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**RISK ASSESSMENT OF HYDROTHERMAL LIQUEFACTION TEST EQUIPMENT**  
Using Failure Mode and Effects Analysis

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# ABSTRACT

Elias Saarinen: Risk assessment of hydrothermal liquefaction test equipment using failure mode and effects analysis  
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The depletion of fossil fuels and the air pollution caused by them have led researchers to look for alternative and more sustainable methods to replace them. One of the most promising methods for this is hydrothermal liquefaction (HTL) which can be utilized to produce biofuels with good efficiency even from undried biomass. This process takes place at a high pressure and at a moderately high temperature, allowing the biomass to be converted into bio-oil in less than an hour.

The purpose of this work is to perform a risk assessment for the hydrothermal liquefaction test equipment of the Efficient Hydrothermal Applications project. In addition, the basic principles of hydrothermal liquefaction technology and legislation related to HTL test equipment in Finland are introduced. The work uses a failure mode and effects analysis (FMEA) as a risk assessment method, as it is well suited for the equipment in question. In the failure mode and effects analysis, the system under consideration is divided into components for which failure modes are formed, after which the causes and effects of these are identified. The method can also be combined with a criticality assessment to assess the significance of the risks, as well as the determination of protective measures, as has been done in this work.

The risk assessment was conducted as a telemeeting with my bachelor supervisor Tero Joronen along with Mika Karttunen and Antti Nuottajärvi, who are also involved in the project. In accordance with the failure mode and effects analysis, the equipment was reviewed component by component at the meeting. Possible fault forms were developed for the components and the causes and most significant consequences of these faults were determined. In addition, the meeting assessed the magnitudes of the risks of the faults and devised effective safety measures to reduce the risks.

The assessment was recorded on an assessment form which can be found in the appendices of the work. It was used to determine the results of the risk assessment. In general, the risks of the equipment are quite small or well controlled. A substantial reason for this is the unmanned process container while the process is running. This significantly reduces the risk to humans, such as the effects of high temperatures or leakage of corrosive substances. Additionally, investments are made in component design and high-quality materials. However, significant failure modes can still occur for the equipment. For example, clogging of the feed pump, solidification of slurry in the preheater and sensor failure in the automation system are quite notable risks. These risks can be mitigated by protective measures, including feed pre-grinding, selection of suitable feeds and an alarm system which warns of process container conditions.

**Keywords:** failure mode and effects analysis, FMEA, hydrothermal liquefaction, HTL, risk assessment, biofuel

# TIIVISTELMÄ

Elias Saarinen: Riskienarviointi hydrotermisen nesteytyksen testilaitteistolle käyttäen vika- ja vaikutusanalyysiä  
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Fossiilisten polttoaineiden väheneminen ja niiden aiheuttamat ilmansaasteet ovat saaneet tutkijat etsimään vaihtoehtoisia ja kestävämpiä menetelmiä korvaamaan niitä. Yksi lupaavimmista menetelmistä tähän on hydrotermisen nesteytyksen (HTL), jolla pystytään tuottamaan biopolttoaineita hyvällä hyötysuhteella myös kuivaamattomasta biomassasta. Tämä prosessi tapahtuu korkeassa paineessa ja melko korkeassa lämpötilassa, jolloin biomassasta saadaan muutettua bioöljyksi alle tunnissa.

Työn tarkoituksena on suorittaa riskienarviointi Efficient Hydrothermal Applications -projektin hydrotermisen nesteytyksen testilaitteistolle. Lisäksi työssä tutustutaan hydrotermisen nesteytyksen teknologian peruseräisiin ja HTL-testilaitteistoon liittyvään lainsäädäntöön Suomessa. Työssä riskienarviointimenetelmänä käytetään vika- ja vaikutusanalyysiä (VVA), sillä se sopii kyseiselle laitteistolle hyvin. Vika- ja vaikutusanalyysissä tarkasteltava systeemi jaetaan komponentteihin, joille muodostetaan vikamuotoja, minkä jälkeen tunnistetaan näiden syyt ja vaikutukset. Menetelmään voidaan yhdistää myös kriittisyysanalyysi arvioimaan riskien merkitystä, sekä suojatoimenpiteiden määritys kuten tässäkin työssä on tehty.

Riskienarviointi suoritettiin etäkokouksena kandidohjaajani Tero Jorosen sekä projektissa myös mukana olevien Mika Karttusen ja Antti Nuottajärven kanssa. Kokouksessa käytiin vika- ja vaikutusanalyysin mukaisesti laitteisto läpi komponentti kerrallaan. Komponenteille kehiteltiin mahdollisia vikamuotoja ja näiden vikamuotojen syyt ja merkittävimmät seuraukset määritettiin. Lisäksi kokouksessa arvioitiin vikamuotojen riskien suuruuksia ja keksittiin riskien pienentämiseksi tehokkaita turvatoimenpiteitä.

Analyysi kirjattiin analyysilomakkeelle, joka löytyy työn liitteistä. Sen avulla riskienarvioinnin tulokset saatiin määritettyä. Yleisesti ottaen laitteiston riskit ovat melko pieniä tai ne ovat hyvin hallinnassa. Suuri syy tälle on ajon aikana miehittämätön prosessikontti. Tämä vähentää merkittävästi ihmisiin kohdistuvia vaaratekijöitä, kuten korkean lämpötilan tai syövyttävien aineiden vuotamisesta aiheutuvia haittoja. Lisäksi komponenttien suunnitteluun ja laadukkaisiin materiaaleihin panostetaan. Kuitenkin laitteistolle voi koitua merkittäviäkin vikamuotoja. Esimerkiksi syötepumpon tukkeutuminen, lietteen pohjaan palaminen lämmittimessä ja anturivika automaatiojärjestelmässä ovat suuruudeltaan melko huomattavia riskejä. Näitä riskejä voidaan vähentää suojatoimenpiteillä, joita ovat muun muassa syötteen esijauhanta, sopivien syötteiden valinta ja prosessikontin olosuhteista varoitava hälytysjärjestelmä.

Avainsanat: vika- ja vaikutusanalyysi, VVA, hydrotermisen nesteytyksen, HTL, riskienarviointi, biopolttoaine

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APPENDIX A: FMEA FORM

# LIST OF SYMBOLS AND ABBREVIATIONS

EHTA	Efficient HydroThermal Application
FMEA	Failure mode and effects analysis
FTA	Fault tree analysis
HAZOP	Hazard and operability study
HTL	Hydrothermal liquefaction

# 1. INTRODUCTION

Hydrothermal liquefaction (HTL) is a process used to convert biomass into bio-oil. The first steps in the development of this technology were made in the 1920s when early experimental data supported the possibility of producing oil from biomass in hot water. The initial research into hydrothermal liquefaction started in the United States after the oil embargo of 1974. Even though further research has been conducted after that, the technology is still at the development stage. [1, pp. 1792]

This work will focus on HTL pilot plant of the Efficient HydroThermal Application (EHTA) and will conduct a risk assessment of it by using failure mode and effects analysis (FMEA). A previous risk assessment of the plant has been conducted using hazard and operability study (HAZOP) but this work will be a separate assessment from it. Thus, the objective of this work is to present the basic functions of HTL test equipment and to discover risks using FMEA. In addition, the study will introduce several ways of reducing the risks or limiting their effects.

At first, general subjects will be reviewed that relate to conducting safety assessments such as relevant legislation and some of the most common methods of risk assessments that are used in process industry. Chapter 3 will present an overview of hydrothermal liquefaction as a technology but not explore the subject too thoroughly. The main reason for this thesis is the risk assessment using FMEA and it will be conducted in chapter 4. To conclude this work, some conclusions will be presented to summarize the findings of this study.

## **2. SAFETY ASSESSMENT**

One of the most important and effective ways to improve safety in work environments and industrial processes is conducting safety analyses and using the results gained from those analyses to make risk reducing decisions. Risks are a part of every industrial process, and that is why analyses should be conducted regularly to decrease them. Safety assessments should be an integral part of process design through its whole life cycle to prevent accidents and other hazards that are caused by poor process management and planning. The next chapter will review relevant restrictions and requirements imposed by legislation on the test equipment being examined in this study.

### **2.1 Legislation**

There are many laws that need to be considered when designing, building or using an industrial plant of any sort. The HTL test plant uses high pressures and moderately high temperatures in its processes which increases the requisite safety requirements. Moreover, there are additional laws that affect HTL bio-oil production due to the usage of different kind of wastes and substances as feed, as well as the production of biocrudes by the plant. [2]

These laws are there to improve occupational safety and to protect the environment. Nowadays, people are starting to pay more attention to environmental issues and that is why companies need to operate ecologically to succeed. In the next chapters some relevant and valid Finnish laws will be presented to display what kind of requirements they have imposed on present HTL test plants and future commercial HTL plants.

#### **2.1.1 Occupational safety and health act**

Occupational safety and health act (738/2002) is a law that defines the basic principles of work safety matters in Finnish legislation. The act deals mostly with obligations of employers and employees in contractual relationships and also with cooperation of the aforementioned parties related to improving work safety. [3]

Employers are required to take care of safety and health of their employees with necessary measures. Employers must consider circumstances related to work, working conditions, working environment and employees' personal capabilities. Taking the nature of the work into account, employers are required to systematically and adequately analyze

and identify hazards and risk factors caused by work, workspace, other aspects of the working conditions and working environment. If the hazards and risk factors cannot be neutralized the consequences to the employees' health and safety need to be assessed. [3]

Employers are required to give employees necessary knowledge of the hazards and risk factors of the workplace and, taking the individual factors of the employees into account, ensure that the employees receive an adequate orientation to the work and instructions and guidance in order to eliminate the hazards of the work and avoid any risk that could endanger safety and health. In addition, instruction and guidance are to be given to employees about disturbances and exceptional situations. [3]

Occupational safety and health act instructs employers and employees to cooperate in maintaining and improving work safety. Employers are required to in good time give employees information on any factors that impact health and safety in workplace and employees in turn have the right to submit proposals on health and safety in the workplace to the employer. Employees also have the obligation to exercise such care and caution in their work that is necessary for maintaining health and safety. [3]

The safety of machinery, work equipment and other devices is also regulated by this act. Only such devices that are suitable for the work and working conditions may be used at work. Regarding this, employees have the obligation to inform the employer and the occupational safety and health representative of faults and defects they have detected in the machinery, work equipment or other devices which may cause harm to the employees' health or safety. [3]

### **2.1.2 Environmental protection act**

The purpose of the environmental protection act (527/2014) is to prevent the contamination of the environment, prevent and reduce emissions and prevent environmental damage. Promotion of sustainable use of natural resources and reduction of the amount and harmfulness of waste along with prevention of adverse effects caused by waste are also part of the purpose of this act. [4]

Activities that pose a risk of environmental pollution require an environmental permit that is granted by the state permit authority. However, a permit is not necessary for an experimental short-term activity when the purpose is to test an emerging technology, a manufacturing method or raw material of fuel. In situations like this, a written notification of such activity must be submitted to the environmental permit authority at the latest 30

days before the start of the operations. [4] This section also applies to EHTA's HTL test plant.

Environmental protection act obligates operators to be aware of the environmental risks and impacts of their operations and of ways to mitigate adverse effects of these operations. Operators also need to have knowledge of the management of risks and impacts. [4]

Operators are required to organize their operations in such a way that environmental pollution can be prevented or at least diminished as low as possible beforehand. In case of activities that pose a risk of environmental pollution must adhere to the general obligations and principles of the Waste act and the Chemicals act. [4]

### **2.1.3 Waste act**

The purpose of the act (646/2011) is to prevent hazard and harm to health and environment induced by waste and waste management, to reduce waste and its harmfulness and to further the sustainable use of natural resources [5]. HTL technology fulfills this purpose by utilizing waste and turning it into usable products, therefore reducing waste and realizing the sustainability requirement.

This act applies to waste, waste management and to activities and products generating waste. Waste is defined as substance or object which is discarded, will be discarded or is required to be discarded. One of the most relevant definitions detailed by this act regarding HTL is waste recovery. Recovery of waste means any operation whose principal result is using waste in production facilities or elsewhere in the economy in such a manner that it replaces other materials or objects which would have otherwise been used for that purpose. [5]

Because HTL plants produce different kinds of waste, those engaged in production must be aware of the waste originating from their production. They are also required to recognize the health and the environmental impacts of their waste and the possibilities of developing their production in such a way as to reduce the amount and harmfulness of waste. [5]

### **2.1.4 Chemicals act**

The purpose of the act (599/2013) is the protection of health and environment from the hazards and harms caused by chemicals. The law defines also the general principles

governing the operation and the obligations of the operator [6]. Because some alkaline catalysts can be used in HTL processes, they are subject to this act [7, pp. 2335–2337].

During activities that involve the use of chemicals, the operators need to be sufficiently aware of the chemical's consequences to health and the environment. Due care must be taken to prevent harm to health and the environment by taking into account the amount and hazardousness of the chemical. One of the general principles is also that, where reasonably possible, the least hazardous chemical or method should be selected from the possible chemicals and methods to prevent the harm caused by chemicals. [6]

### **2.1.5 Pressure equipment act**

This act (1144/2016) applies to all kinds of pressure equipment. For the purposes of this act, pressure equipment means a tank, piping or other technical unit where there is over-pressure, or it may develop. Technical units intended for the protection of pressure equipment are also defined as pressure equipment. [8]

The purpose of the act is to ensure the safety of pressure equipment throughout its life cycle [9]. Pressure equipment are required to be designed, manufactured, maintained, operated and inspected in such a way that it does not endanger the health, safety or property of anyone. In addition, pressure equipment must have adequate safety devices and equipment systems and they need to function properly. [8]

The owner and the possessor must position the pressure equipment in a way that minimizes the risks during damage or malfunction and that the pressure equipment can be used, inspected and maintained appropriately. Also, it must be ensured that the spaces and structures surrounding the pressure equipment are designed and constructed in such a way that the risks during damage or malfunction are kept to a minimum. [8]

## **2.2 Methods of risk assessment**

There are many different risk assessment methods which all have their own characteristics and uses. Although basic guidelines for risk assessments are usually the same, many of the different methods can be used for certain situations or to accomplish certain results which could not be acquired with another method [10, pp. 3]. To better understand risk assessment methods, defining some of the most relevant concepts relating to them is useful.

To start with, the difference between the terms hazard and risk must be defined. Hazard is the potential for harm or an adverse effect to people's health, to the environment or to

the equipment or property of organizations. On the other hand, risk is the combination of the probability of the occurrence of the harm and the severity of it. [11] In risk assessments, risk levels are determined by these two properties of the harm [12, pp. 6].

Risk assessment is a significant part of risk management and it can be roughly divided into 3 phases. The first phase is hazard identification where hazards are found, listed and characterized. The second phase is risk analysis which is a process for comprehending the nature of hazards and determining the level of risk. The level of risk is ascertained with risk estimation. The final phase of risk assessments is risk evaluation where the significance of the risk is determined by comparing the estimated risk against given risk criteria. [13]

The subsequent process of risk management after risk assessment is risk control where appropriate ways to eliminate or at least reduce the risk are determined [13]. Risk control is covered in this chapter because it is often included in risk assessment methods even though it is not officially part of risk assessment process. Risk control phase typically includes risk-related decision making, implementation of those decisions and monitoring of the implemented actions to examine the effects of the risk reducing decisions [14].

In the following chapters a few of the most notable risk assessment methods will be showcased. More precisely the methods are hazard identification methods which is a subclass of risk assessment methods. These methods are suitable for detailed examination of defined objects, functions and working stages. [10, pp. 5]

### **2.2.1 Failure mode and effects analysis**

The objective of failure mode and effects analysis is to identify potential hazards caused by equipment or system defects. FMEA can be used to determine the reliability of a given system especially well [15, pp. 119]. The basic function of FMEA is to divide the system under review into smaller components or subsystems and after that each component's failure modes are recognized and their causes and effects on the system are identified. [10, pp. 14]

FMEA can be performed by a single analyst who is familiar with this method. FMEA starts with establishing the scope of the analysis which helps the analyst to focus on certain parts of the system more reliably [15, pp. 121]. After that, the division of the system into sufficiently simple components or subsystems is carried out. The system components' functions in the system should be discussed to help understand the results of the FMEA [15, pp. 121–122]. The components and subsystems must have potential failure modes

for them to be included in the analysis. Next the failure modes for each system component are defined and for each failure mode the causes and effects of the failure are determined. After that, some suggested improvements to the components or subsystems can be thought out to reduce the effects of the failures. All the data gathered in the analysis is recorded on an analysis form. [10, pp. 14]

FMEA can also include a criticality assessment. In the assessment the criticality of the effects of a failure are estimated to determine the harmfulness of these effects to personnel, the environment or the system. [15, pp. 123] A risk matrix can be used to determine the level and significance of the risks [13].

FMEA offers great versatility in a risk assessment process because the scope of the analysis can be freely chosen to fulfill the demand of the risk assessment [15, pp. 132]. However, FMEA also has some limitations and drawbacks. It is not well-suited to analyze large and complex technological systems. The method does not consider the effect on failures that maintenance work and people have. In addition, failures are only examined independently which makes concurrent failures hard to analyze. [10, pp. 14]

### **2.2.2 Hazard and operability study**

The hazard and operability study is a technique used to examine potential deviations of operations from design conditions that could create hazards or operating problems. HAZOP was originally developed for the petrochemical industry to try to reduce the risks of a major incident occurring. Since then the method has become the most utilized process hazard analysis method in the world. [15, pp. 167] In addition to chemical processes it can be used for material flow examination, product analysis and many other applications [10, pp. 8].

When conducting a HAZOP, the complexity and scope of the subject system needs to be taken into account. The study is led by a leader who gathers a group consisting of experts from different fields. A considerable amount of work and in-depth knowledge of the system is required for a successful analysis. [10, pp. 9] After the scope has been set, the design intention for the system is formulated. The design intention includes the intended operations for all the system components which makes it easier to recognize deviations from the process parameters. Next, special guidewords are combined with the parameters to develop meaningful deviations. Guidewords can be words such as no, less, more, higher, slower or any word like that. Some of the essential parameters are for example flow, pressure, temperature, chemical composition or fluid level. Each mean-

ingful deviation is analyzed separately and for each causes and consequences are identified. Furthermore, safeguards are evaluated to clarify their adequacy and to decide based on that if a change or further study is required. [16, pp. 13–19]

One of the biggest limitations this method has is its major need for resources. HAZOP gives information only of hazards that relate to the environment or failures of equipment. On the other hand, hazards relating to human activities or work assignments are more difficult to analyze. Also, this method does not consider complex chains of events and it is not suitable for examining standardized systems. [10, pp. 8–9]

### **2.2.3 Fault tree analysis**

The objective of fault tree analysis (FTA) is to find component failures and failure combinations that influence chosen system faults. FTA is a deductive method which can be used to examine the possibilities of serious accidents. This method also enables the quantitative comparison of alternative solutions. [10, pp. 20] FTA is a “top-down” analysis because it starts with selecting a top event for which different failure paths are identified by use of a fault tree drawing. A fault tree consists of events and logical operators that connect different events. An event is either a system operation or a component failure. [17, pp. 156]

The top event can be any problem, accident or damage relating to the system [10, pp. 20]. After the selection of the undesired top event, the immediate events that can in some way cause the top event are determined. The causes are examined in turn for these lower events and the process is repeated to the more detailed lower levels. These levels form a branching fault tree that illustrates every way an undesired event can occur. [17, pp. 159]

The most important phase of FTA is the definition of the top event. Flaws in this can affect the results of FTA negatively. It is important to understand that this method does not give an overall view of the level of security in the system under review. Also, dependencies and external factors of faults are difficult to take into account during FTA. Additionally, fault tree analysis is not suitable for analyzing the consequences of the top event [10, pp. 20] and the construction of fault trees can require a lot of effort and become expensive [17, pp. 165].

### 3. HYDROTHERMAL LIQUEFACTION TECHNOLOGY

Increasing energy demand and urbanization in the past decades have raised concerns over finding an alternate source for energy production that is both sustainable and can be utilized as a substitute for traditional fossil fuels. One such source is biomass which is a versatile and a renewable energy source that can be used in fuel production and as a feedstock for chemical manufacturing for the petrochemical industry. Bio-oil produced from biomass is one of the more promising products for the future. Still, world biofuel production has been mainly centered around the first generation biofuels (bioethanol and biodiesel) and only a small percentage of biofuels are produced from lignocellulose materials such as wood and waste biomass. [2, pp. 1378]

To be usable as a fuel, biomass needs to be converted from its solid state to liquid which is not a spontaneous process. Many different conversion technologies have been developed for converting varying kinds of biomass into liquid fuels. These technologies can be roughly divided into biochemical and thermochemical conversion methods. In general, thermochemical methods are much faster than biochemical methods. Thermochemical conversions are processed at much higher temperatures than biochemical conversions and appropriate catalysts are used to increase biofuel yields. Thermochemical conversion methods can be categorized for example into direct combustion, pyrolysis, gasification and liquefaction. Among these methods, hydrothermal liquefaction is estimated to have the most potential in producing sustainable biofuels. [2, pp. 1378–1379]

Hydrothermal liquefaction is a synonym for hydrous pyrolysis but HTL technology uses much lower temperatures and heating rates compared to pyrolysis [2, pp. 1379]. HTL is typically carried out in temperatures between 280–370 °C and pressures between 10–25 MPa but the process conditions can also differ from these values. In these conditions water is still in a liquid state and acts as reactant, solvent and catalyst in the HTL process. Both subcritical and supercritical water can be used in HTL but most of the time subcritical conditions are preferred. [7, pp. 2328] When biomass is processed in a hot and pressurized water environment for some time biomass starts losing its solid polymeric structure and breaks down to mainly liquid components [2, pp. 1379]. When compared to pyrolysis, HTL process can be utilized without drying the biomass first because the water in the biomass can be used as a component in the process [7, pp. 2329]. In addition, low operating temperatures, low tar yield and high energy efficiency when compared to pyrolysis are some of the other advantages of HTL [2, pp. 1379].

### 3.1 Biomass and possible feedstocks

Biomass comprises of carbon, hydrogen, nitrogen, sulphur and oxygen and the ratios of these elements affect the HTL process and the product biofuels. During combustion carbon is converted into CO<sub>2</sub> and it contributes to the overall heating value of the biomass the most. Wood biomass has higher carbon content than herbaceous biomass. Hydrogen is the other major element that contributes to the heating value. During combustion it converts to water. Similarly, woody biomass has slightly higher content of hydrogen than herbaceous biomass. Nitrogen and sulphur are also oxidized during combustion. Oxygen on the other hand does not contribute to the heating value of the biomass. Herbaceous biomass has both higher contents of nitrogen and sulphur when compared to wood biomass. High oxygen contents in biomass is problematic because it lowers the quality of biocrudes obtained from HTL processes. [2, pp. 1379]

At a less precise level biomass consists of carbohydrates, lignin, lipids and protein. Carbohydrates can be further divided into cellulose, hemicellulose and starch. Cellulose is a long chain polysaccharide that has a high molecular weight and it has a high degree of crystallinity due to its hydrogen bonds. Cellulose is insoluble in water at ambient temperature and pressure. However, at subcritical conditions cellulose becomes soluble to water. [2, 7] Hemicellulose is a heteropolymer with amorphous structure that consists of various monosaccharides. Because of this random structure hemicelluloses are solubilized and hydrolyzed in water more easily than cellulose. [2, 7] Starch on the other hand is a polysaccharide that consist of glucose monomers. It is relatively easily hydrolyzed when compared to cellulose. [7, pp. 2331]

Lignin together with cellulose and hemicellulose is a significant component of plant material. It is an aromatic heteropolymer that is relatively hydrophobic. Because of this it dissolves into water quite poorly like cellulose. Lignin has a high energy content in comparison to the carbohydrates which leads to better heating value in the product biofuel. On the other hand, higher lignin content in the biomass increases the amount of solid residue. [7, pp. 2332]

Lipids in this case mean fats and oils that are insoluble in water at ambient temperatures. Again, at subcritical conditions they can be hydrolyzed even without added catalysts. Chemically they are triglycerides, in other words triesters of fatty acids and glycerol. When hydrolyzed they form glycerol and fatty acids which have a high energy content. Finally, proteins are found primarily in animal and microbial biomass. Proteins are formed by amino acids that are linked together by peptide bonds. The peptide bonds are stronger than the bonds in cellulose and because of this high temperature is required to break the

bonds. In addition, due to the heterogeneous nature of amino acids degrading them is challenging. [7, pp. 2333–2334]

Possible sources of biomass for the sustainable use of HTL process in biofuel production are extensive. Potential feedstocks include sources such as wood, corn stalk, plastic, lignite, wastes, microalgae, macroalgae and black liquor. [2, 7, 18] For example, black liquor can be directly utilized as a feed for the HTL process to improve the efficiency of existing pulp mills. This could be done by directing the black liquor to an integrated HTL plant before it is concentrated by evaporation and burned in the recovery boiler of the mill. This would make the slurry more pumpable to the HTL process and avoid the evaporation step. [18, pp. 105]

Feedstocks can be divided into dry and wet feedstocks. Dry feedstocks are woody and have a high lignocellulosic content making them tough to process. Therefore, woody biomass requires grinding to reduce its particle size after which it can be used in slurry. Also, recovery and reuse of water is especially important in slurry preparation when using dry feedstock to reduce water consumption. [19, pp. 148] Although biofuel production using dry feedstocks is more researched, wet feedstock is more suitable to be used in HTL processes due to the required liquid process conditions. Algal biomass is estimated to be one of the most promising sources to produce biofuels using HTL. This is because microalgae are fast to grow, do not need arable soil and produce biomass with good photosynthetic efficiency. [2, pp. 1379] Microalgae contains a lot of lipids and additionally hydrocarbons and proteins which can all be formed into biocrude. Additionally, microalgae feedstock does not need drying prior to processing. It is however generally advantageous to increase the solid content of the microalgae to be able to process more biomass in a given volume and to reduce the energy required to preheat the process water. On the other hand, this consumes energy due to the dewatering process and can affect the pumpability of the feed slurry. [20, pp. 268–269]

### **3.2 Basic hydrothermal liquefaction mechanisms**

The basic HTL reaction mechanisms for any type of feedstock are depolymerization of the biomass, decomposition of biomass monomers and lastly recombination of reactive fragments. Depolymerization occurs due to the high pressure and temperature of the process. These conditions change the long chain polymers to shorter hydrocarbons releasing energy into the process water at the same time. This mimics the formation of fossil fuels in natural geological processes in smaller and faster scale. [2, pp. 1386]

After depolymerization happens the decomposition of the biomass. It can happen by dehydration, decarboxylation, deamination and cleavage. These reactions include the loss of water molecule (dehydration), the loss of CO<sub>2</sub> molecule (decarboxylation) and elimination of amino acids (deamination). The first two reactions advance the removal of oxygen from the biomass which better the product biocrude. In the final step reactive fragments repolymerize in a reverse way when compared to the first step due to the shortage of hydrogen compounds. High molecular weight char compounds are formed as a result of this. [2, pp. 1386–1387]

The HTL process produces a liquid biocrude that can be further hydrotreated to commercial grade bio-oils. Along with it some gaseous, aqueous and solid phase byproducts are formed as well. The process mechanism is defined for both dry and wet feedstock types. Both processes are similar with only a few differences. The HTL process for dry feedstock requires pretreatment as was mentioned in the chapter 3.1. After this the slurry is processed for approximately 15 minutes under the HTL process conditions that depend on the biomass being used. In these conditions phase separation happens spontaneously into the aforementioned phases. To improve the efficiency of the process, the aqueous phase can be recirculated to the process and the solids can be utilized for example as a fertilizer. [2, pp. 1387]

The biggest difference between wet feedstock and dry feedstock is that with wet feedstock the pretreatment step is usually not needed. On the other hand, excess water needs to be removed when using microalgae to improve the biocrude yield. The feed slurry after dewatering is usually around 20 % solids. Processing of algae biomass has great potential because most of the byproducts are recoverable. The aqueous phase and the separated nutrients can be recirculated back to the algae cultivation process. Additionally, CO<sub>2</sub> can be used for the photosynthesis reaction of the algae and the solids can be utilized as a fertilizer in this case too. This HTL process consumes only 10–15 % of the energy in the biomass which means that the energy efficiency of this process is between 85–90 %. Furthermore, the produced biocrude oil does not require much upgrading in comparison to other biomass sources. [2, pp. 1388]

Catalysts other than water are also used in HTL processes to reduce tar and char formation and to improve gasification efficiency. Homogenous catalysts such as alkali salts are used more frequently in HTL than heterogenous catalysts like nickel catalysts. The use of alkali salts can accelerate the water-gas shift and boost biocrude yields in addition to the other listed effects. Especially potassium salts were proved to increase the liquid yields significantly. Heterogenous catalysts are mostly used to improve gasification

which is needed in HTL to remove oxygen from the slurry. However, biocrude yields can diminish due to excessive gasification. [7, pp. 2335–2336]

### **3.3 Scale-up challenges**

Hydrothermal liquefaction is still very much a developing technology that is not used in a commercial setting on a large scale yet. Most HTL plants are in lab- or bench-scale. A few pilot plants exist such as hydrothermal upgrading (HTU) and Lawrence Berkeley Laboratory (LBL) [7, pp. 2328] but there are still major scale-up challenges that need to be solved for HTL to become a mature and competitive technology. The harsh process conditions and the characteristics of different biomass slurries cause problems. Corrosion is one such issue and to protect from it requires the use of expensive alloys which brings the investment costs up. [7, pp. 2328] Also, large-scale pumpability testing must be done to define optimal particle size of the slurry and its concentration. [21, pp. 9]

Other challenges include reactor design, heat transfer, depressurization, solids removal and biocrude/aqueous phase separation. When it comes to reactor design, capital costs need to be minimized by favoring efficient plug flow reactors instead of continuous stirred tank reactors that have been used in bench-scale applications [19, pp. 150]. Rapid liquefaction is needed to get the best products which means that heat transfer coefficients must be determined accurately to optimize the process. [21, pp. 9–12] Depressurization on the other hand is important because if a large-scale HTL plant had buildup pressure in some part of it with no way to depressurize it, the risks of this situation would be considerable. In the worst case an explosion could occur causing massive damage. [22] In the case of solids removal, backflush will improve it and filter pressure drop. Lastly, biocrude recovery aqueous needs to be improved. Continuous phase separation and product collection is also required when moving to commercial scale HTL plants. [21, pp. 12]

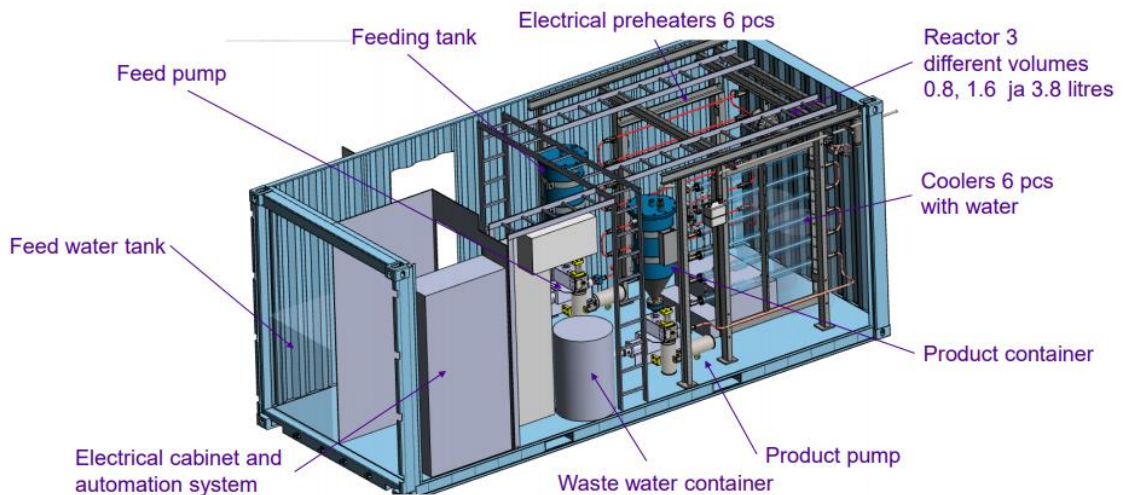
## 4. RISK ASSESSMENT USING FMEA

The subject of this risk assessment is EHTA's hydrothermal liquefaction testing facility. The test plant is not yet built so a visit to the plant was not possible during this assessment. This assessment is conducted with the help of industry professor Tero Joronen, research engineer Mika Karttunen and laboratory engineer Antti Nuottajärvi. An online meeting was arranged with the aforementioned people to discuss safety-related issues about the test plant. A FMEA worksheet was utilized to record the information gathered during the meeting. The worksheet can be found in appendix A.

The scope of the analysis is determined to start with. The testing facility can be divided into two different containers which are the office container and the process container. The process is managed and monitored from the office container and the process container is unmanned during runs and holds all the HTL equipment inside. This assessment focuses only on the process container and its system components.

### 4.1 Dividing the system into components

Next, the system should be divided into suitable components. The division can be performed using the figure 1.



**Figure 1:** HTL process container and its components

The division of components presented in figure 1 is good enough for the assessment's purposes. Before the assessment can be started it is essential to know what the function of every component is in the system.

The feeding tank is the first component in the system we have outlined. It contains the feed that is going to be used in the HTL process. From there the feed is pumped to the required pressure of over 300 bars with the feed pump. The feed pump needs to be able to pump feedstock that has a consistency of paste to high pressures reliably. After the feed pump pressurized water is mixed with the feed to accelerate the HTL process. The water comes from the feed water tank. The feed water tank is left out of the assessment due to it having little to no notable failure modes that could cause harm to anything.

After the mixing of water and the feed, the wet slurry goes to the preheaters where the slurry is heated to over 400 °C with electric heaters. From the preheaters the process slurry is directed to one of the reactors. The reactors are plug flow reactors where the residence time is 10 to 30 minutes depending on the composition of the slurry. After the reactor, the slurry has become liquefied product which contains biocrude, aqueous phase, gas and possible solids. The product is cooled in the coolers to about 80 °C with cooling water. The aqueous phase is be separated from the product after the cooling.

The product is depressurized with the product pump to atmospheric pressure. The product pump is not analyzed further because it effectively shares the same failure modes as the feed pump. The biocrude product is stored in the product tank which is periodically emptied before it gets full. The gas which formed during HTL process is removed from the tank and cooled with water before being directed out of the container. All of the water used in cooling processes and the separated aqueous phase of the product is lead to the waste water container which is likewise periodically drained.

The last components of the system are the electrical cabinet and the automation system. The electrical cabinet contains for example the fuses that protect the equipment from being damaged by high voltage. The automation system controls and monitors the processes with sensors that send data to back to the system. The process basically works in accordance with the command the automation system gives to it.

## **4.2 Risk assessment**

The components of the system are analyzed in the same order as in the previous chapter. The assessment follows the FMEA worksheet from appendix A.

The potential failure modes of the feeding tank are leakage and overflow. Leakage can be caused by mechanical damage to the tank and overflow can be caused by sensor error in which the sensor transmits false information about the feed levels in the tank. Both failures result in mess and a risk of exposure due to the alkaline black liquor feed.

The failure modes of the feed pump are clogging and leakage. Clogging can be caused by mechanical obstruction in the flow path which basically means too large particle size of the feed slurry. It can cause damage to the pump or harm to the equipment's usability. Leakage can be caused by either gasket damage or material fatigue of the pump. These can cause risk of exposure, mess and spraying of harmful slurry inside the container.

Preheaters have three possible failure modes. They are overheating of the electric heaters, solidification of the product and clogging. Overheating can be caused by malfunction in the system such as sensor or software errors or staying on for too long. It ruins the run and can at worst cause a severe leakage due to the breakdown of the pressure equipment. Product's solidification in the preheater can be attributed to the feed's unsuitability to the preheater. Solidification of the product ruins the biocrude's properties and therefore the run. Clogging is caused by feed's high viscosity which hinders the flow of the feed in the pipes. It causes unusability of the equipment and additional cleaning work for the personnel.

The reactor has only one failure mode and it is leakage. It is caused by gasket damage and the effects of the failure are mess, risk of exposure and spraying of the harmful and scalding slurry. The failure modes of the coolers are clogging and malfunction of cooling. Clogging is caused the same way as in the preheaters. The failure likewise causes unusability of the equipment and additional cleaning work. The malfunction of cooling is due to the supply of water to the process being hindered somehow. It can cause damage to the product pump, mess and unusability of the equipment.

Waste water container's failure mode is overflow. It can incur if the draining of the container is neglected. Leakage is also a possibility in waste water container's case. Leakage can be caused by mechanical damage to the container. Both failures cause mess and risk of exposure. The product container's failure modes are also overflow and leakage. Negligence of emptying the container can cause overflowing as was the case with the waste water container. Leakage is similarly caused by mechanical damage to the container. Again, both failures inflict mess and risk of exposure.

Failure modes of the electrical cabinet are overvoltage and short circuit. Overvoltage can be caused by an external voltage charge such as lightning. It results in blown fuses which need to be checked. Short circuit is caused by equipment failure and it can cause a risk of fire and a hazardous voltage to the cabinet. Automation system's failure modes are sequence malfunction and sensor error. Sequence malfunction can emanate from a programming error. Sequence malfunction can cause significant damage to the pumps if the pumping process happens out of order. Also, the run is ruined for certain. Sensor errors

are caused by damage to the sensors which can lead to unusability of the process or a potentially dangerous situation due to false information about the process that the sensors have given, For example someone can think that it is safe to go inside the process container because the sensors tell so when in reality something hazardous could have happened in the process container.

### 4.3 Criticality assessment

The criticality assessment in this risk assessment is done by evaluating the level of risk of each failure mode with the help of a custom risk matrix. The risk matrix is presented in table 1.

**Table 1:** *The risk matrix*

	Frequency	f1	f2	f3	f4
Severity	<b>Risk matrix</b>	less than once a year	once a year	once a month	once a week
s1	No/Minor harm to people/environment/equipment	very low	low	med	high
s2	Medium harm to people/environment/equipment	low	med	high	very high
s3	Major harm to people/environment/equipment	med	high	very high	very high
s4	Severe harm to people/environment/equipment	high	very high	very high	very high

The level of risk is determined as a function of the frequency of the risk happening and the severity of it. The frequency is split into 4 different levels that each represent different frequencies of the risk being realized. The frequencies are rather rough estimations of the probabilities of the risks happening in a certain period of time. The severity of the risk is split in the same manner as frequency. For a risk to have a certain severity rating it does not have to cause harm to every subject presented in the matrix but harming even one suffices.

Both of the feeding tank's failure modes are categorized to have a very low risk level due to them being really rare and having only a little to harm to the equipment. Clogging in the feed pump gets a high risk level because it has a frequency of 3 and a severity of 2. Leakage is estimated to have a medium risk level due to it being more infrequent than clogging of the feed pump.

All of the preheater's failure modes get a severity level of 2 due to them causing medium-level harm to the product or to the usability of the equipment. Solidification of the product and clogging are more likely to happen than overheating so they get a frequency rating

of 2 and 1 respectively. This gives overheating a risk level of low and the others medium. Reactor's leakage is estimated to have a risk level of medium with the frequency and severity of 2.

Clogging in the cooler is estimated to have a frequency of 2 and a severity of 2 which gives it a risk level of medium. The malfunction of cooling is approximated to be more infrequent but to have a more severe effect on the system, but the risk level stays on medium. Waste water container's failure mode is assessed to have a very low risk level because the probability and effects of it are really small. The product container's failure modes get a risk level of low because the effects of the oil product leaking are a bit more severe than with waste water. The frequency of overflow in both cases is so small because the failure happens only if a person responsible for the emptying of the containers forget or neglects to do it.

In the case of the electrical cabinet, overvoltage is rare because for it to happen an external voltage charge such as lightning needs to hit the process container. Also, the effects are trivial due to the fuses that protect the equipment from voltage. Overvoltage gets a risk level of very low because of it. Short circuit gets a risk level of medium because it has bigger chance of happening and the effects in the worst-case scenario are more severe.

Lastly the automation system's both failure modes get a risk level of high. Programming errors are quite possible, and sensors can get damaged during the processes. Furthermore, the effects of both failure modes can cause major harm to the equipment or even people in the case of sensor errors.

#### **4.4 Precautions and safety measures**

In this chapter the analysis will go through precautionary measures that are going to be in place when the HTL testing facility is built and safety measures that could reduce the risk levels. These measures are there to ensure that legislative restrictions and requirements are fulfilled by the testing facility. To begin with, the most important safeguard against any human harm is that the process is run unmanned. Unmanned process container means that harm due to any component's leakage or overflow failure mode can be minimized with careful monitoring of the process container and safety regulations.

Probability of the leakage failure mode of the feeding tank is reduced with the tank's high-quality mechanical design and good junctures. Risk of its overflow can be decreased with a mechanical surface monitor such as a webcam or an ultrasonic surface level

measuring device. Both could be used to track the feed levels and alert the operator if it rises too high.

Clogging of the feed pump can be decreased with the reduction of feed's particle size by wet grinding the feed before the process. Careful design of the flow path and feed recovery before the feed pump are also good ways to limit this risk. To prevent leakage from the feed pump it is essential to prepare for it well before the system is installed. Some ways to reduce this risk are careful sequence design and mechanical mounting of the feed pump, as well as inspection of gaskets during the preparatory maintenance.

In the case of the preheater, thermal protection around it can protect from risks relating to the overheating failure mode. Pre-selection of feeds is used to lessen the risk of solidifying the product in the preheater. In addition, pretreatment of material such as reduction of the particle size can be used to prevent clogging in this component too. On the other hand, risk of the reactor leaking can be reduced for example with sturdy copper gaskets and thorough design and installation of the reactor.

Design of experiments is utilized to prevent the product from clogging in the cooler. To reduce the risk of cooling malfunction regular inspections of the cooling water levels can be conducted. This could be done with a camera monitoring the levels.

Since waste water and product containers both have the same failure modes and are structurally fairly similar their safeguards are thus quite similar. Probability of the overflow failure mode can be reduced with a mechanical float that tracks the water or product levels in the containers so they can be emptied when necessary. Leakage failure mode can be prevented with high-quality mechanical design of the containers.

Both failure modes of the electrical cabinet have the same suggested safety measures to try to reduce the risk of overvoltage and short circuiting. With the use of high-quality components and a design based on existing standards both are less likely to occur. Current limitation additionally diminishes the severity of these risks. In the case of the automation system, programming errors can be noticed, and that way reduced with thorough commissioning testing and quality assurance of programming. Lastly, the probability of sensor errors happening can be decreased with the use of high-quality sensors. In addition, the severity of this failure mode could be reduced with alarms that warn if the conditions inside the process container are hazardous.

## 5. CONCLUSIONS

Several potential risks in EHTA's HTL process container were assessed during this risk assessment. The results were recorded on a worksheet that is found in appendix A. Failure mode and effects analysis was utilized to conduct the risk assessment and additionally criticality assessment and safety measure consideration were carried out to expand the analysis to more practical level.

Most of the risks of the failure modes were found to be relatively small in both severity and frequency. Nonetheless some failure modes had higher risk levels. These failures were mainly connected to clogging of some process component such was the case with feed pump, preheaters, cooler. The effects on the system of these failure modes were only harmful to the equipment and the usability of the process. Some of the other relevant failure modes were the possible solidification of the product in the preheaters and leakages relating to gasket damage in some components. Additionally, the failure modes of the automation system had possibly some of the more severe consequences. Sequence malfunction could cause considerable damage to the equipment and sensor errors could put personnel at risk if false information about the process container was transmitted.

Overall, a large majority of the risks to humans was removed due to the separate office and process containers. Harm to equipment was still possible so effective precautions and safety measures were identified and discussed for every failure mode. With these measures in place the HTL process is sufficiently safe to operate and major mishaps could be averted.

Failure mode and effects analysis was effective in assessing the risks relating to this process but in my opinion HAZOP study is even better suited to objects like this. Obviously because a HAZOP was already conducted a different approach to hazard identification was reasonable. When it comes to the reliability of the analysis there are certainly failure modes and risks that were unintentionally left outside of the assessment. In addition, only the components included in the assessment were analyzed and some other components might indeed have some potential risks connected to them or their operation. Also, the risk matrix used in the criticality assessment is quite inaccurate and there could certainly be better ways to model the level of risks. For example, the risk level categories such as very low, low and medium are not that descriptive or unambiguous of the real risks. Further research on the safety of HTL plants is needed to better understand the potential risks associated with this developing technology.

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## APPENDIX A: FMEA FORM

SYSTEM: EHTA's hydrothermal liquefaction testing facility				FAILURE MODE AND EFFECTS ANALYSIS		Date of analysis: 9.4.2020	
				Analysr: Elias Saarinen Board members: Tero Joronen, Mika Karttunen, Antti Nuottajarvi		Page 1(2)	
Part name	Failure mode	Cause of failure	Effects on system and personnel	Frequency (1-4)	Severity (1-4)	Risk level	Safety and precautionary measures
Feeding tank	1. leakage 2. overflow	1. mechanical damage 2. sensor error	1-2. risk of exposure, mess	1. 1 2. 1	1. 1 2. 1	1. very low 2. very low	1. high-quality mechanical design and good junctures 2. mechanical surface monitor if substance is harmful or used unmanned, visual monitoring (webcam), ultrasonic surface level measuring
Feed pump	1. clogging 2. leakage	1. mechanical obstruction in the flow path 2a. gasket damage 2b. material fatigue	1. harm to equipment's usability 2. risk of exposure, mess, spraying of harmful substance	1. 3 2. 2	1. 2 2. 2	1. high 2. med	1. reduction of feed's particle size, careful design of the flow path and recovery of feed 2. mechanical mounting, careful sequence design, inspection of gaskets during the preparatory maintenance, unmanned process container
Preheater	1. overheating of the electric heater 2. burning of the product 3. clogging	1. malfunction (stays on constantly, sensor error, software error) 2. feed's unsuitability to the preheater 3. feed's high viscosity	1. ruins the run, at worst can cause a severe leakage due to breakdown of the pressure equipment 2. ruins the run 3. additional cleaning, unusability	1. 1 2. 2 3. 2	1. 2 2. 2 3. 2	1. low 2. med 3. med	1. thermal protection 2. pre-selection of feeds 3. pretreatment of material

<b>Part name</b>	<b>Failure mode</b>	<b>Cause of failure</b>	<b>Effects on system and personnel</b>	<b>Frequency (1-4)</b>	<b>Severity (1-4)</b>	<b>Risk level</b>	<b>Safety and precautionary measures</b>
Reactor	leakage	gasket damage	risk of exposure, mess, spraying of harmful substance	2	2	med	copper gaskets, mechanical installation, thorough design
Cooler	1. clogging 2. malfunction of cooling	1. product's high viscosity 2. the supply of water is hindered	1. additional cleaning, unusability 2. damage to product pump, mess, unusability	1. 2 2. 1	1. 2 2. 3	1. med 2. med	1. design of experiments 2. regular inspections of the cooling water levels (with a camera possibly)
Waste water container	1. overflow 2. leakage	1. negligence of draining the tank 2. mechanical damage	1-2. risk of exposure, mess	1. 1 2. 1	1. 1 2. 1	1. very low 2. very low	1. mechanical float tracking water levels 2. high-quality mechanical design
Product container	1. overflow 2. leakage	1. negligence of draining the tank 2. mechanical damage	1-2. risk of exposure, mess	1. 1 2. 1	1. 2 2. 2	1. low 2. low	1. ultrasonic surface level measuring, mechanical float 2. high-quality mechanical design
Electrical cabinet	1. overvoltage 2. short circuit	1. external voltage charge 2. equipment failure	1. blown fuses 2. risk of fire and hazardous voltage	1. 1 2. 2	1. 1 2. 2	1. very low 2. med	1-2. current limitation, design based on existing standards, use of high-quality components
Automation system	1. sequence malfunction 2. sensor error	1. programming error 2. damage to sensors	1. ruins the run, possible damage to the equipment, mess 2. potentially dangerous situation due to false information, unusability	1. 2 2. 2	1. 3 2. 3	1. high 2. high	1. thorough commissioning testing, quality assurance of programming 2. use of high-quality sensors, use of alarms for observation