



Treball Final de Grau

Study of a biodiesel batch production from degummed soybean oil.

Estudio de la producción de biodiesel en discontinuo a partir del aceite de soja desgomado.

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*“Una vez que aceptamos nuestros límites,
podemos ir más allá de ellos.”*

Albert Einstein

En primer lugar, agradecer a mis tutores, Dr. Manel Vicente y Dra. Esther Chamarro, por toda su dedicación y apoyo durante estos largos y duros meses. Han sido, y serán, una inspiración y un modelo a seguir.

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SUMMARY

The use of renewable resources as replacement for petroleum-derived chemicals is gaining importance. This is mainly due to the serious pollution problems with increasing fuel prices increase and the scarcity of fossil resources.

In Spain, the “Plan de energías renovables 2011 – 2020” has been carried out to improve the difficult situation that the biodiesel industry is experiencing in our country, due to according to the European directive, from 2020, 20% of the energy consumed should be renewable.

One of the most widely used type of biofuels is biodiesel and it can be obtained from vegetable oils. One of the most common is soybean oil.

The production process is split in three different stages: degumming, transesterification and purification. Usually, plant for large productions operate continuously. Nevertheless, if the production is not very high, the possibility of working in discontinuous (per batches) the transesterification stage can be considered.

Hence, the final project's goal is to consider the viability of working in discontinuous the transesterification stage, for a production plant of refined biodiesel (without impurities) of 30,000 tonnes per year.

In transesterification stage, the soybean oil is converted to biodiesel, using methanol excess as a reactant and sodium methoxide as catalyst. Biodiesel and glycerin, are the products of the reaction, which are separated by gravity.

Due to the reaction is an equilibrium reaction, the glycerine must be removed in order to force the reaction to the right to obtain more product. Therefore, the transesterification step will consist of two vessels and two settlers.

A batch size of 20,500 kg of refined biodiesel (RB) has been chosen, corresponding to 21,000 kg of non-refined biodiesel (NRB). From this value and the physical-chemical product

properties have been dimensioned the main equipment. Also, the block and process diagrams have been made with their corresponding mass balance.

The necessary pumps, valves and pipes have been chosen, as well as the automation and control system that is presented in the corresponding in the P&ID. Block and process diagram and P&ID have been drawn with AutoCad.

Moreover, it has been estimated the times of each equipment that it should be carried out, as well as, the period of occupation of the equipment. Working in two campaigns with overlapping, batch time and cycle time have been determined. In this case, the batch time is 7.5 (BT = 7.5 h) and the cycle time is 3 hours (CT = 3h).

Different scheduling strategies of the transesterification batch production have been proposed.

The most convenient has been the weekly production of 31 or 32 batches, in two campaigns of 15 and 16 batches, the first one, and 15 and 17 batches, the second one. In addition, even if the purification stage and the recovery methanol excess operates continuously without disruption, the transesterification stage will work only 5 days per week, with 3 shifts per day, without working the weekends.

Working with two campaigns per week, necessarily involves an intermediate shutdown, which it will be used for cleaning work and maintenance. Moreover, this shutdown has been programmed in such a way that it takes place coinciding with the central shift of the plant.

Keywords: refined biodiesel, non-refined biodiesel, transesterification, batch production, basic design.

RESUMEN

El uso de recursos renovables como reemplazo de productos químicos derivados del petróleo está ganando importancia, debido a los graves problemas de contaminación junto con el aumento de los precios de los combustibles y la escasez de recursos fósiles.

En España se ha llevado a cabo el plan de energías renovables 2011 – 2020 para mejorar la difícil situación que atraviesa la industria del biodiesel en nuestro país, ya que, según la directiva europea, a partir del 2020 el 20% de la energía consumida, ha de ser renovable.

Uno de los biocombustibles más utilizados es el biodiesel, y puede ser obtenido de diversos aceites vegetales. Uno de los más comunes es el aceite de soja.

El proceso de obtención del biodiesel a partir del aceite de soja consta de tres etapas: desgomado, transesterificación y purificación. Normalmente, las plantas para grandes producciones operan en continuo. Sin embargo, si la producción no es muy elevada, se puede considerar la posibilidad de trabajar en discontinuo (por lotes) la etapa de transesterificación.

Así pues, el objetivo final de este proyecto es, para una planta de producción de biodiesel refinado (sin impurezas) de 30.000 toneladas al año, plantear la viabilidad de trabajar la etapa en discontinuo de transesterificación.

En la etapa de transesterificación, el aceite de soja se convierte en biodiesel, utilizando metanol en exceso como reactivo y metóxido sódico como catalizador. El biodiesel y la glicerina, son los productos de la reacción, que se separarán por gravedad.

Puesto que la reacción, es una reacción de equilibrio, se retirarán la glicerina para poder producir más biodiesel. Por lo tanto, el proceso de transesterificación constará de dos reactores y dos decantadores.

Se ha elegido un tamaño de batch de 20,5 kg de biodiesel por batch, que corresponde a 21,0 t de biodiesel no refinado (NRB). A partir de este valor y de las propiedades físico-químicas de los productos se han dimensionado los equipos principales. Asimismo, se han realizado los diagramas de bloques y de proceso con su correspondiente balance de materias.

Se han elegido las bombas, válvulas y las tuberías necesarias, así como el sistema de automatización y control que se presenta en el diagrama P&ID correspondiente. Los diagramas de bloques y de proceso y el P&ID, se han elaborado con AutoCad.

Por otra parte, se han estimado los tiempos de cada una de las tareas que se deben llevar a cabo y los tiempos de ocupación de los equipos involucrados, determinándose el batch time y el cycle time, si se produce secuencialmente en campañas con solapamiento. En este caso, han resultado ser el batch time de 7,5 h (BT = 7,5), y el cycle time de 3 h (CT = 3 h).

Se han planteado distintas formas de programación de la producción en discontinuo de la etapa de transesterificación, resultando la más conveniente la producción semanal de 31 o 32 lotes, en dos campañas de 15 y 16 lotes y de 15 y 17 lotes respectivamente. Además, aunque la etapa de purificación y recuperación del metanol funcione en continuo sin interrupción, la etapa de transesterificación trabajará únicamente 5 días a la semana a tres turnos por día, no siendo necesario trabajar los fines de semana.

El trabajar con dos campañas por semana, comporta necesariamente una parada intermedia, que se utilizará para trabajos de limpieza y mantenimiento. Asimismo, está parada se ha programado de tal forma que se lleve a cabo coincidiendo con el turno central de la planta.

Palabras claves: Biodiesel refinado, biodiesel no refinado, transesterificación, producción en discontinuo, diseño básico.

1. INTRODUCTION

During the last century, the fossils fuels (coal, oil and natural gas) have been be the worlds primary energy source. However, the use of non-renewable fuels has been being reckless. Fuel gases, emissions from refineries and factories, etc., together with exhaust emission from vehicles are the leading sources of air pollution. It is necessary to begin using to renewable energy sources to avoid massive economic disruption caused by a global energy crisis. ⁽¹⁾

Any hydrocarbon fuel that is produced from organic material in a short period of time is considered a biofuel. Although, biofuels can also be made through chemical reactions carried out in a laboratory or industrial setting, that use organic matter (biomass) to make fuel. The only real requirements for a biofuel are firstly that the starting material must be CO₂, was fixed by a living organism and the final fuel product must be produced relatively quickly and not over millions of years. ⁽²⁾

The two most widely used types of biofuels are bioethanol and biodiesel. They are made from sugar, starch or vegetable oil and they are produced through well-understood technologies and processes such as fermentation, distillation and transesterification. ⁽³⁾

	Bioethanol	Biodiesel
Process	Fermentation	Transesterification
Compatibility	Must be blended with fossil fuel	Able to run in any diesel generated engines
Costs	Cheaper; less land use 420 (gal/ac)	Expensive; more land use 60 (gal/ac)
NEB	High; provides 93% more net energy	Low; provides 25% more net energy
GHG emissions	12% less than the production and combustion of regular diesel	41% less than the production and combustion of conventional gasoline

Table 1. Bioethanol and Biodiesel comparison. ⁽³⁾

We can stage that the advantages of biodiesel manufacturing are better than bioethanol manufacturing.

1.1. BIODIESEL PRODUCTION BY SOYBEAN OIL

Normally, the composition of soybean oil is composed primarily of triglycerides with different free fatty acids (FFA). Nevertheless, the FFA of soybean oil are primarily unsaturated (approximately 68.3%), like linoleic, linolenic and oleic acid. Palmitic acid and stearic acid are saturated FFA. ⁽⁴⁾

To simplify this model, it is assumed that there is only one kind of component acid or FFA, oleic acid and therefore, one type of triglyceride, triolein. It is important to know the chief part of FFA are triglycerides. Hereunder, the composition of soybean oil can be seen. ⁽⁴⁾

Compound	Weight fraction
Triolein	0.975
Oleic acid	$1.910 \cdot 10^{-2}$
Water	$6.030 \cdot 10^{-3}$
PE-Ca	$1.670 \cdot 10^{-4}$
Total	1.000

Table 2. Soybean oil's composition. ⁽⁴⁾

As documented in the above table, triolein is the major soybean oil component whose purity is at 97.5 % in soybean oil. For that reason, for the mass balance and the process dimension, triolein will be the raw material instead of soybean oil, to facilitate the calculations that it will be carried out.

Generally, the less expensive the raw material, the less refining the oil has experienced, accordingly the raw material contains more variety of impurities such as phosphatides or gums and higher free fatty acids (FFAs). Therefore, these impurities must be removed as if they remain they will cause problems during biodiesel manufacturing. ⁽⁵⁾

Soybean oil, has been pre-treated to remove the impurities, specifically the phospholipids in soybean oil. This section is known as degumming. After that, the soybean oil goes to the transesterification section where triolein is converted into methyl oleate or biodiesel. This section comprises two steps: transesterification reaction and phase separation by settling. ⁽⁵⁾ The methyl oleate obtained, contains impurities. Thus, after the transesterification section should come the purification section to obtain a refined biodiesel.

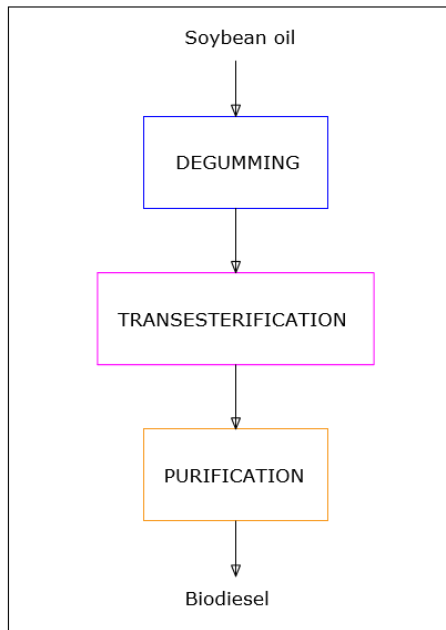


Figure 1. Biodiesel production by soybean oil.

Global process is produced continuously but, in some cases, transesterification process can be produced in batches. Even if the production was to be 30,000 – 40,000 tonnes per year it is considered as small volume compared to the quantities that can be produced from biodiesel. However, the benefit of small scale production always exists. If it works with small volumes and the process needs less capital, the plant will have more *flexibility* than when working continuously. Although the final decision is economical function. ⁽⁶⁾

1.1.1. Transesterification reaction

Transesterification is the conversion of a carboxylic acid ester into a different carboxylic acid ester. ⁽⁷⁾ Hence, it is necessary to know the stoichiometry of the reaction.

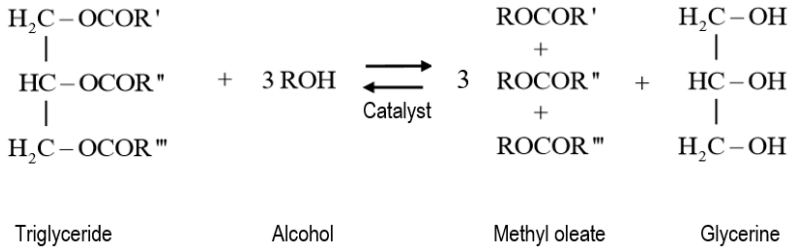


Figure 2. Transesterification reaction.

Transesterification is an equilibrium reaction. This implies that the product must be removed in order to force the reaction to the right to obtain more product. In fact, transesterification consists of a number of consecutive and reversible reactions because triglyceride is converted into diglyceride, monoglyceride and, finally, glycerine. ⁽⁸⁾ Consequently, diglyceride and monoglyceride are sub-products of the reaction.

The conversation and yield of the transesterification reaction are affected by variables such as the molar ratio of alcohol to oil, temperature, impurities and catalyst. A molar ratio of 6:1 (alcohol: oil) is recommended for optimum triglyceride conversion. Minor relationships result in low conversions. ⁽⁸⁾ In this case, the yield of the transesterification has a conversion of approximately 99.85% and it will be seen in section number 3.

Furthermore, the reaction needs a catalyst. Typically, sodium hydroxide is the alkaline catalyst used for the transesterification of triglycerides but one of the disadvantages is that contributes to the formation of soaps. Thus, this formation hinders the separation process. To avoid that, this catalyst is replaced by sodium methoxide (CH_3ONa). The disadvantage in this compound is that if water is present it reacts with the catalyst, which then reverts to flammable methanol and sodium hydroxide. For that reason, the sodium methoxide is sold dehydrated in 30% methanol solution, without the associated water. In addition, this catalyst should be 0.2% of the initial mass of triolein. ⁽⁹⁾

2. OBJECTIVES

The main aim of this project is to study the transesterification stages per batches allowing to answer the production demands of 30,000 annual tonnes of refined biodiesel from degummed soybean oil. In order to achieve the goal, the following tasks have been proposed:

- Selecting all the necessary equipments to reach the desired annual throughput.
- Selecting a batch size of biodiesel for design all the involved equipments in transesterification stage.
- Block and process diagram.
- Piping and instrumentation diagram (P&ID).
- Determining the necessary time to produce one batch of biodiesel.
- Production scheduling.
- Determination of the operation strategy.

Plant utilities are out of the battery limits of the project.

3. BASIC DESIGN

Biodiesel production has the following reagents: soybean oil and methanol, using sodium methoxide as a catalyst, and the products are biodiesel, glycerine and waste. As it has been commented in introduction section. Comment that in this project, the soybean oil will come without phosphates, that is mean degummed soybean oil.

The three stages for obtaining biodiesel are produced in continuous, although the transesterification stage is going to be studied by batches. Another important aspect is the methanol's recovery. The stoichiometry of the reaction shows that 3 methanol moles are needed by one mole of soybean oil but for obtaining a better conversion, 6 methanol moles are needed, this mean an excess of methanol. This excess must be recovered to minimize raw material costs and, in consequence, increase the process benefits. Depending on how this recovery is, the benefits will be higher or lower.

Hence, purification stage does not only imply the impurities elimination from biodiesel, but also separates glycerine from biodiesel and it recovers the methanol excess. To be able to treat methanol and to recover it, the bibliography shows that 15% of methanol is gone with glycerine stream. ⁽¹⁰⁾

Global process is designed to produce 30,000 t/y of refined biodiesel continuously. Knowing the conversion and the reaction stoichiometry, the annual amount of the reactants and products to be treated are known by global mass balance and it is show below.

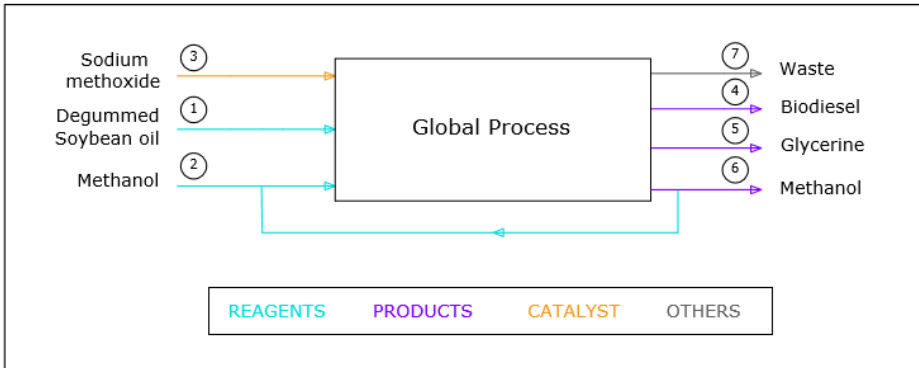


Figure 3. Overall diagram of the inputs and outputs of Biodiesel production.

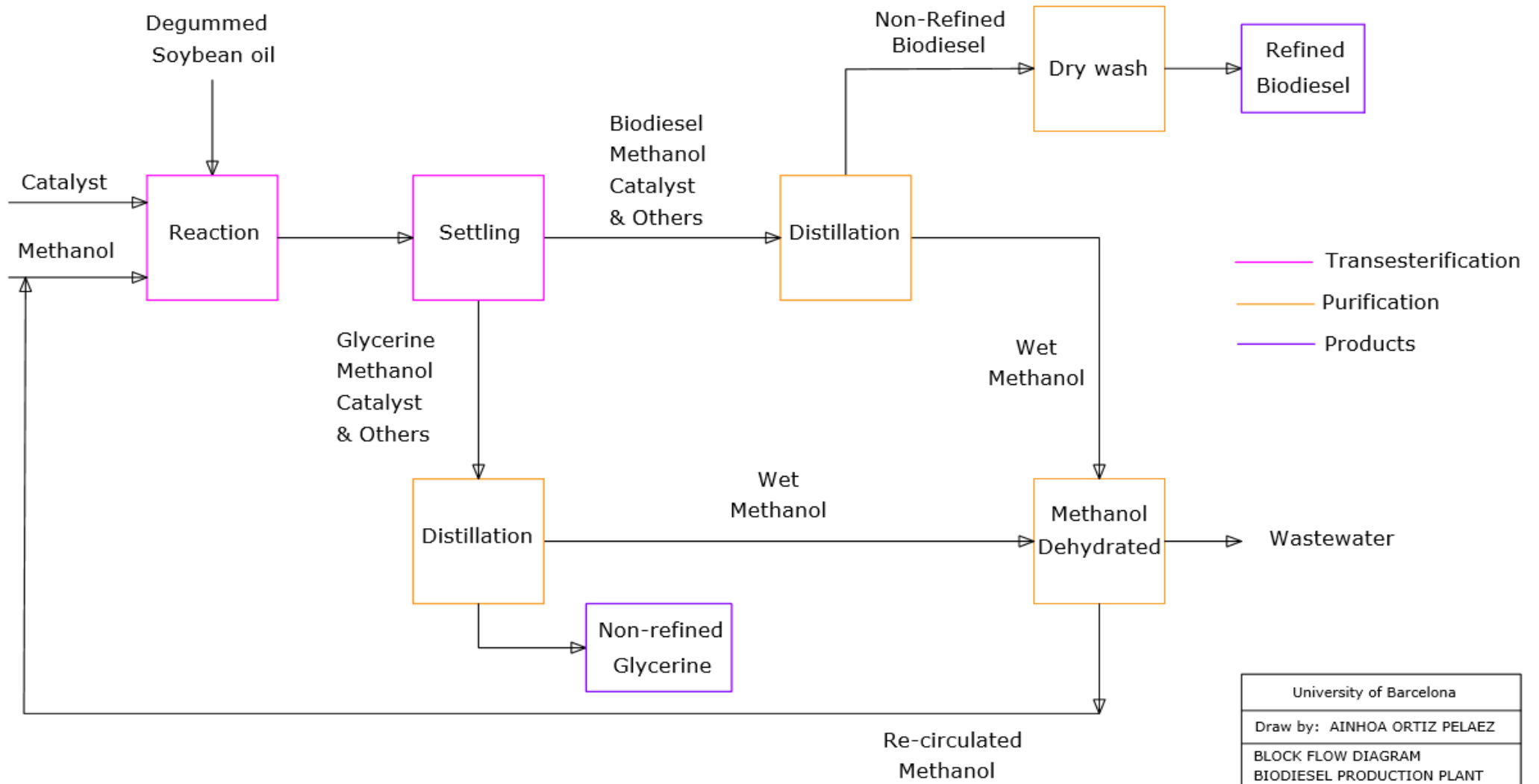
Stream in	1	2	3	Total
Mass [t/y]	32,714	4,969	75	37,758

Stream out	4	5	6	7	Total
Mass [t/y]	30,000	3,106	1,067	3,585	37,758

Table 3. Annual global mass balance.

3.1. BLOCK DIAGRAM

Block flow diagram gives a general notion about the process is going to be studied. For that reason, in this section, it is shown to understand better the process. It is represented by all the inputs and outputs, in which the transesterification and purification stages are differentiated as well as the final products.



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BLOCK FLOW DIAGRAM BIO DIESEL PRODUCTION PLANT TRANSESTERIFICATION SECTION	
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3.2. DESIGN BASES

Hereunder, the assumptions are shown that were used to develop the front-end engineering design package to produce biodiesel from soybean oil.

- One of the aims of this project is producing 30,000 tonnes/year of refined biodiesel (RB). This amount is the calculation base for the design of production process. In addition, the biodiesel impurities will be set at 2.50% in non-refined biodiesel.
- All process stages are defined in continuously except one of them, transesterification process is defined in discontinuous.
- The global process will be operating per batches, 330 out of 365 days by year, working 7 days/weeks (24 hours/day) for producing this amount of biodiesel. That means 7,920 hours/year.

3.3. REACTION

As in any process, it is important to know the kinetics of the reaction. The *Noureddini* paper shoes conversion versus reaction time for a transesterification reaction considering the intermediary diglycerides and monoglycerides. This graphic shows two axes, triglycerides (TG), diglycerides (DG) and monoglycerides (MG) are referenced to the second axis while methyl esters or biodiesel (ME) are referenced to the primary axis. The reaction is carried out in just one reactor. ⁽¹¹⁾

According to the heuristics, the reaction is carried out in two reactors: twenty minutes in the first reactor is enough to achieve the maximum conversion (85% approximately), as set out *Noureddini* paper explains. After this time, the glycerine and biodiesel are separated by settling, to force the reaction to the right to obtain more product, as it has been explained previously. This biodiesel containing non-reacted triglycerides, diglycerides and monoglycerides is sent to the second reactor for another twenty minutes. In the second reactor, reaction achieves a conversion of 95%. Therefore, the whole transesterification reaction has a conversion of approximately 99.85%.

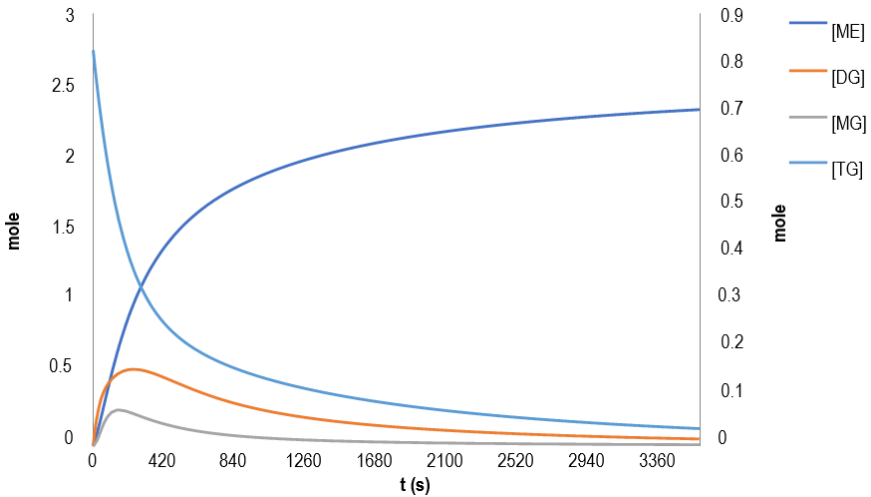


Figure 4. Conversion vs reaction time. ⁽¹¹⁾

Looking the graphic, the 1200 seconds is approximately 85% conversion to methyl esters.

3.4. SETTLER

Once the transesterification reaction is finished in the reactors, the output is transferred to a settler to carry out the settling process. Biodiesel and glycerine are immiscible liquids and separation of two immiscible liquid phases with differing densities by settling is basically governed by Stokes law.

Glycerine is denser than biodiesel, hence, glycerine will be in the bottom part of decanter while biodiesel will be at the top.

On the other hand, methanol and triolein are miscible liquids with biodiesel and glycerine. To simplify the model, it is assumed that triolein is miscible, only, with biodiesel, and to resolve the mass balance it is necessary to know the partition coefficient between biodiesel and glycerine for knowing the methanol mass. So, according to the bibliography ⁽¹⁰⁾ methanol partition coefficient between biodiesel and glycerine is $S_B/S_G = 0.015$, where S_B means the methanol solubility in biodiesel and S_G means the methanol solubility in glycerine.

As has been commented previously, the glycerine is removed to displace the balance in order to obtain more product. For that reason, the biodiesel from S-01 is sent to the second reactor while the biodiesel from S-02 is sent to purification process.

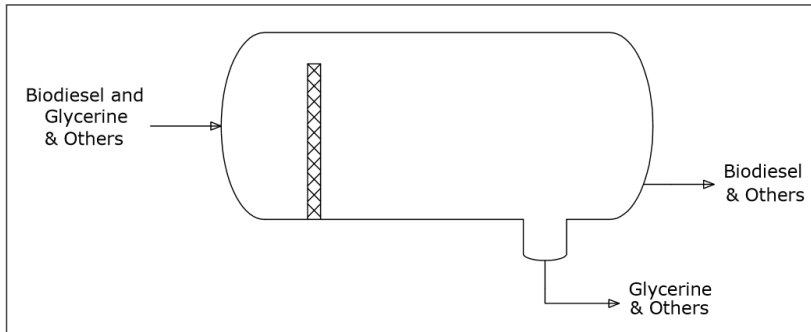


Figure 5. Settler inputs and outputs.

3.5. RECIPIE

Initially, degummed soybean oil will be kept in a storage process tank, T1, while methanol will be in another storage tank T2 and sodium methoxide will be dissolved in 2% with methanol in T3.

Previously, the degummed soybean oil stream, at 20 °C, will increase its temperature to 75°C through a double tube heat exchanger. Instead of dimensioning three heat exchangers for three fluids to reach 60 °C, it has been chosen to dimension one. Thus, the soybean oil will come out at 75 °C from the heat exchanger, when it will be added into the reactor as well as methanol and glycerine, the mixture will be at 60 °C approximately. The heating fluid will be water steam, at 8 bars and 170 °C (boiling temperature) provided by plant services. After the exchange, the heating fluid will have cooled, but being a steam at a boiling temperature it will produce a phase change and the phase changes are maintained at constant temperature. Accordingly, water steam will come out of the heat exchanger in a liquid state and a 170 °C.

After that, the reagents and the catalyst will be set to the first vessel, V-01. The reaction is produced at atmospheric pressure and at a temperature of 60 °C. For this reason, the vessel will have a jacketed to keep the reactor temperature constant. The heating fluid will be water steam, at 8 bars and 170 °C the same as the heat exchanger.

Once the reaction is finished, to force the reaction to the products, the compounds will be sent to a settler, S-01, to separate the immiscible phases of biodiesel and glycerine, considering that all the triolein goes with the biodiesel. On the other hand, part of methanol dissolves in glycerine and in biodiesel but, by distribution coefficients it is known that it is more soluble in glycerine than in biodiesel. In any case, this solubility will be explained later in settler section.

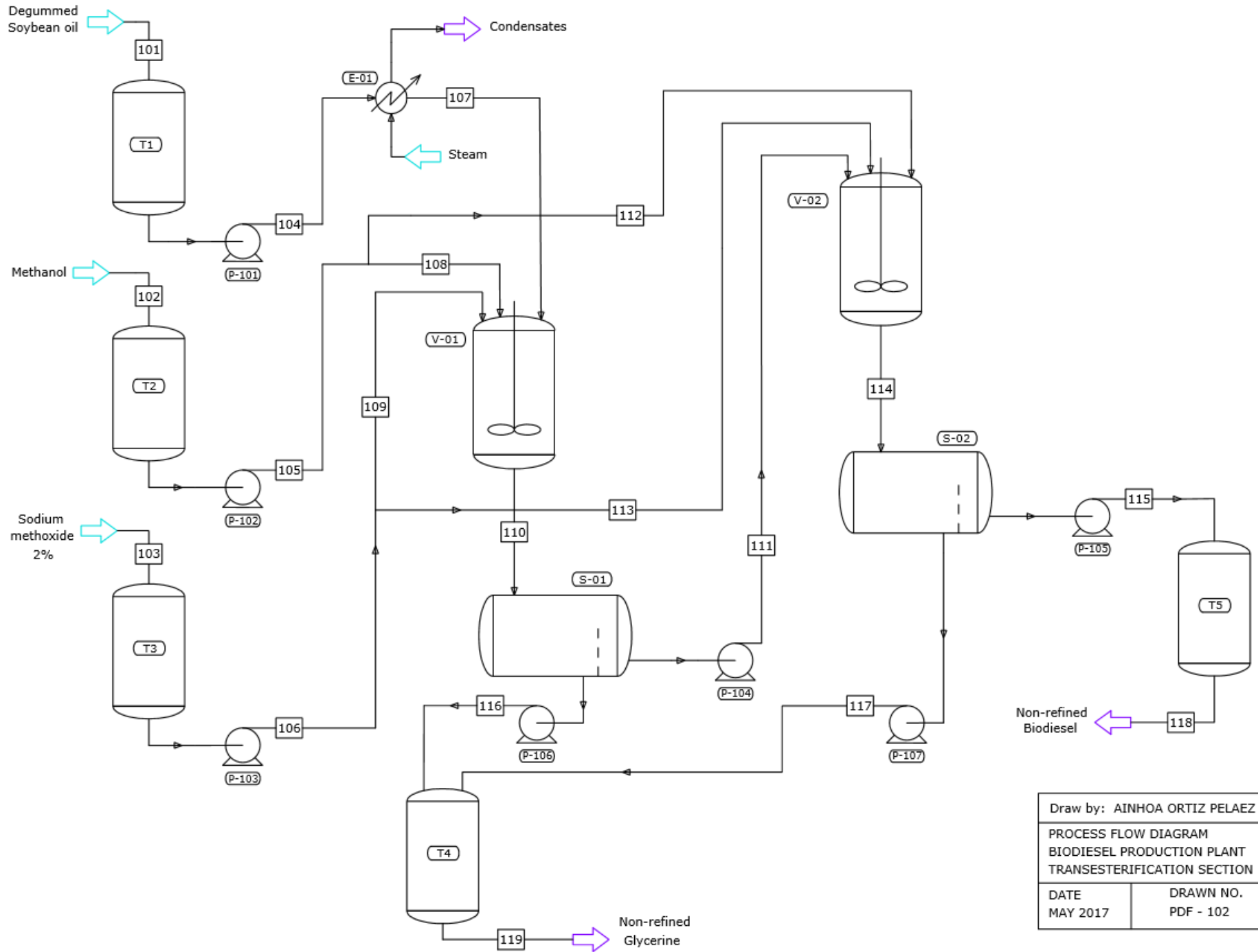
The mixture will be allowed to cool until 50 °C, where finally the separated non-refined glycerine will go to a storage tank, T4, and the biodiesel phase will be sent to the second reactor, V-02.

In the second reactor, the methanol and sodium methoxide amount necessary to carry out the reaction is added. Working at the same conditions as the first vessel. It will also appear of a jacketed whose heating fluid will be water steam at the same conditions as the first vessel too.

In the same way, the compounds will be sent to the second settle, S-02, where non-refined glycerine phase will be sent to the same storage tank used in the first settler, T4. Rather, non-refined biodiesel obtained will be store in another storage tank, T5.

3.6. PROCESS FLOW DIAGRAM

Once the batch size has been chosen, 20,970 tonnes of non-refined biodiesel (NRB), as it will be seen in section number 7, the mass balance has been solved for that batch size. Mass balance and the process flow diagram for transesterification stage by batches is shown below.



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PROCESS FLOW DIAGRAM BIO DIESEL PRODUCTION PLANT TRANSESTERIFICATION SECTION	
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Mass [kg/batch]											
	104	108	109	110	111	112	113	114	115	116	117
Soybean Oil	22300	0	0	3077	3077	0	0	144	144	0	0
Methanol	0	2808	2000	2562	371	384	343	684	132	2191	552
Sodium methoxide	0	0	44	44	19	0	7	26	12	25	15
Biodiesel	0	0	0	17515	17515	0	0	20451	20451	0	0
Glycerine	0	0	0	1813	5	0	0	309	0	1808	309
MG	0	0	0	368	1	0	0	6	0	367	6
DG	0	0	0	1275	4	0	0	17	0	1271	17
Impurities	0	0	0	498	298	0	0	386	232	200	154
TOTAL	22300	2808	2044	27152	21290	384	350	22024	20971	5862	1053

V - 01	Stream in: 104 + 108 + 109	27152
S - 01	Stream out: 111+ 116	27152
V - 02	Stream in: 111 + 112 + 113	22024
S - 02	Stream out: 115 + 117	22024

Table 4. Mass balance by batch.

4. PROJECT SPECIFICATIONS

In this section, it will be detailed the specifications of the mentioned equipments previously. It will be talked about their size, materials and their auxiliary equipments in case they are needed.

As it has been seen previously, the process has two vessels and two settlers. Both vessels and settlers have the same dimensions due to economic and flexibility issues. Consequently, the vessel's auxiliary equipment will be the same for both.

Highlight that all the physical-chemical properties of the process have been found by the chemical process optimization software Aspen Plus®.

The material used, for all equipments, will be stainless steel as it is known for its high corrosion resistance. The most utilized are AISI 304 and AISI 316. The difference between them is that 304 stainless steel contains 18% chromium and 8% nickel while 316 stainless steel contains 16% chromium, 10% nickel and 2% molybdenum. With the molybdenum addition, corrosion resistance is improved, proving a better corrosion resistance for 316 steel. In this case, due to it is not corrosive fluids, AISI 304 will be chosen for all the equipments and pipes.

4.1. VESSELS V-01 AND V-02

In vessel V-01, the next stages take place: feeding of raw materials and reaction.

According to the legislation, the surface of those equipments through the interior of which circulates a fluid at temperatures below 5 °C or exceeding 50 °C, should be isolated. Therefore, the outside of the jacketed with glass wool of 100 mm thick, the amount used for this type of process. ⁽¹²⁾

Vessel's design calculations can be seen with detail in the Appendix 2. However, the next table contains a summary of his characteristics.

Diameter [m]	3
Section [m²]	7.07
Height [m]	4.5
Wall thickness [m]	0.01
Volume [m³]	32

Table 5. Characteristics of vessel V-01.

4.1.1. Auxiliary equipments

For all tasks that are performed in both vessels, it is required:

- Good level of homogenization. Therefore, it will be necessary to include an agitation system.
- One jacket to keep the reactor temperature at 60 °C, favouring the reaction and avoiding the temperature increase due to it is an exothermic reaction.

Agitators

As it's typical in the industry, for this kind of process, the agitation system is carried out by means of propeller stirrer. Due to it is a viscous fluid and the degree of homogeneity must be ensured, the stirrer will be consisted of 3 propellers. In addition, the lower propeller will be shorter than the other two because it will be closer to the concavity of the bottom. Nevertheless, the stirrer will be anchored to the bottom to prevent vibration of the reactor.

Design calculation of agitation system can be seen in Appendix 2. Anyway, the next table contains a summary.

Stirrer diameter [m]	1
Height [m]	3
Rotational speed	60
Agitation power [kW]	6

Table 6. Agitation system characteristics.

Jacket

The vessels should have a heating system that supplies or removes power needed to ensure the vessel development.

The heating system will be necessary once all the reagents has been added and the reaction will be occurring.

The catalyst stream and methanol stream will be at 20 °C, for the first reactor, while soybean oil temperature stream will be 75 °C, as it has been commented before.

To maintain the vessel at 60 °C, through the jacket, will circulate water steam at 8 bars and 170 °C. This water stream is a plant services.

Operating conditions of the second vessel are not identical of the first vessel. However, the jacket dimension for both vessels will be the same. Consequently, this fact would allow, if it is necessary, to use V-02 as V-01 in a hypothetical issue.

According to the legislation, the surface of those equipments through the interior of which circulates a fluid at temperatures below 5 °C or exceeding 50 °C, should be isolated. Therefore, the outside of the jacketed with glass wool of 70 mm thick, which is considered the minimum required or industrial equipment will be covered. ⁽¹²⁾

The calculations can be seen in Appendix 2, but here below it shows the summary of the jacketed vessels characteristics:

Area of contact [m²]	8.6
Q [J/h]	$2.3 \cdot 10^9$
W_{steam} [kg/h]	$1.1 \cdot 10^3$

Table 7. Jacketed vessels characteristics.

4.1.2. Operating conditions

As discussed previously, in T4 will be store NRG while T5 will store NRB. By the mass balance, and once the batch size has been chosen, the mass in each current is known by batch, as it will be shown in section number 7. It is considered interesting to know how much can be stored in these storage tanks because they have finite dimensions.

In addition, mass changes over time, in other words, total mass in the storages tanks will not be the same after 5 hours as 20 hours. This interpretation is shown below:

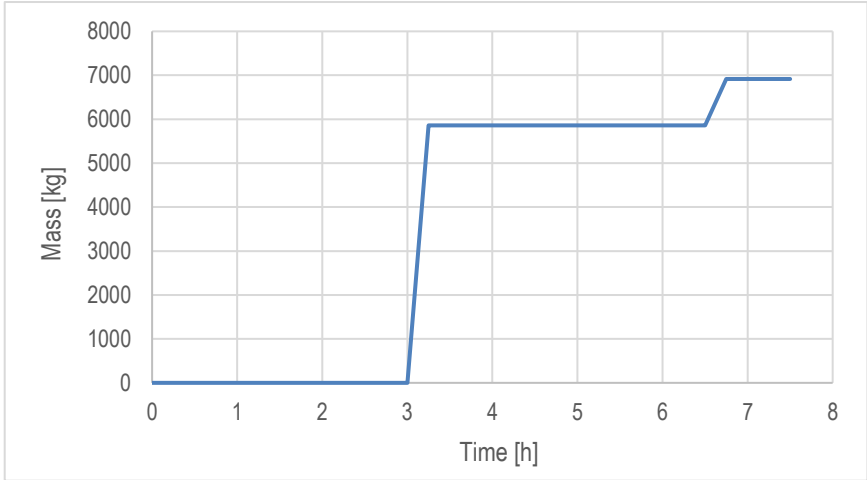


Figure 6. Mass variation in T4 per batch due to production

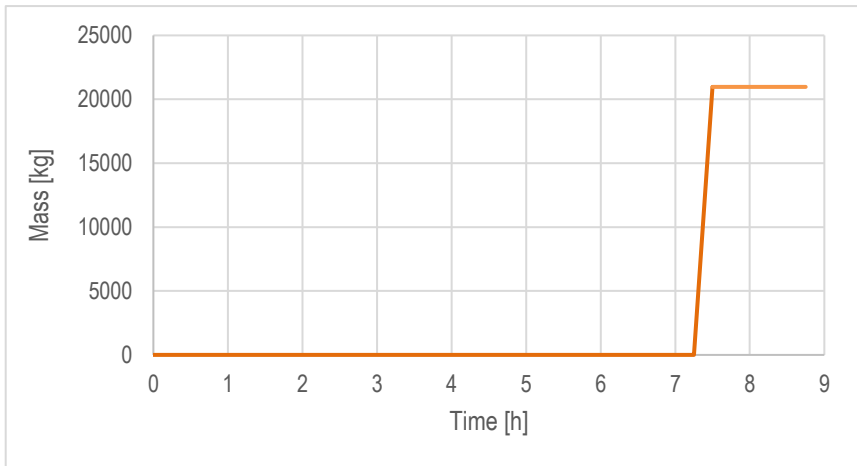


Figure 7. Mass variation in T5 per batch due to production.

As documented in the above figures, biodiesel amounts are larger than glycerine quantities. For that reason, storage tanks are shown separated in the graphics.

4.2. SETTLERS S-01 AND S-02

The settlers will be cylindrical and it will be in a horizontal position. In addition, it will consist of a coalescer for helping the separation of the phases.

Known the settler dimensions, the coalescer will have chosen by vendor. The design of the decanters can be seen in Appendix 2, although here below is a summary of their characteristics:

Diameter [m]	3
Section [m²]	7.07
Height [m]	4.5
Wall thickness [m]	0.01
Volume [m³]	32

Table 8. Settlers characteristics.

In addition, the height of each phase (biodiesel and glycerine) inside the settlers, has been calculated for knowing the liquid occupation (the maximum height will be the settler diameter, 3 meters), and here below are shown. In any case, the calculations are found in Appendix 2.

h [m]	S-01	S-02
Biodiesel phase	2.13	2.10
Glycerine phase	0.61	0.19
Total	2.74	2.29

Table 9. Settlers height.

Comment that in the decanter the liquid-liquid mixture will have cooled up to 50 °C. As it has been mentioned above, if inside of the any equipment exceeds 50 °C, the equipment must be isolated. For that reason, the settlers will not be insulated.

4.3. HEAT EXCHANGER

In order to select the type of heat exchanger, the next factors must be considered: thermal and hydraulic requirements, operating conditions, cleaning of the exchange, available space, tolerable weight, economics aspects. ⁽¹³⁾ Thermal and hydraulic requirements mean it is necessary to know the amount of heat that should be exchanged. In addition, heat transfer is always linked to the pressure drop experienced by the fluids.

Considering these factors, a shell-and-tube exchanger has been chosen. The characteristics of this heat exchanger are: ⁽¹³⁾

- It allows to operate a wide range of pressure and temperature.
- It can be used for many functions, including phase change.
- Depending on the operation's needs, is built with various types of materials.
- Construction methods are standardized by TEMA (Tubular Exchanger Manufacturers Association).

The shell-and-tube exchanger is divided into three zones: front header, shell types and rear header. Many combinations of these three zones can be made according to the type designations for shell-and-tube heat exchangers adopted by TEMA. Although, the combination BEM is the type chosen in this design with one step. ⁽¹⁴⁾

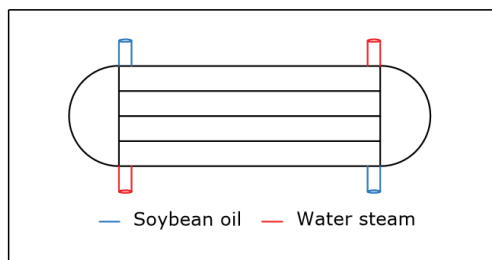


Figure 8. BEM Heat Exchanger with one step.

The fluid will be degummed soybean oil. The inlet temperature will be 20 °C and the outlet temperature will be 75 °C, working at atmospheric pressure. As commented above, the outlet temperature is 75 °C, because once all the compounds are in the reactor, the mixture will be at 60 °C approximately. Furthermore, the heating fluid will be water steam at 170 °C and 8 bars.

The design of the heat exchanger has imposed the tubes number that the exchanger requires, 60 tubes, and the speed at which the fluid circulates inside the tubes, 1 m/s.

Furthermore, the following heuristic may be used to estimate tube counts for various bundle sizes and tube passes. The estimated values include the removal of tubes to provide an entrance area for shell nozzle sizes of one-fifth the shell diameter. The formula/heuristic indicates that triangular tube layouts and square tube layouts must be separated 1.25 times the tube outside diameter. ⁽¹⁵⁾

Chosen the tubes that should go inside the exchanger, the characteristics of the tube-and-shell exchanger are shown below, although the calculations can be found in Appendix 2:

CONDITIONS	
Exchange area [m²]	16.40
Heat exchange [W]	1.12·10 ⁶
Tubes number	60
v [m/s]	1.00
TUBES	
External diameter [mm]	21.34
Thickness [mm]	2.11
Internal diameter [mm]	17.12
Length [m]	7.00
SHELL	
External diameter [mm]	224.8

Table 10. Characteristics of shell-and-tube exchanger.

4.4. VALVES & PIPES

Valves

In this project two types of valves are clearly differentiated, ball valves and butterfly valves. The last one, it is the control valves.

Process plants consist of a lot of control loops all networked together. Each of these control loops is designed to keep some important process variable such as pressure, level, temperature, etc. The problem is the process has disturbances that influence the process variable. For that reason, the most common final control element in the process control industries is the control valve. The control valve manipulates a flowing fluid to compensate for the load disturbance and keep the regulated process variable as close as possible to the desired set point. ⁽¹⁶⁾

A ball valve is a form of quarter-turn valve which uses a hollow perforated and pivoting ball to control flow through it. It is open when the ball's hole is in line with the flow and closed when it is pivoted 90 degrees by the valve handle. The handle lies flat in alignment with the flow when open, and it is perpendicular to it when is closed. There are valves closing securely even after long periods of disuse. For that reason, these kinds of valves are excellent choice for shutoff valves. Moreover, supporting pressures up to 1000 bar and temperatures up to 400 °C, depending on design and materials used. ⁽¹⁶⁾

A butterfly valve is a valve that isolates or regulates the flow of a fluid. Operation is similar to that of a ball valve, which allows for quick shut off. The disc is positioned in the center of the pipe. A rod passes through the disc to an actuator on the outside of the valve. Rotating the actuator turns the disc either parallel or perpendicular to the flow. Unlike a ball valve, the disc is always present within the flow, so it induces a pressure drop, even when open. ⁽¹⁶⁾

Pipes

Initially a speed has been imposed to know the pipes dimensions, knowing that the fluid velocity should be between 1 and 2 m/s. The lowest has been chosen. It has to be taken into account that reducing the speed, increases the diameter and, consequently, reduces pressure drop.

Subsequently, through a pipeline catalogue, piping's have been selected according to the calculated dimensions. Therefore, next step is to recalculate the speed to know if it is between commented interval. All the calculations can be found in Appendix 2.

Thus, the pipes dimensions are:

	104 ^a	105 ^b	117 ^c
d_{ext} (mm)	114.30	48.26	21.36
e (mm)	3.05	2.77	2.11
d_{int} (mm)	108.20	42.72	17.14

Table 11. Pipe dimensioning.

- a. Pipes number: 104, 107, 110, 111, 114 and 115 have the same dimensions.
- b. Pipes number: 105, 106, 108, 109, 112, 113 and 116 have the same dimensions.
- c. Pipe number 117 has these dimensions.

4.5. PUMPS

Generally, there are two types of pumps: centrifugal and positive displacement pumps. Although centrifugal pumps are the most common, positive displacement come in a wider variety such as: gear, lobe, screw, etc. ⁽¹⁷⁾

Main difference between these two types of pumps is that centrifugal pumps create a pressure differential resulting in flow, while, positive displacement pumps, the pressure differential results from flow created by the pump. ⁽¹⁸⁾

Pumps are usually chosen by a vendor; different data must be submitted to the company to be able to choose the pump type. The vendor needs data such as:

- Pressure difference (Pump ΔP). Pressure at the beginning and at the end of the pump.
- Total dynamic head (Pump TDH). The total equivalent height that a fluid is to be pumped, taking into account friction losses in the pipe.
- NSPH (Net Positive Suction Head). It helps to know the proximity of the installation to the cavitation.

Cavitation is a hydrodynamic effect. It occurs when the fluid, in liquid phase, rushes into the pump so fast and produces a fluid decompression. In other words, when vapor pressure is reached, the molecules immediately change to gaseous state and form bubbles known as cavities. Therefore, the pressure in the stream in must be greater than the vapor pressure of the mixture inside the pump. The NPSH amount of the process must always be greater than the NSPH amount provided by the manufacturer.

In this project, centrifugal pumps have been selected because for these characteristics and operation condition, the most used in the industry are centrifuges pumps. Moreover, the toxicity and corrosivity of the material and the fluid are low.

4.6. PLANT SERVICES

The following points will be offered by plant services:

- Water steam. During the process, and average pressure of 8 bars is required for the heating fluid for carrying out the temperature increase of degummed soybean oil input. Also, it is used to keep the vessel temperature constant at 60 °C.
- Nitrogen stream. It will be used to inert storage tanks, T2 and T3, and vessels, V-01 and V-02, when it will be necessary. Moreover, in the vessels are used to download faster. In other words, according to Bernoulli equation, for closed containers, discharge velocity is independent of the diameter of the pipe (the opposite of the open containers) but providing more pressure it will lead to discharge faster.
- Cooling water. It refers to the water used for cooling processes such a public channel catchment water (rivers, lakes, etc), seawater and cooling tower circuit water (natural or forced draft, in this case, the design variable is the we bulb temperature of the zone.

4.7. STORAGE TANKS

It is important to know that storage tanks T1, T2, T3 are process tanks. In other words, the storage quantities come from larger tanks. This means that these tanks are designed for keeping the mass between 1 – 3 days. Hence, T1 will be designed for working one day while T2 and T3 will be designed for working 3 days.

By contrast, the sizing of the storage tanks T4 and T5 comes from an optimization. Depending on the operation mode of the plant will have one dimensions or others, as it will be seen in section 7.

However, the dimensions are shown below:

Storage Tank	T1	T2	T3	T4	T5
Volume [m ³]	180	80	60	130	405

Table 12. Storage tanks dimensions.

By mass balance, the necessary amount of degummed soybean oil, methanol, and sodium methoxide diluted 2% is known. Knowing the batch time (it will be seen in section 7) and the days that each tank is going to store, the total mass is known. Volume is relating with the mass through the density, so knowing the mass and the density, volume shall be set.

Furthermore, it should be commented that for T4 and T5 tanks will be composed of two tanks each. The total stored volume for T4 will be 130 m³, and for T5, 405 m³.

The fact of having two tanks instead of one is for the operation strategy, while one is filling up, the other one is discharging.

5. PI&D

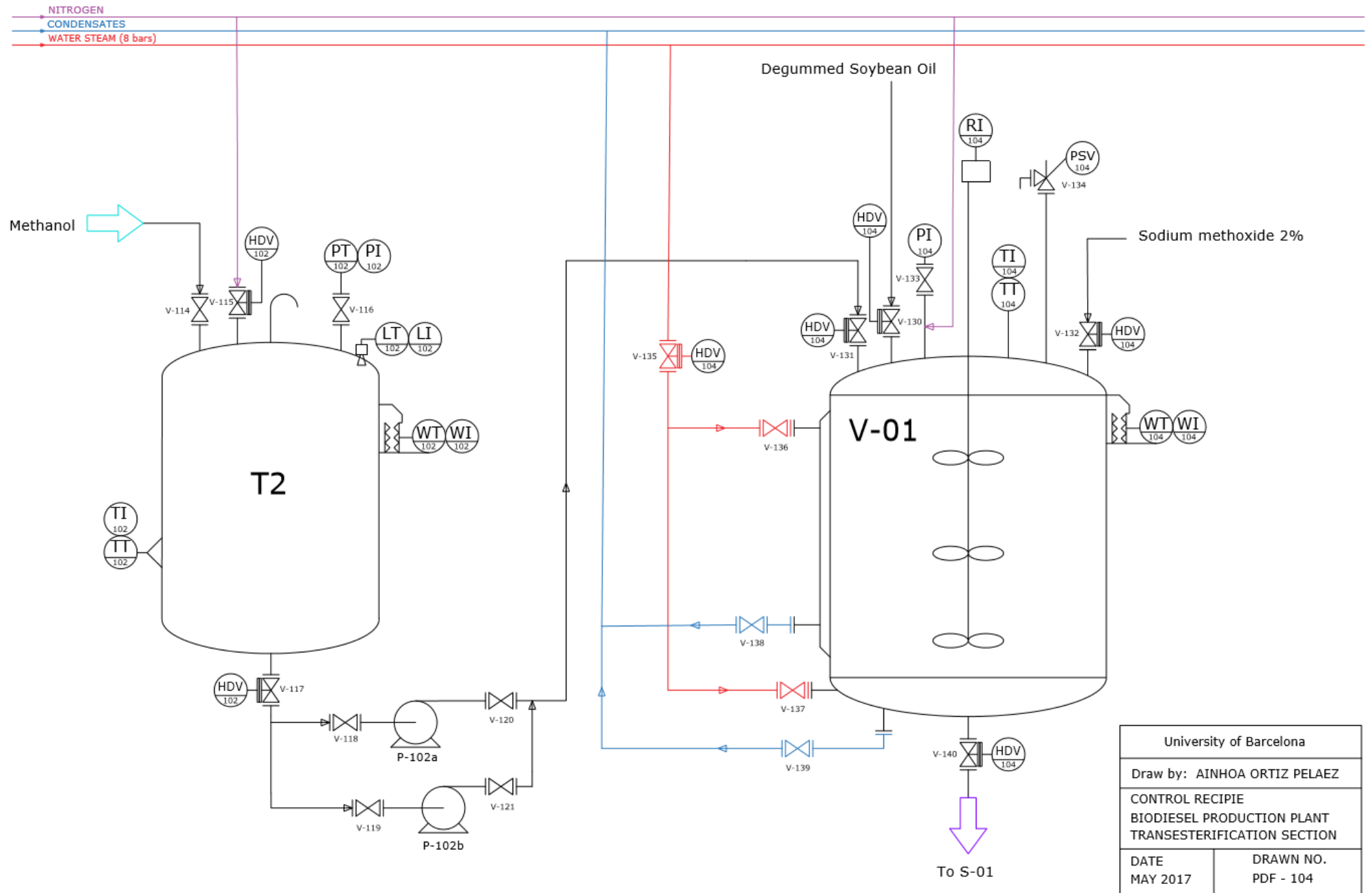
This section will show the P&ID (Piping & Instruments Diagram) of the process. The design has been created using the program AutoDesk® AutoCad.

Firstly, it will be shown an example, as it should be the methanol loading in the vessel. Therefore, the control recipe will be explained.

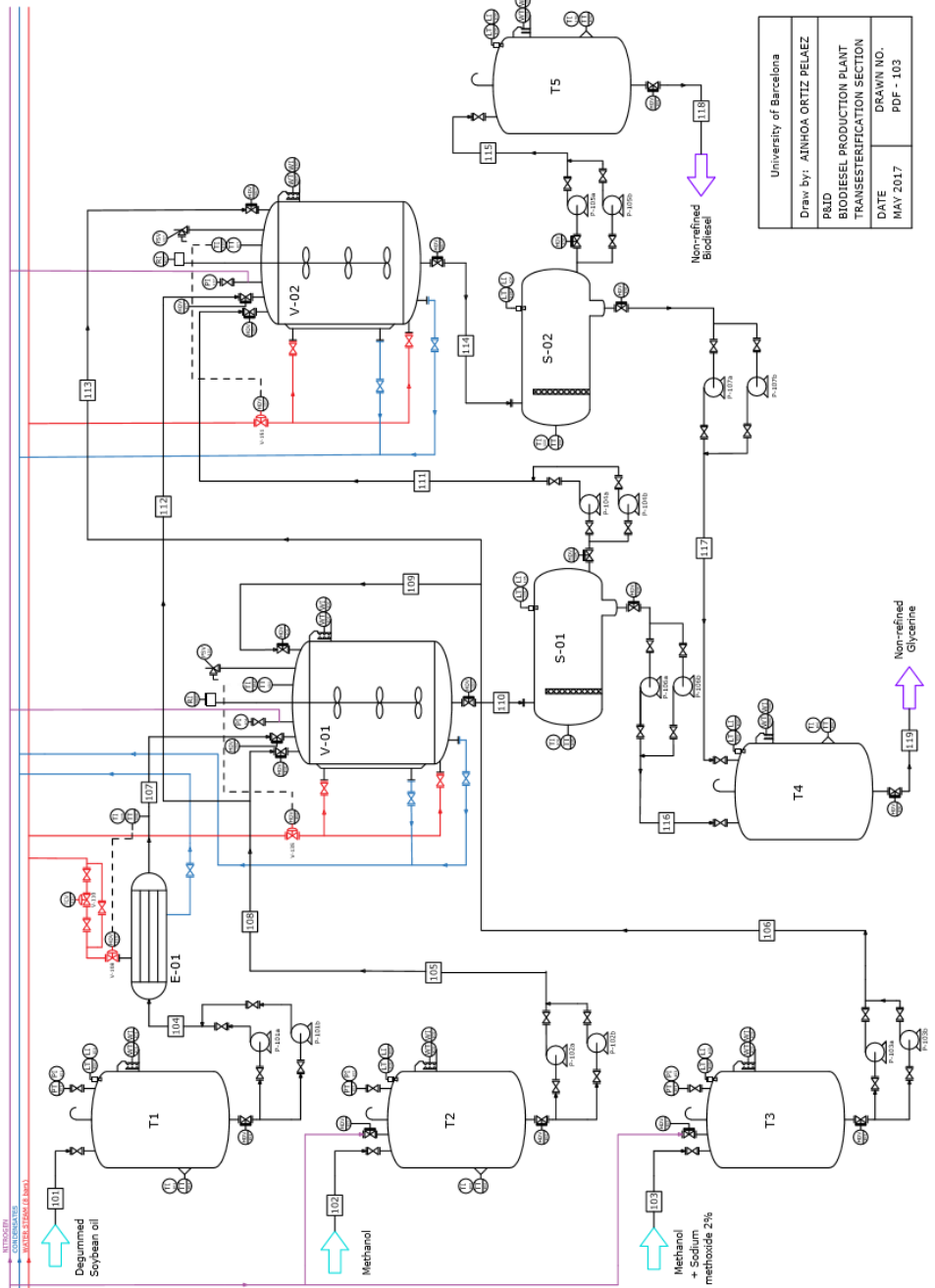
Secondly, it will be show the P&ID. To observe better the P&ID diagram, the diagram in DIN-A3 can be found at the end of the memory.

Charge V-01 with Methanol Storage Tank

1. First, checking V-140 valve must be closed. If it is not closed, it must close.
2. Secondly, select the time and the quantity to load in T2 (kg) knowing that at the end of the load, it does not exceed the maximum capacity of T2.
3. The valve V-114 will open and the storage tank, T2, will be filled.
4. After the T2 filling time, valve V-114 will close.
5. Once the previous action has finished, the valve V-117 must be opened to discharge T2 which it will span 30 min.
6. Simultaneously, it must open the valves V-118 and V-120 (involving V-119 and V-121 have to be close) to push the fluid towards the vessel. Therefore, V-131 must be open too, for charging the vessel which it will span the same as discharge T2, 30 minutes.
7. Upon of vessel completion. Valves V-117, V-118, V-120 and V-131 will be closed.
8. Once finished the reaction, 30 min, valve V-140 will open to empty the reactor and, by gravity, the mixture will go to S-01.



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CONTROL RECIPIE BIODIESEL PRODUCTION PLANT TRANSESTERIFICATION SECTION	
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P&ID BIO DIESEL PRODUCTION PLANT TRANSESTERIFICATION SECTION
DATE MAY 2017
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6. EQUIPMENT LIST

Equipment illustrated in P&IDs are shown hereunder:

Item	Service	Type
Agitators		
A-01	V-01 Agitator	Propeller stirrer
A-02	V-02 Agitator	Propeller stirrer
Heat exchanger		
E-01	DSO heater	BEM
Pumps		
P-101a/b	DSO storage tank output pump	Centrifugal
P-102a/b	MeOH storage tank output pump	Centrifugal
P-103a/b	SM 2% storage tank output pump	Centrifugal
P-104a/b	1st stage biodiesel pump	Centrifugal
P-105a/b	2nd stage biodiesel pump	Centrifugal
P-106a/b	1st stage glycerine pump	Centrifugal
P-107a/b	2nd stage glycerine pump	Centrifugal
Settlers		
S-01	1st stage settling	Settler
S-02	2nd stage settling	Settler
Vessels		
V-01	1st stage transesterification reactor	-
V-02	2nd stage transesterification reactor	-

Table 13. Transesterification P&ID.

Moreover, specification sheets can be found in Appendix 3.

7. SCHEDULING

Once known in depth the process, it has made the equipments sizing and it has decided which tasks will be carried out in each, it is time to set the pace of production to achieve the annual of product to produce.

7.1. GENERAL CONSIDERATIONS

Scheduling is a critical issue in process operations and is crucial for improving production performance. For batch processes, short-term scheduling deals with the allocation of a set of limited resources over time to manufacture one or more products following a batch recipe.

There are a great variety of aspects that need to be considered when developing scheduling models for batch processes. In order to provide a systematic characterization a general roadmap for classifying most relevant problem features is summarized in Table 14 and considers not only equipment and material issues, but also time and demand-related constrains. As can be seen, the main features involve 13 major categories, each of which are linked to central problem characteristics. ⁽¹⁹⁾ The results of the application of the roadmap in this project is showed in the same Table 14.

It should be stressed that the annual production of 30,000 tonnes of refined biodiesel correspond to the annual mass balance, that it involves that 30,764 tonnes of non-refined biodiesel (NRB) are required in the annual production of the transesterification stage.

Although the purification process of the biodiesel and the methanol recovery are carried out continuously (330 days per year, 24 h/day), the transesterification section is carried out per batch. It is proposed to work 5 days per week in 3 shifts per day and a total of 47 weeks per year. Therefore, the weekends will only work the purification section and the methanol recovery.

PROCESS TOPOLOGY	Sequential. Single product. Dedicated Batch Plant
EQUIPMENT ASSIGNMENT	Fixed
EQUIPMENT CONNECTIVITY	Full. It has been assumed it is able to interconnect all equipment needed.
INVENTORY STORAGE POLICIES	Finite Intermediate Storage (FIS). The storage tanks have a specific volume.
	Zero Wait (ZW). The rest of the process steps must occur one after another one, with no possibility to stop the process between stages.
MATERIAL TRANSFER	Time consuming. Pipes. There are available all needed pipes to transfer the reagents and the products from one stage to another one.
BATCH SIZE	Fixed. It has been fixed a batch size in 21,000 kg of NRB.
BATCH PROCESSING TIME	Fixed. Unit dependent. Batch processing time is fixed and it depends only on the units.
DEMAND PATTERNS	Scheduling horizon. Fixed requirements. It has set an annual production target, 30,000 tons of Biodiesel
CHANGE OVERS	None It has not had in account.
RESOURCE CONSTRAINS	None (only equipment). It has been assumed not to have limitations of raw materials and energy. Only limits the operation of process equipment.
TIME CONSTRAINS	Non-working periods. It has been assumed that the production is possible for 24 hours, five days a week, 47 weeks by year. It means that weekends are non-working periods.
COST	None. It has not taken in account.
DEGREE OF CERTAINLY	None. It has not taken in account.

Table 14. Roadmap for scheduling in the transesterification stage.

The difficulty is finding a batch size it allows to get 30,764 t/y of NRB. To get it, an iteration is required to find a suitable batch size for the required working conditions. So, it is necessary to determine the batch size. Stressing that roadmap is an iterative process, firstly, a value is assumed. Secondly, the scheduling resolution is carried out and, finally it is checked if the obtained values fulfil all the problem restrictions. After a study, the batch size of 20,970 kg of NRB per batch was chosen. In addition, it will allow to work with more flexibility as it will be shown later.

7.2. BATCH TIME

In this section, it will show the batch time for this kind of production. The following table shows the estimated time duration of each stage considering the filling and emptying of each equipment.

	Stage / Equipment	Time [h]
1	Charge V-01	0.5
2	Reaction V-01	0.5
3	Discharge V-01 / Charge S-01	1.0
4	Settling S-01	1.0
5	Discharge S-01 / Charge T4	0.25
6	Discharge S-01 / Charge V-02	0.75
7	Reaction V-02	0.5
8	Discharge V-02 / Charge S-02	1.0
9	Settling S-02	1.0
10	Discharge S-02 / Charge T4	0.25
11	Discharge S-02 / Charge T5	0.75

Table 15. Stages time.

From these times, it is estimated occupation time (OT_i) of each equipment stage and the process limiting stage is determined. Where t' is absolute time and OT_i is equipment operation time.

Equipment	t' [h]		OT_i [h/batch]
	Start	End	
V-01	0	2	2
S-01	1	4	3
T4	3	3.25	0.25
V-02	3.25	5.5	2.25
S-02	4.5	7.5	3
T4	6.5	6.75	0.25
T5	6.75	7.50	0.75

Table 16. Occupation time of equipments.

As it can be seen, the limiting required for biodiesel production per batch is 7.5. In addition, the limiting stage of the process is the settler step. This time is known as cycle time (CT) and its value is 3h.

In any case, knowing the batch time and the time required for each stage it is possible to make a Gantt Diagram displayed below.

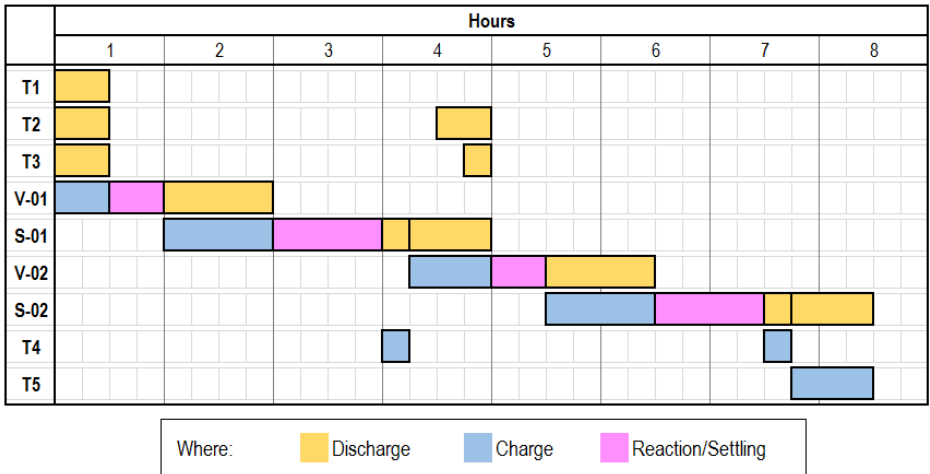


Figure 9. Batch time.

7.3. PRODUCTION CAPACITY

The production capacity has two alternatives:

- Producing in campaign, namely, working with overlapping.
- Producing in campaign with non-overlapping.

A study has been made to know which methodology is the most suitable for this process.

7.3.1. Production in non-overlapping campaign

Production in non-overlapping campaign consists to produce a batch after another one successively. Meaning the second batch will not start if the first one has not finished yet.

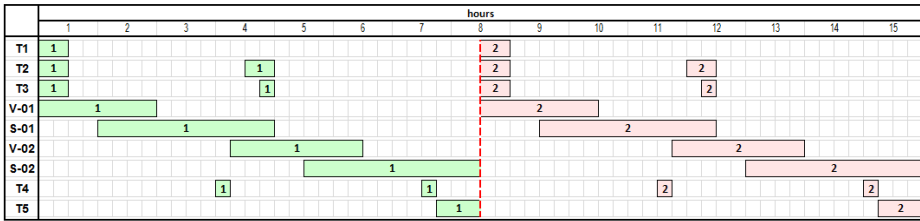


Figure 10. Production in non-overlapping campaign.

In this case, production time of a biodiesel batch matches the duration of the cycle. In consequence:

$$MS_N = BT \cdot N$$

$$N = \frac{MS_N}{BT} = \frac{7,920 \text{ h/year}}{7.5 \text{ h/batch}} = 1,056 \text{ batches/year}$$

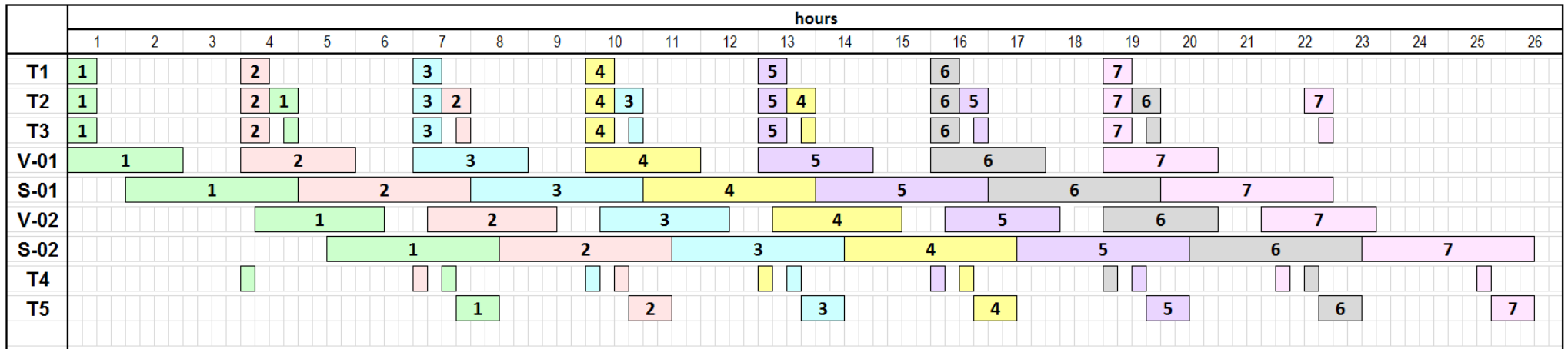
$$1,056 \frac{\text{batches}}{\text{year}} \cdot \frac{20,970 \text{ kg}}{\text{batch}} \cdot \frac{1 \text{ tone}}{1,000 \text{ kg}} = 22,144 \text{ tone/year}$$

It will not be able to work with non-overlapping campaign because of the main aim is to produce 30,000 t/y of RB. Henceforth, it is not a possibility to work with non-overlapping campaign.

This is not the best operating method due to the dead times of the equipments are very raised, as it can be seen in the previous figure.

7.3.2. Production in overlapping campaign

Production in overlapping campaign consists in optimizing the production time. Next batch can start although the last one has not finished. In such a way to produce a batch after another one successively. Meaning the second batch can start although the first one has not finished. In this way, the batches are overlapping, considering that between batches should leave enough time's space lest limiting stage is not be a problem.



Batch	Time [h]	
	Start	End
1	0	7.5
2	4	11.5
3	7	14.5
4	10	17.5
5	13	20.5
6	16	23.5
7	19	26.5

Figure 11. Production in overlapping campaign.

7.4. HIGHEST CAPACITY

Highlight that optimizing time is seen the first reactor charge in the second batch, it begins before that the second reactor charge in the first batch.

As it has been commented before, CT is “cycle time” and it refers to the longer time per batch in biodiesel production. In consequence:

$$MS_N = BT + (N - 1) \cdot CT$$

$$N = \frac{MS_N - BT}{CT} + 1 = \frac{7,920 \text{ h/year} - 7.5 \text{ h/batch}}{3 \text{ h/batch}} + 1 = 2,638 \text{ batches/year}$$

$$2,638 \frac{\text{batches}}{\text{year}} \cdot \frac{20,970 \text{ kg}}{\text{batch}} \cdot \frac{1 \text{ tone}}{1,000 \text{ kg}} = 52,760 \text{ tone/year}$$

It will be able to work with overlapping campaign because one of the main aim is to produce 30,764 t/y of non-refined biodiesel and it is being obtained 53,000 t/y roughly. So, working 330 days per year, 7 days by week, the maximum capacity of the plant will be 53,000 t/y of NRB.

The difficulty is producing 30,000 t/y of RB, involving 30,764 t/y NRB. If the plant works 7 days by week (highest capacity), it is required:

$$30,764 \frac{\text{t}}{\text{y}} \cdot \frac{1 \text{ y}}{7,920 \text{ h}} \cdot \frac{24 \text{ h}}{1 \text{ day}} \cdot \frac{7 \text{ days}}{1 \text{ week}} = 653 \text{ t/w}$$

To produce 653 t/w it needs:

$$653 \frac{\text{t}}{\text{w}} \cdot \frac{1,000 \text{ kg}}{1 \text{ t}} \cdot \frac{1 \text{ batch}}{20,970 \text{ kg}} = 31.14 \frac{\text{batches}}{\text{week}}$$

By contrast, as it has been commented before. The transesterification stages works 5 days per week with a batch size of 20,970 kg/batch of NRB, producing annually 30,764 t/y of NRB, it is required:

$$5 \frac{\text{days}}{\text{week}} \cdot \frac{24 \text{ h}}{1 \text{ day}} = 120 \frac{\text{h}}{\text{d}}$$

$$N = \frac{MS_N - BT}{CT} + 1 = \frac{120 - 7.5}{3} + 1 = 38 \text{ batches/week}$$

$$38 \frac{\text{batch}}{\text{week}} \cdot \frac{20,970 \text{ kg}}{1 \text{ batch}} \cdot \frac{1 \text{ t}}{1,000 \text{ kg}} = 796.86 \text{ t/w}$$

Working 7 days per week is required 653 t/w while working 5 days is produced 796.86 t/y. To set up, there is an overcapacity as it can be seen in the previous calculations. Overcapacity is related to an excess of time. This excess can be destined, for example, in case of incidence it will have the necessary time to act.

So, working 5 days per week, 47 weeks per year means that it needs 1467 batch/year to carry out 30,764 of NRB.

7.5. DETERMINATION OF THE OPERATION STRATEGY

It will work on overlapping campaign, to a maximum of 120 hours per week. It is not going to work at the maximum capacity of the plant. It is decided the plant must have one day of safety stock. This represents a restricción against the model. This safety stock will be 94 tonnes. It is not going to be below that amount.

In normal conditions to produce 653 tonnes per week, 31.9 batches are needed, so there are two options. The transesterification stage can either produce 31 batches or 32.

It is observed that working with 32, production exceeds (causing an increase in the security stock), and 31 batches, the required production is reached (the safety stock is consumed). Therefore, both options have to be blended throughout the year.

Two production possibilities will be studied:

- Working in a single campaign per week with 31/32 batches.
- Working in two campaigns per week with an intermediate shutdown, with 31/32 batches per week.

It should be highlighted that the following sections is going to be discussed the determination of the operation mode of the NRB storage tank, as an example. Thus, it will be possible to know the tank volume. Once the T5 sizing has been determined, the T4 sizing is going to be commented.

7.5.1. Working in one campaign

The following graphics show the variations of NRB required, NRB production and NRB stock in tonnes. It is working in a campaign with 32 and 31 batches per week.

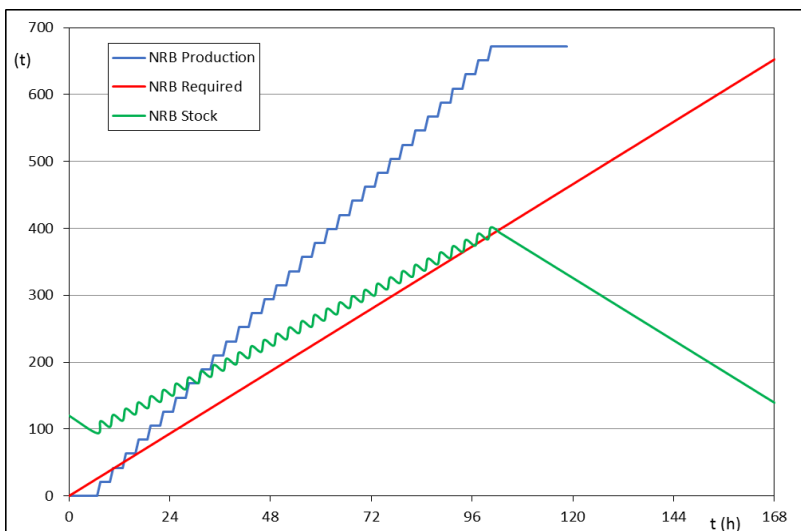


Figure 12. 32 batches per week of NRB.

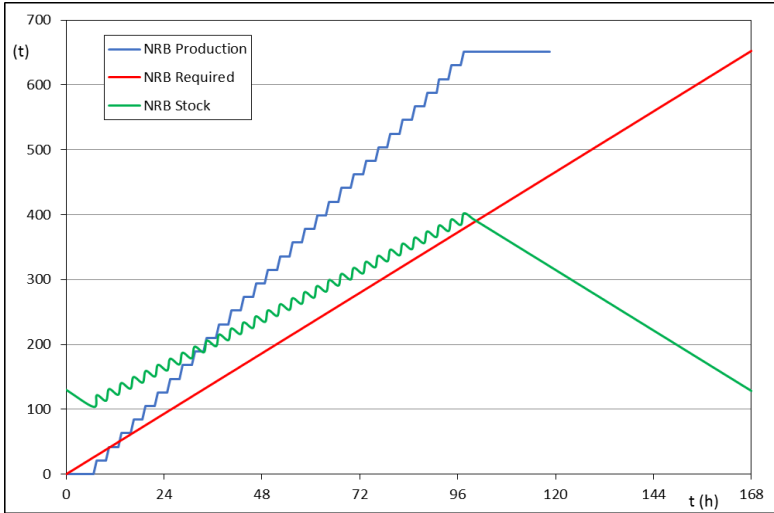


Figure 13. 31 batches per week of NRB.

If the stock line is observed, the NRB stock, and therefore, the volume to be stored is very high. The storage tank should be designed to have a maximum NRB stock of 400 tonnes per week. If it is related to density, the required volume would be approximately 455 m³ in both cases.

On the other hand, the maximum time worked is 120 hours. Working on a campaign of 31 and 32 batches, effective process time and non-effective process time are shown below:

	MS₃₂	MS₃₁
Effective process time [h]	100.5	97.7
Non-effective process time [h]	19.5	22.5

Table 17. Effective and non-effective process time in one campaign.

Non-effective process time can be used for cleaning and maintenance work. Note that from 98 hours, at the beginning of the fifth day, the NRB production is stopped and the NRB stock is started to be consumed.

7.5.2. Working in two campaign

In this case, it is worked in two campaigns, the first is with 15 and 17 batches (MS_{15}/MS_{17}), and the second with 15 and 16 batches (MS_{15}/MS_{16}). It must be taken into account that if campaigns increase, the make span is greater, therefore more time is lost. Hereunder, the effective and non-effective process time is shown:

	MS_{15}/MS_{17}	MS_{15}/MS_{16}
Effective process time [h]	105	102
Non-effective process time [h]	15	18

Table 18. Effective and non-effective process time in two campaigns.

It has been decided that the intermediate shutdown will be 10 hours. In the case of producing 31 batches (MS_{15}/MS_{16}), it will remain 5 hours for maintenance and cleaning at the end of the campaign and, in the case of 32 batches (MS_{15}/MS_{17}), it will remain 8 hours for maintenance and cleaning.

In the same way as working in one campaign, the NRB production, NRB required and NRB stock working in two campaigns are shown:

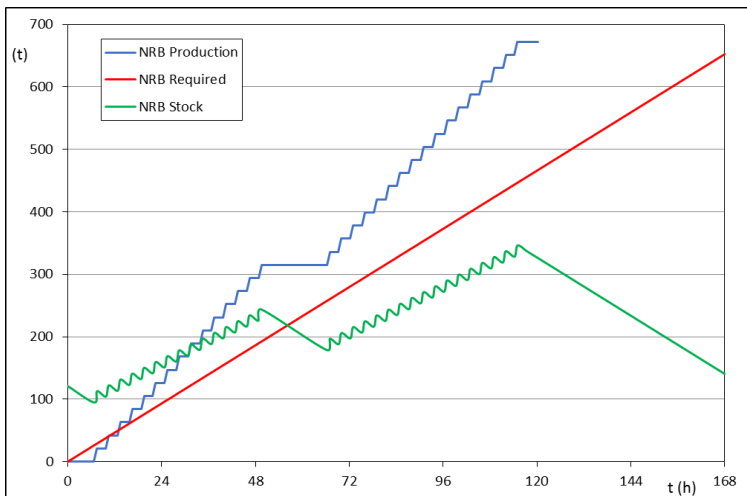


Figure 14. Weekly production of 15 and 17 batches of NRB with an intermediate shutdown.

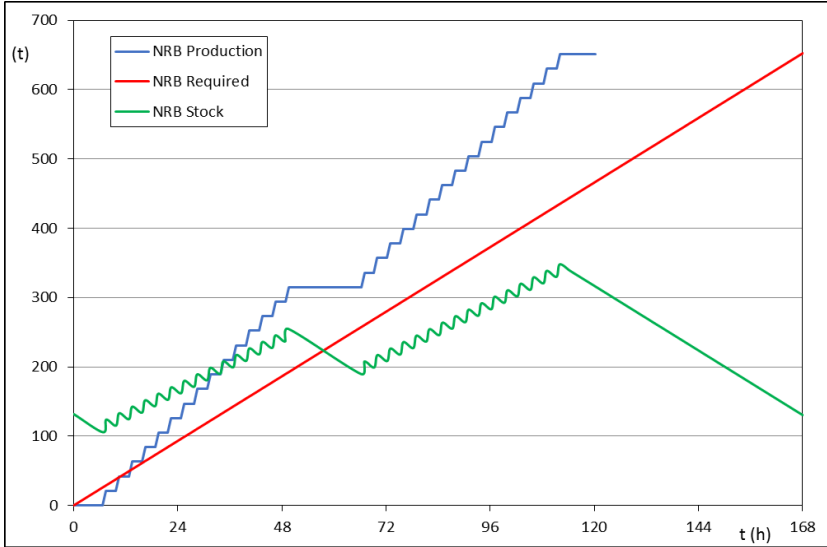


Figure 15. Weekly production of 15 and 16 batches of NRB with an intermediate shutdown.

As documented in the above figure, if the stock line is observed, the storage tank should be designed to keep a maximum of 356 NRB tonnes, implying a volume of 405 m³. As it has been discussed previously, the NRB amount to be stored has decreases. That is the reason because of the T5 size is 405 m³.

In both graphics can be observed that the intermediate shutdown will occur at 49.5 hours (at the end of the first campaign). In other words, after 2 days, the hour and a half of beginning the third day. As the intermediate stop has been set at 10 hours, it will end at 5.30 pm on the 3rd day, when the second shift ends.

As a summary, it should be noted that working in two campaigns:

- At the capacity level, it is observed that the NRB volume stored is less than working in one campaign.
- Provides more flexibility.

Finally, note that there is no single solution, only one of the best has been found. However, one of the best solutions for doing the shut down is when:

- It must be done between the first and second shift because of there are more people in the plant.
- Productions per campaign make up for reducing the maximum stock.

Non-refined glycerine storage tank

The non-refined glycerine (NRG) storage tank, T4, can be established in the same way as the optimization of NRB storage tank has been done. The difference is that, now, the values are determined by what it has been established for NRB fact. In other words, it will not be compared working with one or two campaigns because it has already been decided that it will work in two campaigns.

Likewise, the safety stock should always be above one day as for NRB tank, but, for NRG is 31 tonnes. The NRG storage tank, T4, stores less matter, for that reason, the safety stock is going to be smaller than T5.

In the same way as working in two campaigns for NRB, the NRG production, NRG required and NRG stock.

As documented in the next figures, if the stock line is observed, the NRG storage tank should be designed to store a maximum of 126 tonnes, implying a volume of 130 m³.

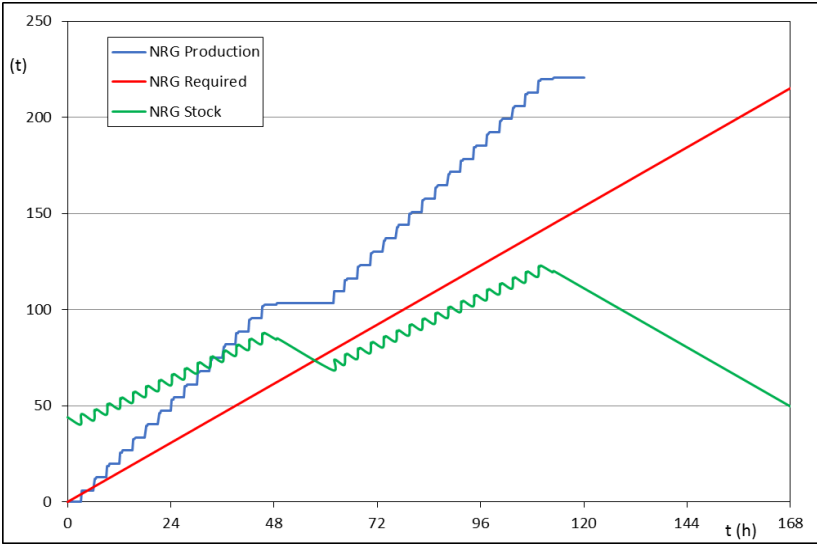


Figure 16. Weekly production of 15 and 17 batches of NRG with an intermediate shutdown.

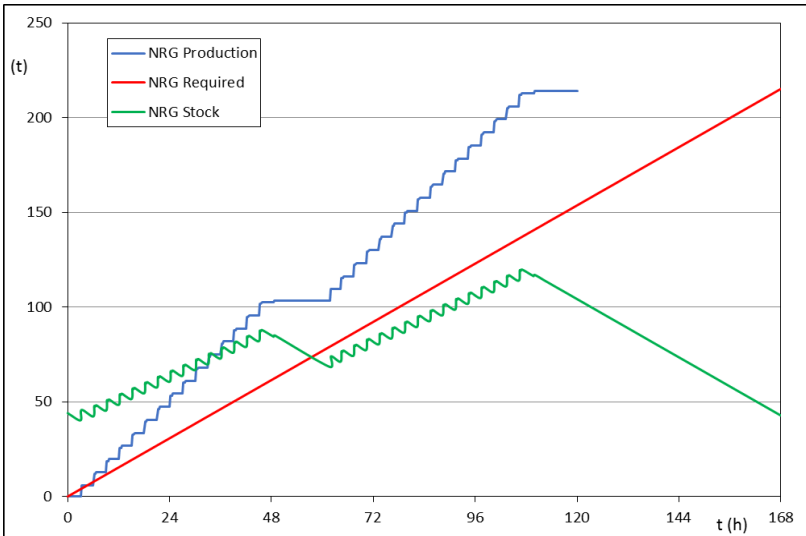


Figure 17. Weekly production of 15 and 16 batches of NRG with an intermediate shutdown.

8. CONCLUSIONS

The transesterification stage in the process of obtaining biodiesel has been studied and designed. Although, the overall production process is continuously, it has been shown that the transesterification step can work per batches as long as the production volumes will be low.

Quantities in mass of the different raw materials needed to produce 30 thousand tonnes per year of refined biodiesel, are shown in the overall mass balance of the process (Figure & Table 3).

Transesterification stage will consist mainly of two vessels and two settlers, with the necessary pumps, valves and pipes have been chosen, as well as the automation and control system that is presented in the corresponding in the P&ID.

All the equipment was sized and specified with the exception of pumps and coalescer which are designed by vendor.

Piping and Instrumentation Diagram (P&ID) as well as the block and process diagram were drawn using AutoCad software.

Working in two campaigns with overlapping, batch time and cycle time have been determined. In this case, the batch time is 7.5 (BT = 7.5 h) and the cycle time is 3 hours (CT = 3h).

The most convenient has been the weekly production of 31 or 32 batches, in two campaigns of 15 and 16 batches, the first one, and 15 and 17 batches, the second one. In addition, even if the purification stage and the recovery methanol excess operates continuously without disruption, the transesterification stage will work only 5 days per week, with 3 shifts per day, without working the weekends.

Working with two campaigns per week, necessarily involves an intermediate shutdown, which it will be used for cleaning work and maintenance. Moreover, this shutdown has been programmed in such a way that it takes place coinciding with the central shift of the plant.

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ACRONYMS

μ	Absolute (Dynamic) viscosity [$\text{kg}\cdot\text{m}^{-1}\cdot\text{s}^{-1}$]
$^{\circ}\text{C}$	Celsius
$^{\circ}\text{F}$	Faraday
λ	Latent heat [J/kg]
σ_{LE}	Yield strength [Pa]
ΔP	Pressure drop [Pa]
ΔT	Temperature difference [K]
ΔT_{ml}	Average temperature logarithmic [K]
ρ	Weight density of fluid [kg/m^3]
A	Area [m^2]
BT	Batch time
BS	Batch size
BTU	British thermal unit
CT	Cycle time [h]
C_p	Calorific capacity [$\text{J}/(\text{kg}\cdot\text{k})$]
DG	Diglyceride
DSO	Degummed soybean oil
dext	External diameter [m]
dint	Internal diameter [m]
D	diameter [m]
e	Thickness [m]

E-01	Heat-Exchanger
FFA	Free Fatty Acids
FIS	Finite Intermediate Storage
ft	feet
gal	Gallon
GHG	Green – House Gas
h	height [m]
HDV	Charge and discharge valve
lb	Pounds
L	Length [m]
LT	Level Transmit
LI	Level Indicate
MG	Monoglyceride
ME	Methyl ester
MeOH	Methanol
MS _n	Make span [h]
NEB	Net Energy Balance
N _p	Power's number
N	Turn speed [rpm]
NSPH	Net Positive Suction Head
NRB	Non-Refined Biodiesel
OT _i	Equipment operating time
P	Power [W]
P	Equipment Pump
P&ID	Piping and Instrumentation Diagram
PT	Pressure Transmit
PI	Pressure Indicate
PSV	Pressure Switch Valve

Q	Exchanged heat [W]
Re	Reynold's number [dimensionless]
R	Radius [m]
RT	Rotation Transmit
RB	Refined Biodiesel
SM	Sodium methoxide
S	Section [m ²]
S-01	First settler
S-02	Second settler
TG	Triglyceride
TEMA	Tubular Exchanger Manufacturers Association
TDH	Total Dynamic Head
t	tonne
t'	absolute time [s]
T	Temperature [°C]
TT	Temperature Transmit
TI	Temperature Indicate
T1	Soybean oil storage process tank
T2	Methanol storage process tank
T3	Sodium methoxide 2% storage process tank
T4	Non-refined glycerine storage tank
T5	Non-refined biodiesel storage tank
U	Heat Transfer Coefficient [W/(m ² ·K)]
V	Volume [m ³]
V-01	First vessel
V-02	Second vessel
W	Watts [J/s]

WT	Weight Transmit
WI	Wight Indicate
y	year
ZW	Zero Wait

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Block flow diagram

Process flow diagram










Control recipe

P&ID

APPENDICES

APPENDIX 1: SAFETY DATA SHEETS

Fichas Internacionales de Seguridad Química












METANOL		ICSC: 0057 Abril 2000	
Alcohol metílico		Carbínol	
CAS:	67-56-1	CH ₂ O / CH ₂ OH	 
RTECS:	PC1400000	Masa molecular: 32.0	
NU:	1230		
CE Índice Anexo I:	603-001-00-X		
CE / EINECS:	200-659-6		
TIPO DE PELIGRO / EXPOSICIÓN	PELIGROS AGUDOS / SÍNTOMAS	PREVENCIÓN	PRIMEROS AUXILIOS / LUCHA CONTRA INCENDIOS
INCENDIO	Altamente inflamable. Ver Notas.	Evitar las llamas, NO producir chispas y NO fumar. NO poner en contacto con oxidantes.	Polvos, espuma resistente al alcohol, agua en grandes cantidades, dióxido de carbono.
EXPLOSIÓN	Las mezclas vapor/aire son explosivas.	Sistema cerrado, ventilación, equipo eléctrico y de alumbrado a prueba de explosión. NO utilizar aire comprimido para llenar, vaciar o manipular. Utilícense herramientas manuales no generadoras de chispas.	En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.
EXPOSICIÓN		¡EVITAR LA EXPOSICIÓN DE ADOLESCENTES Y NIÑOS!	
Inhalación	Tos. Vértigo. Dolor de cabeza. Náuseas. Debilidad. Alteraciones de la vista.	Ventilación. Extracción localizada o protección respiratoria.	Aire limpio, reposo. Proporcionar asistencia médica.
Piel	¡PUEDE ABSORBERSE! Piel seca. Enrojecimiento.	Gautes de protección. Traje de protección.	Quitar las ropas contaminadas. Aclarar con agua abundante o ducharse. Proporcionar asistencia médica.
Ojos	Enrojecimiento. Dolor.	Gafas ajustadas de seguridad, o protección ocular combinada con la protección respiratoria.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
Ingestión	Dolor abdominal. Jadeo. Vómitos. Convulsiones. Pérdida del conocimiento (para mayor información, véase Inhalación).	No comer, ni beber, ni fumar durante el trabajo. Lavarse las manos antes de comer.	Provocar el vómito (¡UNICAMENTE EN PERSONAS CONSCIENTES!). Proporcionar asistencia médica.
DERRAMES Y FUGAS		ENVASADO Y ETIQUETADO	
Evacuar la zona de peligro. Ventilar. Recoger el líquido procedente de la fuga en recipientes precintables. Eliminar el residuo con agua abundante. Eliminar vapor con agua pulverizada. Traje de protección química, incluyendo equipo autónomo de respiración.		No transportar con alimentos y piensos. Clasificación UE Símbolo: F, T R: 11-23/24/25-39/23/24/25; S: (1/2)-7-19-36/37-45 Clasificación NU Clasificación de Peligros NU: 3 Riesgos Subsidiarios de las NU: 6.1; Grupo de Envasado NU: II	
RESPUESTA DE EMERGENCIA		ALMACENAMIENTO	
Ficha de emergencia de transporte (Transport Emergency Card): TEC (R)-30S1230. Código NFPA: H 1; F 3; R 0;		A prueba de incendio. Separado de oxidantes fuertes, alimentos y piensos. Mantener en lugar fresco.	
      	Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © IPCS, CE 2000		

VÉASE INFORMACIÓN IMPORTANTE AL DORSO

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METANOL		ICSC: 0057
DATOS IMPORTANTES		
<p>ESTADO FÍSICO; ASPECTO: Líquido incoloro, de olor característico.</p> <p>PELIGROS FÍSICOS: El vapor se mezcla bien con el aire, formándose fácilmente mezclas explosivas.</p> <p>PELIGROS QUÍMICOS: Reacciona violentamente con oxidantes, originando peligro de incendio y explosión.</p> <p>LÍMITES DE EXPOSICIÓN: TLV: 200 ppm como TWA, 250 ppm como STEL; (piel); BEI establecido (ACGIH 2004). MAK: Riesgo para el embarazo: grupo (DFG 2004). LEP UE: 200 ppm; 260 mg/m³ como TWA (piel) como TWA (UE 2006).</p>	<p>VIAS DE EXPOSICIÓN: La sustancia se puede absorber por inhalación, a través de la piel y por ingestión.</p> <p>RIESGO DE INHALACIÓN: Por evaporación de esta sustancia a 20 °C se puede alcanzar bastante rápidamente una concentración nociva en el aire.</p> <p>EFFECTOS DE EXPOSICIÓN DE CORTA DURACIÓN: La sustancia irrita los ojos la piel y el tracto respiratorio. La sustancia puede afectar al sistema nervioso central, dando lugar a pérdida del conocimiento. La exposición puede producir ceguera y muerte. Los efectos pueden aparecer de forma no inmediata.</p> <p>EFFECTOS DE EXPOSICIÓN PROLONGADA O REPETIDA: El contacto prolongado o repetido con la piel puede producir dermatitis. La sustancia puede afectar sistema nervioso central, dando lugar a dolores de cabeza persistentes y alteraciones de la visión.</p>	
PROPIEDADES FÍSICAS		
<p>Punto de ebullición: 65 °C Punto de fusión: -98 °C Densidad relativa (agua = 1): 0.79 Solubilidad en agua: miscible Presión de vapor, kPa a 20 °C: 12.3 Densidad relativa de vapor (aire = 1): 1.1</p>	<p>Densidad relativa de la mezcla vapor/aire a 20 °C (aire = 1): 1.01 Punto de inflamación: 12 °C c.c. Temperatura de autoignición: 464 °C Límites de explosividad, % en volumen en el aire: 5.5-44 Coeficiente de reparto octanol/agua como log Pow: -0.82/-0.66</p>	
DATOS AMBIENTALES		
NOTAS		
Arde con llama azulada. Está indicado examen médico periódico dependiendo del grado de exposición. Esta ficha ha sido parcialmente actualizada en octubre de 2006: ver Límites de exposición.		
INFORMACIÓN ADICIONAL		
Límites de exposición profesional (INSHT 2011):		
VLA-ED: 200 ppm; 266 mg/m ³		
Notas: vía dérmica.		
VLB: 15 mg/L en orina. Notas F, I.		
Nota legal	Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.	
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METANOLATO DE SODIO			ICSC: 0771 Octubre 2006				
CAS: RTECS: NU: CE Índice Anexo I: CE / EINECS:	124-41-4 PC3570000 1431 603-040-00-2 204-699-5	Metóxido sódico Metilato sódico CH ₃ ONa Masa molecular: 54,0					
TIPO DE PELIGRO / EXPOSICIÓN	PELIGROS AGUDOS / SÍNTOMAS	PREVENCIÓN	PRIMEROS AUXILIOS / LUCHA CONTRA INCENDIOS				
INCENDIO	Altamente inflamable. Muchas reacciones pueden producir incendio o explosión.	Evitar las llamas, NO producir chispas y NO fumar. NO poner en contacto con agua.	NO utilizar agua. NO utilizar agentes hídricos. Polvo. Arena seca.				
EXPLOSIÓN	Riesgo de incendio y explosión en contacto con. agua, aire húmedo, metales.		En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua pero NO en contacto directo con agua.				
EXPOSICIÓN		¡EVITAR TODO CONTACTO!	¡CONSULTAR AL MÉDICO EN TODOS LOS CASOS!				
Inhalación	Dolor de garganta. Tos. Sensación de quemazón. Dificultad respiratoria. Jadeo.	Extracción localizada. Protección respiratoria.	Aire limpio. reposo. Posición de semincorporado. Respiración artificial si estuviera indicada. Proporcionar inmediatamente asistencia médica.				
Piel	Enrojecimiento. Dolor. Quemaduras cutáneas graves.	Guantes de protección. Traje de protección.	Aclarar con agua abundante durante 15 minutos como mínimo, después quitar la ropa contaminada y aclarar de nuevo. Proporcionar inmediatamente asistencia médica.				
Ojos	Enrojecimiento. Dolor. Visión borrosa. Quemaduras profundas graves.	Pantalla facial y protección ocular combinada con protección respiratoria.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad). Proporcionar asistencia médica inmediatamente.				
Ingestión	Quemaduras en la boca y la garganta. Sensación de quemazón en la garganta y el pecho. Shock o colapso.	No comer, ni beber, ni fumar durante el trabajo.	Enjuagar la boca. NO provocar el vómito. Proporcionar asistencia médica inmediatamente.				
DERRAMES Y FUGAS		ENVASADO Y ETIQUETADO					
Eliminar toda fuente de ignición. Evacuar la zona de peligro. Consultar a un experto. Protección personal adicional: traje de protección completa incluyendo equipo autónomo de respiración. Cubrir el material derramado con arena seca. Barrer la sustancia derramada e introducirla en un recipiente seco tapado de plástico. Recoger cuidadosamente el residuo, trasladarlo a continuación a un lugar seguro. NO verterlo en el alcantarillado.		Hermético. Envase irrompible; colocar el envase frágil dentro de un recipiente irrompible cerrado. No transportar con alimentos y piensos. Clasificación UE Símbolo: F, C R: 11-14-34 S: (1/2)-8-16-26-43-45 Clasificación NU Clasificación de Peligros NU: 4.2 Riesgos Subsidiarios de las NU: 8 Grupo de Envasado NU: II Clasificación GHS Peligro Se calienta espontáneamente; puede inflamarse. Provoca graves quemaduras en la piel y lesiones oculares.					
RESPUESTA DE EMERGENCIA		ALMACENAMIENTO					
		Mantener en lugar fresco y seco. A prueba de incendio. Separado de oxidantes fuertes, ácidos, metales y de alimentos y piensos. Bien cerrado. Almacenar en un área con suelo de hormigón resistente a la corrosión. Almacenar en un área sin acceso a desagües o alcantarillas.					
Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © CE, IPCS, 2007							
 IPCS International Programme on Chemical Safety		 WHO	 ILO	 UNEP		 MINISTERIO DE TRABAJO Y EMIGRACION	 INTERNACIONAL DE EMIGRANTES DEL TRABAJO

VÉASE INFORMACIÓN IMPORTANTE AL DORSO

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METANOLATO DE SODIO		ICSC: 0771
DATOS IMPORTANTES		
<p>ESTADO FÍSICO: ASPECTO Polvo higroscópico, blanco.</p> <p>PELIGROS QUÍMICOS El calentamiento intenso puede originar combustión violenta o explosión. Reaccionaviolentemente con agua produciendo metanol inflamable e hidróxido sódico corrosivo. La sustancia puede inflamarse espontáneamente en contacto con aire húmedo. La sustancia es un agente reductor fuerte y reacciona violentamente con oxidantes. La sustancia es una base fuerte, reacciona violentamente con ácidos y es corrosiva. Ataca a muchos metales formando un gas combustible (hidrógeno - ver FISQ.0001).</p> <p>LÍMITES DE EXPOSICIÓN TLV no establecido. MAK no establecido.</p>	<p>VÍAS DE EXPOSICIÓN Efectos locales graves por todas las vías de exposición.</p> <p>RIESGO DE INHALACIÓN Puede alcanzarse rápidamente una concentración nociva de partículas suspendidas en el aire cuando se dispersa.</p> <p>EFFECTOS DE EXPOSICIÓN DE CORTA DURACIÓN La sustancia es corrosiva para los ojos, la piel y el tracto respiratorio. Corrosivo por ingestión. La inhalación puede causar edema pulmonar, pero sólo tras producirse los efectos corrosivos iniciales en los ojos o las vías respiratorias.</p>	
PROPIEDADES FÍSICAS		
<p>Se descompone a > 50°C. Densidad: 1,3 g/cm³</p> <p>Solubilidad en agua: reacciona</p>	<p>Temperatura de autoignición: >50°C Límites de explosividad, % en volumen en el aire: 7,3 - 36</p>	
DATOS AMBIENTALES		
<p>Esta sustancia puede ser peligrosa para el medio ambiente; debe prestarse atención especial a los organismos acuáticos</p>		
NOTAS		
<p>Esta sustancia es un sólido muy reactivo y es manipulado en disolución en la mayoría de casos. El sólido es muy higroscópico y se descompone rápidamente. Solo es estable en ausencia de aire y humedad. Reacciona violentamente con agentes extintores de incendio tales como agua. Enjuagar la ropa contaminada con agua abundante (peligro de incendio). Otro número NU es NU 1289 Solución de metilato sódico en alcohol, clasificación de peligro: 3, riesgo subsidiario: 8, grupo de envasado: II, III. Esta ficha ha sido parcialmente actualizada en abril de 2010; ver Explosión.</p>		
INFORMACIÓN ADICIONAL		
NOTA LEGAL	<p>Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.</p>	
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Fichas Internacionales de Seguridad Química

GLICEROL		ICSC: 0624
		Abril 2006
Glicerina 1,2,3-Propanotriol 1,2,3-Trihidroxipropano		
CAS:	56-81-5	C₃H₈O₃ / CH₂OH-CHOH-CH₂OH
RTECS:	MA8050000	Masa molecular: 92,1
CE / EINECS:	200-289-5	

TIPO DE PELIGRO / EXPOSICIÓN	PELIGROS AGUDOS / SÍNTOMAS	PREVENCIÓN	PRIMEROS AUXILIOS / LUCHA CONTRA INCENDIOS
INCENDIO	Combustible. En caso de incendio se desprenden humos (o gases) tóxicos e irritantes.	Evitar las llamas.	Agua pulverizada. Espuma resistente al alcohol. Polvo. Dióxido de carbono.
EXPLOSIÓN			En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.

EXPOSICIÓN			
Inhalación		Ventilación.	Aire limpio, reposo.
Piel	Piel seca.	Guantes protectores.	Aclarar la piel con agua abundante o ducharse.
Ojos		Gafas ajustadas de seguridad.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
Ingestión	Diarrea.	No comer, ni beber, ni fumar durante el trabajo.	Enjuagar la boca.

DERRAMES Y FUGAS	ENVASADO Y ETIQUETADO
Ventilar. Recoger el líquido procedente de la fuga en recipientes tapados. Absorber el líquido residual en arena o absorbente inerte y trasladarlo a un lugar seguro.	
RESPUESTA DE EMERGENCIA	ALMACENAMIENTO
Código NFPA: H1; F1; R0;	Separado de oxidantes fuertes.

IPCS

International
Programme on
Chemical Safety

Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © CE, IPCS, 2006

VÉASE INFORMACIÓN IMPORTANTE AL DORSO

Fichas Internacionales de Seguridad Química

GLICEROL

ICSC: 0624

DATOS IMPORTANTES

ESTADO FÍSICO; ASPECTO:

Líquido viscoso, higroscópico e incoloro.

PELIGROS QUÍMICOS:

La sustancia se descompone al calentaría intensamente, produciendo humos corrosivos de acroleína. Reacciona con oxidantes fuertes originando peligro de incendio y explosión.

LÍMITES DE EXPOSICIÓN:TLV: niebla 10 mg/m³ como TWA; (ACGIH 2005).
MAK: 50 mg/m³ (fracción inhalable); Categoría de limitación de pico: I(2); Riesgo para el embarazo: grupo C (DFG 2006).**RIESGO DE INHALACIÓN:**

La evaporación a 20 °C es despreciable; sin embargo, se puede alcanzar rápidamente una concentración molesta de partículas en el aire por pulverización.

PROPIEDADES FÍSICAS

Punto de ebullición: 290 °C

Punto de fusión: 18 °C

Densidad relativa (agua = 1): 1,26

Solubilidad en agua: miscible

Presión de vapor, Pa a 25 °C: 0,01

Densidad relativa de vapor (aire = 1): 3,2

Punto de inflamación: 176 °C c.c.

Temperatura de autoignición: 393 °C

Límites de explosividad, % en volumen en el aire: 2,6 - 11,3

Coeficiente de reparto octanol/agua como log Pow: -1,76

DATOS AMBIENTALES

NOTAS

Esta ficha ha sido parcialmente actualizada en agosto de 2007: ver Límites de exposición.

INFORMACIÓN ADICIONAL

Límites de exposición profesional (INSHT 2011):

VLA-ED: niebla 10 mg/m³**Nota legal**

Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.

APPENDIX 2: CALCULATION MANUAL

SIZING OF THE EQUIPMENTS

Following is a manual of calculus to know the size of the both vessels and the agitation system.

V-01 and V-02

If the total mass is known, the volum will known since both magnitudes are related by density. Supposing that the volumens are additives, the volumetric maximum content of the vessel will be the following:

Magnitude	V-01	V-02
Mass [kg]	27152	22024
Volume [m ³]	30.9	25.1

Table 19. Vessel contents.

By heuristics its known that the height should be 1.5 of the diameter. Consequently, the specifications of V-01 and V-02 are:

	V-01	V-02
Diameter [m]	3	3
Section [m²]	7.07	7.07
Height [m]	4.5	4.5
Capacity [m³]	32	32

Table 20. Specifications of both vessels.

The liquid volume is similar to design volume in the case of the reactor. Nonetheless, vessels have at the top and at the bottom a concavity.

This concavity will be free volume which is considered as the excess that the volume must have for safety. Furthermore, it has been decided that both reactors have the same dimensions because it comes out cheaper than if they had different dimensions.

Agitation system

It has been commented in the section of description of the equipments, both of the vessels need an propeller stirrer. The dimensions of the impellers are directly related to the size of the vessels, the length of the propeller is equivalent to 1/3 of the diameter of the container.

The power of agitation has to be calculated, knowing that this magnitude depends of the fluid and the rotational speed. Moreover, due to it is a viscous fluid, the revolutions of the agitator should be low.

First, it is necessary to calculate the Reynold's number, expressed by (where N is turn speed):

$$Re = \frac{\rho \cdot N \cdot D^2}{\mu}$$

Secondly, the power's number (N_p) will be determined. By definition, power's number is directly related by Reynold's number. The parameters b and x depend of the type of circulation's regime:

$$N_p = b \cdot Re^x$$

Regime	b	x
Laminar	35	-1
Turbulent	2	0

Table 21. Agitation's parameters depending of the regime.

Finally, the agitation's power is determined from the following empirical correlation:

$$P = N_p \cdot \rho \cdot D^5 \cdot N^3$$

With all these expressions, the power has been determined. However, as both vessels have the same fluid and dimensions, the agitation system will be the same.

Thus, the characteristics of agitation system are:

Stirrer diameter [m]	1
Height [m]	3
Rotational speed [rpm]	60
Agitation power [kW]	6

Table 22. Characteristics of agitation system.

Jacketed vessels

It has been commented in the section of description of the equipments, both of the vessels need a jacketed to keep the temperature around 60 °C. To be able to initiate the design of the heat interchanger is necessary to know first the necessary heat exchanged and after the area of exchange .

Firstly, the heat interchanger is calculated by next expression:

$$Q = m \cdot C_p \cdot (T_2 - T_1)$$

Secondly, the area of exchange can be calculate by next formula, knowing for water the global heat exchange coefficient must be between 50 – 150 (BTU·°F⁻¹·ft²·h⁻¹) for organic solvents – water system. ⁽¹⁾ (The figure will be shown on page 80).

$$A = \frac{Q}{U \cdot \Delta T_{ml}}$$

Where

$$\Delta T_{ml} = \frac{(T_w - T_2) - (T_w - T_1)}{\ln\left(\frac{T_w - T_2}{T_w - T_1}\right)}$$

Finally, with the latent heat at 170 °C and 8 bar (steam condition), the steam flow is defined as:

$$w_{stream} = \frac{Q}{\lambda}$$

Therefore, the jacketed characteristics are:

Area of contact [m²]	8.6
Q [J/h]	2.3 · 10 ⁹
w_{steam} [kg/h]	1.1 · 10 ³

Table 23. Jacketed characteristics.

Settler S-01 and S-02

For knowing the settler dimensions, the same procedure as for the vessels will be followed. If the total mass in the equipments are known and its density, the volume is fixed. Supposing that the volumens are additives, the volumetric maximum content of the vessel will be the following:

Magnitude	S-01	S-02
Mass [kg]	27152	22024
Volume [m ³]	30.9	25.1

Table 24. Contained of both settlers.

By heuristics its known that the height should be 1.5 of the diameter. Consequently, the specifications of S-01 and S-02 are:

	S-01	S-02
Diameter [m]	3	3
Section [m²]	7.07	7.07
Height ([m]	4.5	4.5
Capacity [m³]	32	32

Table 25. Specifications of both settlers.

The same as vessels, the concavity at the ends of the decanter, will provide the volume of safety to be added to the liquid's volume. Usually, this safety volume is around 15 % - 20 %.

In each phase, the mass is known, consequently the volume in each phase is known too. This variable is related to height of a half cylinder by the followin expression:

$$V = L \left[\frac{\pi R^2}{2} + (h - R)\sqrt{2 R h - h^2} + R^2 \arcsin \frac{-R + h}{R} \right]$$

Where L is the decanter's length; V is liquid's volume in each phase; R is the radius and h is each phase's height.

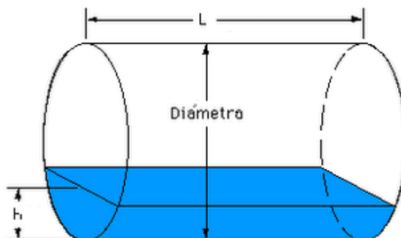


Figure 18. Settlers volume.

Therefore, the height of each phase in both settlers are defined. Down below are the results:

h [m]	S-01	S-02
Biodiesel phase	2.13	2.10
Glycerine phase	0.61	0.19
Total	2.74	2.29

Table 26. Settlers height.

Heat Exchanger

It has been commented in the heat exchanger section, the speed and tubes number has been imposed: 60 tubes and 1 meter per second the speed. For dimensioning the heat exchanger it is necessary to know the overall heat-transfer coefficient in tubular heat exchangers. For these kind of fluids (organic solvents and water) the typical overall heat-transfer coefficient is between 50 – 150 BTU/(°F·ft²·h). ⁽¹¹⁾ It has been imposed the amount will be 100 BTU/(°F·ft²·h), in units of international system (SI) is equivalent to 567.83 W/(m²·°C). ⁽¹¹⁾

TABLE 11-3 Typical Overall Heat-Transfer Coefficients in Tubular Heat Exchangers
 $U = \text{Btu}/(^{\circ}\text{F} \cdot \text{ft}^2 \cdot \text{h})$

Shell side	Tube side	Design U	Includes total dirt	Shell side	Tube side	Design U	Includes total dirt
Liquid-liquid media							
Aroclor 1248	Jet fuels	100-150	0.0015	Dowtherm vapor	Dowtherm liquid	80-120	.0015
Cutback asphalt	Water	10-20	.01	Gas-plant tar	Steam	40-50	.0055
Demineralized water	Water	300-500	.001	High-boiling hydrocarbons V	Water	20-50	.003
Ethanol amine (MEA or DEA) 10-25% solutions	Water or DEA, or MEA solutions	140-200	.003	Low-boiling hydrocarbons A	Water	80-200	.003
Fuel oil	Water	15-25	.007	Hydrocarbon vapors (partial condenser)	Oil	25-40	.004
Fuel oil	Oil	10-15	.008	Organic solvents A	Water	100-200	.003
Gasoline	Water	60-100	.003	Organic solvents high NC, A	Water or brine	20-60	.003
Heavy oils	Heavy oils	10-40	.004	Organic solvents low NC, V	Water or brine	50-120	.003
Heavy oils	Water	15-50	.005	Kerosene	Water	30-65	.004
Hydrogen-rich reformer stream	Hydrogen-rich reformer stream	90-120	.002	Kerosene	Oil	20-30	.005
Kerosene or gas oil	Water	25-50	.005	Naphtha	Water	50-75	.005
Kerosene or jet fuels	Oil	20-35	.005	Naphtha	Oil	20-30	.005
Jacket water	Trichlorethylene	40-50	.0015	Stabilizer reflux vapors	Water	80-120	.003
Lube oil (low viscosity)	Water	230-300	.002	Steam	Feed water	400-1000	.0005
Lube oil (high viscosity)	Water	25-50	.002	Steam	No. 6 fuel oil	15-25	.0055
Lube oil	Water	40-80	.003	Steam	No. 2 fuel oil	60-90	.0025
Naphtha	Oil	11-20	.006	Sulfur dioxide	Water	150-200	.003
Naphtha	Water	50-70	.005	Tall-oil derivatives, vegetable oils (vapor)	Water	20-50	.004
Organic solvents	Water	50-150	.003	Water	Aromatic vapor-stream azeotrope	40-80	.005
Organic solvents	Brine	35-90	.003	Gas-liquid media			
Organic solvents	Organic solvents	20-60	.002	Air, N ₂ , etc. (compressed)	Water or brine	40-80	.005
Tall oil derivatives, vegetable oil, etc.	Water	20-50	.004	Air, N ₂ , etc., A	Water or brine	10-50	.005
Water	Caustic soda solutions (10-30%)	100-250	.003	Water or brine	Air, N ₂ (compressed)	20-40	.005
Water	Water	200-250	.003	Water or brine	Air, N ₂ , etc., A	5-20	.005
Wax distillate	Water	15-25	.005	Water	Hydrogen containing natural-gas mixtures	80-125	.003
Wax distillate	Oil	15-23	.005	Vaporizers			
Condensing vapor-liquid media							
Alcohol vapor	Water	100-200	.002	Anhydrous ammonia	Steam condensing	150-300	.0015
Asphalt (450°F)	Dowtherm vapor	40-60	.006	Chlorine	Water condensing	150-300	.0015
Dowtherm vapor	Tall oil and derivatives	60-80	.004	Chlorine	Light heat-transfer oil	40-60	.0015
				Propane, butane, etc.	Steam condensing	200-300	.0015
				Water	Steam condensing	250-400	.0015

NC = noncondensable gas present.
 V = vacuum.
 A = atmospheric pressure.
 Dirt (or fouling factor) units are (h · ft² · °F)/Btu.
 To convert British thermal units per hour-square foot-degrees Fahrenheit to joules per square meter-second-kelvins, multiply by 5.6783; to convert hours per square foot-degree Fahrenheit-British thermal units to square meters per second-kelvin-joules, multiply by 0.1761.

Figure 19. Typical overall heat-transfer coefficient in tubular heat exchangers.

1. The necessary heat exchange is calculated:

$$Q = w \cdot C_p \cdot (T_2 - T_1)$$

$$Q = 41026 \left[\frac{kg}{h} \right] \cdot 1786 \left[\frac{J}{kg \cdot K} \right] \cdot (75 - 20)[K] = 4.03 \cdot 10^9 \left[\frac{J}{h} \right]$$

2. The necessary area of exchanged is calculated:

$$\Delta T_1 = T_s - t_e = 170 - 20 = 150 \text{ } ^\circ\text{C}$$

$$\Delta T_2 = T_e - t_s = 170 - 75 = 95 \text{ } ^\circ\text{C}$$

$$\Delta T_{ml} = \frac{\Delta T_2 - \Delta T_1}{\ln \left(\frac{\Delta T_2}{\Delta T_1} \right)} = \frac{95 - 150}{\ln \left(\frac{95}{150} \right)} = 120.41 \text{ } [^\circ\text{C}]$$

$$A = \frac{Q}{U \cdot \Delta T_{ml}} \quad ; \quad A = \frac{4.03 \cdot 10^9 \left[\frac{J}{h} \right]}{567.83 \left[\frac{W}{m^2 \cdot K} \right] \cdot 120.41 [K]} \quad ; \quad A = 14.60 \text{ } m^2$$

3. The total mass per tube is:

$$w = \frac{w_{total}}{N_T} = 684 \text{ } kg \text{ per tube}$$

4. Below, it can be found the internal diameter:

$$S = \frac{w}{\rho_{triiolein} \cdot v}$$

$$S = \frac{41026 \left[\frac{kg}{h} \right]}{791.8 \left[\frac{kg}{m^3} \right] \cdot 1 \left[\frac{m}{s} \right]} = 2.4 \cdot 10^{-4} m^2$$

$$d_{int} = \sqrt{\frac{4 \cdot S}{\pi}} = 1.75 \cdot 10^{-2}$$

5. Thickness has been imposed, 2 mm, knowing that for stainless steel tubes, thickness has to be less than 3 mm. ⁽¹²⁾

$$d_{ext} = d_{int} + 2 \cdot e = 1.75 \cdot 10^{-2} + 2 \cdot 0.002 = 2.15 \cdot 10^{-2} m$$

6. Once the internal diameter of the tube is known, the length of the tube is defined as:

$$L = \frac{A}{N_T \cdot \frac{d_{int}}{2} \cdot \pi} = 7.0 m$$

7. Thus, the characteristics of the tubes are fixed. So now it has to know the characteristics of the tube bundle. In this section is necessary to apply the heuristic commented in Heat exchanged section. The triangular tube layouts and square tube layouts must be separated 1.25 time the tube outside diameter.

$$S_{tube\ bundles} = \frac{\pi \cdot d_{int}^2}{4} \cdot 1.25 \cdot N_T = 2.72 \cdot 10^{-2} m^2$$

$$d_{tube\ bundles} = \sqrt{\frac{4 \cdot S_{tube\ bundles}}{\pi}} = 0.226 m$$

8. Once the dimensions of the heat exchanger are known, in a pipes catalog the pipes dimensions are chosen and all the magnitudes are recalculated again, doing the reverse process. The following table shows the recalculation and final dimensions of the heat exchanger.

CONDITIONS	
Exchange area [m²]	16.40
Heat exchange [W]	1.12·10 ⁶
Tubes number	60
v [m/s]	1.00
TUBES	
External diameter [mm]	21.34
Thickness [mm]	2.11
Internal diameter [mm]	17.12
Length [m]	7.00
TUBE BUNDLE	
External diameter [mm]	224.8

Table 27. Characteristics of shell-and-tube exchanger.

Piping

Initially a speed has been imposed to know the pipes dimensions, knowing that the fluid velocity should be between 1 and 2 m/s. The lowest has been chosen. Knowing the mass flowing through each pipe, the speed and the density, the section has been calculated by:

$$S = \frac{w}{\rho \cdot v}$$

Secondly, the internal diameter is set by:

$$d_{int} = \sqrt{\frac{4 \cdot S}{\pi}}$$

Once the internal diameter is fixed, it can be determined the thickness. But it is important to know the maximum pressure that the pipe can withstand. Typically, the pipes are designed to withstand a pressure of 1.5 times the working pressure, it is a heuristic.

Depending on the material type of the pipe, the thickness will be one or other due to it depends on the yield strength. The yield strength depends on the temperature, for that reason it has been extrapolated at the desired temperature. It has been decided that as the temperature variations are not high as well as the yield strength variations, the yield strength amount is $1.52 \cdot 10^5$ Pa for AISI 304.

Thus, once the maximum pressure and yield strength are known, thickness is fixed as:

$$e (m) = \frac{P_{max} (Pa) \cdot d_{int} (m)}{\sigma_{LE} (Pa)}$$

Hence, external diameter is: $d_{ext} = d_{int} + 2 \cdot e$

Following the last recipe, hereunder is shown the pipe's characteristics.

	104^a	105^b	117^c
S (m²)	$1,56 \cdot 10^{-2}$	$2,24 \cdot 10^{-3}$	$2,32 \cdot 10^{-1}$
d_{int} (mm)	141,14	53,39	0,54
e (mm)	$1,12 \cdot 10^{-4}$	$4,23 \cdot 10^{-5}$	$1,36 \cdot 10^{-5}$
d_{ext} (mm)	0,141	0,053	0,017

Table 28. First pipe dimensioning.

- a. Pipes number: 104, 107, 110, 111, 114 and 115 have the same dimensions.
- b. Pipes number: 105, 106, 108, 109, 112, 113 and 116 have the same dimensions.
- c. Pipe number 117 has these dimensions.

Subsequently, through a pipeline catalogue, piping's have been selected according to the calculated dimensions. Therefore, next step is to recalculate the speed, with the expression that relates section with speed, to know if it is between commented interval. Calculations are shown below:

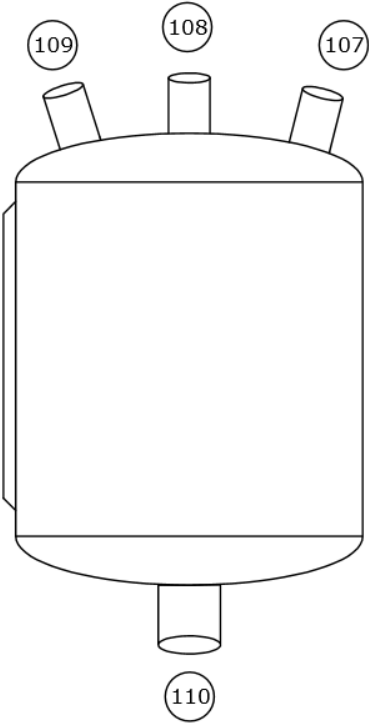
	104	105	117
dext (m)	114.3	48.26	21.36
e (m)	3.05	2.77	2.11
dint (m)	108.20	42.72	17.14
v (m/s)	1.70	1.56	2.00

Table 29. Pipe dimensioning.

As the speed is between the range, the selected pipes dimensions are correct.

APPENDIX 3: SPECIFICATION SHEETS

PROJECT: Study of a biodiesel batch production from DSO		NUMBER OF EQUIPMENT: V-01	
		FUNCTION: Reactor	
Functioning of the unit			
Feed [kg]	27152		
Temperature of operation [°C]	60		
Pressure of operation [bar]	1.013		
Design specifications			
Volume [m³]	31.5		
Wall thickness [mm]	*		
Diameter 107 [mm]	114.30		
Diameter 108mm]	48.26		
Diameter 109 [mm]	48.26		
Diameter 110 [mm]	114.30		
Time of occupation [h]	2		
Painting	-		
Number of legs	*		
Supports	*		
Cylinder			
Height [m]	4.5		
Diameter [m]	3.0		
Materials			
Tank	Steel AISI 304		
Flanges	*		
Tubes	*		
Screws	*		
Joints	*		
Inputs and outputs			
Number	Function		
107, 108, 109	Input		
110	Output		



* To be specified by the manufacturer

Table 30. V-01 specifications sheet.

PROJECT: Study of a biodiesel batch production from DSO		NUMBER OF EQUIPMENT: V-02	
		FUNCTION: Reactor	
Functioning of the unit		<p>The diagram shows a vertical cylindrical reactor vessel. At the top, there are three ports labeled 111, 112, and 113. At the bottom, there is a single port labeled 114. The vessel has a rounded top and bottom. A vertical line on the left side indicates a support or leg.</p>	
Feed [kg]	22024		
Temperature of operation [°C]	60		
Pressure of operation [bar]	1.013		
Design specifications			
Volume [m ³]	25.5		
Wall thickness [mm]	*		
Diameter 111 [mm]	114.30		
Diameter 112 [mm]	48.26		
Diameter 113 [mm]	48.26		
Diameter 114 [mm]	114.30		
Time of occupation [h]	2		
Painting	-		
Number of legs	*		
Supports	*		
Cylinder			
Height [m]	4.5		
Diameter [m]	3.0		
Materials			
Tank	Steel AISI 304		
Flanges	*		
Tubes	*		
Screws	*		
Joints	*		
Inputs and outputs			
Number	Function		
111, 112, 113	Input		
114	Output		

* To be specified by the manufacturer

Table 31. V-02 specifications sheet.

PROJECT: Study of a biodiesel batch production from DSO		NUMBER OF EQUIPMENT: S-01	
		FUNCTION: Settler	
Functioning of the unit			
Feed [kg]	27152		
Temperature of operation [°C]	50		
Pressure of operation [bar]	1.013		
Design specifications			
Volume [m³]	31.5		
Wall thickness [mm]	*		
Diameter 110 [mm]	114.30		
Diameter 111 [mm]	114.30		
Diameter 116 [mm]	48.26		
Time of occupation [h]	3		
Painting	-		
Number of legs	4		
Supports	*		
Cylinder			
Height [m]	4.5		
Diameter [m]	3.0		
Materials			
Tank	Steel AISI 304		
Flanges	*		
Tubes	*		
Screws	*		
Joints	*		
Inputs and outputs			
Number	Function		
110	Input		
111, 116	Output		
		* To be specified by the manufacturer	

Table 32. S-01 specifications sheet.

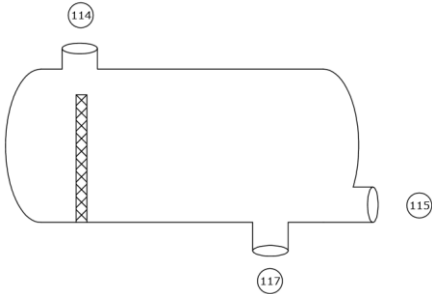
PROJECT: Study of a biodiesel batch production from DSO		NUMBER OF EQUIPMENT: S-02	
		FUNCTION: Settler	
Functioning of the unit			
Feed [kg]	22024		
Temperature of operation [°C]	50		
Pressure of operation [bar]	1.013		
Design specifications			
Volume [m³]	25.5		
Wall thickness [mm]	*		
Diameter 114 [mm]	114.30		
Diameter 115 [mm]	114.30		
Diameter 117 [mm]	21.36		
Time of occupation [h]	3		
Painting	-		
Number of legs	4		
Supports	*		
Cylinder			
Height [m]	4.5		
Diameter [m]	3.0		
Materials			
Tank	Steel AISI 304		
Flanges	*		
Tubes	*		
Screws	*		
Joints	*		
Inputs and outputs			
Number	Function		
114	Input		
115, 117	Output		
		* To be specified by the manufacturer	

Table 33. S-02 specifications sheet.

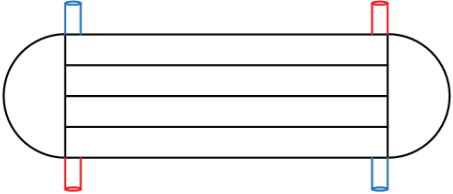
PROJECT		Study of a biodiesel batch production from DSO			
NUMBER OF EQUIPMENT		E-01			
SERVICE		Increasing temperature			
TYPE	X	HORIZONTAL		VERTICAL	
Functioning of the unit					
		TUBES SIDE		SHELL SIDE	
Circulating fluid		Degummed soybean oil		Water steam	
		INPUT	OUTPUT	INPUT	OUTPUT
Steam [kg/h]		-	-	$2.64 \cdot 10^4$	-
Condensed fluid [kg/h]		Water Steam			
Molecular weight [kg/kmol]		18			
Latent heat soybean oil [kJ/kg]		153			
Temperature input [°C]		20			
Temperature output [°C]		75			
Pressure [bar]		1.013			
Exchanged heat [kW]		1120			
Global coefficient of heat transfer [W/(m²·K)]		568			
Exchange area [m²]		16.40			
SPECIFICATIONS					
		TUBES SIDE		SHELL SIDE	
Pressure [bar]		1.013		1.013	
Temperature [°C]		75		75	
Lenght [m]		7.00		*	
External diameter [m]		0.021		0.22	
Number os passes		1		1	
Number of tubes by pass		60			
Pass		TRIANGULAR			
Tubular bundle		X	FIXED	FLOATING	
 <p>— Soybean oil — Water steam</p>					
MATERIALS					
SHELL		Steel AISI 304			
TUBES		Steel AISI 304			

Table 34. E-01 specifications sheet.

