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Development, Testing and Evaluation of the Second Generation of the Small-Scale Biodiesel Production Line "BioMax"

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Mechanics and Process Technology

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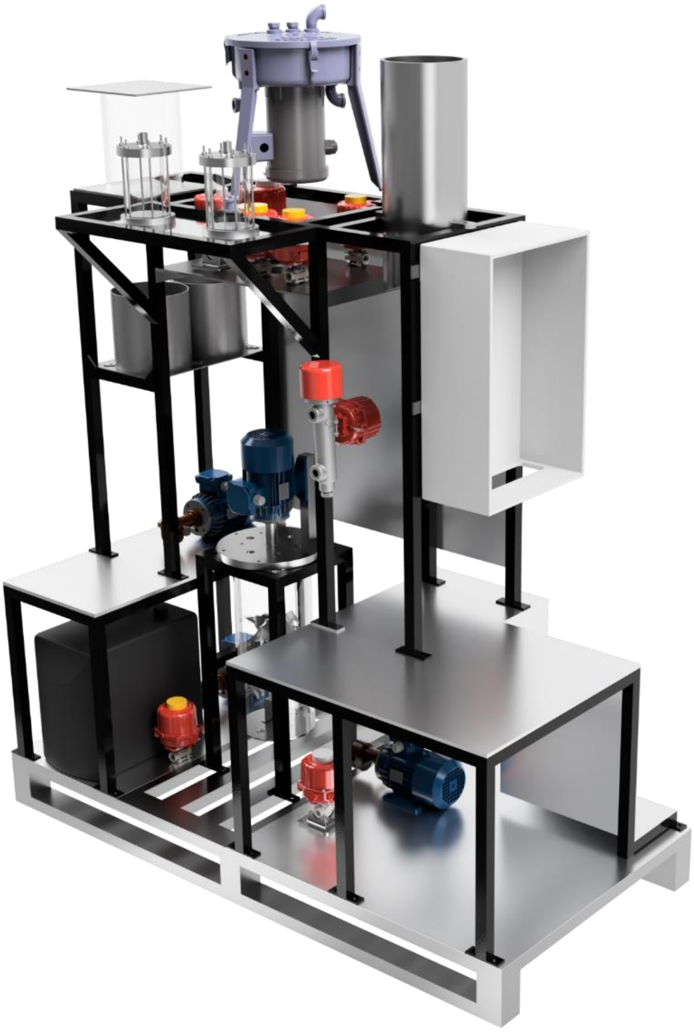

Dokumentasjon, Testing og Evaluering av Andre generasjon av det Småskala Biodiesel Produksjonsanlegget «BioMax»

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Preface

With this thesis I finish a five-year Master of Science in Mechanics and Process engineering at the Norwegian University of Life Sciences. The thesis has been written during the spring of 2019 and has a scope of 30 study points.

Since I was introduced to the "BioMax" project in February of 2017 and was allowed to help with programming the first generation of "BioMax", I have spent a considerable amount of time working on the production line. This work has provided me with insight into how much time, resources and knowledge that goes into creating a production line, albeit small-scale. Building the second generation of "BioMax" has challenged me intellectually and technically, and it is my hope that the production line will be of some use to students at NMBU going forward.

Ås, May 10th

Pål Ryssdal Tveit

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Thanks to "Mannskoret Over Rævne" for five great years with many memorable moments.

And finally, I wish to thank my family for always supporting me.

Abstract

Finding suitable and economically viable fuel alternatives, that might help mitigate the effect of global warming, is a field of continuous research. Biodiesel is a fuel derived from lipids from biomass that is renewable. Although biodiesel has desirable qualities as a fuel, the current methods of producing biodiesel still require improvements. NMBU is a university dedicated to the green shift, and as such, an effort is being made to research biofuels.

The project for the first generation of “BioMax” was started in January of 2016 by a team of students at “Eik ideverksted” at NMBU and received 500 000 NOK in funding from “Sparebankstiftelsen”, the Faculty of Science and Technology (REALTEK) and “Eik Idéverksted”. Developing a functional biodiesel production line has been the subject of several reports and theses at the Faculty of Science and Technology, and this thesis aims to provide a platform for future testing. During the span of this thesis, the second generation of the production line has been built, tested, improved upon and tested again.

“BioMax” is a small-scale biodiesel production line for utilizing heterogeneous catalyst technology. The production capacity of the production line is between 6 and 13 liters of reaction mixture. The production line is controlled by a PLC and a semi-automated program has been developed for controlling the production process. Process parameters such as temperature, reactor flow, reaction time and mixing speed can be varied to enable further research into biodiesel production with heterogeneous catalysts.

A FAME yield of approximately 27% was obtained with a methanol to oil ratio of 6:1 during the preliminary test of the second generation. After this, the production line was improved upon and tested again. The results from the tests has been presented and the function of the production line has been evaluated along with propositions for future development work.

Sammendrag

Å finne passende og økonomisk levedyktige drivstoffalternativer som kan bidra til å redusere effekten av global oppvarming, er et kontinuerlig felt for forskning. Biodiesel er et fornybart drivstoff avledet av lipider fra biomasse som er fornybar. Selv om biodiesel har gode egenskaper som drivstoff har de nåværende metodene for å produsere biodiesel fortsatt behov for forbedringer. NMBU er et universitet dedikert til det grønne skiftet, og som sådan blir det gjort en innsats for å forske på biodrivstoff.

BioMax-prosjektet ble startet i januar 2016 av et team av studenter på "Eik ideverksted" på NMBU og mottok 500 000 kroner i finansiering fra Sparebankstiftelsen, fakultetet for realfag og teknologi (REALTEK) og Eik Idéverksted. Utvikling av et funksjonelt produksjonsanlegg for biodiesel har vært temaet for flere rapporter og avhandlinger på fakultetet for realfag og teknologi, og denne oppgaven vil bidra med en plattform for fremtidig testing. I løpet av denne oppgaven har anlegget blitt bygget, testet, forbedret og testet igjen.

"BioMax", i sin nåværende tilstand, er et lite biodieselproduksjonsanlegg for bruk av heterogen katalysatorteknologi. Produksjonsanlegget har en kapasitet på rundt ti liter biodiesel. Anlegget kontrolleres av en PLS og et delvis automatisert styringsprogram har blitt utviklet for å kontrollere produksjonsprosessen. Prosessparametere som temperatur, reaktorstrøm, reaksjonstid og blandingshastighet kan varieres for å tilrettelegge for videre forskning innen biodieselproduksjon med heterogene katalysatorer.

Et biodieselutbytte på omtrent 27% ble oppnådd med et metanol- til oljeforhold på 6:1 under den første testen. Etter dette, ble produksjonsanlegget forbedret og anlegget ble testet igjen. Resultater fra testing av anlegget har blitt presentert og anleggets funksjon har blitt evaluert sammen med forslag til fremtidig utviklingsarbeid.

List of Figures

List of Abbreviations

Abbreviation	**Explanation**
A/D	Analog to Digital
AI	Analog Input
AO	Analog Output
AOCS	American Oil Chemists' Society
ASTM	American Society for Testing and Materials
CEN	European Committee for Standardization
CFPP	Cold Filter Plugging Point
CN	Cetane Number
DAG	Diacylglycerol
D/A	Digital to Analog
DI	Discrete Input
DO	Discrete Output or Relay Output
FAAE	Fatty Acid Alkyl Ester
FAEE	Fatty Acid Ethyl Ester
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acids
FKM	Fluroelastomer
I/O	Input/Output
IP	Ingress Protection
JSA	Job Safety Analysis
LECA	Lightweight Expanded Clay Aggregate
LEL	Lower Explosive Limit
MAG	Monoacylglycerol
MeOH	Methanol, but also called methyl alcohol
NMBU	Norges Miljø- og Biovitenskapelige Universitet
PBR	Packed Bed Reactor
PFD	Process Flow Diagram
PID	Proportional-integral-derivative
P&ID	Piping and Instrumentation Diagram
POU	Program Organization Unit
PLC	Programmable Logic Controller
PTFE	Polytetrafluorethylene
PVC	Polyvinyl Chloride
RCD	Residual Current Device
SOP	Standard Operating Procedure
TAG	Triacylglycerol
TOF	Off-Delay Timer
TON	On-Delay Timer
UCO	Used Cooking Oil
UEL	Upper Explosive Limit
VAC	Volts Alternating Current

VDC	Volts Direct Current
VFD	Variable Frequency Drive

List of Tables

List of Equations

Table of contents

1 Introduction

1.1 Motivation for the Thesis Work

Liquid fuel as an energy source is a pillar of our society. Fuels are used in large quantities every day to transport people and goods, and a global shortage of fuel would have a damaging impact on the global economy. Greenhouse gas emissions from the consumption of petroleum products are rising along with the rapidly increasing global population. The limitation or elimination of these emissions might prove to be the most difficult task of our time.

Norway is currently working on fulfilling the obligations, related to the reduction of greenhouse gas emissions, as stated in the Paris Agreement. The Norwegian government has put forward a goal to reduce emissions from road traffic by 35-40% compared to 2005, by 2030 [1]. A part of the governmental plan to accomplish this goal will be by enforcing an escalation plan, where the goal is to incorporate 20% blends of biofuel by 2020 [2]. Biofuels have the potential of being climate neutral, making it a more viable alternative than fossil fuels in the long term. In 2016, only about 7% of the total consumed biofuels in Norway were produced in locally [3]. Biodiesel accounts for approximately 90% of the biofuels in use in Norway today [4]. It is miscible with petroleum diesel, can be used as a blend stock and, in most cases, as an alternative to petroleum diesel [5]. In addition to this, it is the objective of the Norwegian government to go from using conventional biofuels to advanced biofuels [6].

Biodiesel has comparative advantages to petroleum derived diesel, such as low toxicity, being biodegradable and renewable [7]. Transesterification is the most common method for conversion of biodiesel feedstocks to biodiesel [8].

However, there are some aspects of biodiesel itself and biodiesel production that must be further researched to make biodiesel a viable replacement for petroleum diesel. Biodiesel has a relatively low shelf life and poor cold flow properties [5], making it unsuitable for cold climates such as Norway. Most methods of producing biodiesel relies on the consumption of electrical energy, which is in its own becoming a more viable energy source for transportation. Therefore, further research must be performed in order to decrease the energy consumption and the cost of producing high grade biodiesel.

Bio4Fuels is a Research Excellence Center at NMBU with the main goal of creating sustainable biofuels [9]. As there is a need for testing different types of biodiesel feedstocks and production methods, a versatile means of doing this is needed. Furthermore, an effort is being made to give process engineering students at NMBU a practical insight into production methods. The purpose of the small-scale biodiesel production line “BioMax” will be to provide a technology for these needs.

1.2 Background

A biodiesel production process based on heterogeneous catalyst technology was justified by Kristian Omberg in his master thesis [10]. Because of this, a design report was created by students during the course TMP299 at NMBU in June of 2016 [11]. The vision of the design report was to design a small-scale biodiesel production line that could be used in education and instruction at NMBU. From August of 2016 to January of 2017, a group of students at "Eik idéverksted" at NMBU was brought together to create the first generation of "BioMax". In the end of January 2017, the production line was assembled, and work began to test the equipment. In 2017, Tord Reinaas published his master thesis "*Dokumentasjon, videreutvikling, programmering og innledende testing av BioMax – et anlegg for produksjon av biodiesel til undervisningsbruk*. During his thesis, the production line was documented, and the equipment was tested. However, the entire production line was never tested due to time constraints. Figure 1.1 shows the previous work on the "BioMax" project.

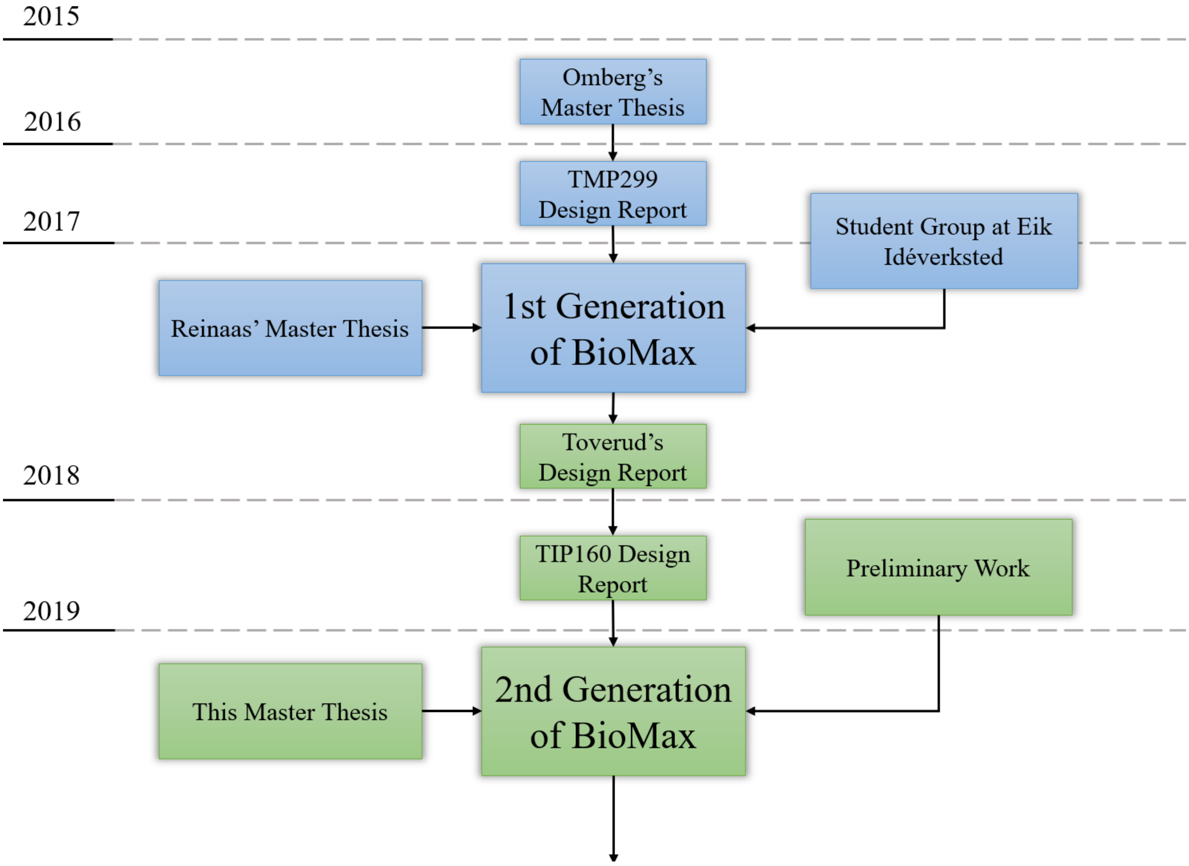


Figure 1.1: Previous work on BioMax.

The design and the P&ID from Reinaas' master thesis can be seen in figure 1.2 and 1.3.

Figure 1.2: The first generation of "BioMax" [12].

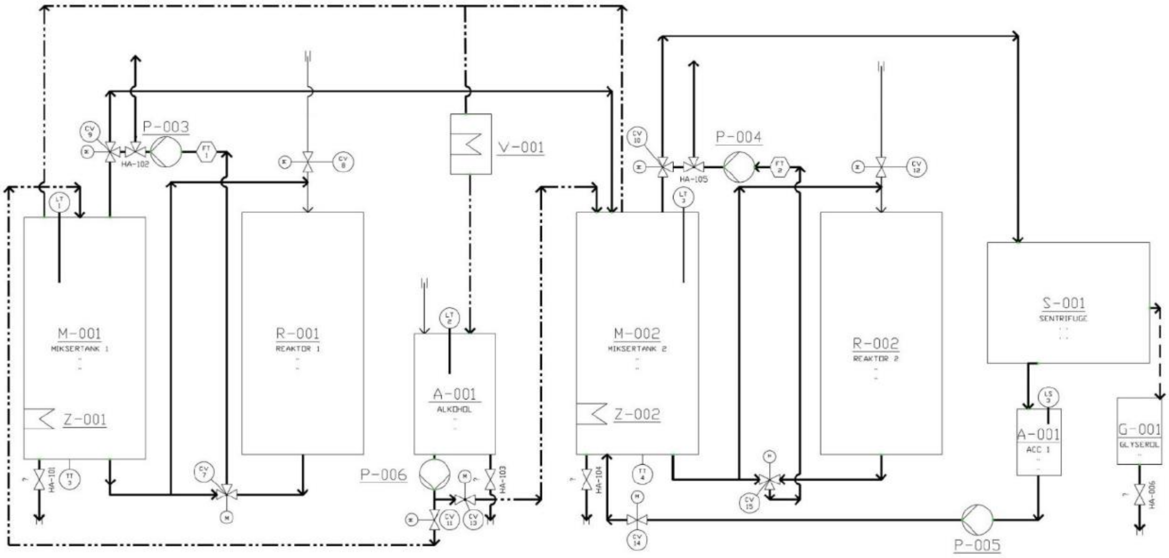


Figure 1.3: P&ID for the first generation of "BioMax" [12].

During the testing in Reinaas' thesis, it became apparent that the production line had some issues. The following recommendations were suggested for future work [12]:

- → Where it is possible, pump use should be replaced by gravity feeding.
- → The heating and alcohol recovery system should be more efficient, and an effort should be made to minimize the number of components.
- → The feeding into the packed bed reactors should be done from the bottom and up.
- → The use of one mixer tank should be enough for the production line, unless extra separation steps are added to the production process.

A new design report named “Prosjekt BioMini” [13] was written in June of 2017, and the first generation of the production line was disassembled. During the “BioMini” project, an aluminum pallet and two jacket heaters were acquired. The design and the P&ID from the design report can be seen in figure 1.4 and 1.5.

Figure 1.4: "BioMini" design [13].

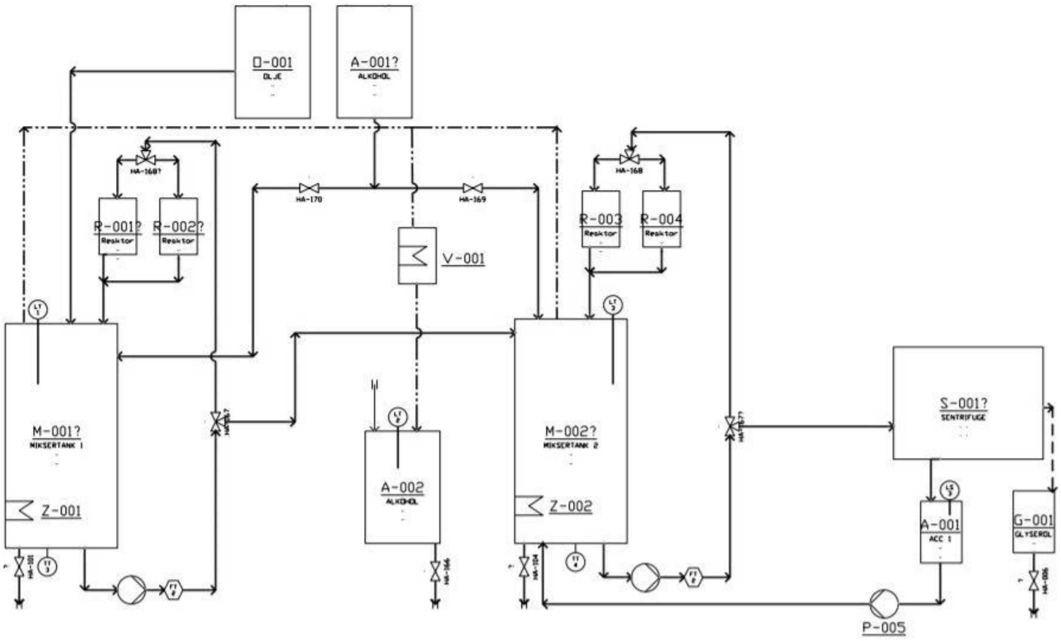


Figure 1.5: "BioMini" P&ID [13].

“BioMini” was not built due to time constraints. The following issues were identified with constructing the “BioMini” version:

→ The design did not allow for gravity feeding in a major way.

→ No extra separation steps had been added to the process and so the use two mixing tanks was unnecessary.

A new design report was made during the course TIP160 in January of 2018 [14]. The design and the process were simplified, which can be seen in figure 1.6, figure 1.7 and table 1.1.

Figure 1.6: "BioMax V3" design [14].

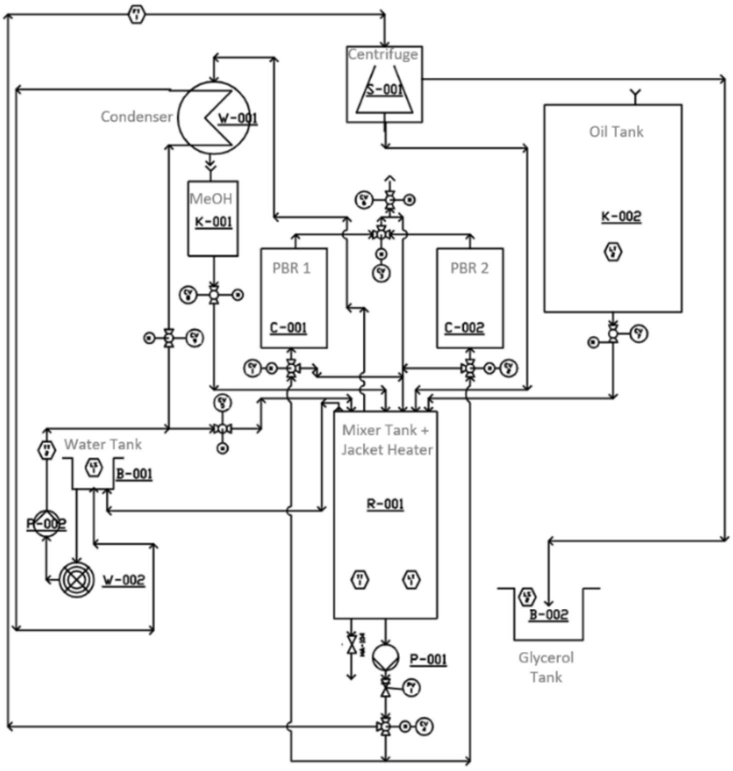


Figure 1.7: "BioMax V3" P&ID [14].

The number of components on the “BioMax V3” were significantly reduced from the previous versions, as it can be seen from table 1.1.

Table 1.1: Comparison of Previous Versions of BioMax

System	**BioMax V1 [12]**		**BioMini [13]**		**BioMax V3 [14]**	
	Equipment	#	**Equipment**	#	**Equipment**	#
Mass transfer	Pumps	6	Pumps	4	Pumps	2
Heating	Water heating	1	Jacket heaters	2	Jacket heater	1
Valve	Actuated valves	16	Actuated valves	11	Actuated valves	8
Reactor	Large acrylic PBR	2	3D-printed PBRs	4	Small acrylic PBR	2
Mixing tank	Mixing tanks	2	Mixers tanks	2	Mixer tank + Static mixer	1
Temp. measurement	Thermocouples	5	Thermocouples	3	Thermocouples	3
Flow measurement	Flow transmitters	2	Flow transmitters	2	Flow transmitter	1
Level measurement	Level transmitters	2	Level transmitters	2	Level transmitters	1

“BioMax V3” was assembled and tested as a part of the preliminary work for this thesis during the spring of 2018.

1.3 Objectives

1.3.1 Primary Objective

The primary objective of this thesis is to develop a small-scale biodiesel production line so that it can be safely employed in the education of students and research activities.

1.3.2 Secondary Objectives

- → Present a literature study about process requirements for producing biodiesel of high purity.
- → Evaluate the previous work on “BioMax” and develop a new design based on improving the energy and process efficiency of the production line.
- → Ensure that the safety of operators and students is maintained when operating the production line.
- → Develop a user interface for the control system so that the production line can be used by students with little to no technical training.
- → Implement a PLC to ensure flexibility of process parameters.
- → Provide documentation and recommendations for future development work and operations on the biodiesel production line.

1.3.3 Specifications of the Thesis’ Objectives

Due to limited time and resources, the following tasks have not been carried out in this thesis:

- → Testing of the proposed purification system.
- → Testing of the esterification system.
- → Testing of the centrifuge pretreatment of feedstock.
- → Catalyst optimization.
- → Optimization of process parameters.

→ Economic calculations concerning the production of biodiesel.

Furthermore, it should be noted that most of the components employed at the production line stems from previous work on the BioMax project.

1.4 Method

The foundation of this thesis has been the preliminary work done during the spring of 2018. The preliminary work is presented and used as supporting material for many of the decisions throughout the course of this thesis. A literature study into biodiesel production has been performed.

Autodesk Plant3D 2019 has been used for creating the P&ID and Autodesk Electrical has been used for creating the wiring diagrams. The PLC has been programmed using the free ladder logic software SoMachine Basic. All mechanical drawings, assemblies and renderings have been created by using Autodesk fusion 360. The construction of custom-made components has been done by the workshop at NMBU. Minor components for the assembly of the production line has been acquired from local vendors. The production line has been assembled using the mechanical, electrical and P&ID drawings. The practical work of assembling the production line has been done by the writer of this thesis.

The biodiesel production line has been tested with water to ensure a proper function and control of the system. Testing with oil, alcohol and catalyst has been performed and sensory data has been logged throughout the testing. The different systems that makes up the production line has been evaluated and recommendations for further development of the production line has been suggested.

2 Literature Study

This section will provide some theoretical insight into the chemical reactions, process parameters and production methods that are used to make biodiesel.

2.1 What is Biodiesel?

Biodiesel is a term for fuels that consists of mono alkyl esters of long chain fatty acids derived from renewable lipid feedstock [7]. The most common types of biodiesel are fatty acid methyl ester (FAME) and fatty acid ethyl ester (FAEE), depending on the type of alcohol used for the biodiesel production. Biodiesel is made by chemical reaction of lipid feedstock such as triacylglycerides (TAG) or free fatty acids (FFA) with an alcohol, usually by a catalytic reaction. As long as the lipid feedstock derives from renewable biomass, the produced biodiesel is considered a renewable fuel, in contrast to fossil fuels [15].

It should be stated that there are other types of biodiesel and biodiesel production methods, as can be seen from figure 2.1. Hydrogenated vegetable oil (HVO) is a synthetic biodiesel that has many comparative advantages to FAME and FAEE [16]. However, the hydrogenation process requires high temperature and pressure, making it less suitable for use in education.

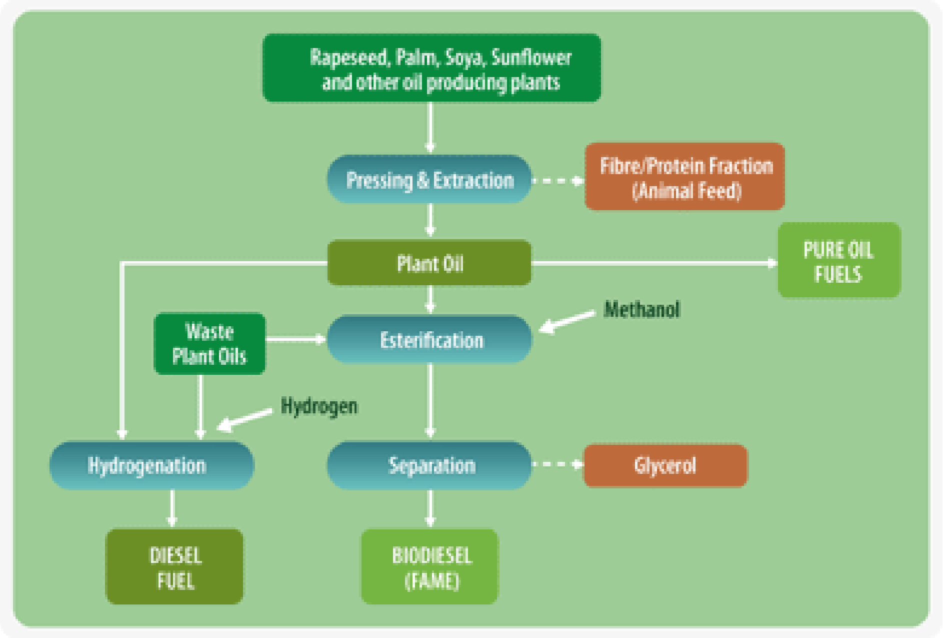


Figure 2.1: Production of biofuels from vegetable oils [17].

There are classifications, or generations, associated with the origin of the biodiesel feedstock:

(1) **Conventional biodiesel** (first-generation) is made from edible oils, such as canola or soybean oil. This is a problem because the use of agricultural land for fuel production raises the ethical question of food versus fuel. Currently, most of the consumed biodiesel in Norway falls under this category [18].

(2) **Advanced biodiesel** (second, third and fourth generation) uses oil produced on non-agricultural land, usually non-edible.

   a. The second generation of biodiesel resolves the fuel vs. food debate, as the non-edible oil plants used to produce the biodiesel feedstock can be cultivated in areas unsuitable for agriculture [19]. The main issue with second generation biodiesel is that the production is limited by land.

b. The third generation of biofuels is based on the use of microalgae, which can be cultivated at sea or in ponds, meaning that land area is not required for production [20].
c. The fourth generation of biodiesel is based on using genetically modified algae to achieve enhanced biodiesel production [21].

Of these generations, only the first and second generation are currently being produced on a commercial scale [21].

Biodiesel can be blended with fossil diesel to blends such as B20, meaning 20% biodiesel and 80% fossil diesel, or it can be used in its pure form as B100. The European standard for B100 biodiesel is EN14214 which describes the requirements and test methods for FAME [22, 23]. The American counterpart for the EN14214 standard is ASTM D6751. Table 2.1 shows some of the criteria for biodiesel.

Table 2.1: Biodiesel Purity Specifications [23]

Property	**ASTM D6751 (2012)**		**EN 14214 (2012)**		**EN 590 (2004)**	
Concerning	Biodiesel blend stock		Biodiesel		Diesel	
Flash Point (Min)	93	°C	101	°C	55	°C
Water Content (Max)	0.050	vol%(1)	0.050	wt%	200	mg/kg
Kinematic Viscosity	1.9 - 6.0	mm^2/s	3.5 - 5.0	mm^2/s	2.0 - 4.5	mm^2/s
Density	-	-	860 - 900	kg/m^3	820 - 845	kg/m^3
Ester Content (Min)	-	-	96.5(3)	%	-	-
Sulphur Content (Max)	15(2)	ppm	10.0	mg/kg	10.0(4)	mg/kg
Cetane Number (Min)	47	-	51	-	51	-
Oxidation Stability (Min)	3	h	8	h	25	g/m^3
Alcohol Content (Max)	0.2	wt%	0.20	wt%	-	wt%
MAG Content (Max)	0.4	wt%	0.7	wt%	-	wt%
DAG Content (Max)	-	-	0.2	wt%	-	wt%
TAG Content (Max)	-	-	0.2	wt%	-	wt%
Free Glycerin (Max)	0.020	wt%	0.02	wt%	-	wt%
Total Glycerin (Max)	0.240	wt%	0.25	wt%	-	wt%
Phosphorus content (Max)	0.001	wt%	4.0	mg/kg	-	mg/kg

(1) Water and sediment content. (2) S15 Biodiesel quality. Can also be S500 and S5000. (3) Linolenic acid methyl ester can be max. 12 wt% and polyunsaturated methyl esters can be max. 1.0 wt%. (4) Can also be 50 mg/kg.

The European standard is generally stricter than the American due to different approaches with regards to how biodiesel is utilized as a fuel. In the ASTM standard, biodiesel is treated as a blend stock for petroleum diesel rather than as a neat fuel as in the EN14214 standard [23, 24]. The properties described in the standards are important for the biodiesel to operate in diesel engines without causing issues with the fuel system, the engine or the environment.

As can be seen from table 2.1, the produced biodiesel must consist of 96.5% esters to meet the specifications from the EN14214 standard. This is an important number to note, as the separation of unreacted feedstock is difficult. Therefore, a high conversion of feedstock to biodiesel is needed. The density and kinematic viscosity of the biodiesel are factors that can be evaluated relatively easy. Testing these parameters can therefore give a quick indication of whether the production process has been successful or not.

The cloud point and the cold filter plugging point (CFPP) are indications of how the biodiesel will operate during low temperatures. This property is especially important when the biodiesel is to be used in cold climates, such as Norway. The cloud point is the temperature at which crystals start to form as the fuel is gradually cooled. The CFPP is the point at which the fuel starts to plug a test filter due to gel consistency or crystallizing of the fuel [25].

The flash point can be defined as the lowest temperature at which a fuel still releases enough vapors to ignite. This point is important to note as biodiesel with high alcohol content will often have a low flash point. Biodiesel, in general, will have a flash point above 150℃ [5].

The cetane number of a diesel fuel describes the ignition quality of the fuel. The cetane number speaks to the amount of time that passes from compression of the diesel fuel until combustion. High cetane numbers ($CN = 100$) are assigned to diesel fuels that ignite rapidly. Low cetane numbers ($CN < 15$) are assigned to diesel fuels that have a delayed ignition. Neither high or low cetane numbers are desirable as the scale is based on the correct cetane number being somewhere in between [5]. FAME and FAEE will generally have a higher cetane number (48-65) than petroleum derived diesel fuel (40-55) [26]. HVO will generally have a higher Cetane number than FAME and FAEE [16].

Free glycerol is the remaining glycerol after purification that is either dissolved into or suspended in the biodiesel. Bound glycerol is the glycerol connected to unreacted MAG, DAG and TAG. The amounts of free and bound glycerol make up the **total glycerol** content. Given enough time, the free glycerol will settle out and accumulate in storage or fuel tanks. Large amounts of total glycerol can plug filters or cause combustion issues [5, 25].

The oxidation stability is an indication of the fuel's reactivity with air and thereby the amount of time the fuel can be stored. Low oxidation stability is an issue that limits the use of biodiesel, as the biodiesel will decompose in a short amount of time compared to petroleum derived diesel. The decomposition of the biodiesel can be caused by the biodiesel feedstock having a high degree of polyunsaturated fatty acids that are susceptible to oxidation in storage [27]. Adding antioxidants to biodiesel may improve the lifetime of the fuel [28].

2.2 What is Biodiesel Made From?

2.2.1 Feedstock

The feedstock in biodiesel production is the lipids that are reacted into fatty acid alkyl esters (FAAE). As the transesterification process does not alter the chemical composition of the fatty acid groups, the produced FAEE will reflect the feedstock that was used to produce it. The quality of the feedstock for the biodiesel production is an important factor to consider, as it will have a strong impact on the result. Refined vegetable oils can be transesterified directly, however these are expensive to obtain and may still be used for food purposes. Lower quality oils such as used cooking oil (UCO) or yellow grease will have high FFA levels, along with other impurities, but are significantly cheaper.

Sanford et al. [25] evaluated 36 different feedstocks for biodiesel production. Conventional feedstocks such as canola, soybean, sunflower and used cooking oil were tested, as well as advanced feedstocks such as jatropha, fish, algae and tung oil. Some of their findings after testing these feedstocks can be seen in table 2.2.

Table 2.2: Feedstock Properties from Sanford et al. [25]

Feedstock	**Algae**	**Canola**	**Jatropha**	**Soybean**	**Sunflower**	**UCO**
Moisture Content (wt%)	0.021	0.085	0.073	0.029	0.020	0.242
FFA Content (wt%)	0.45	0.34	1.17	0.07	0.04	2.72
Kinematic Viscosity (mm^2/s)	10.99	34.72	33.90	28.87	35.84	27.00
Saponification Value (mg KOH/g)	160.60	189.80	200.80	195.30	193.14	198.50
Oxidation Stability (hours)	51.3	14.1	15.6	5.3	10.1	2.8
Sulphur Content (ppm)	28.1	5.7	3.5	0.8	0.1	3.4
Phosphorus Content (ppm)	339.7	17.9	322.9	3.7	< 0.1	27.0

Of the properties in table 2.2, there are some that can be altered by pretreatment, and some that cannot be so easily altered. The moisture content can be lowered by evaporation, centrifugation or adsorbents, and the FFA content by esterification. The sulfur and phosphorous content will often depend on the washing method for purifying the biodiesel. The moisture content and the FFA content will be discussed in section 2.3.2.

The saponification value is based upon how much potassium hydroxide is needed to create soap from one gram of fat or oil [25]. In short, the saponification value is an indication of the chain length or the average molecular weight of the fatty acids that are present in the feedstock. High or low saponification values indicates that the feedstock contains shorter or longer length chains, respectively [5]. High saponification values may also indicate high levels of FFA, due to the low molecular weight of FFA [29].

The biodiesel will inherit many properties from the feedstock used to produce it. Table 2.3 shows some of the results that Sanford et al. obtained from creating biodiesel from various feedstocks. The produced biodiesel was tested against the ASTM D6751 standard.

Table 2.3: Biodiesel Characteristics Compared to Feedstock from Sanford et al. [25]

Property	Algae	Canola	Jatropha	Soybean	Sunflower	UCO
Flash Point (C)	> 160	> 160	> 160	> 160	> 160	> 160
Water and Sediment (% vol)	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Kinematic Viscosity (mm^2/s)	4.519	4.439	4.253	4.039	4.439	4.332
Density (S.G.)	0.8780	0.8820	0.8795	0.8840	0.8800	0.8555
Sulphur Content (ppm)	5.1	1.4	1.2	0.8	0.2	2.4
Oxidation Stability (hours)	8.5	7.6	2.3	2.1	0.9	1.0
MAG Content (wt%)	0.265	0.363	0.291	0.473	0.387	0.411
DAG Content (wt%)	0.078	0.127	0.104	0.088	0.092	0.161
TAG Content (wt%)	0.020	0.000	0.022	0.019	0.000	0.000
Free Glycerin (wt%)	0.009	0.006	0.006	0.012	0.007	0.012
Total Glycerin (wt%)	0.091	0.114	0.100	0.149	0.121	0.143
Phosphorus content (ppm)	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
CFPP (C)	-7	-13	0	-4	-3	-2
Cloud Point (C)	-5.2	-3.3	2.7	0.9	3.4	2.4
Picture (Oil on the left and biodiesel on the right)	 *Figure 2.2: Algae oil and biodiesel.*	 *Figure 2.3: Canola oil and biodiesel.*	 *Figure 2.4: Jatropha oil and biodiesel.*	 *Figure 2.5: Soybean oil and biodiesel.*	 *Figure 2.6: Sunflower oil and biodiesel.*	 *Figure 2.7: UCO and UCO biodiesel.*

The results from the research of Sanford et al. indicates the suitability of some feedstocks over others in cold climates as can be seen in figure 2.8.

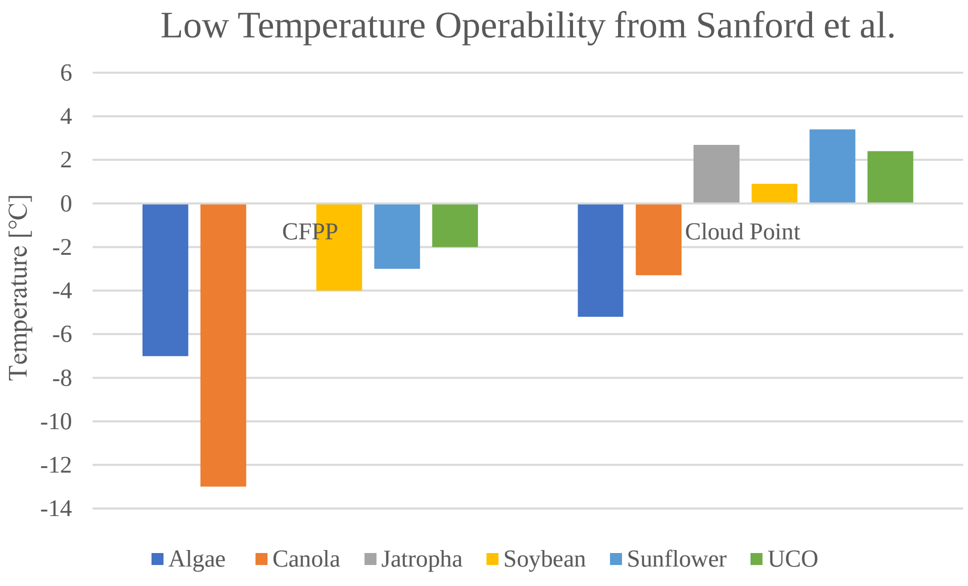


Figure 2.8: Low temperature operability of biodiesel from different feedstocks [25].

The algae sample shows a good cloud point and CFPP along with a relatively high oxidation stability. Canola oil, which is a more commercially available feedstock than algae oil, shows similarly good characteristics. It can be seen from table 2.2 and 2.3 that the oxidation stability of the feedstock was significantly reduced when the feedstock was converted into biodiesel.

2.2.2 Alcohol

Although there are many types of alcohol, methanol and ethanol are the most readily available and cheapest on the market. First and foremostly, although both methanol and ethanol are toxic and highly flammable, methanol is much more hazardous. Ingestion or inhalation may lead to blindness, or in the worst case, death [30]. Ethanol is more suitable in a learning environment, as the effects of exposure are less severe compared to methanol. Table 2.4 shows some properties of methanol and ethanol.

Table 2.4: Methanol and Ethanol Properties [31-33]

Description	Methanol	Ethanol
Molecular formula	CH_3OH	CH_3CH_2OH
Boiling Point [°C]	64.7	78.2
Flash point (closed cup) [°C]	9.0	14.0
Auto-ignition point [°C]	440	363
Density $[kg/m^3]$	0.79	0.79
Lower Explosive Limit (vol%)	6.7	3.3
Upper Explosive Limit (vol%)	36.0	19.0
Miscibility with water	Miscible	Miscible

The boiling point is the temperature at which a liquid phase will be transformed into a gaseous phase at atmospheric pressure. The boiling point of the alcohol is an important factor, as the excess alcohol after transesterification is often removed by evaporation. **The auto-ignition point** is the temperature at which a substance will spontaneously ignite at atmospheric pressure.

The lower and upper explosive limit (LEL and UEL) is the lower and upper concentration at which the gas-air mixture will be to lean or too rich to burn, respectively [33]. The LEL and UEL in table 2.4 are given at room temperature and atmospheric pressure.

The miscibility of a liquid is related to its polarity. Whereas methanol and ethanol are polar substances that are miscible with water, oil is non-polar. As different polarities do not mix, oil and methanol/ethanol are immiscible.

2.2.3 Catalysts

A catalyst is a substance that will increase the rate of the reaction, while chemically not reacting with any of the reactants. Although FAAE can be produced without a catalyst [34], most conventional biodiesel production methods include the use of a catalyst. Biodiesel can be produced by using alkali, acid and enzymatic catalysts. Moreover, these catalysts are often classified as either homogeneous or heterogeneous. Homogeneous catalysts are in the same phase as the other substances in the process and heterogeneous catalysts are in a separate phase. Figure 2.9 shows the variety of ways biodiesel can be produced.

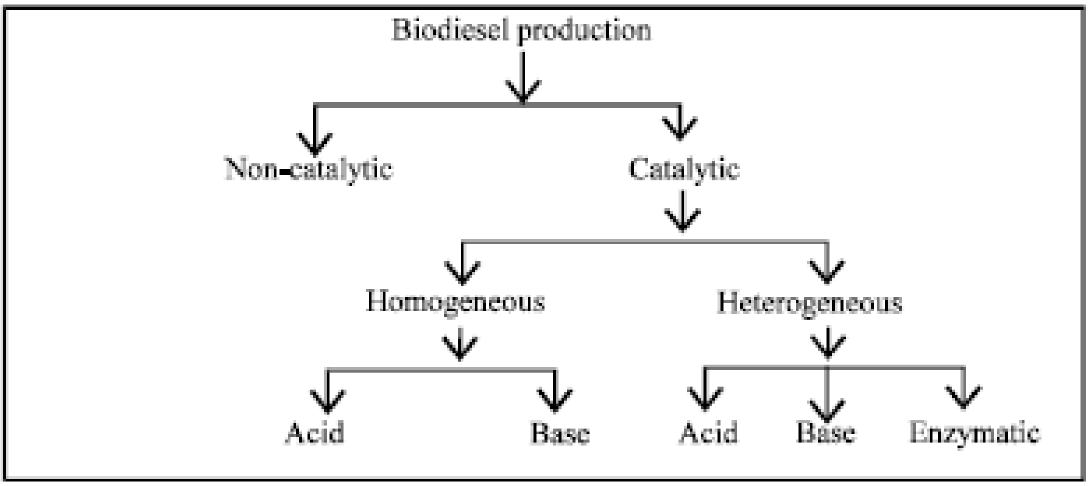


Figure 2.9: Biodiesel production methods [35].

The most conventional approach is to employ an acid-catalysed esterification process and an alkaline-catalysed transesterification process. Homogeneous catalyst are generally cheaper, more available and more effective catalysts than heterogeneous catalyst, and are therefore more widely used. However, the issue with using a homogeneous catalyst, is that it cannot be regenerated and that its separation from the product requires several extra process steps [5, 36]. As heterogeneous catalysts are in a different phase from the reaction mixture, separation of it is much easier, and the catalyst can be regenerated and reused several times. By using heterogeneous catalyst, a more sustainable way of acid-catalysed esterification and alkaline-catalysed transesterification can be obtained [10].

2.3 Production of Biodiesel by Catalytic Reactions

There are some commercial small-scale biodiesel processors on the market such as Springboard Biodiesel's BioPro series [37] or CTsystems' Biotron series [38]. These biodiesel processors use technology ranging from conventional tank technology to cavitation reactor technology. The scope of "BioMax" will be limited to conventional tank technology.

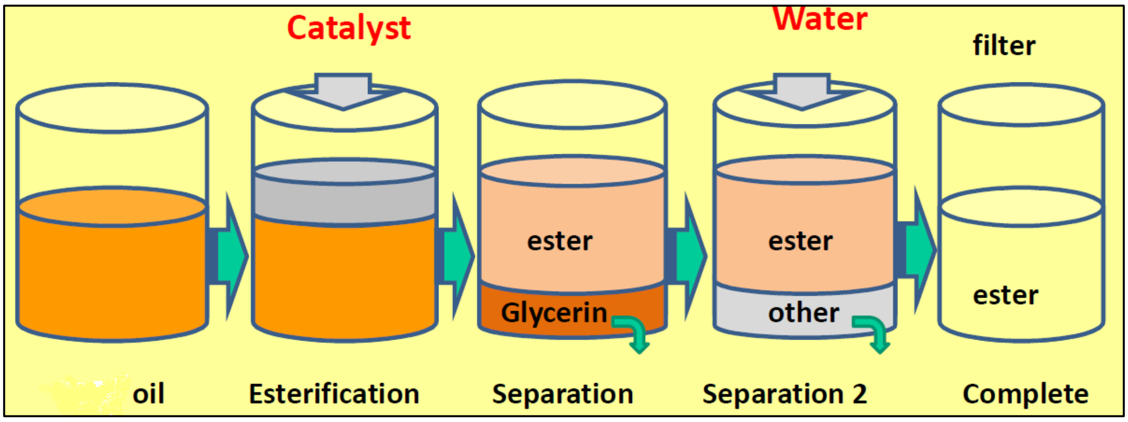


Figure 2.10: Biodiesel production by catalytic reaction [39].

2.3.1 Transesterification

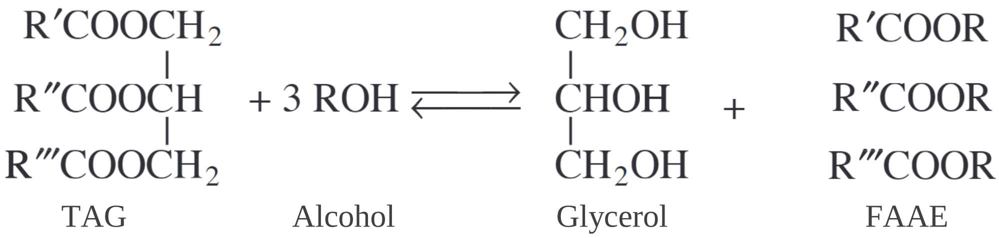
Transesterification is a chemical reaction where triacylglycerol (TAG) reacts with an alcohol to produce glycerol and fatty acid alkyl esters (FAAE). The transesterification process is a series of stepwise reversible reactions as shown in equations 1, 2 and 3 [40].

$$TAG + ROH \Leftrightarrow DAG + R'COOR \qquad \text{Eq. 1}$$

$$DAG + ROH \Leftrightarrow MAG + R''COOR \qquad \text{Eq. 2}$$

$$MAG + ROH \Leftrightarrow Glycerol + R'''COOR \qquad \text{Eq. 3}$$

Tri-, di- and monoacylglycerol are three, two or one fatty acids, respectively, bound to a glycerol molecule. TAG, DAG and MAG molecules may consist of many combinations of fatty acids, usually with a chain length between 12 and 24 carbon atoms [41]. For simplicity, the overall transesterification reaction can be written as it is shown in figure 2.11.



$$\begin{array}{c} R'COOCH_2 \\ | \\ R''COOCH \\ | \\ R'''COOCH_2 \end{array} + 3\,ROH \rightleftharpoons \begin{array}{c} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array} + \begin{array}{c} R'COOR \\ R''COOR \\ R'''COOR \end{array}$$

Figure 2.11: The overall transesterification reaction [15]

The desired outcome when producing biodiesel is to push the reaction towards the right side of the equation in figure 2.11. This is usually done by adding a larger amount of alcohol than what is needed to balance the reaction equation [5].

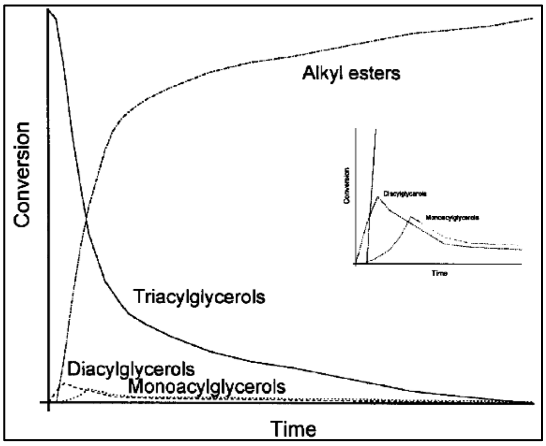


Figure 2.12: Conversion vs. time for transesterification of TAG to FAAE [5].

Figure 2.12 shows the reaction time for conversion of TAG to FAAE. Although a 100% conversion of TAG to FAAE is theoretically possible, this requires optimal conditions for the conventional production method. The reaction time of the transesterification process may depend on many factors such as mixing characteristics, temperature, type and dosage of catalyst, type and molar ratio of alcohol and the feedstock [42]. Although these factors may vary, most transesterification processes using conventional process technologies use a reaction time of 1-3 hours, a temperature around 60 °C and an alcohol-to-oil ratio of around 6:1 [5].

2.3.2 Esterification

Many low-cost feedstocks for biodiesel production will have a high free fatty acid content and moisture content, such as used cooking oils or animal fats. Moisture is commonly removed by evaporation or adsorption. FFAs are fatty acid molecules that are not bound to a glycerol molecule. FFAs are unwanted during the transesterification reaction as the FFAs can form metal soaps and water when reacted with a base [43].

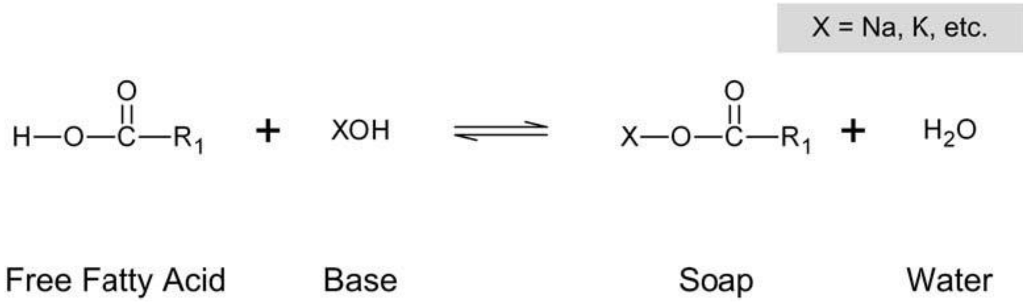


Figure 2.13: Saponification reaction between FFA and base catalyst [44].

As can be seen by comparing figure 2.13 and 2.14, the saponification reaction is fairly similar to the hydrolysis of TAG. Hydrolysis of TAG will usually require water to be in a gaseous phase, and so is unlikely to be prevalent during conventional transesterification conditions [45]. As most catalysts used for transesterification are alkaline, esterification is a common pretreatment when the FFA content in the feedstock exceeds 1-2% [46]. However, high FAAE yields may still be obtained up to around 5-7% FFA content [5, 47]. Esterification is usually done by using a strong acid catalyst, as acids will not react with the FFA. The esterification reaction equation can be seen in figure 2.14.

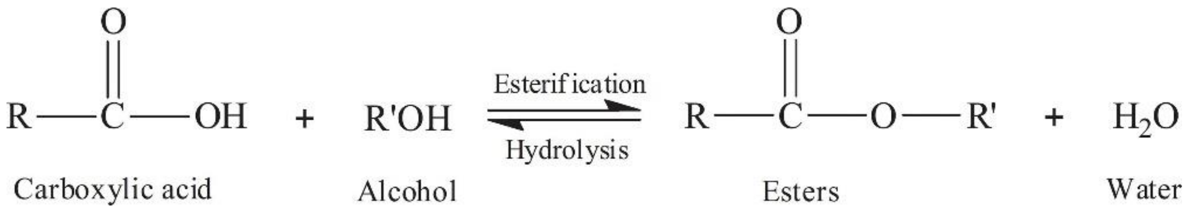


Figure 2.14: Esterification Reaction between FFA (Carboxylic acid) and alcohol by an acid catalyst [48].

The byproduct of the process is water, which is important to remove from the process as it can lead to a decrease in catalyst activity and a lower yield [49, 50].

2.3.3 Mixing

An important process parameter of the esterification and transesterification process is the mixing of oil and alcohol. Due to oil and alcohol being immiscible, creating a good emulsion between the two substances is essential to increase the interfacial area at which a reaction can occur [51]. There are several methods of mixing immiscible phases. Static mixers provide a relatively simple and inexpensive method of mixing. However, this method of mixing will only work provided that both liquids are somewhat mixed beforehand.

The most common method of mixing on an industrial scale, is by employing an agitator. The agitator usually consists of a motor connected to a shaft with attached impellers that are submerged into the liquid in a tank. Lakshmi et al. [51] proposed a minimum agitator speed of 600-650 rpm for karanja oil and 700-750 rpm for rice bran oil to ensure a strong emulsion on a laboratory scale.

Adding cosolvents, a substance which is soluble with both methanol and oil, is another method of creating an emulsion. Many cosolvents, such as hexane, are highly toxic and must therefore be removed safely after the process is finished. Todorovic et al. [52] researched a method of using crude biodiesel as a cosolvent during the transesterification of sunflower oil by methanol and calcium oxide. 10wt% (of oil weight) of crude biodiesel was added together with the catalyst, methanol and oil.

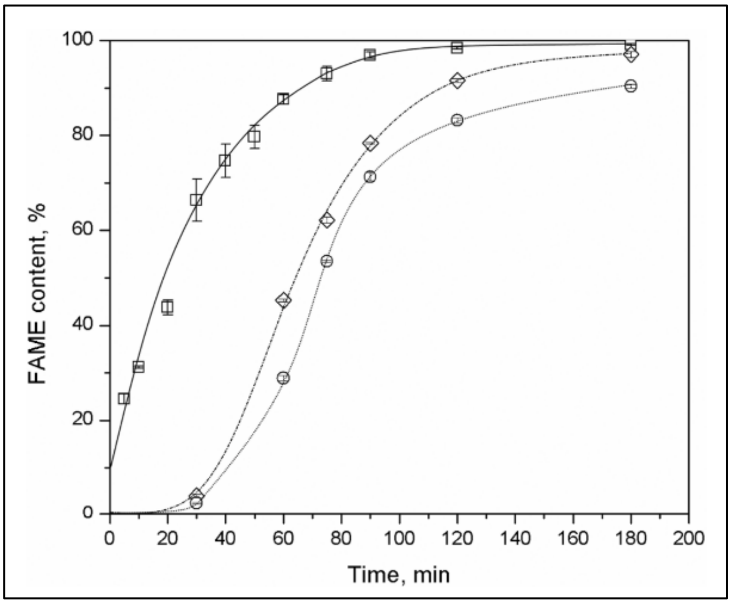


Figure 2.15: The effect of adding cosolvents where □ are with cosolvent at 50°C, ○ are without cosolvent at 50°C and ◊ are without cosolvent at 60°C [52].

The addition of crude biodiesel lead to a reduction in the initial induction period for the calcium oxide catalyst, as can be seen in figure 2.15. Moreover, the addition of crude biodiesel to the process does not require any extra separation steps.

2.3.4 Purification of Biodiesel

Purification of the biodiesel is a necessity due to the strict standards imposed on the biodiesel's quality, as can be seen in table 2.1. As the production line is going to be based on heterogeneous catalyst technology, only purification processes specific to heterogeneous catalyst technology will be presented.

Removing the excess alcohol is usually the first step in the purification process [53]. Methanol and ethanol are often removed by evaporation due to the relative low boiling points of these substances. The next step is removing the bulk of the glycerol, which is often done by gravity settling or centrifugation, due to the relative high density of glycerol. If necessary, minor contaminants are removed, such as soap, remaining alcohol, catalyst traces and free glycerol.

Water washing is a commonly employed method of biodiesel purification. The use of hot water to remove impurities in the biodiesel can give the product a high purity and yield [54]. Water washing can be done by spraying hot water on top of the crude biodiesel. As the water travels down through the biodiesel, polar substances are dissolved into the water. After the water has settled down, a two-phase liquid system will be present, and the water can be drained from the bottom. The issue with water washing is that the excess water in the biodiesel must be removed after the washing cycle. The efficient removal of water from biodiesel will require evaporation, meaning a use of temperatures above 100°C.

Dry washing is a term for purification processes that does not include the use of water in the purification process. Dry washing methods includes the use of ion exchange resins or adsorbents [53, 54]. Dry washing can be done by feeding the crude biodiesel through a column containing the dry washing medium or by adding the dry washing medium into a stirred tank [53, 55].

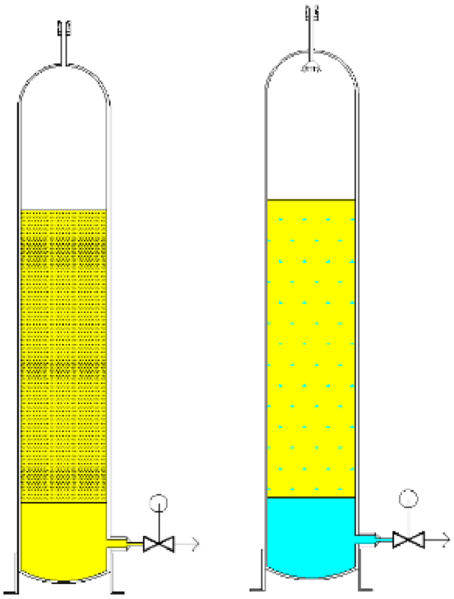


Figure 2.16: Simplified visualization of dry wash (Left) and water wash (right) configuration in a purification column. Made with AutoCAD 2018.

The use of a heterogeneous catalyst should only produce small amounts of soap, if any [56]. Although there are no direct restrictions on soap in EN14214 or ASTM D6751 [23], emulsions created by soap will alter the viscosity of the biodiesel and thereby altering its flow properties. Soap is also an issue during the separation of glycerol, alcohol and water from the FAAE.

3 Preliminary Work for the Second Generation of "BioMax"

The framework for the second generation of "BioMax" was constructed by the workshop at NMBU during the spring of 2018. The biodiesel production line was assembled during the spring of 2018 and tested in May of 2018 as a part of this thesis. This version of the production line can be seen in figure 3.1.

Figure 3.1: The assembly of BioMax during the spring of 2018.

3.1 Testing of the Production Line

An automated control program was made for testing the production line. The control program was verified with oil before the test. The testing consisted of adding 10 liters of refined rapeseed oil and 2,8 liters of methanol to the mixer tank. The liquid was circulated through PBR 1 with 0.5 kg of lab grade calcium oxide rocks with a mean particle size of approximately 1 cm. The liquid in the mixer tank was heated to and kept at approximately 60 °C throughout the process by using the jacket heater. The frequency on the VFD for the main pump was set to 10 Hz during the circulation, corresponding to approximately 0.8 L/min (Figure 7.26). The liquid was circulated through the PBR for two hours. After the circulation, the contents of the mixer were circulated between the centrifuge and the mixer for 30 minutes. Finally, the liquid was cooled and stored in four plastic containers of approximately 3 liters and one plastic container of 1.5 liter.

Figure 3.2: Adding oil to the production line during testing in June of 2018.

3.2 Evaluation of the Production line

3.2.1 Design

After testing the biodiesel production line, it was apparent that there were some challenges that needed to be resolved. In the design drawing of the production line (figure 1.6), the tubing, wiring and minor components on the production line were not accounted for. As a result of this, there were several challenges with getting everything to fit onto the frame during assembly. Modifications to make the production line functioning were difficult to perform due to tightly packed components. The PBRs and the tubing was found to be vulnerable to the methanol, as the reactors were made of acrylic material and the tubing was made from fluoroelastomer and PVC.

Figure 3.3: Damaged acrylic top lid of PBR 1 after testing the production line in June of 2018.

3.2.2 Mixing

The oil and the methanol were premixed by shaking it into a seemingly homogeneous mix before adding it to the production line. A static mixer was placed after the main process pump to enforce the emulsion of the premixed liquid. The liquid was circulated by the main pump at 50 Hz between the static mixer and the mixer tank until the liquid was 60 °C during the heating process. After this, the circulation through the PBR was initiated. Figure 3.4 shows the liquid moving through the reactor. After approximately one hour, a strong emulsion was observed as the liquid changed to a misty white color.

Figure 3.4: Flow through PBR 1 during testing in June of 2018.

3.2.3 Heating

The only employed heating equipment on the production line was a jacket heater with an adjustable thermostat. The thermostat was set to 140 °C and liquid was transferred from the bottom of the tank to the top with a VFD setting of 50 Hz. The temperature of the liquid was measured by a temperature transmitter mounted in the bottom of the tank. Heating was slow, with a heating time of 45 minutes to raise the temperature from 20 to 60°C. During the transesterification, the frequency on the VFD was reduced to 10 Hz, meaning a significant reduction in flow rate from the heating program (50 Hz). The absence of any stirring mechanism in the mixing tank resulted in a hotter liquid fraction at the mixer walls and a cooler liquid fraction towards the middle of the mixer. It is therefore likely that the actual temperature of the liquid was lower than what was measured.

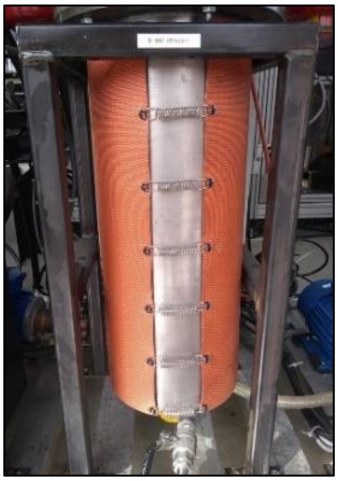


Figure 3.5: The jacket heater configuration during testing

3.2.4 Centrifugation

The liquid was heavily emulsified after the circulation through the reactor. The centrifuge was running at 6000 rpm and the VFD setting on the main pump was 40 Hz. Centrifugation of the reaction mixture was performed for 30 minutes. After the centrifugation, the contents of the sump collection tank were allowed to settle. Only a few small droplets of glycerol were discovered when draining the tank. This may have been as result of the strong emulsion in the liquid or the flow rate into the centrifuge being too high.

3.2.5 Cooling

A steel coil tube was mounted in the mixer tank for cooling the reaction mixture after centrifugation. When the cooling started, the water in the cooling tank was at 20°C and the reaction mixture was at 50°C. After the cooling process had proceeded for 10 minutes, the cooling water was at 40°C. The reason for this was likely due to the cooling unit being too low power to continuously cool the water.

3.3 Results

The product of the test was approximately 13 liters of reaction mixture, which was stored in four containers of three liters and one container of 1.5 liters. The reaction mixture was drained from the bottom of the mixer into the containers. The containers were labeled sequentially, with container 1 being the first liquid drawn from the mixer, and so on. After the liquid had been allowed to settle for a week, container 1 consisted of two liquid phases as can be seen in figure 3.6. Container 2 and 3 was also in a two-phase system, but the dark phases were significantly smaller than in container 1. Similarly, container 4 had two phases, with the bottom phase accounting for approximately half of the volume. Container 5 consisted of approximately one liter in one phase of light brown non-viscous liquid.

Figure 3.6: The product in container 1

Although the liquid in the containers was not tested, some assumptions can be made with regards to their contents. There was a darker and more viscous phase present in the bottom of container 1,2 and 3. It is likely that this substance was glycerol due to the high density and viscosity of this substance [57, 58]. Glycerol can be the product of hydrolysis of FFA [45], but as no significant amounts of water was present during testing, this is considered unlikely. Therefore, it stands to reason that if there was glycerol present, FAME was produced. The contents of container 5 and the top of container 4 was most likely the unreacted methanol, due to methanol having the lowest density and viscosity [57, 58].

The total amount of glycerol from the process can be approximated to be 200 ml, which corresponds to 0.24 kg [57]. Assuming that the feedstock consisted solely of TAG with oleic acid groups, the amount of each reactant needed to produce this amount of glycerol can be calculated from the stoichiometric reaction equation for transesterification in Figure 2.11. From the balanced equation, 0.25kg of glycerol will correspond to 2.43 kg of FAME (appendix A). The density of the oil that was used was found to be 0.91 kg/L by measurement, meaning that the added weight of oil was 9.1 kg. The FAME yield of the transesterification process can be calculated from the formula in equation 4 [59].

$$Yield\ (\%) = \frac{Weight\ of\ FAME\ produced\ (g)}{Weight\ of\ oil\ used\ (g)} \times 100\% \qquad \text{Eq. 4}$$

A FAME yield of approximately 27% was obtained during the preliminary test (appendix A). The low yield was likely due to some key factors. The absence of a mixer in the mixing tank likely created a two-phase system where most of the methanol was on top of the feedstock. Furthermore, the temperature during the process was between 50 and 60 °C, which is slightly lower than most commonly employed transesterification temperatures. Finally, the calcium oxide was not calcinated and was not properly stored. This may have resulted in poisoning of the catalyst by air or moisture.

4 Description of the Production Line

The current version of “BioMax” is the continuation of “BioMax V3” that was built during the preliminary work for this thesis. “BioMax” is a small-scale FAME or FAEE production line for education and research activities. The production is based upon heterogeneous catalyst technology with an acid-catalyzed esterification and a base-catalyzed transesterification. After the preliminary testing, the production line has been improved further to fix the previous issues. Figure 4.1 shows a schematic representation of the improved production line.

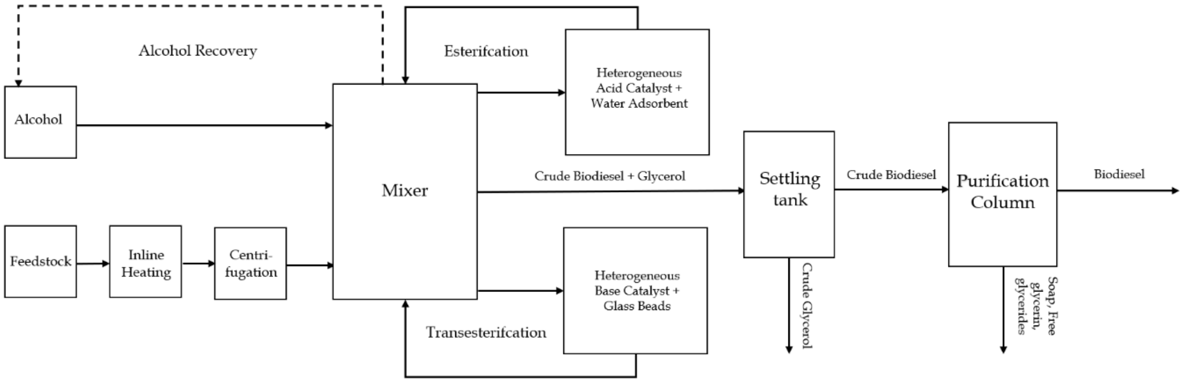


Figure 4.1: The biodiesel production method for BioMax

The referenced literature on biodiesel production indicates that there are certain parameters that are of high importance. To enable optimization of the production line at a later stage, it has been set up so that these parameters can be varied by the control system. The main parameters that can be varied by the control system are:

→ Reaction temperature
→ Reaction time
→ Flow
→ Mixing speed

A production volume between 6 and 13 liters of reaction mixture can be operated with at the production line (appendix B). Additional space has been left on the framework and hoses has been used, so that components can easily be added or removed. The production line should be able to accommodate a variety of feedstocks. The six main steps of the biodiesel production line can be outlined in the following order:

1. Pretreatment and heating of feedstock
2. Esterification
3. Transesterification
4. Alcohol recovery
5. Glycerol removal
6. Washing of product

Due to limited time, only steps 1 to 5 will be built and only steps 3 to 5 will be tested, as refined vegetable oil will be used as the feedstock during testing. However, the description will include all process steps and the production line’s functional capabilities.

4.1 Overall Process Description

To start the production process, alcohol, feedstock and catalyst must be added to the system. The feedstock is added to the process through gravity feeding, either directly into the centrifuge or by adding it to the oil tank.

If pretreatment of the feedstock is needed, adsorbents must be added together with an acid catalyst. Moisture is removed from the feedstock by heating and centrifugation. Once the centrifugation is finished, and the feedstock has reached the temperature setpoint, the feedstock will be ready for esterification.

To start the esterification, alcohol is added either by pump mixing or by addition through the oil tank. The liquid is then circulated for a certain period between the mixer tank and PBR 2. Water formed during the esterification process is removed by using adsorbents. If refined feedstock is used, the centrifugation and esterification steps are not needed. Transesterification can then start once the feedstock has reached the temperature setpoint. Alcohol is then added again, and the liquid is circulated between the mixer tank and PBR 1 for a certain period of time.

Once the transesterification is finished, the first step is to remove the alcohol. This is done by raising the temperature in the mixer tank to a setpoint above the alcohol's boiling point. The evaporation continues until it is stopped on the display unit or by a predefined timer. The alcohol vapors are continuously led out through the condenser and away from the production line into a receptacle.

After the alcohol is removed, the agitator is stopped, and the liquid is allowed to settle in the mixer tank for a certain period of time. The reaction mixture is then transferred to the glycerol removal tank, where the glycerol is drained off. The crude biodiesel can then be purified by either water washing or dry washing.

4.2 Design Description

The design of the biodiesel production line is based on the recommendations for further work from Reinaas's thesis [12], the preliminary work and practicality. A stirred tank system has been constructed and implemented to ensure a proper mixing of alcohol and lipid feedstock.

4.2.1 Framework

The main consideration with regards to re-designing the framework, has been to make the production line more functional and efficient. The framework used during the preliminary work has been raised 50cm to create more room for components.

Figure 4.2: The framework.

The aluminum pallet allows for transporting the production line by forklift or pallet jack to the desired location of operation. The framework is made from welded 30x30mm construction steel hollow profiles. Parts of the framework and the pallet is covered with aluminum sheet metal to enable multilevel placement of components. Some space has been designated at the end of the production line to allow for placing a permanent stationary computer. The process equipment is mainly fastened to the framework by bolted connections with aluminum plates and mounts. The construction steel in use at the production line has not been surface treated, so corrosion may occur during prolonged exposure to moisture.

4.2.2 Stirred Tank Design

Figure 4.3: Stirrer constructed by the workshop at NMBU.

The preliminary test results showed that the previously established biodiesel production line needed an agitator to create better heat transfer and mixing characteristics. As there was an extra VFD from the first version of BioMax, it was decided to repurpose one of the pump motors and a VFD to the agitator. The agitator was created by the mechanics at the workshop at NMBU. The stirrer shaft is connected to a motor driven by a VFD, allowing for adjustable stirring speed. All metal components and welds used for the stirrer mechanism are stainless steel to prevent corrosion. The stirrer shaft is led though a nylon bushing with a sealing system to prevent vapors from exiting the mixer. Nylon has a low reactivity with both biodiesel and methanol, in addition to being able to operate under the process temperatures [60]. The blades on the stirrer are connected to the shaft by welded-on lugs with bolts and locknuts. The stirrer blades are replaceable to allow for future testing and optimization of the mixing process. The VFD controlled motor is able to provide stirring speeds from approximately 30 to 1360 rpm. The production drawing for the assembly can be seen in appendix C.

Figure 4.4: Visualisation of the stirred tank configuration in the mixer tank

4.2.3 Electrical System

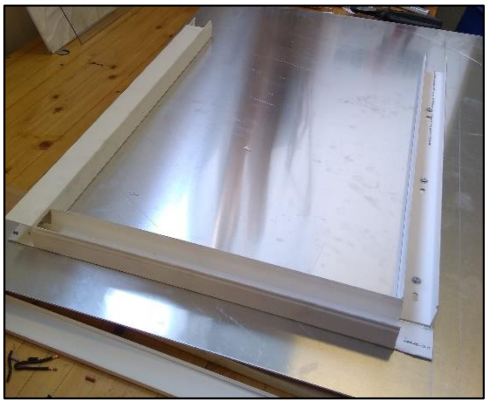


Figure 4.7: Plastic cable ducts for cable management.

The wiring from the process equipment has been placed into plastic cable ducts at the most exposed areas to reduce the chance of damaging or wetting wires during the operation of the production line. The wiring for the 230VAC equipment has been sized according to the current-draw of the appliances. All 230VAC equipment, except the heaters and the 24VDC power supply, is earthed to the framework of the production line, which in turn is grounded to the power supply. The contactors used for controlling the power supply to the one-phase pumps and the centrifuge are equipped with thermal overload relays, giving protection in the event of motor overload or phase failure. The control valves and the transmitters have an ingress protection of IP65 or higher. All electrical connections are shielded to at least IP44 [61]. A residual-current device (RCD) has not be been connected to the power source. However, this is an essential safety feature that must be implemented before further testing and operation of the production line. The setup of the components in the electrical cabinet can be seen in figure 4.6 and 4.7.

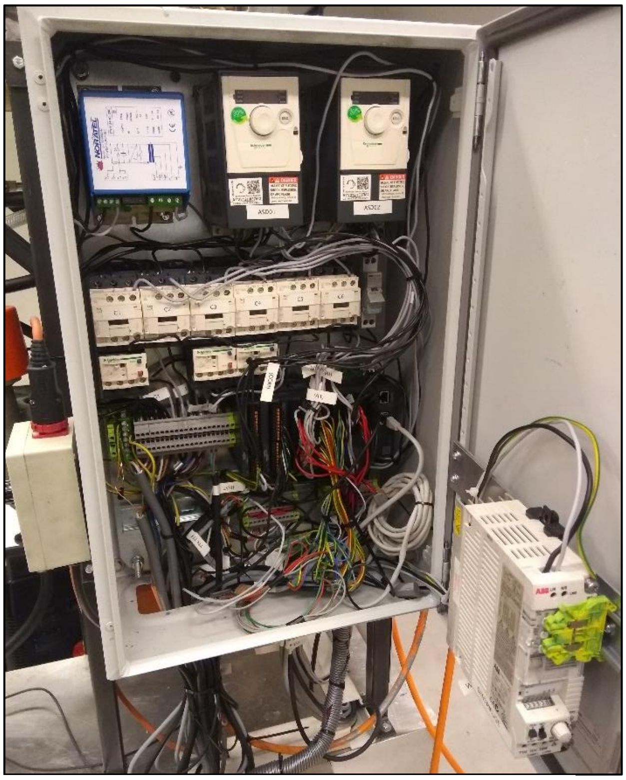


Figure 4.6: The electrical cabinet on "BioMax"

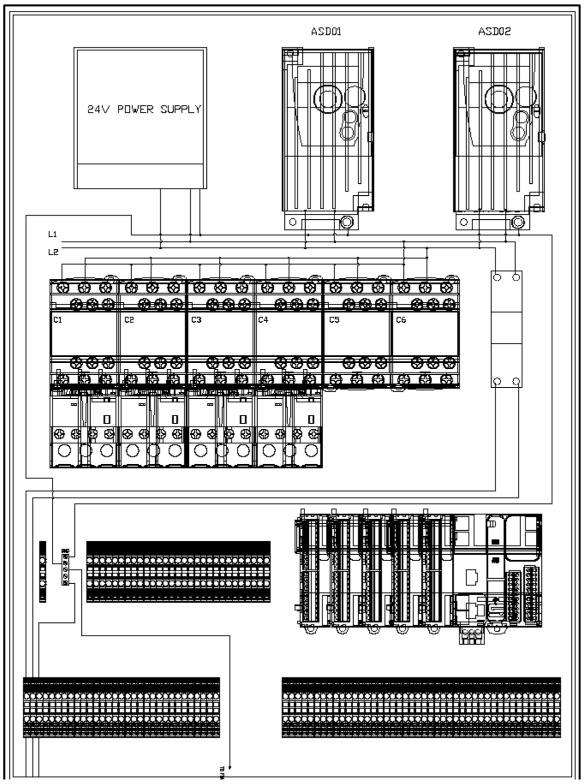


Figure 4.5: Schematic of the wiring of the electrical cabinet

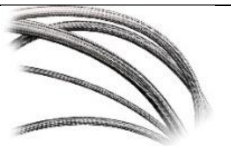
The complete wiring diagram for the electrical system can be found in appendix D.

4.2.4 Piping

The tubing, valves and fittings of the production line has been chosen for their chemical resistance and their temperature and pressure ratings. All piping components are of ½" size.

The piping used for all processes that requires contact with ethanol, methanol, biodiesel and oil are wire braided hoses from TESS. The hoses are lined with PTFE which is chemically compatible for the use with methanol, ethanol, esters and oil [62]. The PTFE hoses were acquired because the previously used fluoroelastomer (FKM) hoses were found to be unsuitable for processes requiring the use of methanol [63]. For the alcohol recovery system, where only water is used, PVC tubing is used.

Table 4.1: Tubing

Component	Description	Internal Tubing Material	Pressure Rating	Temperature rating	Picture
Water Hose	Hose for water in the alcohol recovery system	PVC	11 Bar	-10 to +60 °C	 *Figure 4.8: Miljøtex Hose [64]*
Gasohol Hose	Hose for Oil and Biodiesel without methanol	FKM	20 Bar	-40 to +135 °C	 *Figure 4.9: Gasohol Hose [65]*
Teflon Hose	Hose for reaction mixture	PTFE	161 Bar	-60 to +260 °C	 *Figure 4.10: Tessflon Hose [66]*

Pipe fittings such as 90^0 elbows, t-fittings and nozzles conforming to the piping class rating cl150, meaning a pressure rating of 20 Bar [67], are used at the production line. To keep the piping system sealed, plumber's hemp and Loctite 577 has been applied to all threaded joints.

Table 4.2: Fittings and Valves

Component	Description	Material	Pressure Rating	Picture
T-Fittings	3-way ½" fitting	Stainless Steel / Bronze	20 Bar (cl 150)	 *Figure 4.11: T-Fitting from TESS [67]*
90º Elbows	90-degree ½ "fitting	Stainless Steel / Bronze	20 Bar (cl 150)	 *Figure 4.12: 90-degree elbow from TESS [68]*
Nozzles	½" nozzle	Stainless Steel	20 Bar (cl 150)	 *Figure 4.13: Nozzle from TESS [69]*
Check Valves	Pressure activated check valves	Brass	16 Bar	 *Figure 4.14: Check valve from Biltema*
Manual Valves	2-way ball valves with attached levers for manual draining.	Stainless Steel	69 Bar	 *Figure 4.15: Manual Valve from TESS [70]*

4.2.5 The Production Line

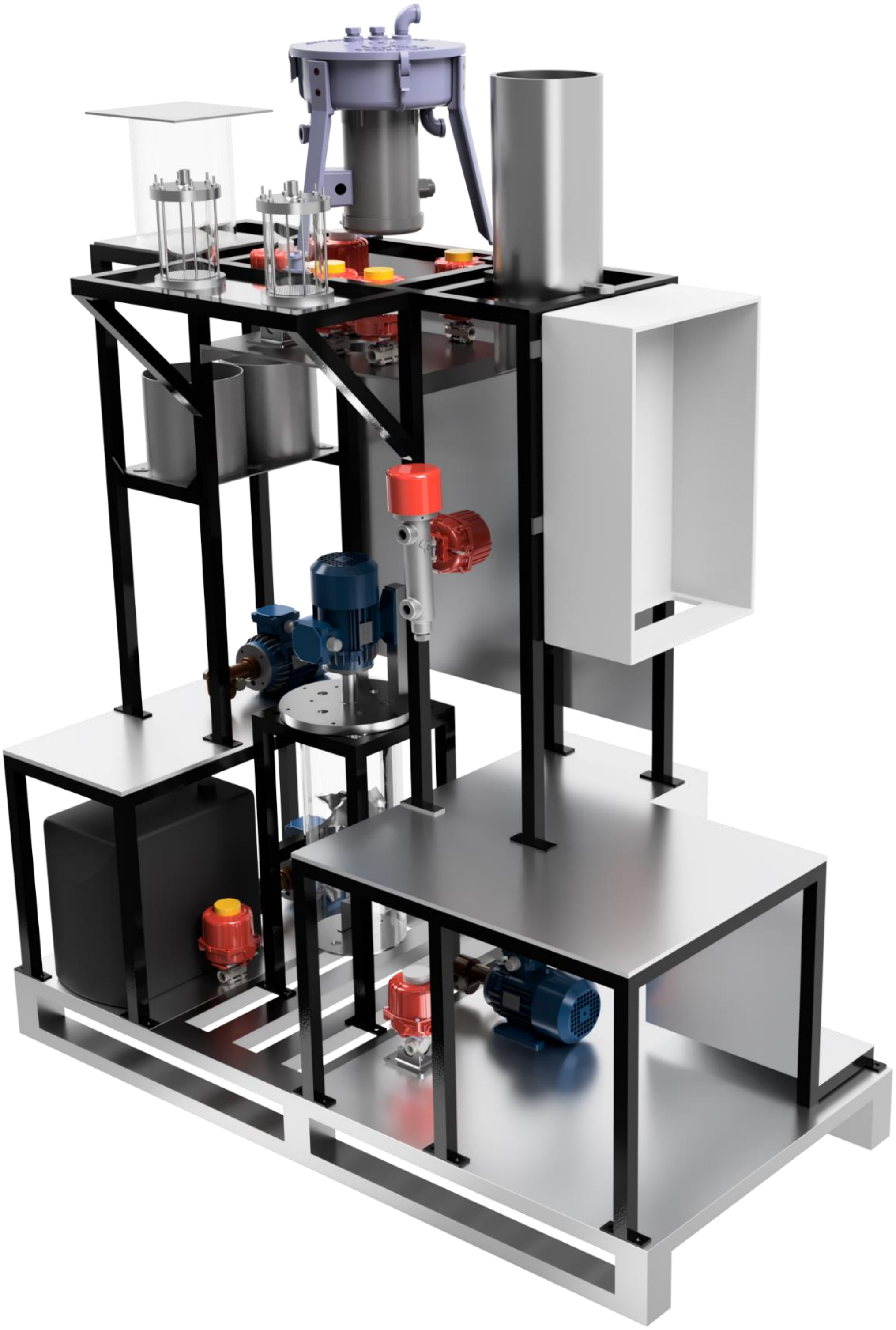


Figure 4.16: Design of the 2nd generation of "BioMax"

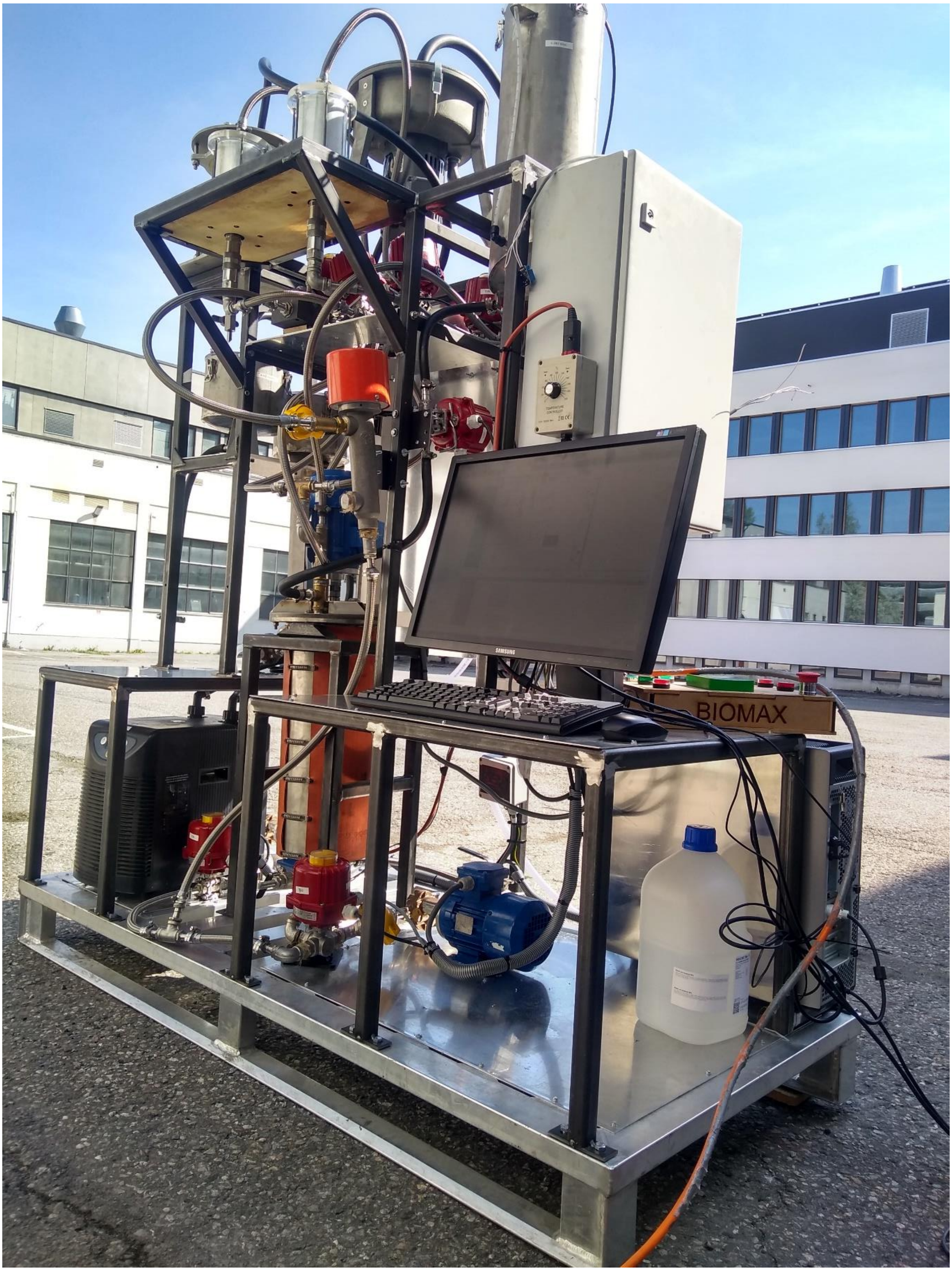


Figure 4.17: The 2nd generation of "BioMax"

4.3 Description of Components

4.3.1 Process Control Equipment

Figure 4.18: Built-in relief valve

There are three pumps in use at the production line: a water pump, an alcohol pump and a main process pump. The water and the alcohol pumps have set flow rates at approximately 3 L/min. The main process pump is controlled by a VFD, which is needed in order to adjust the flow for the different processes. The pumps are self-priming and has built-in pressure relief valves that are set to 3.5 bar [71] (figure 4.18 and 4.19). The datasheet for the pumps can be seen in appendix E.

Figure 4.20: The inside mechanics of the gear pump

The heating equipment is an inline heater and a jacket heater. The inline heater provides relatively quick heating of liquid and the jacket heater provides continuous heating of liquid in the mixer tank. Continuous cooling of water flowing through the heat exchanger is done by a 165W cooling unit. The lowest temperature setting on the cooler is 12℃.

A centrifuge is employed at the production line for pretreatment of the feedstock or glycerol separation. The centrifuge is controlled by a VFD which allows the centrifugation speed to be adjusted up to 6000 rpm [72]. There are three inlets and two outlets as can be seen in figure 4.20. The outlets are for the sump and the processed oil.

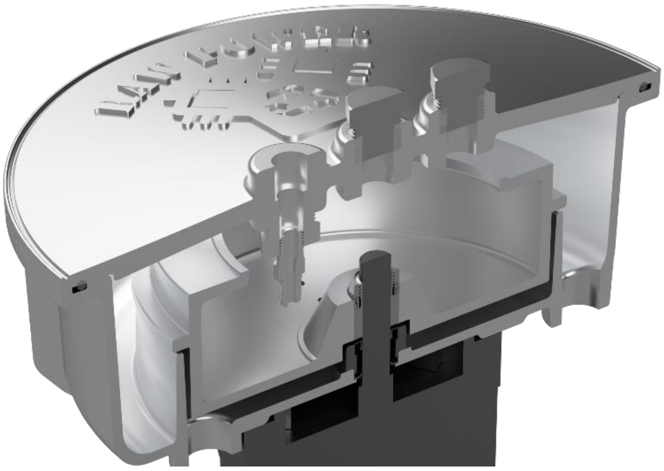
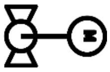
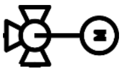


Figure 4.19: Cross-section of the centrifuge

The control valves consist of an actuator housing mounted on two- or three-way valves. The valves are opened or closed by a 24VDC servo motor. The valve position can be adjusted by mechanically positioning connector lobes within the actuator housing (appendix F). Table 4.3 shows the equipment used to control the processes on the production line.

Table 4.3: Process Control Equipment

Component	Description	#	V_{in}	W	IP	P&ID	Picture
Gear Pump	Oberdorfer N999RE Self-priming gear pump with a built-in pressure relief valve set to 3.5 Bar.	3	230 VAC	370W	IP65		 *Figure 4.21: N999RE Oberdorfer gear pump [71]*
Inline Heater	Inline Heater from Tempco with an adjustable thermostat up to 80°C.	1	230 VAC	1000W	IP44		 *Figure 4.22: Tempco inline heater [72]*
Jacket heater	From Norske Backer with an adjustable thermostat from 40°C to 200°C.	1	230 VAC	1800W	IP44	-	 *Figure 4.23: Jacket heater from "Norske Backer"*
Cooler	Hailea HC 150A cooling unit with recommended flow rate between 4 and 20 l/min.	1	230 VAC	165W	-		 *Figure 4.24: Hailea water cooling unit [73]*
Centrifuge	WVO Raw Power bowl centrifuge with recommended flow rate of approx. 1.0 l/min at 6000 RPM and 65C. 3800G.	1	230 VAC	1100W	-		 *Figure 4.25: WVO raw power centrifuge[74]*
Actuated Valves	2- and 3-way RCEL003 actuated valves with a revolution time of 12 and 36 seconds, respectively	7	24 VDC	5W	IP67	 	 *Figure 4.26: RCEL003 actuator housing [75]*

4.3.2 Electrical Control Equipment

A Modicon M221 PLC from Schneider Electric has been employed on the production line. Five expansion modules have been added to the PLC to accommodate the number of components in use on the production line. A TMH2GDB display unit is used for accessing the PLC during operation. The control system is powered by a 24VDC 180W power supply.

There are three VFDs in use on the production line: One for the main process pump, one for the agitator and one for the centrifuge. The process pump and the agitator use Altivar 312 VFDs which are controlled from the PLC. The centrifuge is controlled by an ACS55 VFD, which uses a manual knob to adjust the speed. Contactors are used to turn the 230 VAC appliances on or off. Thermal overload relays are connected to three contactors. If the motors connected to the contactor experiences overload or phase failure, the thermal overload relays will turn off the power to the connected appliance.

Table 4.4: Electrical Control Equipment

Component	Description	#	Voltage	Effect	Picture
PLC	Schneider Electric TM221M16R PLC controller with 8 DO, 8 DI and 2 AI.	1	24 VDC	22.5W with max. amount of expansion modules.	 Figure 4.27: TM221M16R PLC from Schneider Electric [76]
Display Unit	Schneider Electric TMH2GDB 240x160px display unit.	1	5VDC (Modbus)	1 W	 Figure 4.28: TMH2GDB display unit from Schneider Electric [77]
PLC DI Expansion Module	Schneider Electric TM3DI16G DI expansion module with 16 DI.	1	24 VDC	N/A	 Figure 4.29: TM3DI16G DI module from Schneider Electric [78]
PLC DO Expansion Module	Schneider Electric TM3DQ16R DO expansion module with 16 DO.	2	24 VDC	N/A	 Figure 4.30:TM3DQ16R DO module from Schneider Electric [79]

PLC AI Expansion Module	Schneider Electric TM3AI8G AI expansion module with 8 AI.	1	24 VDC	N/A	 *Figure 4.31: TM3AI8G AI module from Schneider Electric [80]*
PLC AI/AO Expansion Module	Schneider Electric TM3AM6 AI and AO expansion module with 4 AI and 2 AO.	1	24 VDC	N/A	 *Figure 4.32: TM3AM6 AI/AO module from Schneider Electric [81]*
24 VDC Power supply	NORATEL DC power supply for instruments and control. Max 10% ripple voltage.	1	230 VAC	180W (55W heat dissipation)	 *Figure 4.33: 24VDC Power supply from Noratel [82]*
Variable Frequency Drive	Altivar 312 0.37 kW variable frequency drive for one-phase connections with a speed range from 1 to 50.	2	230 VAC	370W (41W heat dissipation)	 *Figure 4.34: Altivar 312 VFD from Schneider Electric [83]*
Variable Frequency Drive	ABB ACS55 1.1 kW variable frequency drive for one-phase connections with a potentiometer for speed control.	1	230 VAC	1100 W (74W heat dissipation)	 *Figure 4.35: ACS55 VFD from ABB [74]*
Contactors	Schneider Electric TeSys LC1D09BD Contactors.	6	24 VDC	N/A	 *Figure 4.36: Contactor from Schneider Electric [84]*
Overload Relay	Schneider Electric LRD08 overload relay for protection against motor overload.	3	230 VAC	N/A	 *Figure 4.37: Overload relay from Schneider Electric [85]*

4.3.3 Sensors

The sensory equipment on the production line consists of temperature transmitters, a level transmitter, a flow transmitter and two gas sensors. The 24VDC sensory equipment are analog inputs and are wired as current giving devices at 4-20mA.

The temperature transmitters are thermocouples, meaning that the resistance of the electrical conductor within the sensor will change with the changes in temperature. The level transmitter has a piezo resistive element, that will change resistance when the sensing element is under stress from hydraulic pressure. The flow transmitter works on the principle of electro-magnetically induced voltage. There are turbines within the enclosed housing that will revolve when there is a flow through the transmitter. The movement is detected by an electromagnetic pickup on the outside of the enclosed housing that sends a signal to the PLC.

Six illuminated pushbuttons, two LED lights and an emergency stop pushbutton have been employed to operate the production line. The pushbuttons and LEDs have been mounted on a laser cut control panel along with the display unit.

In addition to the 24VDC sensors, an alcohol sensor configuration has been created by students at Eik Ideverksted by using a MQ-3 gas sensor with additional control equipment. The MQ-3 gas sensor is able to sense alcohol from a concentration of 0.05 to 10 ppm [86]. The alcohol sensor configuration can be seen in figure 4.38.

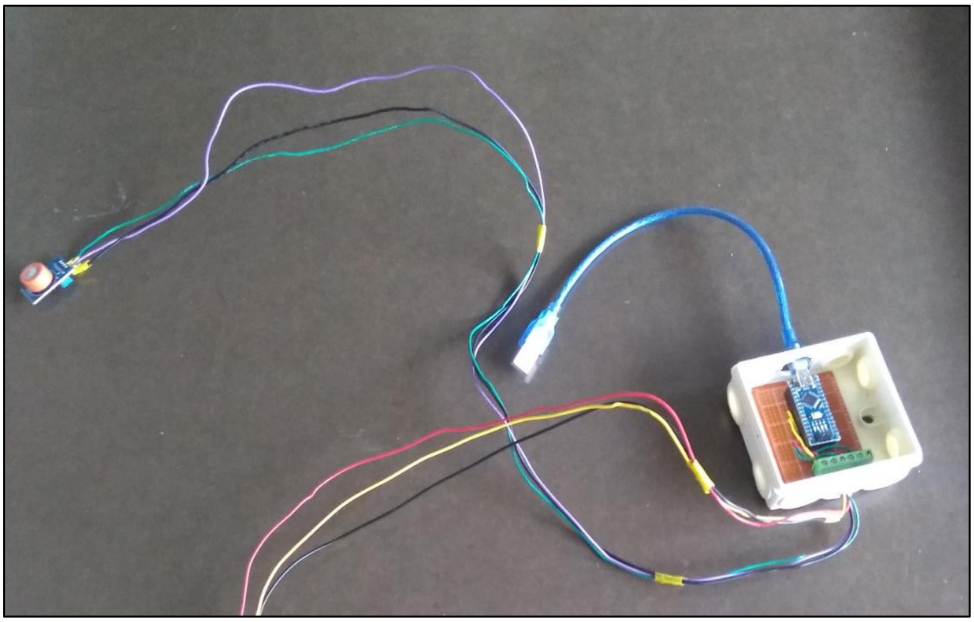


Figure 4.38: The alcohol sensor setup. Wiring schematic can be found in appendix D

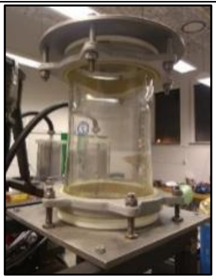
Table 4.5: Sensors and Inputs

Component	Description	#	V_{in}	Signal out	Signal Type	Picture
Level Transmitter	SGE-25 piezoresistive level transmitter for oil and water applications with range from 0 to 1000mm.	1	24 VDC	4-20 mA	Analog	 *Figure 4.39: SGE-25 level transmitter from OEM Automatic [87]*
Temperature Transmitter	CTGB1 Temperature Transmitter with range from 0 °C to 150 °C.	4	24 VDC	4-20 mA	Analog	 *Figure 4.40: CTGB1 temperature transmitter from OEM Automatic[88]*
Flow Transmitter	VHZ-020GA050E flow transmitter with range from 0.5 to 50 l/min for viscous fluids (1-2500cSt) up to 80 °C.	1	24 VDC	4-20 mA	Analog	 *Figure 4.41: VHZ-020GA050E flow transmitter from OEM Automatic[89]*
Pushbuttons and lights	Illuminated pushbuttons, LED lights and an emergency stop pushbutton from Schneider Electric and Biltema.	6 2 1	24 VDC	24 VDC	Discrete	 *Figure 4.42: Laser cut wooden control panel with pushbuttons from Schneider Electric and Biltema*
Alcohol Sensor	Alcohol sensor consisting of a MQ-3 sensor, a 5V relay and an Arduino Nano.	1	230 VAC	24 VDC	Analog / Discrete	 *Figure 4.43: MQ3 gas sensor from Banggood.com [86]*

4.3.4 Vessels

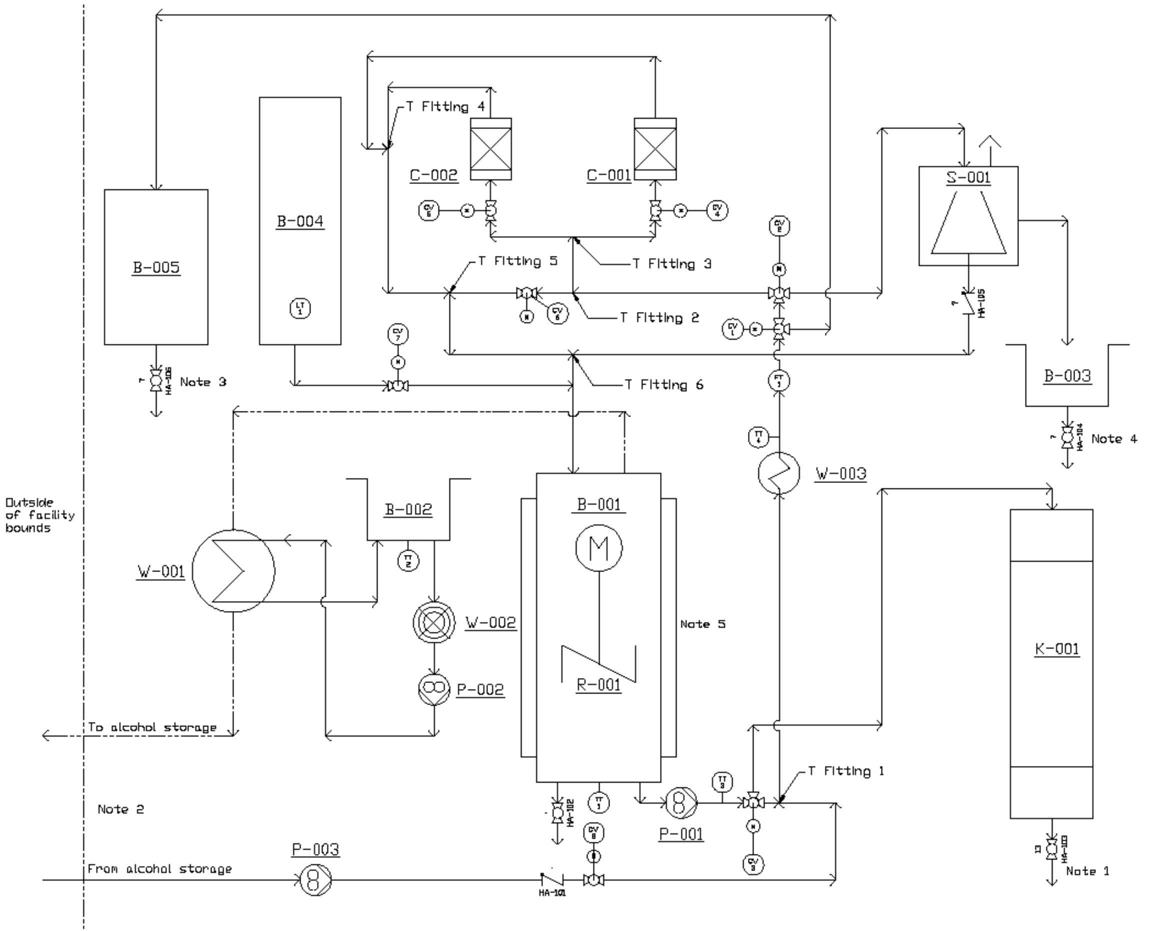
Table 4.6: Vessels

Component	Description	Purpose	Capacity	Picture
Water Tank	Tank for containing the cooling water.	Acts as a reservoir for water in the alcohol recovery system.	Approx. 6 liters	 *Figure 4.44: Water tank made by the workshop at NMBU*
Centrifuge Sump Collection Tank	Tank for collecting the centrifuge sump.	Catches the contents of the centrifuge upon centrifuge shutdown (approx. 0.7L).	Approx. 6 liters	 *Figure 4.45: Centrifuge sump collection tank made by the workshop at NMBU*
Oil Tank	Tank for adding oil to the system with a level gauge.	Works as a filtering, storage and measurement tank.	Approx. 16 liters	 *Figure 4.46: Oil tank made by the workshop at NMBU*
Mixer Tank	Stirred tank containing the mixing process and the reaction mixture.	Heats and mixes the reaction mixture. Works as a junction point for all processes.	14.7 Liters	 *Figure 4.47: Vessel made by Dynatec AS and mixer assembly made by the workshop by NMBU*

Purification Column	Biodiesel purification column for washing of biodiesel.	Works as a vessel for containing the purification process. Can be used in a dry or wet wash configuration.	Approx. 16 liters	 Figure 4.48: Washing Column
Glycerol removal tank	Glass vessel for glycerol removal and visual inspection of the finished biodiesel.	For visual inspection and for draining the glycerol from the biodiesel after the trans-esterification.	Approx. 6 liters	 Figure 4.49: Glass vessel
Condenser	Shell and tube heat exchanger with inlayed steel wool for alcohol condensation.	For cooling the alcohol vapor coming out from the mixer tank.	N/A	 Figure 4.50: Condenser
Packed Bed Reactors	Two identical packed bed reactors for esterification and trans-esterification.	Used for containing the catalyst during the esterification and transesterification processes.	Approx. 1 liter	 Figure 4.51: Packed bed reactors made by the workshop at NMBU

4.4 P&ID

The piping and instrumentation diagram (P&ID) can be seen in figure 4.52. The full drawing can be seen in appendix G.



LEGEND

Tag	B-001	B-002	B-003	B-004	B-005	W-001	W-002	W-003	W-004	K-001	S-001	C-001	C-002
NAME	Mixer tank	Water tank	Centrifuge sump collector tank	Oil tank	Glycerol removal tank	Shell and tube heat exchanger	Cooler unit	Inline heater	Jacket heater	Wash column	Centrifuge	Packed bed reactor 1	Packed bed reactor 2

Figure 4.52: P&ID for BioMax

5 Process Parameters

Due to the previous research of Omberg [10] and Reinaas [12], the production line is based on heterogeneous catalyst technology. A method of producing biodiesel based on the literature study and the previous work will be proposed. This section will go through how biodiesel can be made from both refined and unrefined feedstock on "BioMax".

5.1 Feedstock

The production line should be able to handle different feedstocks for biodiesel production. However, the limiting factor is that the feedstock must be liquid and coarsely filtered before adding it to the production line. Moisture and solids in the feedstock are removed by heating the feedstock and adding it to the centrifuge. A commercially available refined feedstock (figure 5.1) will be used during testing to simplify the production process and to ensure a proper transesterification process before attempting esterification. Sanford et al. [25] determined the fatty acid profile of sunflower oil as can be seen in table 5.1 by using Ce 1c-89 from AOCS.

Figure 5.1: Commercially available sunflower oil

Table 5.1: Fatty Acid Profile of Sunflower Oil [25]

Fatty Acid Designation and Name	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	C20:0	C22:0	C24:0	C24:1
	Palmitic	Palmitoleic	Stearic	Oleic	Linoleic	Linolenic	Arachidic	Behenic	Lignoceric	Nervonic
Sunflower Oil	4.2	-	3.3	63.6	27.6	0.2	-	0.7	0.4	-

As sunflower oil mainly consists of fatty acids with 18 carbon atoms, and most of it is oleic acid, the simplification of TAG molecules with 100% oleic acid will be made in the mass balances.

5.2 Alcohol

Although the production line has been designed for the use of both ethanol and methanol, ethanol has been chosen as the alcohol for the transesterification process, mainly due to safety concerns. Rectified ethanol of 99% purity will be used to minimize the addition of water into the process. The transesterification equation is balanced by a molar ratio of 3:1 of alcohol to oil. A higher molar ratio is often used in biodiesel production to push the production over to the product side of the equation. The reaction temperature will be set to 70 °C as this is close to the boiling point of ethanol. For the test, a molar ratio of 6:1 will be used as the ethanol to oil ratio.

5.3 Esterification

For the esterification, a solid acid catalyst needed. Cationic ion exchange resin, such as Amberlyst 15 and Amberlyst BD20, can be used as heterogeneous acid catalysts. Park et al. [50] utilized a 20 wt% loading of Amberlyst 15 or Amberlyst BD20 and a methanol to oil ratio of 6:1. The FFA content was reduced from 2.5 wt% to below 0.5 wt% in under 2 hours. It was reported from the study that water formed during the esterification reduced the activity of the catalyst.

Water has a molecular size of 2.8 angstrom [90], which is significantly smaller than the other substances involved in the process. Zeolites are natural adsorbents that can be used for removing substances from the process, based on the molecular size of the substance. Zeolites with sieves down to 3 angstroms are commercially available, that can adsorb 21 wt% of their own weight in water [91]. The mass balance for esterification of UCO with 2.72 wt% FFA content can be seen in figure 5.2.

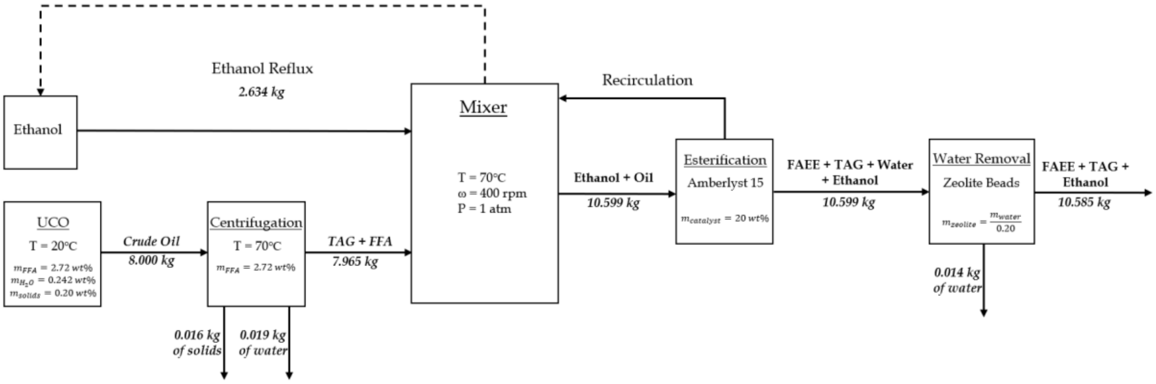


Figure 5.2: Esterification mass balance. Full calculations can be found in appendix H.

It is important to note that the process drawings in figure 5.2 and figure 5.3 assumes 100% efficiency for all processes, although this is not realistic in practice.

5.4 Transesterification

Lab-grade calcium oxide was used during the preliminary work. Calcium oxide is a strong alkaline catalyst that can be used in a packed bed reactor or in a stirred tank reactor. Calcium oxide will typically require thermal activation at high temperatures to remove adsorbed CO_2 and moisture [47]. Calcium oxide will be used as the catalyst for transesterification during the test with ethanol and refined sunflower oil. The drawing in figure 5.3 shows the transesterification of the pretreated UCO from figure 5.2.

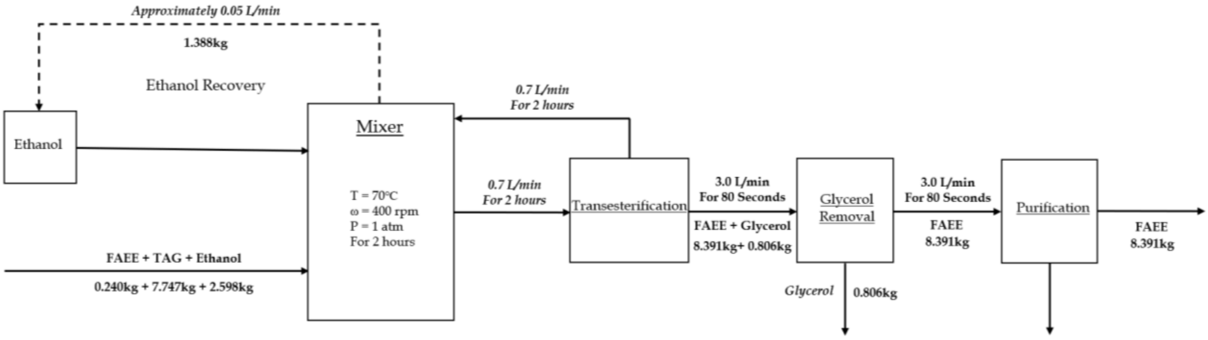


Figure 5.3: Transesterification mass balance. Full calculations can be found in appendix I.

5.5 Purification

In reality, the product in figure 5.3 will not be pure FAEE. Until optimization of the esterification and transesterification process, a total conversion of TAG to FAEE is unrealistic, and therefore unreacted feedstock will be present in the biodiesel. Although the bulk of the glycerol in the biodiesel can be effectively removed by gravity, the free glycerol cannot.

The purification method must be determined by testing to find out which approach is the most effective. The advantage of using a dry purification process is the reduction in washing time and no added steps for water removal [53]. The water washing method on the other hand is simple and does not require regeneration.

5.6 Process Flow Diagram

The PFD for the overall process can be seen in figure 5.4. The proposed flow rates are based on equipment ratings and experiences from the preliminary work.

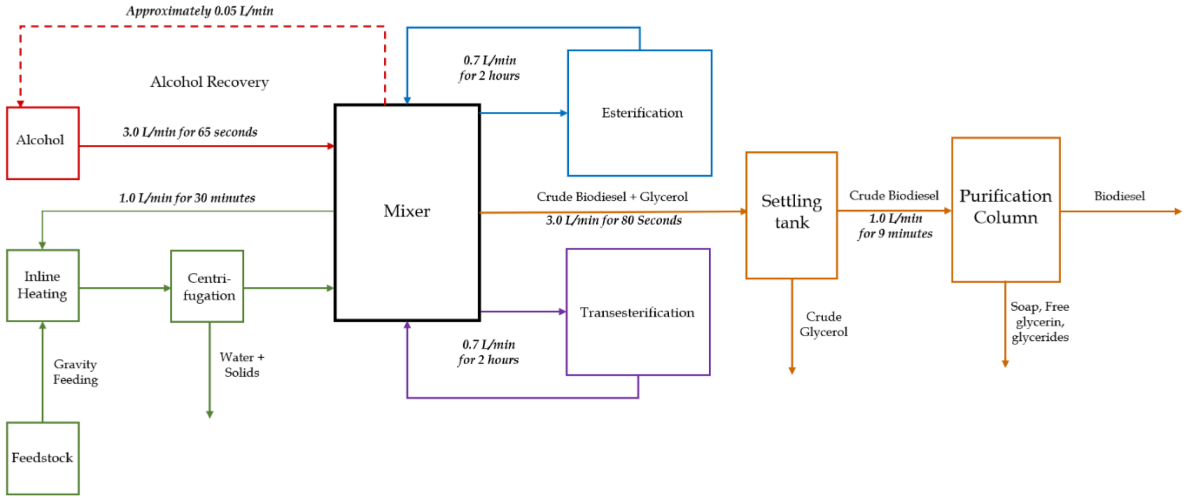


Figure 5.4: PFD for the overall process. The PFD can be seen in appendix J.

5.7 Mass Balance for Testing the Production Line

For the test, an amount of 8 kg of sunflower oil will be used and the molar ratio of ethanol-to-oil is 6:1. The sunflower oil is assumed to be solely consisting of TAG molecules, which consists of 3 oleic acids and one glycerol molecule. The TAG molecule can then be identified as triolein with a molecular formula of $C_{57}H_{104}O_6$ and a molecular weight of 885.4 g/mol [92]. The reaction equation can then be written as the following:

$C_{57}H_{104}O_6$	+	$3C_2H_5OH$		$3C_{20}H_{38}O_2$	+	$C_3H_8O_3$
Triolein (TAG)		Ethanol	$\leftrightarrow$	Ethyl Oleate (FAEE)		Glycerol

The calculated amount of each of the reactants for the transesterification process can be seen in table 5.2.

Table 5.2: Mass Balance for the Transesterification Reaction

Type	Name	Mass [kg]	Density@20°C [kg/l]	Volume [L]
Reactants	**Sunflower oil**	8.000	0.91 [93]	8.79
	Ethanol (6:1 ethanol-to-oil)	2.497	0.79 [31]	3.16
Product	**Ethyl Oleate (FAEE)**	8.417	0.87 [94]	9.67
	Glycerol	0.832	1.26 [57]	0.66

The full calculations for the mass balance for the tests with alcohol and oil can be seen in appendix K.

6 Control System

The control of the production line has been developed with some key factors taken into consideration. First, the safety of the operators must be maintained. Dangers related to the use of alcohol in the production process are especially important to account for. If the production line is to be used for education, it is advantageous that the students control the processes. In other cases, where the product is the most important, a more automated solution might be preferable. In both cases the control program will be virtually the same, but the interaction between the operators and the production line will differ. This section will go through the structure of the control system.

6.1 Programmable Logic Controller

The control system is based on a programmable logic controller (PLC). The PLC is a Modicon M221 from Schneider Electric (Figure 4.27). The controller has 8 discrete inputs and 8 discrete outputs along with 2 analog inputs built in. The controller has been expanded with two discrete output modules, a discrete input module, an analog input module and an analog I/O module. A programmable logic controller (PLC) is a microprocessor-based device with a programable memory that stores instructions. The instructions, usually programmed using a ladder program, are used to control processes by using functions such as logic, sequencing, timing, counting and arithmetic [95].

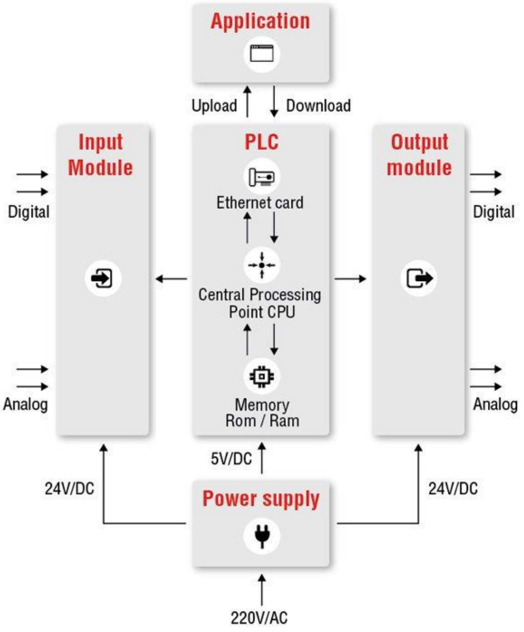
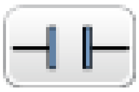
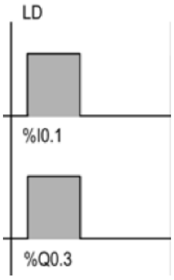
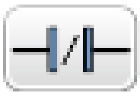
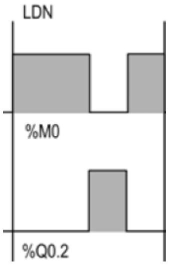
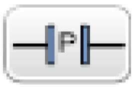
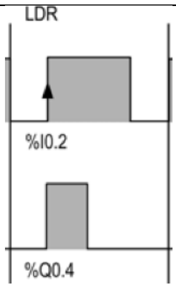
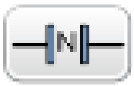
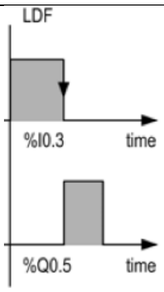


Figure 6.1: Key features of the PLC [96]

6.2 Ladder Programming

Ladder programs are comprised of rungs which consist of two vertical lines that represents power rails. A high output signal will be given once there is a connection between the rails. The ladder program will usually consist of at least a contact and a coil. Typical contacts used by Schneider Electric's SoMachine Basic program can be seen in table 6.1.

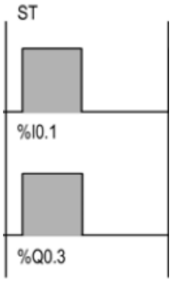
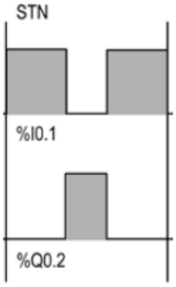
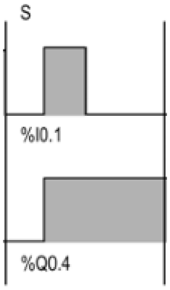
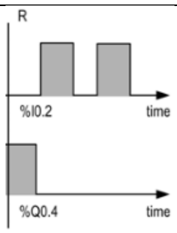
Table 6.1: Typical Contacts in Ladder Programming [97]

Name	Graphic Element	Timing diagram	Function
Normally open contact			High when bit object is high
Normally closed contact			High when bit object is low
Rising edge contact			Detects a change in bit state from low to high
Falling edge contact			Detects a change in bit state from high to low

A bit object, i.e. a memory bit or an input signal from a pushbutton, will be connected to the contact. The type of contact will depend on how the connected bit object is to be interpreted by the program. In the case of a normally open pushbutton, the button will give a rising edge signal once it is pushed, a high signal as it is being pushed and a falling edge signal as it is released. To visualize this, timing diagrams are often used.

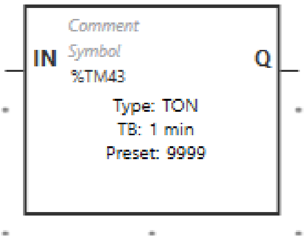
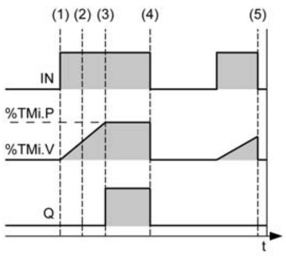
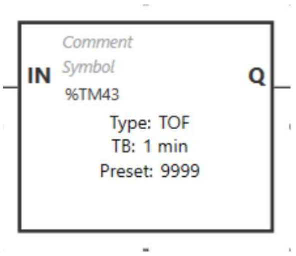
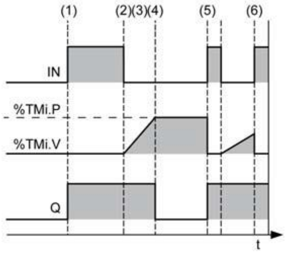
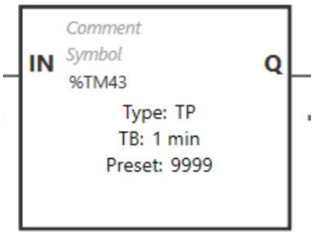
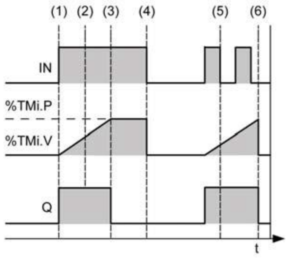
Coils are only used on the right side of the ladder program, as they signify the rung state. An output or a memory bit will typically be connected to the coil. When the direct coil is in a high state, current is allowed to flow through I/O address connected to the output. Set coils will not necessarily signify the rung state, as they will latch as soon as they get a change in signal. To reset a set output or a memory bit, a high signal must be given to a reset coil that is connected to the same output or memory bit. Some common coils are shown in table 6.2.

Table 6.2: Typical Coils in Ladder Programming [97]

Name	Graphic Element	Timing diagram	Function
Direct coil	-( )		Assigns high signal to bit object when rung state is high
Inverse coil	-(/)		Assigns high signal to bit object when rung state is low
Set coil	-(S)		Sets bit object to high when rung state is high
Reset Coil	-(R)		Resets bit object to high when rung state is high

Operation and comparison blocks have been used for writing values to analog outputs or by setting up conditions for analog signals. Different timers can be used for specific process control applications. On-delay timers are typically used to start or stop a process after a certain amount time. Off-delay timers are often used to keep appliances going a certain amount of time after shutdown.

Table 6.3: Typical Functions Blocks in Ladder Programming [97]

Name	Graphic Element	Timing diagram	Function
On-Delay Timer (TON)			Starts counting operation when a rising edge is sensed. High when preset is reached. Retentive or non-retentive.
Off-Delay Timer (TOF)			Starts counting operation when a falling edge is sensed. High while counting. Non-retentive.
Pulse Timer (TP)			Starts counting operation when a rising edge is sensed. High while counting. Retentive.
Operation block		-	Performs a function or operation
Comparison block		-	High when operation expression is true.

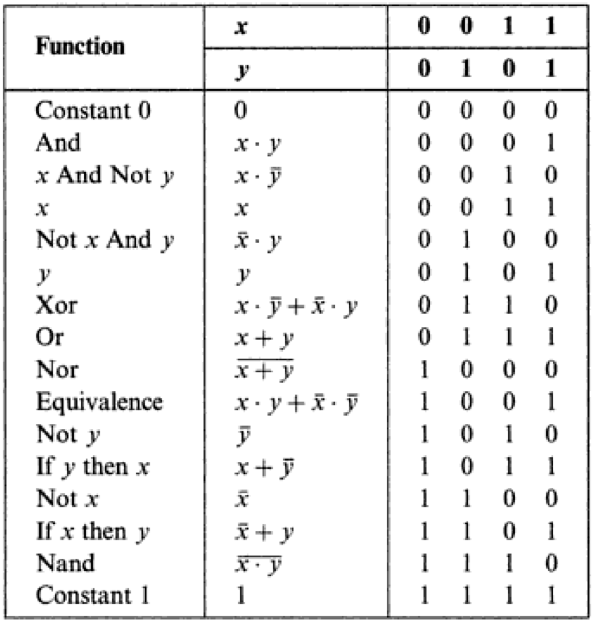
6.3 Ladder Logic

In ladder programming several simple logic principles are used together to create a more complex logic. The method of programming a PLC will often vary depending on the programmer, although there are some fundamental principles and methods that must be understood.

6.3.1 Logic Gates

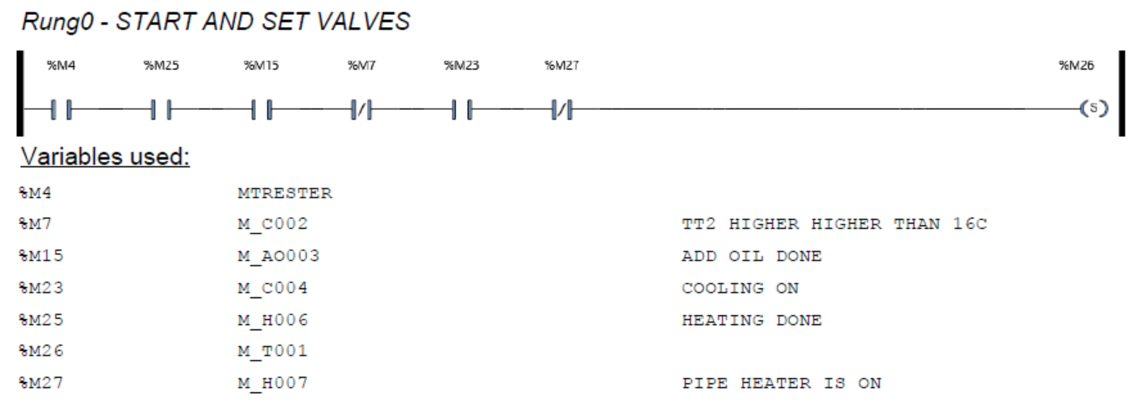
The basis for ladder programming is the use of logic gates, also called Boolean functions. Logic gates such as AND, OR, NAND, NOR and XOR can be used when creating a ladder program. Correct use of Boolean functions can significantly reduce the length of a ladder program, although increasing the complexity. Boolean functions and logic of two variables can be seen in figure 6.2.

The *and* logic gate is used when several conditions must be fulfilled before a task is performed. For instance, before starting the transesterification process, several conditions must be met, as can be seen in figure 6.3.



Function	x	**0**	**0**	**1**	**1**
	y	**0**	**1**	**0**	**1**
Constant 0	0	0	0	0	0
And	$x \cdot y$	0	0	0	1
x And Not y	$x \cdot \bar{y}$	0	0	1	0
x	x	0	0	1	1
Not x And y	$\bar{x} \cdot y$	0	1	0	0
y	y	0	1	0	1
Xor	$x \cdot \bar{y} + \bar{x} \cdot y$	0	1	1	0
Or	$x + y$	0	1	1	1
Nor	$\overline{x + y}$	1	0	0	0
Equivalence	$x \cdot y + \bar{x} \cdot \bar{y}$	1	0	0	1
Not y	$\bar{y}$	1	0	1	0
If y then x	$x + \bar{y}$	1	0	1	1
Not x	$\bar{x}$	1	1	0	0
If x then y	$\bar{x} + y$	1	1	0	1
Nand	$\overline{x \cdot y}$	1	1	1	0
Constant 1	1	1	1	1	1

Figure 6.2: Boolean functions of two variables [98].



Rung0 - START AND SET VALVES

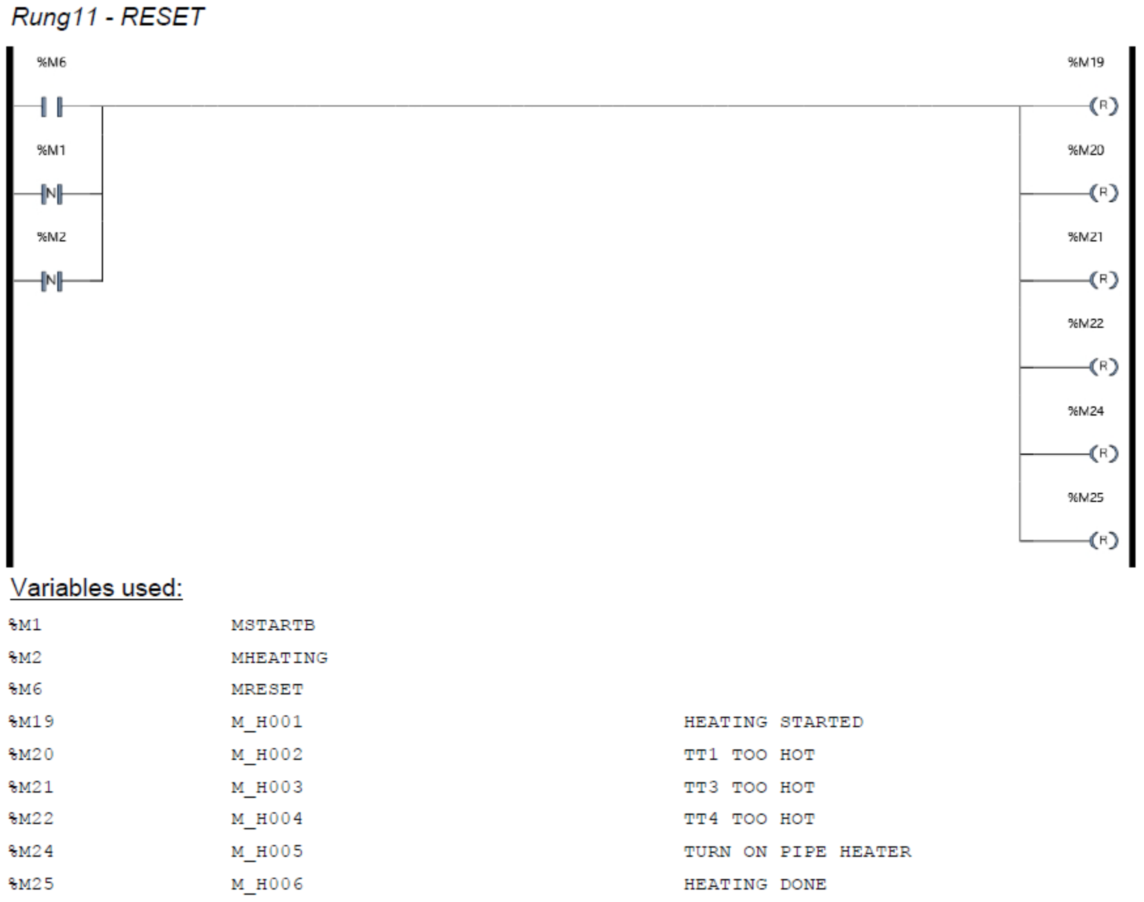
Variables used:

%M4	MTRESTER	
%M7	M_C002	TT2 HIGHER HIGHER THAN 16C
%M15	M_AO003	ADD OIL DONE
%M23	M_C004	COOLING ON
%M25	M_H006	HEATING DONE
%M26	M_T001	
%M27	M_H007	PIPE HEATER IS ON

Figure 6.3: Logic for starting the transesterification program

The pushbutton assigned to “Trester” must be pushed *and* the alcohol recovery system must be on *and* the heating task must be done, and so forth.

The *or* logic gate is often used when at least one condition must be true before a task is performed. This is used in the ladder program when equipment is turned on. SoMachine basic's protocol does not allow for an output to be used in a coil more than once in a program. As outputs must often be turned on or off several times during a process, an *or* logic gate can be used. Figure 6.4 shows the resetting of the memories connected to the heating master task.



Variables used:

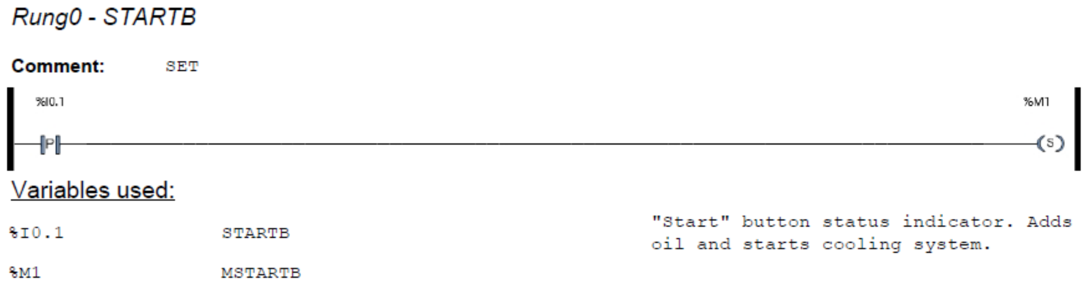
%M1	MSTARTB	
%M2	MHEATING	
%M6	MRESET	
%M19	M_H001	HEATING STARTED
%M20	M_H002	TT1 TOO HOT
%M21	M_H003	TT3 TOO HOT
%M22	M_H004	TT4 TOO HOT
%M24	M_H005	TURN ON PIPE HEATER
%M25	M_H006	HEATING DONE

Figure 6.4: Rung for resetting all set memories in the heating master task

All the set memories for the heating master task is reset when either the reset button is pushed, *or* the start button has been reset, *or* the heating button has been reset.

6.3.2 Memorizing Events

Memorizing events has been done in the ladder program by setting memories. Once the memory bit is set, it will remain set, no matter the value of the rung. An example of this, is with the operation of the pushbuttons. Figure 6.5 shows how the event of pushing the "start" pushbutton is memorized within the program.



Rung0 - STARTB

Comment: SET

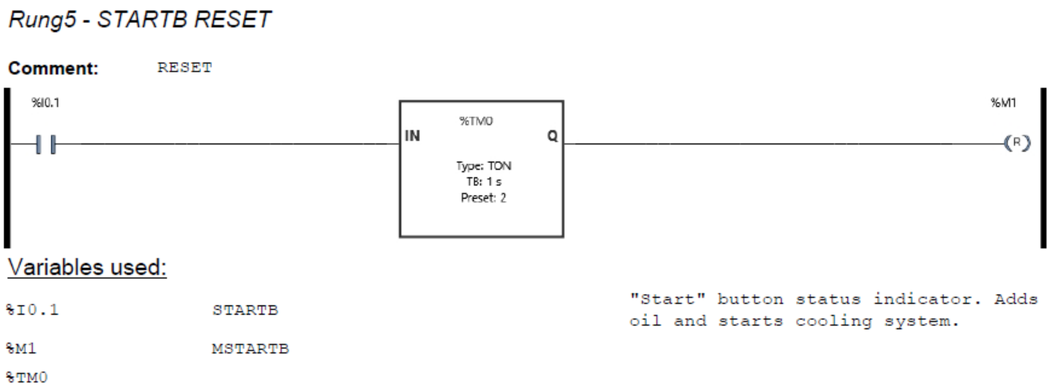
Variables used:

%I0.1	STARTB	"Start" button status indicator. Adds oil and starts cooling system.
%M1	MSTARTB	

Figure 6.5: Setting the "start" pushbutton

When using set memory bits, it is important to reset the memory bit at some point. If a memory bit is not reset, the program will only be able to run once. All memory bits connected to a pushbutton in the program are reset if:

1. The pushbutton is pushed consecutively for two seconds, as can be seen in figure 6.6.
2. The reset button is pushed.
3. The entire control program is finished.



Rung5 - STARTB RESET

Comment: RESET

Variables used:

%I0.1	STARTB	"Start" button status indicator. Adds oil and starts cooling system.
%M1	MSTARTB	
%TM0		

Figure 6.6: Resetting the "start" pushbutton

6.3.3 Comparison

Comparison blocks are used for evaluating analog input values against a numerical value. The comparison block will give a high signal if the condition within the block is true.

Rung3 - TT1 HIGH TEMPERATURE CHECK

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M20	M_H002	TT1 TOO HOT
%M57	M_H008	

Figure 6.7: Temperature check - Set

In figure 6.7 the rung can be read as if TT1 is higher than 76 °C, set memory bit 20 to high. Memory bit 20 is then used to turn off the heater. However, when the heater has been off for a time, the temperature will fall. In figure 6.8, another comparison is used to reset memory bit 20 if TT1 is lower than 73 °C.

Rung6 - TT1 LOW TEMPERATURE CHECK

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M20	M_H002	TT1 TOO HOT
%M57	M_H008	

Figure 6.8: Temperature check - Reset

This control configuration is commonly referred to as on/off control and is a simple feedback controller. The issue with this method of regulating the temperature, is that the temperature will fluctuate. Nevertheless, this method is used as the heaters are controlled by contactors, meaning that they are strictly on or off devices with the current setup.

6.4 User Interface

For the operation of the production line, the user interface that can be seen in figure 6.9 was made. There are five main processes that are associated with the buttons that can be seen in table 6.4.

Table 6.4: User Interface

Name	**Function**	
Start	Set all valves, start cooling and add oil.	 Figure 6.9: User interface for BioMax
Heating	Start heating the oil.	
Ester	Add alcohol, start mixing and circulation through PBR 2.	
Trester	Add alcohol, start mixing and circulation through PBR 1.	
Purify	Start alcohol evaporation and transfer liquid to glycerol removal.	
Running	Signifies that the program is running.	
Alarm	Signifies that there is an alarm active on the display unit.	
Reset	Resets all set memories and all pushbuttons.	
Stop	Stops all 230VAC appliances.	
Display unit	Shows process parameters and alarms.	

The stop button is not hardwired as the button has been used for other tasks during testing. A pushbutton has been acquired to replace the emergency stop button, so that the emergency stop button can be hardwired. In addition to the pushbuttons, the production line can be controlled by the TMH2GDB display unit or by a USB to the computer. These methods of controlling production line is advantageous when testing new programs or controlling single components.

6.5 Wiring Schematics

The wiring schematics are essential for setting up the PLC and are important for how the ladder program is set up. Figure 6.10 shows how the discrete inputs and outputs are connected to the PLC.

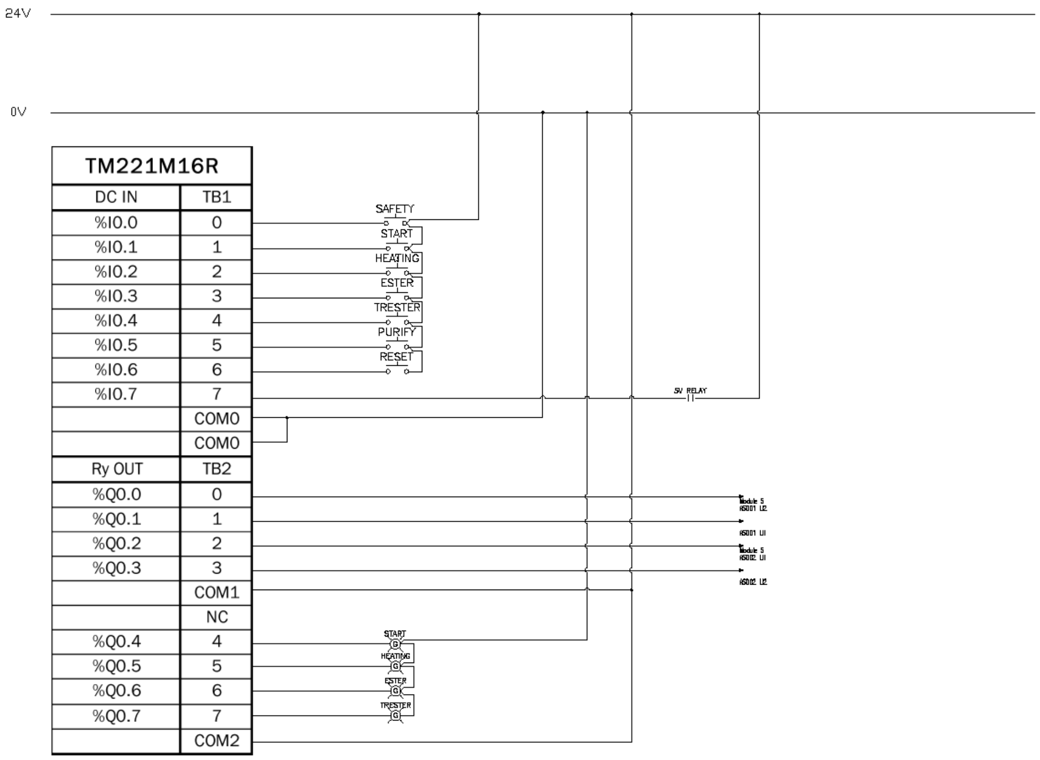


Figure 6.10: Wiring schematic for the TM221M16R. %I0.X are discrete inputs and %Q0.X are discrete outputs.

The discrete inputs are wired as sinking inputs, meaning that a positive logic is used when programming the PLC. The discrete outputs are wired as sourcing outputs, which also results in a positive logic.

The PLC has a built-in analog to digital (A/D) converter and a digital to analog (D/A) converter. The A/D converter takes an analog signal, usually voltage, current or resistance, and maps it onto the digital domain. The sensors in use on the production line are wired to transmit a 4-20 mA signal. The 4-20 mA signal is then mapped onto the sensor's rated measurement range in the A/D converter to obtain the correct measured value. The setup for the analog inputs can be seen in figure 6.11.

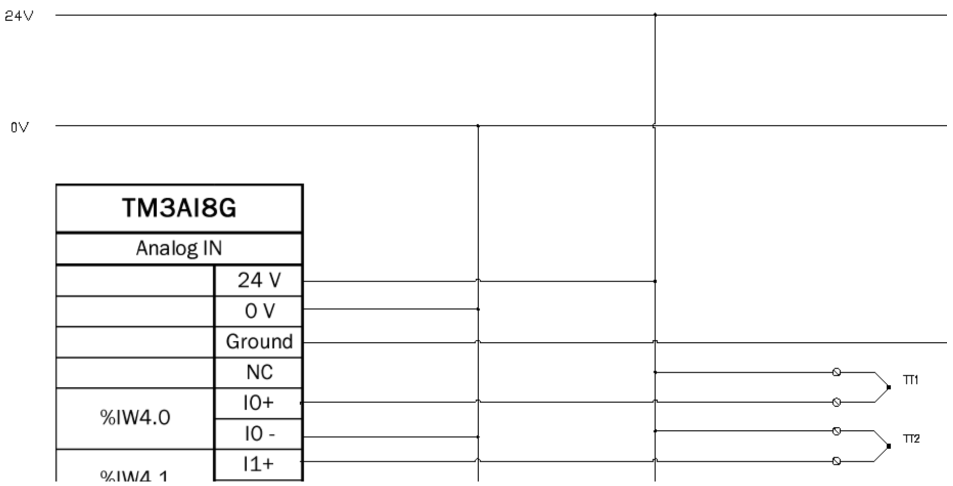


Figure 6.11: Analog input configuration

Analog outputs are used for controlling the Altivar 312 VFDs. As the VFDs can vary their frequency outputs from 0 to 50 Hz, a digital signal between 0 and 50 is written onto the digital output. This is in turn transposed by the D/A converter down to a 0-10V signal range, which the VFDs can use for setting the output frequency for the connected motors. Figure 6.12 shows the wiring for the analog outputs connected to the VFDs.

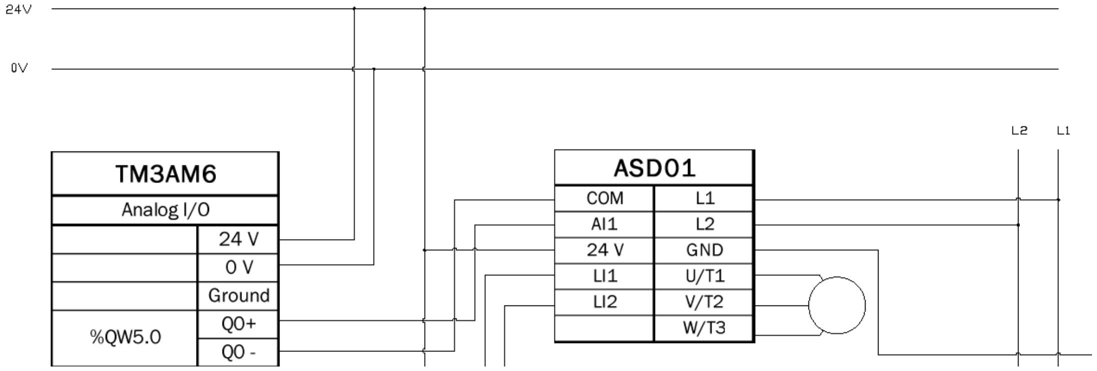


Figure 6.12: Analog output configuration

The complete wiring diagrams for the PLC and the expansion modules can be found in appendix D for further study.

6.6 System Safety

Alarms and an alarm light have been added to the control system. Once an alarm is triggered, the alarm light will turn on and a short word will be displayed on the display unit. Table 6.5 shows the “Warning” alarms in the control system.

Table 6.5: Warning Alarms

Alarm	Description
H -> AO	Heating button pushed before “start” task is finished.
E -> H	Esterification button pushed before “Heating” task is finished.
T -> H	Transesterification button pushed before “Heating” task is finished.
P -> T	Purify button pushed before “Transesterification” task is finished.
TT2>25	TT2, the TT in the water tank, shows a temperature greater than 25 °C.
LT1<50	The liquid level in the oil tank is below the minimum amount of liquid when the start button has been pushed.

"Warning" alarms are incidents that the operator should be aware of but will not result in a shutdown of the system. "Critical" alarms are alarms that results in a shutdown of some appliances or the entire production line.

Table 6.6: Critical Alarms

Alarm	Description	Action	Causes
ALCO	Alcohol is detected by MQ3 sensor after alcohol has been added.	Shutdown of all 230 VAC appliances	- Leakage of alcohol vapors - insufficient cooling of vapors
NO FLOW	The pump is on, but there is no signal coming from FT1.	Shutdown of P-001	- Pickup is not adjusted properly - Flow is obstructed by closed valves or clogged reactor filter - Pump malfunction
TT4 HOT	TT4, the TT after the inline heater, shows a temperature greater than 85 °C.	Shutdown of W-003	- Heating control disabled - Contactor malfunction
TT3 HOT	TT3, the TT between P-001 and the inline heater, shows a temperature greater than 80 °C.	Shutdown of W-003 and W-004	- Heating control disabled - Contactor malfunction
TT2 HOT	TT2, the TT in the water tank, shows a temperature greater than 35 °C.	Shutdown of W-003 and W-004	- Cooling unit overloaded.
TT1 HOT	TT1, the TT in the mixer tank, shows a temperature greater than 90 °C.	Shutdown of W-003 and W-004	- Heating control disabled - Contactor malfunction

6.7 Functional Description

A ladder program has been created from the functional description that can be seen in appendix L. The scope of this subsection is to provide a general understanding of how the real-life events that the program controls.

The ladder program is divided into POUs, which are further divided into rungs. The program associated with each of the main processes is placed within a POU, referred to as a "Master Task" within the ladder program. The program accounts for use of unrefined feedstock, as centrifugation and esterification steps has been added. The equipment tags from the P&ID drawing will be used to reference the different equipment. A program for washing the biodiesel has not been included, as the equipment will not be tested in this thesis.

6.7.1 Master Task 1 – Start

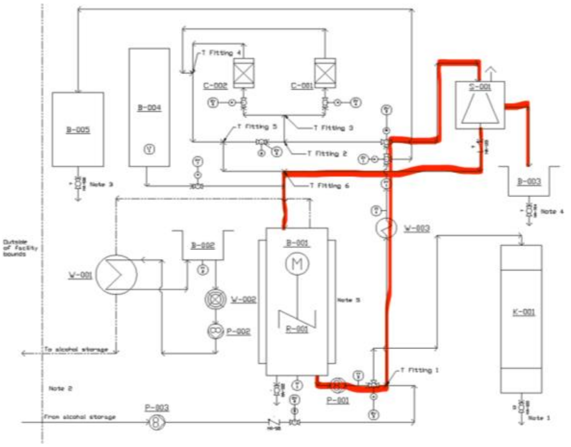
After the "Start" button has been pushed, the data logging will start, and all valves will be set into the initial position as can be seen in table 6.7. Once all valves have reached the initial position, P-002 and W-002 will start up. If there is more than 6 liters of oil in the oil tank, CV7 will open to allow oil to be added to the mixer tank. LT1 will log the initial level of the oil tank and CV7 will close once the difference between the initial level and the real-time measurement is 10 liters. The "Start" task will be finished once CV7 is in closed position.

6.7.2 Master Task 2 – Heating

After the “Heating” button has been pushed, the control system will check if:

1. Oil has been added to the system.
2. The alcohol recovery system is on.
3. The alcohol recovery system temperature is below 20 °C.
4. All valves are in the initial position (Table 6.7)

Table 6.7: Initial Position of Valves for the “Heating” Master Task

P&ID TAG	State	From	To	Process Path
CV1	Open	W-003	CV2	
CV2	Closed	CV1	S-001	
CV3	Closed	P-001	W-003	
CV4	Closed	CV2	Reactor 1	
CV5	Closed	CV2	Reactor 2	
CV6	Closed	CV2	Mixer tank	
CV7	Closed	Oil tank	Mixer tank	
CV8	Closed	Methanol pump	T-fitting 3	

If centrifugation pre-treatment is not used, the table in appendix M is used.

If all these criteria are met, W-004, the agitator, S-001 and a 30-minute TON timer will be turned on. The frequency on the agitator will be set to 8 Hz. The contents of the mixer will be transferred towards W-003 by P-001 which is set to 40 Hz. 45 seconds after P-001 has been turned on, W-003 will be turned on. The oil will then circulate between the mixer and the centrifuge. If TT1, TT3 or TT4 shows a temperature higher than 71°C, W-003 and W-004 will be turned off. If TT1, TT3 and TT4 shows a temperature lower than 69°C, W-003 and W-004 will be turned on. The “Heating” task will be finished once the 30-minute TON timer is finished.

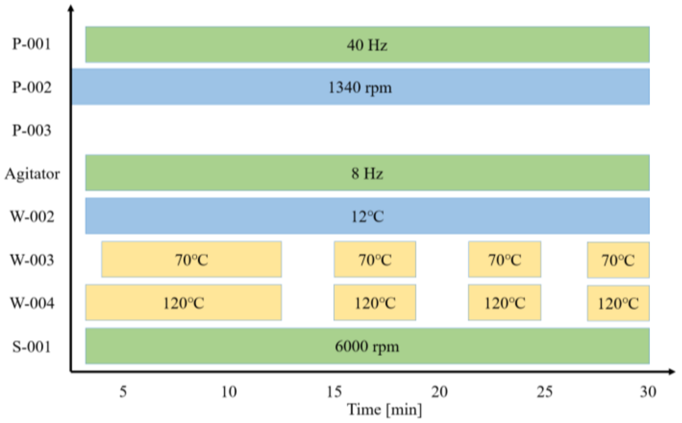


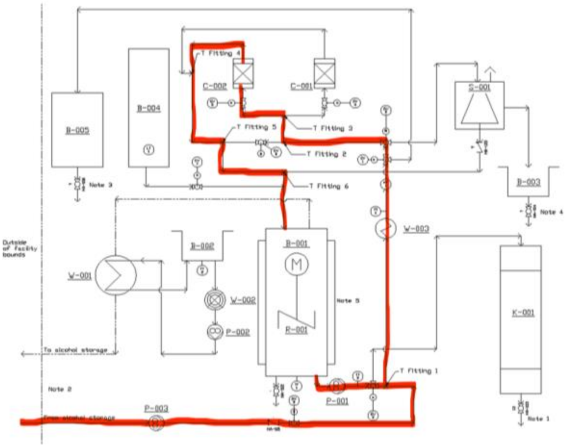
Figure 6.13: Visualization of the function of each component during the “Heating” Master Task.

6.7.3 Master Task 3 – Esterification

After “Ester” has been pushed, the control system will check if:

1. The heating is done.
2. The pipe heater is off.
3. Oil has been added.
4. The alcohol recovery system is on.
5. The alcohol recovery system temperature is below 20°C.
6. All valves are in the correct position (Table 6.8).
7. The transesterification program has not been performed.

Table 6.8: Initial Position of Valves for the “Ester” Master Task

P&ID TAG	State	From	To	Process Path
CV1	Open	W-003	CV2	
CV2	Open	CV1	T-fitting 2	
CV3	Closed	P-001	W-003	
CV4	Closed	CV2	Reactor 1	
CV5	Open	CV2	Reactor 2	
CV6	Closed	CV2	Mixer tank	
CV7	Closed	Oil tank	Mixer tank	
CV8	Open	Methanol pump	T-fitting 1	

The functional description for the “Ester” master task will be the same as the “Trester” master task. This is because esterification has not yet been attempted and optimization of parameters has not yet been performed.

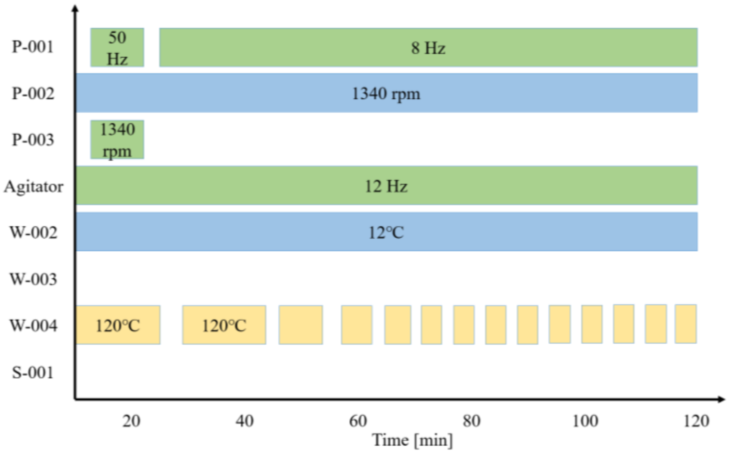


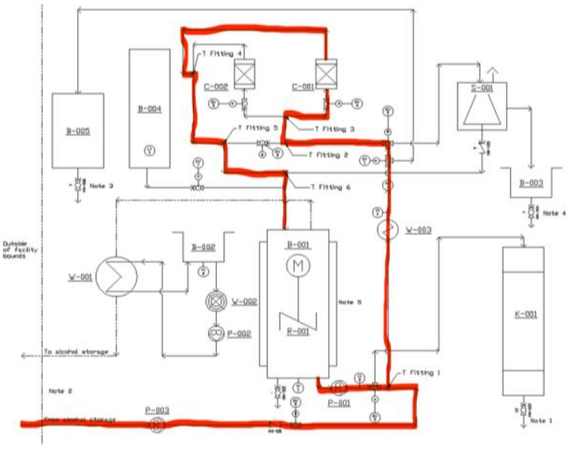
Figure 6.14: Visualization of the function of each component during the “Ester” and “Trester” Master Tasks.

6.7.4 Master Task 4 – Transesterification

After “Trester” has been pushed, the control system will check if:

1. The heating is done.
2. The pipe heater is off.
3. Oil has been added.
4. The alcohol recovery system is on.
5. The alcohol recovery system temperature is below 20 °C.
6. All valves are in the correct position (Table 6.9).

Table 6.9: Initial Position of Valves for the “Trester” Master Task

P&ID TAG	State	From	To	Process Path
CV1	Open	W-003	CV2	
CV2	Open	CV1	T-fitting 2	
CV3	Closed	P-001	W-003	
CV4	Open	CV2	Reactor 1	
CV5	Closed	CV2	Reactor 2	
CV6	Closed	CV2	Mixer tank	
CV7	Closed	Oil tank	Mixer tank	
CV8	Open	Methanol pump	T-fitting 1	

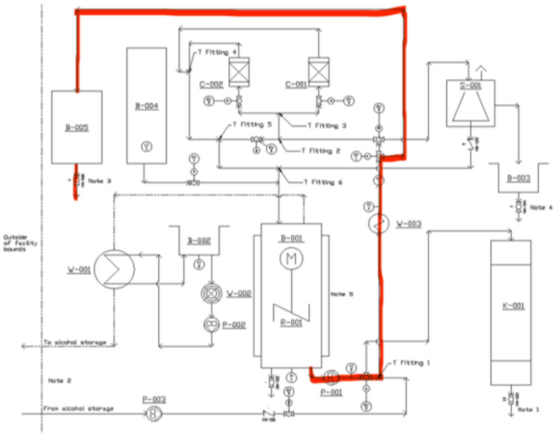
If all these criteria are met, P-001 and a 2-hour TON timer will be turned on. P-003 is turned on by the display unit and the frequency of P-001 will be turned up to 50 Hz as long as P-003 is on. P-001 will be set to 8 Hz after the alcohol pump has been turned off. W-004 and the agitator will already be on from the “Heating” task, but the frequency on the agitator will be turned up to 12 Hz. The temperature control system will keep a liquid temperature of around 70 °C throughout the transesterification process, as described in the heating master task. After the 2-hour TON timer is finished, a 60-second TOF timer will be turned on to give CV6 an “open” signal. The “Trester” task will be finished once the 60-second TOF timer is finished. The heater and the agitator will be turned off once the “Trester” task is finished.

6.7.5 Master Task 5 – Purification:

After “Purify” has been pushed, the control system will check if:

1. The transesterification program is done.
2. The alcohol recovery system is on.
3. The alcohol recovery system temperature is below 20 °C.
4. All valves are in the correct position (Table 6.10).

Table 6.10: Initial Position of Valves for the “Purify” Master Task

P&ID TAG	State	From	To	Process Path
CV1	Closed	W-003	B-005	
CV2	Open	CV1	T-fitting 2	
CV3	Closed	P-001	W-003	
CV4	Closed	CV2	Reactor 1	
CV5	Closed	CV2	Reactor 2	
CV6	Open	CV2	Mixer tank	
CV7	Closed	Oil tank	Mixer tank	
CV8	Open	Methanol pump	T-fitting 1	

If all of these criteria are met, a 30-minute TON timer and W-004 will start, and the agitator will start up with a frequency of 6 Hz. If TT1, TT3 or TT4 shows a temperature higher than 85 °C, W-003 and W-004 will be turned off. If TT1, TT3 and TT4 shows a temperature lower than 83 °C, W-003 and W-004 will be turned on. After the 30-minute timer is finished, the agitator and the heater will be turned off, and an 80-second TOF timer will turn on P-001 and assign it a frequency of 50 Hz. The purification task is finished once the 80-seconds TOF timer is finished.

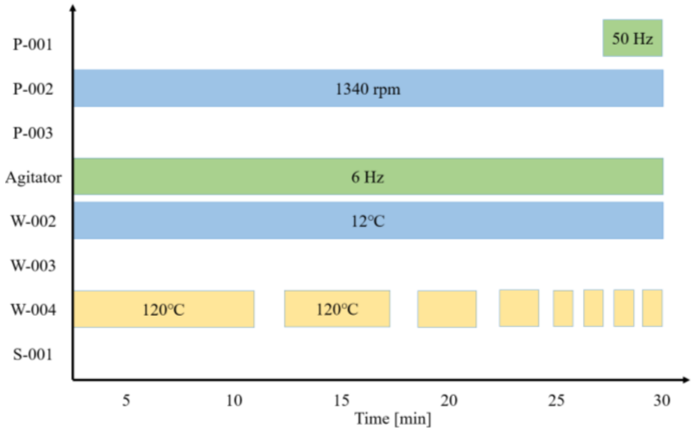
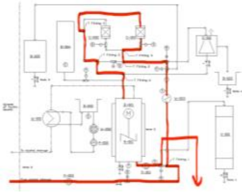
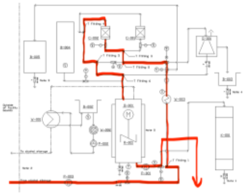
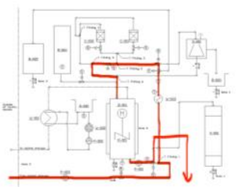
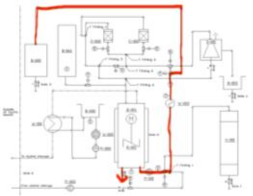


Figure 6.15: Visualization of the function of each component during the “Purify” Master Task.

6.7.6 End of the Process Control Program

The process control program is finished once the liquid has been transferred to the glass storage container. An additional program was made for flushing the piping system with alcohol or air. The program can be operated from the display unit.

Table 6.11: Flushing Pattern

P&ID TAG	Flush 1	Flush 2	Flush 3	Flush 4
Path				
CV1	Open	Open	Open	Closed
CV2	Open	Open	Open	Open
CV3	Closed	Closed	Closed	Closed
CV4	Open	Closed	Closed	Closed
CV5	Closed	Open	Closed	Closed
CV6	Closed	Closed	Open	Open
CV7	Closed	Closed	Closed	Closed
CV8	Open	Open	Open	Open

During flush 1, 2 and 3, pressurized air is used in the inlet of CV8 and CV3 is used as the outlet. Each flush sets the valves and starts a 60-second TON timer. During flush 4, pressurized air is added through the top of the glass storage vessel.

7 Testing the biodiesel production line

The testing of the production line will be done in three steps:

- **(1)** Individual testing of components
- **(2)** Verification of the control system by water testing
- **(3)** Testing of the production line by transesterification of refined sunflower oil, rectified ethanol and calcium oxide.

Due to difficulties encountered in the first test with alcohol and feedstock, a second test of the production line was carried out.

7.1 Testing of Components

Before the production line was assembled, every component was tested at a testbench. The results from the tests are presented below.

The Flow Transmitter

It is difficult to get a reading with water at low flows. Testing with water gave results, but only briefly when the pickup was positioned precisely in the transmitter housing (figure 7.7).

The Temperature Transmitters

A deviance of -2°C was found when TT4 was tested against a handheld thermometer. There were several sources of error, as the measurement by the handheld thermometer was performed downstream from TT4. Furthermore, TT4 is exposed to conduction from the inline heater.

The Level Transmitter

LT1 showed accurate level readings when tested with water and a ruler. The density of oil must be known beforehand when tested with oil.

The Alcohol Sensor

The sensor reacts to alcohol levels much lower than the LEL of ethanol [33, 86]. The sensor was triggered when positioned 20cm away from an open alcohol container and it took around five minutes for the sensor to reset after alcohol had been sensed. A calibration for the odor threshold of ethanol was performed. It has not been tested with methanol.

Pushbuttons and LEDs

The five pushbuttons from “Biltema” emits a weaker light than the pushbutton from Schneider Electric, as the pushbuttons from “Biltema” are rated for a 230 VAC power supply. No light was observed from the LEDs. The wiring was checked, but no faults were uncovered.

Control Valves

The valve position of the three-way valves was tested before they were placed on the production line and physically marked with a “T” that indicates the position of the valve. All valves have been adjusted so that the input lobe hits the switch before the actuator lobe (appendix F). The valves rotate slowly, at approximately 7.5° per second.

VFD Controlled Gear Pump

The pump flow vs. VFD frequency is mostly linear (Figure 7.8). The Pump emits a “squeaking” sound when running. The nozzles are 3/8“, as 3/8” to 1/2" reducers cannot be mounted due to the bypass valve getting in the way.

Fixed Speed Gear Pump

Pump flow was measured to be higher than 3 l/min when testing with water (Figure 7.7). There are significant vibrations coming from the pumps compared to the VFD controlled pump. Emits a loud “humming” sound.

Agitator

Emits a “clanking” sound, which is likely due to the motor being slightly tilted compared to the bushing hole. The torque is high even at low frequencies. The VFD cannot be run in “forward”. This can be fixed by reprogramming the VFD.

Inline Heater

The temperature can be varied by a thermostat, which is fairly precise. Convergence to the setpoint is reached within a few minutes. The heating element will overheat or short circuit if liquid is not present in the pipe when turned on.

Jacket Heater

The jacket heater has a high heat loss as it has not been insulated. The temperature must therefore be set to at least 40 degrees above the setpoint, if effective heating is to be obtained.

Centrifuge

The centrifuge is efficient in cleaning oil when provided with the right flow at higher temperatures. The centrifuge sump is approximately 0.7 liters.

Cooling Unit

The cooling unit is under-dimensioned for the intended use. Heat removal from the water is extremely slow.

7.2 Verification of the Control System

After testing the components individually, the production line was assembled. To validate that the reaction mixture followed the intended path for each program, the mixer was removed and replaced with a transparent plastic box. The reactors were packed with dry peas to imitate the pressure drop from the catalyst. The mixer was tested separately.

7.2.1 Adding “Oil”

The “add oil” program was tested by filling between 12 and 15 liters of water into the tank. Before the test, it was calculated that a pressure of 325 mmH_2O corresponded to 10 liters of water for the oil tank (appendix B).

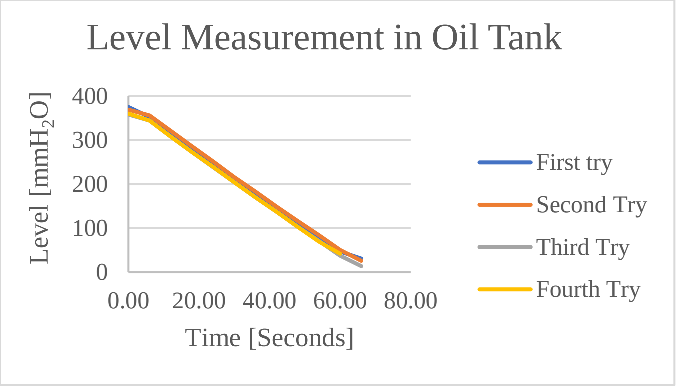


Figure 7.1: Level in the oil tank during the four tests of the "add oil" program.

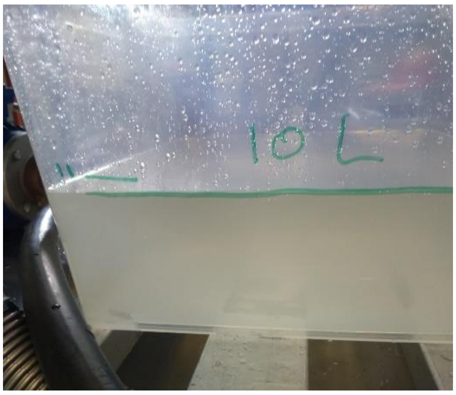


Figure 7.2: Result after adjusting the add "oil" program.

The level calculation in the PLC uses the initial level of liquid in the tank and subtracts the real-time measurement. The difference can then be looked upon as the added volume. However, the slow rotation of CV7 had to be accounted for. The difference was recalculated to 290 mmH_2O, which yielded the result that can be seen in figure 7.2. Figure 7.1 shows that the slope of mmH_2O over time through the oil tank is linear and nearly constant.

7.2.2 The Alcohol Recovery System

Before the test, 4 liters of water at room temperature (approximately 18℃) was added to the water tank. The water cooling was found to be slow, at around 0.5℃/min, as can be seen in figure 7.3.

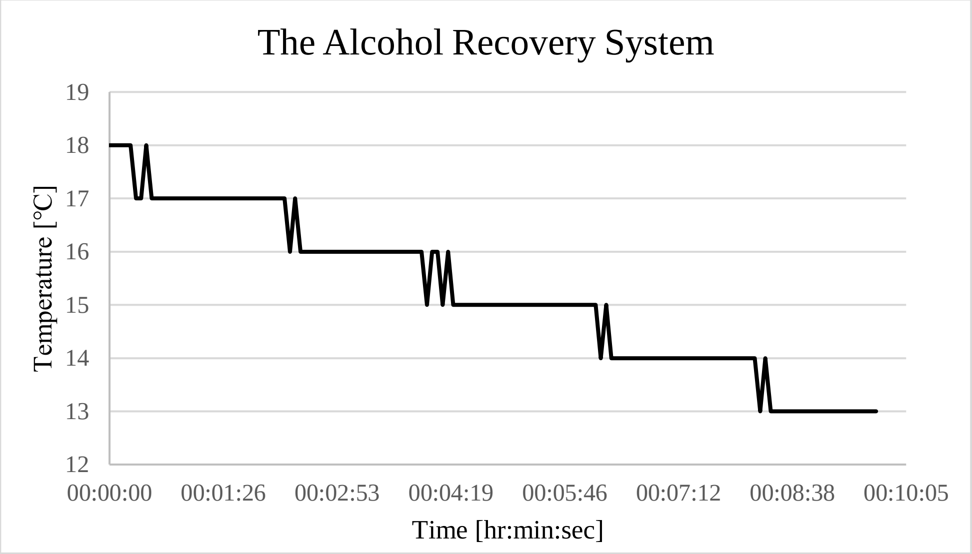


Figure 7.3: The alcohol recovery system curve

7.2.3 The Heating System

The inline heater was tested by pumping water at 25 ℃ through it. The thermostat setting on the inline heater was set to 60 ℃. It was discovered that some of the water vaporized within the inline heater, which led to pressure within the hoses. The outlet temperature of the inline heater can be seen in figure 7.4.

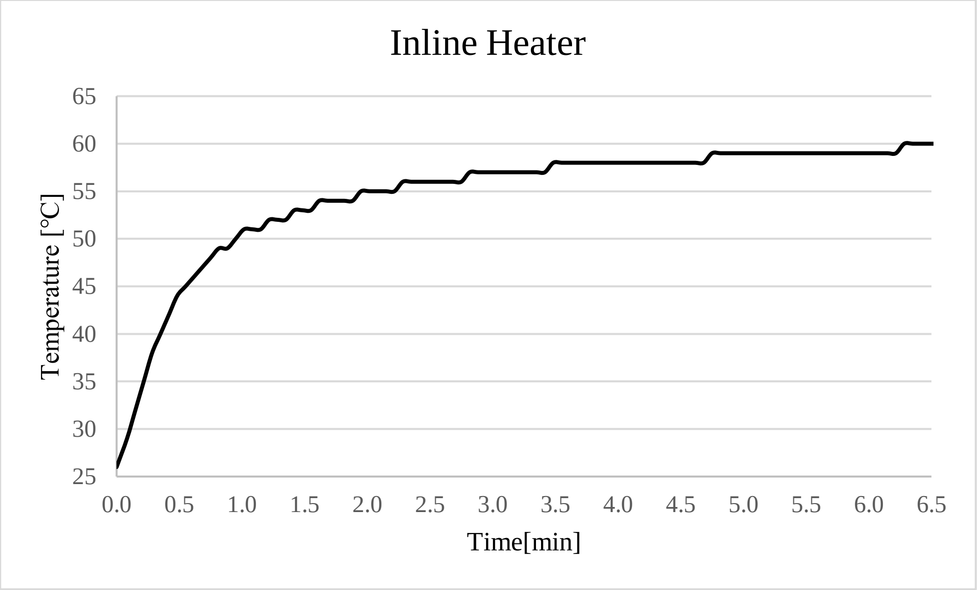


Figure 7.4: Inline heater curve

The jacket heater was tested after reinstallation of the mixer, where the mixer was filled with 10 liters of water at approximately 20 °C. The thermostat on the jacket heater was set to 120 °C and the agitator was set to 8 Hz. The heating curve of the jacket heater can be seen in figure 7.5.

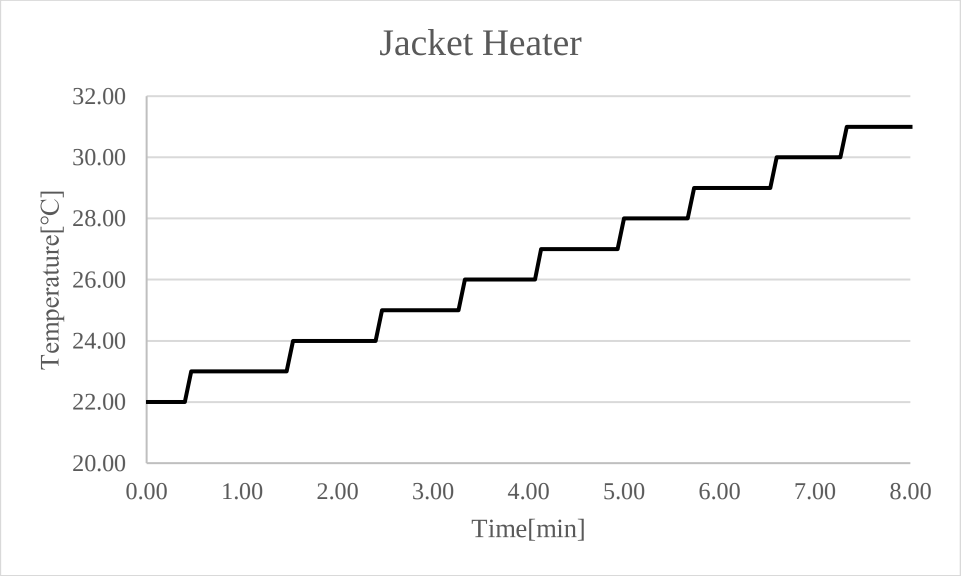


Figure 7.5: Temperature curve for the Jacket Heater (TT1)

The heating rate for the water test was approximately 1.2 °C per minute. However, as the LMTD gets smaller, the heat exchange between the mixer wall and the liquid will be slower.

7.2.4 The Reactor System

Figure 7.6: The reactor packed with peas

The reactors were packed with dry peas with a diameter of approximately one centimeter to test the proficiency of the pumps. The reactors were packed to the brink as can be seen in figure 7.6. After several tests, the peas had started swelling, and thereby increasing the required pump pressure. No difference in operation was observed because of this, as liquid was still coming out of the discharge line at the same rate.

7.2.5 The Mass Transfer System

The pump system proved proficient for the water testing. However, there were difficulties with getting the flow transmitter to work. No signals were measured at a 4-20mA or a 0-10V signal configuration. It was discovered that the electromagnetic pick-up must be precisely positioned at the right height and rotation compared to the gears within the transmitter housing. The signal range within the PLC was set between 0.5 to 50 l/min. The measurement results can be seen in figure 7.7.

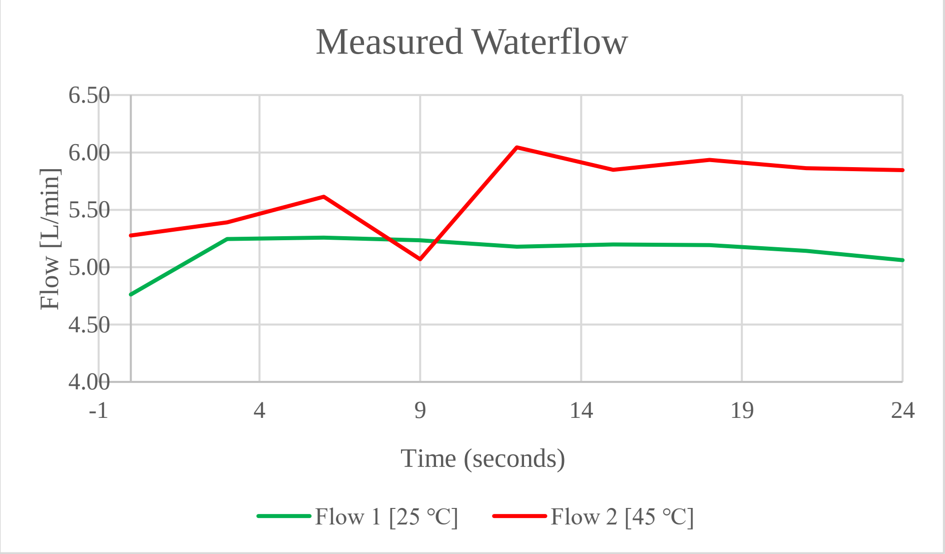


Figure 7.7: Flow Transmitter Curve for Water

The transmitter only showed results when the frequency of the pump was set to 50 Hz. The results in figure 7.7 are conflicting with the rated pump flow, which is around 3 l/min. The flow transmitter is rated to work within a viscosity range of 1-2500 cSt [89]. Water at 25 °C has a dynamic viscosity of 0.89 cSt [99], which might have impacted the measurement.

The relationship between the pump flow and the frequency on the VFD was examined by measurement. The measurement in figure 7.8 was performed by turning on the pump and filling up a measuring glass while measuring the time. The liquid was rapeseed oil at 20 °C.

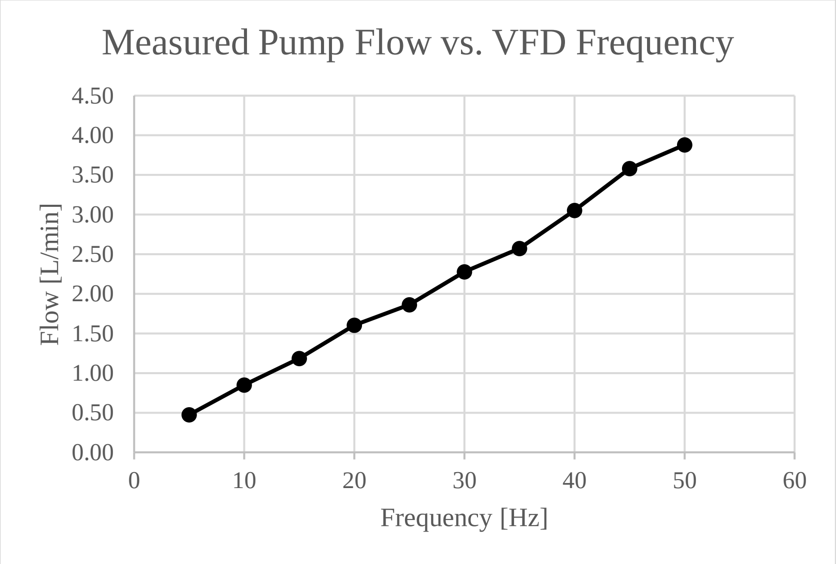


Figure 7.8: Measured Volumetric Pump Flow

The measurement method was not precise but figure 7.8 gives a general picture of the flow provided by the pump. Although the rated pump flow is 3 L/min, it seems that flows up to 4 L/min can be obtained.

7.3 First Biodiesel Production Test on the Production Line

Prior to testing with alcohol and oil, a job safety analysis (JSA) was performed to ensure that safety was maintained throughout the testing. Since refined vegetable oil was used, the pretreatment and esterification steps were removed from the ladder program. Furthermore, the "add oil" program was removed, and the ethanol was added though the oil tank.

7.3.1 Job Safety Analysis

The JSA in table 7.1 was used for determining the potential hazards associated with testing of the production line.

Table 7.1: Job Safety Analysis

Hazard	Causes	Action	Preventive measures
Fire	Electrical equipment failure	Disconnect power supply and put out with fire extinguisher.	- Low process temperatures - Outdoors operation - External alcohol storage - Correct wire sizing
Vapor Explosion	Ignition of alcohol vapors	Keep safe distance and disconnect power supply.	- Alcohol detection sensor - Outdoors operation - Visual check of electrical connections before test - Keeping a safe distance during operation - No open fires allowed near the production line - Visual check of piping and flanged connections before test - External alcohol storage

Toxicity	Exposure to alcohol vapors	Call 113 (Norwegian ambulance) and disconnect power supply.	- Alcohol detection sensor - Outdoors operation - Outdoors operation - Visual check of piping and flanged connections before test - Use of 3M fully covering mask for volatile organic compounds when near the production line - External alcohol storage - Stay upwind from the production line
Electrical Shock (230 VAC)	Exposed wiring or faulty connection	Call 113 (Norwegian ambulance) and disconnect power supply.	- Grounding to frame - Check grounding before test - Check wires before test - Check wire connections before test
Pipe Rupture	High differential pressure due to clogging of reactor filter mesh	Keep safe distance and disconnect power supply.	- Maximum pump pressure (10 Bar [71]) lower than rating of piping system. - Visual check of piping and flanged connections before test - Keep safe distance - Built-in bypass valve on pumps set to 3.5 Bar.

There are several safety considerations that has been made during the design of the production line. The most dangerous aspect of the production line is the use of alcohol, which is highly flammable. The alcohol tank is kept at least 2 meters away during operation to limit the effects in case of fire. The alcohol vapor is led through a 3-meter hose and transferred away from the production line.

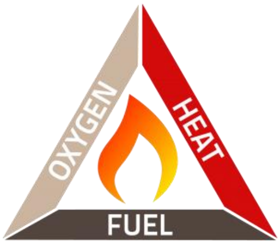


Figure 7.9: The fire triangle [100]

It was decided to use ethanol during the testing of the production line to eliminate the risk of methanol poisoning. Ethanol is also toxic, but the effects are milder compared to methanol. A 3M 6900 full face mask with 6098 filters, for organic vapors with a boiling point lower than 65 °C [101], has been acquired. The mask can be seen in figure 7.10 and allows for future testing of the production line with methanol.

Figure 7.10: 6900 full face mask from 3M [99]

7.3.2 Preparation

A custom-made catalyst was made by covering lightweight expanded clay aggregate (LECA) beads with calcium carbonate powder of high purity. The LECA beads and the calcium carbonate was mixed is a bowl, as can be seen in figure 7.13. For calcium carbonate to be converted to calcium oxide, high temperatures are required. The calcination process is typically done at temperatures between 850 °C and 1340 °C. The chemical reaction equation for calcination of calcium carbonate can be seen in equation 5 [102].

$$CaCO_3 \rightarrow CaO + CO_2 \qquad \text{Eq. 5}$$

The LECA catalyst was calcinated at 950℃ for 8 hours (figure 7.12). After the calcination, the catalyst was cooled down to 200℃ in the furnace, and subsequently placed into aluminum foil for transport to the production line. The PBR was packed to the brink, as can be seen in figure 7.11.

Figure 7.13: Mixing of water, calcium carbonate and LECA balls

Figure 7.12: Catalyst after heating

Figure 7.11: Catalyst placed in the reactor

An amount of 8 kg of oil was weighed and added to a container, as it can be seen from figure 7.14. 2.5 kg of ethanol of 99% purity was weighed and added to a separate container, giving an ethanol-to-oil ratio of 6:1 (appendix K).

7.3.3 Process Parameters

The parameters used during the first test can be seen in table 7.2.

Table 7.2: Process Parameters for Ethanol Test

Parameters	Heating	Transesterification	Ethanol Evaporation
Temperature Setpoint [℃]	70	70	85
Pump Flow [l/min]	1.80	0.70	-
Mixing Speed [rpm]	220	330	160
Duration [min]	30	120	30

The temperature for the transesterification process was set to 70 °C, due to ethanol having a boiling point of 78.4 °C [31]. A reaction temperature close to the alcohols setpoint seems to be the preferred method for most biodiesel research. A relatively long reaction time was chosen to ensure that a reaction would happen. The flow through the packed bed reactor was set low so that a slow flow through the reactor could be obtained. Due to concerns regarding the mechanical robustness of the mixer, the mixing speed was reduced by half from what the literature study recommended [51]. The heating and evaporation times were set to 30 minutes due to the performance of the heating equipment during the water test.

Figure 7.14: 8 kg of sunflower oil for the test

7.3.4 Experimental Procedure

An energy measuring device was connected to the power inlet of the production line and used to measure the energy consumption of the developed production line. The production line was powered up and the custom test program was downloaded to the PLC. An SD card was inserted into the PLC for data logging.

To start the process, the oil was added to the system through the oil tank. After this, the heating system, the alcohol recovery system and the agitator were turned on. The jacket heater was set to 120℃ and the inline heater was set to 70℃. After 30 minutes the oil had reached a temperature of 70 °C, and the alcohol was added through the oil tank. The empty alcohol container was placed approximately 2 meters away from the production line as can be seen in figure 7.15.

Figure 7.15: Placement of the alcohol receptacle during testing.

The process pump was turned on and the reaction mixture was transferred to PBR 1. A test was taken after the liquid had circulated for 30 minutes. Alcohol was re-added two times, after 1 hour and 1 hour and 30 minutes, due to the large amount that evaporated during the transesterification process. The water in the water tank was replaced two times with colder water, due to safety concerns. After the 2-hour timer was finished, the reaction mixture was transferred to the glass container in figure 7.16.

7.3.5 Results

The product after the transesterification process was a slightly darker liquid as can be seen in figure 7.16. No glycerol was observed in the bottom of the liquid after it had been allowed to settle. In addition to this, the color was darker than the oil in figure 7.14. The total energy consumption for the production was 4.31 kWh. This section will go through the performance of each system on the production line.

Figure 7.16: Product after the first test

The Alcohol Recovery System

The ambient temperature was 7 °C when the testing started, which is to say that the alcohol recovery system was performing under relatively good conditions. However, when the ethanol started to evaporate, and the system came under load, the temperature in the water increased rapidly. The log data from the temperature trans-mitter at the bottom of the water tank can be seen in figure 7.17.

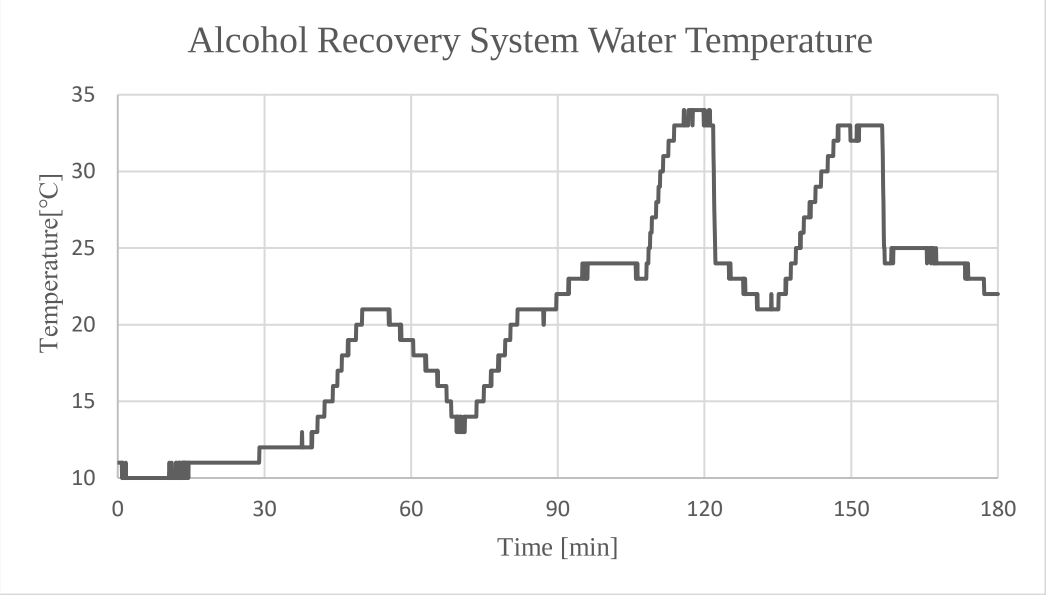


Figure 7.17: Alcohol recovery system temperature curve

After 30 minutes ethanol was added, and soon after the ethanol had started to evaporate. The water was replaced after 50, 115 minutes and 160 minutes with colder water. The testing showed that the cooling capacity of the cooling unit was too low to continuously cool the water. Nevertheless, the alcohol was condensed during the test. Removal of the ethanol after the transesterification process worked well, as little to no ethanol was observed in the final product.

The Heating System

The heating system performed well during the initial heating of the oil. However, during the transesterification program the temperature in the tubes started to decrease.

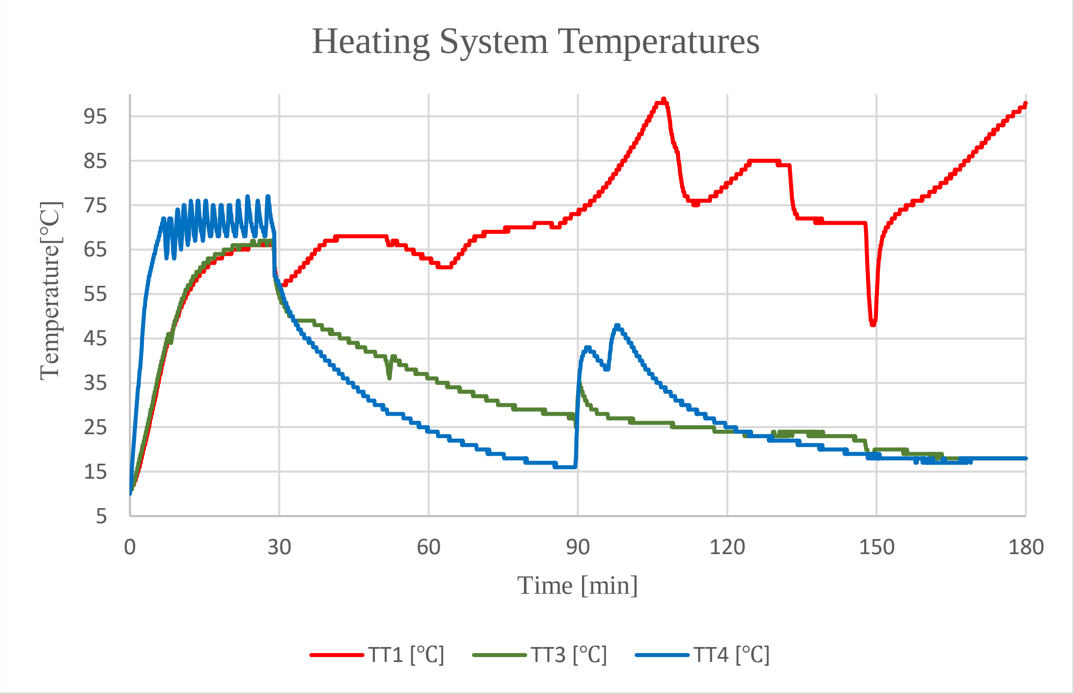


Figure 7.18: Heating system curves

Added to this, was the fact that a signal could not be obtained from the flow transmitter. As there had been previous issues with the flow transmitter, it was assumed that it was malfunctioning.

Once the circulation through the PBR started, the temperature reading from TT3 and TT4 started to decrease. When TT3 reached 30℃, the control program was overridden to counteract the decreasing temperature. The inline heater was turned on at 40℃ after one hour, to raise the temperature. However, shortly after turning on the inline heater, a far more likely scenario was deduced; the reactor filter had been clogged by the catalyst. This theory was corroborated when opening the reactor after the test was finished as can be seen in figure 7.19. It is likely that some of the LECA beads were crushed upon sealing the reactor, as the reactor was packed to the brink. Because of this, the circulation through the PBR was unsuccessful, which explains the lack of glycerol in the product.

Figure 7.19: The reactor after the first test

Mass Transfer System

The mass transfer system worked well considering that the test was unsuccessful. During the transesterification program, it is likely that the reaction mixture was circulated through the built-in bypass valve on the pump. The pump was checked several times to see if it was running, and no significant heat was felt coming off it. No signal could be obtained from the flow transmitter, most likely because of the reactor being clogged.

The Mixing System

During the test, samples were drawn from the bottom of the mixer every 30 minutes to check the mixing characteristics of the agitator. All samples appeared to be in one phase and smelled strongly of alcohol. Approximately five minutes after the samples were taken, a thin layer of alcohol could be observed on top of the oil.

The Control System

Some of the automated features and capabilities of the ladder program were disabled before or during the test. The disabled features before testing were those concerning either pretreatment of feedstock or the addition of alcohol and feedstock. During the test, the control system stopped the heating as soon as TT2 exceeded 20°C. This feature was disabled to continue testing, as the ethanol was still condensing. Furthermore, as it was assumed that the heating system was not working properly, it was also disabled. This resulted in the high temperatures for TT1 in figure 7.18.

7.4 Second Biodiesel Production Test on the Production Line

It was decided to perform a second test of "BioMax" under better circumstances. 800 grams of calcium oxide rocks were acquired for the test, as this catalyst yielded relatively good results during the preliminary test. The calcium oxide rocks were calcinated at 950℃ for 8 hours and subsequently crushed into smaller pieces. The catalyst was added to the PBR together with medium sized pieces of plastic as packing material. The same JSA procedure and parameters were used for the second test except for the transesterification temperature being set to 65 ℃. This was done as the first test proved the cooling system to be insufficient.

7.4.1 Experimental Procedure

The experimental procedure for the test was similar to the first test. However, the temperature was lowered as the alcohol recovery system proved insufficient in handling the high rate of ethanol evaporation during the first test. No alcohol was observed coming out of the heat exchanger during the transesterification program. Once the evaporation process started, and ethanol was observed, the alcohol sensor was used to determine whether there was any leakage of ethanol vapors.

Figure 7.20: Reaction mixture flowing through the transparent tube during the second test.

A transparent tube was placed in the discharge line between PBR 1 and the mixer during the test to monitor the liquid coming out of PBR 1. This was done as it was unsure if the flow transmitter would operate as intended and to prevent a repeat of the first test. It should be noted that this is a temporary measure, as the PVC pipe is not well suited for use with the temperatures and liquids at the production line. Another difference for this test was that the alcohol was added by the alcohol pump.

After the test, approximately half of the reaction mixture was added to the glass container, allowed to settle for five minutes and drained into a plastic container. The remaining reaction mixture was drained into another plastic container and re-added through the oil tank. The reaction mixture was circulated between the mixer tank and the centrifuge for 8 minutes with a VFD frequency of 8 Hz on the main pump.

7.4.2 Results

The product after the transesterification process can be seen in figure 7.21. Approximately two hours after the liquid had been added to the container, the appearance of the reaction mixture in figure 7.21 went from transparent to misty. After one week, the ethanol had settled out in a thin layer at the top. The reaction mixture that was added to the centrifuge did not change appearance and had less ethanol in the top layer.

The reaction mixture was less viscous liquid than the initial oil feedstock, when it was cooled to the ambient temperature. A small amount of glycerol was observed in the bottom of the reaction mixture after it had been allowed to settle. The total energy consumption for the production process was 4.36 kWh. Some notable problems occurred during the test, as is evident from the charts below. The heating program had to be restarted two times as a result of faults in the PLC program.

Figure 7.21: The product after the second test

The Alcohol Recovery System

Due to a lower process temperature being used, no ethanol was observed evaporating during the transesterification program, which is evident by comparing figure 7.17 and 7.22.

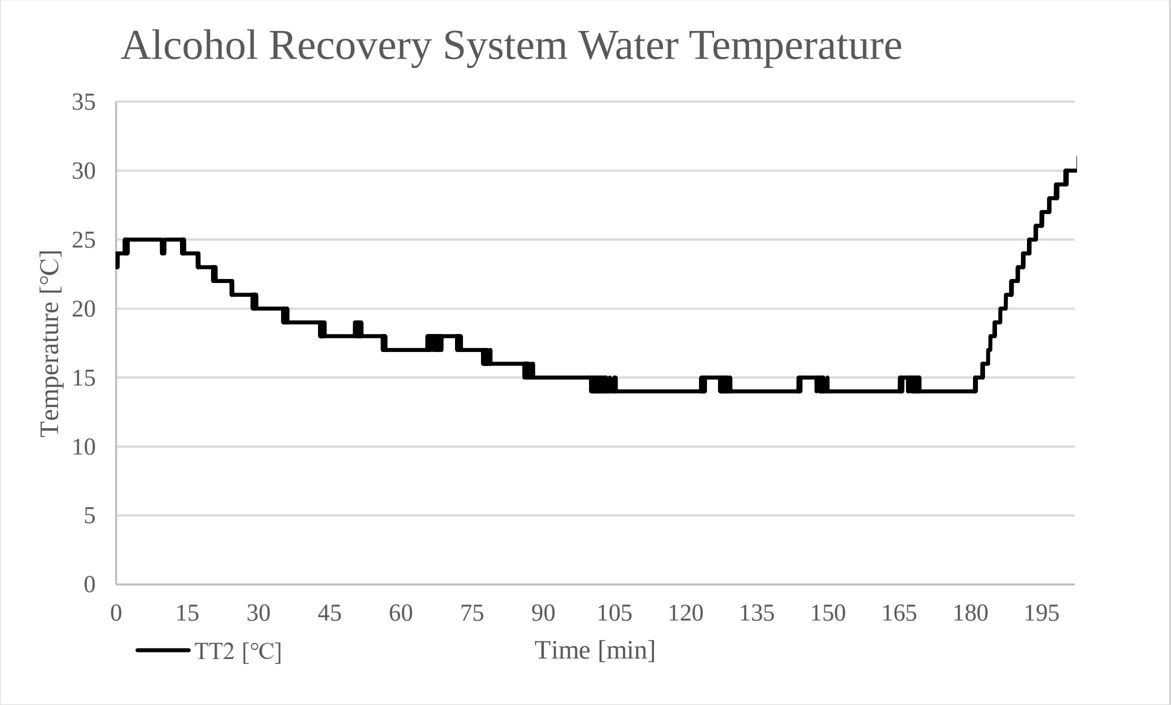


Figure 7.22: The alcohol recovery system curve during the second test

The alcohol sensor was used to check the production line for ethanol vapor leaks during the purification program. The sensor was placed at several spots on the production line, but there was no indication of any significant ethanol levels.

The Heating System

The frequency on the VFD of the main pump during the heating program was set to 8 Hz, due to a programming error. This pump flow proved to be too low, as a “sizzling” sound could be heard coming from the inline heater. The heating program was restarted and the VFD was set to 40 Hz. After the heating program, the temperature setting in the control program was discovered to be 60°C instead of 65°C. Because of these technical difficulties, the data will be presented separately for the heating, transesterification and the purification program. The complete chart can be viewed in appendix N.

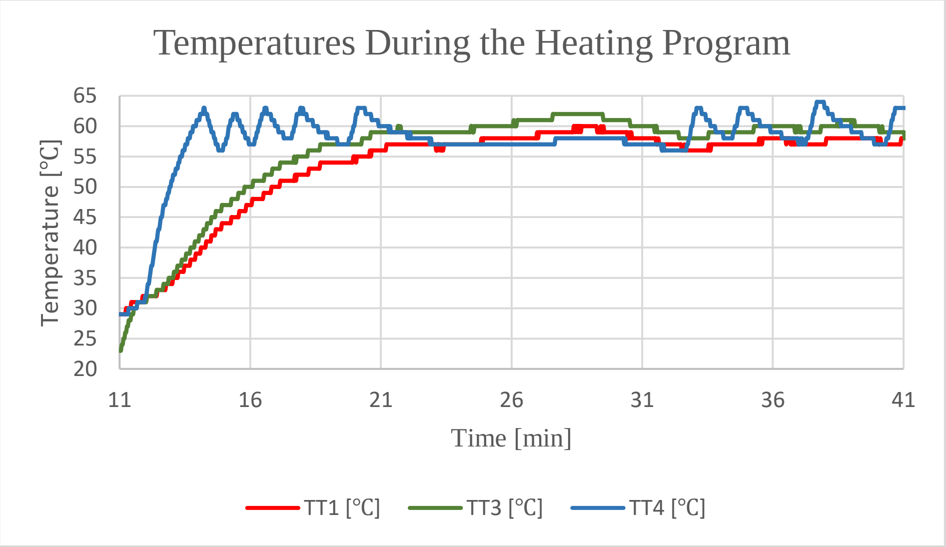


Figure 7.23: Heating Program Temperatures

The heating program worked well, as the equilibrium at 60°C was reached quickly, compared to the first test as can be seen by comparing figure 7.18 and figure 7.23. Figure 7.24 shows the heating during the transesterification program.

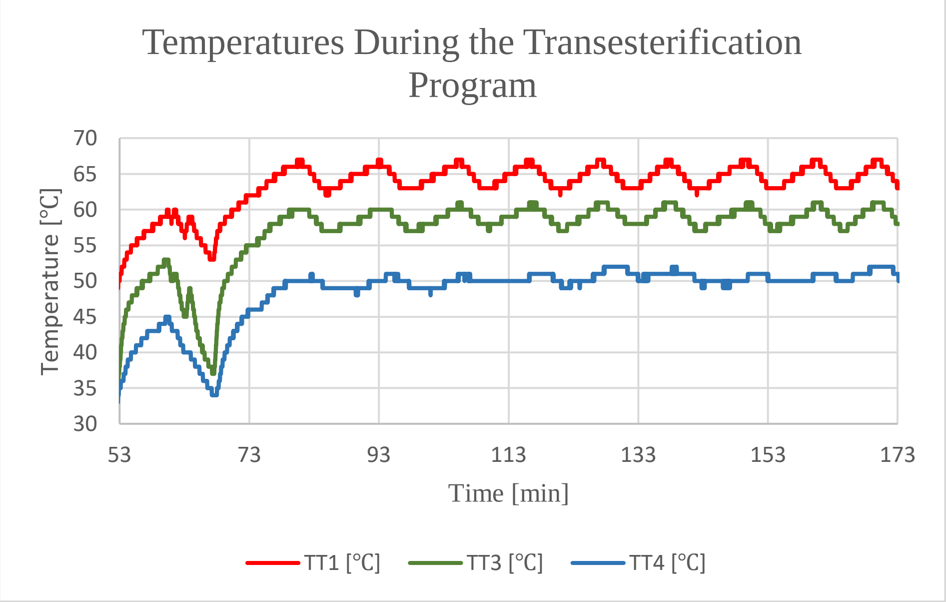


Figure 7.24: Transesterification Program Temperatures

The drop in temperature between 60 and 65 minutes was due to the transparent tube being installed. Figure 7.25 shows the temperatures during the purification program.

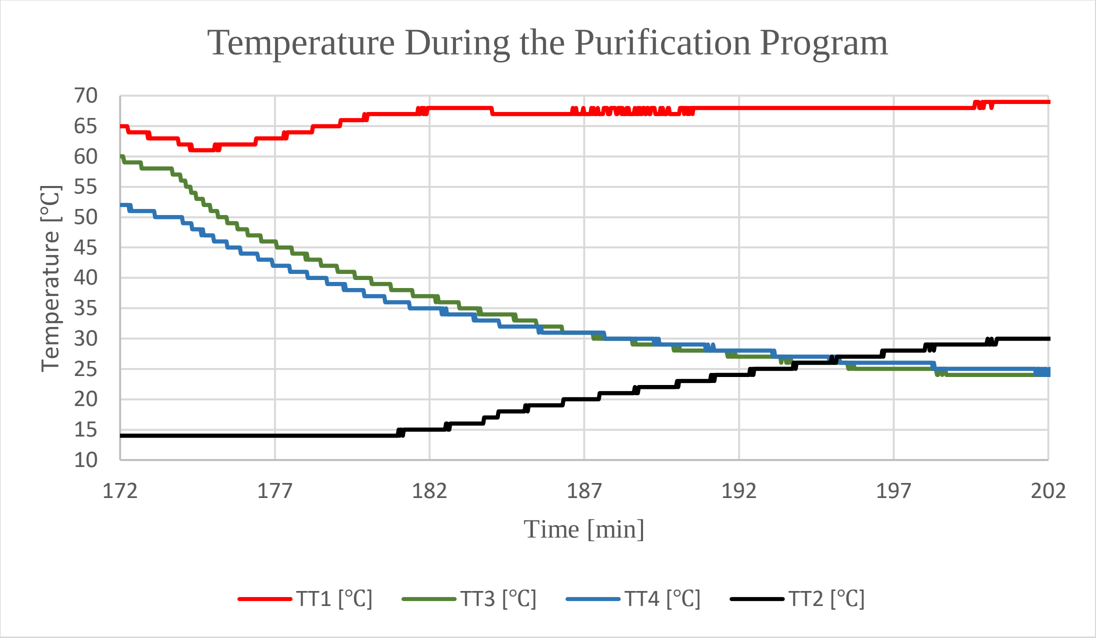


Figure 7.25: Purification Program Temperatures

Mass Transfer System

The flow transmitter was adjusted after the first test and the results can be seen in figure 7.26.

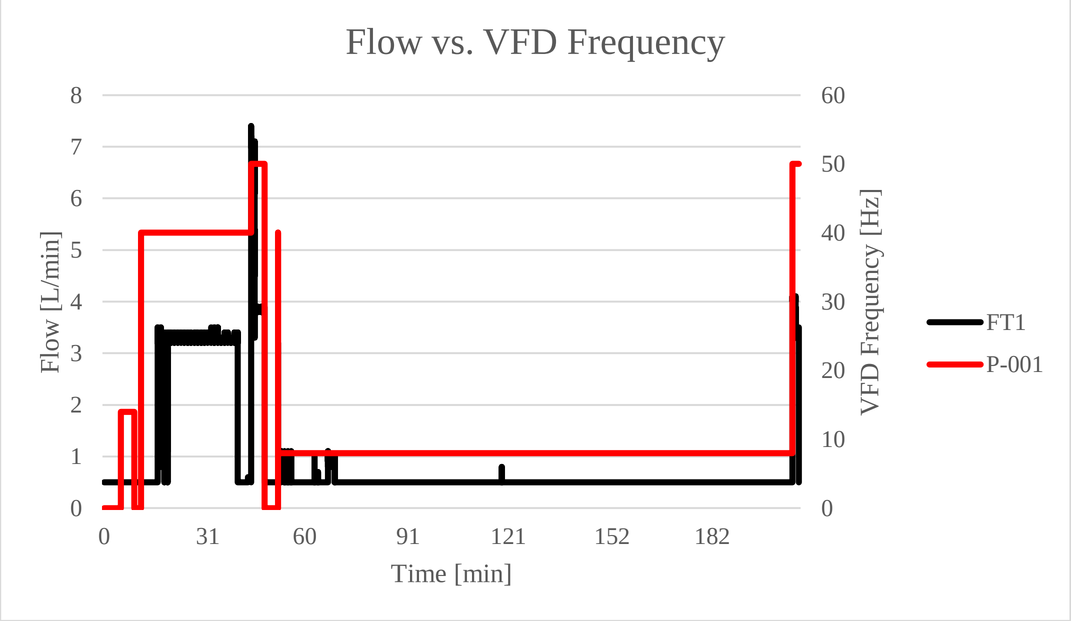


Figure 7.26: Flow vs. VFD frequency

The flow when the VFD was set to 40 Hz showed 3.3 L/min. This corresponds well with the measured flows in figure 7.8, considering the temperature difference. However, a signal was only registered at high pump flows, as the flow transmitter barely gave a signal at 8 Hz during the transesterification program. The flow was increased during the pump mixing of ethanol and oil before the transesterification process, as can be seen in figure 7.27.

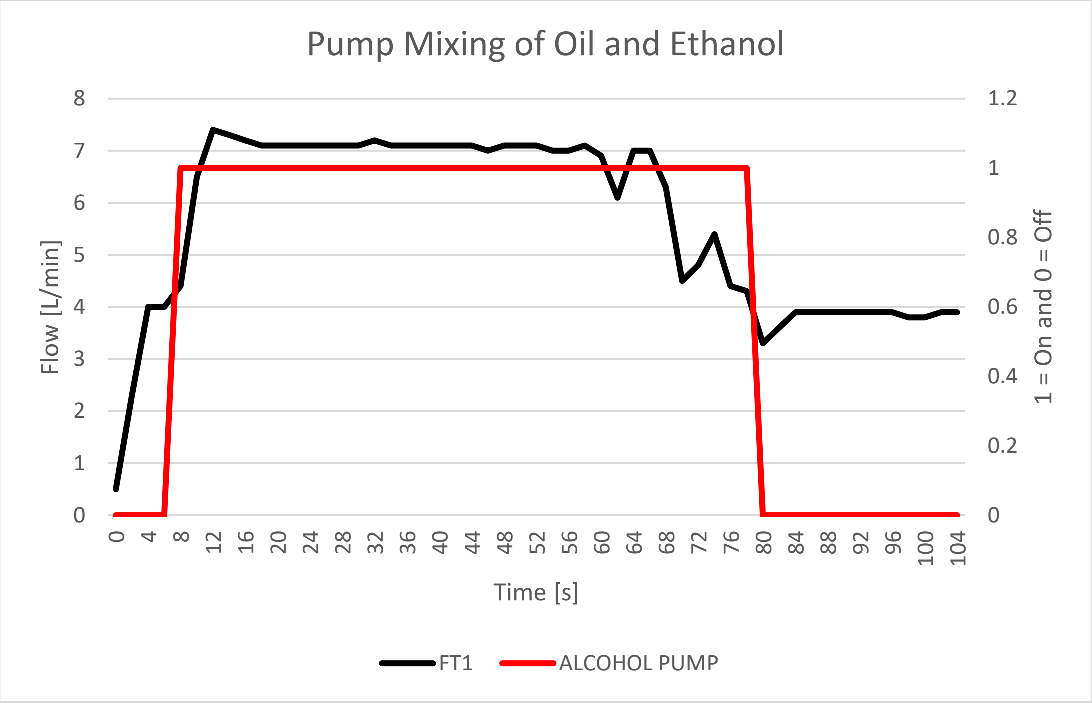


Figure 7.27: Pump mixing of oil and alcohol during the second test

The reason for the fluctuating measurement before the alcohol pump was turned off, was that the alcohol container was empty. As previously mentioned, the reaction mixture was less viscous than the oil feedstock. Figure 7.28 shows the level measurements in the oil tank during the second test with data from the water test for reference.

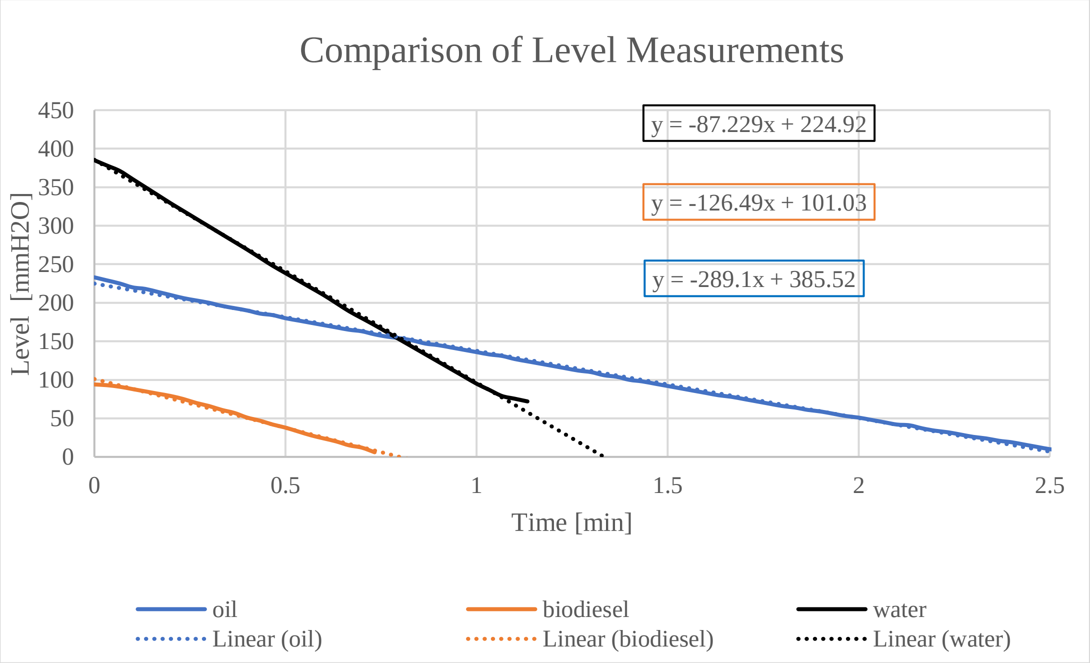


Figure 7.28: Flow characteristics of water, the oil feedstock and the reaction mixture upon addition through the oil tank.

The slope of the biodiesel is higher than the oil feedstock, but also significantly lower than the water. Nevertheless, a change in viscosity was observed and is supported by the sampled data.

Control System

The performance of the on/off regulator can be seen in figure 7.29.

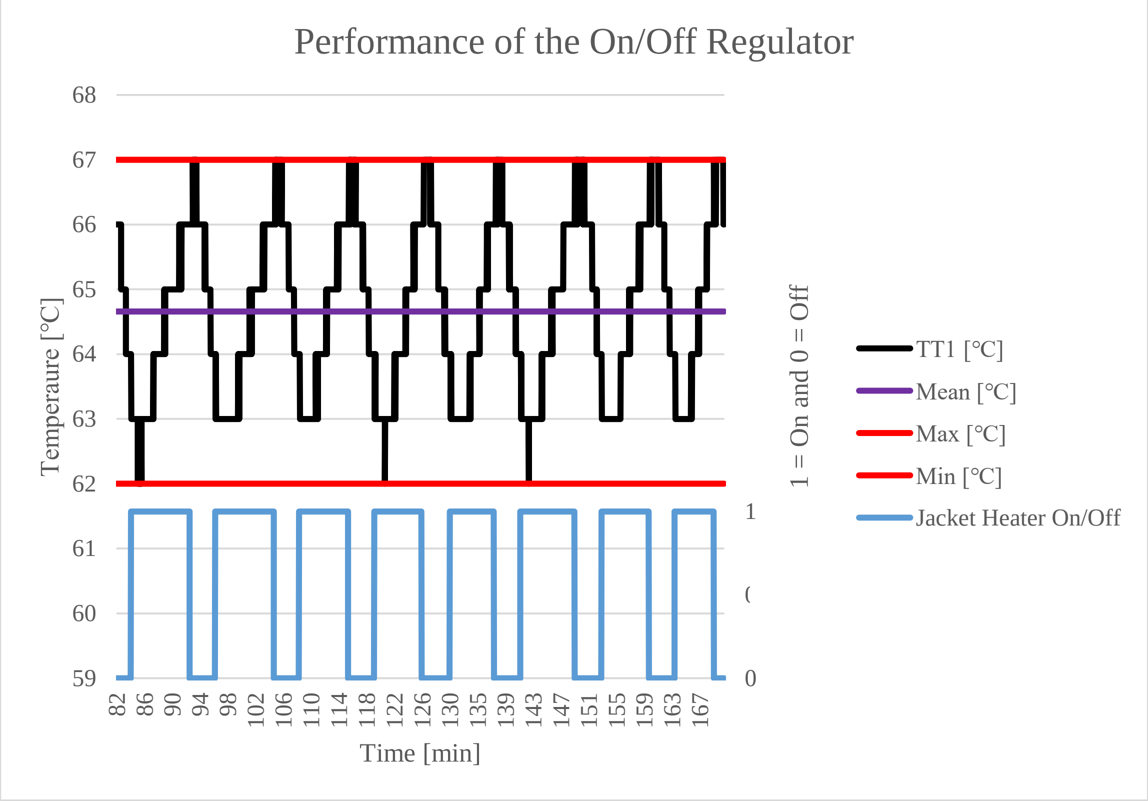


Figure 7.29: Performance of the on/off regulator during the transesterification program

The high temperature setpoint was set to 66°C and the low was set to 64°C. The recorded mean temperature was 64.7°C, which is close to the intended mean of 65°C.

8 Discussion

In this section, the performance of the components, the systems and the production line as a whole will be discussed. The safety and suitability of the production line for use in education and research will also be evaluated.

8.1 Safety

Safety is the most important part when using the production line and must always come before production.

8.1.1 Safety During Work at the Production Line

Care must be taken to ensure that people carrying out electrical work and operations on the production line have experience with electrical work and are responsible. Figure 8.1 shows a proposition for a simplified standard operating procedure (SOP) when doing electrical work on the production line.

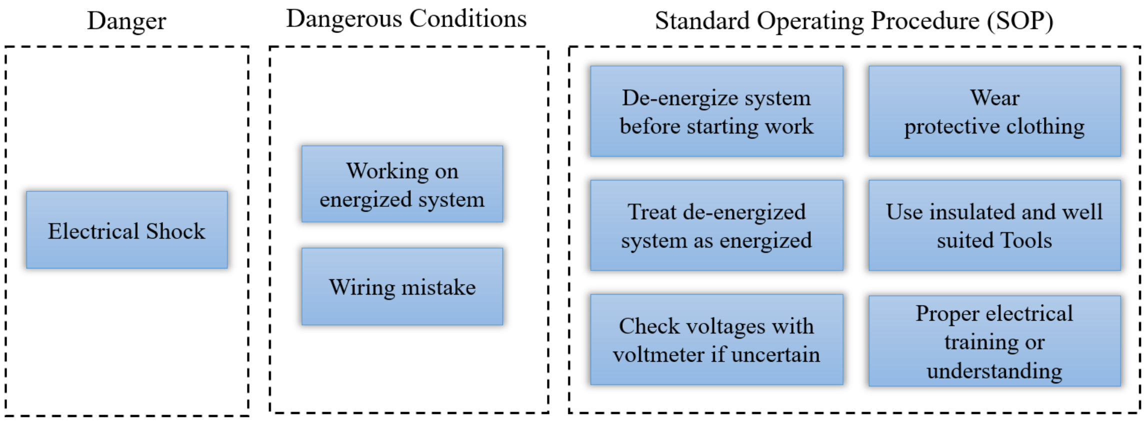


Figure 8.1: Proposition for standard operating procedure (SOP) when performing electrical work on the production line

8.1.2 Safety During Operation at the Production Line

Equipment failure can occur if the production line is used in the wrong way. A method of safeguarding operators is to place barriers between the operators and the hazards. The key point of this philosophy is to create the barriers so that the operator is not hindered from doing the required work, whilst still ensuring the worker’s safety. The main hazards on the production line are electrical shock and vapor ignition. Figure 8.2 shows the proposed barriers on the production line.

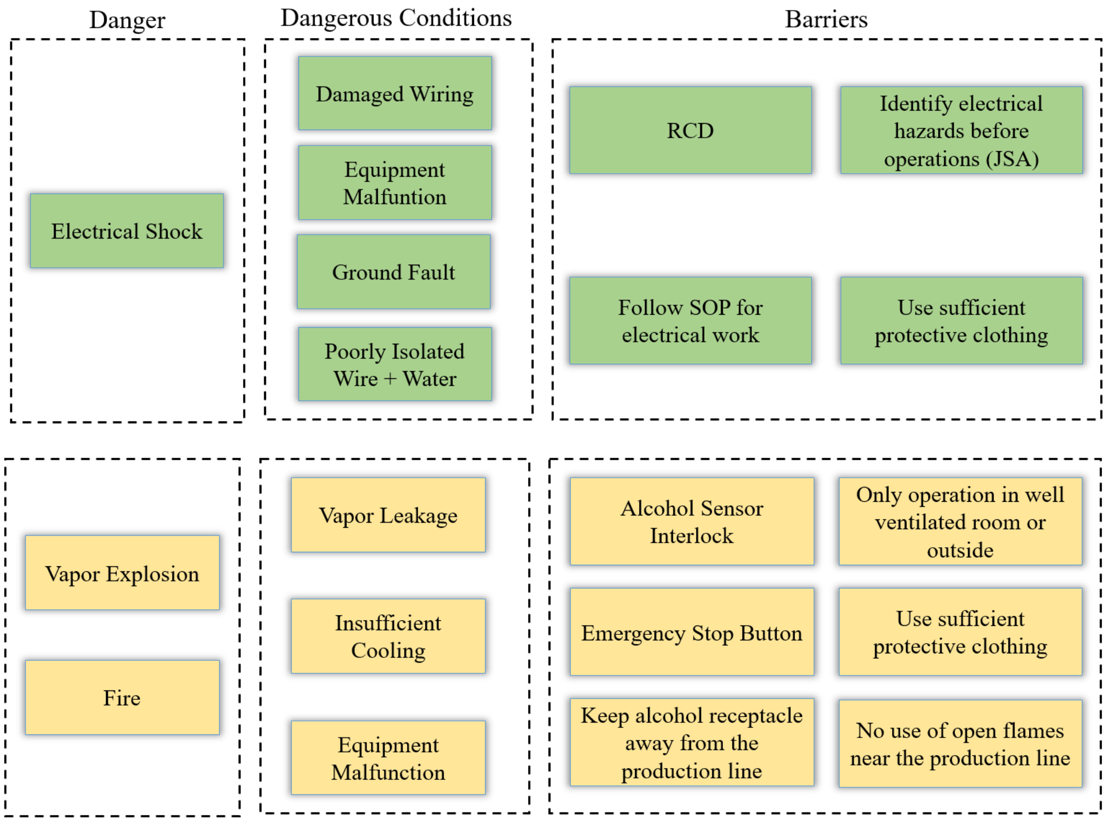


Figure 8.2: Barriers for the main dangers when operating the production line

In addition to the main hazards, there are other hazards as presented in the JSA in table 7.1. Alcohol vapors, especially methanol, and calcium oxide powder can be dangerous for operators of the production line to inhale, ingest or come in contact with. When handling these materials, it is important that the operators use the fully-covering face mask from 3M that was acquired.

The first biodiesel production test proved that the system is able to withstand the pressure of a clogged reactor. However, this was mainly due to the function of the bypass valve. Malfunction of the bypass valve could occur, although this is unlikely as the bypass valve is a purely mechanical device. Nevertheless, in such an event the system will have a weak point. It is uncertain what the weak point of the production line is, but it is likely that the hose/nozzle connections will be the first to yield. During the first test, the treaded joint of the inline heater was observed to be leaking small amounts of liquid during the transesterification program. It is probable that operators will have indication in good time from leaking joints before any rupture or disconnection of hoses occur.

The alcohol sensor proved to be effective at detecting ethanol. The sensitivity of the sensor was found to be high, as it would trigger when within 20 cm of an open alcohol container. When the alcohol source was removed from the sensor, it took around five minutes to get below the trigger value. The reset time for the sensor is of little concern, as the production line will need repair should any alcohol vapor be present during the production process. The alcohol sensor was not implemented in the system to its intended purpose, which was to be a passive sensor component of the control system. During the testing it was rather used to check whether the system was vapor tight. No alcohol vapors were detected by the sensor or by the operators nearby during the second test. Even though the production line was found to be vapor proof during the test, there is no guarantee that it will stay vapor proof with wear and tear. The implementation of the sensor should therefore be of high priority for future work on the production line.

The inline heater requires high flows, preferably up to 3 l/min, to ensure that the feedstock does not overheat within the heater. During the first test, the heater was turned on while there was ethanol in the reaction mixture. The result of this was that the tubes were pulsating from the vapor pressure from the evaporating ethanol. It is important to note that this should not be attempted should there be any future operations on the production line.

Although the safety features on the production line has been improved, there is still a need for further improvements. The safety interlocks were not implemented during the tests but were rather used as “warning” alarms. The 230VAC pushbuttons and LEDs were acquired due to budgetary concerns. Although the 230VAC pushbuttons illuminated at 24 VDC, the “alarm” LED did not. A better way of alerting the operators of alarms should be implemented in the control system, as a blinking light is easy to miss. An audible alarm paired with a visual alarm could be used for alerting operators of errors or dangerous conditions at the production line.

A user manual has been made (appendix O), which explains how the production line is operated and how parameters are changed. It is important that students and operators of the production follow these instructions during operation with the current control program. If custom ladder programs are to be made, it must be made sure that the proper safety interlocks are in place. It is recommended that a JSA is performed before trying out new programs or making any changes to the hardware of the production line.

8.2 The Biodiesel Production Process

The literature study was used as a guideline when setting up the biodiesel production process. Low FAAE yields were obtained during the testing with ethanol, refined feedstock and different calcium oxide catalysts in this thesis. As the production process has not yet been optimized, there are several sources of error.

8.2.1 Preliminary Test Results

The preliminary test had the best results, which is likely due to methanol and calcium oxide of certified laboratory grade being used. Although the catalyst may have been subject to some poisoning, there was likely a relatively high catalytic activity. As no sensor data was acquired from the test, it is difficult to compare the preliminary test to the first and the second test. Currently, the only major change in the equipment of the production line from the preliminary to the second test, is the addition of an agitator instead of static mixers. The Static mixers that were used during the preliminary test were not included during the first and second test. The reason for this was that the material of the static mixer was not thought to be compatible for use with alcohol. Furthermore, the mixers would have lowered the pressure rating of the production line drastically. It then seems likely that the reason for the preliminary test being more successful than the second test, was the difference in process parameters.

8.2.2 First Test Results

The first test was unsuccessful due to the LECA catalyst clogging the filter mesh in the reactor outlet. There was little flow through the reactor, which means that no conclusions can be drawn with regards to the suitability of the LECA catalyst. It is likely that the clogging of the PBR occurred due to some of the LECA being crushed upon addition to the PBR. In hindsight, the LECA catalyst should have been rinsed beforehand with alcohol to remove debris and smaller particles.

8.2.3 Second Test Results

The catalyst during the second test was acquired from an online vendor which listed the rocks as being 97% calcium oxide [103]. The calcium oxide rocks had a diameter of approximately 4 cm and were calcinated before they were crushed. The calcination of $CaCO_3$ to CaO releases CO_2, as can be seen from equation 5. If it is assumed that the rocks were mostly $CaCO_3$, calcination at 950°C for 8 hours would have released large amounts of CO_2, which should have resulted in the rocks breaking. However, all the rocks were intact when they were removed from the furnace. As no tests were performed to determine the purity of the catalyst, there is no certainty to the claim that the catalyst was of the listed purity. However, it can be reasoned that the rocks were of high purity calcium oxide, as the rocks did not crack during calcination.

8.2.4 Possible Sources of Error

For the following section it will be assumed that the calcium oxide used during the preliminary and the second test had similar catalytic activity. The flow through the reactor and the reaction time during the second test was also similar to the preliminary test. Furthermore, the mixing characteristics were likely better during the second test.

During the preliminary test, methanol was used for the transesterification process with a temperature setpoint of approximately 60°C. The first and second tests utilized ethanol with temperature setpoints of 70°C and 65°C, respectively. For the second test it was decided to lower the reaction temperature due to the high ethanol evaporation rate during the first test. The reason for this was due to safety concerns, as the cooling unit was unable to continuously cool the water flowing through the heat exchanger. If the alcohol recovery system is improved, it is possible to use the heat exchanger as a reflux column, so that the alcohol is returned to the mixer. This can be done by simply changing the position of the column. Another method of reflux is by placing the hose from the alcohol pump directly into the alcohol receptacle. In this way, the energy efficiency will be lower, but it will be safer with regards to process control.

The reaction temperature during the transesterification process of the preliminary and the second test is a source of error with regards to the low FAAE yields. During the second test, he temperature of the reaction mixture through the PBR was below 50°C, as TT4 in figure 7.25 shows. During the preliminary work, it is likely that the heat loss between the mixer and the

PBR was lower, as the hoses were shorter and less heat conductive. Therefore, the reaction temperature during the preliminary work was likely closer to the optimal reaction temperature than during the second test.

Finding a suitable heterogeneous catalyst, decreasing the heat loss and improving the reactor design is essential for future operations on “BioMax”. The flow through the packed bed reactor is easily obstructed when a catalyst of non-uniform particle size is used. It is likely that the low yield during the second test was due to a lower reaction temperature and a larger particle size when comparing it to the preliminary test. In both the preliminary and the second test, relatively large catalyst particles were used. Large catalyst particles will have a lower area for catalytic activity per weight than smaller particles. Identifying a method for using smaller catalyst particles without obstructing the flow is essential for future work if PBRs are to be used.

8.2.5 Electric Energy Consumption

The consumed energy during the first and second test was 4.31 kWh and 4.36kWh, respectively. If pure biodiesel was produced during the tests, this would mean that the consumed power per kilo produced would be at approximately 0.51 kWh/kg for the production process.

For conventional biodiesel production technology on an industrial scale, the electric energy for transesterification should be around 0.15 kWh/kg [104]. Furthermore, there are newer technologies such as cavitation reactors, that may provide an even lower energy consumption [105]. However, most of the electric energy for biodiesel production will not be used during transesterification. Janulis [104] found that an additional 8.3 kWh/kg was used in agriculture, extraction of oil and refining of chemicals for biodiesel production with conventional feedstock. Furthermore, the use of fuel in engines will also result in a major loss of energy, due to the low thermal efficiency of engines.

The lower calorific value of biodiesel is approximately 11.25 kWh/kg [106]. For biodiesel production to become viable, the kWh/kg for production must be lowered. With the coming of more advanced biodiesel feedstocks, it is possible that the consumed energy for biodiesel production can be lowered. However, for this to be feasible, more improvements to the cultivation of biomass and the extraction of lipids are required.

The consumed electrical energy at “BioMax” is relatively high compared to industrial production methods. However, a higher energy consumption is to be expected at small-scale production. The energy consumption of the production line can be significantly improved upon by reducing the production time, reducing the use of heating equipment and better insulation of components.

8.3 Untested Processes

Due to time restraints, all aspects of the biodiesel production process could not be tested. The following discussion will examine the likeliness for success of the untested processes.

8.3.1 Moisture Removal

It is uncertain at this point whether removal of moisture from the feedstock by using the inline heater and the centrifuge will work. No water was observed coming out of the top of the centrifuge during the water testing, even though the water was measured to be at 60°C. The centrifuge was tested at a testbench by pump mixing water and oil and adding it to the centrifuge at room temperature. The centrifuge sump was found to contain more water than the mixture from the discharge nozzle. However, the issue with the centrifuge is that it discharges 0.7 liter of liquid upon shutdown. This is a large part of the total added volume of feedstock, as the maximum volume of feedstock is 10 liters. One possibility is to add an extra 0.7 liter of feedstock for each operation to account for the loss. The feedstock can then be separated further and re-added during the next cycle of the production line.

8.3.2 Esterification

The esterification process has not been tested during the scope of this thesis. A catalyst and an adsorbent have been proposed from the literature study. However, the combined function of the catalyst and the adsorbent is uncertain. Park et al. [50] reported reduced catalytic activity for the Amberlyst catalyst as the amount of water increased. Furthermore, water is an unwanted component in the production line, as it can lead to a decrease in transesterification yield and soap formation. The proposed method of using adsorbents must be tested before any permanent changes are made to the production line.

Other methods of removing water is by evaporation or centrifugation. Evaporation is undesirable as it requires high process temperatures for effective removal and will result in downstream issues with removing alcohol from water. Centrifugation is a method that can easily be tested on the production line but will as mentioned result in the loss of some of the reaction mixture.

8.3.3 Stirred Tank Esterification and Transesterification

It is possible to use the stirred tank as a reactor. This could be done by adding calcium oxide powder directly to the mixer instead of into the PBRs. This would reduce the need for several components and could prove more efficient, as well as more comparable to laboratory tests. However, the production line is currently not equipped to handle this, as the calcium oxide would likely damage pumps and other components. For future work, the possibility of using the mixer as a stirred tank reactor should be investigated.

8.3.4 Purification

Two purification processes have been presented in this thesis. During Omberg's thesis, two columns were acquired of the type that is displayed in Figure 4.48. Figure 2.16 shows how a dry or wet wash configuration of the columns could be set up.

The major drawback of using water for the purification process is that there will be introduced water in the product. This must then be separated from the biodiesel. With the current equipment there are two possibilities for how this can be done:

The first is by reintroducing the product to the production line and utilize the existing heating equipment. However, this will introduce water into the system. If a flushing system is devised that uses a fluid that is able to purge the system of water, water washing could be a part of the integrated system.

The second possibility is to install a heating element in or around the purification column. The columns would then have to be fitted with extra equipment such as temperature transmitters and a sprinkler nozzle.

Dry washing has the benefit that it can be done in a packed bed or in a stirred tank reactor. An extra inlet was added to the top of the mixer tank, in the event that adsorbents or dry washing media were to be added directly to the mixer. This is a possibility, but a simple way of removing the dry washing media must then be devised. The columns were originally made for use with ion exchange resins or adsorbents in a packed bed [10]. The use of ion exchange resin or adsorbents for dry washing of biodiesel has some benefits compared to water washing. The ion exchange resin can be used up to several times and will likely not require any modifications or instrumentation to be added to the column. The major drawback with ion exchange resin or adsorbents is that it must be backwashed and regenerated [55]. The backwash is usually done by utilizing alcohol, which could be implemented by using the condensed alcohol from the production process.

8.3.5 Flushing

There is currently no good way of purging the hoses of reaction mixture after the production process is finished. A flushing pattern using pressurized air was proposed in table 6.11. However, this requires that the production line is operated close to or with an air compressor. This will increase the energy consumption of the production line and might not be the best way of going about it. A flushing system using the condensed alcohol from the production process could be used to purge the system of reaction mixture. However, this would require a down-stream separation and regeneration process for the alcohol. Currently the proposed method for flushing the system is to use pressurized air as it is accessible at the production site. For future work an integrated method should be considered.

8.4 The Function of the Production Line

The overall design of the processes and components of the production line has performed above what was expected. In the following section the overall function of "BioMax" will be discussed.

8.4.1 Addition of Feedstock and Alcohol to the Production Line

The automated addition of oil worked well when tested with water. The reason for this system being employed was to limit the possibility of the production line running dry. The oil tank was also designed to serve other purposes, such as straining "dirty oil" and performing a controlled addition of feedstock. During the first generation of "BioMax" and the preliminary work, the mixer tanks had built-in level transmitters. This made gave some additional insight into the process but was not an essential part of the process. When the agitator was implemented into the mixer, the level transmitter had to be removed. The current placement of the level transmitter is useful in that it can provide some details with regards to the flow of the feedstock, as seen in figure 7.28. Furthermore, the sampled data can be used to verify the amount of added feedstock. Currently the add oil program is not functioning as the density of the added liquid must be known beforehand for the program to be precise. As density is temperature dependent, it will require an on-site measurement for it to be accurate.

During the first test the alcohol was added through the oil tank. Although there were no difficulties with the first addition of alcohol, alcohol was re-added due to the high rate of alcohol evaporation. This was difficult as there was pressure in the system. Once CV7 was opened, the ethanol went slowly into the system. It is likely that the ethanol was hindered by the steam pressure within the mixer. Because of this, it is not recommended that the oil tank is used for returning alcohol to the system after alcohol has been added. The use of the alcohol pump is safer and is likely to provide a better mixing of alcohol and feedstock.

8.4.2 The Mixing System

Ethanol was added to the system through the alcohol pump during the second test. Figure 7.27Figure 7.26 shows the measured flow from the flow transmitter during the pump mixing. The feasibility of pump mixing on the production line was examined when the components were tested at the testbench. Employment of a static mixer after t-fitting 1 might improve the mixing of oil and alcohol. The results of the pump mixing were not examined during the second test, but it is recommended that future work on the production line examine this method of mixing.

The stirred tank system worked well, as an emulsion of alcohol in oil was obtained, judging by the samples that were drawn during the first test. The sealing mechanism that was used to prevent vapors from escaping through the bushing hole worked well, as no ethanol vapors was detected by the alcohol sensor during the second test. It is not recommended that the VFD that controls the agitator is set to higher frequencies than 25 Hz. High speeds of the shaft will result in significant vibrations and might damage the sealing mechanism or the nylon bushing.

The research of Todorovic et al. showed that adding crude biodiesel to the biodiesel feedstock reduced the induction period for the transesterification process. It is recommended that this is tested for future work at the production line, as it has the potential of reducing the reaction time, and thereby the consumed energy.

8.4.3 The Alcohol Recovery System

The alcohol recovery system worked well during the first and second test, considering that there were some significant issues with the cooling unit. The ethanol vapors were cooled sufficiently for condensation, even though the cooling water reached temperatures as high as 35°C. For future work on the production line, a calculation of the required cooling power should be performed. It is possible that the current cooling system can be improved upon in the current configuration, but this will be left for future work on the production line.

8.4.4 The Heating System

The limitations of heating system have in large parts been discussed in the sections below. However, the heating system has been significantly improved from previous work at "BioMax". The inline heater proved to be a more time efficient way of heating the feedstock. Furthermore, a far more effective heating was achieved in the mixer tank compared to the preliminary work, due to the addition of the agitator.

However, there are some factors that must be considered with regards to the heating system. It must be verified that the temperature transmitters are showing accurate values with regards to the temperature of the reaction mixture. TT1 is directly connected to the mixer tank, which has the jacket heater connected to it. The temperature of the jacket heater has been 120°C during testing, and so it is likely that the mixer tank itself will have high temperatures due to the heat characteristics of stainless steel [107, 108]. TT3 has the highest possibility of showing accurate values, as it is removed from any heat sources and is in direct contact with the liquid, as can be seen in figure 8.3.

Figure 8.3: TT'3's placement in the pipe system

Although the time efficiency has been increased by adding the inline heater, the system is still in need of optimization. It is evident from Figure 7.24 that there is a high heat loss through the hoses. This is likely due to the tubes not being insulated. Insulation material for the pipes can be acquired at a relatively low cost, as the production line operates on low temperatures. There is also a large heat loss from the surface of the jacket heater, which could be reduced by covering it in an insulating material. Insulation for the mixer tank will require the use of a material that is suitable for temperatures up to at least 120 °C.

The mean temperature of the on/off regulator is fairly accurate. However, reaching the setpoint temperature is impossible with this regulator. SoMachine Basic has the possibility of using P, PI and PID regulators (figure 8.5). This is difficult when using a device that is either on or off. If the heaters can be configured so that the effect is varied by an analogue signal, it is possible to implement a PID controller.

8.4.5 The Mass Transfer System

As demonstrated with the data from the first test, there are currently no good indications of whether there is flow through the PBRs or not. The flow of liquid through the system seems to be on the low end of the flow transmitter's range. Because of this, the flow transmitter cannot currently be relied upon. If a new flow transmitter is acquired, it must be able to measure at lower flows and viscosities. A pressure gauge or a pressure transmitter could be placed between the main pump and the PBRs to indicate clogging. Data sampled from a pressure transmitter could be used in the optimization of a new reactor design.

8.4.6 The Design

A major change on the production line from the preliminary test to the first and second test, is that the FKM hoses has been replaced with wire-braided PTFE hoses. As there is no recorded data with regards to the temperature drop when using the FKM hoses, comparison is difficult. However, it is likely that the heat loss was higher for the wire-braided hoses due to the relatively high conductivity [108].

The design of the second generation of “BioMax” is a product of the previous design reports and work on the production line. In this thesis, the focus has been to make a functional assembly based on the components and the previous designs. The previous versions brought along some technical difficulties that has been resolved to some extent. The reason for increasing the size of the framework from the preliminary version was to make working on the facility easier and to enable for later modifications and additions. Currently, the facility is approximately 2.2 meters tall from ground level and has a center of gravity approximately 0.8 meters from the lifting points (appendix P). The high center of gravity makes the transport of the production line difficult. The center of gravity can be lowered during transport by adding weights to the bottom level of the framework beforehand.

The new mixing system was constructed by the workshop at NMBU. As there were two pumps with VFDs from Reinaas’ version of BioMax, one of the pump motors was repurposed. The design worked well, although there are significant vibrations at high mixing speeds. The durability of the design is uncertain, as heat from the jacket heater carries to the bushing. Debris has been observed coming off the rubber connection between the motor and the shaft.

The design of the framework for the second generation of “BioMax” was made with the use of gravity feeding in mind. As the feedstock must be added through the oil tank, operators will need a ladder to add the oil. Ladder steps could be welded on the framework as a permanent feature.

Some cable management was done by adding the cable channels. However as was pointed out in Reinaas’ master thesis, the electrical cabinet is too small. The optimal design would be to have more room for adding cable channels, which would provide some cable management within the electrical cabinet as well. A bottom plate for the electrical cabinet equipped with cable glands was created, but not implemented due to time constraints.

The mechanism for adding and removing the catalyst in the reactor could also be improved upon. A solution where the catalyst could be easily removed without having to loosen or fasten any bolts would be beneficial. Replacing the catalyst in the reactor is reasonably simple but can be subject to user error, as experienced during the first test.

8.5 Suitability for Use in Education and Research

A large part of the main objective of this thesis was to create a production line that is suitable for use in education and research. Although the production line is in need of some improvements, there is currently a high potential for the production line to be educational for students.

8.5.1 "BioMax" for Use in Education

During Reinaas' master thesis, the production line had several components that were made of acrylic material. Although this material proved to be incompatible with the use of alcohol, it made the production line more visually appealing. Currently, there are no permanent transparent sections or components where students or operators can watch what is happening in the production process. The areas of most interest in the production process are currently the reactors, the mixer and the centrifuge.

As the reactors will have to be rebuilt, it would be advantageous if the reactors were created in a transparent material that is suited for the production process. With regards to the mixer tank, there is the possibility of inserting a sight glass to the tank. The least intrusive way of achieving a more visual production process is by adding sight glasses between components (figure 8.4). This will aid operators and students in understanding how the production process is set up.

Figure 8.4: 1/2" Sight glass from AliExpress [105]

A natural way of improving the production line is to use it as a part of the education. All the programs used during the span of this thesis are free for students to use and download. Students will be introduced to P&ID drawings, wiring schematics and process control, which are used in most manufacturing companies. There is a great potential in having students design their own control programs for specific tasks and also testing them. Furthermore, operation of the production line requires mass balance calculations and testing of the product will provide students with hands-on experience with laboratory work.

8.5.2 "BioMax" for Use in Research

For "BioMax" to be suitable for research, the control system has to be improved. This can be done by changing the way the on/off regulator is set up. The on/off regulator for temperature control is currently operating on inputs from all temperature transmitters, except TT2. The goal of the regulator is to ensure that the temperature of the reaction mixture is at the temperature setpoint. As TT1 is most likely too close to the heat source, this transmitter should not be used during the transesterification program. During the transesterification program, the temperature in the reactors should be at the temperature setpoint. There are currently two extra temperature transmitters from the first generation of "BioMax" that has not been utilized. These transmitters could be attached to the new reactor design. The measurement values from these sensors could be used in the on/off regulator.

Furthermore, as flow is an important parameter and different feedstock will have different viscosities, there is need for a better control of the flow. As the main pump is operated by a VFD, that can be controlled by analogue outputs from the PLC, there is the possibility of employing a PID regulator. SoMachine Basic has built in PID regulators that can be programmed to operate on the signal from the flow transmitter. However, for this to work, it is imperative that a flow transmitter that is suitable for the flow domain is employed. The PID regulator setup in SoMachine Basic can be seen in figure 8.5.

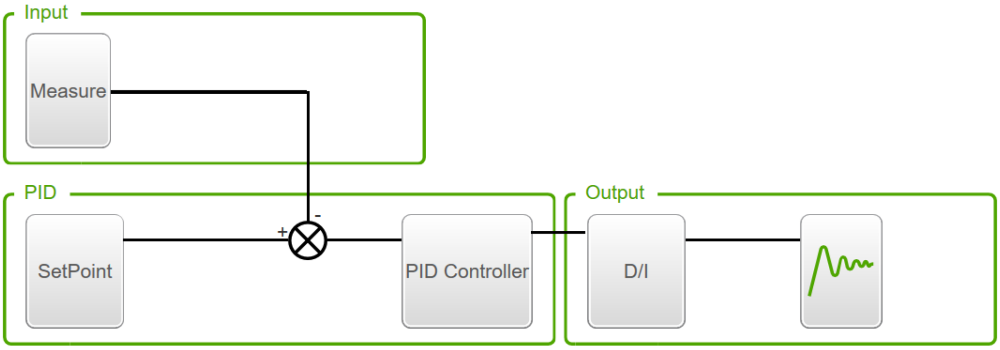


Figure 8.5: PID regulator setup in SoMachine Basic

8.6 Further Work

"BioMax" is operational, but it is evident from testing the production line that there is still a need for improvements. For high-grade biodiesel to be produced on "BioMax", laboratory tests must be performed that are transferable to the production process of the production line. In the meantime, optimization of the control system and improvements on the production line can be performed. The following are propositions for improvements that can increase the safety, performance or efficiency of "BioMax".

Design Improvements

- → Add sight glasses to the production line i.e. between components or in the mixer.
- → Research the feasibility of creating a transparent lid for the centrifuge.
- → Research the feasibility of creating the PBRs in a transparent material.

Electrical System Improvements

- → Acquire and install an RCD for the safety of operators and students.
- → Hardwire the emergency stop button as normally closed to the power supply. The current function of the emergency stop button can assigned to a red pushbutton.
- → Acquire a larger electrical cabinet and install cable channels. If this is not done, a bottom plate must be installed on the current electrical cabinet and an effort must be made to rewire the cabinet as orderly as possible.

Mechanical Design Improvements

- → The center of gravity of the production line should be lowered. This can for instance be done with a more tactical placement of components.

- → New reactor lids must be made, as the acrylic material has started to crack from the testing. If the reactors are redesigned and constructed, the construction material should be transparent. The new reactors should be designed for the catalyst.
- → Construct a cover for the connection between the agitator shaft and the motor.

Equipment Improvements

- → Acquire a better suited flow transmitter.
- → Improve the alcohol recovery system. A new cooling unit can either be acquired or a new configuration can be devised.
- → Insulate piping and the mixer tank with a suitable material.

Control System Improvements

- → Implement the alcohol sensor into the control system, so that it shuts down the production line when alcohol is sensed.
- → Improve the temperature controller by adding temperature transmitters near, or directly into, the PBRs. In the long term it should be considered to implement a more sophisticated temperature regulator, such as a PID regulator
- → Implement a PID regulator for the main process pump once a new flow transmitter has been acquired and tested.
- → Replace the LED lights that are used in the control panel with lights that are more suited for the 24VDC system.
- → Consider installing an enhanced alert system for alerting operators.
- → Create a user interface on the stationary computer that displays the processes with parameters (with i.e. Visual Basic).

Biodiesel Production

The improvement of the biodiesel production should be done on a laboratory scale. More research is needed into finding a suitable method of using heterogeneous catalysts in the production process.

- → Perform further tests of the production line to determine whether biodiesel can be produced in the current configuration of "BioMax".
- → Determine optimal catalyst loading and size and perform laboratory tests to determine optimal process parameters for production with the catalyst.
- → Evaluate the need for a reflux configuration of the alcohol recovery system. This will make it easier to utilize process temperatures close to the boiling point of the alcohol.
- → Determine the feasibility for producing biodiesel with heterogeneous catalyst in a stirred tank reactor configuration on the production line.
- → Determine feasibility of producing biodiesel from low-cost feedstock, such as waste cooking oil, from the proposed methods.
- → Test pretreatment of unrefined feedstock by centrifugation.
- → Implement a purification system using depending on results from testing.

9 Conclusion

The second generation of the small-scale biodiesel production line "BioMax" has been built and tested. The production line has been deemed safe for use in education and research, in the context of section of 8.1.1 and 8.1.2. During the span of this thesis, the following has been accomplished:

- → The previous work on "BioMax" has been presented and evaluated. The production line has been built and the process and energy efficiency has been improved.
- → A detailed JSA was performed before testing the production line. Safety interlocks has been implemented into the control system and recommendations for conduct concerning work and operations on the production line has been put forward.
- → A user manual and a user interface has been created to enable simplified operation of the production line. The production line can be operated with little to no prior technical training.
- → A ladder program has been made and tested for control of the production line with success. A detailed section on programming of the PLC and the control system has been added to ease the induction period for future work.
- → The function of the production line has been tested and evaluated.
- → Documentation of the production line has been made and recommendations for further work has been suggested.

Methods for pretreatment and esterification of unrefined feedstock and purification of biodiesel has been proposed and discussed but remains to be tested. Catalysts has been proposed and tested, but with varying results.

The production line is suitable for use in education, but some effort must be made to make the production process more visual.

Currently, there are too many sources of error to say anything certain with regards to the suitability of the production line for biodiesel production. The production line has been tested three times, where only one test resulted in production of biodiesel. Optimization and testing of the parameters of the biodiesel production process is required before the production line can be employed with confidence in scientific research.

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11 Appendixes

Appendix A: Calculation of FAME Yield from the Preliminary Test

The amount of feedstock added during the preliminary test was approximately 10 liters of refined rapeseed oil. It will be assumed that the rapeseed oil consists of 100% triolein, due to the fatty acid profile from Sanford et al. in table A1.

Table A1: Fatty Acid Profile in Canola and Sunflower Oil [25]

Property	**C16:0**	**C16:1**	**C18:0**	**C18:1**	**C18:2**	**C18:3**	**C20:0**	**C22:0**	**C24:0**	**C24:1**
Name	Palmitic	Palmitoleic	Stearic	Oleic	Linoleic	Linolenic	Arachidic	Behenic	Lignoceric	Nervonic
Sunflower	4.2	-	3.3	63.6	27.6	0.2	-	0.7	0.4	-
Rapeseed	3.8	0.3	1.9	63.9	19.0	9.7	0.6	0.4	0.2	0.2

The oil used during the test had a density that was determined by weighing the oil in a 300 ml glass. The weight of the oil was measured to be 273 grams, which gives the density:

$$\rho_{oil} = \frac{m_{oil}}{V_{oil}} = \frac{0.273kg}{0.300L} = 0.91\ kg/L$$

The weight of the oil is then be given by:

$$m_{oil} = \rho_{oil} \cdot V_{oil} = 0.91\frac{kg}{L} \cdot 10.0L = 9.1kg$$

The amount of glycerol was approximated to be 200 ml. Glycerol has a density of 1.26 kg/L, which gives a mass of:

$$m_{gly} = \ \rho_{gly} \cdot V_{gly} = 1.26\frac{kg}{L} * 0.2\ L = 0.252\ kg$$

The molecular weight of a glycerol molecule is 92.09 g/mol which means that the amount of mols is given by:

$$n = \frac{m}{M} = \frac{252g}{92.09\ g/mol} = 2.736\ mol$$

From the balanced equation the amount and mass of each reactant can be calculated, as can be seen from table A2

Table A2: Mass Balance for the Preliminary Test

Molar Ratio	1	3	$\longleftrightarrow$	3	1
Compound	Triolein	Methanol		Methyl Oleate	Glycerol
Molecular Formula	$C_{57}H_{104}O_6$	CH_3OH		$C_{19}H_{36}O_2$	$C_3H_8O_3$
C (12.011 g/mol)	57	1		19	3
H (1.008 g/mol)	104	4		36	8
O (15.999 g/mol)	6	1		2	3
Molecular weight (g/mol)	885.43	32.04		295.49	92.09
Amount (mol)	2.736	8.209		8.209	2.736
Mass (g)	2423	263		2434	252

As can be seen from table A2, 2434 gram of methyl oleate (FAME) was produced during the preliminary test, assuming that the approximation of 200 ml of glycerol is correct. The FAME yield can then be calculated from equation 5.

$$yield\ (\%) = \frac{Weight\ of\ FAME\ produced\ (g)}{Weight\ of\ oil\ used\ (g)} \times 100\% = \frac{2.434\ kg}{9.100\ kg} \times 100\% = 26.7\%$$

Appendix B.1: Calculation of Production Volume

The inside diameter and height of the mixer tank was measured to be 199mm and 505mm, respectively. The area and the volume of the mixer tank can then be given by:

$$A_{mixer} = \frac{\pi}{4} \cdot d_{mixer}^2 = \frac{\pi}{4} \cdot (199mm)^2 = 31102.6\, mm^2$$

$$V_{mixer} = A \cdot h = 31102.6mm^2 * 505mm = 15706789\, mm^3 \cdot 10^{-6} \frac{L}{mm^3} = 15.7L$$

However, the volume of the agitator must be accounted for and it must be possible for the alcohol vapor to get thought the outlet. Therefore, the maximum liquid level in the mixer tank is set to be 85% of the maximum volume.

$$V_{max} = 0.85 \cdot 15.7L = 13.3L \approx 13.0L$$

Furthermore, the inline heater was measured to hold 0.25 liters and there will be some liquid moving though the pipes and the PBR during the transesterification. There is approximately 4 meters of pipe in the transesterification and esterification line. The volume of liquid in the ½” pipes can be calculated from the following:

$$d_{pipe} = 12.5mm$$

$$V_{pipe} = \frac{\pi}{4} \cdot d^2 \cdot l = \frac{\pi}{4} \cdot 12.5mm^2 \cdot 4000mm = 490874mm^3 \cdot 10^{-6} \frac{L}{mm^3} = 0.49L$$

The volume of the PBRs

$$d_{PBR} = 90mm$$

$$A_{PBR} = \frac{\pi}{4} \cdot d_{PBR}^2 = \frac{\pi}{4} \cdot 90mm^2 = 6361.7mm^2$$

The height of the PBRs were measured to be 140mm. The volume of the PBRs without catalyst is then given by:

$$V_{PBR} = A_{PBR} \cdot h_{PBR} = 6361.7mm^2 \cdot 140mm = 890642mm^3 \cdot 10^{-6} \frac{L}{mm^3} = 0.89L$$

During the transesterification and esterification, the PBRs will be filled with catalyst. To be conservative, a packing of 50 vol% is assumed. The volume of the PBR with catalyst is then given by:

$$V_{PBR,catalyst} = 0.89L \cdot 50\% \approx 0.45L$$

The maximum volume of liquid outside of the mixer tank is then given by:

$$V_{tot,outside} = V_{pipe} + V_{inline} + V_{PBR,catalyst} = 0.49L + 0.25L + 0.45L \approx 1.2L$$

The minimum amount that can be produced effectively at the production line is in large parts dictated by the height of the impellers on the agitator. The agitator has two levels of impellers. The lowest level is 152mm above the bottom lid. The minimal effective level of reaction mixture in the mixer can then be calculated:

$$V_{min,eff} = A_{mixer} \cdot h = 31102.6mm^2 \cdot 152mm = 4727588mm^3 \cdot 10^{-6} \frac{L}{mm^3} = 4.7L$$

$$V_{min} = V_{min,eff} + V_{tot,outside} = 4.7L + 1.2L = 5.9L \approx 6.0L$$

The minimum production volume on the production line is 6 liters and the maximum is 13 liters.

Appendix B.2: Calculation of Oil Tank Volume

The diameter of the oil tank was measured to be 198mm. The oil tank has a level gauge that was calibrated for water at 20℃, that can be used to verify the added volume of liquid. The level transmitter is placed at the line of the weld between the cylinder and the bottom dish. Therefore, it must be kept in mind that there will be some liquid below the level transmitter. The volume of the cylindrical tank can then be given as a function of the height:

$$V_{tank} = \frac{\pi}{4} \cdot d^2 \cdot h = \frac{\pi}{4} \cdot 198mm^2 \cdot h = 30790.75mm^2 \cdot h$$

The height of the tank can then be found as a function of volume:

$$h = \frac{V_{tank}}{30790.75mm^2}$$

When testing the system, a volume of 10 liters of water was added through the oil tank. The level transmitter is calibrated for water, and so the level that must be added can be calculated:

$$h_{10L,H_2O} = \frac{10L \cdot 10^6 \frac{mm^3}{L}}{30790.75mm^2} = 324.8mm \approx 325mm$$

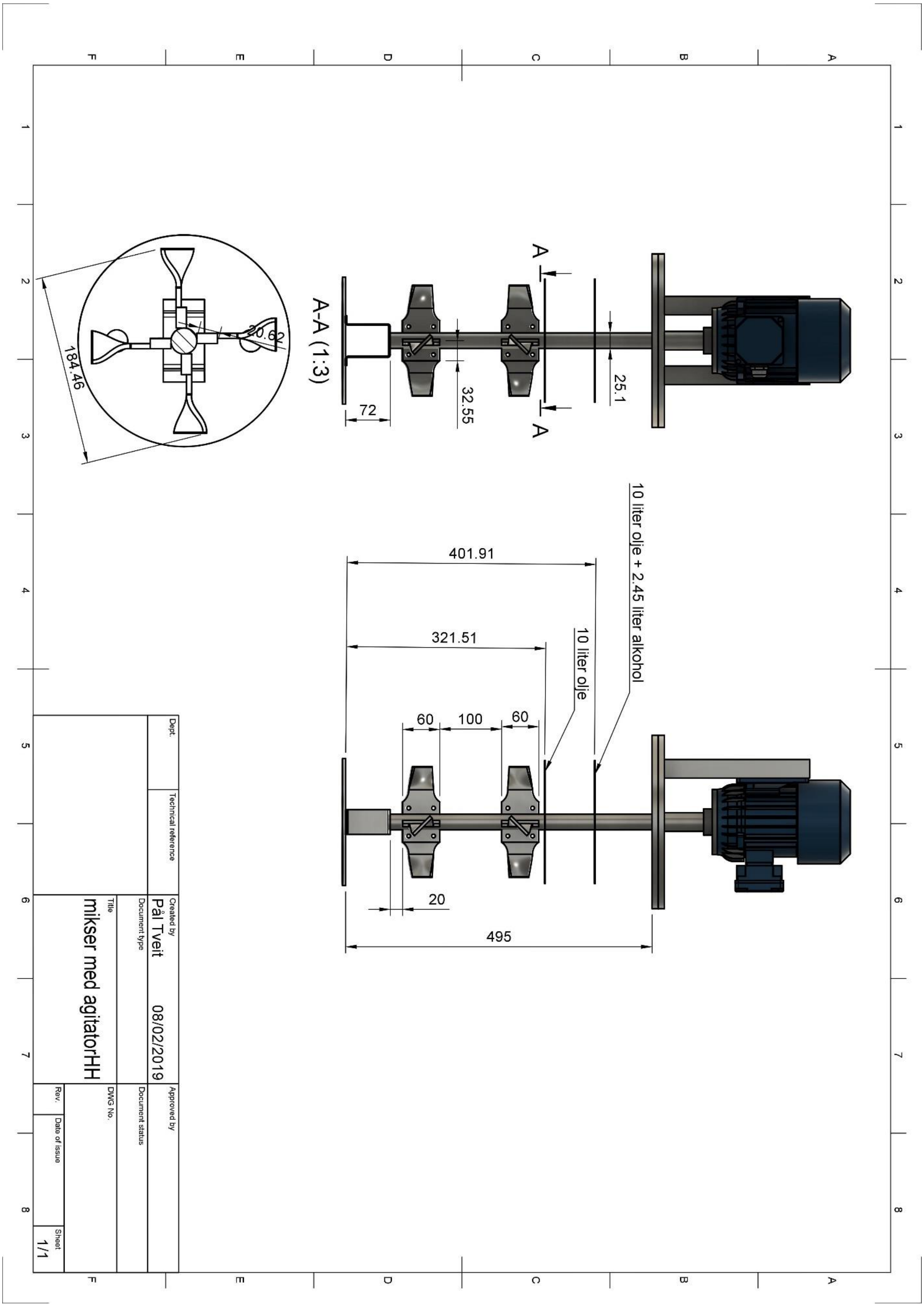
However, during testing it was discovered that more liquid was after CV7 received the “Close” signal, as the valve moves slowly. The difference between the initial and the real-time measurement was inserted as 290mm, meaning a difference at 35mm.

Oil has a higher viscosity than water. The flow of water was found to be approximately 3.3 times slower than water though the tank from Figure 7.28. Furthermore, sunflower oil has a lower S.G. (0.91) than water, and so the level must be recalibrated. The calculation for adding 10 liters of oil to the system can be written as the following:

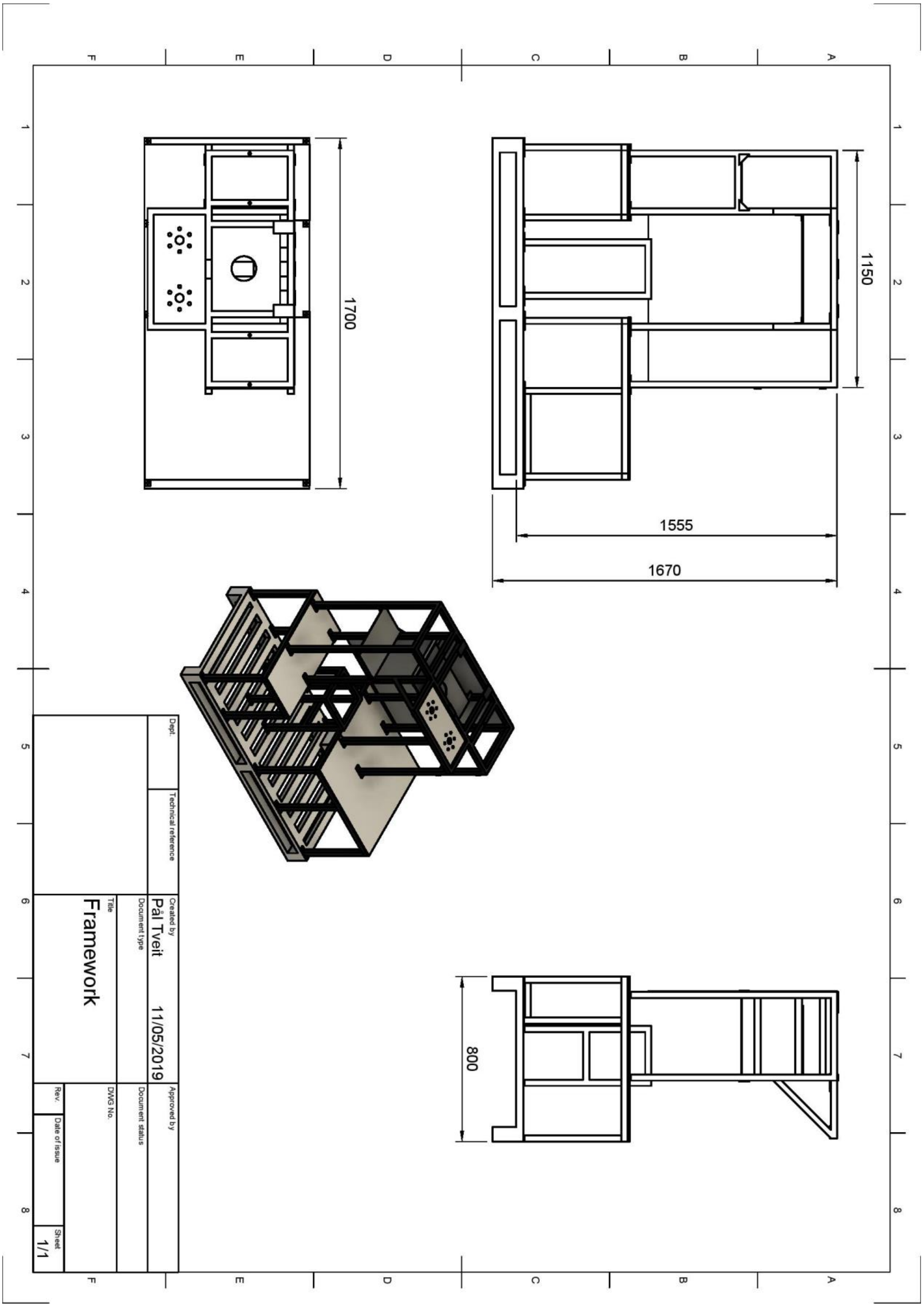
$$h_{10L,H_2O} = 0.91 \cdot \frac{10L \cdot 10^6 \frac{mm^3}{L}}{30790.75mm^2} - \frac{35mm}{3.3} = 284.93mm \approx 285mm$$

The difference between the initial and the real-time measurement can be inserted in the ladder program as 285mm if 10 liters of sunflower oil is to be added.

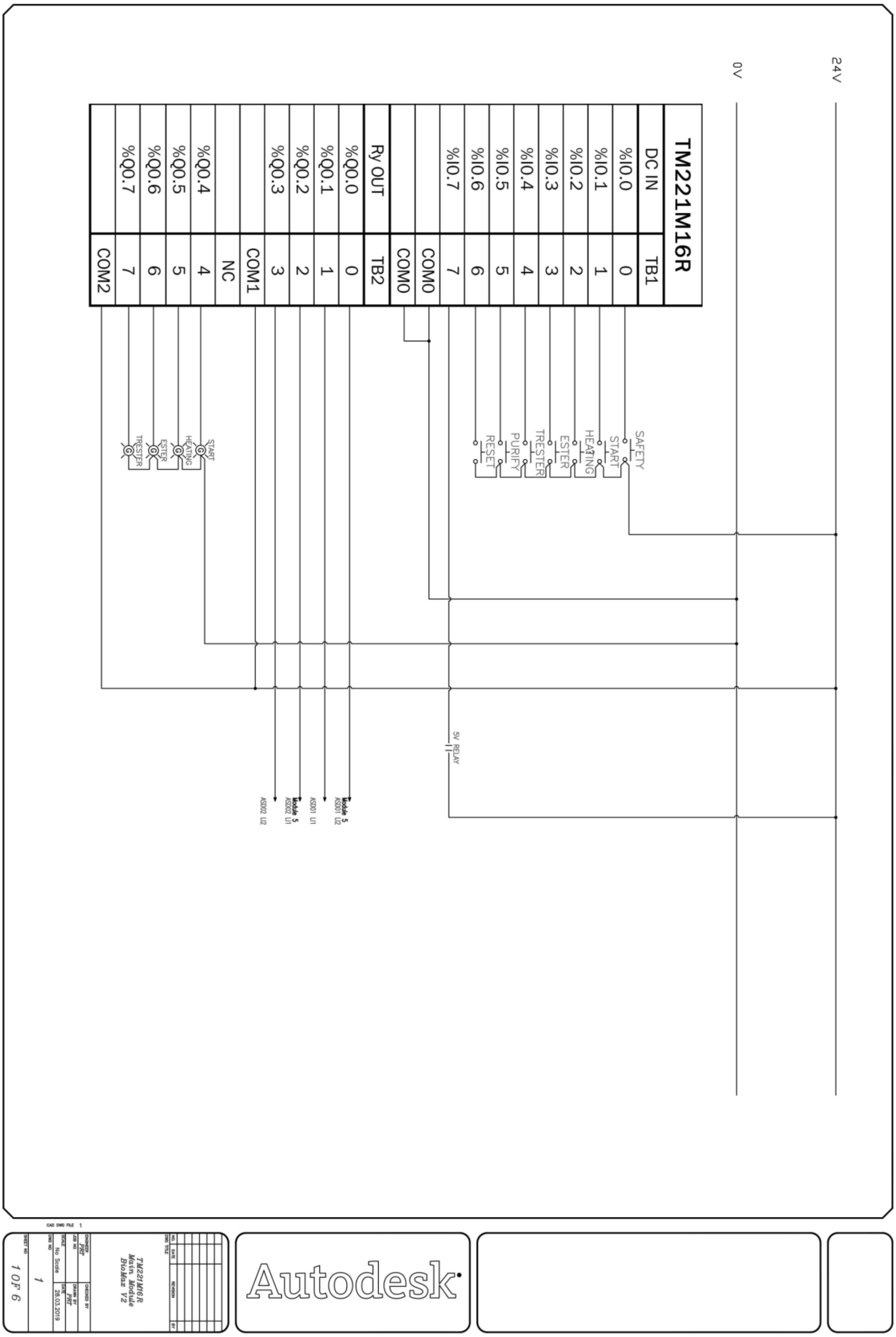
Appendix C.1: Construction Drawing for the Agitator



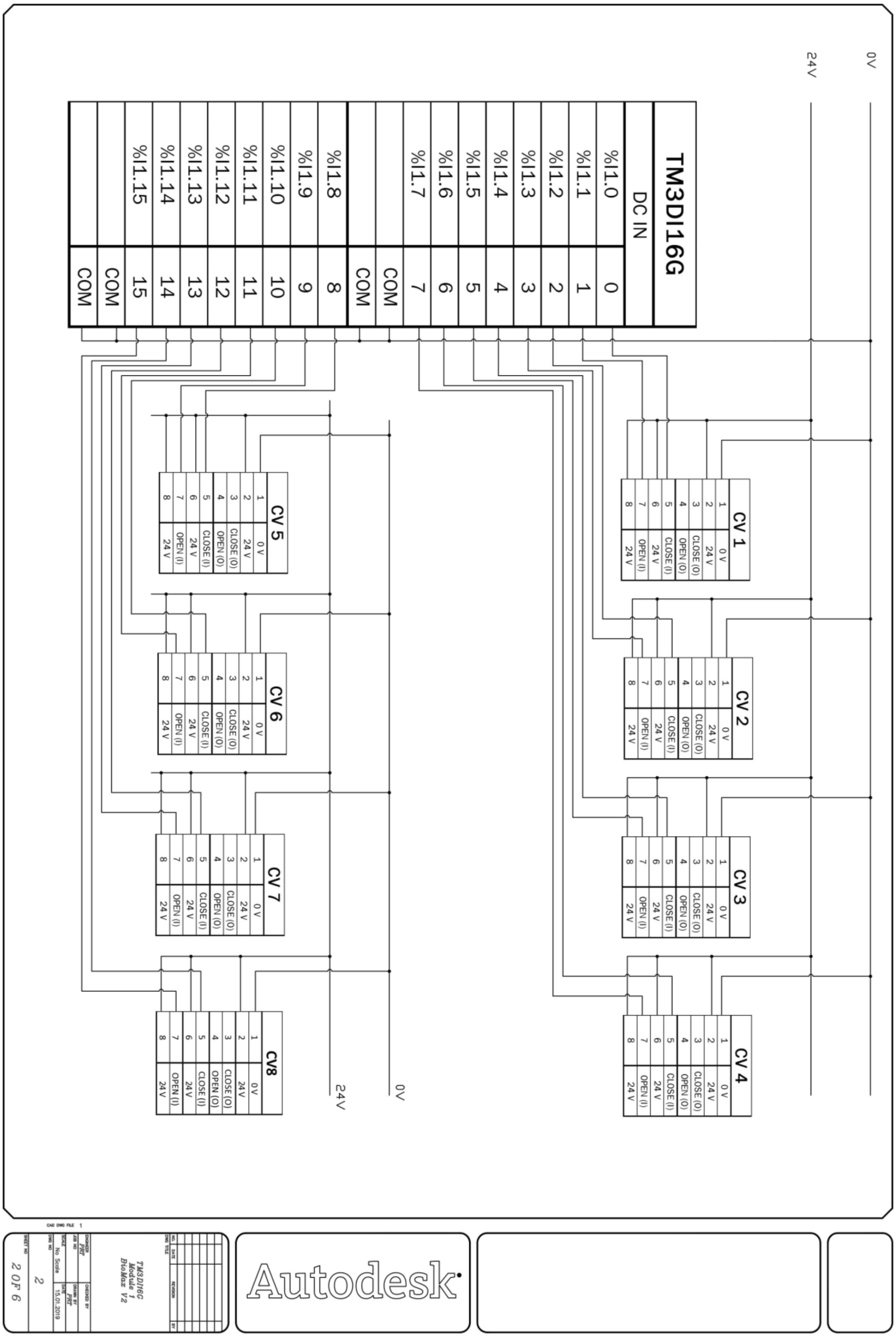
Appendix C.2: Dimensions for the Framework



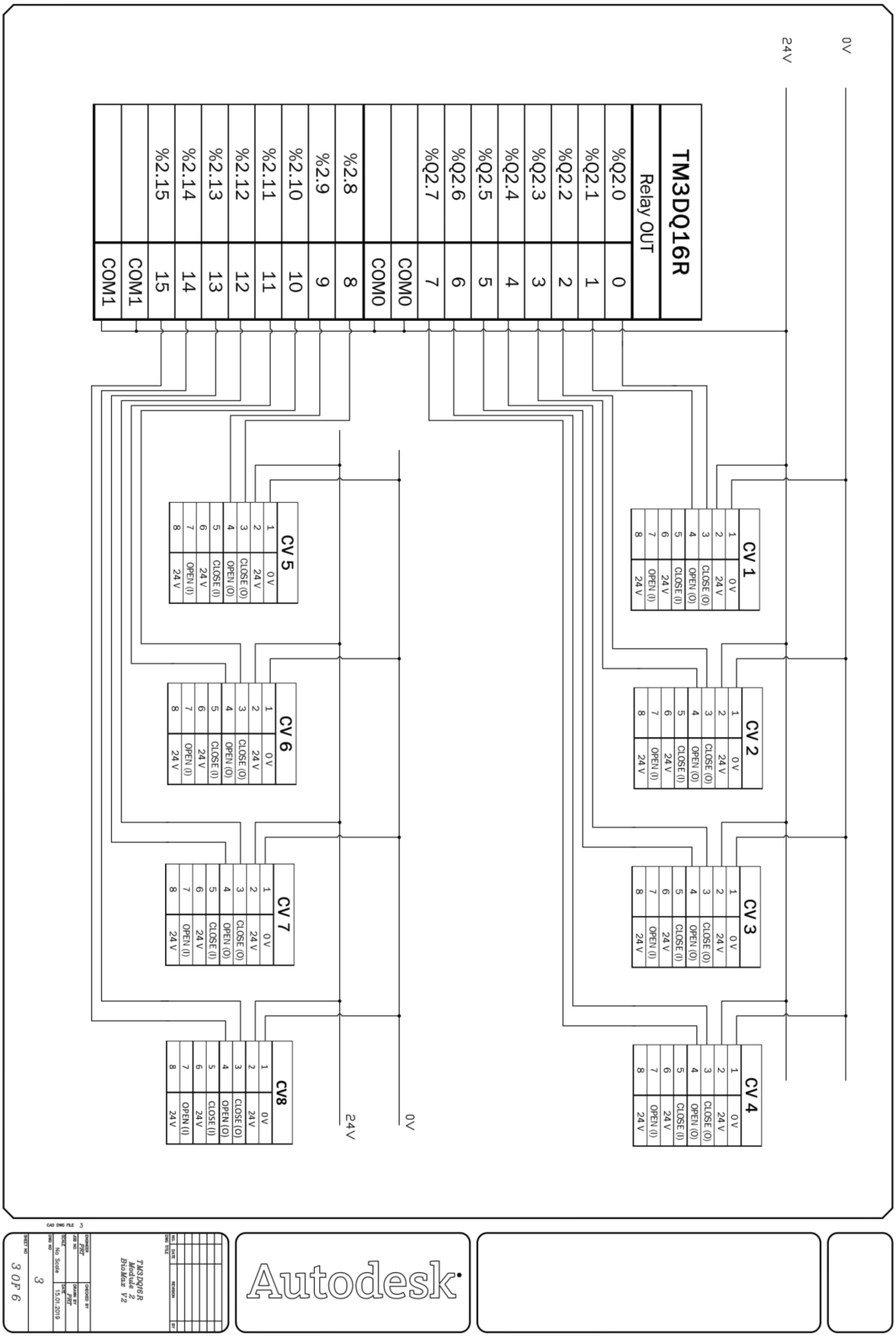
Appendix D.1: Wiring Diagram for the Main Module



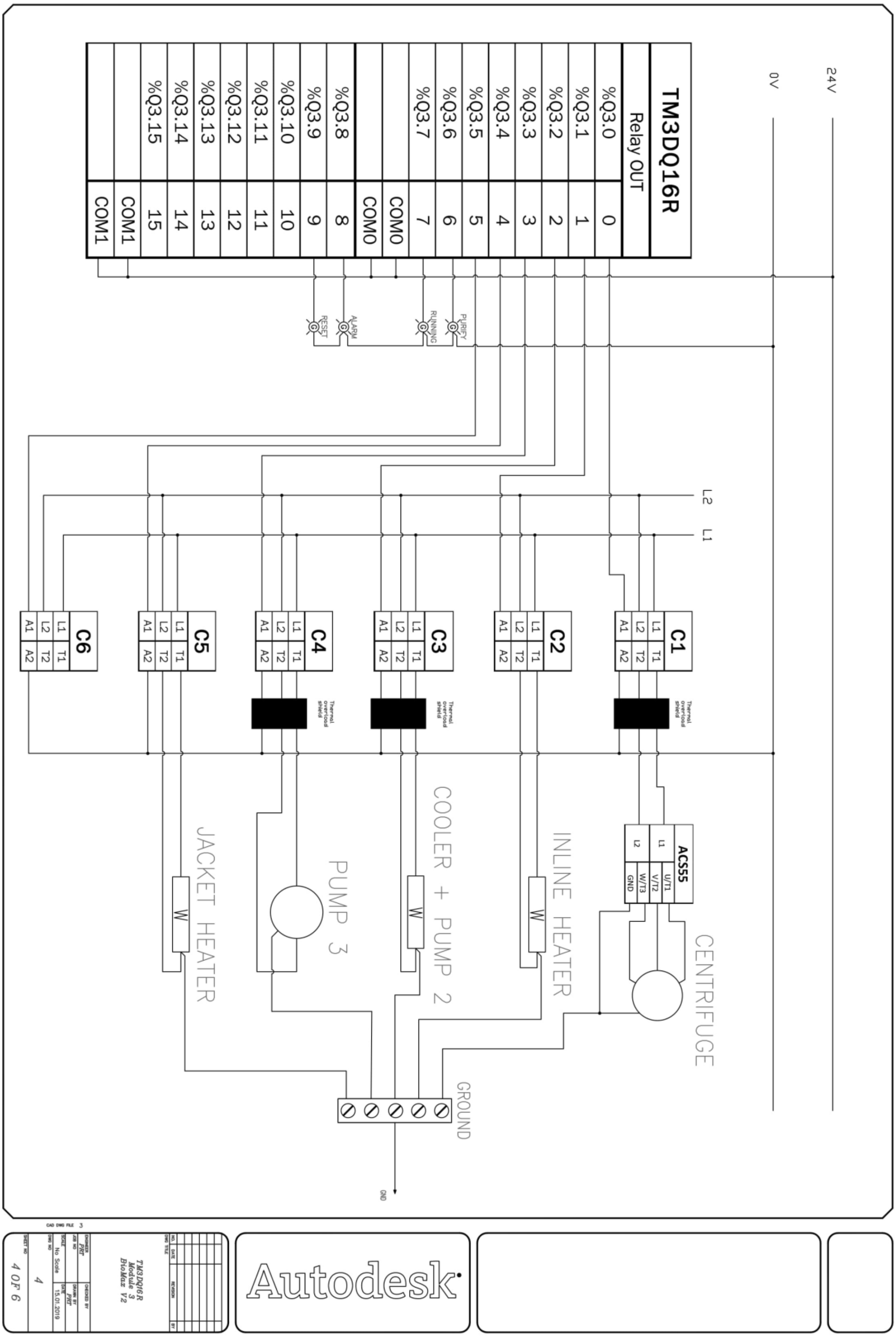
Appendix D.2: Wiring Diagram for Module 1



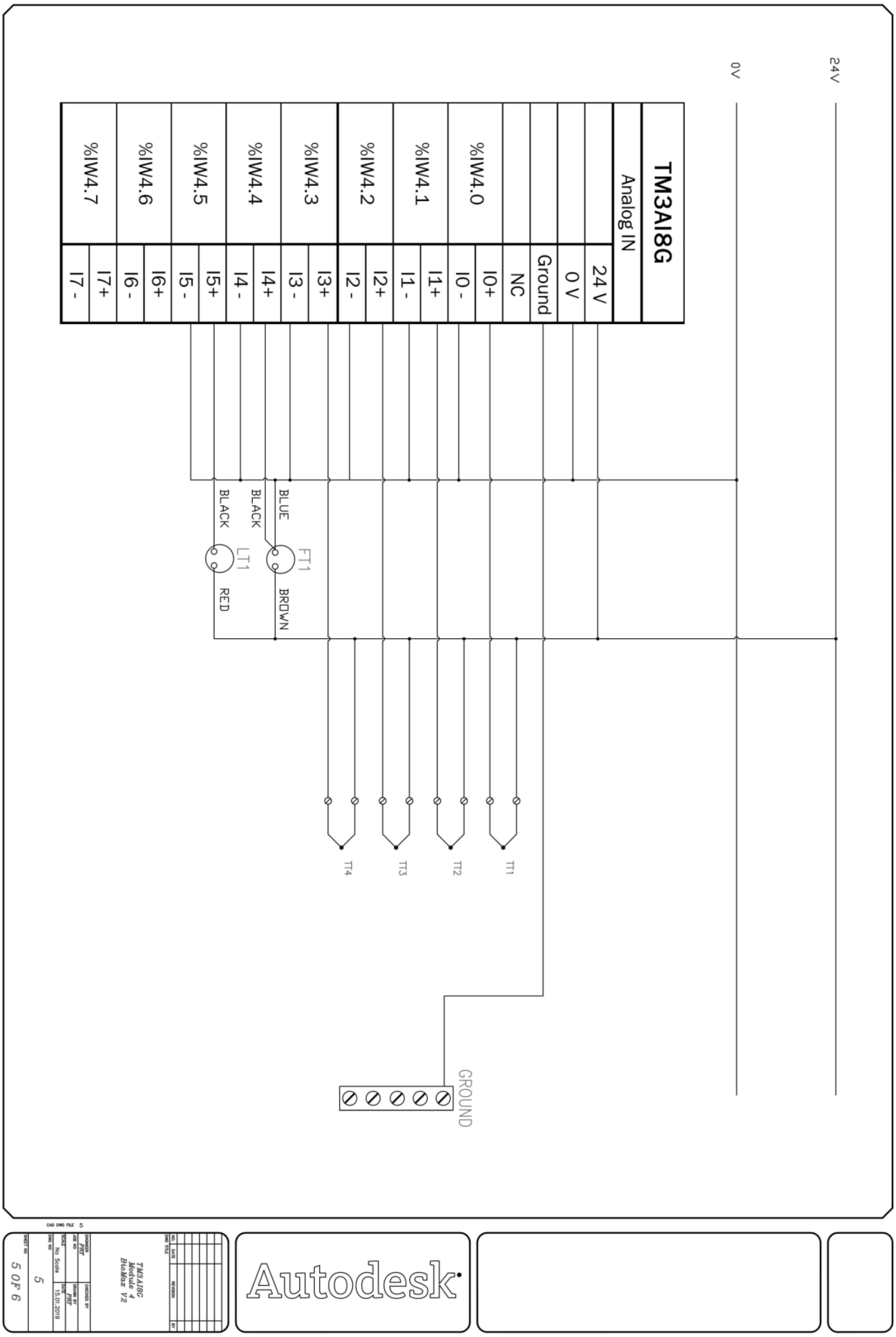
Appendix D.3: Wiring Diagram for Module 2



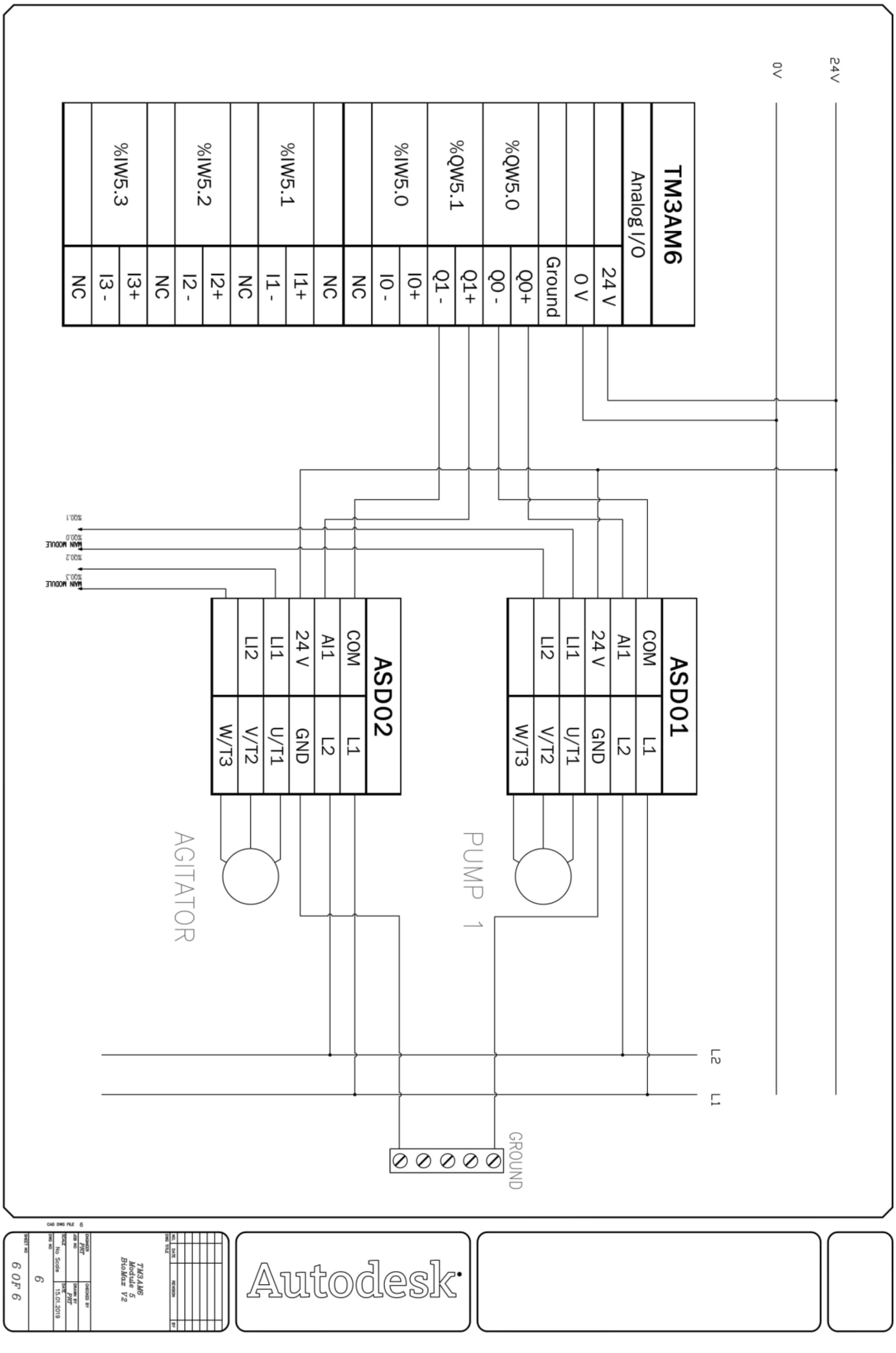
Appendix D.4: Wiring Diagram for Module 3



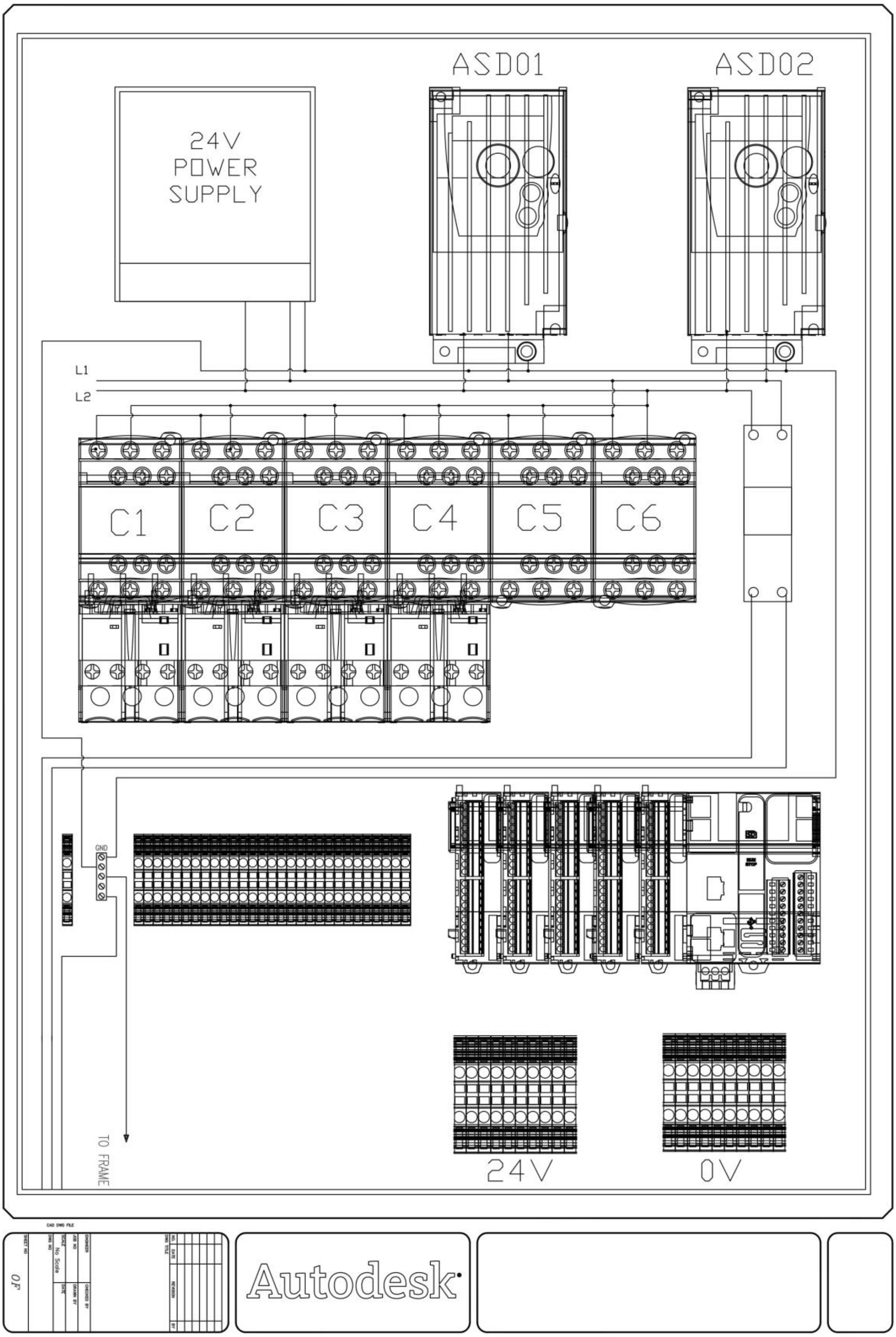
Appendix D.5: Wiring Diagram for Module 4



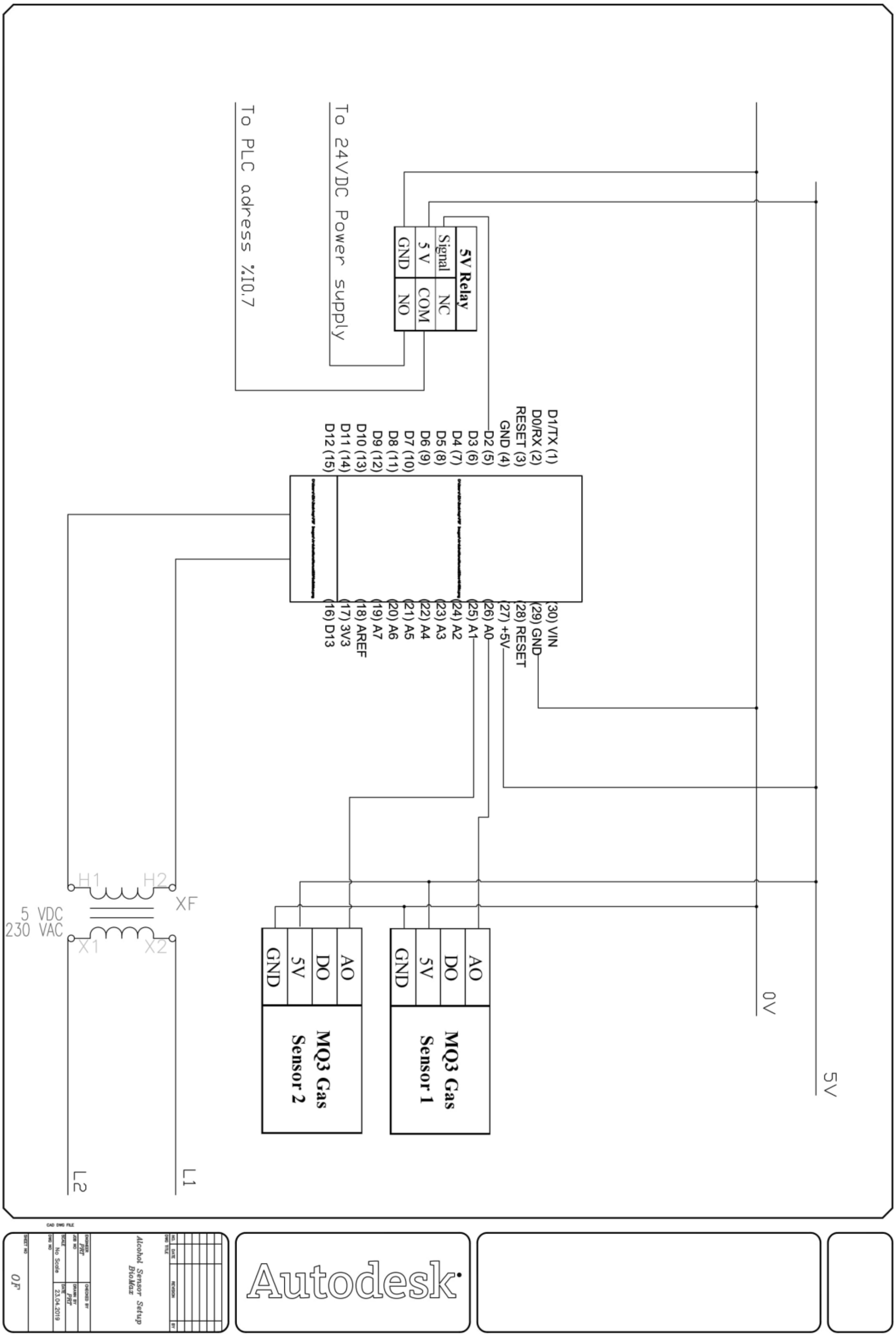
Appendix D.6: Wiring Diagram for Module 5



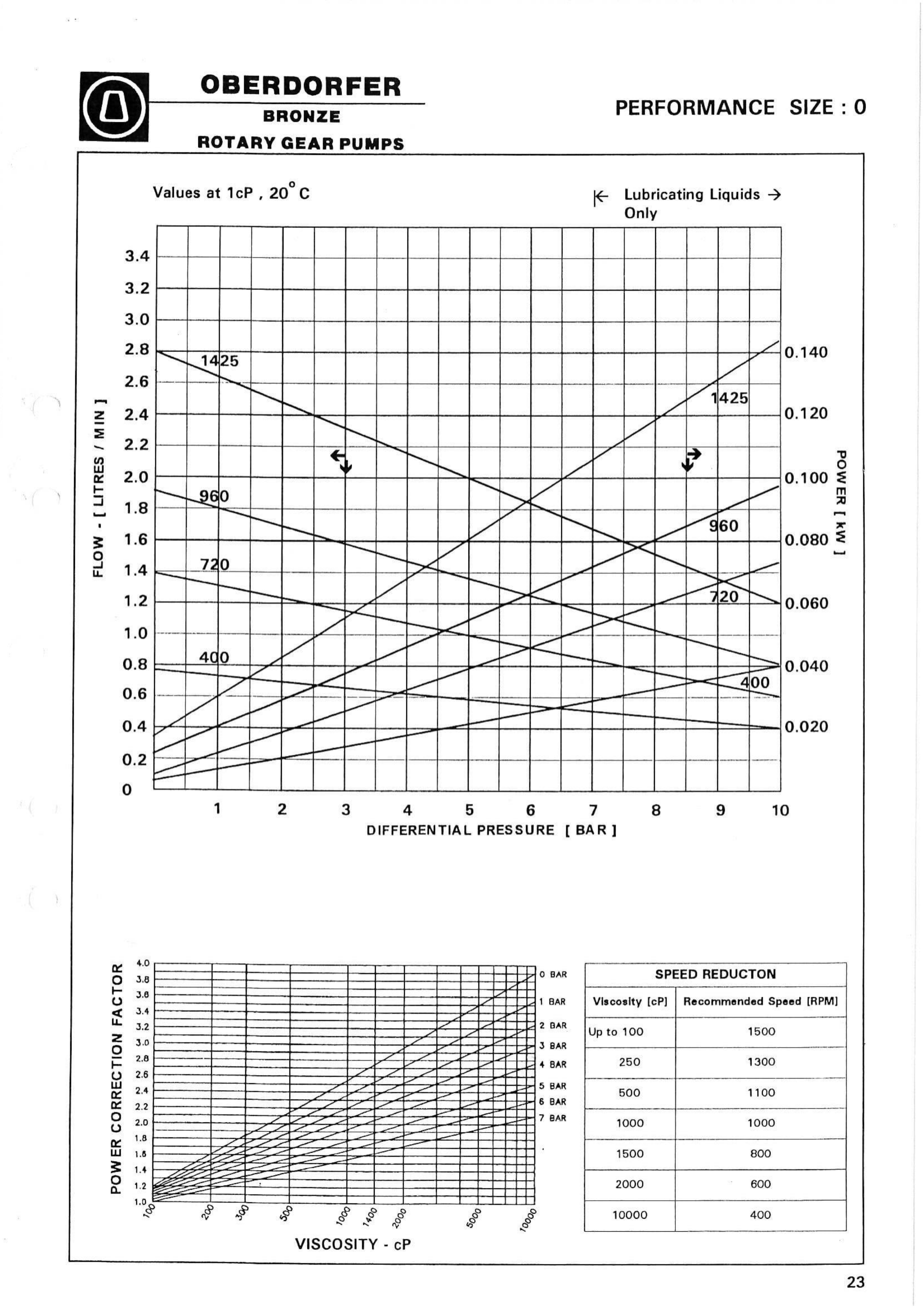
Appendix D.7: Wiring Diagram for the Electrical Cabinet



Appendix D.8: Wiring Diagram for the MQ3 Gas Sensor



Appendix E: Pump Datasheet



SPEED REDUCTON

Viscosity [cP]	Recommended Speed [RPM]
Up to 100	1500
250	1300
500	1100
1000	1000
1500	800
2000	600
10000	400

23

Appendix F: Valve Positioning

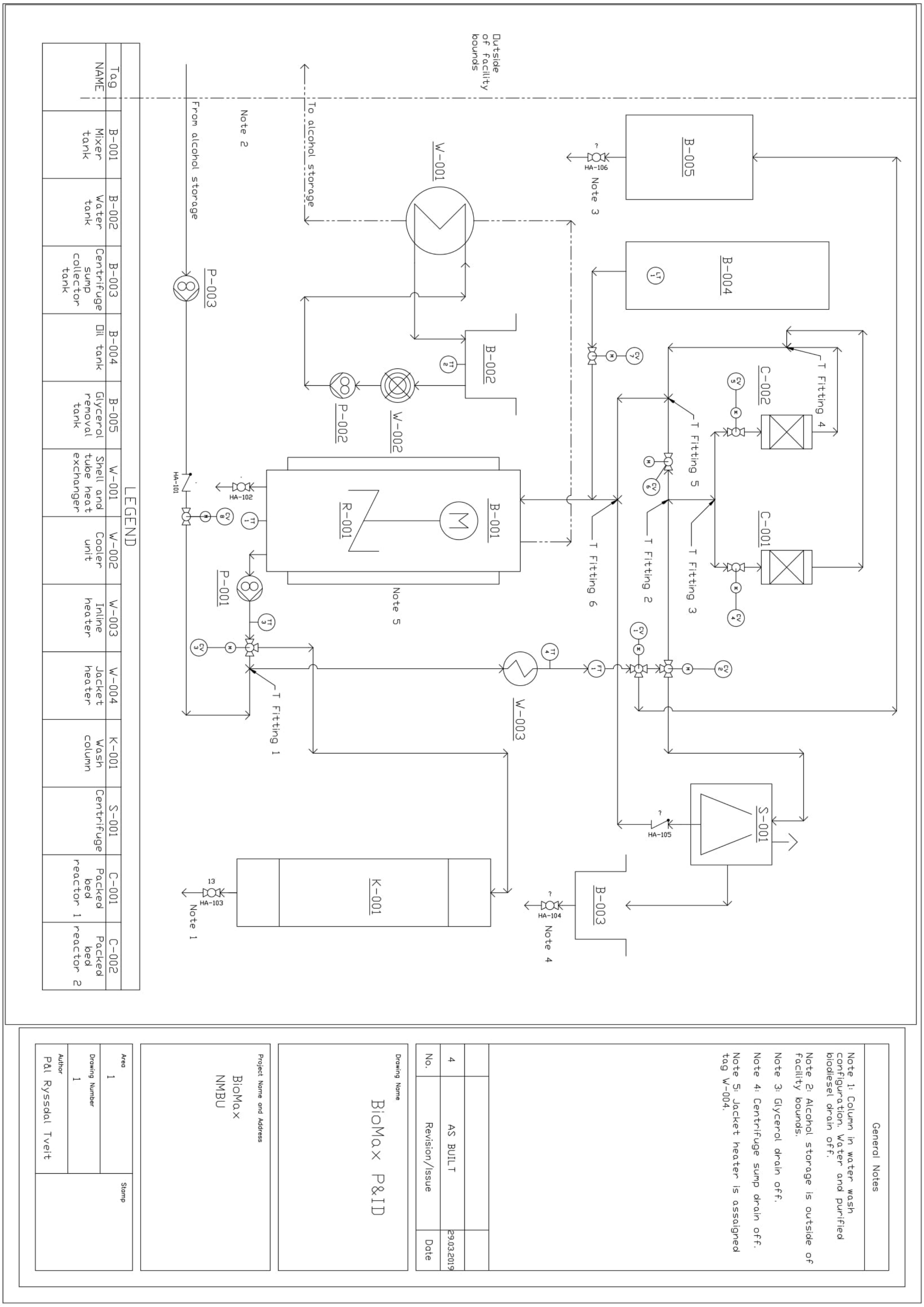
It is important for the program to know how the valves are positioned at any time, as the position of the valve will dictate where the liquid flows. It was discovered during the preliminary work that the valve positioning posed an issue. The PLC program has been programmed so that is does not allow for sending the actuator a “Close valve” and “Open valve” signal simultaneously. This complicates the control of the valve. For instance, the required steps for turning a valve from “open” to “close” position, and back to open position can be stated as such:

1. The actuator gets a “Close valve” signal from the PLC.
2. The actuator turns off once it has reached the closed position.
3. The “Close valve” signal from the PLC is turned off.
4. The actuator gets a “Open valve” signal from the PLC.
5. The actuator turns off once it has reached the open position.
6. The “Open valve” signal from the PLC is turned off.

The issue is when turning off the signal from the PLC. During the preliminary work, this signal was turned off by an on-delay timer, as it was measured that the servo moves at a speed of approximately 7.5 degrees per second. Although somewhat functioning, this method proved to be complicated and somewhat unreliable.

For this thesis, an extra input module was added to the PLC. This makes it possible to use the position switches on the actuator to turn off the signal from the PLC. As can be seen in the figure to the right, the valves have four lobes that are listed from the top. The first and third lobe switches off the servomotor after reaching the closed or open position, respectively. Similarly, the second and fourth lobes are used for position indication. Because of the difficulty in positioning the corresponding lobes so that they reach the switches at the same time, the positioning lobe is turned slightly more towards the rotation direction. When the input signal from the position switch reads as high, a 5-second timer is turned on to allow the servo to finish its rotation. The position switch indicates whether the valve is in open or closed position. The input signal from the position switches is used to check that the valves have reached the intended position before a new task is performed.

Appendix G: Piping and Instrumentation Diagram (P&ID)



LEGEND

Tag	NAME
B-001	Mixer tank
B-002	Water tank
B-003	Centrifuge sump collector tank
B-004	Oil tank
B-005	Glycerol removal tank
W-001	Shell and tube heat exchanger
W-002	Cooler unit
W-003	Inline heater
W-004	Jacket heater
K-001	Wash column
S-001	Centrifuge
C-001	Packed bed reactor 1
C-002	Packed bed reactor 2

General Notes

Note 1: Column in water wash configuration. Water and purified biodiesel drain off.

Note 2: Alcohol storage is outside of facility bounds.

Note 3: Glycerol drain off.

Note 4: Centrifuge sump drain off.

Note 5: Jacket heater is assaigned tag W-004.

No.	Revision/Issue	Date
4	AS BUILT	29.03.2019

Drawing Name
BioMax P&ID

Project Name and Address
BioMax
NMBU

Area
1

Drawing Number
1

Author
Pål Ryssdal Tveit

Stamp

Appendix H.1: Mass balance for Esterification of UCO

For the esterification, the UCO from Sanford et al. [25] will be used. The UCO feedstock had a FFA content of 2.72 wt%, a moisture content of 0.242wt% and 0.20 wt% of solids. Before the UCO can be transesterified, the moisture and solids must be removed. This will be done by heating the UCO to 70°C and adding it to the centrifuge. The loss of feedstock through the sumpt during centrifuge shutdown will not be included. 8 kg of UCO will be used for the calculation. The amount of water and solids can be calculated from the weight of oil:

$$m_{H_2O} = m_{UCO} \cdot 0.242wt\% = 8kg \cdot\ 0.242wt\% = 0.0194kg$$

$$m_{solids} = m_{UCO} \cdot 0.20wt\% = 0.0160kg$$

After the centrifugation, the moisture and solids are assumed to be completely removed. The remaining UCO will consist of TAG (Triolein) and FFA (Oleic acid). The amount of TAG and FFA can then be calculated:

$$m_{FFA} = m_{UCO} \cdot 2.72wt\% = 8kg \cdot 2.72wt\% = 0.2176kg$$

$$m_{TAG} = m_{UCO} - \left(m_{H_2O} + m_{solids} + m_{FFA}\right)$$

$$m_{TAG} = 8kg - (0.0194 + 0.0160 + 0.2176)kg = 7.747kg$$

An alcohol to oil ratio of 6:1 is used. The amount ethanol can then be calculated from the combined mass of the FFA and the TAG:

$M_{TAG} = 885.4g/mol$

$$n_{TAG} = \frac{m_{TAG}}{M_{TAG}} = \frac{7747g}{885.4g/mol} = 8.750mol$$

$M_{FFA} = 282.5g/mol$

$$n_{FFA} = \frac{m_{FFA}}{M_{FFA}} = \frac{218g}{282.5g/mol} = 0.772mol$$

$$n_{oil} = n_{TAG} + n_{FFA} = 8.750mol + 0.772mol = 9.522mol$$

$$n_{ethanol} = n_{oil} \cdot 6 = 9.522mol \cdot 6 = 57.132mol$$

$M_{ethanol} = 46.1\ g/mol$

$$m_{ethanol} = n_{ethanol} \cdot M_{ethanol} = 57.132mol \cdot 46.1\ g/mol = 2634g$$

The reaction equation for the esterification of FFA can then be calculated as seen in table H1

Table H1: Mass Balance for Esterification of Oleic Acid

Molar Ratio	1	1	$\leftrightarrow$	1	1
Compound	Oleic Acid	Ethanol		Ethyl Oleate	Water
Molecular Formula	$C_{18}H_{34}O_2$	C_2H_5OH		$C_{20}H_{38}O_2$	H_2O
C (12.011 g/mol)	18	2		19	3
H (1.008 g/mol)	34	6		36	8
O (15.999 g/mol)	2	1		2	3
Molecular weight (g/mol)	282.5	46.1		310.5	18.0
Amount (mol)	0.772	0.772		0.772	0.772
Mass (g)	218	36		240	14

After the esterification, the water will be removed from the reaction mixture by adsorbents (zeolites). The zeolites are able to adsorb 20% of their own weight in water.

$$m_{Zeolites} = \frac{m_{H_2O}}{0.2} = \frac{14g}{0.2} = 70g$$

The minimum amount of added adsorbents is 70 grams. After the esterification the reaction mixture will consist of:

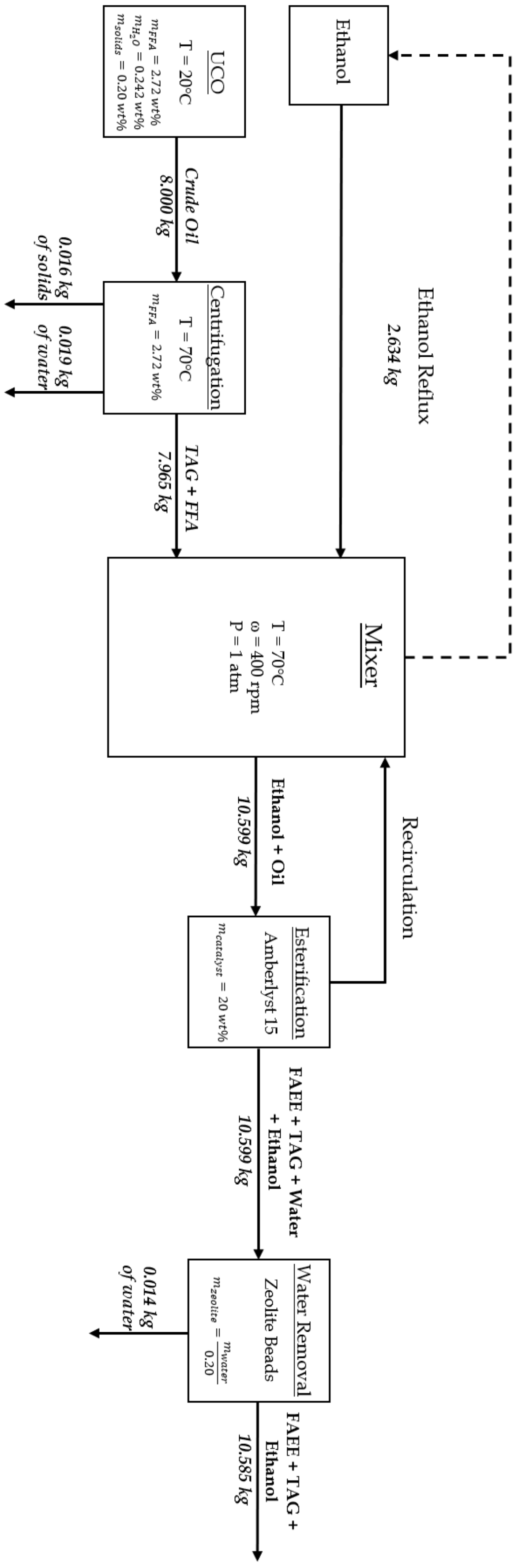
$m_{TAG} = 7747g$

$m_{FAEE} = 240g$

$m_{etanol} = 2634g - 36g = 2598g$

$m_{total} = m_{TAG} + m_{FAEE} + m_{ethanol} = 7747g + 240g + 2598g = 10585g$

Appendix H.2: PFD for Esterification of UCO



Appendix I.1: Mass balance for Transesterification of UCO

The pre-treated and esterified feedstock from appendix H.1 will be used for the following calculations. The ethanol from the esterification process will be used and no more will be added for the transesterification process.

$m_{TAG} = 7747g, n_{TAG} = 8.750mol$

$m_{FAEE} = 240g, n_{FAEE} = 0.773mol$

$m_{etanol} = 2634g - 36g = 2598g, n_{ethanol} = 56.356\,mol$

Table H1: Mass Balance for Transesterification of Triolein

Molar Ratio	1	3	$\leftrightarrow$	3	1
Compound	Triolein	Ethanol		Ethyl Oleate	Glycerol
Molecular Formula	$C_{57}H_{104}O_6$	C_2H_5OH		$C_{20}H_{38}O_2$	$C_3H_8O_3$
C (12.011 g/mol)	57	2		20	3
H (1.008 g/mol)	104	6		38	8
O (15.999 g/mol)	6	1		2	3
Molecular weight (g/mol)	885.4	46.1		310.5	92.1
Amount (mol)	8.75	26.25		26.25	8.75
Mass (g)	7747	1210		8151	806

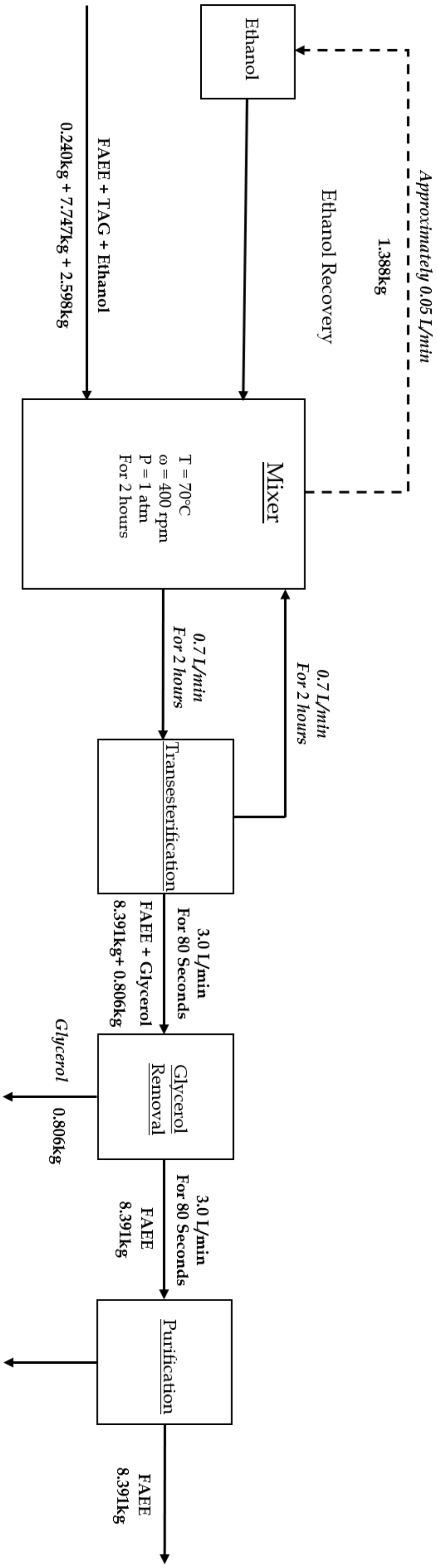
After esterification the reaction mixture will consist of:

$m_{FAEE} = 240g + 8151g = 8391g$

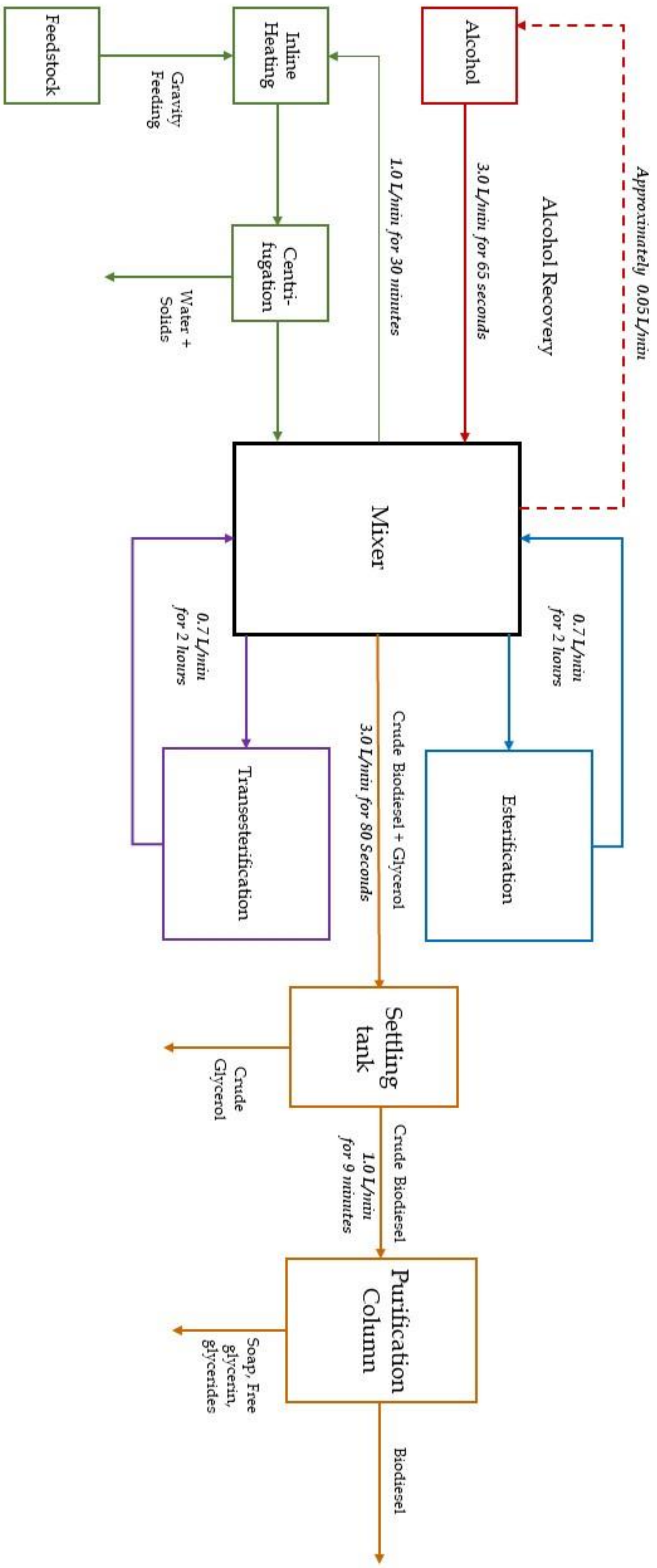
$m_{ethanol} = 2598g - 1210g = 1388g$

The ethanol is then evaporated, and the FAEE is added to the purification column.

Appendix I.2: PFD for Transesterification of UCO



Appendix J: PFD for "BioMax"



Appendix K: Mass Balance for the First and Second Test

The amount of added sunflower oil was 8kg for the first and second test. A 6:1 molar ratio of alcohol of oil was utilized during the tests. A simplification is made by assuming that the fatty acid profile of the sunflower oil is 100% oleic acid.

$m_{TAG} = 8000g,\ M_{TAG} = 885.4g/mol,\ n_{TAG} = 9.035mol$

Table K1: Mass Balance for Transesterification of Triolein

Molar Ratio	1	3	$\leftrightarrow$	3	1
Compound	Triolein	Ethanol		Ethyl Oleate	Glycerol
Molecular Formula	$C_{57}H_{104}O_6$	C_2H_5OH		$C_{20}H_{38}O_2$	$C_3H_8O_3$
C (12.011 g/mol)	57	2		20	3
H (1.008 g/mol)	104	6		38	8
O (15.999 g/mol)	6	1		2	3
Molecular weight (g/mol)	885.4	46.1		310.5	92.1
Amount (mol)	9.035	27.105		27.105	9.035
Mass (g)	8000	1250		8416	832

The total amount of added ethanol will be 2.5 kg.

After esterification the reaction mixture will consist of:

Appendix L.1: Ladder Program for the "Start" Master Task

Below, the Master Tasks for each major task of the control system is presented. The program references to outputs such as valves, heaters and pumps. The logic of these will not be presented, due to limited space.

13 - Initial Positioning

Master Task

Rung0 - START AND SET VALVES

Comment: `Set valves per table 6.5`

Legend:

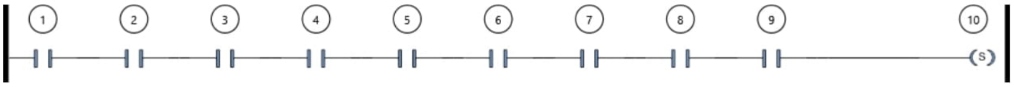
1	%M16

Variables used:

%M1	MSTARTB	Start button has been pushed
%M16	M_IP001	Set valves HIGH

Rung1 - CHECK VALVE POSITIONING

Comment: `Check that valves are in posistion per table 6.5`



Legend:

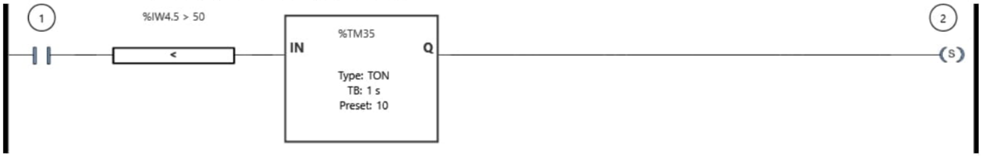
1	%M16
2	%M42
3	%M44
4	%M45
5	%M47
6	%M49
7	%M52
8	%M53
9	%M55
10	%M17

Variables used:

%M16	M_IP001	Set valves HIGH
%M17	M_IP002	Cut off HIGH signal
%M42	MCV1_O_I	CV1 is Open per Valve Tracking
%M44	MCV2_O_I	DIFFERENT FROM INDICATOR ON VALVE
%M45	MCV3_C_I	CV3 is Closed per Valve Tracking
%M47	MCV4_C_I	CV4 is Closed per Valve Tracking
%M49	MCV5_C_I	CV5 is Closed per Valve Tracking
%M52	MCV6_O_I	
%M53	MCV7_C_I	CV7 is Closed per Valve Tracking
%M55	MCV8_C_I	CV8 is Closed per Valve Tracking

Rung2 - CHECK LEVEL IN OIL TANK

Comment: Check that there is a minimum amount of oil in the oil tank and wait for 10 seconds before opening CV7



Legend:

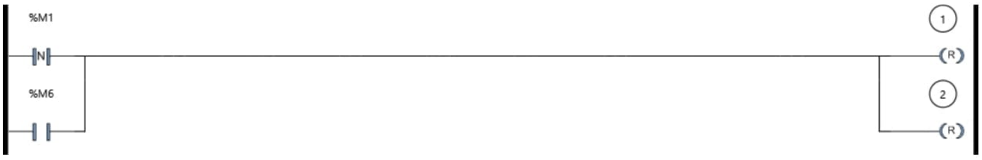
1 %M17

2 %M18

Variables used:

%IW4.5	LT1	Leveltransmitter in oiltank. Measuring unit is mmH20.
%M17	M_IP002	Cut off HIGH signal
%M18	M_IP003	There is oil in the oil tank
%TM35		Delay after oil has been added to the tank

Rung3 - RESET



Legend:

1 %M18

2 %M17

Variables used:

%M1	MSTARTB	Start button has been pushed
%M6	MRESET	The reset button has been pushed
%M17	M_IP002	Cut off HIGH signal
%M18	M_IP003	There is oil in the oil tank

14 - Add Oil

Master Task

Rung0 - INITIAL LEVEL MEASUREMENT

Comment: Log the level in the oil tank at the start of the program

Legend:

1 %M18

2 %MW2 := %IW4.5

Variables used:

%IW4.5	LT1	Leveltransmitter in oiltank. Measuring unit is mmH20.
%M18	M_IP003	There is oil in the oil tank
%MW2		

Rung1 - OPEN CV7

Comment: Open CV7 to allow for oil being added to the mixer

Legend:

1 %M18

2 %M10

Variables used:

%M10	M_AO001	Set CV7 as HIGH during Add Oil
%M18	M_IP003	There is oil in the oil tank

Rung2 - REAL TIME MEASUREMENT

Comment: `Subtract the real-time measurement from the initial level measurement until the difference is a set level`

Legend:

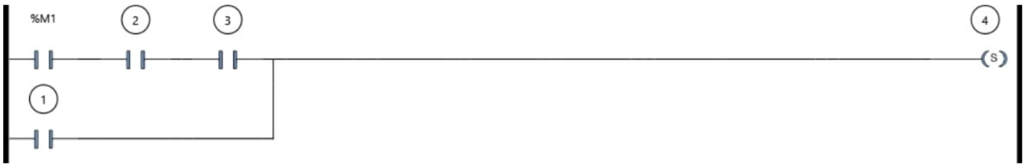
1	%M18
2	%MW2 - %IW4.5 = 205
3	%M11

Variables used:

%IW4.5	LT1	Leveltransmitter in oiltank. Measuring unit is mmH20.
%M11	M_AO002	Cut off HIGH signal from add oil and CLOSE CV7
%M18	M_IP003	There is oil in the oil tank
%MW2		

Rung3 - END OF PROGRAM

Comment: `Program is finished when cv7 is closed`



Legend:

1	%M58
2	%M11
3	%M53
4	%M15

Variables used:

%M1	MSTARTB	Start button has been pushed
%M11	M_AO002	Cut off HIGH signal from add oil and CLOSE CV7
%M15	M_AO003	ADD OIL DONE
%M53	MCV7_C_I	CV7 is Closed per Valve Tracking
%M58	AO_BYPASS	

Rung4 - RESET MEMORIES

Comment: `Reset set memories used in the POU`

Legend:

1	%M11
2	%M15
3	%M10

Variables used:

%M1	MSTARTB	Start button has been pushed
%M6	MRESET	The reset button has been pushed
%M10	M_AO001	Set CV7 as HIGH during Add Oil
%M11	M_AO002	Cut off HIGH signal from add oil and CLOSE CV7
%M15	M_AO003	ADD OIL DONE

Appendix L.2: Ladder Program for the “Heating” Master Task

15 - Heating

Master Task

Rung0 - START AND SET VALVES

Comment: Set valves per table 6.6

Legend:

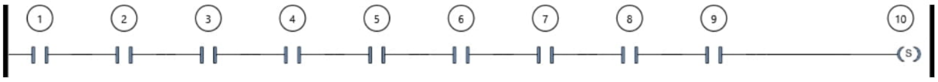
1	%M15
2	%M23
3	%M19

Variables used:

%M2	MHEATING	Heating button has been pushed
%M7	M_C002	TT2 HIGHER HIGHER THAN 16C
%M15	M_AO003	ADD OIL DONE
%M19	M_H001	Set valves HIGH
%M23	M_C004	COOLING ON

Rung1 - CHECK VALVE POSITIONING

Comment: Check valves per table 6.6



Legend:

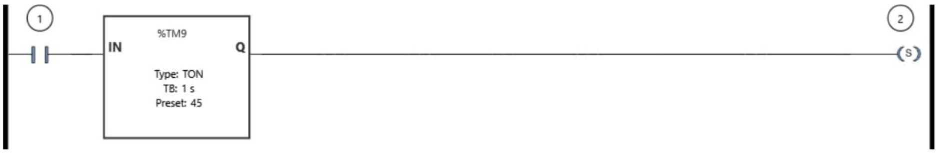
1	%M19
2	%M42
3	%M44
4	%M45
5	%M47
6	%M49
7	%M52
8	%M53
9	%M55
10	%M57

Variables used:

%M19	M_H001	Set valves HIGH
%M42	MCV1_O_I	CV1 is Open per Valve Tracking
%M44	MCV2_O_I	DIFFERENT FROM INDICATOR ON VALVE
%M45	MCV3_C_I	CV3 is Closed per Valve Tracking
%M47	MCV4_C_I	CV4 is Closed per Valve Tracking
%M49	MCV5_C_I	CV5 is Closed per Valve Tracking
%M52	MCV6_O_I	
%M53	MCV7_C_I	CV7 is Closed per Valve Tracking
%M55	MCV8_C_I	CV8 is Closed per Valve Tracking
%M57	M_H008	Cut off HIGH signal

Rung2 - TURN ON PIPE HEATER

Comment: Turn on the inline heater after the liquid has filled the inline heater



Legend:

1	%M57
2	%M24

Variables used:

%M24	M_H005	turn on inline heater
%M57	M_H008	Cut off HIGH signal
%TM9		Wait for liquid to fill inline heater

Rung3 - HIGH TEMPERATURE

Comment: The high setpoint in the on/off regulator

Legend:

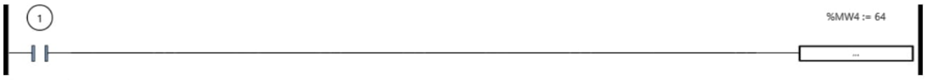
1 %M57

Variables used:

%M57	M_H008	Cut off HIGH signal
%MW3		High temperature

Rung4 - LOW TEMPERATURE

Comment: The low temperature in the on/off regulator



Legend:

1 %M57

Variables used:

%M57	M_H008	Cut off HIGH signal
%MW4		Low temperature

Rung5 - TT1 HIGH TEMPERATURE CHECK

Legend:

1 %M57

2 %IW4.0 > %MW3

3 %M20

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M20	M_H002	TT1 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW3		High temperature

Rung6 - TT3 HIGH TEMPERATURE CHECK

Legend:

1 %M57

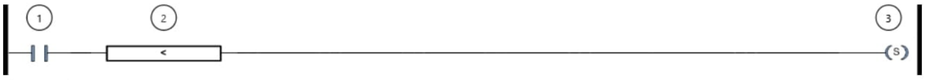
2 %IW4.2 > %MW3

3 %M21

Variables used:

%IW4.2	TT3	Process line temperature. Measurement unit is Celsius.
%M21	M_H003	TT3 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW3		High temperature

Rung7 - TT4 HIGH TEMPERATURE CHECK



Legend:

1 %M57

2 %IW4.3 > %MW3

3 %M22

Variables used:

%IW4.3	TT4	Heater outlet temperature. Measurement unit is Celsius.
%M22	M_H004	TT4 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW3		High temperature

Rung8 - TT1 LOW TEMPERATURE CHECK

Legend:

1 %M57

2 %IW4.0 < %MW4

3 %M20

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M20	M_H002	TT1 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW4		Low temperature

Rung9 - TT3 LOW TEMPERATURE CHECK

Legend:

1 %M57

2 %IW4.2 < %MW4

3 %M21

Variables used:

%IW4.2	TT3	Process line temperature. Measurement unit is Celsius.
%M21	M_H003	TT3 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW4		Low temperature

Rung10 - TT4 LOW TEMPERATURE CHECK

Legend:

1 %M57

2 %IW4.3 < %MW4

3 %M22

Variables used:

%IW4.3	TT4	Heater outlet temperature. Measurement unit is Celsius.
%M22	M_H004	TT4 TOO HOT
%M57	M_H008	Cut off HIGH signal
%MW4		Low temperature

Rung11 - TIMER

Comment: Amount of time for heating the oil

Legend:

1 %M57

2 %M25

Variables used:

%M25	M_H006	HEATING DONE
%M57	M_H008	Cut off HIGH signal
%TM10		heating timer

Rung12 - RESET

Comment: Reset all set memories in the rung

Legend:

1	%M57
2	%M20
3	%M21
4	%M22
5	%M24
6	%M25

Variables used:

%M1	MSTARTB	Start button has been pushed
%M2	MHEATING	Heating button has been pushed
%M6	MRESET	The reset button has been pushed
%M20	M_H002	TT1 TOO HOT
%M21	M_H003	TT3 TOO HOT
%M22	M_H004	TT4 TOO HOT
%M24	M_H005	turn on inline heater
%M25	M_H006	HEATING DONE
%M57	M_H008	Cut off HIGH signal

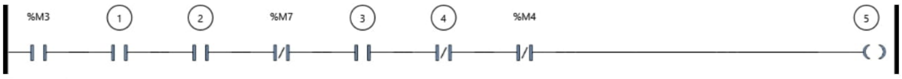
Appendix L.3: Ladder Program for the "Ester" Master Task

16 - Esterification

Master Task

Rung0 - START AND SET VALVES

Comment: Set valves per Table 6.7



Legend:

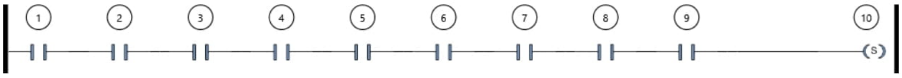
1	%M25
2	%M15
3	%M23
4	%M27
5	%M31

Variables used:

%M3	MESTER	Ester button has been pushed
%M4	MTRESTER	Trester button has been pushed
%M7	M_C002	TT2 HIGHER HIGHER THAN 16C
%M15	M_AO003	ADD OIL DONE
%M23	M_C004	COOLING ON
%M25	M_H006	HEATING DONE
%M27	M_H007	PIPE HEATER IS ON
%M31	M_E001	Set valves HIGH

Rung1 - CHECK VALVE POSITIONING

Comment: Check valves per Table 6.7



Legend:

1	%M31
2	%M42
3	%M44
4	%M45
5	%M47
6	%M50
7	%M51
8	%M53
9	%M56
10	%M32

Variables used:

%M31	M_E001	Set valves HIGH
%M32	M_E002	Cut off HIGH signal
%M42	MCV1_O_I	CV1 is Open per Valve Tracking
%M44	MCV2_O_I	DIFFERENT FROM INDICATOR ON VALVE
%M45	MCV3_C_I	CV3 is Closed per Valve Tracking
%M47	MCV4_C_I	CV4 is Closed per Valve Tracking
%M50	MCV5_O_I	
%M51	MCV6_C_I	CV6 is Closed per Valve Tracking
%M53	MCV7_C_I	CV7 is Closed per Valve Tracking
%M56	MCV8_O_I	CV8 is Open per Valve Tracking

Rung2 - ADD ALCOHOL

Comment: Alcohol can be added if esterification is started

Legend:

1	%M32
2	%M89
3	%M100

Variables used:

%M32	M_E002	Cut off HIGH signal
%M89	M_M001	Start alcohol pump manually for esterification
%M100	M_E008	

Rung3 - ADD ALCOHOL DONE

Comment: Signifies that the Add alcohol task is finished

Legend:

1	%M89
2	%M101

Variables used:

%M89	M_M001	Start alcohol pump manually for esterification
%M101	M_E009	

Rung4 - TIMER

Comment: Time for circulation during the esterification process

Legend:

1	%M101
2	%M61

Variables used:

%M61	M_E007	Esterification Done
%M101	M_E009	
%TM12		esterification timer

Rung5 - EMPTY REACTOR

Comment: Wait for Open CV5 and CV6 and empty into mixer tank

Legend:

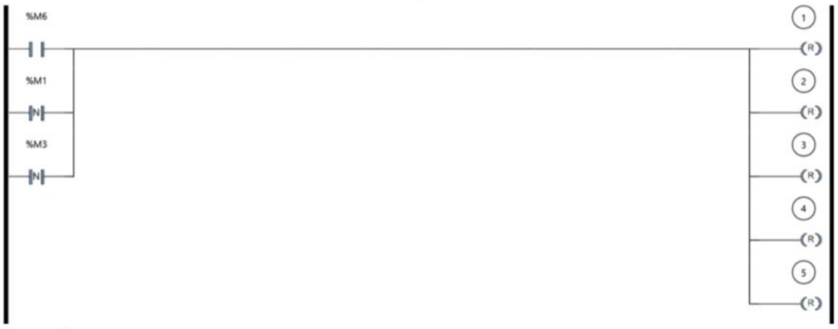
1	%M61
2	%M30

Variables used:

%M30	M_E003	Esterification is DONE
%M61	M_E007	Esterification Done
%TM18		empty reactor 2 timer

Rung6 - RESET

Comment: Reset all set memories in the rung



Legend:

1	%M31
2	%M32
3	%M30
4	%M38
5	%M101

Variables used:

%M1	MSTARTB	Start button has been pushed
%M3	MESTER	Ester button has been pushed
%M6	MRESET	The reset button has been pushed
%M30	M_E003	Esterification is DONE
%M31	M_E001	Set valves HIGH
%M32	M_E002	Cut off HIGH signal
%M38	M_E005	
%M101	M_E009	

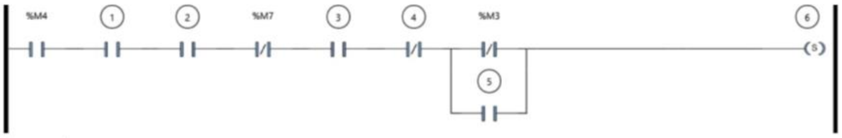
Appendix L.4: Ladder Program for the “Trester” Master Task

17 - Transesterification

Master Task

Rung0 - START AND SET VALVES

Comment: Set valves per Table 6.8



Legend:

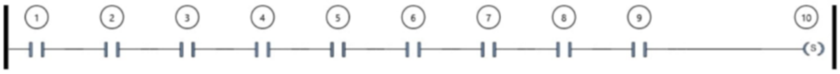
1	%M25
2	%M15
3	%M23
4	%M27
5	%M30
6	%M26

Variables used:

%M3	MESTER	Ester button has been pushed
%M4	MTRESTER	Trester button has been pushed
%M7	M_C002	TT2 HIGHER HIGHER THAN 16C
%M15	M_A0003	ADD OIL DONE
%M23	M_C004	COOLING ON
%M25	M_H006	HEATING DONE
%M26	M_T001	Set valves HIGH
%M27	M_H007	PIPE HEATER IS ON
%M30	M_E003	Esterification is DONE

Rung1 - START AND CHECK VALVES

Comment: Check valves per Table 6.8



Legend:

1	%M26
2	%M42
3	%M44
4	%M45
5	%M48
6	%M49
7	%M51
8	%M53
9	%M56
10	%M28

Variables used:

%M26	M_T001	Set valves HIGH
%M28	M_T002	Cut off HIGH signal
%M42	MCV1_O_I	CV1 is Open per Valve Tracking
%M44	MCV2_O_I	DIFFERENT FROM INDICATOR ON VALVE
%M45	MCV3_C_I	CV3 is Closed per Valve Tracking
%M48	MCV4_O_I	CV4 is Open per Valve Tracking
%M49	MCV5_C_I	CV5 is Closed per Valve Tracking
%M51	MCV6_C_I	CV6 is Closed per Valve Tracking
%M53	MCV7_C_I	CV7 is Closed per Valve Tracking
%M56	MCV8_O_I	CV8 is Open per Valve Tracking

Rung2 - ADD ALCOHOL

Comment: Alcohol can be added if transesterification is started

Legend:

1	%M28
2	%M90
3	%M98

Variables used:

%M28	M_T002	Cut off HIGH signal
%M90	M_M002	Start alcohol pump manually for transesterificatio
%M98	M_T008	

Rung3 - ADD ALCOHOL DONE

Comment: Signifies that the Add alcohol task is finished

Legend:

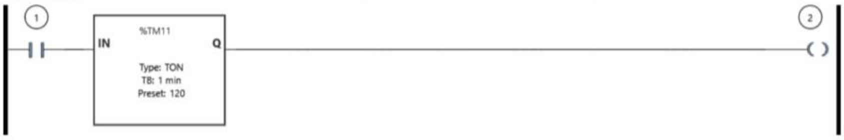
1 %M90
2 %M99

Variables used:

%M90	M_M002	Start alcohol pump manually for transesterificatio
%M99	M_T009	

Rung4 - TIMER

Comment: Time for circulation during the transesterification process



Legend:

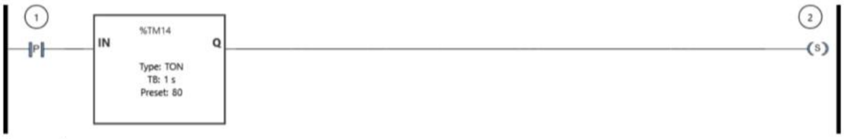
1 %M99
2 %M63

Variables used:

%M63	M_T007	Timer is DONE
%M99	M_T009	
%TM11		Transesterification timer

Rung5 - EMPTY REACTOR

Comment: Wait for Open CV4 and CV6 and empty into mixer tank



Legend:

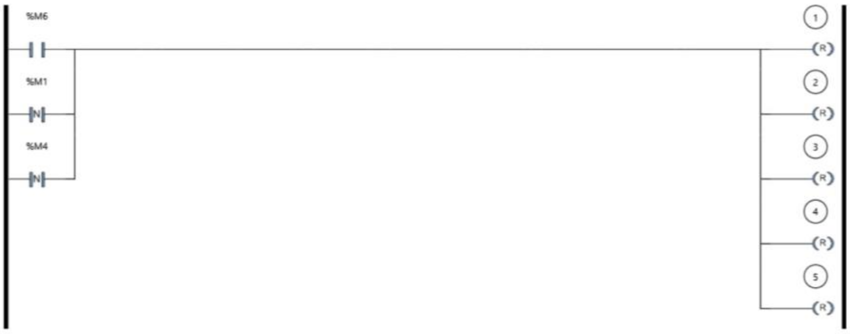
1 %M63
2 %M29

Variables used:

%M29	M_T003	Transesterification is DONE
%M63	M_T007	Timer is DONE
%TM14		

Rung6 - RESET

Comment: Reset all set memories in the POU



Legend:

1 %M26
2 %M28
3 %M29
4 %M36
5 %M99

Variables used:

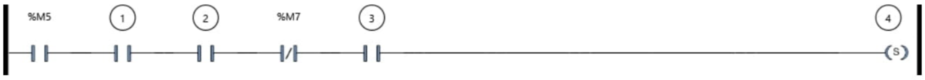
%M1	MSTARTB	Start button has been pushed
%M4	MTRESTER	Trester button has been pushed
%M6	MRESET	The reset button has been pushed
%M26	M_T001	Set valves HIGH
%M28	M_T002	Cut off HIGH signal
%M29	M_T003	Transesterification is DONE
%M36	M_T005	
%M99	M_T009	

Appendix L.5: Ladder Program for the “Purify” Master Task

18 - Purification

Master Task

Rung0 - START AND SET VALVES



Legend:

1 %M25
2 %M29
3 %M23
4 %M80

Variables used:

%M5	MPURIFY	Purify button has been pushed
%M7	M_C002	TT2 HIGHER HIGHER THAN 16C
%M23	M_C004	COOLING ON
%M25	M_H006	HEATING DONE
%M29	M_T003	Transesterification is DONE
%M80	M_P001	Set valves HIGH

Rung1 - START AND CHECK VALVES

Legend:

1 %M80
2 %M41
3 %M81

Variables used:

%M41	MCV1_C_I	
%M80	M_P001	Set valves HIGH
%M81	M_P002	Cut off High signal

Rung2 - HIGH TEMPERATURE

Comment: High temperature setting for the on/off regulator during alcohol evaporation

Legend:

1 %M81

Variables used:

%M81	M_P002	Cut off High signal
%MW5		

Rung3 - LOW TEMPERATURE

Comment: Low temperature setting for the on/off regulator during alcohol evaporation

Legend:

1 %M81

Variables used:

%M81	M_P002	Cut off High signal
%MW6		

Rung4 - TT1 HIGH TEMPERATURE CHECK

Legend:

1 %M81
2 %IW4.0 > %MW5
3 %M82

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M81	M_P002	Cut off High signal
%M82	M_P003	TT1 too high
%MW5		

Rung5 - TT3 HIGH TEMPERATURE CHECK

Legend:

1 %M81
2 %IW4.2 > %MW5
3 %M83

Variables used:

%IW4.2	TT3	Process line temperature. Measurement unit is Celsius.
%M81	M_P002	Cut off High signal
%M83	M_P004	tt3 too high
%MW5		

Rung6 - TT4 HIGH TEMPERATURE CHECK

Legend:

1 %M81
2 %IW4.3 > %MW5
3 %M84

Variables used:

%IW4.3	TT4	Heater outlet temperature. Measurement unit is Celsius.
%M81	M_P002	Cut off High signal
%M84	M_P005	tt4 too high
%MW5		

Rung7 - TT1 LOW TEMPERATURE CHECK

Legend:

1 %M57
2 %IW4.0 < %MW6
3 %M82

Variables used:

%IW4.0	TT1	Mixertank temperature. Measurement unit is Celsius.
%M57	M_H008	Cut off HIGH signal
%M82	M_P003	TT1 too high
%MW6		

Rung8 - TT3 LOW TEMPERATURE CHECK

Legend:

1	%M81
2	%IW4.2 < %MW6
3	%M83

Variables used:

%IW4.2	TT3	Process line temperature. Measurement unit is Celsius.
%M81	M_P002	Cut off High signal
%M83	M_P004	tt3 too high
%MW6		

Rung9 - TT4 LOW TEMPERATURE CHECK

Legend:

1	%M81
2	%IW4.3 < %MW6
3	%M84

Variables used:

%IW4.3	TT4	Heater outlet temperature. Measurement unit is Celsius.
%M81	M_P002	Cut off High signal
%M84	M_P005	tt4 too high
%MW6		

Rung10 - TIMER

Comment: Time for the evaporation of ethanol

Legend:

1	%M81
2	%M85

Variables used:

%M81	M_P002	Cut off High signal
%M85	M_P006	end of purification
%TM41		purification timer

Rung11 - TRANSFER TO STORAGE

Comment: Transfer the liquid to the glass container manually

Legend:

1	%M85
2	%M91
3	%M88

Variables used:

%M85	M_P006	end of purification
%M88	M_P008	Turn on pump
%M91	M_M003	

Rung12 - END OF PROGRAM

Comment: Purification is finished

Legend:

1	%M91
2	%M86

Variables used:

%M86	M_P007	Purification DONE
%M91	M_M003	

Rung13 - Reset

Comment: Reset all set memories in the POU

Legend:

1	%M80
2	%M81
3	%M82
4	%M83
5	%M84
6	%M85

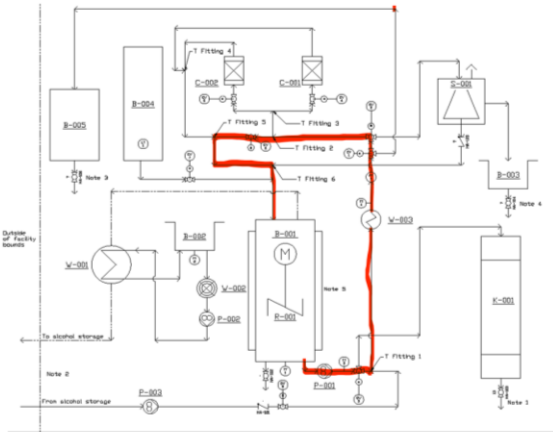
Variables used:

%M1	MSTARTB	Start button has been pushed
%M5	MPURIFY	Purify button has been pushed
%M6	MRESET	The reset button has been pushed
%M80	M_P001	Set valves HIGH
%M81	M_P002	Cut off High signal
%M82	M_P003	TT1 too high
%M83	M_P004	tt3 too high
%M84	M_P005	tt4 too high
%M85	M_P006	end of purification

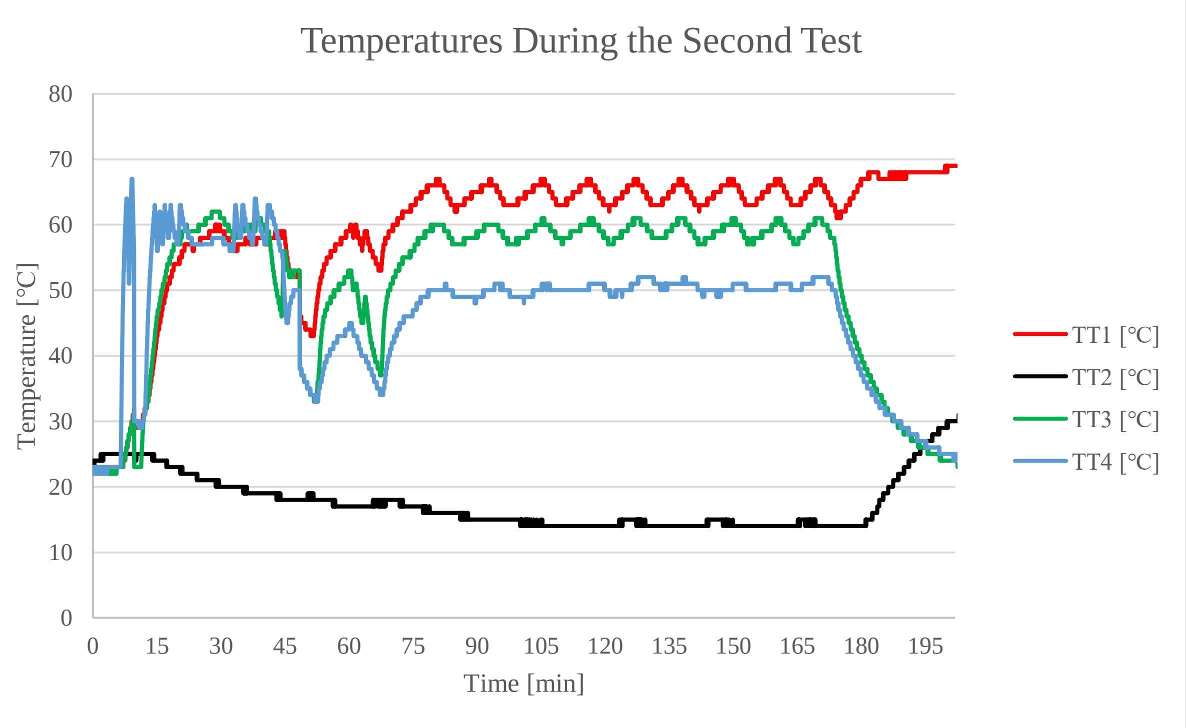
Appendix M: Alternate Valve Position During the "Heating" Master Task

If refined feedstock is used on the production line, there is no need for pre-treatment by centrifugation. In this case, the valve positioning will be as can be seen from Table M1.

Table M1: Initial Position of Valves for the "Heating" Master Task

P&ID TAG	State	From	To	Process Path
CV1	Open	W-003	CV2	
CV2	Open	CV1	S-001	
CV3	Closed	P-001	W-003	
CV4	Closed	CV2	Reactor 1	
CV5	Closed	CV2	Reactor 2	
CV6	Open	CV2	Mixer tank	
CV7	Closed	Oil tank	Mixer tank	
CV8	Closed	Methanol pump	T-fitting 3	

Appendix N: Complete Data from the Second Test

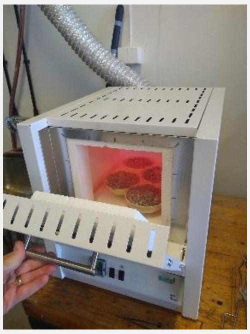
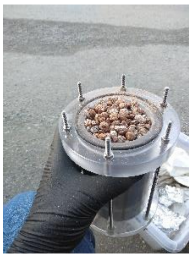


Appendix O: User Manual for "BioMax"

The user manual for the control program used during testing of the production will be presented in the following section. The user manual will only cover the transesterification of refined feedstock.

Preparatory work

Before "BioMax" is powered up, there are some task that must be performed.

#	Task	Description and Information	Picture
1	Prepare catalyst	a) This will be dependent on the type of catalyst. b) If calcium oxide is used, the catalyst will have to be calcinated before use. Crush the calcium oxide into the desired size and place it into a furnace for at least a couple of hours, depending on the catalyst size. c) When removing the calcium oxide catalyst from the furnace, be aware that the catalyst is hot. Place the catalyst directly into aluminium foil for storage	
2	Pack the reactors with catalyst	a) When packing the reactor, transfer the catalyst directly from the aluminium foil packing into the reactor. b) Fill the reactor to the top with catalyst and packing material. c) When closing the reactor, ensure that the catalyst is not crushed during tightening and sealing	
3	Set the thermostat temperature on the inline heater.	a) The cover is removed by loosening three flathead bolts on the orange casing b) The thermostat is set by rotating the potentiometer wheel to the desired temperature	
4	Set the thermostat temperature on the jacket heater	a) Set the temperature to 120℃ for effective heating, but no more than 140℃.	
5	Check that all manual valves are in the correct position	a) Upon starting "BioMax", all two-way manual valves must be in the closed position. b) The lever of the three-way manual valve must be pointing towards the mixer.	

6	Weight feedstock and alcohol	a) A molar ratio of alcohol-to-oil must be decided before weighing the inputs. b) The total added volume of reaction mixture must be between 6 and 13 liters.	
7	Power up the production line	a) Connect the orange power cord to a power supply. b) If the consumed energy is to be measured, a kWh counter must be connected to the power supply.	

The switches in figure O1 are used for turning the power supply on or off. The off position can be seen to the left and the on position to the right.

Figure O1: The power switch in the electrical cabinet

User Interface

The production line is operated by the utilizing the user interface in figure O2. The function of the pushbuttons is presented below:

- → If a pushbutton is activated, it will light up
- → If not, you do not have permission to push the button
- → The task associated with the button can be reset by pushing in the button for two consecutive seconds
- → All task can be reset by pushing the reset button for three consecutive seconds.
- → If the stop button is pushed, all 230VAC appliances will be turned off. The water pump will remain on for 30 second before it is shut off.

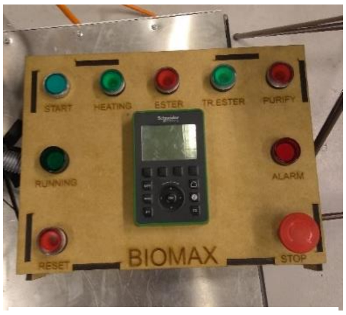


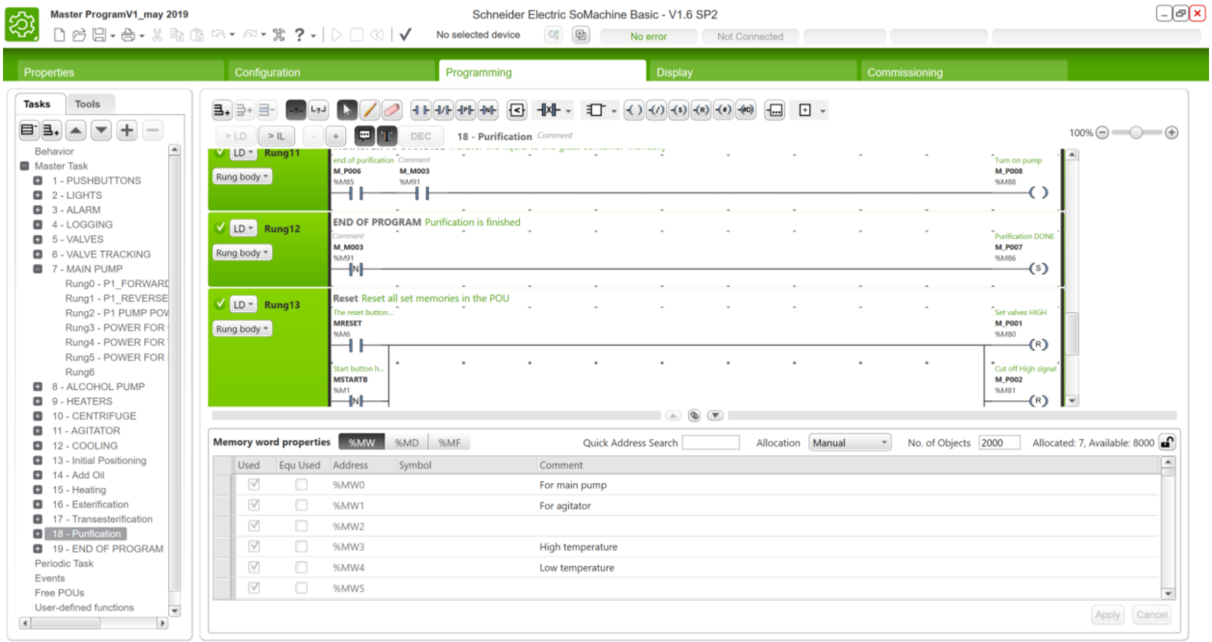
Figure O2: The user interface

Setting up Process Parameters

After this thesis, the PLC will have the correct program downloaded to it. However, the parameters for production must be set. This is done by opening the SoMachine Basic program on the stationary computer (figure O3) and performing the following instructions:

1. Once SoMachine Basic is open, press CTRL + N on the keyboard (Create new project)
2. Check that the computer is connected to the PLC and go to the commissioning tab.
3. Check that M221 is connected under “Local Devices” or “Ethernet Devices”.

4. Press Login
5. Press Controller to PC (upload)
6. Go to the save icon, and choose Save as and choose a suitable filename, preferably with a date.



Quick Address Search

Allocation: Manual

No. of Objects: 2000

Allocated: 7, Available: 8000

Used	Equ Used	Address	Symbol	Comment
☑	☐	%MW0		For main pump
☑	☐	%MW1		For agitator
☑	☐	%MW2		
☑	☐	%MW3		High temperature
☑	☐	%MW4		Low temperature
☑	☐	%MW5		

Figure O3: Program for creating a ladder program in SoMachine Basic

Set Process Temperatures

1. Go to programming and click the “15 - Heating” Master Task (Tasks).
2. In rung 3 and 4, change %MW3 and %MW4 to the upper and lower limit for the on/off temperature controller, respectively. Insert temperatures that is 1°C higher for %MW3 and 1°C lower for %MW4 than the desired setpoint.

Set Process Times

1. Go to programming and click “Tools” on the left side of the screen.
2. Go to “Software Objects” and press timers.
3. In Timer Properties, identify %TM10, %TM11, %TM12 and %TM41. Change the presets to the desired times.

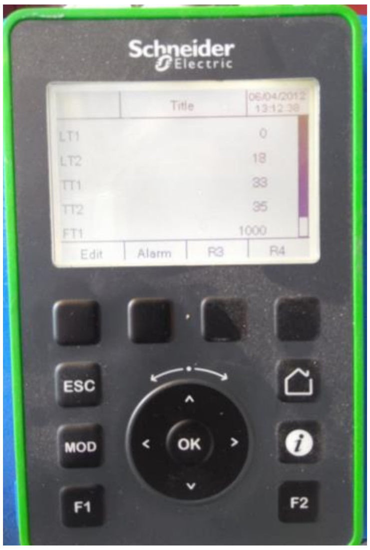
Set Process Flows

1. Go to programming and click the “7 – MAIN PUMP” Master Task (Tasks)
2. Change the value for %MW0 in rung 3 and 4 to the desired frequency on the VFD (0 to 50Hz).

Set Mixing Speeds

1. Go to programming and click the “11 – AGITATOR” Master Task (Tasks)
2. Change the value for %MW1 in rung 3, 4 and 5 to the desired frequency on the VFD (0 to 50Hz).

Operating the Production Line



During operation, the display unit can be used for monitoring. Two predefined displays have been created. By pressing the F1 key, the position of the valves and the sensor data can be seen. The F2 key is used for turning on the alcohol pump. The control panel is used to operate the production line.

The “Start” Master Task

1. Ensure that the “Stop” button is not in latched position
2. Press the “Start” button. This will start the cooling system and sett all valves in the initial position.
3. Remove the lid from the oil tank and fill oil into the tank.
4. Close the lid. The oil will be added to the tank after approximately 10 seconds.

The “Heating” Master Task

1. Press the “Heating” button

The”Trester” Master Task

1. Press the “Trester” button
2. Place the hose from the alcohol pump into the alcohol container.
3. On the display unit, press F2 and press “Add alcohol off (T)”. This will turn on the alcohol pump. The alcohol pump is turned off by pressing “Add alcohol on (T)”
4. After the alcohol container is emptied, place the hose from the heat exchanger into the empty alcohol container.
5. Seal the container and hose connection with aluminum foil.

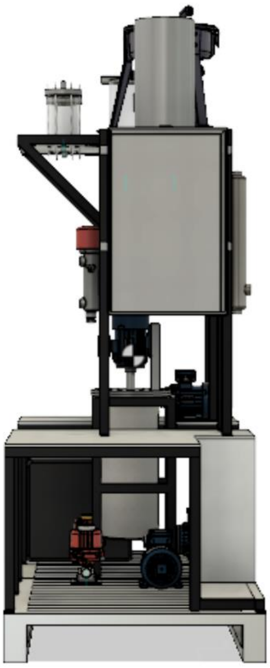
The “Purify” Master Task

1. Press the “Purify” button
2. The evaporation program will continue until 30 minutes has passed or the “Stop Evap” on the F2 display is pushed.
3. Turn on P001 to transfer liquid to the glass container

When “BioMax” is finished, all set memories will be reset automatically.

Appendix P: Centre of Gravity

The center of gravity has been calculated from the Fusion 360 model. X-direction is the longest side, y-direction is the width and z-direction is the height.



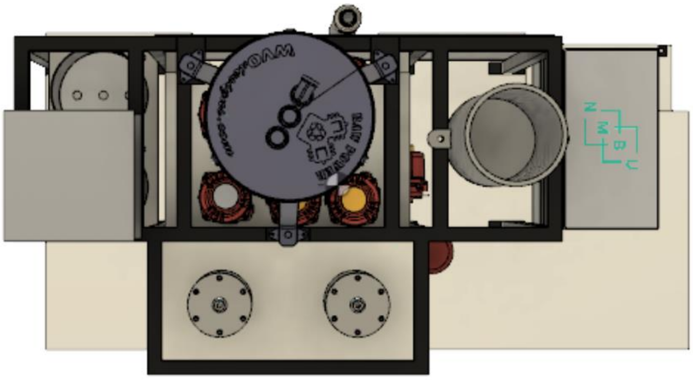


Table F1: Centre of Gravity Coordinates

	X	Y	Z
Distance from Marked Corner (mm)	763.2	414.4	913.4
Middle Point (mm)	850	400	1050

Appendix Q: Project Budget

Vendor	Item	Description	Price per Item	Amount	Price (NOK)
Biltema	Isolation material	180x50x0.8cm	59.90	1	59.90
Biltema	Vibration Dampeners	Rubber material for reducing vibrations from water pump	39.90	1	39.90
Biltema	Cable Glands	10 cable glands	19.90	1	19.90
Biltema	Cutting discs	For Cutting plates and mounts	49.70	1	49.70
Biltema	Strips	200 pk of strips for wires	29.90	2	59.80
Biltema	Thread sealing tape	14mm - 5 meter	39.90	1	39.90
Biltema	1/2" Check valve	For the vent valve	44.90	1	44.90
Biltema	230VAC pushbutton	For the control panel	21.23	8	169.84
Biltema	230 VAC LED light	2 pk - For the control panel	8.73	1	8.73
Biltema	Cable staples	200 pk - for the control panel	11.73	1	11.73
Biltema	Heavy duty gloves	for material handling during testing	99.90	1	99.90
Biltema	Plumbers Hemp	60 grams	29.90	1	29.90
Biltema	3/8"-1/2" Reducer	For the main pump	39.90	1	39.90
Biltema	3-phase connector	For the kWh counter	44.90	1	44.90
Biltema	Terminals	For connections in the junction boxes	19.90	1	19.90
Biltema	1"-2" Reducer	For the purification column	44.90	1	44.90
Biltema	Junction box	IP65 - For the cooler and the water pump	49.90	1	49.90
TESS	PVC Hose	1/2" Miljøtex hose for the water cooling	50.00	5	250.00
TESS	PTFE Hose	1/2" Tessflon	300.00	12	3600.00
Better Price	Sunflower Oil	5 Liter jugs of refined sunflower oil	34.50	4	138.00
Elfa Distrelec	Control wiring	4x0.75mm^2 – 25 meter coil	383.75	1	383.75
Elektroimportøren	Cable Channel	2 meter cable channel	149.00	2	298.00
Elektroimportøren	DIN rail	328mm DIN rail for the centrifuge VFD	49.00	1	49.00
Elektroimportøren	Power cable	1 cable for the juction box	7.00	2	14.00
Europris	Plastic boxes	For testing	39.30	3	117.90
Europris	Rapeseed oil	3 liter jugs for testing	69.90	4	279.90
NMBU Workshop	Agitator	Labor Costs for the agitator (NOK/hour)	650	12	7800
NMBU Workshop	Framework	Labor Costs for the framework (NOK/hour)	650	8	5200
SUM (NOK)					18964

The expenditures were paid by Eik Idéverksted and the Faculty for Science and Technology at NMBU. The cost of the components used on the production line from the first generation of “BioMax” can be found in Tord Reinaas’ Master’s Thesis.

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