



Sizing the auxiliary system capacities for a pilot plant project

Juho Leinonen

Bachelor's thesis April 2020 Technology Bachelor of engineering, Energy and Environmental Technology

Jyväskylän ammattikorkeakoulu JAMK University of Applied Sciences

jamk.fi

Description

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Author	Type of publication	Date	
Leinonen, Juno	Bachelor's thesis	April 2020	
		Language of publication: English	
	Number of pages	Permission for web publi-	
	62	cation: x	
Title of publication Sizing the auxiliary system capaciti	es for a pilot plant project		
Degree programme Degree programme in Energy and E	nvironmental Technology		
Supervisor(s) Honkanen, Hannariina & Hytönen, H	Kari		
Assigned by Stora Enso Oy			
Abstract			
scale plant where lignin can be processed. The aim of processing lignin is to improve its properties so that it can replace fossil-based materials in the future. The thesis focused on the design of auxiliary systems. The main objectives of the thesis were to determine the requirements for the nitrogen supply system, the exhaust gas treatment system, and the cooling water system and to select suitable equipment based on these requirements. The study was done by gathering material from heat treatment furnaces which are the main objectives of the furnaces provided the main requirements of the process.			
quirements for the auxiliary systems. The material was analyzed and the requirements from each furnace were compared with each another so that uniform requirements could be made for the systems. Once the requirements were set, literature, studies, and the knowledge of the auxiliary system suppliers were utilized to select the equipment for the systems.			
A generator with PSA-technology was chosen for the nitrogen supply system because it was found to be the most cost-efficient solution that met the requirements. An oil-free screw-compressor was selected as a compressor for the system. The system is equipped with the necessary vessels and cleaning equipment. For the exhaust gas treatment system, the solution included two separate combustion chambers. The main equipment manufacturers design and supply both combustion chambers. The cooling water system was designed to have a closed loop. The closed loop requires a heat exchanger with 200kW cooling power to keep the water temperature inside the loop stable. The scarcity and secrecy of the data, as well as the lack of references, affected the sizing accuracy of the systems. These are normal problems for a pilot-project. However, most of the results are reliable and these results can be utilized in the process design.			
Keywords/tags (<u>subjects</u>) Pilot plant, designing, nitrogen, coo	ling water, lignin		

Miscellaneous (<u>Confidential information</u>) Appendixes 1, 2, 3, and 4 are confidential and they have been removed from the public thesis. Grounds for secrecy: Act on the Openness of Government Activities 621/1999, Section 24, 17: business or professional secret. Period of secrecy is ten years and it ends 1.6.2030.



Kuvailulehti

Cekijä(t)	lulkaisun laii	Päivämäärä
	Julkaisuli laji	Falvallaala
_einonen, Juho	Opinnäytetyö, AMK	Huhtikuu 2020
	Sivumäärä	Julkaisun kieli
	62	Englanti
		Verkkojulkaisulupa
		myönnetty: x
[vön nimi		

Käyttöhyödykejärjestelmien kapasiteettien mitoitus

Tutkinto-ohjelma

Energia- ja ympäristötekniikka

Työn ohjaaja(t)

Honkanen, Hannariina & Hytönen, Kari

Toimeksiantaja(t)

Stora Enso Oy

Tiivistelmä

Opinnäytetyö on osa Stora Enso Oy:n projektia, jonka tarkoituksena on rakentaa Sunilan tehdasalueelle pilotmittakaavan tehdas ligniinin jatkojalostusta varten. Jatkojalostuksen tarkoituksena on parantaa ligniinin ominaisuuksia, niin että se voisi korvata fossiilipohjaisia materiaaleja tulevaisuudessa. Opinnäytetyö keskittyi käyttöhyödykejärjestelmien suunnittelun ympärille. Tarkoituksena oli selvittää jäähdytysvesijärjestelmän, typpijärjestelmän ja savukaasujenkäsittelyjärjestelmän vaatimukset ja niiden perusteella mitoittaa järjestelmät.

Työ toteutettiin keräämällä aineistoa päälaitteina toimivista lämpökäsittelyuuneista. Nämä laitteet määrittelevät tärkeimmät vaatimukset apujärjestelmille. Kerättyä ainestoa analysoitiin ja erilaitteiden vaatimuksia vertailtiin, jotta voitiin määritellä järjestelmälle yhtenäiset vaatimukset. Vaatimusten määrittelyn jälkeen sopivan ratkaisun valinnassa hyödynnettiin kirjallisuutta, tutkimustietoa ja apujärjestelmätoimittajien tietotaitoa.

Typpijärjestelmäksi valittiin PSA-teknologialla toimiva generaattori, koska se oli kustannustehokkain järjestelmä, joka pystyi täyttämään vaatimukset. Kompressoriksi järjestelmälle valittiin öljytönruuvikompressori. Järjestelmä varustettiin tarvittavilla säiliöllä ja puhdistuslaitteilla. Savukaasujen käsittelyjärjestelmän osalta päädyttiin ratkaisuun, joka sisälsi kaksi erillistä polttokammiota. Päälaitevalmistajat suunnittelevat ja toimittavat molemmat polttokammiot. Jäähdytysvesijärjestelmäksi suunniteltiin suljetuksi kierroksi. Suljettukierto vaatii lämmönvaihtimen, jäähdytysteholtaan n.200kW, jotta nesteen lämpötila pysyy tasaisena.

Järjestelmien mitoituksen tarkkuuteen vaikuttivat lähtötietojen vähäisyys ja salassapitoasiat sekä referenssien puute. Nämä ovat tyypillisiä ongelmia pilotprojekteille. Suurin osa saaduista tuloksista on luotettavia ja niitä voidaan hyödyntää myöhemmin prosessisuunnittelussa.

Avainsanat (asiasanat)

Typpi, Jäähdytysvesijärjestelmä, ligniini, pilotlaitos, suunnittelu

Muut tiedot (salassa pidettävät liitteet)

Liitteet 1, 2, 3 ja 4 ovat salassa pidettäviä, ja ne on poistettu julkisesta työstä. Salassapidon perusteena on viranomaisten toiminnan julkisuudesta annetun lain (621/1999) 24 §:n kohta 17: yrityksen liike- tai ammattisalaisuus. Salassapitoaika on kymmenen (10) vuotta. Salassapito päättyy 1.6.2030.

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1 Introduction

Climate chance has recently been one of the biggest topics of discussion. Companies around the world are trying to invent with new ways to get rid of fossil fuels and fossil-based materials. Energy storages play a major role in the transition from fossil fuels to renewable sources. The global energy storage market is predicted to grow from 59 billion dollars to 546 billion dollars by year 2035 (Global Energy Storage Market 2019). According to a research by Lux the demand for energy storage, especially for mobility applications, will grow rapidly in the future. Mobility applications include fuel cell electric vehicles and battery electric vehicles. Overall, it is expected that stationary storage Market 2019.) The anode of lithium batteries are traditionally made of graphite, which is a fossil material (Anode Materials. n.d.). Stora Enso, who is the assignor of the thesis, answers to this problem with a new processing technology of lignin. Processed lignin could replace fossil-based material in rechargeable batteries in the future. (Tillaeus 2020.) The thesis is part of a project to build a pilot-scale factory where this technology will be utilized first time in the world.

Stora Enso is a global forest industry company. Stora Enso was listed as the world's fifth largest company in the forest, paper, and packaging industries in 2015 (Global Forest, Paper & Packaging Industry Survey 2016). Stora Enso has divided its operations into six divisions: Biomaterials, Forest, Packaging materials, Packaging solutions, Paper, and Wood products. The six different divisions have a total of 26,000 employeees. The biomaterials division is responsible for pulp production and the utilization of biomass by-products. (Stora Enso in 2019.) The research department of the biomaterials division has studied the further processing of lignin. They have found promising results from research and decided to invest 10 million euros in pilot-scale plant in 2019. Designing and construction works will last until of the end of the year 2020. The pilot-plan is scheduled to start running in early 2021. (Kykkänen 2019.)

The pilot plant will be built in Sunila mill area. Sunila mill, located in Kotka on the shores of the Baltic Sea, is part of Stora Enso's biomaterial division and it produces

softwood pulp, lignin, tall oil, and turpentine. Sunila mill is founded in 1938 and it produces 375 000 tons of pulp per a year. Comparing the producing capacities, Sunila mill is one of the smallest pulp mills in Finland. On the other hand, Sunila mill is one of the biggest lignin producers in the world and Stora Enso's only pulp mill that produces lignin. The mill produces 50 000 tons of lignin per a year (Sunila mill. n.d.)

As mentioned, designing the pilot plant was underway. Most of the main components had already been bought and the rest of the main components were to be bought during spring 2020. The main components are heat-treatment furnaces where lignin will be processed. (Tillaeus 2020). These heat-treatment furnaces need different types of auxiliary systems. The furnaces consume nitrogen gas to create an oxygen-free atmosphere and because lignin is heated in the furnaces, it must be cooled afterwards, so cooling water system is needed. Heating of lignin generates exhaust gases, that need an exhaust gas treatment system which ensures gases are controlled properly.

The thesis focused on these auxiliary systems. The main objective was to investigate and calculate the capacity required and based on the capacities and other requirements select the main devices for the auxiliary systems. For the nitrogen system, the object was determining the total demand of nitrogen gas and the other requirements for the system. Secondary objective was designing how to ensure the supply of nitrogen during power outages. The supply of nitrogen is critical for the process and the nitrogen flow must be continuous to ensure the safety of the process. The main question for the cooling system was how much cooling power is needed as total. Once cooling power was calculated the objective was to determinate other requirements for the cooling water system. The safety of the process also had to be considered in the design of the cooling system. Thus, the secondary question was how to ensure the sufficient availability of cooling water in all situations. Calculating how much each process step generates exhaust gas was the main objective for an exhaust gas system. The equipment choices for the exhaust gas system were to be done based on result of the calculations.

2 Research methods and material

The material used in the thesis was the information available on the furnaces. The whole project was in the early stages, so a different amount of information was available for each furnace. Because some furnaces had just been bought, there was no further information available and the technical information of the offers were the only source that could be utilized as material. The first versions of the offers mainly contained information about the furnace itself. They defined the most important information for the process, the key measurements of the equipment, and delivery limits. There was little information for the auxiliary system designing. Nitrogen consumption maximum and minimum values were given in the offers. But there was no explanation for when the furnaces consume the most nitrogen. Only one manufacturer reported average consumption and instantaneous maximum consumption separately. The same issue was with cooling water consumptions. The offers clarified the maximum and minimum consumptions but not the situation in which the oven consumes the most. There was even less information about the exhaust gases in the offers. Amount of the gas depends on the properties of the input material, the feed rate and heating rates. Therefore, it is hard to provide exact values. Manufacturers made several different versions of the offers. The offers were amended and updated based on the issues noted during the negotiations.

Once Stora Enso accepted the offer, the manufacturers had a deadline to provide more detailed documentation of the equipment. Most of the offers had been accepted when the thesis process began. But for the most equipment, the more detailed documents were yet to be provided. The detailed documents included more detailed drawings, connection data lists, and overall, more detailed information about the equipment. These documents were very useful and gave much more information that could be utilized. However, even these documents did not provide complete information for the designing of the auxiliary systems. Some further information was later requested by email. In spite of all this, some assumptions had to be made and the analysis of the material had to be done carefully. The maximum consumptions were used in the most cases if there was not further information. Because the furnaces are pilot-scale equipment even the maximum consumptions are not so high. Therefore, basing the design of auxiliary systems on the maximum values did not affect so much. This also ensured that the auxiliary systems would not be a bottleneck in the process. Because the offers or the technical documents did not provide information on the amount of exhaust gas generated Stora Enso's research department made its own laboratory test. The results of the tests were utilized in the design of the exhaust system. Based on the analysis of the material, the intention was to determine the requirements for the systems.

The theoretical part describes the properties of substances, what they are commonly used for, what systems are commonly used, and how they work. The results of the material analysis are compared with the theoretical knowledge and based on this the decisions on the systems are suitable for this project were made. The knowledge of the suppliers was also utilized in decision making because they have a deep knowledge of their products and when the requirements for the equipment are clearly stated, they can easily tell the suitability of the equipment for the project. Once materials were analyzed Stora Enso, as the customer, had idea what kind of equipment would be suitable. If, based on the information given to them, the supplier offered us a solution that we did not expect, we negotiated why we would need that kind of equipment. The supplier could know better what their equipment is capable of and what would be the workable solutions. Because deals had been made with suppliers in the past and were known to be reliable, their suggestions were listened to and utilized in the design process.

The analysis of material includes both comparing different values and calculations and collecting other kinds of information about the requirements and comparing them with each other. Thus, both quantitative and qualitative methods were utilized in the thesis. The project was progressing all the time and more information was available during the project, but there was a deadline for completing the thesis, the thesis is partly based on limited information and the results are displayed as they were at the stage of completing the thesis work.

3 Lignin and thermal processing

Nowadays 98% of produced lignin is used for energy production, as most of the lignin is produced as a by-product of the pulp industry. As an aromatic polymer, lignin has high potential to be much more than just a renewable fuel. It is the complicated structure, heterogeneity, and the industrial processing costs limit its use. There have been several researches on lignin utilization over the last decade. (Faruk & Sain 2015, 52.) Lignin is found a suitable copolymer, polymer additive, and a filler material in thermoplastics. Lignin is also added to concrete to improve its properties and used as binding agent in animal food. (Wool & Sun 2011, 1038.) Recent studies have shown that lignin could be modified to carbon fibers or carbon nanofibers and it could replace fossil-based materials in many applications. Carbon fibers can be made of different precursors, polyacrylonitrile (PAN) being currently the most popular option. Polyacrylonitrile is heat treated at several process steps to produce carbon fibers. Lignin-based carbon fibers can be produced with the same technology. (Faruk & Sain 2015, 488.)

3.1 Lignin

Lignin is one of the three major macromolecular components of wood. The other two are hemicellulose and cellulose. All woody species contain lignin and its content ranges from 15% to 40% depending on the species. Thus, lignin is a very abundant natural polymer. Typically, softwoods have higher lignin content than hardwood. Lignin was discovered in 1838 by the French scientist Payen. The name of lignin comes from Latin word lignum, which means wood. (Faruk & Sain 2015, 33.)

Lignin provides mechanical support for a plant in the cell walls. The rigidity and stiffness of plants comes from lignin. It also protects the woody tissue from microorganisms and insects. Lignin has a complex chemical structure and its exact structure cannot be terminated. It is descripted to be a complex three-dimensional polymer. Lignin contains three different monolignols: coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol. There are various types of lignin and their structure depends on the source of lignin and the processing method. (Faruk & Sain 2015, 35.)

The primary source of lignin is wood because of its high lignin content and lignin from the wood is extracted during the pulping process. Other potential lignin sources are sugarcane bagasse and agricultural residues. Sugarcane bagasse are residues of sugar production and it is currently used as a low-grade fuel. Sugarcane production generates over 54 million tons of bagasse annually, so it is a potential lignin source even though its lignin content is 6-12% which is much lower than in wood. Agriculture generates large amounts of residues which are also a potential source of lignin. For example, rice straw has a lignin content around 15-28% and its annual production is 525 million tons. (Faruk & Sain 2015, 44-51.)

As mentioned, lignin is unwanted in the paper making process and it is extracted from wood fibers during the pulping process. Nowadays kraft pulping (a.k.a. sulfate pulping) is the most used pulping method in the pulping industry. Lignin extracted from the kraft pulp process is known as a kraft lignin. Kraft pulping is a chemical method where wood is treated with sodium hydroxide and sodium sulfide to separate cellulose from lignin. In a normal kraft-pulp process, lignin is mixed in the black liquor which is burned in a recovery boiler to recover inorganic components. (Kuinka sellua valmistetaan n.d. [How to make pulp]) Burning black liquor provides more energy than the total consumption of the mill is. (Faruk & Sain 2015, 488.)

Extracting lignin from black liquor has been raising recently because the technologies have developed. The mills can raise their pulping capacity without meeting the recovery boiler limits by removing lignin from the black liquor. The easiest way to utilize the produced lignin is to use it as a fuel for lime kilns. Traditionally natural gas has been used as fuel in lime kilns. Changing the fuel to lignin-based will reduce the CO₂ emissions of the mill. Sunila mill uses Valmet's extracting technology called LignoBoost. In LignoBoost, part of black liquor is taken from an evaporation plant and it is precipitated with CO₂ and then dewatered, washed, and dewatered again. The produced lignin is dry powder. (LignoBoost process n.d.)

3.2 Thermal treatment process

Producing carbon fibers from lignin can be considered one the most economically attractive applications of lignin. Fibers that contain over 90% carbon are called carbon fibers and fibers with over 99% carbon content are called graphite fibers. Carbon fiber is a high-performance product and it is used in applications when the properties of material are more important than the price. Carbon fiber products are expensive because of the high price of polyacrylonitrile which is commonly used as a precursor for carbon fibers. Polyacrylonitrile is fossil-based and therefore its availability is limited. Lignin has a natural carbon content of around 60% and it is less expensive than polyacrylonitrile. Moreover, have shown that processing lignin fibers to carbon fibers is faster than the traditional process. 50% of the price of carbon fiber comes from a precursor and 50% from processing. Thus, lignin is an attractive option because it is an inexpensive precursor, its availability is high, and it needs less processing. (Faruk & Sain 2015, 490.)

Producing carbon fibers from purified lignin includes three major process steps: spinning, thermal stabilization, and carbonization in this order. Lignin must be pure to produce high quality carbon fibers and lignin that has less heterogeneity is also better for processing. The idea in spinning is to control the tenacity, diameter, and morphology of the precursor fibers. Spinning can be done by melt, dry, wet, electro, or by solvent-assisted spinning. The nature of lignin determines a suitable spinning technology. In dry and wet spinning, lignin is dissolved with a suitable solvent. In dry spinning, the solvent evaporates while in wet spinning the solvent does not evaporate and it needs to be chemical washed. In melt spinning lignin is heated rapidly to 100-200°C after which the melted lignin is passed through an air blower. (Faruk & Sain 2015, 598.)

Thermostabilization is a heating process where the precursor is heat treated under noncontrolled atmosphere. The objective is to form cross-links between the lignin

molecules. During the stabilization phase, reactions as oxidation, dehydrogenation, elimination, and condensation occur. The mass lost in those reactions is converted to gasses. Gasses contain both hydrocarbon components and water vapor. Thermostabilization is done at around 250°C and the heating rate is low, typically 1°C/min. The temperatures and heating rate depend on the type of the lignin. (Faruk & Sain 2015, 600.)

Once fibers have gone through thermostabilization, they go to the carbonization phase. Carbonization is done in controlled atmosphere at high temperatures. The temperature inside the furnaces can be from around 700°C up to 3000°C. Controlled atmosphere is created via injecting nitrogen or argon into the process. The objective is to eliminate non-carbon elements such as oxygen, hydrogen, and sulfur, to increase the carbon-carbon bonds of the fibers and to improve the mechanical and electrical properties of the fibers. It's possibile that carbon fibers have the graphit structure if the heat treatment conditions and precursors are correct. The process is controlled by adjusting the heating rate. Changes in the heating rate affect the morpohology and brittleness of the product. Graphitization process can be used as an additional step if object is to produce graphite fibers. (Faruk & Sain 2015, 602). Faruk and Sai cite a study by the Chinese called "Lignin-based electrospun carbon nanofibrous webs as free-standing and binder-free electrodes for sodium ion batteries". The results of the study showed that lignin-based carbon electrode materials are suitable high-performance and low-cost solution for sodium ion batteries. (Faruk & Sain 2015, 531.)

3.3 Further processing of lignin from the circular economy perspective

Although further processing of the separated lignin is done to improve its selling price, it is also a step towards the circular economy. The circular economy is a holistic paradigm. The idea is to move from using products to using services. Recycling, the bioeconomy and renewable energy sources are part of the circular economy thinking. The materials used are utilized as efficiently as possible in the circular economy. The aim is also to minimize the amount of waste by inventing new reuse purposes. The main objective is to secure the future of the planet. Behind the circular economy is the fact that the earth will not withstand current consumption habits. We would need 1.6 globes to have sufficient resources for current consumption. (Mikä ihmeen kiertotalous? [What on earth is the circular economy] N.d.)

The Finnish Independence Celebration Fund (SITRA), which works to promote a fair and sustainable future, presents solutions that promote the circular economy under the name "The most interesting in the circular economy". The list includes forest industry companies such as Spinnova, which manufactures textile fibers from pulp, Sulapa, which manufactures wood packaging materials to replace plastics, and Forchem Oyj, which offers refined tall oil that can replace fossil raw materials in paints. From this it can be concluded that further processing of lignin can be considered as a circular economy idea. (Kiertotalouden kiinnostavimmat [The most interesting in the circular economy]. N.d.)

As mentioned, lignin is obtained mainly as a by-product in the production of pulp. By simply burning it as black liquor in a recovery boiler, it can already be considered part of the circular economy. The mill thus receives renewable energy and the non-organic components can be recycled back to the pulping process. Separating lignin from the pulp process and utilizing it, for example, in a lime kiln, is also in line with circular economy thinking, because fossil fuels are being replaced by renewable lignin. Further processing of lignin results in it being reused for a longer period of time than for energy production alone. In this way, it can replace fossil materials and is an even better option from a circular economy perspective.

4 Use and manufacture of nitrogen in industry

79 percent of the volume of air is nitrogen, making it an abundant element. As air, pure nitrogen is a colorless, tasteless, and odorless gas at the atmospheric pressure and ambient temperature. Nitrogen was discovered during investigations of air in the late 18th century. Scientists discovered that a part of air does not support respiration

or combustion. That part of air was later named as nitrogen. The first applications that utilize nitrogen were invented in the early 20th century. (Häussinger, Leitgeb & Schmücker 2000, 1.)

4.1 Properties of nitrogen

The chemical symbol for nitrogen is N and its atomic number is 7. It belongs to group 15 of the periodic table. Nitrogen contains two stable isotypes: ¹⁴N and ¹⁵N. Nitrogen forms molecular nitrogen known as dinitrogen (N₂). That strong trivalent bond between nitrogen atoms is responsible for its inertness. At room temperature lithium is the only element that reacts with nitrogen. Also, solubility of nitrogen is very low. Its boiling point is at -195,80°C and its melting point is at -209,86°C at ambient pressure. So, nitrogen is extremely cold in liquid form. The critical point of nitrogen is Tc: - 146.95°C and Pc: 3,39908MPa. (Häussinger et. Al. 2000, 1.)

Because nitrogen is unreactive and noncombustible, it is relative safe to use. But even though nitrogen gas is classified as non-toxic, it is an inert gas and replaces oxygen in the air, which can cause even death by asphyxiation. Asphyxiation can happen without any warning signs. If nitrogen is inhaled the first aid measure is removing the victim to fresh air as quickly as possible. When nitrogen leakage is detected, the area must be ventilated. Nitrogen itself is non-flammable but when stored in a pressurized container heat from the outside will increase the pressure of the gas and can cause the container to explode. Liquid nitrogen may cause a frostbite in direct contact. (Typen käyttöturvallisuustiedote [safety data sheet of nitrogen] 2015.)

Nitrogen applications

Nitrogen is used in both gaseous and liquid forms in various applications. A third of the produced nitrogen is used as liquid and the rest of it as gas. There are a few common applications represented in table 1. It can be seen that liquid nitrogen is used as a refrigerant and gaseous nitrogen as an inert. Basically, gaseous nitrogen is used when oxygen atmosphere could cause harm to a product or process and liquid nitrogen when fast or/and lot of cooling power is needed. For example, food industry uses nitrogen gas to preserve the freshness of packaged or bulk foods and liquid nitrogen in flash freezing to prevent damage to the cell walls in the cooling process. Gaseous nitrogen prevents any kind of contamination from happening and therefore is used in the pharmaceutical industry, for example. (Häussinger et. 2000, 18-23; Nitrogen gas applications. n.d.).

Table 1. Common uses of the nitrogen (Häussinger et. 2000, 18-23; Nitrogen gas applications. n.d.)

Liquid nitrogen		Gaseous nitrogen
Flash freezing of foods		Producing ammonia
Keeping goods cold during transport		Protective gas in metallurgy
Flash removal		Inert-gas shielding
Shrink-Fitting		Food storing
Cold polishing		Tire inflation
Cryomedical applications		Light pulp industry
Concrete cooling		Electronics
Control chemical reactions		pharmaceutical industry

However, the largest volume of nitrogen is used to product ammonia (Häussinger et. 2000, 18). Ammonia is a hydrogen-nitrogen compound (NH₃), that is mostly used in fertilizers. Ammonia is produced by the synthesis of hydrogen and nitrogen. The process is carried out under high pressure with the aid of a catalyst. Ammonia production is one of the most important industries in the world and the demand of ammonia is steadily rising all the time because when the world's population rises more fertilizers are needed. (Pattabathula & Richardson 2016.)

4.2 Methods of manufacturing nitrogen gas

There are two major methods of manufacturing nitrogen: fractional distillation and mechanical generation. Fractional distillation is also called the cryogenic method and mechanical generation is called the non-cryogenic method because in the fractional distillation air needs to be cooled to a cryogenic temperature and mechanical generation operates at ambient temperature. The mechanical methods are organized into subcategories: pressure swing adsorption technology and membrane technology, according to their working principle. (How Is Nitrogen Produced for Industrial Applications? 2018.) Advantages and disadvantages that are listed for each technology are from the manufacturers information so they must be viewed critically.

4.2.1 Pressure swing adsorption

The pressure swing adsorption (PSA) is a mechanical generation technology that generates nitrogen gas from compressed air. The technology is based on oxygen molecules being smaller than nitrogen molecules. Compressed air enters a PSA-generator where the oxygen molecules are captured with an absorbent material. Typically, a substance called a carbon molecular sieve (CMS) is used. Nitrogen molecules pass through the carbon molecular sieves and exit from the top of the vessel for use. The process is illustrated in figure 1. The grey pieces in the figure represent carbon molecular sieves and the close-up picture shows that smaller oxygen molecules attach to the pores of the carbon molecular sieve and the bigger nitrogen molecules bypass them. (Generating Nitrogen with Pressure Swing Adsorption (PSA) Technology 2017.)



Figure 1. PSA process illustration (Dobreva 2018.)

In the pressure swing generator, there must be two pressure vessels filled with carbon molecular sieves to produce nitrogen gas continuously. One vessel is in absorption phase and another in the regenerating phase. Regenerating is needed because after a while the pores of the carbon molecular sieves are saturated with oxygen molecules. When that happens, the air flow is switched to another vessel which was regenerated. The regenerating is done with a low flow of nitrogen, which enters through purge line from another vessel and by depressurizing the vessel. Oxygen molecules detach from the carbon molecular sieves because of depressurizing and exit through the exhaust pipe because flow of nitrogen. The name 'pressure swing' for the process comes from this. In a lower pressure the molecules that are captured in a higher pressure are released. After that the vessel is ready to go the adsorption phase again. The running interval of the phases is a few minutes and the switching system is automatically controlled and the PSA-generator produces nitrogen continuously without a need to operate. (Generating Nitrogen with Pressure Swing Adsorption (PSA) Technology 2017.) The schematic of a pressure swing adsorption generator is shown in figure 2. The main equipment and material flows are show in the figure.



Pressure swing adsorption generator

Figure 2. PSA-generator schematic

The generator sets the requirements for the compressed air. The temperature and pressure of the intake air are determined by the generator and because dirt and moisture could damage the carbon molecular sieves, the air must be filtered and dried after compressing. There are several measurements for the generator to make it a-fail-safe. (Generating Nitrogen with Pressure Swing Adsorption (PSA) Technology 2017.)

The advantages of a pressure swing adsorption are the purity of nitrogen, the reliability of the system, producing flexibility, low operating costs and a long-life cycle. The main advantage of the PSA-technology is the purity of nitrogen produced; A PSAgenerator can provide up to 99,9995% nitrogen. On the other hand, the generating capacity is directly related to the purity. The generated flow is lower when required high purity that means more expensive investment if high purity-high flow rate is needed. The reliability of the system means that it does not need much maintenance and nitrogen is available all the time. Flexibility means that generators are adjustable. Purity and flow requirements can be changed any point. Even though the investment can be expensive, operating costs are low. When nitrogen is needed continuously, an on-site generator is a cheaper solution than bottled nitrogen. PSAgenerators typically last at least a decade if the maintenance is properly taken care of so they offer a longtime solution for nitrogen supply. (Dobreva 2018.)

4.2.2 Membrane technology

Membrane generators are another mechanical method to generate nitrogen from compressed air. As the name implies the method is based on membrane technology. The technology is simple and is based on the different molecules of the air having different permeation rates. Oxygen molecules have a faster permeation than nitrogen, therefore oxygen molecules exit first through the membrane. The process is done in membrane modules. The modules are filled with very small hollow membrane fibers. Feed air is compressed and guided inside the fibers. The oxygen molecules pass through the membrane fibers and nitrogen molecules exits from the other end of the fibers. The purity of nitrogen depends from feed air pressure, temperature, and processing. (Generating Nitrogen using Membrane Technology 2018.) The process is illustrated in figure 3.

Membrane module schematic



Figure 3. Schematic of membrane generator

The advantages of a membrane generator are its simple working principle, its need little or no maintenance, it is quietness compared to PSA-generators and its easy and fast start up. Also, the flow and pressure of membrane generator are very stable. The main disadvantage of a membrane generator is that it can generate nitrogen purity of only approximately up to 99.9%. If the required purity is above that, membrane generators are not an option. A high air factor is another disadvantage of membrane generator. The air factor shows how much compressed air is needed to generate the demanded amount of nitrogen. For example, if required nitrogen flow is 100 m³/h and the air factor is 2,5, a compressor must compress 250m³/h air to the generator. (Generating Nitrogen using Membrane Technology 2018.)

4.2.3 Fractional distillation

Fractional distillation (a.k.a. cryogenic distillation) of air is another method to generate nitrogen from an air. Distillation is separating method which utilizes the fact that matters have different boiling point; the boiling point of oxygen is -183°C which higher than the boiling point of nitrogen which is -196°C. For distillation of air, the air must be in liquid form. Liquid form of the air can be achieved by cooling it below its critical point. Boiling point of air depends on temperature and pressure, higher pressures less cooling is required to condensate the air. Under ambient pressure air condenses at -192°C (or 81,5K). (Häussinger & et. 2000, 6-10.)

In the single column process, which is the simplest technology, a clean and dry air is compressed and then precooled. After that it is dried and cleaned again to remove water, carbon dioxides, and other impurities. Then the air enters to a main heat exchanger where it is cooled again. After the main heat exchanger air temperature is close to its liquefaction temperature. The air, under operating pressure, enters to a rectification column (a.k.a. distillation column) where distillation takes place. In the column liquid oxygen exits from the bottom and part of gaseous nitrogen exits from the top of column. That very cold nitrogen gas stream is utilized by passing it through the main heat exchanger. There is a reboiler-condenser where the rest of the nitrogen vapor is condensed and then mainly used as reflux. Part of the condensed nitrogen can be withdrawn as a liquid nitrogen product. The liquid oxygen that exits from the bottom of the column is vaporized in the reboiler-condenser. That vaporized oxygen stream is then warmed by the feed air in the main exchanger and expanded in a turbo-expander to provide even more cooling power for the main heat exchanger. After passing through the heat exchanger a second time the oxygen waste stream is almost at ambient pressure and temperature and it exits through an exhaust. The process is shown in figure 4. (Agrawal & Herron 2000; Häussinger & et. 2000., 6-10.)



Figure 4. Single column process schematic (Agrawal & Herron 2000)

Process efficiency can be raised with a two-column process (a.k.a. double-column process). Basis are the same as in the single column process. Air goes through the same treatment as for a single column process but as the name implies there are two distillation columns instead of the one. The columns are categorized to the high-pressure column where feed air enters first and the low-pressure column. Inside the columns, the process is the same as in the single column process, but it is done two times in different pressures. With the two-column process nitrogen product flow rate is about 72 mol per 100 mol of the feed air when with the single column process flow rate is about 40-50 mol per 100 mol of the feed air. (Agrawal & Herron 2000; Häussinger et. 2000, 6-10).

Advantage for the cryogenic distillation process is that it can meet almost all product requirements. Nitrogen generated with a distillation has oxygen content about 5ppm and plant is scalable. It is the most cost-effective solution when required nitrogen flow is very high, no matter what purity demand is. At lower flow rates a distillation plant is cost-effective if purity demand is high. The cost-effectiveness of the technologies depends from the sources. Source from year 2000 says that if needed purity is above 99,9% the fractional distillation system or delivered liquid nitrogen bottles are the most cost-effective solutions. (Häussinger et. 2000, 5). But thanks to development of the membrane and specially PSA- generators cost-effectiveness of the systems has changed. Nowadays PSA-generators can provide almost as pure nitrogen as fractional distillation systems. Distillation method is still dominant in a large-scale production. Bottled liquid nitrogen and high pressurized gaseous nitrogen for delivery are produced in large-scale cryogenic plants that are producing nitrogen as their primary product. Therefore 70 % of a total nitrogen production is done with fractional distillation. Disadvantages of the distillation systems are high capital cost and power costs. (Ivanova & Lewis 2012)

5 Cooling water systems

5.1 Cooling water circuits

The function of the cooling system is to remove heat from the process medium. That is done by transferring heat from the process medium to cooling medium. A coolant can be discharged from the process once it is warmed in heat exchanger or it can be cooled in another heat exchanger and reused. Cooling circuits are called oncethrough cooling systems or recirculating cooling system depending if the coolant is reused or not. (Chapter 23 - Cooling Water Systems-Heat Transfer n.d.)

5.1.1 Once-through water cooling systems

Once-through cooling (OTC) system is the simplest cooling method. It requires a water source, a pump, and a heat exchanger. Water is pumped from a water source, for example from sea, to a heat exchanger where it is discharged back to the sea, where heat transfers from the water to the environment. Because water is not reused, demand for it is high but on the other hand energy consumption is lower than in recirculating systems. Once-through systems have an environmental impact because warmed water is discharged back to the water source and in case of leakage, chemicals from the process medium may end up in the water source. There are two types of once-through systems. In direct systems the process medium is cooled with water from the source. In indirect systems there are primary and secondary circuits. Primary circuit uses water from the source as a coolant and it passes through heat exchanger where it cools the secondary circuit. Secondary circuit is a closed loop between process heat exchanger and cooling heat exchanger. With the secondary loop there are no worries that the process medium could end up in the environment through the cooling system. Once-trough cooling circuits are shown in figure 5. (Chapter 30 – Once-through cooling n.d.)

Once-through circuits are used commonly in locations where a natural source of water is available. The minimum achievable temperature of the process medium depends on the ambient temperature of the water source but with the once-through technology it is possible to get lower temperatures than with the recirculation technology. The temperature of the discharged water is kept as close as possible to the ambient temperature to prevent energy losses and to minimize the environmental impact. The general objective is to improve energy efficiency because all energy discharged to the environment is wasted money. (BREF Cooling 2001.)



Figure 5. Once-through systems

5.1.2 Recirculating water cooling systems

Recirculating (a.k.a. closed circuit or closed loop) cooling system's basic difference to a once-through system is that it reuses cooling water and does not need a large water source. Reusing the water is often done by transferring heat from coolant water to ambient air. In an open recirculating system, a coolant is cooled directly in cooling tower, spray bond, or in an evaporative condenser, where heat evaporates to air and water is guided back to the reservoir tank. Closed recirculating systems have a secondary and a primary loop. They are connected in a liquid-to-liquid heat exchanger where the heat transferred from process to secondary loop is transferred again to the primary circuit. There the heat is transferred to the atmosphere with evaporation. Thus, there is very little mass loss of water and so no constant water treatment is needed. In a wet closed circuit, primary loop's cooling is done in another liquid-toliquid heat exchanger and transferred heat is used in other processes. Depending from temperatures and size of the process there can be several cooling circuits. (Chapter 32 - Closed Recirculating Cooling Systems n.d.)

5.2 Heat exchangers

Every cooling application requires at least one heat exchanger where most heat transfer occurs. Heat exchangers are devices where heat energy is transferred from a hot medium to a cold medium. In the heat exchanger, the process medium is cooled without involving fluids to mix that differs them from mixing chambers. In heat exchangers, heat is transferred by conduction and convection. (Cengel & Ghajar 2015, 647.)

Heat exchangers can be classified various ways. One way to classify is to sort them according to their process functions into condensers, heaters, coolers, chiller, and phase-change exchangers. Another way classify is by flow path configuration: In parallel flow (a.k.a. concurrent flow) both fluids enter to exchanger at one end and flow same direction and exit from another end. In countercurrent flow (a.k.a. counterflow) fluids enter from opposite ends and flow in opposite directions. In single-pass crossflow fluids enter from different angles and flow crosses in heat exchanger. In multi-pass cross flow fluids enter from different angles and one fluid crosses another fluid multiple times. Construction type is another way of classification. Heat exchangers can be sorted to tubular, plate, plate-fin, tube-fin, and regenerative heat exchangers. (Zohuri 2017.)

This chapter introduces a few most common heat exchangers used in industrial cooling applications. One of the most used types of heat exchanger is the shell-and-tube heat exchanger. It is a tubular heat exchanger that contains a shell with lots of tubes inside (one shell can contain hundreds of tubes). Tubes are connected to front- and rear-end headers. One fluid enters to the front-end header and flows inside tubes to the rear-end header where it exits through the outlet. Other fluid enters from a shell inlet and it flows outside the tubes through the shell and exits from a shell outlet. Fluids can flow concurrent or countercurrent. The shell is usually equipped with baffles to improve heat transfer. The two most common shell-and-tube heat exchangers are shown in figure 6. Shell-and-tube heat exchangers are subcategorized by the number of shells and tube passes they involve. In the figure left one is called as oneshell pass and one-tube pass and right one as one-shell pass and two-tube passes. There are many different variants of shell-and-tube heat exchanger: they can have one or two headers, different shapes of headers can be used, tubes can take several U-turns inside the shell or tubes can take U-turn outside of the shell. The shell-andtube heat exchangers are suitable for many applications, they can be used as a liquidto-liquid, a liquid-to-gas, or a gas-to-gas heat exchanger. (Cengel & Ghajar 2015, 650).



Shell-and-tube heat exchangers

Figure 6. Shell-and-tube heat exchangers

The simplest heat exchanger used in cooling operations is a double pipe exchanger. It is made of two pipes with different diameters. Pipe with smaller diameter goes inside of another pipe. One fluid flows through a smaller pipe and the other fluid flows in the space between the pipes. Fluids can flow in parallel or in countercurrent. Parallel flow double-pipe heat exchanger is shown in figure 7. (Cengel & Ghajar 2015, 648).

Double-pipe heat exchanger



Figure 7. Double-pipe heat exchanger

Plate (a.k.a. plate and frame) heat exchanger is another typical option for cooling systems. Plate heat exchanger is a compact heat exchanger that is made of a series of thin metal plates packed together. Plate package is compressed with frame plates and tightening bolts. The frame plate has nozzles where fluids enter to the heat exchanger. Each plate has portholes that allows only one fluid to enter between plates. Plates have gaskets that seal channels between each other. Heat transfers from hot fluid to cold one through plates. Each cold stream flows between two hot fluids to provide effective heat transfer. The more plates, the greater the heat transfer. Material of the gasket depends on fluids properties. Typically, molded elastomers are used. There are different corrugations used in plates to enhance heat transfer even more. Liquid-to-liquid plate heat exchanger is shown in figure 8. (Carvalho, Mota & Ravagnani 2015; Cengel & Ghajar 2015, 650-651.)



Figure 8. Exploded View of a Plate Heat Exchanger (adapted from Carvalho et. 2015.)

Plate exchanger's advantages are its compactness, great temperature control, heat transfer efficiency and low manufacturing costs. Compactness of the plate heat exchanger comes from its great efficiency. Plate heat exchangers can occupy even ten times less floor space than tubular heat exchanger for the same cooling power. Producing costs are low because manufacturing does not involve much welding when the plates are compressed together. The heat exchanger is also easy to maintain and clean because plate packages can be opened when needed. Material of plates can be selected based on fluid properties. Disadvantages are high pressure drop, temperature, pressure, and fluid limitations. High pressure drop occurs due to the small flow space between the plates and corrugation of plates. Small flow space is also a reason to avoid phase change in the plate heat exchanger. Gaskets can break at high temperatures or if fluid flowing inside is not compatible for the gasket. Because of these limitations the plate heat exchanger is good at liquid-to-liquid applications where hot and cold fluid streaks are about at the same pressure. (Carvalho et. 2015.)

5.3 Heat transfer in heat exchangers

Heat transfer follows the laws of thermodynamics in heat exchangers. First heat transfers to a separating wall by convection and through the wall by conduction. Then it is transferred from the separating wall to the cold fluid by convection. The easiest way to start analyzing a heat exchanger is calculate heat transfer rate from fluid's temperature difference. It can be done via formula (1): (Cengel & Ghajar 2015, 651-660)

$$\dot{Q} = \dot{m} \cdot c_{ph} \big(T_{h,in} - T_{h,out} \big) \tag{1}$$

Where:

 $\dot{Q} = Heat \ transfer \ rate \ [W]$

$$\dot{m} = Mass flow rate of the fluid \left[\frac{kg}{s}\right]$$

$$c_{ph} = specific heat of hot fluid \left[\frac{J}{Kg \cdot K}\right]$$

 $T_{h,in} = hot fluid inlet temperature [K]$

 $T_{h,out} = hot fluid outlet temperature [K]$

All thermal resistances are combined in the analysis of heat exchangers. The walls separating fluids are very thick and made of a highly conductive material to lower its thermal resistance and it is usually negligible. Therefore, the overall heat transfer coefficient can be calculated with formula (2):

$$U = \frac{1}{\frac{1}{h_i} + \frac{1}{h_o}}$$
(2)

Where:

$$U = Overall heat transfer coefficient \left[\frac{W}{m^2 \cdot K}\right]$$
$$h_i = convection coefficient of fluid inside \left[\frac{W}{m^2 \cdot K}\right]$$
$$h_o = convection coefficient of fluid outside \left[\frac{W}{m^2 \cdot K}\right]$$

Convection coefficients can be terminated if properties of fluids and heat transfer surface are known. In the water-to-water heat exchangers the overall heat transfer coefficient is from 850 W/m²·K up to 1700 W/m²·K. In practice, the overall heat transfer coefficient deteriorates over time because of fouling. Impurities accumulating on the heat transfer surfaces cause a heat resistance. Also, corrosion and other chemical fouling can affect the overall heat transfer coefficient. Fouling factor should be considered especially in applications where it is likely to occur. Thermal resistance of water due to fouling is 0,0001 (m²·K)/W below 50°C and 0,0002 (m²·K)/W above 50°C. Thus, it is not a big deal in the water-cooling systems. Fouling affects more to designing in heat exchangers where one of the fluids is for example fuel gas. (Cengel, & Ghajar 2015, 651-655).

The overall heat transfer coefficient is needed to calculate heat transfer rate in heat exchanger via Newton's law of cooling formula (3):

$$\dot{Q} = UA_s \Delta T_m \tag{3}$$

Where:

 $\dot{Q} = Heat \ transfer \ rate \ [W]$

$$A_s = The heat transfer surface [m^2]$$

 $\Delta T_m = An \ appropriate \ mean \ temperature \ between \ fluids \ [K]$

Because temperature difference is various along the heat exchanger mean temperature difference is needed. Logarithmic mean temperature difference is commonly used in the analysis of the heat exchangers. It represents an average temperature difference between hot and cold fluids. It can be calculated with a formula (4):

$$\Delta T_{lm} = \frac{\Delta T_1 - \Delta T_2}{\ln\left(\frac{T_1}{T_2}\right)} \tag{4}$$

Where:

$$\Delta T_{lm} = Logarithmic mean temperature difference [K]$$

 $\Delta T_1 = T_{h,in} - T_{c,in}$ in parallel flow and $T_{h,in} - T_{c,out}$ in counter flow [K]

 $\Delta T_2 = T_{h,out} - T_{c,out}$ in parallel flow and $T_{h,out} - T_{c,in}$ in counter flow [K]

These calculations are used to analyze a simple parallel-flow or counter-flow heat exchanger. Correction factor F is needed to calculate heating rate for multipass shelland-tube heat exchangers. Logarithmic mean temperature difference is multiplied with correction factor. Value of the correction factor depends on the design of the heat exchanger and temperature of the fluids. Once you know the mass flows of fluids and the inlet and outlet temperatures of at least one fluid you can calculate the heat transfer surface for the heat exchanger. In that case, it is assumed that the heat exchanger is well insulated and there are not heat losses. (Cengel & Ghajar 2015, 663- 670).

Heat transfer rate of the heat exchanger is the most important thing when selecting heat exchanger. The heat exchanger must be able to transfer enough heat to reach the desired temperatures. Other factors that affect selecting the heat exchanger are the cost of the heat exchanger, size and weight, type, materials, and specified

requirements. Usually the one with the lowest price tag is selected from the heat exchangers suitable for the process. The size of the heat exchanger influences the price. The size of the heat exchanger should also be considered in cases where space for it is limited. There are various types of heat exchanger available. The type and properties of the fluids, size and weight limitations, and the possibility of phase change limit the choice. The material of the heat exchanger must be suitable for fluids and it must withstand the process conditions. A big temperature difference between fluids and corrosive fluids can cause problems if the wrong material is selected. Different applications have different specific requirements that must be considered when selecting the heat exchanger. For example, leak-tightness, safety, and reliability are important factors when using toxic fluids. Ease of maintenance and the price of maintenance may also be influencing factors. Selecting a heat exchanger is a complicated process and results obtained from heat transfer calculations do not give a definite answer to what kind of heat exchanger is needed. Therefore, heat exchangers are often overdesigned. This in turn leads to higher costs. The bigger the heat exchanger is the higher the price it has, and overdesigned heat exchanger will lead to increased pumping costs due to the greater pressure drop in the heat exchanger. (Cengel & Ghajar 2015, 684).

6 Nitrogen gas supply system design

6.1 Use of nitrogen gas in the process

The carbonization process requires a controlled atmosphere which can be done by inert gases or vacuum. Nitrogen was designed to be inert in this project. Nitrogen gas replaces air in a furnace and creates an oxygen-free atmosphere inside. Some furnaces have vacuum pump technology that creates necessary vacuum first and then nitrogen is injected into the furnace. Nitrogen ensures that no combustion reaction can occur even when temperature is very high inside the furnace. Although the lignin does not burn in the furnace, part of the lignin is evaporated as gases. These gases exit through an exhaust gas outlet from the furnace. The nitrogen gas exits through the same outlet. Therefore, the supply of nitrogen gas must be continuous in order to maintain an oxygen-free atmosphere inside the furnaces.

6.2 Requirements for the nitrogen gas supply system

The manufacturers of the furnaces define requirements for the auxiliary systems in quotations. Nitrogen supply system must be able meet requirements for every furnace. So, first you must gather all requirements from all quotations before you can set exact requirements for the nitrogen gas supply system. The main requirements for the nitrogen supply system are producing capacity requirement and purity requirement. Other requirements are maximum and minimum inlet pressure for the nitrogen gas. Every manufacturer has reported nitrogen gas consumption for each furnace in technical data sheets. In some cases, manufacturers have also defined requirements for the amount of emergency nitrogen in quotations. Emergency nitrogen gas is a nitrogen gas that must be available when the main source of nitrogen fails. One of the furnaces works under controlled oxygen atmosphere and does not need nitrogen gas continuously, but it also needs connection from nitrogen source.

Required purity of nitrogen is the second important thing that affects designing a nitrogen gas supply system. Nitrogen gas can be produced several ways, but purity requirements limit the options. If the purity requirements are at ppm level (parts per million), membrane generators cannot meet the requirements. The purity value indicates how much there is oxygen in the nitrogen gas. For example, 100 ppm means that 0.01% of the gas produced is oxygen and rest of it is pure nitrogen. The purity value may also be expressed as the nitrogen content of the gas produced. For example, the nitrogen gas having a purity of 99% has an oxygen content of 1% which is equal to 10000 ppm.

Furnaces require different purity, but because a generator can generate one purity at the time the highest purity requirement provides the basis for the design of the

nitrogen gas supply system. Manufacturers have defined inlet pressure for nitrogen gas. Typically, they are given scales where inlet pressure should be settled. In some quotations there is given only maximum inlet pressure. The pressure requirement must be considered when choosing a nitrogen generator.

6.3 Capacity analysis

At this point capacity analysis is based on initial data available from manufacturers quotations. Each manufacturer has its own way of presenting the nitrogen consumption of the furnace. Consumption can be shown as an approximate value or as a scale value. There is also a pointed out instantaneous maximum value in some cases. Based on this information it is difficult to know exactly the total consumption of nitrogen gas at each moment. In the quotations, it is not defined when furnace demands the highest nitrogen flow. Another thing is that there is no final decision made on that which of the furnaces will be running at the same time. Of course, this affects the total instantaneous consumption.

So, it is clear that the only viable option at this point is to design the nitrogen gas supply system with a production capacity that high that every furnace could run at the same time. Theoretical maximum capacity can be clarified, and calculations are done with maximum flow values represented in the quotations. Table 2 shows demand for nitrogen gas flow for every furnace.

Equipment	Nitrogen consumption
Furnace 1	average. 25m ³ /h, instantaneous maximum
	70m³/h
Furnace 2	max. 24m³/h
Furnace 3	max. 15m ³ /h
Furnace 4	No constant need for nitrogen gas, inert gas sup-
	ply required
Furnace 5	No constant need for nitrogen gas, needs nitro-
	gen only for purging and flushing

Furnace 1 manufacturer specified their average consumption and instantaneous maximum consumption, other manufacturers just specified consumption scale. Based on these values and assumptions, it was decided that the required production capacity of the nitrogen generator would be about 40 m³/ h. It is clear that a generator with such production capacity will not be able to supply the required nitrogen flow in all situations. The idea is that the nitrogen gas supply system is equipped with buffer vessel after the nitrogen generator. Instantaneous consumptions of nitrogen are not affecting generator sizing when the system is equipped with that vessel.

6.4 Choosing the nitrogen generator

Once the nitrogen supply system requirements have been specified it is possible to search options for a nitrogen generator supplier. There are several suppliers with the same kind of generator systems. A nitrogen supply system is a big investment that it is always done with consulting the supplier and negotiation about price, options, delivery terms and conditions. It is important to define your requirements for the generator as precisely as possible to suppliers, so they can do an exact quote for you.

A supplier was selected by tender. The supplier proposed a PSA-generator because it is the most cost-effective solution that can meet our requirements. The supplier offered generators that could produce nitrogen flow about 55m³/h as total. Its production is slightly higher than the calculated consumption, but it is better to be over capacity rather than having the nitrogen system as a bottleneck. Their generators are modular, that means there are several generators connected in parallel. Therefore, they can offer solution for every demand. The generators are adjustable that mean the operator can adjust purity and flow demand if needed. Overcapacity is acceptable in this stage because there may still be equipment that uses nitrogen coming to the plant that is not involved in calculations at this point of design. Thanks to the modularity of the generator, even if the demand for nitrogen gas increases significantly in the future, it is easy to increase capacity by adding more generators in parallel. Possibility of that just must be noticed when doing layout designing of the plant.

6.5 Safety

Safety of process is very important factor when designing a nitrogen gas supply system. Nitrogen gas exits through the exhaust gas outlet from the furnace so if the nitrogen gas supply system fails then there is no nitrogen gas flow into the furnace. If air gets into the furnace instead of nitrogen, there occurs a combustion reaction. The combustion reaction in the furnace can cause danger. To avoid that situation, it is defined that nitrogen gas supply system must be redundant or fail-safe. The easiest way doing that is acquire nitrogen gas cylinders. The nitrogen gas is pressurized to high pressure and stored in these cylinders. If the nitrogen generator breaks down during the process, the nitrogen source is automatically switched to nitrogen cylinders. Nitrogen from cylinders provides enough time to shut process down safely. When nitrogen is stored at high pressure, less cylinders are needed because nitrogen gas obeys the laws of ideal gases. Once the required volume of the emergency nitrogen is known capacities of the cylinders can be calculated by combined gas law formula (5):

$$\frac{P_1 \cdot V_1}{T_1} = \frac{P_2 \cdot V_2}{T_2}$$
(5)

Where

 P_1 =Pressure inside cylinder [bar]

 V_1 = Gas volume inside cylinder [m3]

 T_1 =Temperature inside cylinder [K]

P₂= Operating pressure [bar]

 V_2 = Volume of the emergency nitrogen demand at operating pressure [m3]

 T_2 = Operating temperature [K]

As the temperature is constant the formula can be reduced to the form:

$$P_1 \cdot V_1 = P_2 \cdot V_2$$

Gas volume inside cylinder is solved by dividing equation by the pressure inside cylinder, thus the final form is:

$$V_1 = \frac{P_2 \cdot V_2}{P_1}$$

Once furnace manufacturer's emergency nitrogen demand is 120m³ at 1 bar pressure and pressurized cylinders can handle pressure up to 300bar the volume of cylinder is:

$$V_1 = \frac{120m^3 \cdot 1bar}{300bar} = 0.4m^3$$

Typically, a capacity of the cylinder is 80 liters, thus there are needed a total of 5 cylinders.

$$\frac{0,4m^3}{0,08m^3} = 5$$

Another safety thing of the nitrogen system is taking care of ventilation. Small nitrogen gas leakages are not dangerous for the surrounding area if ventilation is properly designed. If the ventilation of the nitrogen gas supply system area or any area where the nitrogen gas pipe passes is poorly designed, there is a potential for serious injury. In case of massive nitrogen leakage, there must be a change to ventilate the area without accessing the area. Leakages can be detected for example by pressure measurements. Critical areas can also be equipped with multi-gas meters that detect if oxygen levels in the air are falling. Automation has also a big role in the safety of nitrogen supply systems. There must be different kinds of interlocks that operate automatically if example pressure of nitrogen gas drops or purity measurement alerts.

6.6 Other equipment in the nitrogen gas supply system

The nitrogen gas supply system includes components other than the nitrogen generator alone. The system needs compressor, vessels of different sizes, filters, and various measuring instruments.

6.6.1 Compressor

Normally the choice of compressor is based on the size of the generator and usually generator supplier offers optimum sized compressor. However, there are other machines in the process that also need compressed air and the idea is that the compressor could produce all compressed air that is needed. Therefore, it is important to clarify also the whole compressed air consumption before doing a business with the generator/compressor supplier. Requirements for the compressor designing are consumption and pressure. The highest consumption and the highest-pressure demand define the size of the compressor. In this project, there are special requirements for the compressor. The whole process of the pilot plant is designed to be clean as possible to avoid contamination. The objective is to ensure that no unwanted particles enter in the final product. That is why it was defined that the compressor must be oil free. The compressor oil does not mix with compressing air during normal operation, but it is possible if the compressor breaks down. Oil leakage would ruin the nitrogen generator and filtering systems. Oil-free model ensures that no oil can enter the process under any circumstances. Oil-free model is more expensive, but you do not want to take risks in this type of project. On other hand, there will be a cooling water circuit, so the compressor can be a water-cooled model instead of an air-cooled model. After discussions with the supplier, the selected compressor was a water-cooled oilfree rotary screw compressor.

6.6.2 Filters and vessels

The generator suppliers often offer whole nitrogen gas supply system from the compressor to the nitrogen gas buffer vessels. They know best what kind of cleaning and drying components are needed for the generator. After selecting generator and compressor units, they offered optimum sized cleaning and drying components. The final tender included three different filters, an adsorption dryer, and active carbon filter which were sized based on the compressed air flow.

A buffer tank for nitrogen was required, so that the production capacity of the generator is not needed to be so high. The supplier sized capacity of the vessel based on given information. A buffer tank for the compressed air is needed to keep pressure steady. If the compressed air consumption rapidly rises, for example in process equipment start up the compressor cannot keep up and the pressure drops temporarily.

6.7 Summary of the nitrogen gas supply system

The Nitrogen gas supply system designed for the project is shown in the figure 9. The system shown in figure 9 can meet all the key requirements that were set for it. In addition to this there will be the emergency nitrogen system that was mentioned in the safety chapter. It includes high pressurized nitrogen cylinders with fail safe valves. Main system includes (numbers on the list referring numbers in figure):

- 1. A water-cooled oil-free rotary screw compressor, power of 160kW
- 2. A buffer vessel for the compressed air, volume of 5m³
- 3. Cleaning and drying system, which includes all filters needed
- 4. Nitrogen generators, nitrogen flow of appr. 50m³/h
- Buffer vessel for the nitrogen gas, 3m³
 Measurement instruments and valves are not shown in the figure but those are also



Figure 9. The theoretical chart of a nitrogen gas supply system

Once these main components of the nitrogen gas supply system have been properly sized, there should be no surprises that affect the rest of the design process. Thus, it is possible to start more accurate layout and process design. Process design includes piping designing and automation designing for the system.

7 Exhaust gas treatment system design

An exhaust gas treatment system is needed to control gases that exit from the furnaces. Hydrocarbon gases generated during heat treatment and nitrogen gas injected to control atmosphere exit through the furnace outlet. The easiest way to treat those gases is to guide them to a combustion chamber where burnable gases are burned with the help of auxiliary burner, and all gases are safely guided to the outside through a chimney.

7.1 Overview of the exhaust gas treatment system

Every manufacturer has its own kind of exhaust gas treatment system, but the basics of the systems are the same. Exhaust gases are guided from the furnace to the combustion chamber where burnable gases are burnt with help of auxiliary fuel. From the combustion chamber gases are guided to the chimney via exhaust fan. The main components of the exhaust gas treatment system are a combustion chamber with burners, a burner for auxiliary fuel, chimney, fans, and bypass systems for in-case of failure. The auxiliary fuel is needed because the exhaust gas flow is not steady enough to keep combustion reaction going on continuously.

7.2 Designing

When buying the furnaces, the manufacturers usually offer an optional gas treatment system for the furnace. There will be several furnaces in this project, so it is not wise to buy a separated exhaust gas treatment system for every furnace. The exhaust gas flows from the furnaces are so low that all gases could be treated in one larger combustion chamber. Exhaust gas flows from each furnace must be known when designing the combustion chamber. The gas flows from furnaces can be calculated when knowing which process step is going on in the furnace. The furnaces are designed for complete a single process step. The exception to this is the furnace number one, that is designed to be multifunctional and it can go all process steps at one go, and the furnace number three that can complete two process steps. Therefore, not all process steps, even in theory, can be running at the same time.

In laboratory tests, the mass yield of each process step was calculated by measuring the weight of the material before and after the process step. The mass of the closed system must remain constant over time according to the law of conservation of mass, so the mass lost during the process is converted to gases. The exhaust gas flow generated during the process step can be calculated with the formula:

$$(1 - mass yield) \cdot \dot{m_1} = \dot{m_2}$$

Where:

mass yield = average mass yield given from laboratory tests

 \dot{m}_1 = raw material mass flow [kg/h]

 \dot{m}_2 =Exhaust gas mass flow [kg/h]

Mass flows are shown in kg/h because manufacturers use those units in their designing. Results of the calculations are shown in appendix 1. The required maximum exhaust flow is obtained when the maximum mass flow of raw material is used for calculation. It can be seen that most of the mass loss happens at the process step two, thus most of the gas is generated there. The appendix also includes a comparison of the maximum flows between all possible running models. The running models that are most likely to be used are in bold. There are four process steps in calculations, however there will be a total of six furnaces that generate exhaust gases. The two furnaces are not in the calculations because those mass yields are not known. But those furnaces are running less frequently and have low capacities thus those do not generate much exhaust gases. Of course, these furnaces are also taken into account in the design process. These exhaust streams do not greatly affect system design, but they still require connections to the system.

In addition to burnable gases there will be non-burnable gases, mostly nitrogen gas, that are also guided to the combustion chamber. Those gases must be calculated and considered when setting requirements for the exhaust gas treatment system, because if nitrogen flow is higher than the design values it may affect the combustion reaction.

7.3 Summary of the exhaust gas system

Once the exhaust gas flows were examined and calculated from every furnace, there was decided to choose two separated systems. The multifunctional furnace number 1 is more complex than the others and it will have its own exhaust gas treatment system. This ensures that the exhaust gas treatment system has sufficient capacity in all situations. The system will be ordered from the furnace manufacturer so, because they have designed the furnace, they have also accurate information about the gas flows.

There will be another treatment system for the gases from the other furnaces. The system is sized for maximum gas flows from every furnace. Even though all furnaces will not be running at the same time it will not affect sizing much because the exhaust gas flows are so low. The flow chart of the whole exhaust gas treatment system is represented in figure 10. There are two burners for exhaust gases in the combustion chamber two. Furnaces two, three, and four are connected to one burner and furnaces five and six are connected to another burner. Both combustion chambers are equipped with natural gas burners. There is already a natural gas network in the Sunila mill area. So, it is the best option to use natural gas as an auxiliary fuel. When using the existing gas network, only the pipeline design needs to be done. Another fuel option for auxiliary burner would have been propane gas.



Exhaust gas treatment system flow chart

Figure 10. Exhaust gas flow chart

8 Design of the cooling water system

Furnaces operate at high temperatures, so the product needs to be cooled before further processing. All furnaces have integrated water-cooling. Idea was to design a cooling water system that can ensure cooling water to all equipment at every situation.

8.1 Overview of the cooling water system

Indirect once-through cooling water circuit was the best choice for this plant. System have a closed loop where fluid, in this case water, flows. The water circulating inside the closed loop is cooled in a heat exchanger by the primary coolant which is also water. The closed loop includes a feedwater tank, two pumps, pipelines to furnaces, the water-to-water heat exchanger, and process and safe equipment. In figure 11 you can see a rough illustration of the system. The closed loop is designed so that water is pumped through a single pipe from the feed water tank to a header. There are pipelines for each furnace from the header. From furnaces pipelines are connected to another header and from that header a single pipeline goes to the heat exchanger and after that back to the buffer tank. There are some measurements in the figure but in the final version there will be many more. The actual process design begins after the main equipment has been selected and the safety of the process has been reviewed in a hazard and usability study.



Figure 11. Cooling water circuit

8.2 Process safety

The cooling water supply is important for the process safety. If cooling water supply fails it can cause mechanical damage to furnaces and danger for operators. Therefore, the cooling water supply system must be redundant or otherwise fail-safe. Pump of the cooling water circuit will be redundant to provide option for maintenance. There will be several measurements for cooling water temperature and pressure in order to notice possible faults.

In this project, it was decided to design the fail-safe system. The idea is there will be a water tank located on the upper floor. During power failure or pump breakdown water flows through the process without pumping due to the hydrostatic pressure. Warmed water is then led to drain through an outlet which is normally closed. Therefore, the water loop is not closed anymore, and water supply lasts as long as there is water in the tank. Sunila mill have back-up pumps that provide water supply to critical parts of the mill during power outages. There will be connection from those backup pumps to the tank. However, it takes some time to get those pumps running so the capacity of the tank must be large enough to ensure continuous water supply.

8.3 Cooling power calculations

The total cooling capacity is calculated by first calculating the cooling power for each furnace. Furnace manufacturers have given the technical data in which calculations are based. Cooling water consumption is given in technical data for every furnace and for some furnaces required cooling power is given by the manufacturer. For other furnaces there is given maximum inlet and outlet temperatures of cooling water. Cooling water pressure is also defined in technical data. Thus, theoretical maximum cooling power for each furnace can be calculated from the enthalpy difference with the formula (6):

$$\dot{m}_i \cdot h_i + Q_f = \dot{m}_o \cdot h_o \tag{6}$$

Where:

$$\dot{m}_i = Mass flow cold water in \left[\frac{kg}{s}\right]$$

$$h_i = Cold water enthalpy \left[\frac{kJ}{kg}\right]$$

 $Q_f = heat flow to water [kW]$

$$\dot{m}_o = mass flow out \left[\frac{kg}{s}\right]$$

$$h_o = Exiting \text{ water enthalpy } \left[\frac{kJ}{kg}\right]$$

There occurs no mass transfer because cooling is indirect, so mass flow in is equal to mass flow out. Formula to calculate heat flow to water is:

$$Q_f = \dot{m}_i \cdot (h_o - h_i)$$

Furnace 2 for example:

 $\dot{m}_i = 0,33333 \left[\frac{kg}{s}\right]$

$$h_o = 230,27 \left[\frac{kJ}{kg}\right]$$

$$h_i = 84,294 \left[\frac{kJ}{kg}\right]$$

$$Q_f = 0,33333 \left[\frac{kg}{s}\right] \cdot (230,27 - 84,294) \left[\frac{kJ}{kg}\right]$$

$$Q_f \approx 49kW$$

All results are shown in appendix 2. Using given minimum inlet and maximum outlet temperatures in calculations do not give realistic results. Outlet temperature depends on the cooling water mass flow and when given maximum mass flow is used, we can assume the actual outlet temperature is much lower than the given maximum temperature. The assumption is confirmed by the fact that the cooling systems are very similar in every furnace and cooling power values given for the other furnaces are way lower than values calculated by temperature difference. Therefore, there is another calculation which is based on assumption that the water temperature will raise 15°C in furnaces which have not been given the cooling power information. Results of these calculations are shown in appendix 3. Those results neither

does give an exact answer to the required cooling power but comparing the results will help you choosing a heat exchanger.

Cooling water pipelines from furnaces are designed to connect header where water exits through a single pipeline. Calculation of exiting water mass flow and temperature is done by formula (7)

$$\sum \dot{m}_n \cdot h_n = \dot{m}_e \cdot h_e \tag{7}$$

where:

$$\dot{m}_n = water\ mass\ flow\ from\ each\ furnace\left[\frac{kg}{s}\right]$$

$$h_n = enthalpy of each water mass flow \left[\frac{kJ}{kg}\right]$$

$$\dot{m}_e = water mass flow exiting from header \left[\frac{kg}{s}\right]$$

$$h_e = enthalpy \, of \, water \, exiting \, from \, header \left[\frac{kJ}{kg}\right]$$

In this case form is:

$$\dot{m}_1 \cdot h_1 + \dot{m}_2 \cdot h_2 + \dot{m}_3 \cdot h_3 + \dot{m}_4 \cdot h_4 + \dot{m}_5 \cdot h_5 = \dot{m}_e \cdot h_e \tag{7}$$

Where

$$\dot{m}_1 = 0,833 \left[\frac{kg}{s}\right]$$

$$\dot{m}_2 = 0,333 \left[\frac{kg}{s}\right]$$
$$\dot{m}_3 = 0,333 \left[\frac{kg}{s}\right]$$
$$\dot{m}_4 = 0,750 \left[\frac{kg}{s}\right]$$
$$\dot{m}_5 = 1,389 \left[\frac{kg}{s}\right]$$

Thus

$$\dot{m}_e = (0,833 + 0,333 + 0,333 + 0,75 + 1,389) \left[\frac{kg}{s}\right]$$

 $\dot{m}_e = 3,638 \left[\frac{kg}{s}\right]$

And

$$h_e = \frac{(\dot{m}_1 \cdot h_1 + \dot{m}_2 \cdot h_2 + \dot{m}_3 \cdot h_3 + \dot{m}_4 \cdot h_4 + \dot{m}_5 \cdot h_5)}{m_e}$$

$$h_e = 169,06 \left[\frac{kJ}{kg}\right]$$

Water must be cooled in a primary heat exchanger back to starting the temperature to keep closed cycle stable. Thus, cooling power needed from the primary heat exchanger can be calculated by formula:

$$\dot{m}_e \cdot h_e - Q_p = \dot{m}_b \cdot h_b$$

There occurs no mass transfer in the heat exchanger, so formula gets form:

$$Q_p = \dot{m}_e \cdot (h_e - h_b) \tag{8}$$

Where:

$$Q_P = Cooling \ power \ required \ [kW]$$

 $h_b = Enthalpy of water exiting from heat exchanger$

For example, situation 1 in the appendix 4:

$$\dot{m}_e = 2,25 \left[\frac{kg}{s}\right]$$

$$h_e = 182,871 \left[\frac{kJ}{s}\right]$$

$$h_b = 83,965 \left[\frac{kJ}{s}\right]$$

$$Q_p = 2,25 \left[\frac{kg}{s}\right] \cdot (182,871 - 83,965) \left[\frac{kJ}{kg}\right]$$
(8)

 $Q_p \approx 223 \ kW$

All results are shown in appendix 4. At this point in time, there is no further information about furnace 5 because negotiations about it are still ongoing. So, there are calculations where it has not been considered but also calculations where it is included with assumption it will work with the same principles as the other furnaces. Basically, every possible running schema is calculated. It gives perspective how required cooling power changes between schemas. Results are shown in figure 12. It can be seen that cooling power needed to keep closed cycle stable is between 42 kW and over 300 kilowatts. As mentioned, calculation 1 results are higher and probably not correct ones, but they give maximum theoretical required cooling power based on manufacturers information. Calculation 2 is done with assumptions and choosing a heat exchanger based on it is not recommended. In situation seven is just calculation one showed because assumptions made in calculation 2 does not affect at this situation and it would give same answer.



Figure 12. Cooling power at different running schemas

8.4 Summary of the cooling water system

8.4.1 Feed water tank

Capacity of the tank is designed based on water consumption and how fast back-up water is available. Water consumption depends on the running schedule. Different water consumptions are shown in figure 13.



Figure 13. Cooling water consumptions

Back-up water pumps start running within 15 minutes after power failure so the capacity of the tank should be at least 3,75m³ at maximum water consumption. As said before, all furnaces will not be running at the same time so even smaller capacity would be enough in many situations. Capacity of three cubic meters would last over half hour in running situations one, four and five and over 15 minutes in other running situations. Larger capacity ensures continuous waters supply even if there are problems starting up the back-up pumps. Final decision about the capacity of the tank is done later because it depends how much there is space for the tank in the building. But based on calculations capacity suggested is between two to four cubic meters.

8.4.2 Heat exchanger

Requirements for the heat exchanger are enough cooling capacity and small footprint and liquid-to-liquid technology. Water in a closed secondary loop will be cooled with a primary coolant which is cold water. The mill is near a big water source (Baltic sea) so there always will be water available for cooling. Therefore liquid-to-liquid heat exchanger is the most reasonable option. Liquid-to-liquid heat exchangers are the most common type of heat exchanger and it does not limit options. Tubular and plate heat exchangers are very common in industrial cooling applications. Based on calculations and discussions in the previous chapter the maximum heat transfer rate needed is around 200kW. It is rather small in industrial size and many types of heat exchangers are capable of it. The pilot-plant will be constructed in an old warehouse building. Therefore, space inside the building is limited and all equipment must be designed to fit inside. Another factor is the purity thing. The operating area is intended to be kept as clean as possible that means footprint design must be done carefully. Plate and frame heat exchangers offer smaller footprint than tubular heat exchangers with same cooling power. That is the main reason to choose the plate heat exchanger instead of tubular. Thus, based on information given this far the plate heat exchanger size about 200kW is the way to go.

8.4.3 Other equipment

The main equipment beside the heat exchanger and the tank is a pump. The pump provides needed pressure for the cooling units of furnaces. Mass flow of water is not very high so various types of pumps are suitable. Centrifugal pumps are commonly used in industrial because those are inexpensive, reliable and the working principle is quite simple. The centrifugal pumps also provide stable water flow and a throttling valve can be used to control the flow. Thus, the centrifugal pump is a good choice for this cooling system. Final decision for the pump unit will be made in the future. There will most likely to be two pumps to enhance usability, so the process does not need to stop if the running pump breakdown.

Valves are needed to control water flows. Each pipeline needs its own valve to control mass flow and to prevent unnecessary water flow through the furnace when it is not running. Fail-safe valves are needed for power failure situations. One which opens the pipeline to drain where heated water is guided and one which opens the pipeline from back-up pumps to tank. Cooling system will be equipped with enough measurements that provide needed information. Temperature and pressure measurements are major measurements. The number and locations of the measurements will be decided later.

9 Conclusions

In summary, the answers to the research questions were found. The requirements for the nitrogen supply system were found and nitrogen consumption could be calculated quite well. The result was that a PSA-generator with the required safety devices would be the best solution for this project. The most important factors in the choice of technology were the required purity and total consumption. Negotiations on the details and price of the system were initiated with suppliers.

The exhaust gas system was challenging but the capacities were determined. The furnace manufacturers were selected as system suppliers and will make a more detailed design of the exhaust system based on the information obtained from the thesis. The thesis provided information to manufacturers on how much gas the combustion chamber should be able to treat.

The cooling water system is the simplest of the systems and does not require special components. Thus, it takes less time to construct the cooling water system and delivery times of the components are shorter compared to other systems. Thus, at this point, its design was mainly based on the calculation of the cooling capacities and doing rough design of the process. Safety factors was also taken under consideration. As a result, the cooling powers calculated in two different ways and a description of the system were obtained. The result of these can be utilized in the future.

Designing auxiliary systems for a pilot-plant projects is a bit different than it is in normal industrial situations. Two major differences are confidentiality and the uniqueness of the project. The objective is to build a plant which is first of a kind. Therefore, the information about the process is kept a secret. The suppliers only have the information that is crucial for designing. That affects how accurate offers they can make. Uniqueness makes designing a little harder because there are no references to consult. Furnace manufacturers have their own setups, but nobody has built a plant with several furnaces from different manufacturers running as a unit. It is obviously more efficient to build one auxiliary system for furnaces rather than every furnace having their own. Thus, gathering information and investigating requirements for every furnace were key steps in this project. Another factor differentiating a pilot-plant designing from normal industry designing is the life cycle of the plant. The objective of a pilot-plant is to perform test runs and ensure that the technology is working as assumed and the product is what is expected. Once that is done the technology will be scaled up to an industrial level and the pilot-plant is abandoned. Thus, the life cycle of a pilot-plant is shorter than it is in a normal industry investment project. It is a good factor to keep in mind when designing and choosing equipment. Designing must be done carefully and the right equipment have to be chosen, but you can, for example, choose an inexpensive solution with a 10 years' expected life cycle rather than an expensive one with a longer life cycle.

On the other hand, auxiliary systems, in addition to exhaust gas systems, are basic systems in the industry. The designed cooling water system is no different from a normal industrial cooling system. It is a simple closed loop circuit and all components are basics for industrial cooling system. The back-up-water system was designed to be the simplest possible solution for this plant. Nitrogen is widely used in industrial applications and generating it on site is normal in places where its consumption is continuous and high. Therefore, designing the nitrogen system for this project was almost the same as it is for any project. The only difference was that suppliers do not get as much information as they usually get. Objective of the project organization was set requirements for the system as exact as possible, so that the supplier needed to know as little as possible about the process. Difficulties were related to the lack of information about nitrogen consumptions and the requirements from the furnace manufacturers. That was because the furnaces had just been bought and the specified technical information was to be given in the future. However, it is not possible to do test runs without nitrogen supply. Thus, the nitrogen supply system had to be available right from the beginning. Designing and building nitrogen supply systems takes several months so it was important to get the requirements for the system soon as possible. Therefore, some assumptions and over-sizing had to be done with the system. Overall, the chosen system should be suitable for the process. Both the literature and supplier recommend a described nitrogen supply system for this kind of a process. Exhaust gas treatment system was a challenging part. Basically, the

combustion chamber for the exhaust is quite simple and has no difference to other combustion chambers. However, calculating the amount of gas coming to the chamber was difficult. The amount of gas depends on the properties of the input product and heating conditions, so manufacturers cannot give exact values for the amount. Therefore, designing is based on laboratory scale tests. Exhaust gas system does not affect the product properties and its objective is to burn and lead gases safely to the atmosphere. Thus, there is no harm if the combustion chamber is a little oversized.

10 Discussion

The assignor, Stora Enso is a big world-wide company with a large research division. The project behind this thesis had started much earlier. The objective of the thesis was providing new information to the project and thus it is truly connected to the project. Because the study is part of the real project, all of the used material is from real suppliers. This material can be assumed to be accurate and it was utilized as the main source of material for the study. This provides the basis for the reliability of the study. In addition, information obtained from auxiliary system suppliers was utilized in the search for the right solution. It has ensured that the information on technologies presented in the thesis are up-to-date and correct. The information from the suppliers followed the information from literature sources. Therefore, it can be assumed that the literary sources chosen for the thesis are reliable. The limited time has an impact on the reliability of the study. There was no time to wait for more specified information about some equipment and some assumptions had to be made.

Those assumptions affected calculations especially in the designing process of the cooling water system. Therefore, the input data was not precise, and the results were only indicative. Because the project was a pilot plant, there was no references to which the results could be compared. Thus, it was necessary to calculate the cooling power in two different ways. The results of the calculations can be used to reliably state that the practical cooling capacity requirement is somewhere between the

minimum and maximum values. Even the theorical maximum values (results of the calculation 1) are rather small in the industrial scale because the furnaces are pilot-scale equipment thus energy losses will not be very high if the cooling water system is designed based on these values.

Pilot-plants are unique and built to test the technology and equipment. If the results from pilot-plant are promising the next phase is building an industrial scale plant. Then it would be a good time to do further research. Scaling the plant to an industrial scale affects the designing of the auxiliary systems. The systems now designed for the pilot-plant may not be the best solutions for a bigger plant. For example, bigger furnaces consume more nitrogen and fractional distillation could be better solution for the bigger plant. The cooling water system would be under closer examination because as the water consumption rises, more attention needs to be paid to energy efficiency.

References

Anode Materials. N.d. Article on Targays' website Accessed on 15.2.2020. Retrieved from https://www.targray.com/li-ion-battery/anode-materials

Agrawal, R. & Herron, D. 2000. *Air liquefaction: distillation*. Allentown: Air Products and Chemicals, Inc.

BREF Cooling. 2001. Integrated Pollution Prevention and Control (IPPC) Reference Document on the application of Best Available Techniques (BAT) to Industrial Cooling Systems.

https://eippcb.jrc.ec.europa.eu/reference/industrial-cooling-systems

Carvalho, E. P., Mota, F. A. S. & Ravagnani, M. A. S. S. 2015. *Modeling and Design of Plate Heat* Exchanger. Heat Transfer Studies and Applications. IntechOpen. ISBN: 978-953-51-6381-7. Accessed on 10.3.2020. Retrieved from <u>https://www.intechopen.com/books/heat-transfer-studies-and-applications/modeling-and-design-of-plate-heat-exchanger</u>

Cengel, Y. A. & Ghajar, A.J. 2015. *Heat and mass transfer- Fundamentals & applications*. 5th ed. New York: McGraw-Hill education

Chapter 30 – Once-through cooling. N.d. Chapter from Handbook of Industrial Water Treatment on Suez's website. Accessed on 7.3.2020. Retrieved from <u>https://www.suezwatertechnologies.com/handbook/chapter-30-once-through-cool-ing</u>

Chapter 32 - Closed Recirculating Cooling Systems. N.d. Chapter from Handbook of Industrial Water Treatment on Suez's website. Accessed on 7.3.2020. Retrieved from https://www.suezwatertechnologies.com/handbook/chapter-32-closed-recirculat-ing-cooling-systems

Chapter 23 - Cooling Water Systems-Heat Transfer. N.d. Chapter from Handbook of Industrial Water Treatment on Suez's website. Accessed on 7.3.2020. Retrieved from <u>https://www.suezwatertechnologies.com/handbook/chapter-23-cooling-water-systems-heat-transfer</u>

Dobreva, M. 2018. *How Do PSA Nitrogen Generators Work? – Pressure Swing Adsorption*. Article on Peak Scientific's website. Accessed on 5.2.2020. Retrieved from <u>https://www.peakscientific.com/blog/how-do-nitrogen-generators-work-pressure-</u> <u>swing-adsorption-psa/</u>

Faruk, O. & Sain, M. 2015. *Lignin in Polymer Composites*. Elsevier Science. ISBN: 9780323355667 Accessed on 16.3.2020. Retrieved from https://www.scribd.com/book/288390168/Lignin-in-Polymer-Composites

Generating Nitrogen with Pressure Swing Adsorption (PSA) Technology. 2017. Article on Atlas Copco compressors' website Accessed on 3.2.2020. Retrieved from https://www.atlascopco.com/en-us/compressors/wiki?utm_source=undefined&utm medium=undefined&utm campaign=undefined

Global Energy Storage Market 2019. 2019. Report from lux research company. Accessed on 8.4.2020. Retrieved from

https://www.luxresearchinc.com/global-energy-storage-market-forecast-executivesummary

Global Forest, Paper & Packaging Industry Survey. 2016. Report by PwC. Accessed on 8.4.2020. Retrieved from

https://www.pwc.com/gx/en/industries/assets/pwc-annual-fpp-industry-survey-2016-10.pdf

How Is Nitrogen Produced for Industrial Applications? 2018. Article on Generon's website. Accessed on 17.2.2020. Retrieved from https://www.generon.com/how-is-nitrogen-produced-for-industrial-applications/

Häussinger, P., Leitgeb, P. & Schmücker, B. 2000. Nitrogen. In Ullmann's Encyclopedia of Industrial Chemistry

Ivanova, S. & Lewis, R. 2012. Producing Nitrogen via Pressure Swing Adsorption. Article on American Institute of Chemical Engineers' website. Accessed on 10.2.2020. Retrieved from

https://www.aiche.org/resources/publications/cep/2012/june/producing-nitrogenpressure-swing-adsorption

Kiertotalouden kiinnostavimmat [The most interesting in the circular economy]. N.d. List of companies with the circular economy products made by SITRA. Accessed on 2.5.2020 retrieved from:

https://www.sitra.fi/hankkeet/kiertotalouden-kiinnostavimmat/

Kuinka sellua valmistetaan [how pulp is made] N.d. Article on UPM's website. Accessed on 6.4.2020. Retrieved from https://www.upmpulp.com/fi/vastuullinen-sellu/kuinka-sellua-valmistetaan/

Kykkänen, V. & Veteli, A. 2019. Stora Enso investoi 10 miljoonaa euroa koelaitokseen Kotkassa – yhtiö tähtää akkumarkkinoille [Stora Enso invested EUR 10 million in a pilot plant in Kotka - the company aims to the battery markets]. Article on YLE's website. Accessed on 11.1.2020. Retrieved from https://yle.fi/uutiset/3-10885254

LignoBoost process. N.d. Process illustration on Valmet's website. Accessed on 4.2.2020. Retrieved from

https://www.valmet.com/pulp/other-value-adding-processes/lignin-separation/lignin-uses/

Mikä ihmeen kiertotalous? [What on earth is the circular economy] N.d. Article on Confederation of Finnish Industries website. Accessed on 2.5.2020 retrieved from: <u>https://ek.fi/syty-kiertotaloudesta/mika-ihmeen-kiertotalous/</u>

Nitrogen gas applications. N.d. Article on omega air's website. Accessed on 17.2.2020. Retrieved from https://www.omega-air.si/news/news/nitrogen-gas-applications

Pattabathula, V. & Richardson, J. 2016. *Introduction to Ammonia Production*. Article on American Institute of Chemical Engineers' website. Accessed on 19.2.2020. Retrieved from

https://www.aiche.org/resources/publications/cep/2016/september/introductionammonia-production

Stora Enso in 2019. 2019. Summary of the annual report 2019. Accessed on 13.4.2020. Retrieved from https://www.storaenso.com/en/download-centre

Sunila mill. N.d. Information about Sunila mill on Stora Enso's website. Accessed on 13.1.2020. Retrieved from https://www.storaenso.com/en/about-stora-enso/stora-enso-locations/sunila-mill

Tillaeus, J. 2020. *Metsäjätti rakentaa uutta koelaitosta vanhaan varastorakennukseen Kotkassa: "Tässä mennään tulevaisuuden asioihin"* [The forest industry giant is building a new pilot plant in an old warehouse in Kotka "This is the things of the future"] Article on YLE's website. Accessed on 13.1.2020. Retrieved from <u>https://yle.fi/uutiset/3-11168372</u>

Typen käyttöturvallisuustiedote [Safe data sheet nitrogen] 2015. Woikoski. Accessed on 20.2.2020. Retrieved from http://www.woikoski.fi/sites/default/files/typpi.pdf

Zohuri, B. 2017. *Heat Exchanger Types and Classifications*. Chapter from book Compact Heat Exchangers. Accessed on 6.3.2020. Retrieved from https://www.researchgate.net/publication/308464270 Heat Exchanger Types and Classifications

Wool, R. & Sun, X.S. 2011. *Bio-Based Polymers and Composites*. Academic Press. ISBN:9780080454344 Accessed on 26.3.2020. Retrieved from https://www.scribd.com/book/282478093/Bio-Based-Polymers-and-Composites

Appendices

Appendix 1. Exhaust gas flow calculations

Appendix 2. Calculation 1. Heat flow to cooling water

Appendix 3. Calculation 2. Heat flow to cooling water

Appendix 4. Cooling power calculations