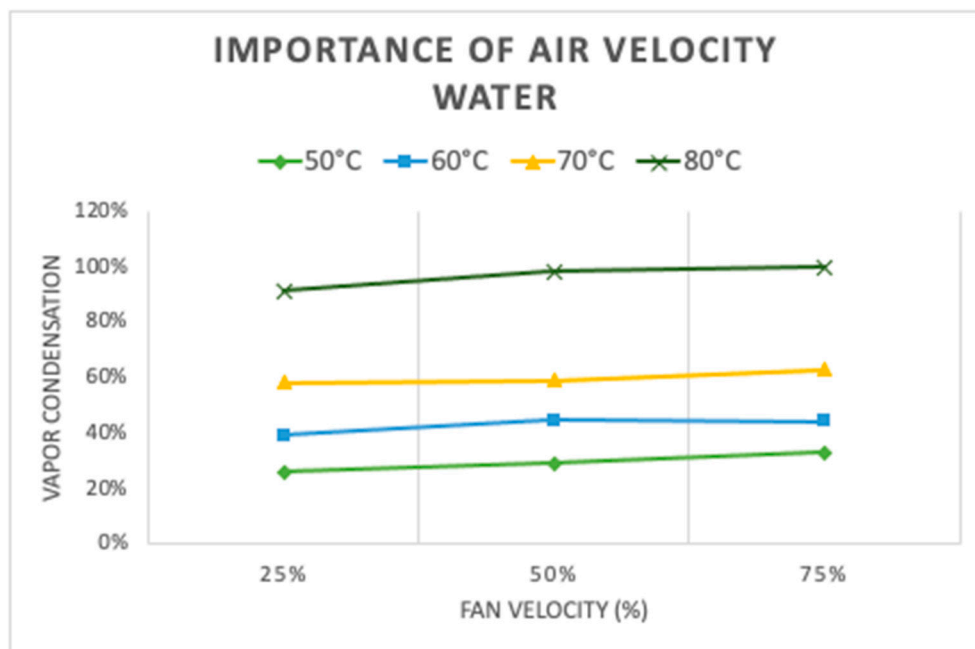
Figure 17: Test results on the importance of the air velocity at a feed flowrate of.

Temperature dependence: in this interval, air velocity seems to have no significant influence on condensation rate (Figure 18). The temperature dependence appears that at higher temperatures the evaporation increases, as can be expected.
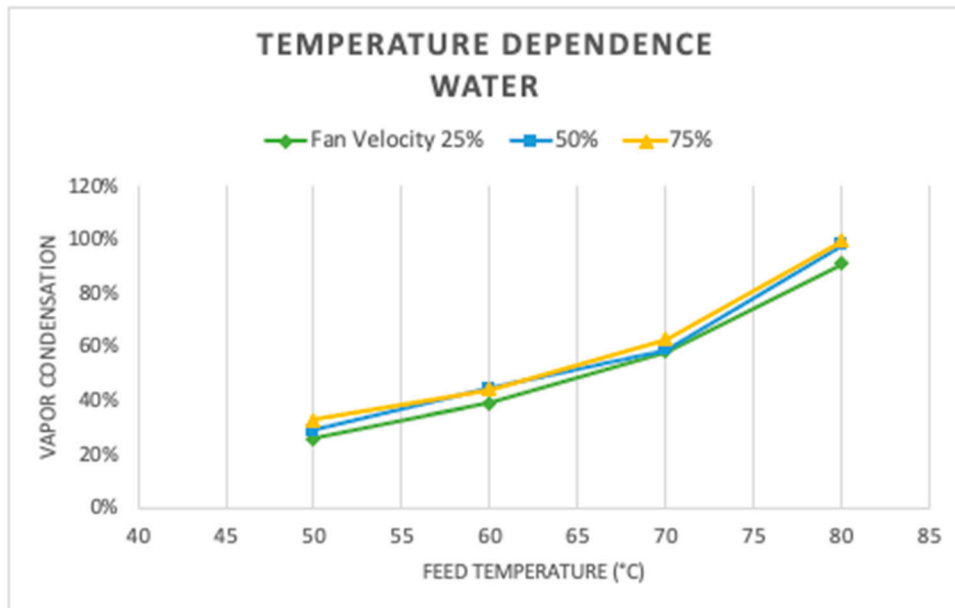
Figure 18: Vapor condensation rate as a function of temperature for tests with water.

5.3 Pilot test: Black Liquor

Potential and feasibility of Helios technology in pulp and paper industry, rests in the technical validation of evaporation of thin black liquor. Material tests have shown initial results that suggest that the packing material can withstand process conditions even at elevated temperatures. In these tests, additional stress was applied as both air and liquid are forced across the cellulose blocks. Like water, experimental results found in Figure 19 show that there is a clear correlation on increased vapor condensation at increasing temperatures. Dry substance tests on original black liquor were 17 w/w% by weight. Dry substance was measured by a small sample of black liquor being put in an oven at 105°C for 24 hours. The remaining weight divided by the original weight results in fraction dry substance. During testing, dry substance varied and it was evidently hard to have the same exact composition for every test. Tests were run in series and when the black liquor started to thicken, the black liquor was disposed of and new black liquor at known dry substance was used. A larger tank could have been used to prevent the quick concentration increases.

As appear in Figure 19 there is a strong correlation between amount of vapor that condensates as a function of temperature. Data points all have the same mass flow for comparability.
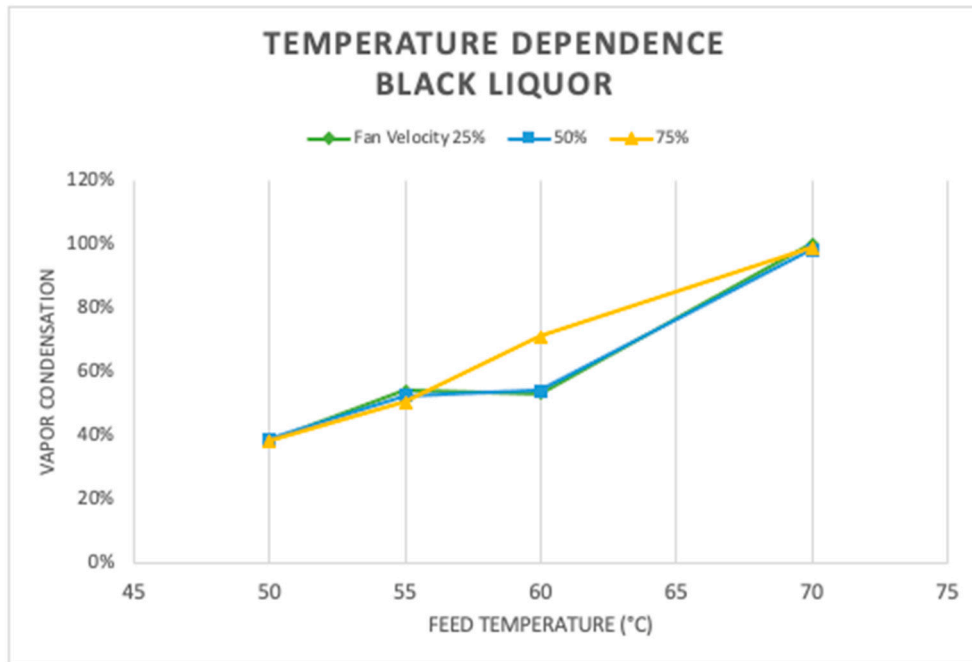
Figure 19: Vapor condensation rate as a function of temperature for tests with water.

More interestingly is the comparison of water versus thin black liquor. It is expected that solutes should decrease the vapor pressure and thus lower evaporative capacity. In Figure 20, it is confirmed that the vapor pressure for thin black liquor is between 15-20% lower in the interval of 50-60°C.

At 70°C the difference is only 1%, that could be explained by some foaming that occurred during the run-on black liquor. The hypothesis is that the foaming isolated the temperature sensor, registering a lower temperature than the actual temperature of the fluid. This was compensated for by increasing the amount of hot steam to get back to operational temperature. This would have led to a higher vapor pressure than intended at the original setpoint of 70°C, causing the thin black liquor to approach condensate rates of pure water. A dry substance test showed that the behavior of foaming occurred at 39.6 w/w%. It is uncertain if this was a loss of temperature of the liquid or foaming occurred. The prototype unit is a closed system, there is yet no answer to this.
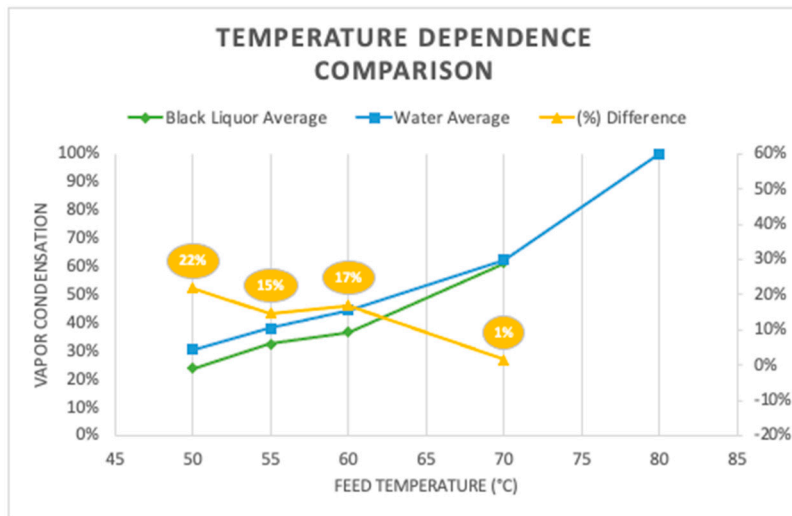
Figure 20: Temperature dependence comparison of water versus black liquor. The primary axis (left) represents percentual data on black liquor and water average in terms of vapor condensation rate. The secondary axis (right) is the relative difference between black liquor and water experiments.

From the psychrometric chart available in the Chemical Engineering Handbook[29], partial pressures for water and black liquor can be estimated. The dry and wet temperature of air was measured for each data point on the pilot unit. From Figure 21 the differences between pilot runs for water and black liquor are plotted at different liquid feed temperatures. Note that data points for each temperature, with similar flow settings have been averaged.
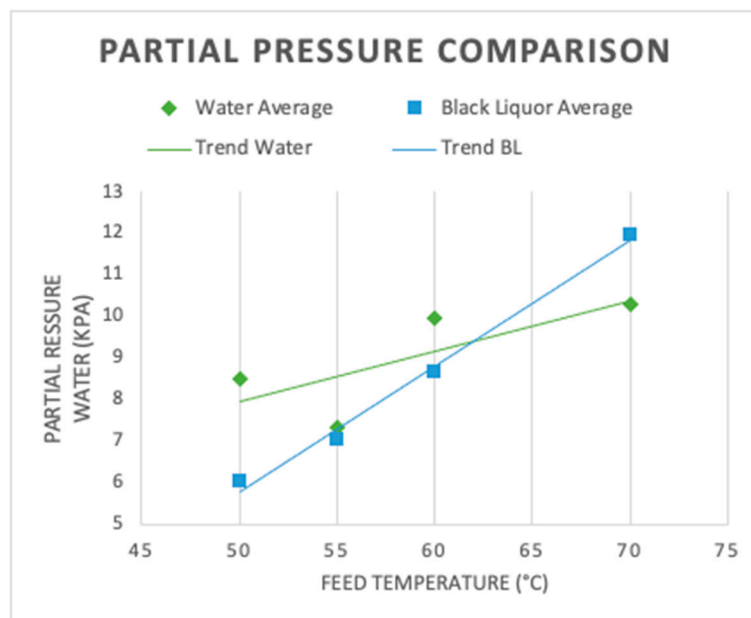
Figure 21: Average partial pressures for water and black liquor at different temperatures.

It appears that water shows a slightly higher partial pressure than black liquor. This is expected, as the dry substance of black liquor induces a boiling point elevation, or lower partial pressure than pure water. The data is not perfect, but the main trend of

a lower partial pressure for black liquor is noticeable. At 70°C, it is very likely that the temperature exceeded its setpoint during pilot unit runs on black liquor. This is important because it means that some runs could have been run on a higher temperature than intended, giving a misconception of a higher vapor condensation rate than what should be expected.

Solving Antoine's equation for temperature and with constants for water, is used to calculate a temperature difference between vapor pressures for black liquor versus water. Vapor pressure for was calculated for feed temperature after passing through the evaporative block. Difference in vapor pressures could then be translated to a difference in temperature, or boiling point elevation. In Figure 22, pilot unit tests are compiled and compared to vapor pressure for water runs, a boiling point elevation is derived. At 55°C, an average BPE of 0.8 °C is derived and as for 60°C resulted in an average BPE of 2.8°C. According to Table 2, a BPE of around 4°C is present at a dry substance black liquor of 26-34 w/w%. For process conditions at 55°C, pilot runs show a BPE of between -1 to 3°C. According to literature, that can be expected at a dry substance of 18-34 w/w% (Table 2). A test on dry substance for each individual run/data point has not been done.
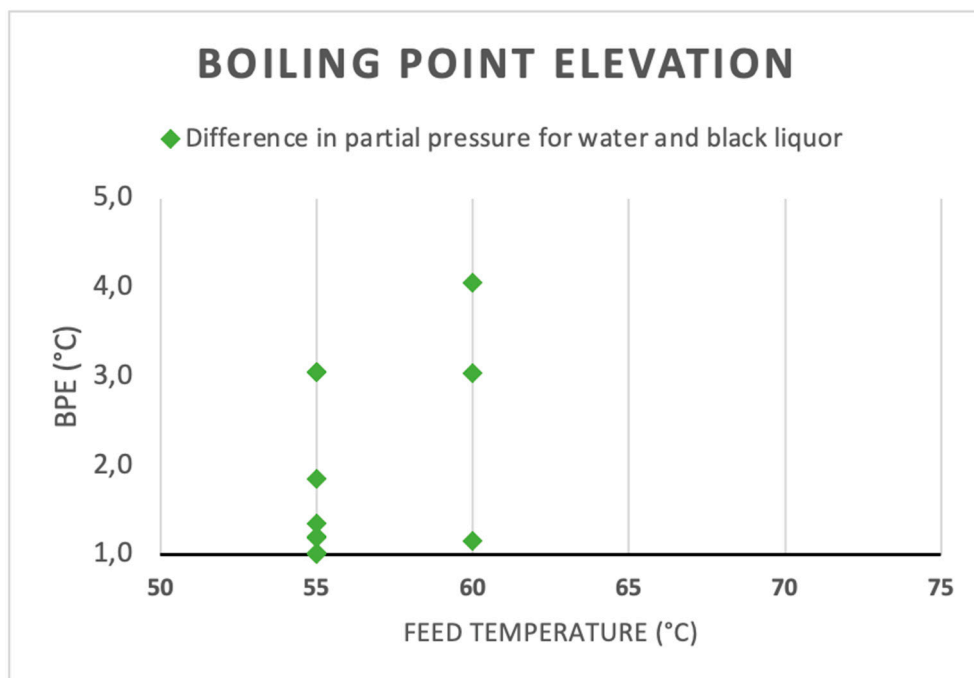
Figure 22: Estimated boiling point elevation compared to partial pressures of water. Calculated by Antoine's equation with constants for water. This figure reveals loss in driving force caused by a loss of vapor pressure in runs on black liquor compared to water.

The next graph (Figure 23) is a summary of experiments of how the pilot unit behaved when dry substance approached 50 w/w%. Results show unexpectedly high vapor condensation rates, indicating that something is off. Results completely contradicts Figure 20 above; vapor condensation rate should be at least 15% lower than that of water, not the other way around.

A possible explanation for false readings on sensors could have been foaming, a discussed above. Another possibility yet to be discussed would be that an air-channel was formed above the evaporative block where the temperature sensor was placed. That would decrease the temperature reading, thus falsely leading the experimental setup to operate at a higher temperature than intended. Unfortunately, this was noted after experimental sessions were finalized.
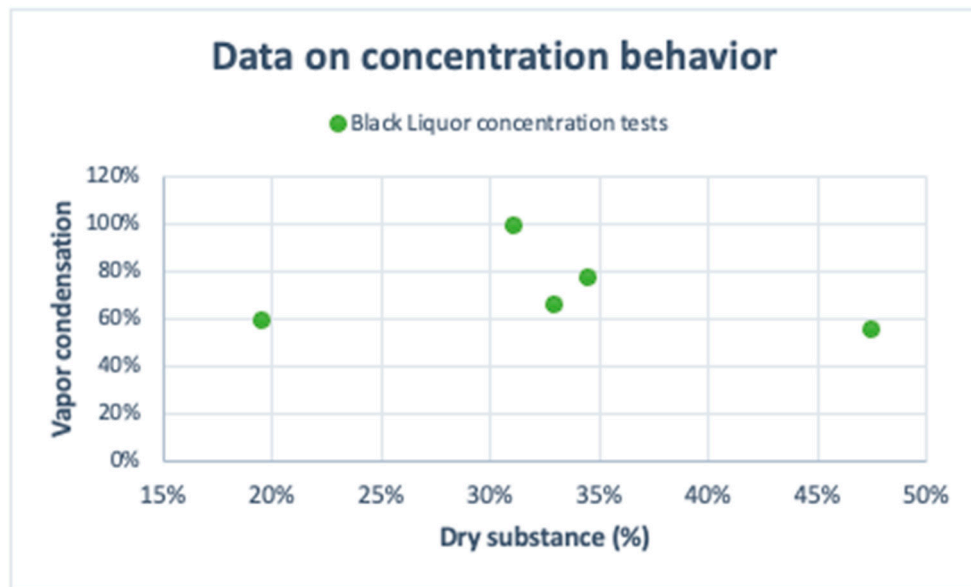
Figure 23: Vapor condensation of concentrated black liquor at higher dry substances at 55°C.

Disregarding the abnormally high vapor condensation rates, the test validate that the pilot unit has the potential to concentrate black liquor above 47% dry substance. More experiments are needed to satisfy quality and reliability on data. However, at an average rate of ≈65% of maximum condensation rate (Figure 20), the data suggests that the pilot unit continue to operate similarly to earlier experiments; assumed that the temperature did not increase too far out of specified range.

Figure 24: Distillate from pilot unit and black liquor at 19% and 47% dry substance from experimental runs.

Figure 24 shows a comparison of condensate from experimental runs on the left; slightly concentrated, weak black liquor at 19% in the middle; heavily concentrated black liquor to a dry substance of 47% on the right. The black liquor at 47% show qualitative increases in viscosity compared to the starting material at 17% dry substance. A look at the distillate versus the 47%, the condensate is drastically less stained by constituents present in the concentrated black liquor. This indicates a successful process where non-volatile species remains in the concentrate and do not escape into the condenser to any large extent.

6   Retrofit

This chapter assessed real cases from Swedish pulp and paper production sites. The first case reviewed the present situation at the pulp and paper production facility. The second case investigated the impact of a retrofit by Helios technology, supported by the technical validation. The facilities will be abbreviated Facility A, B and C.

6.1 Existing operations

In pulp and paper production plants, weak black liquor leaves the digester at approximately 120°C. The overheated stream enters a flash stage, where an initial amount of vapor is removed. Heat exchangers lower the temperature to approximately 90-95°C before going into a mixing tank, prior multiple effect evaporators (Table 8). The mixing tanks serve as a buffer to provide reliable and constant flow to the evaporators.

Table 8: Process data from respondents. Fields marked with '-' indicates no available data. Both heat and electricity consumption in unit kWh per condensed ton of vapor. "Excess Heat" is a side stream at 70°C that is a small fraction of the total 2600 m3/h of 55°C cooling water.

Facility	Facility A	Facility B	Facility C	Unit
Evaporator Feed	1080	930	720	m3 / h
Number of effects	8	7	7	Effects
Heat Consumption	120	120	120	kWh/ton
Electricity Consumption	-	32	90	kWh/ton
Temperature	93	95	90	°C
Feed to Coolers	2600	-	1000-2500	m3 / h
Temperature	55	50	48	°C
Excess Heat	600	-	-	m3 / h
Excess Heat Temperature	70	-	-	°C
Solid Fuels burnt	250	485	185	GWh / year

This data on existing operations is used as a reference to how much capacity can be increased and the extent of energy and electricity that can be saved by a Helios retrofit.

6.2 Facility with Retrofit

Leaving existing operations, Figure 25 is a design made as an end-of-pipe retrofit. Evaporator feed temperature is in range of 90-95°C (Table 8). Incoming thin liquor from mixing tanks is heat exchanged with the returning, concentrated stream. Since a fraction of water vapor is removed, consequently the returning stream is smaller than the ingoing and can transfer enough heat so it will not cool the mixing tanks to any large extent. On the opposite, incoming thin liquor to the retrofit unit will be cooled down to a temperature of close to 55°C so excess heat can be recovered (denoted waste heat in Figure 25), and heat exchanged to drive evaporation (Figure 25). There is, however, another possibility other than waste heat at 55°C. Occasionally at the Facility A facility they have excess heat at 70°C, but at a smaller volumetric flow of 600 m³ per hour.
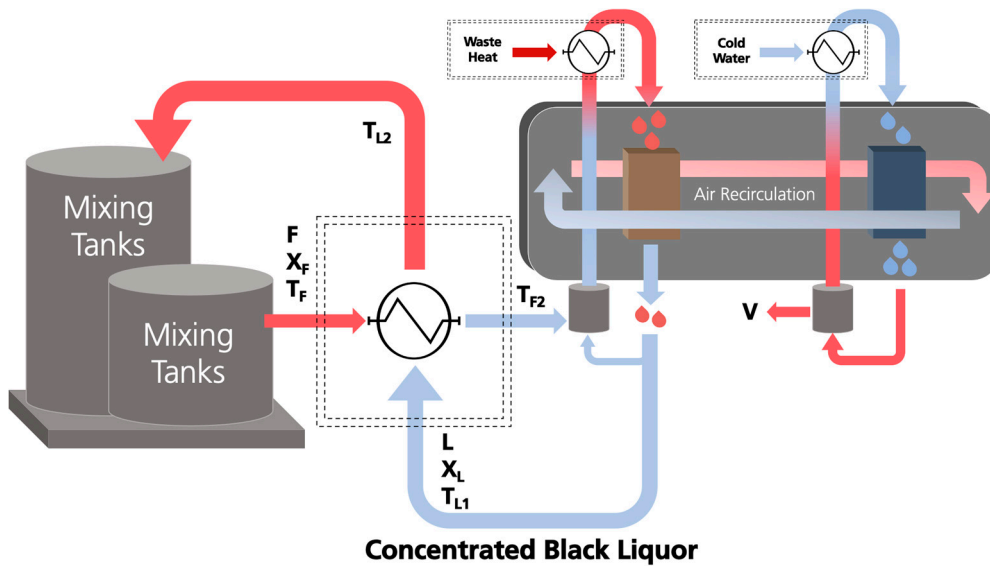
Figure 25. Proposed setup of Helios technology.

A retrofit before the mixing tank is non-desirable. The choice of implementing the Helios retrofit at this stage is beneficial in the sense that a retrofit can be added without risk of disrupting production.

6.2.1 Design one Facility A – 55°C

Available Heat: 2600 m³ per hour, or 722 Liters per second of cooling water at 55°C is available (denoted Waste heat in Figure 25). Assuming a temperature change in heat exchangers of (ΔT) 15°C from 55°C down to 40°C from incoming waste heat. According to Eq. (13), with a heat capacity for water at (C_p) 4180 J/kg K and a flow of (m) 722 Liters per second, this translates to an available effect of (Q) 45.3 MW.

$$Q = m * C_p * \Delta T \tag{13}$$

Heat Consumption: a retrofit unit consumes approximately 2500 kJ/kg evaporated liquid[6] and available energy is enough to evaporate 65 m³ of water per hour. By mass balance equations, available design configurations are listed in Table 9.

Capacity Calculations

Thin Liquor from Digester						
DS in %	17%	17%	17%	17%	17%	17%
Incoming Thin Liquor	1080	1080	1080	1080	1080	1080 m3/h
Amount water	896,4	896,4	896,4	896,4	896,4	896,4 m3/h
Dry solids	183,6	183,6	183,6	183,6	183,6	183,6 m3/h
Retrofit unit						
Thin Liquor In	**150**	**200**	**250**	**300**	**350**	**400** m3/h
Water in	124,5	166	207,5	249	290,5	332 m3/h
Dry solids in	25,5	34	42,5	51	59,5	68 m3/h
Evaporative Capacity	65,2	65,2	65,2	65,2	65,2	65,2 m3/h
Liquid Out	59,3	100,8	142,3	183,8	225,3	266,8 m3/h
Dry solids out	25,5	34	42,5	51	59,5	68 m3/h
DS out %	43%	34%	30%	28%	26%	25%
Total flow to mixing tanks						
Water	831,2	831,2	831,2	831,2	831,2	831,2 m3/h
Dry solids	183,6	183,6	183,6	183,6	183,6	183,6 m3/h
DS to mix tanks	22%	22%	22%	22%	22%	22%
Reduced amount of condensate for evaporator train						
Evaporated	65,2	65,2	65,2	65,2	65,2	65,2 m3/h
Saved effect	7825	7825	7825	7825	7825	7825 kW
Units in GWh	66,7	66,7	66,7	66,7	66,7	66,7 GWh/year
Summary						
Capacity	13,9%	18,5%	23,1%	27,8%	32,4%	37,0% into retrofit unit
Percent Evaporated	7,3%	7,3%	7,3%	7,3%	7,3%	7,3% of incoming water
Percent of total	6,0%	6,0%	6,0%	6,0%	6,0%	6,0% of total mass flow
Excess heat recovered						386 GWh per year
Reduced Heat demand in evaporator train						67 GWh per year

Heat supply 1: large heat exchangers need to be dimensioned to transfer the effect needed. In the retrofit, assuming a temperature change of 15°C, an overall heat transfer coefficient, k, of 1.7 kW/m^2 K and a transferred effect of 45.3 MW of energy results in a minimum transfer area of approximately 5160 m^2 of heat exchange surface. (Figure 25, heat exchanger for waste heat)

Heat Supply 2: after concentration in the retrofit unit, a return stream of black liquor is heated back to initial temperatures close to 90°C. The returning liquor has a smaller mass flow than the incoming liquor. The energy balance will be a net energy surplus in the incoming thin liquor, compared to the concentrated returning liquor. An assumption of a k-value of 2.2 kW/m^2 K sum up to a heat exchanger area of 310 m^2. (Figure 25, heat exchanger for returning black liquor)

Electricity savings: Helios retrofit unit has an approximate electricity consumption of 15 kWh per tonne condensed vapor.[6] If it is assumed that Facility A has similar consumption as Facility B at 32 kWh per tonne condensed vapor, resulting in a save of 17 kWh per tonne condensed vapor. In total a save of 9.4 GWh electricity per year. If instead Facility A would have an energy consumption like Facility C, a save of 75 kWh per tonne condensed vapor could be save, resulting in 41.7 GWh electricity per year.

Steam savings: Heat savings potentials estimated to 67 GWh of thermal energy per year. This is calculated by the amount of energy that would be needed to evaporate 65.2 m^3 per hour in the existing multiple effect evaporators. Given they consume 120 kWh per tonne of condensed vapor (Table 8).

Experimental results (Figure 18) show an average of 15-17% lower vapor condensation rate, compared to pure water. To achieve similar vapor condensation rates, the Helios unit would have to be increased in size by 15-17%, compared to previous water desalination units, to fulfill design specifications.

By knowledge acquired in the experimental section on the pilot unit, it is shown to be technically feasible to evaporate black liquor with low-temperature waste heat at 55°C. From the mixing tanks, tests showed a dry substance content of thin black liquor of 17 w/w%, on thin black Liquor received from Facility B chemical recovery. For these calculations it is assumed that the incoming thin black liquor therefore has a dry substance content of 17 w/w%.

The thin black liquor leaves the mixing tanks and enter evaporation effects at a dry substance content of 20-22 w/w%, depending on facility. To avoid disturbance in their existing operations, a DS as close to the present situation is desirable. By mass balance calculations Eq. (14) and Eq. (15) for a final concentration of 25-43 w/w%:

$$F = L + V \tag{14}$$
$$F * x_F = L * x_L \tag{15}$$

a capacity for a Helios unit of 27.8% of the total incoming stream translates to approximately 300 m^3 per hour. This design will not interfere with existing operations, as concentration when mixed with its origin will arrive at a dry substance content of 22 w/w%. To summarize, the design would increase the incoming thin liquor concentration by only a small fraction.

6.2.2 Design Two Facility A – 70°C

Capacity: Compared to design one, this design has an available waste heat of only 600 m^3 per hour but at a higher temperature. Available heat is calculated with same constants and temperature change as in Design one, according to Eq. (13) and results in available effect of 14 MW of heat. This is enough to evaporate approximately 20.1 m^3 of water per hour. If design capacity is set to 100 m^3 per hour of incoming thin liquor, an output of 27 w/w% dry substance is achieved, as seen in Table 10.

Table 10: Mass balance analysis for available heat at 70°C.

Capacity Calculations

Thin Liquor from Digester						
DS in %	17%	17%	17%	17%	17%	17%
Incoming Thin Liquor	1080	1080	1080	1080	1080	1080 m3/h
Amount water	896,4	896,4	896,4	896,4	896,4	896,4 m3/h
Dry solids	183,6	183,6	183,6	183,6	183,6	183,6 m3/h
Retrofit unit						
Thin Liquor In	**75**	**100**	**150**	**200**	**250**	**300** m3/h
Water in	62,25	83	124,5	166	207,5	249 m3/h
Dry solids in	12,75	17	25,5	34	42,5	51 m3/h
Evaporative Capacity	20,1	20,1	20,1	20,1	20,1	20,1 m3/h
Liquid Out	42,2	62,9	104,4	145,9	187,4	228,9 m3/h
Dry solids out	12,75	17	25,5	34	42,5	51 m3/h
DS out %	30%	27%	24%	23%	23%	22%
Total flow to mixing tanks						
Water	876,3	876,3	876,3	876,3	876,3	876,3 m3/h
Dry solids	183,6	183,6	183,6	183,6	183,6	183,6 m3/h
DS to mix tanks	21%	21%	21%	21%	21%	21%
Reduced amount of condensate for evaporator train						
Evaporated	20,1	20,1	20,1	20,1	20,1	20,1 m3/h
Saved effect	2408	2408	2408	2408	2408	2408 kW
Units in GWh	20,5	20,5	20,5	20,5	20,5	20,5 GWh/year
Summary						
Capacity	6,9%	9,3%	13,9%	18,5%	23,1%	27,8% into retrofit unit
Percent Evaporated	2,2%	2,2%	2,2%	2,2%	2,2%	2,2% of incoming water
Percent of total	1,9%	1,9%	1,9%	1,9%	1,9%	1,9% of total mass flow
Excess heat recovered						119 GWh per year
Reduced Heat demand in evaporator train						21 GWh per year

Heat supply 1: large heat exchangers need to be dimensioned to transfer the effect needed. In the retrofit, assuming a temperature change of 20°C, an overall heat transfer coefficient, k, of 1.7 kW/m^2 K and a transferred effect of 14 MW of energy results in a minimum transfer area of approximately 1440 m^2 of heat exchange surface.

Heat Supply 2: after concentration in the retrofit unit, a return stream of black liquor is heated back to initial temperatures close to 93°C. The returning liquor has a smaller mass flow than the incoming liquor. An assumption of a k-value of 2.2 kW/m^2 K sum up to a heat exchanger area of 120 m^2. Since less water is evaporated compared to design one, the returning thin liquor need to be heated with an effect of 1.6 MW to return to desired temperature in the mixing tanks. This would lower the thermal save by 13.6 GWh per year in total from 119 GWh per year, to approximately 105 GWh per year.

Steam savings: heat must be supplied to heat returning black liquor, steam saved by this design has the potential of 6.9 GWh per year.

Electricity savings: calculations show a potential of up to 12.8 GWh of electricity, if Facility A has the same electricity consumption as Facility C.

7   Discussion

Up until now, cooling towers or heat exchange with nearby water bodies have been the way to get rid of large quantities of heat. Initially, research focus was to determine whether there would be any waste heat streams of interest available. It was unclear how Helios equipment would react on relevant process conditions, especially with black liquor at elevated temperatures. In this research, it has been shown that large quantities of waste heat are available at vast quantities. Experimental findings suggest that for the first time ever, black liquor can be evaporated using temperatures considered waste heat. Pulp and paper producers can drastically decrease both their energy and electricity consumption in evaporators. The available space in evaporators could also be used to increase production for facilities where evaporators have served as a bottleneck.

Experimental findings suggest that more energy can be recovered than previously thought. The importance of this, is a big step closer to achieving the sustainability goals set in Agenda 2030, by reaching a net zero emission by 2045.

7.1 Technical validation

Outcomes of experimental work has been mostly positive. Initially, tests on material resistance of cellulose packing material showed greater resistance than what could have been expected. The material retained most of its load capacity during the timeframe of laboratory tests. Furthermore, flutes and channels remained intact, but resin between flutes was dissolved within 48 hours, at elevated temperatures. When packing material was exposed to external forces, stress, from in situ testing its performance was not significantly worsened to any extent that was noticeable.

Above 80°C, packing material remained completely intact with only dark stains because of thin black liquor exposure (Figure 16). Flutes continued to remain intact and performed surprisingly well. During testing, no visible fouling on packing materials or blockage of liquid nozzles were noted at any time of testing.

For performance tests, increasing temperature resulted in increased vapor condensation rates as the partial pressure of water rose accordingly. For these tests the air velocity had no noticeable impact on condensation rate. It is possible, that the mass transfer of vapor was already satisfied at low air velocities. It could also be that unnecessarily high velocities do not disturb the mass and heat transfer significantly in this "small" pilot unit. Since the validation has only taken place in a small scale, care should be taken into directly translating it into large scale operability. Tests in large scale are desirable and should be subject to future research.

Potential sources of errors include false readings, incorrect mass flows of liquid, fan speed and thermal energy losses both through radiation and evaporation. The system is not thermally insulated and completely made from stainless steel, with exceptions for connections and pipes. Steel has a very high conductivity, and a lot of heat is possibly lost through radiation and conduction to its surroundings. This will influence temperature changes on evaporation block, as some heat is lost and not used in the process of evaporation.

Validation in this thesis have shown general trends in a pilot scale, with promising results on the technical and economic feasibility of Helios evaporation technology in the pulp- and paper industry by evaporation of black liquor by excess heat recovery.

7.2 Implementation

To penetrate pulp and paper industry with new, energy efficient technologies the design of an implementation need to consider these bullet points. Assessing these risks, the retrofit design will not impose major, systemic changes to existing production processes. The potential benefits of an implementation of a retrofit are listed below:

- No risk of production disruptions.
- Induces cost reductions by lower energy usage.
- Payback time for these types of investments is on average 2 years.
- Energy improvements are needed to cope with stricter regulations.
- More renewable electricity can be sold and rewarded with ECS certificates.

Regarding regulatory frameworks, CO_2 taxes are expected to continue rising and the cost per MWh biofuel is also expected to increase as the world demand more renewably sourced fuels.

In the field of energy efficient technology, this novel evaporation method is unmatched. No other studies have been able to technically validate evaporation of black liquor using waste heat to the extent that this thesis have shown. The risk and potential need to be further researched but initial results show great potential, as Helios technology is verified in an industrial environment.[6]

Traditional technology includes steam flash evaporators and multiple effect falling film evaporators. A flash evaporator can only vaporize a small fraction of incoming steam without depressurizing the vessel, thus increasing electricity consumption. Multiple effect evaporators are the best of its kind when it comes to evaporation of black liquor and can reach dry solids content that most other technologies cannot. However, they are restricted by a fresh steam supply and that serves as their bottleneck. This retrofit would serve as either a capacity increasing step or energy reducing measure by exploiting excess heat, traditionally removed by cooling towers or by supply from nearby water bodies.

7.3 Impact

The retrofit, design one, show potential performance of a capacity increase of 6%. The made available space in existing evaporators can either increase production of pulp, or steam can be sold externally and to mitigate CO_2 emissions elsewhere. A net increase of sold steam by 67 GWh can be achieved.

Furthermore, on the topic of savings. Price data in energy district 3, in the last ten years (2011-2021) lands in an average 35.3 öre per kWh electricity (1 öre = 0.01 SEK).[36] Electricity savings has a potential of up to 42 GWh per year, resulting in a save of up to 14.7 MSEK per year. An average emission factor for Nordic electricity generation is estimated to 90 g CO_{2e} per kWh, according to the Swedish Environmental Institute, on behalf of Naturvårdsverket.[37] Thus a potential of 3,700 tons of CO_{2e} from electricity generation can be reduced.

Carbon dioxide net emissions from Naturvårdsverket, is assumed to 345.6 kg CO_2 per MWh burnt biofuel (black liquor, biomass and wooden fuels). A total saving of 66.7 GWh thermal energy has the potential of reducing total direct emissions by 23,000 tons of CO_2 at per year.

In total a mitigation of almost 26,700 tons of CO_2 can be achieved by this design.

8 Conclusion

More research is needed to validate performance in a larger industrial setting, but experimental results show great potential for further development. The main findings and learnings from this study are compiled below.

Technical Validation:

- Technical validation shows potential for excess heat recovery to evaporate black liquor with Helios retrofit equipment.
- On average 15-17% less capacity compared to water can be expected.
- No visible fouling on packing materials or blockage of liquid nozzles.
- Managed boiling point elevation at dry substances and temperature intervals.
- Cellulose can in laboratory environments withstand degradation for extended exposure at elevated temperatures.
- Cellulose blocks withstand black liquor at relevant process conditions.
- A longer timeframe would be interesting to research further. Material withstood range tested in this report. It is uncertain how longer exposure times would affect the cellulose material.
- Results in a small scale should be viewed with caution and not be translated directly into full scale operability. Large scale research is desirable and would be interesting to research further.

Implementation:

- Barriers of implementing energy efficient technology are addressed.
- Retrofit unit will not interfere with existing production.
- Induces cost reductions by lower energy usage.

Impact:

- Excess heat is available in the pulp and paper industry.
- Helios unit can technically recover heat that is presently wasted.
- Excess heat recovery of 386 GWh of thermal energy per year.
- Steam saving potential of 67 GWh per year.
- Potential of 42 GWh per year of electricity.
- Electricity market value up to 14.7 MSEK per year.
- Maximum capacity increase is estimated to 6%.

Climate action and carbon dioxide reducing measures:

- Carbon dioxide savings is approximated to 23,000 tons per year.
- Electricity savings result in potential CO_{2e} reduction of 3,700 tons per year.

Concluding remarks

This design has the potential to save 67 GWh thermal and 42 GWh electric energy.
Increases capacity of evaporators by up to 6%.
Up to 26,700 tons of CO_2 can be saved every year, per facility in size A.

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10 Appendices

Appendix A: Test Schedule for water runs on Helios pilot unit.

Parameter	Fan Speed % of MAX	Hot Feedstock IN [°C]	Hot Feedstock OUT [°C]	Hot Feedstock dT Hot [°C]	Hot Feedstock Mass Flow	Cooling water IN [°C]	Cooling water OUT [°C]	Cooling water dT Cold [°C]	Cooling water Mass flow	Dry Temperature T1 [°C]	Dry Temperature T2 [°C]	Wet Temperature Tw1 [°C]	Wet Temperature Tw2 [°C]	Water content X1	Water content X2	Condensed Mass per time
Process Temp Varies	25%	50			63%	15			63%							
Process Temp Varies	25%	60			63%	15			63%							
Process Temp Varies	25%	70			63%	15			63%							
Process Temp Varies	25%	80			63%	15			63%							
Process Temp Varies	50%	50			63%	15			63%							
Process Temp Varies	50%	60			63%	15			63%							
Process Temp Varies	50%	70			63%	15			63%							
Process Temp Varies	50%	80			63%	15			63%							
Process Temp Varies	75%	50			63%	15			63%							
Process Temp Varies	75%	60			63%	15			63%							
Process Temp Varies	75%	70			63%	15			63%							
Process Temp Varies	75%	80			63%	15			63%							
Process mass Flow Varies	50%	80			63%	15			63%							
Process mass Flow Varies	50%	80			50%	15			63%							
Process mass Flow Varies	50%	80			38%	15			63%							
Process mass Flow Varies	50%	80			25%	15			63%							
Process mass Flow Varies	50%	80			15%	15			63%							
Process mass Flow Varies	25%	80			85%	15			63%							
Process mass Flow Varies	25%	80			63%	15			63%							
Process mass Flow Varies	25%	80			50%	15			63%							
Process mass Flow Varies	25%	80			38%	15			63%							
Process mass Flow Varies	25%	80			25%	15			63%							
Process mass Flow Varies	25%	80			15%	15			63%							
Process Temp Varies	25%	80			100%	15			63%							
Process mass Flow Varies	75%	80			85%	15			63%							
Process mass Flow Varies	75%	80			63%	15			63%							
Process mass Flow Varies	75%	80			50%	15			63%							
Process mass Flow Varies	75%	80			38%	15			63%							
Process mass Flow Varies	75%	80			25%	15			63%							
Process mass Flow Varies	75%	80			15%	15			63%							
Process Temp Varies	50%	80			100%	15			63%							
Process Temp Varies	50%	80			85%	15			63%							
Process mass Flow Varies	25%	55			15%	15			63%							
Process mass Flow Varies	25%	55			25%	15			63%							
Process mass Flow Varies	25%	55			38%	15			63%							
Process mass Flow Varies	25%	55			50%	15			63%							
Process mass Flow Varies	50%	55			15%	15			63%							
Process mass Flow Varies	50%	55			25%	15			63%							
Process mass Flow Varies	50%	55			38%	15			63%							
Process mass Flow Varies	50%	55			50%	15			63%							
Process mass Flow Varies	75%	55			15%	15			63%							
Process mass Flow Varies	75%	55			38%	15			63%							
Process mass Flow Varies	75%	55			50%	15			63%							
Process mass Flow Varies	75%	55			63%	15			63%							

Appendix B: Questions to pulp and paper manufacturers, in Swedish.

Fråga	Namn	Enhet
Indunstare		
Antal effekter indunstare		stycken
Elförbrukning Indunstare		kWh / ton kondensat
Värmeförbrukning indunstare		kWh / ton kondensat
Torrsubstans in (tunnlut från kokeri)		%
Torrsubstans blandlut (till indunstning)		%
Torrsubstans ut indunstare		%
Kapacitetsbrist indunstare		[Ja / Nej]
Mix Cistern		
Temperatur blandlut cistern		°C
Kylning		
Flöde tunnlut till indunstare		m3 / h
Vätskeflöde till kyltorn/kylare		m3 / h
Temperatur vätskeflöde till kyltorn/kylare		°C
Energi		
Internvärde Ånga		Öre/kWh
Internvärde El		Öre/kWh
Fastbränsleförbränning per år		GWh / år

Appendix C: Response from pulp and paper manufacturers, in Swedish. Responding company was the Södra Group.

Fråga	Södra A	Södra B	Södra C	Enhet
Indunstare				
Antal effekter indunstare	8	7	7	stycken
Elförbrukning Indunstare	-	32	90	kWh / ton kondensat
Värmeförbrukning indunstare	120	120	120	kWh / ton kondensat
Torrsubstans in (tunnlut från kokeri)	15-18	16	13-17	%
Torrsubstans blandlut (till indunstning)	20	22	21	%
Torrsubstans ut indunstare	82	80	80	% (inkl. recirkulerad aska, ca. 7% av TS)
Kapacitetsbrist indunstare	Nej	Nej	Ja (i perioder)	[Ja / Nej]
Mix Cistern				
Temperatur blandlut cistern	93	95	90	°C
Kylning				
Vätskeflöde till kyltorn/kylare	600	-	1000-2500	m3 / h
Flöde tunnlut till indunstare	1080	930	720	m3 / h
Temperatur vätskeflöde till kyltorn/kylare	70	50	48	°C
Energi				
Internvärde Ånga	Kondensturbin	Kondensturbin	-	Öre/kWh
Internvärde El	-	-	-	Öre/kWh
Fastbränsleförbränning per år	250	485	185	GWh / år